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Studies of structural properties of char from intumescent coating

Prepared by

Mohamed Azeem Bin Othman

16369

With a supervision from

Assoc Prof Dr Faiz Ahmad

Preliminary report submitted in partial fulfillment of the

requirement for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

STUDIES OF STRUCTURAL PROPERTIES OF CHAR FROM INTUMESCENT COATING

By

MOHAMED AZEEM BIN OTHMAN
16369

A project dissertation submitted to the

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Approved by,

……………………..........
(Assoc. Prof.Dr.Faiz Ahmad)

UNIVERSITY TECHNOLOGY PETRONAS
TRONOH, PERAK
January 2016.

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgement and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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(MOHAMED AZEEM BIN OTHMAN)

ABSTRACT

Intumescent coating system cannot insulate and protect the material effectively if it's char weak in mechanical strength. The purpose of this research was to study the effects of additional micro filler which is alumina (Al_2O_3) as reinforcement to epoxy based coating's char strength. Different wt. (%) of formulation was developed in the experiment. The coated sample was put in the furnace test machine and the test was conducted at 500ºC at 1 hour period in order to determine the physical property of the char. The microstructures of the char were examined through Scanning Electron Microscopy Analysis (SEM). The residual weight and degradation temperature of the chars were determined by Thermo Gravimetric Analysis (TGA). The thermal performance of the coatings were tested through fire testing process (Bunsen Fire Test).In this test, the coated sample of 10cm x 10cm were burned to high temperature and backside temperature of the substrates were recorded using a Data Logger. For mechanical testing (compression test), the chars' strength were determined by examined the ability of the char to resist deformation. In the compression test, mass from 100g to 3600g was continuously added on top of the char and the height of the char was recorded while leaving the load for one minute.

ACKNOWLEDGEMENT

The author for this report is from department of mechanical engineering from University Technology Petronas (UTP). The purpose of this report is to investigate the structural properties of char from intumescent coating.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND STUDY

In the last few decades, dire accidents caused by the fire towards a steel structure increase the awareness about the instability effect of a fire. The steel's structural properties will yield at high temperature due to unsteady microstructure. According to prugarinc website (2015), excessive heat towards a steel structure would decrease the modulus elasticity and the yield strength. This would lead to extreme sagging in a beam of a steel structure. Thermal expansion could occur and eventually damage the welded connection attached on steel. Flame retardant coating is a practical method to protect the steel structure from this risk. Generally, flame retardant coating is divided into two kinds which are intumescent fire retardant coating and non-intumescent fire retardant coating. Intumescent fire retardant coating (IFRC) is a strategy which involves the formation on heating of a swollen of rigid char to isolate steel structure from the flaming process (Camino and Delobel, 2000).This system is used to reduce the transferred and spreading of heat from the fire in order to maintain the purity of the steel's structure.

Intumescent system consists of inorganic acid, carbon source and blowing agent (Zhenyu Wang and Wei Ke, 2006). During thermal degradation, a chemical reaction takes place between these additives and lead to formation of hard char to protect the substrate from heat expansion. This char will act as protective barrier for the steel structure. In this case of study, a different formulation of coating is developed to analyze the production of char structure. The char strength is determined from a few testing. With that, the formulation of the highest strength could be analyzed certainly for further performance improvement opportunity.

1.2 Problem Statement

In intumescent system, the binder of mixture usually is made up from organic binders which have a better char structure and expanding effect. However, organic binders usually release harmful substances like gas and smoke which is malicious if the mixture is heated with high temperature. Nowadays, inorganic binder is used to solve this issue due to low gas emission and smoke during heating process. Inorganic binders ease the combination to more permeable component of the formulation during drying and setting. Besides that, it also improves the insulation properties to protect the coated substrate. Normally, Bisphenol Epoxy Resin (BPA) is used as binder in the formulation of the coating. The binder and the mixture react together to produced foamed char.

However, their fire conductivity and anti-oxidation is weak at elevated condition and eventually will cause the specimen unprotected. Besides that, poor bonding between between char and the substrate grant to weak intumescent coating in protecting substrate from fire. Therefore, modification of the formulation is necessary to significantly maximize the coating performance with subsequently efficiencies. The formulation is modified using additional fillers such as titanium dioxide, alumina, etc. Through this formulation, a better mechanical property of char is achieved. In this case study, alumina is used as the filler for the experiment. J. Green (1996) expounded that alumina possess great mechanical properties at room temperature and high melting point which is 2050˚C. Thus, the microstructure of the alumina is not easily destroyed and this will improve the heat shielding ability of the char.

1.3 OBJECTIVES

Based on the problem stated for intumescent fire retardant coating, the following objectives are considered,

1) To develop a formulation for the intumescent coating
2) To investigate the char formation from the intumescent coating
3) To determine the effect of nano filler which is alumina on char strength and heat shielding effect.

1.4 SCOPE OF STUDY

This project is mainly focus on the different usage of filler affected the char strength of the intumescent coating. The filler used in the project are mainly alumina (Al_2O_3). Different (wt.%) of the filler was used in the formulation for this project. The char produced was analyzed using different mechanical weight in order to test its mechanical strength. The coating then will be characterized to determine the formulation that gives the best fire protection performance. Besides that, heat shielding effect is been investigate in this project to determine which formulation give better result.

CHAPTER 2

LITERATURE REVIEW

2.1 INTUMESCENT FIRE COATING

In recent years, numerous issues had been raised in the industry regarding fire protection for the materials. An intumescent fire coating system is a very practical method to protect materials against fire since it is very economical and easy to manufacture. Intumescent is meant by the swelling of a substance when it is exposed to thermal behavior. Intumescent coating is a highly effective passive fire protection and fire retardant coating, by providing a char as a shielding to steel substrate. This system does not hold flaming process and enlarge when heated and produce a thick insulating char to preserve the substrate from the heat and sustained the endurance of steel structure for longer period (Triantafyllidis and Bisby, 2014). The charred layer of the coatings will prevent heat from diffuse into the substrate and thus preventing flames to spread.

Intumescent coating normally consists of acid, blowing agent and a carbon source .This three source are link together with a binder. When the source is exposed to thermal behavior, the blowing agent will cause the carbon source to solidify in a form of cellular foam. According to (Ebdon and Joseph, 2001), the higher the amount of char produced, the higher the flame retardancy of the material. Most of the acid source used for the intumescent coating is ammonium polyphosphate (APP) .This is a vital source as it increased the speed of char formation and improved the char swelling. Because of its char swelling properties, APP commonly use as both blowing agent and acid source. The ammonium gas release for APP could increase swelling process of the coating. Carbon source is the material that produced char in the intumescent system. The carbon that usually used is the methynol melamine. Other materials such as starch, mannitol, dimer, trimer, monomer are also used as carbon source. Blowing agent is a source that expands the char during heating. The blowing agent is selected to react with the carbonific and phosphorous containing material to form water and phosphorous which functioas fire extinguishers in the coating layer between the fire and substrates. Typical blowing agents are urea and melamine (Wilkie and Morgan, 2010).

2.2 CHAR FORMATION

Char formation in intumescent system is vital to reduce flammability and char can protect substrate from fire action. The formation of char contained 90% of carbon, 3 % of hydrogen and gasification and polymer cross-linking. The distinction of the char in intumescent system can be illustrated in figure 1. A frozen bubble gas produced from the intumescent coating will diffuse into the polymer melt which then solidified the structure of the polymer. This will prevent the fuel mix with the flame and to keep the thermal gradient at sufficient level in order to protect the polymer that unaffected by the flame. On the contrary, a poor char does not contained unclosed cell to prevent polymer melt and gaseous decomposition products from escaping (Wicks, Jones, and Pappas, 1992). This will make the layer of the polymer easily exposed to the flame.

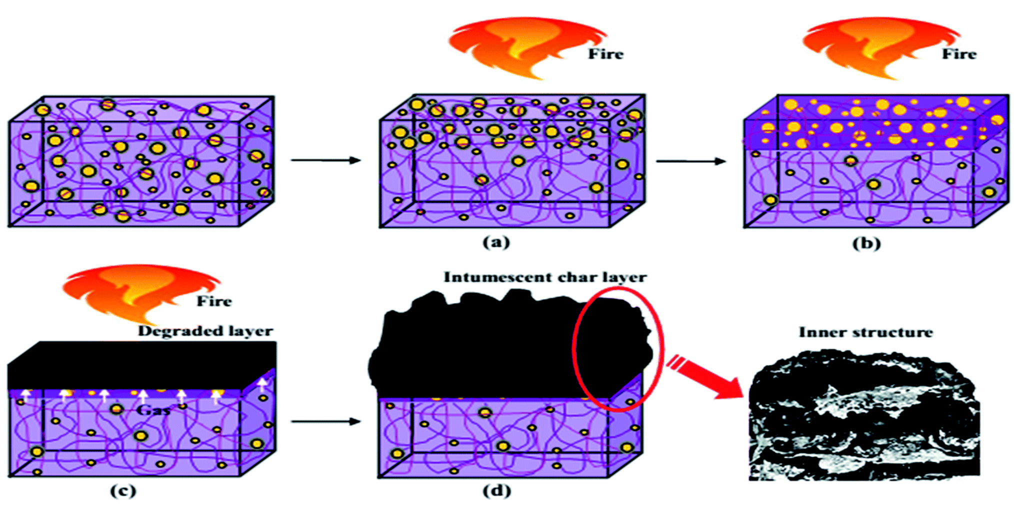


Figure 1: Ideal char formation

During the degradation stage, char is formed and it contained chain of carbon along the way to graphitization. Fire resistance of intumescent fire retardant coating leans on the char formation proposed by the reaction of APP, pentaerythritol (PER) and melamine. The layer of insulated char will decrease the heat produced by the flame from 94 000 calories per gram mol of carbon dioxide to 26 400 calories per gram mol of carbon monoxide to zero for carbon maintained in the stable composition (Hilado, 1990).

2.3 FILLERS IN INTUMESCENT COATING

Fillers is defined as a substance or material added to compounds to decrease usage of expensive binder and to improve the mechanical properties of the compounds. In this system, adding a certain amount of filler can enhance the flame retardant. Besides that, addition of filler will decreased the amount of smoke produced during heating process and lowering the time to retard flame. Filler can be classified in two types which are the inert filler and active filler and they are distinguished by their mode of action (Horrocks and Price, 2001). Inert filler basically reduced the flammability and smoke produced by a few mechanisms. Inert filler reduced smoke by diluting the combustible substrate and absorb heat to decrease the combustibility reaction occur onto substrate. Typical inert filler used intumescent system are silica, calcium carbonate, pumice, talc, calcium sulphate.

Unlike inert filler, active filler consist of special properties which make the filler more efficient at lowering the risk of flammability. Active filler can be decomposed at certain temperature by endothermic process. Endothermic process will absorb the heat during the heating process and slows down the decomposition the rate of substrate (Karbhari, 2007). When active filler decomposed, it will release passive gas such as water vapour and carbon dioxide which then diffused into the fire and reduce the heat produced. Active filler that commonly used are aluminum trihydroxide, $Al(OH)_3$ and molybdenum, (MoO_3)

2.3.1 Alumina (Al_2O_3)

Alumina is compound that significantly used as filler in intumescent coating. This is due to its high melting point and hardness properties which is very suitable as fire retardant filler. The alumina adds mechanical strength and has high resistance to corrosion and wear. In this application, alumina will decomposed through endothermic process by absorbing heat and released water vapor to decrease rate of combustion. Besides that, alumina also will decrease the smoke released during combustion and act as a smoke concealed.

CHAPTER 3

METHODOLOGY

3.1 PROJECT FLOW CHART

At first, previous research papers will be studied in order to have better understanding about the research. The author needs to search for the past research regarding the project through internet. After deep research regarding the project, the material for the formulation is selected after having a discussion with research officer. Coating formulation will be conducted by varying the (wt.%) of filler that need to be used in the formulation. After coated onto substrate, the sample will be dried at least for one day. When the sample is dry, furnace test will be conducted in order to heat the coated sample. Char produced will be prepared for mechanical testing to examine the mechanical properties of the char produced. Finally, the data will be collected based on the mechanical testing conducted and will be further analyzed with other sample.

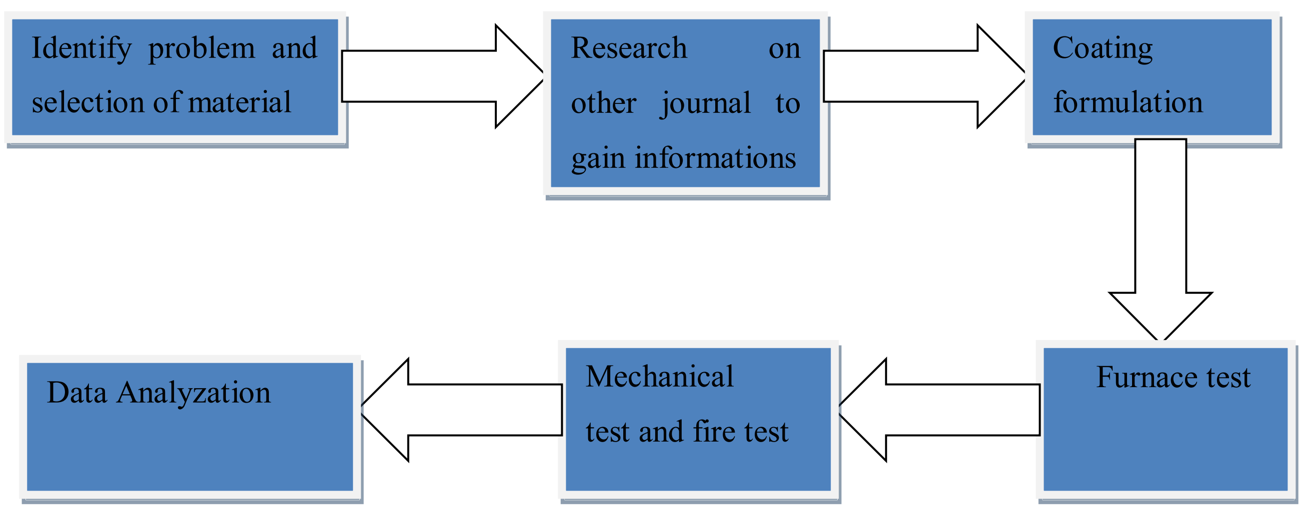


Figure 2: Project flow chart

3.2 PROJECT GANTT CHART AND KEY MILESTONES

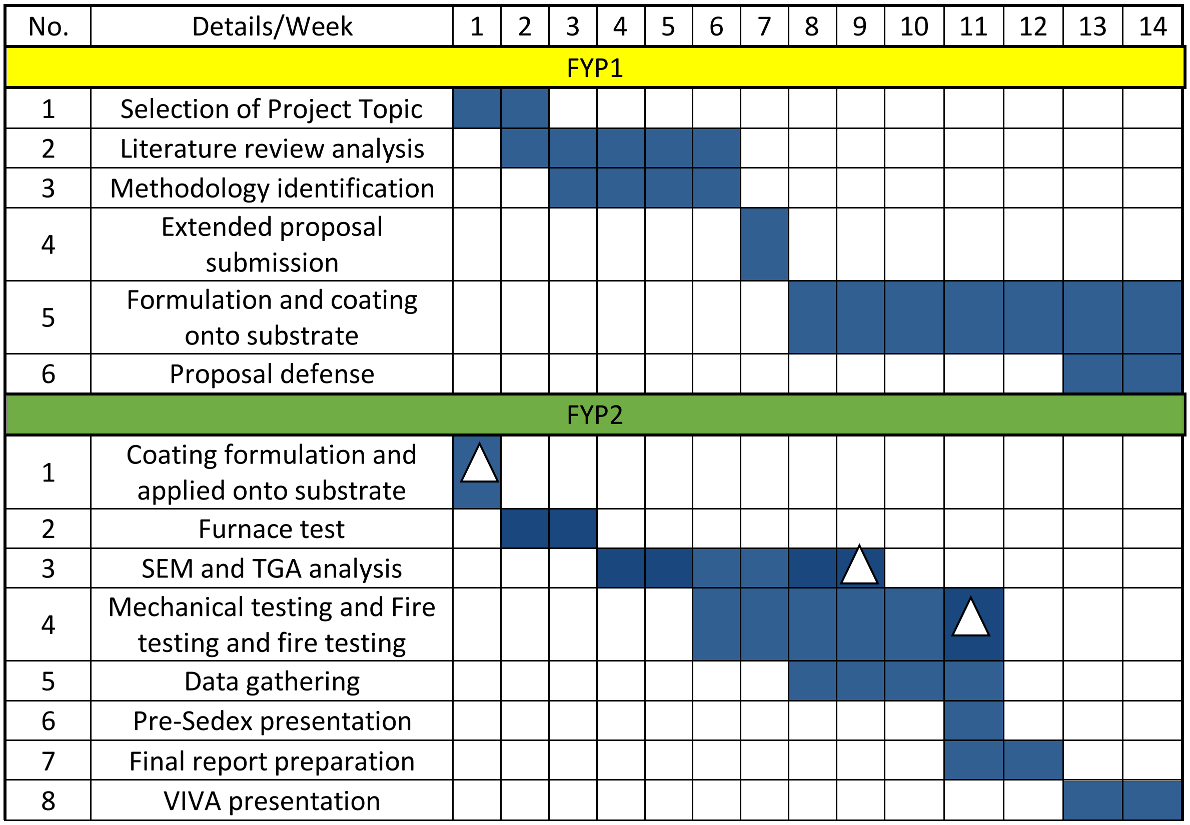


Table 1: Gantt chart and key milestones

△ = **Key milestones**

1) Completion of all coating and applied onto substrate

2) Completion of SEM and TGA analysis

3) Completion of investigation of mechanical properties of char

3.3 MATERIALS SELECTION FOR INTUMESCENT COATING

1) **Ammonium polyphosphate (APP)** – Inorganic salt that contain polyphosphoric acid and ammonia chains. The physical property is colourless, and incombustible. In the intumescent system, the role of APP as an acid source which then reacts with the carbon source to form ester.

2) **Melamine (MEL)** – An organic base compound that contain abundant of nitrogen molecule. It will degrade to commute gaseous such as ammonia gas and nitrogen gas when scorched. Thus, it will be used as a gas source in intumescent coating system.

3) **Expandable graphite (EG) –** Expandable graphite is a composition of graphite that enlarged when exposed to flame. The thickness of graphite is 100 times larger of its original thickness retaining the superior heat resistance properties of graphite. These properties are vital in increasing the efficiency of the intumescent coating system. The role of EG acts as both blowing agent and carbon source in the intumescent coating formulation.

4) **Boric acid (BA) –** Boric acid is an occurring material containing boron, oxygen and hydrogen. It's crystalline structure are white and inodorous. Boric acid can impede the commute of flammable gaseous and releases chemically bonded water which can reduce the combustion rate in coating system.

5) **Zinc Borate (ZB) –** Zinc borate is an inorganic compound that is very discrete in size which makes it dissipate easily. It is used in fire retardant system to abolish smoke and produce char at elevated temperature that can prevent flame from spreading. It also releases water from hydration when burned in order decrease combustion.

6) **Epoxy resin and hardener** – In this project, epoxy resin Bisphenol A BE-188 (BPA) is used as the binding agent and ACR Hardener H-2310 polyamide amine as the curing agent. The role of the binder is to bind fire retardant additives and to provide adhesiveness to the coated substrate.

7) **Alumina (Al_2O_3) -** Alumina is used in the experiment as an additional filler for the coating in order to increase the mechanical strength of the coating. Alumina possessed high mechanical properties at elevated temperature which is 2050˚C. Alumina can be used to provide passivation on a coated substrate to prevent from corrosion.

3.5 Equipment and tools

i. Grinder

- Grinder machine is used to grind all the colourless substance simultaneously. The grinding process took about 10 minutes to crush all the mixture.

Figure 3: Grinder for grinding the formulation

ii. Mixer

- The mixer machine is used to mix the colourless mixture with EG and epoxy. The machine is set with 50rpm rotational speed. The mixing process is carried out approximately 25 minutes.

Figure 4: Shear Mixer for making a coating

3.5.1 Testing Procedure

Scanning Electron Microscopy Analysis (SEM)

- SEM is a method of analysis to examine the microstructures and the material compositions in the samples.

Procedure:

1. The char samples about 5mg from the coating formulation is to be taken.
2. The samples were examined using the SEM machine located at block 17.

Thermo Gravimetric Analysis (TGA)

- TGA is a method of thermal analysis to examine the changes in the weight of the sample when the sample is heated.

Procedure:

1. The sample weighted about 10 mg from the coating formulation is to be taken.
2. The temperature of the test is to be set at range 50°C to 900°C.
3. The starting temperature is to be set at 20°C min^{-1} under gas environment.

Furnace Test

- Furnace test method to investigate the fire behavior toward the coated sample. The char will produce from this test due to high temperature from the furnace

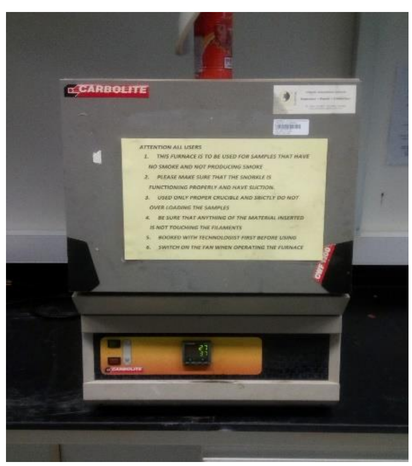


Figure 5: Furnace test machine.

Procedure:

1. The sample is to be placed in the furnace.
2. The temperature is to be set at 500°C and dwell for 1 hour period.
3. The temperature is then to be decreased at room temperature for 30 minutes.
4. The thickness of each burned sample is to be taken after the furnace test for further analysis.

Hardness test (Compression test)

- The test is to determine the char strength produced by the furnace test. The char strength is determined by the ability of the char to endure deformation at certain loads.

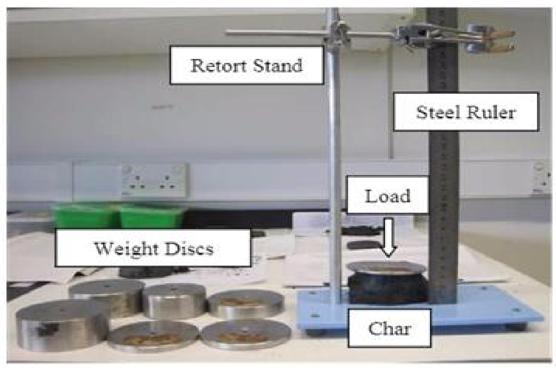


Figure 6: Set up of hardness test

Procedure:

1. The experiment is set up as the above.
2. The load disk is to be prepared which are 3 disc weighted of 1000 g, 1 disc weighted of 500 g, 1 disc weighted of 200 g and 1 disc weighted of 100 g.
3. The loads are to be continuously added on top of the char.
4. The height of the char is to be recorded while leaving the loads on top of the char for one minute.

Fire Test (Bunsen Fire Test)

- Fire test is a method to investigate the shielding effect of each of the coating formulations towards fire.

Figure 7: Set up of fire test

1. The experiment was set up as the above.
2. Thermocouples are attach at the backside of each substrate as shown
3. When the substrate is exposed to fire, the temperature are to be recorded using a Data Logger
4. The temperature is to be recorded for interval of one minute until the temperature become constant.

3.6 PROJECT ACTIVITY

3.6.1 Preparation of the formulation

The procedure began with mixture of colourless powder which is APP, MEL, ZB and BA and fillers (alumina) is grind calmly using a grinder machine. Then, the mixture powder is mixed with EG and epoxy with a shear mixer machine for 25 minute. After that, the mixture is mixed with hardener which is polyamide amine using shear mixer for another 10 minutes. The coating formulation will be applied onto the steel substrate in two different area of sample which is $25cm^2$ and $100cm^2$ using a brush. The experimental procedure can be illustrated as shown below.

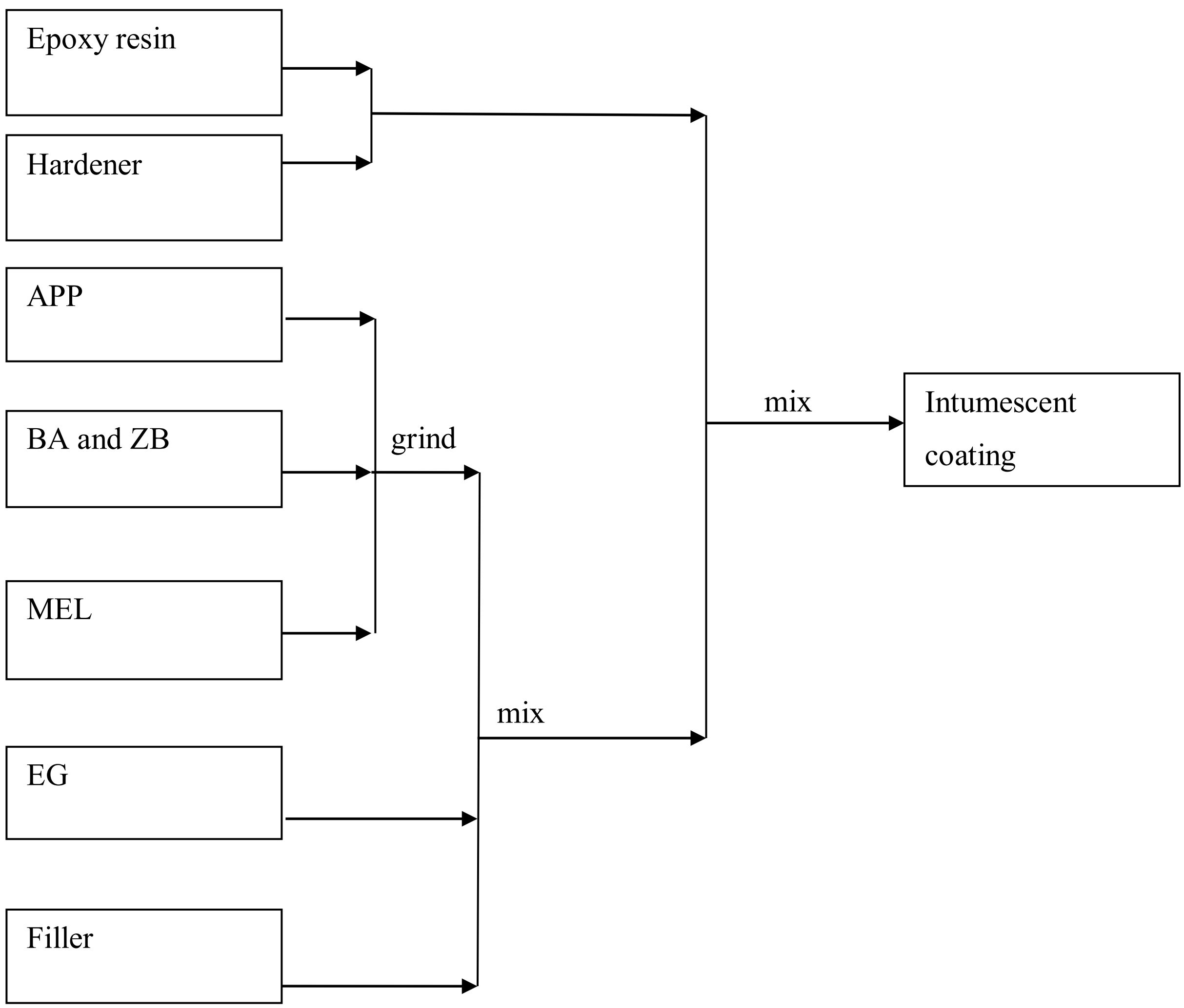


Figure 8: Preparation of intumescent coating

3.6.2 Experimental formulation

The formulation used in the experiment is different to be applied onto substrate. The overall weight percentage is 100% for all samples but different weight percentage is used for the usage of polyamide amine (Hardener) and epoxy resin. The differences is varies with the weight percentage of Alumina used in the formulation.

Component (wt. %)	**1**	**2**	**3**	**4**	**5**	**6**	**7**
APP	11.76	11.76	11.76	11.76	11.76	11.76	11.76
MEL	5.50	5.50	5.50	5.50	5.50	5.5	5.5
BA	6.11	6.11	6.11	6.11	6.11	6.11	6.11
ZB	5.0	5.0	5.0	5.0	5.0	5.0	5.0
EG	5.5	5.5	5.5	5.5	5.5	5.5	5.5
Polyamide Amine	22.22	22.121	22.055	21.989	21.890	21.73	21.56
BPA	44.44	44.242	44.11	43.978	43.78	43.45	43.12
Filler							
Alumina	0	0.3	0.5	0.7	1.0	1.5	2.0

Table 2: Formulations table

3.6.3 Sample Calculation

- Calculation for the Polyamide Amine (hardener) and Bisphenol A (binder) to be used in the formulation

Example for sample 2

Filler used in sample 2 = 0.3 gram

For bisephenol A (binder) = 0.3 x 0.66 = 0.198 gram

44.44 – 0.198 = **44.242 gram**

For Polyamide Amine (hardener) = 0.3 x 0.33= 0.099

22.22 – 0.099 = **22.121 gram**

CHAPTER 4

RESULT AND DISCUSSION

4.1 Apply Coating and Curing Process

After the formulation process was completed, the coatings were applied onto substrate gently using a brush. The samples below were process of coating that fully cured. It takes for about a 2 days to have a fully cured intumescent coating system that will be ready for further testing and experimenting. 14 formulations had been prepared in the lab and left at room temperature for curing process.

Figure 9: Coated substrate (5cm x 5cm sample)

Figure 10: Coated substrate (10 cm x 10cm sample)

The study will be using the nano filler which is alumina as the reinforcement to coating formulation. Author believes that addition of nano filler will strengthen the char strength produced thus enhance the result compared to control formulation. In order to justify the statement, the author will conduct certain testing on the coated substrate. The tests and analysis are describes as follows:

i. Thermo Gravimetric Analysis (TGA) for residual weight measurement

ii. Furnace test for char expansion analysis

iii. Char Hardness testing

iii. Fire testing

iv. Scanning Electron Microscopy (SEM) for microstructure

Besides that, the author was exposed on previous studies for some references and used the data of previous researcher's journal as a benchmark data. This is to ease the author's work in the comparison of the result gained in the experiment with the previous research data.

4.2 FURNACE TEST RESULTS

Furnace test were conducted to all of the samples. All of the samples were tested at 500°C. The test was conducted by using the model CWF 1300 of carbolite furnace. Then, the temperature was dwelled for a 1 hour time. The figure below shows the char produce after conducted the furnace test.

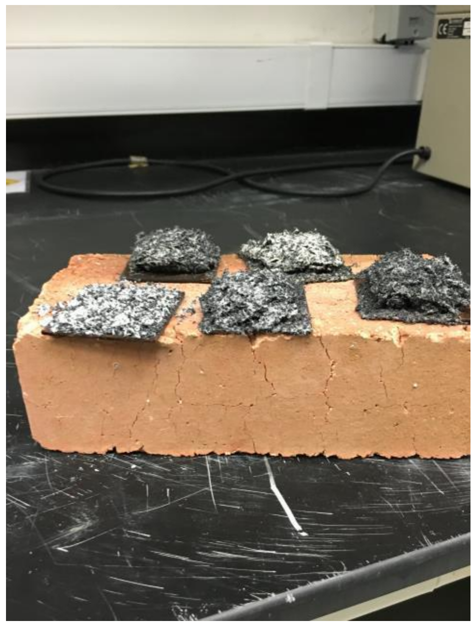


Figure 11: Coating's samples after furnace test

4.2.1 Thickness Measurement

Sample	Temperature (°C)	Thickness(mm)		Expansion (mm)
		Before	After	
Formulation control	500	1.972	6.092	4.120
Formulation of 0.3% Al		1.885	6.40	4.515
Formulation of 0.5% Al		1.991	7.208	5.217
Formulation of 0.7% Al		1.966	7.314	5.348
Formulation of 1.0% Al		1.951	7.47	5.519
Formulation of 1.5% Al		1.915	7.458	5.543
Formulation of 2.0% Al		1.916	8.00	6.084

Table 3: Intumescent coating's thickness expansion for 500℃ test

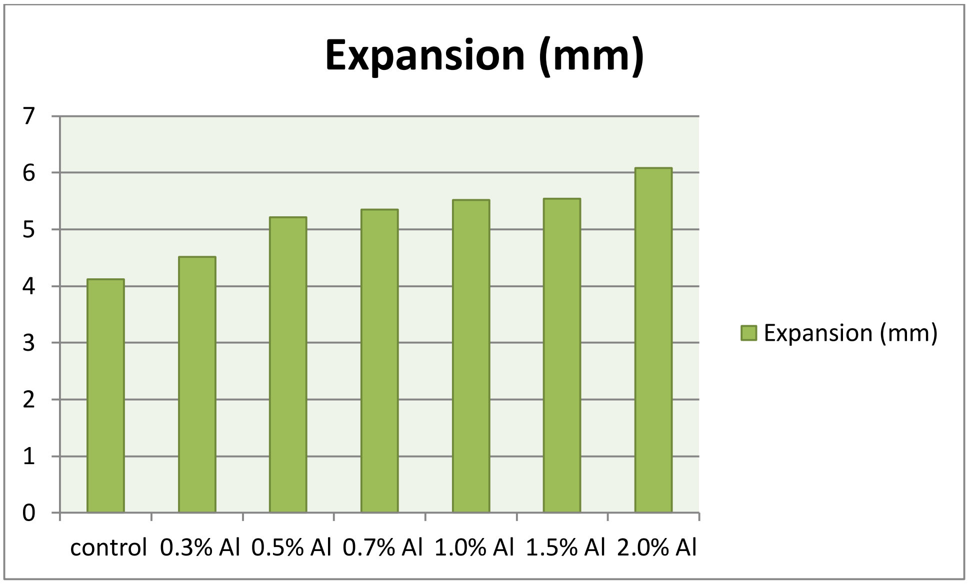


Figure 12: Expansion for Coating at 500℃

<u>Concluding remarks</u>

The intumescent coatings that have been applied on the 5cm x 5cm undergo the furnace test at 500°C. The **table 4** above shows the expansion of the char furnace test process. Based on the table above, the largest char expansion is the formulation that consists of the most alumina percentage, which is formulation 2.0% Alumina with the expansion of 6.084mm. This is followed by the coating with the formulation of 0.7% Alumina and 0.5% Alumina with the expansion of 5.934 mm and 5.509 mm respectively. The least expansion is the coating that has the least alumina which is 0.1% Alumina. The structure of the char after furnace test observed to have a fragile structure and sufficiently attached to the steel substrate.

4.2.2 Intumescent Factor

Intumescent factor determined the expansion of the coating and it is compared to the control initial thickness. The intumescent factor is defined as in the equation of:

$I = D_2 – D_0 / D_1 – D_0$

Where I = Intumescent factor

D_0 = Thickness of steel substrate

D_1= Thickness of coated substrate

D_2= Thickness of substrate after furnace test

Sample	Intumescent Factor
Formulation control	5.5
Formulation of 0.3% Al	6.8
Formulation of 0.5% Al	6.6
Formulation of 0.7% Al	7.1
Formulation of 1.0% Al	6.5
Formulation of 1.5% Al	7.1
Formulation of 2.0% Al	7.6

Table 4: Intumescent Factor

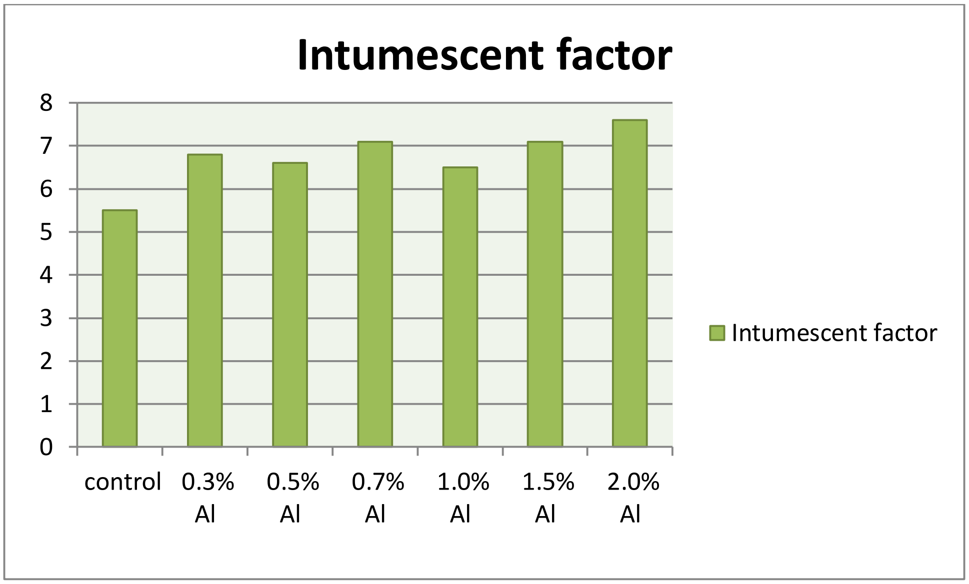


Figure 13: Intumescent factor of Alumina reinforced formulation and control formulation

Concluding remarks

From the figure above, the intumescent factor was increased by reinforcing the coating formulation with nano filler which is alumina. The maximum intumescent factor achieved by the sample was 2.0% Al. The improvement of physical structure of all sample was observed by reinforcing the formulation coating with alumina. The alumina in the coating was well dispersed for all the coating. Besides that, the intumscent factor was increased as the wt% of alumina increased. This will provide a better fire protection of intumescent coating as it provide the non-flammable gases that contribute for expansion of char. The highest value of intumescent factor with proper adhesion to substrate will prevent the penetration of flame into the substrate and spreading of flame to other area. The addition of alumina in the formulation provide greater attachment with the substrate as the char did not detached during the Bunsen test.

4.3 CHAR MORPHOLOGY OF INTUMESCENT COATING

Char is vital in the system as it heat from diffuse into the substrate and thus preventing flames to spread. Char was formed after the furnace test. This char act as a thermal behavior thus preventing the diffusion of the heat to the substrate.

The morphology of char was examined using **Scanning Electron Microscope (SEM)**. The char sample was sent to the block 17 for further analysis to examine the microstructures of the char. There were total of 4 samples has been sent for the analysis in this project. The microstructure of the char was analyzed at different magnifications which was 1000x, 5000x, and 10000x.

4.3.1 Microstructure of Control Formulation

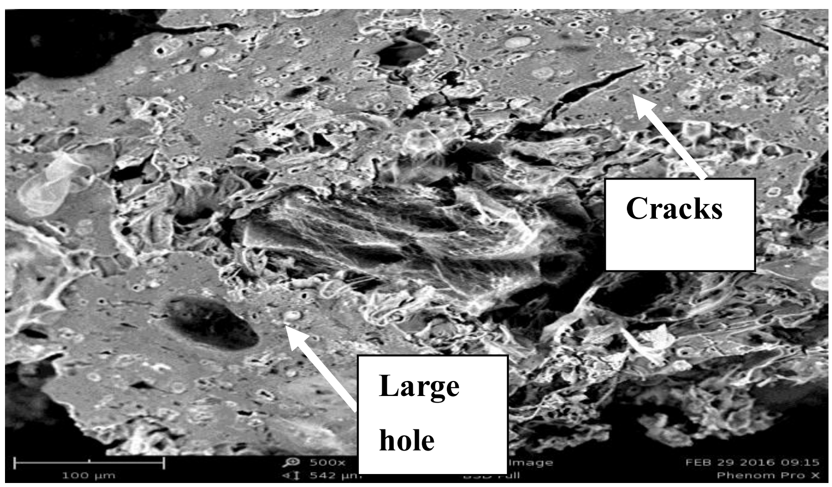


Figure 14: Microstructure of control formulation at 500x magnification

The crack was visible on the structure as shown in the figure. This crack does not provide any good thermal insulation as heat transfer process could propagate through this crack. The big void does not provide any good char expansion and thus char formed was not rigid. Unbalanced structure was shown in the figure that eased the transferring of heat from the flame inside of the substrate that will lead to the decreasing of thermal barrier performance of the coating.

Energy Dispersive Spectroscopy analysis (EDS) was carried out to examine the elements found in the char after furnace test. The graph of EDS of control formulation is shown in figure 14.

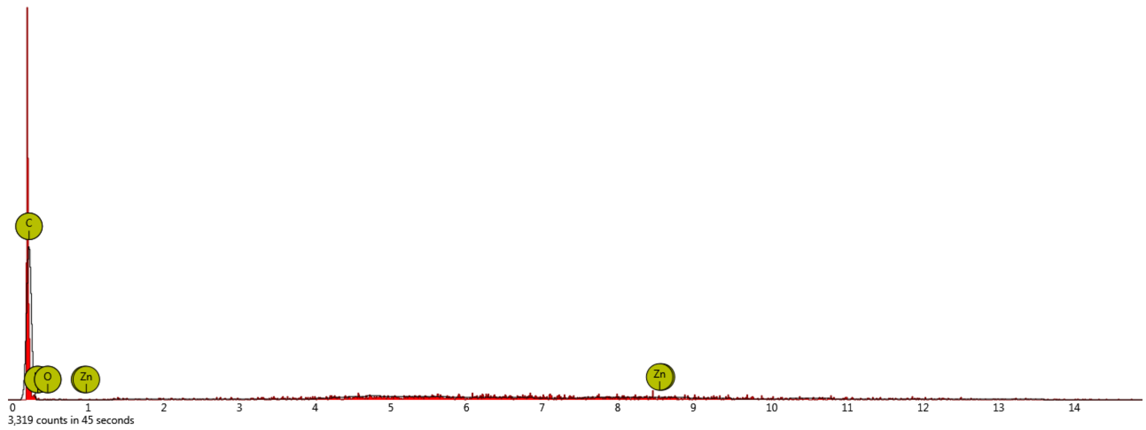


Figure 15: EDS graph of control formulation

Element symbol	Element name	Weight concentration (%)
C	**Carbon**	**40.7**
N	**Nitrogen**	**6.0**
O	**Oxygen**	**38.4**
Zn	**Zinc**	**7.85**
P	**Phosphorus**	**7.05**

Table 5: Element concentration of control formulation

4.3.2 Microstructure of 0.3% Alumina reinforced formulation

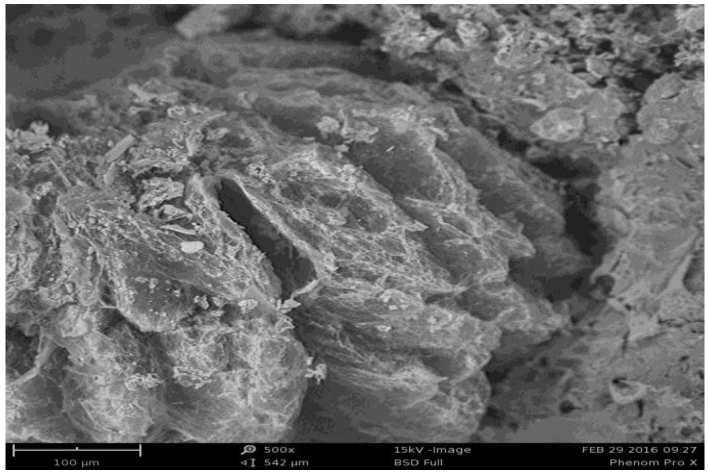


Figure 16: Microstructure of 0.3% formulation at 500x magnification

The crack was not visible on the structure as shown in the figure. It shows an improvement of the structure as filler which was Alumina was added into the formulation. Thus, the char structure was stronger than control formulation. This will provide a better intumescent effect to the structure of the char compared with the control formulation.

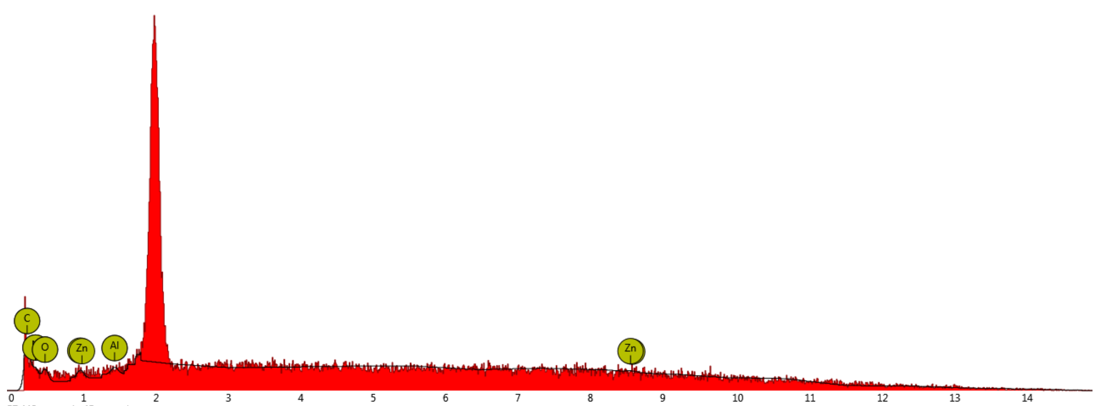


Figure 17: EDS graph of 0.3% Alumina reinforced formulation

Element symbol	Element name	Weight concentration (%)
C	Carbon	42.7
N	Nitrogen	4.0
O	Oxygen	38.4
Zn	Zinc	6.6
P	Phosphorus	5.8
Al	Alumina	2.5

Table 6: Element concentration for 0.3% Al formulation

The oxygen to carbon ratio for this formulation was 0.89. The O/C ratio was increased compared to control formulation. This shows that the good char was formed as the weight concentration of carbon was high. Carbon is needed to produce a strong and rigid structure of char.

4.3.3 Microstructure of 0.5% Alumina reinforced formulation

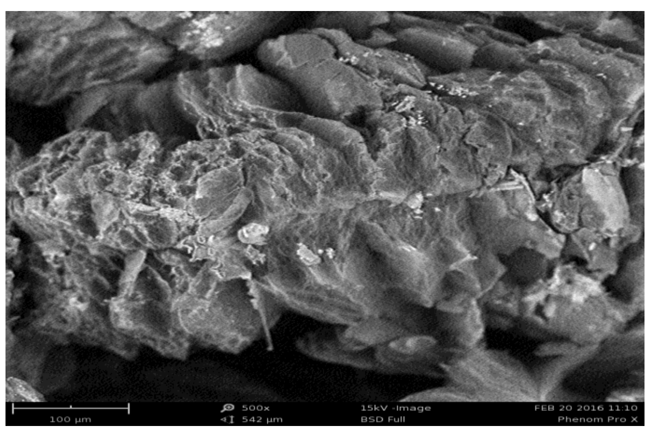


Figure 18: Microstructure of 0.5% formulation at 500x magnification

The crack was not visible in this as shown in the picture. In fact, the structure started to form a homogeneous structure. The homogeneous structure was need in the intumescent coating because of the ability of the structure to entrap the gases produced melamine. The melamine gases produce ammonium gases after it degraded at high temperature. Ammonium gase is needed as the gas helps in the formation of char.

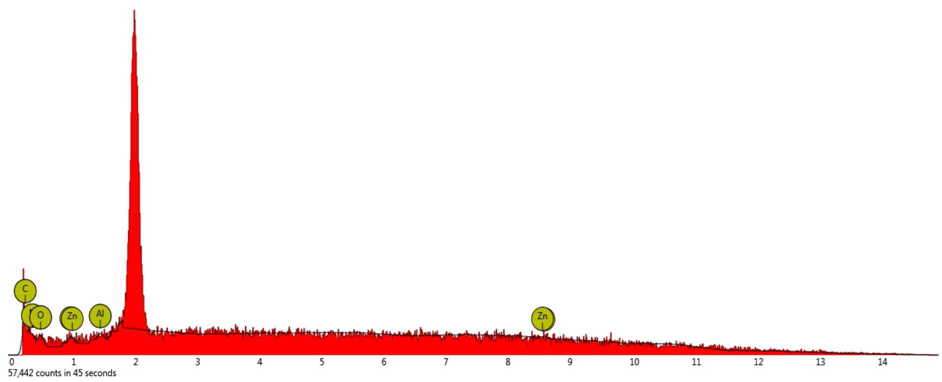


Figure 19: EDS graph of 0.5% Alumina reinforced formulation

Element symbol	Element name	Weight concentration (%)
C	Carbon	41.9
N	Nitrogen	4.2
O	Oxygen	37.8
Zn	Zinc	6.8
P	Phosphorus	6.5
Al	Alumina	2.8

Table 7: Element concentration for 0.5% Al formulation

The oxygen to carbon ratio for this formulation was 0.90. The O/C ratio was increased compared to others formulation. This shows that the good char was formed as the weight concentration of carbon was high. Carbon is needed to produce a strong and rigid structure of char.

4.3.4 Microstructure of 1.0% Alumina reinforced formulation

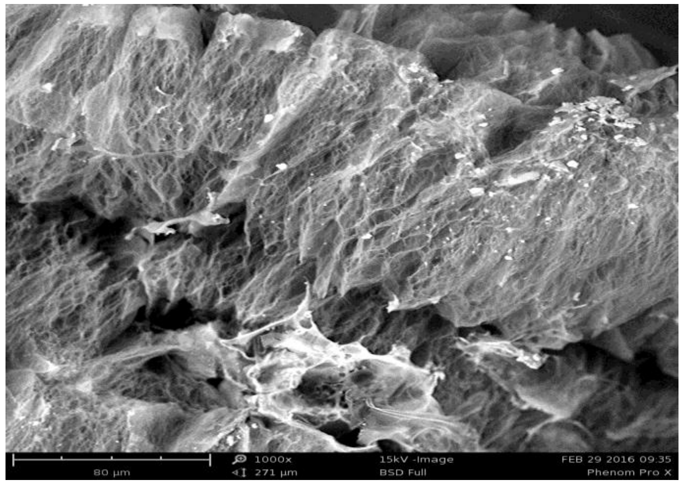


Figure 20: Microstructure of 1.0% formulation at 1000x magnification

The crack was not visible on the structure as shown in the figure. The surface of the the char become more dense. The homogeneous structure became compact than before.

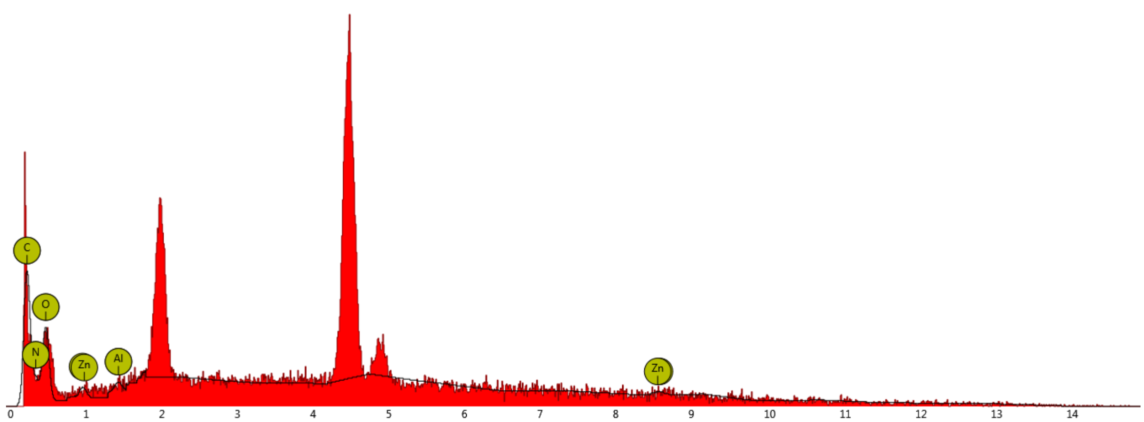


Figure 21: EDS graph of 1.0% Alumina reinforced formulation

Element symbol	Element name	Weight concentration (%)
C	Carbon	42
N	Nitrogen	4.3
O	Oxygen	38.7
Zn	Zinc	3.45
P	Phosphorus	4.1
Al	Alumina	2.85

Table 8: Element concentration for 1.0% Al formulation

The oxygen to carbon ratio for this formulation was 0.92. The percentage of carbon was increased if compared to 0.5% Alumina formulation.

4.3.5 Microstructure of 2.0% Alumina reinforced formulation

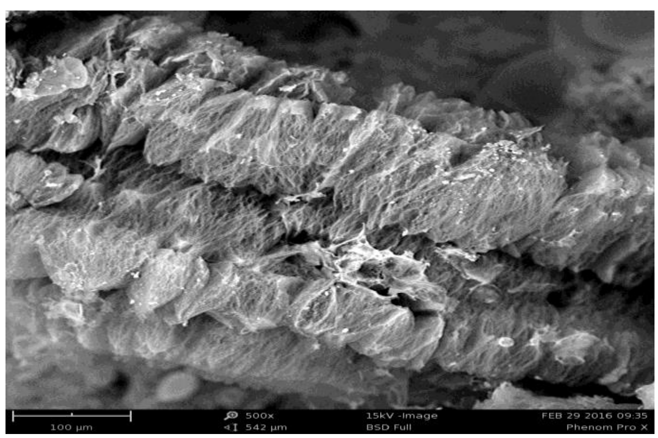


Figure 22: Microstructure of 2.0% formulation at 500x magnification

When the coating was modified to 2.0% of Alumina, the homogeneous structure became more compact and aligned to the other structure. This kind of homogeneous structure will provide a high stabilizing effect on the structure of the char formed.

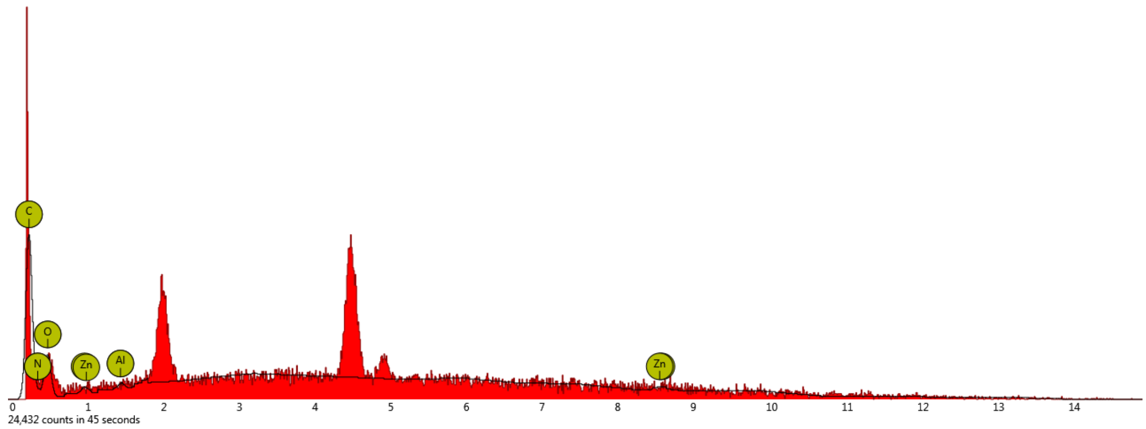


Figure 23: EDS graph of 2.0% Alumina reinforced formulation

Element symbol	Element name	Weight concentration (%)
C	Carbon	42.8
N	Nitrogen	5.0
O	Oxygen	38.2
Zn	Zinc	4.5
P	Phosphorus	6.5
Al	Alumina	3.0

Table 9: Element concentration for 2.0% Al formulation

The oxygen to carbon ratio for this formulation was 0.91. The percentage of carbon is the highest among the coating formulation. This will form a good char with a better intumescent effect if compared to others formulation.

<u>Concluding remarks</u>

From the result shown, the 2.0% of reinforced Alumina formulation had the highest amount percentage of the carbon. Carbon is needed for producing a good and rigid structure of char. The percentage of oxygen became decreased as the percentage of alumina in the formulation increased. As percentage of the oxygen decreased, the possibility of the carbon to react with the oxygen became decreased. If the carbon react with the oxygen, it will produced gases such as CO or even CO_2 which was harmful to the environment. Due to that, an additive such as zinc borate is added to the formulation. Zinc borate will degrade at a certain temperature to become zinc. This zinc will react with oxygen to produce Zinc Oxide (ZnO) .

4.4 RESIDUAL WEIGHT OF INTUMESCENT COATING

Thermo gravimetric analysis (TGA) was carried out to determine the residual weight of the char and degradation temperature of the material present in the formulation. It was described that for a higher residual weight after analysis indicate that the char had higher anti oxidation property. It also indicated the char could withstand high temperature for a longer period.

4.4.1 Residual weight for control formulation

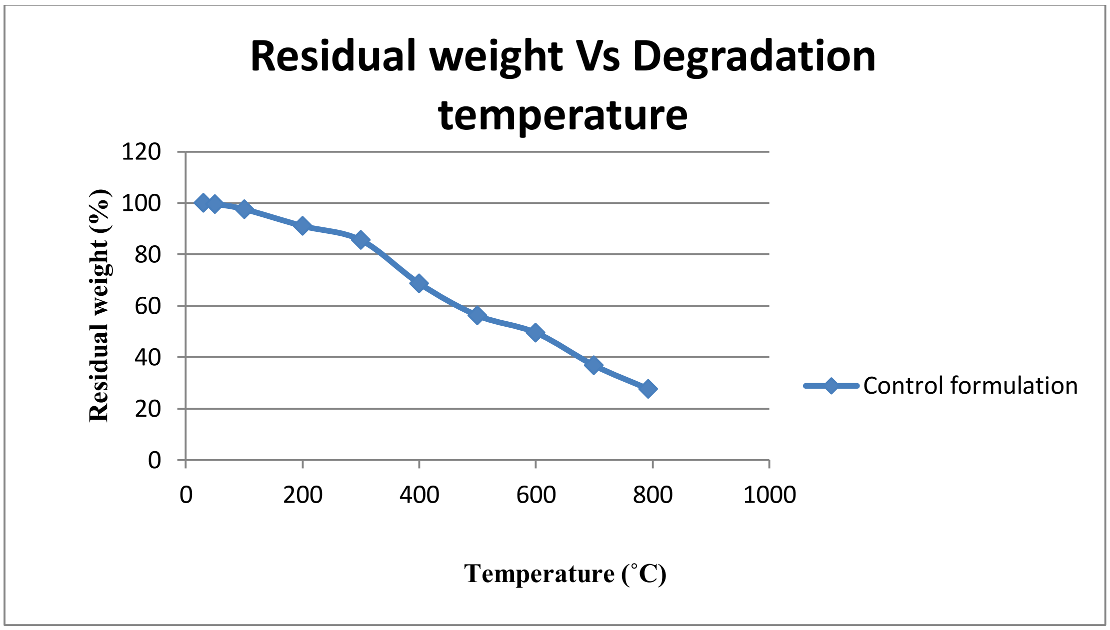


Figure 24: Residual weight (%) for control formulation

The TGA curves shown had 4 stages of degradation. The stages were described as melting, intumescence, formation of char and degradation stage. These stages occurred at (0-200˚C), (200-300˚C), (300-450˚C), and (450-800˚C) respectively. At the first stage which was (0-200˚C), the weight loss occurred approximately about 15% due to the degradation of polymer matrix which was epoxy resin molecules. Besides that, the degradation of boric acid occurred at this stage. At (100 - 140˚C), the boric acid decomposed to become metaboric acid. The metaboric acid was further degraded to become boron oxide at temperature of (140-180˚C) (Jimenez and Duquesne, 2006). The second stage which was in the range of temperature of (200-

300˚C), 12 % weight loss occurred. Ammonium poly phosphate (APP) started to degrade and release ammonia gas and water vapors which then converted into phosphoric acid.

Besides that, Expandable graphite (EG) started to degrade by releasing carbon dioxide. Melamine (MEL) started to decompose within this temperature and release ammonia gas. The importance of ammonia gas was vital as it blows the layer of char formed by the chemical reaction produced by APP, EG, and boric acid. In the third stage (300-450˚C), 25-30% weight loss occurred due to further decomposition of APP which then been converted into polyphosphoric and metaphosphoric acid (Jimenez and Duquesne, 2006). Furthermore, the formation of boron phosphate at this stage occurred due to the degradation of APP and boric acid. This compound was very stable and had higher decomposition temperature which was 1200˚C. During the final stage at (450-800˚C), a layer of char was formed which will provides a thermal insulation to the substrate. This char layer will degrade slowly after certain time and temperature. In the control formulation, the final residual weight obtained was **27.61wt. %**.

4.4.2 Residual weight for Alumina reinforced formulations

Alumina was added into the control formulation in order to study their effect on the residual weight analysis.

	Residual Weight (wt. %)						
Temperature (°C)	Control	0.3%	0.5%	0.7%	1.0%	1.5%	2.0%
30	99.98	99.99	99.93	99.94	99.97	99.95	99.98
50	99.58	99.63	99.68	99.75	99.76	99.84	99.89
100	97.58	97.61	97.65	96.82	96.98	97.45	98.13
200	91.04	90.96	90.87	90.53	91.35	92.56	92.86
300	85.52	84.22	82.34	84.78	85.42	87.41	88.54
400	68.69	67.63	65.67	67.87	65.42	67.97	69.72
500	56.22	59.09	57.64	58.65	57.43	58.12	59.68
600	49.51	51.84	52.68	51.92	53.65	54.49	55.68
700	36.78	36.66	38.43	43.75	46.73	49.52	51.87
793	**27.61**	**28.94**	**32.79**	**35.68**	**37.67**	**39.62**	**41.64**

Table 10: Residual weight (wt. %) for all formulations

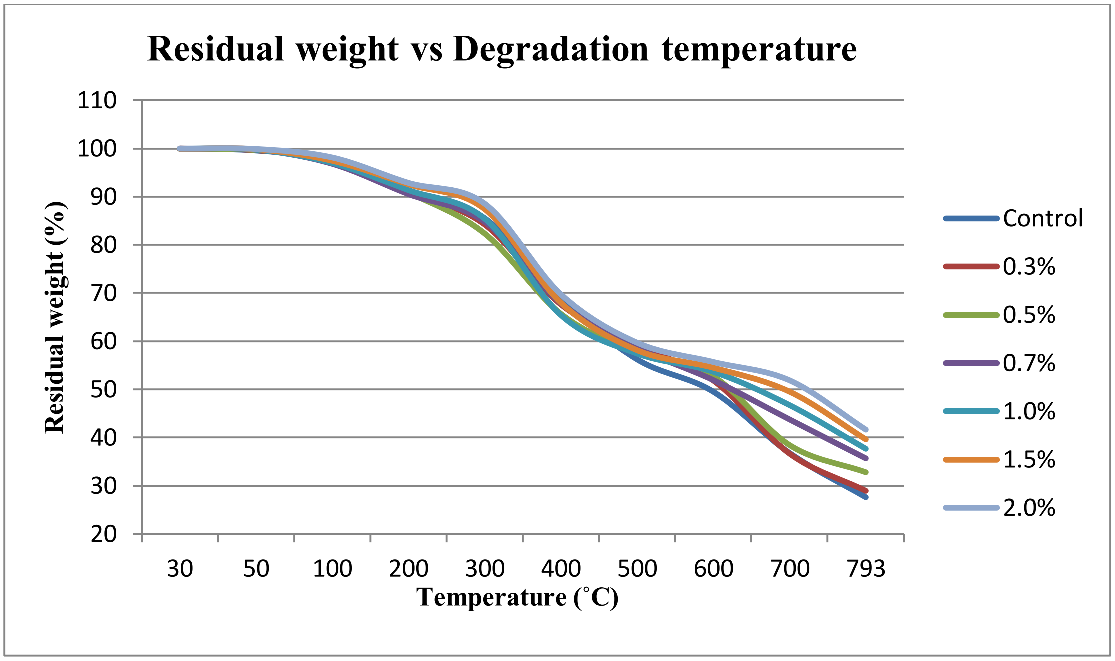


Figure 25: Residual weight against temperature curve for all formulations

Concluding remarks

From the figure above, the residual weight (wt.%) increased as the percentage of alumina increased in the formulation. The final residual weight at 793˚C for control formulation, 0.3% Alumina, 0.5% Alumina, 0.7% Alumina, 1.0% Alumina, 1.5% Alumina and 2.0% Alumina was **27.61, 28.94, 32.79, 35.68, 37.67, 39.62, 41.64 respectively**. Thus the highest residual weight recorded was **41.64** which belong to **2.0%** formulation. During the final stage of the degradation process, it can be seen that the residual weight started to degrade slowly if compared to the initial stage of the degradation process.. The presence of Alumina in the formulation showed that higher amount of residual weight achieved compared to control formulation. Higher amount of residual weight provides a better thermal stability and anti-oxidation property of coating in order to withstand elevated temperature for a longer period. The amount of residual weight for alumina keep increased as the percentage of alumina is high in the formulation. This is because alumina possesses high tolerance of temperature which is 2050˚C J. Green (1996).

4.5 FIRE TESTING (BUNSEN FIRE TEST)

The fire testing was carried out onto 10cm x 10cm coated specimens. The back side temperature for each specimen was recorded for each minute until the temperature measured became constant. A few thermocouples was attached at the backside of the substrates and the temperature was recorded using a Data Logger .**Table 11** showed the physical appearance of the coating obtained after the fire test had been carried out.

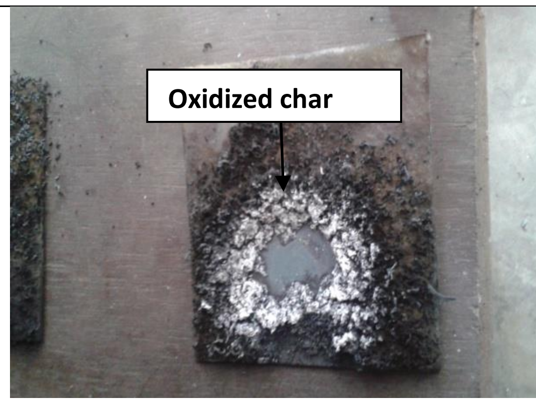
Formulations	Result	Remarks
Control formulation		- Oxidation of char occurred - Some detachment of char from substrate - More smoke were released
2.0% Alumina		- Least oxidation occurred - Least detachment of char - Least smoke were released

Table 11: Appearance of char after fire test for all formulations

time	Alumina 0.3 %	Alumina 0.5 %	Alumina 0.7 %	Alumina 1.0%	Alumina 1.5 %	Alumina 2.0 %	control
0	33.8	34	35.4	34.8	35.6	34	33.4
2	91.8	84.3	89.4	95.7	96.7	91.8	88.2
4	120.4	113.6	105.6	110.6	110.3	100.5	156.6
6	127.4	114.2	124.7	120.4	115.7	109.6	186.7
8	130.5	120.5	129.4	128.9	117.4	110.4	208.5
10	136.5	124.5	135.6	133.4	118.9	112.3	214.3
15	139.7	127.4	137.4	131.5	117.6	111.9	219.5
20	143.6	134.7	138.9	127.4	118.3	115.4	232.6
25	147.8	137.5	142.3	128.3	116.4	114.3	220.7
30	149.8	145.9	141.7	125.4	115.9	114.5	230.9
35	152.6	146.7	140.9	125.9	119.5	115.2	219.4
40	155.8	150.7	138.3	124.3	122.6	114.7	225.6
45	161.6	142.5	137.3	119.3	117.3	114.9	218.3
50	158.7	140.4	135.4	118.2	118.9	116.2	216.7
55	155.4	138.5	136.9	118.6	118.8	115.1	217.9
60	153.9	138.2	134.4	119.5	119.1	115.4	218.2

Table 12: Recorded temperature for all formulations

Sample	**Highest recorded temperature (˚ C)**
Control	232.6
0.3% Alumina	161.6
0.5% Alumina	150.7
0.7% Alumina	142.3
1.0% Alumina	133.4
1.5% Alumina	122.6
2.0% Alumina	116.2

Table 13: Highest recorded temperature for each formulation

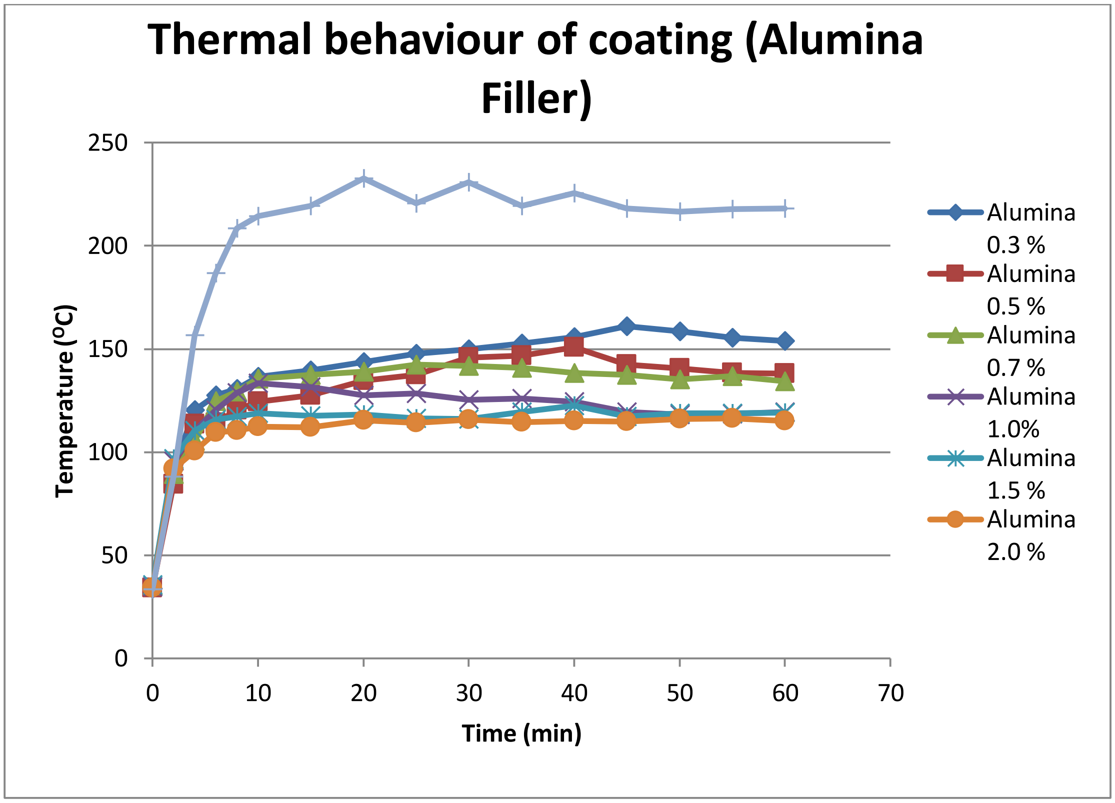


Figure 26: Thermal behaviour curve for all formulations

<u>Concluding remarks</u>

From the data above, the highest temperature recorded by control formulation, 0.3% Alumina, 0.5% Alumina, 0.7% Alumina, 1.0% Alumina, 1.5% Alumina and 2.0% Alumina were 232.6˚ C, 161.6˚ C, 150.7˚ C,142.3˚ C,133.4˚ C,122.6˚ C,116.2˚ C respectively. The present of Alumina showed that the highest temperature recorded decreased as the percentage of Alumina increased in the formulations. The present of Alumina provides a good shielding effect for the coated substrate. Besides that, it was observed that the heat shielding efficiency increased significantly when the coating was modified using Alumina filler. The backside temperature recorded for **2.0% Alumina** formulation had **116.2˚ C** which was better coating formulation compared to others. During the first 15 minutes of the test, it was observed that the temperature rose drastically for all of the formulations. This showed no intumescent effect had been developed during this interval of time. When the test reached at the last 10 minutes, the temperature changes started to become constant. This was due to intumscent effect had been developed and preventing the increment of the temperature. Furthermore, less smoke were released throughout the test of Alumina formulation if compared to control formulation. It was observed that the present of alumina decreased the amount smoke released and therefore acted as a smoke concealed. This proved that the addition of Alumina in the formulation provides a better thermal insulation compared to non-modified formulation.

4.6 HARDNESS TEST (COMPRESSION TEST)

The coated samples were further analyzed through compression test. This test was to analyze the performance of the coatings in term of its strength. Stiffer structure of char will decreased the changes of the height or deformation while the higher drop of the height indicated that the char lack its strength. **Table** below showed some of the char appearance after the compression test.

Formulations	Result	Remarks
Control formulation		- Failed at load of 1200g
2.0% Alumina		- Failed at load of 3200g

Table 14: Physical appearance for all formulations after compression test

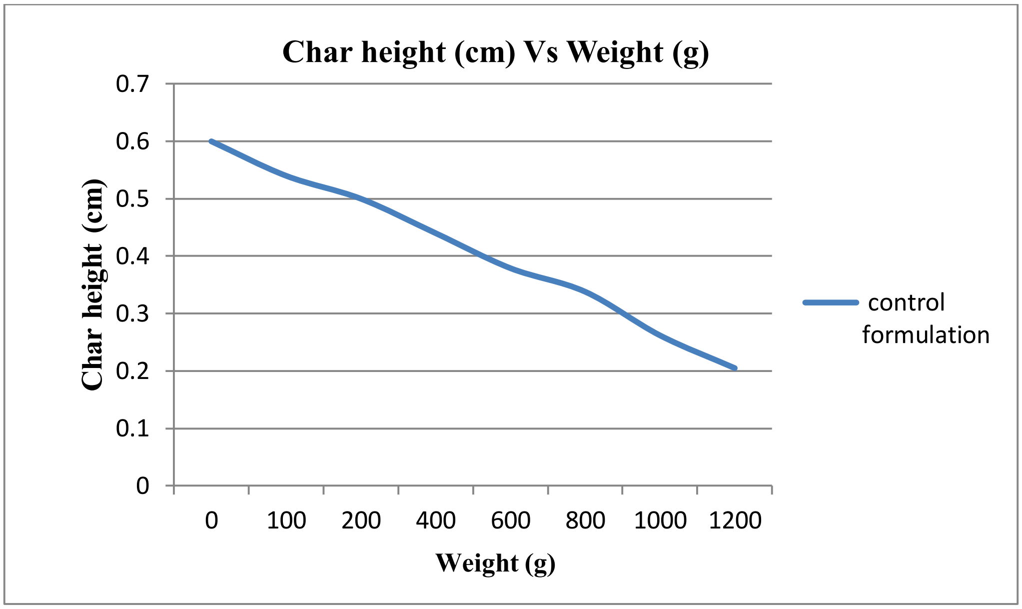


Figure 27: Char height of control formulation after hardness test

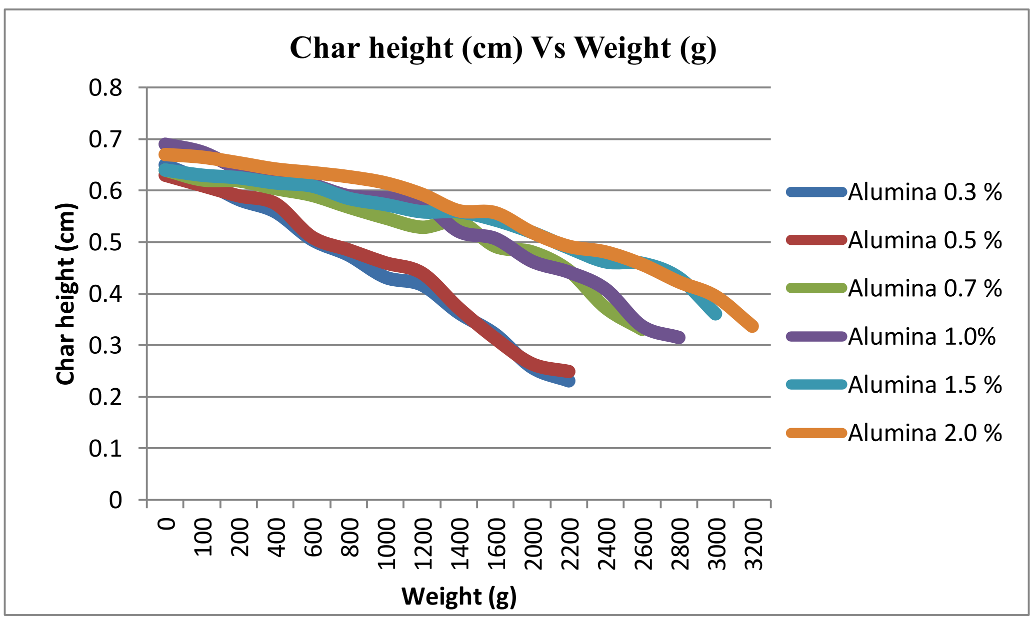


Figure 28: Char height of Alumina reinforced formulation after hardness test

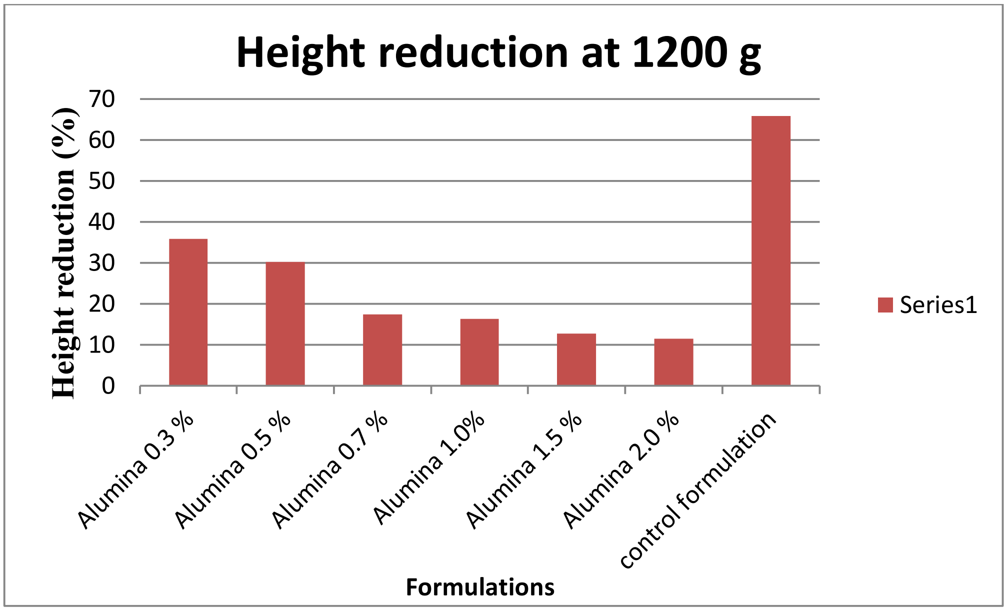


Figure 29: Height reduction (%) of all formulations

<u>Concluding remarks</u>

From the data obtained through hardness test, it was observed that each of the coated samples failed at different weight of load. **From figure 27,** the control formulation of the coating could only withstand up until 1200g. The control formulations ruptured at early stage due to lack of mechanical strength. The better results were showed at **figure 28** after the formulations were modified using Alumina inside the formulations. The result showed that all the modified formulations could withstand more than 1200g of loads. This showed that by adding the Alumina in the formulations increase the strength of the char to resist higher load.

From **figure 28,** the highest load that could be withstands was 3200g by 2.0% of Alumina. This result was followed by 1.5% Alumina (3000g), 1.0% Alumina (2800), 0.7% Alumina (2600g) and 0.5% and 0.3% Alumina (2200g). This showed that higher amount of Alumina in the formulation increase the strength thus resist the height deformation when exposed to external loads eventhough all the samples failed**.** This occurred due compression force from the loads had been attributed to rupture the peak of the char's layer. As all the coatings failed at different loads, the height reduction was plotted at amount of **1200g** loads due to all the coatings could resisted that amount of loads. **From figure 29**, the highest reduction recorded was by **control formulation**. The reduction occurred at this amount of load was **65.8%**. The least reduction occurred at **2.0% Alumina** which was **11.49%**. This showed that the coating possessed higher strength as the coating had higher tendency to resist deformation than the others. The addition of Alumina showed that higher strength of char was formed thus providing a better performance of the coatings in term of insulation and protection of the substrate.

CHAPTER 5

CONCLUSION

5.1 CONCLUSION

Many researches had been conducted shown that intumescent coating system has helped to reduce the fire risks thus saves many life. Intumescent system is a fire insulator and fire that commonly used in the world for fire protection. The coating system expands if it's exposed to fire and produce the char which possessed higher thickness, thus char layer will protect the substrate from heat during fire incident. This coating is not toxic when burned and its thermal expansion could be beneficial for industry usage.

In over the years, many researcher trying to improve the efficiency of intumescent coating effect and there are still many method that can be experimented. The improvement need to be done in term of substrate protection, char expansion, char morphology and residual weight. Some material or filler addition compound such as magnesium oxide, titanium oxide and alumina has shown significant improvement based on past research. The usage of different weightage of alumina in the formulations will produce different char strength heat shielding effect performance. From the experiment been conducted, following conclusions were drawn;

1) The result for intumescent factor showed that reinforcement of Alumina inside the formulation increased the intumescent factor compared to control formulation. Formula modified by 2.0% of Alumina had the highest of the intumescent factor which was **7. 6.**
2) Char morphology were improved with the modification of Alumina inside the formulation. The absent of the cracks for the modified formulation showed that a better performance of the coating can be achieved. A homogeneous structured was seen at 0.5% , 1.0% and 2.0% of Alumina that could trap the melamine gas which is vital in formation of char.
3) TGA results showed that more residual weight was produced when the formulation was modified with Alumina. The highest residual weight

occurred at 793˚C with 41.64(wt.%) recorded by 2.0% of Alumina. Control formulation had 27.61(wt.%) of residual weight. This showed that by adding Alumina in the formulation, much residual weight was present that provides thermal stability and anti oxidation property for the coating to withstand high temperature

4) Thermal behavior of the coating was improved by addition of Alumina in the formulation. The back side temperature recorded for control formulation was 232.6˚C while the formulation with 2.0% of Alumina was 116.2˚C. The decrement of the temperature showed that the heat shielding effect efficiency of Alumina increased.
5) The char strength of the modified formulation increased as compared to control formulation. All the formulations ruptured at some load but the highest load recorded was 3200g by 2.0% of Alumina formulation. The control formulation ruptured at early stage which was 1200g.

From the above conclusions, all the objectives were met throughout the projects. Some recommendations are provided for future work and enhancement for the project.

5.2 RECOMMENDATIONS

1) The performance of the coatings need to be studied in a different environments
2) A further research need to be conducted on using others fillers such as magnesium oxide to improve the performance of the coating
3) The usage of sago starch as a carbon source in the development of the intumescent coating due to abundantly available in Malaysia

REFERENCES

Triantafyllidis, Z & Bisby, L, 2014, Fibre-reinforced epoxy intumescent coatings for steel beam

C . A. Wilkie and A. B. Morgan, *Fire Retardancy of Polymeric Materials*, 2nd ed. Boca Raton, FL: Taylor & Francis Group, 2010.

C. J. Hilado, *Flammability Handbook for Plastics*, 4th ed.: Taylor & Francis, 1990.

Z . W. Wicks, F. Jones, and P. Pappas, *Organic Coating Science and Technology*, 2nd ed. New York, United States of America: Wiley Interscience, 1992.

S . Duquesne, S. Magnet, C. Jama, and R. Delobel, "Intumescent paints: fireprotective coatings for metallic substrates," *Surface and Coatings Technology*, vol. 180-181, pp. 302–307, March 2004.

Zhenyu Wang , Enhou Han, Wei Ke " *Effect of nanoparticles on the improvement in fire- resistant and anti-ageing properties of flame-retardanat coating* " , September 2005.

Camino,G. and R. Delobel, 2000. Intumescence. In: Fire Retardancy of Polymeric Materials, Grand, A.F amd C.A. Wilkie (Eds). Marcel Dekker, New York, pp: 217-243

Effect of fire to steel structure. Retreived October 25, 2015, from http://prugarinc.com/steel/fire-damage-to-structural-steel/

M Jimenez, S. Duquesne, and S. Bourbigot, "Characterization of the performance of an intumescent fire protective coating," *Surface and coatings Technology*, vol. 201, no. 3, pp. 979-987, October 2006.

P . Joseph and J. R. Ebdon, *Recent Developments in flame-retarding thermoplastics and thermosets*. Cambridge, England: Woodhead Publishing Limited, 2001.

A . R. Horrocks and D. Price, *Fire Retardant Materials*. Cambridge, England: Woodhead Publishing Ltd, 2001.

A . Riva, G. Camino, L. Fomperie, and P. Amigouët, "Fire retardantmechanism in intumescent ethylene vinyl acetate compositions," *Polymer Degradation and Stability*, vol. 82, no. 2, pp. 341–346, 2003.

T.A. Roberts, L.C. Shirvill, K. Waterton, I. Buckland "Fire resistance of passive fire protection coatings after long-term weathering,"Process Safety and Environmental Protection, 88 (2010), pp. 1–19

J. W. Gilman, T. Kashiwagi, and J. D. Lichtenhan, "Nanocomposites: a revolutionary new flame retardant approach," *Sampe Journal*, vol. 33, pp. 40-46, 1997.

M. Jimenez, S. Duquesne, and S. Bourbigot, Thermochimica acta, 449, pp. 16-26, (2006)