CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Each material on the earth will face a serious weakness as it is exposed to fire for some duration. Thus, it is same goes to the steel; it will lose its structural strength at elevated temperature. Thus, the main function of intumescent coating is to protect the steel up to two hours, where it can give ample time for evacuate and rescue process if there is any fire accident happens. Moreover, the intumescent coating also act as thermal insulation for a building, as it is can help to resist the fire spread to another building if the adjacent building is on fire. Thus the bonding strength between the substrate and the polymer is a fundamental aspect in intumescent coating. Poor bonding between the coating and the substrate will lead to unprotected steel substrate due to char fall off. As the result its increase the speed of heat transfers to steel substrate. A strong bonding of intumescent coating is desired to form a good protective char layer for steel substrate. Moreover, intumescent coating is a requirement of building regulation in many countries. Hence, this phenomena lead to the grows of intumescent coating technology nowadays

1.2 PROBLEM STATEMENT

Previously, research on formulation intumescent coating by using Expandable Graphite, EG as carbon source had some limitation of poor bonding strength. In theory said that when increasing the EG quantities, the swelling percentage of char become higher, as the weight percentage of EG is increase. However, as it pass to its optimum performance, the swelling percentage will decrease although increasing the percentage of EG in the formulation. Thus, some additives need to be added for improvement of intumescent coating formulation. Hence, by adding inorganic fillers as additive inside the formulation are said can improve the performance of intumescent coating.

1.3 OBJECTIVE AND SCOPE OF STUDY

1.3.1 Objective

i. To develop intumescent coating formulations with inorganic fillers in order to obtain an optimum performance.

ii. To study the effect of inorganic fillers in the formulation in term of bonding strength.

1.3.2 Scope of Study

In order to improve the previous research on intumescent coating, an inorganic filler will be added in the formulation. The selected inorganic fillers are fumed silica and Alumina Trihydrate (ATH). ATH is a very effective flame retardant due to its thermodynamics properties which absorb heat and release water vapor at certain temperature. On the other hand, fumed silica is stated as thermal insulator. Moreover, scope of study also included research on the effect of inorganic filler in the formulation in term of bonding strength. Thus, variations of weight percentage of the inorganic fillers were be made in order to obtain the optimum formulation. The test that will cover are shear test where to examinate the bonding strength of the coating with the substrate and fire test which is to examinate the bonding strength of the char with the substrate. Both of the tests can determine the characteristic of bonding strength of intumescent coating with steel substrate.

CHAPTER 2

LITERATURE REVIEW

2.1 INTUMESCENT COATING

Intumescent coating is a fire protection paint as search on the internet. Moreover it is the oldest and easiest way protect material against fire, yet it is an efficient ways [1,2]. Thus, it have several advantages, which are it does not modify the intrinsic properties of the material (e.g the mechanical properties), it is easily to manufacture and can be used onto several material such as metallic materials [3], polymers [4], textiles [5] and wood [6]. Hence this project is focusing on metallic materials as the material is widely used in the industry. The metallic material is steel. Every metal on the earth has its own characteristic and its limitations. Thus, for steel it will degradation if expose to elevate temperature which is 550^{o}C [7]. The steel decompose and show weakness on its characteristics especially on mechanical properties.

The working principle of intumescent coating is swelling when exposed to fire. Thus, it will form expended multicellular layer, which acts as thermal barrier [8]. The expended multicellular layer is called char. It prevents heat from penetrating and flames from spreading. As a consequence, the char makes intumescent coating particularly suitable for the protection of structural steelwork.

Intumescent coating is composed in three active main ingredients; carbon agent, blowing agent and acid source [9]. All these components are bind with a binder. Here, in this study Expandable graphite (EG) was been used as carbon source, melamine (EN) as blowing agent and ammonium polyphosphate (APP) as acid source. The used of EG has its own advantage: when exposed to the heat, exfoliation of the graphite occurs, i.e expansion of the crystal structure by about hundred times [10]. Thus it generates a protective layer as a result.

There are some limitation of EG in the intumescent coating. Incresing of weight percentage EG will increase the swelling percentage, due to the molecular weight is increasing. However, the swelling percentage decreases, although increasing the weight percentage of EG. Thus, this indicate there is a limitation weight percentage where EG can contribute for better swelling [11].

Hence, some additive needs to be used for reinforce the intumescent coating. Here, study is focusing on inorganic fillers as it gives advantages to intumescent coating; It does not send out organic solvent in the application and have little toxic emission and also smoke output in heating [12]. The inorganic fillers are fumed silica and Alumina Trihydrate (ATH). ATH is a very effective flame retardant due to its thermodynamics properties which absorb heat and release water vapor in certain temperature. Thus, it becomes a good fire protection. In order word ATH is Aluminium hydroxide. Moreover, ATH became aluminium oxide which act as char barrier and water when exposed to heat from flame [13]. The equation can be shown as below

$$Al(OH)_3 \text{ ----- } Al_2O_3 + H_2O \quad (1)$$

On the other hand, fumed silica has potential to reduce heat release and burning rates [14]. Thus it reduce the flammability properties of intumescent coating. Both characteristics of inorganic fillers inside intumescent coating formula will be compared in term of bonding strength.

Several Test will be conducted to achieve the objective. Firstly the shear test will be conducted in order to determine the mechanical properties of the intumescent coating by using tensile machine [12]. The bonding strength (f_b) where calculated using Eq. (2.1.1)

$$f_b = F/A \quad (2.1.1)$$

F_b Bonding strength, MPa

F Force, N

A Sticking Area, m^2

Second, the fire test. The structure of intumescent coating will be analyses in term of bonding strength by using Scanning Electron Microscope, SEM in two condition, before and after fire test. Here, the specimens will exposed to the standard temperature for a certain period of time [15]

2.2 FIRE TEST

The performance of building and structure under fire exposure condition is an item of major importance in securing construction are safe and not menace to neighboring structures nor to the public. In order to ensure this safety, it is necessary that the fire resistive properties of material and assemblies be measured and specified according to a common standard. Hence, there will be two types of fire test, which are Furnace Test and Bunsen Burner Test. Those tests are based on ASTM E119 [17].

2.2.1 Furnace Test

In these tests, building components are subjected to a constantly increasing and decreasing furnace temperature intended to represent a standard fire. The components are then rated, with units of time, on their ability to withstand the exposure up to a criterion that is defined as a failure point. It is expected that a 2-h rated wall would resist failure in a real fire for a longer period of time than a similarly functioning 1-h rated wall, and this is invariably the case [16].

On the procedure section, the furnace test will be using three specimens in order to obtain an accurate result. Those specimens will be placed in the Muffle Furnace. Moreover, these specimens will follow a temperature cycle, called Time-Temperature Curve. The Time-Temperature Curve is shown below;

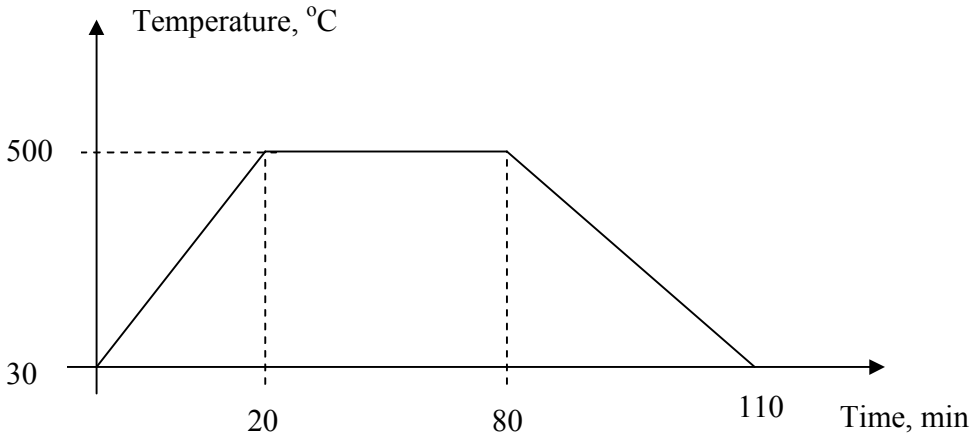


Figure 2.1.1: Time-Temperature Curve for Furnace Test

Hence, several analysis will be covered after obtaining result from Furnace Test, for example, Scanning Electron Microscope for view char formation characterize, X-Ray Diffraction, XRD to examinate the composition of the char formation and Thermo Gravimetric Analysis, TGA for calculate weight loss of the formulation.

For the test specimen, the size that will be used is 10mm x 10mm, with 1 mm thickness of coating. The equipment that will be used is Muffle Furnace.

2.2.2 Bunsen Burner Test

There are two types of Bunsen Burner Test, which are Vertical and Horizontal Bunsen Burner Test. The difference between these two tests is the orientation of the fire source itself. However, in order to study the bonding strength of inorganic fillers based on intumescent coating, it is enough to conduct one of the test. Thus, the scope of this test is determining the resistance of materials to flame when tested according to the 1-h horizontal Bunsen burner tests specified in ASTM E119[17].

The procedure of this test is need to prepare the apparatus on the initially. The apparatus is Bunsen burner, clampers, timer, thermocouple and test specimen. Then, the apparatus were set up according to the figure below;

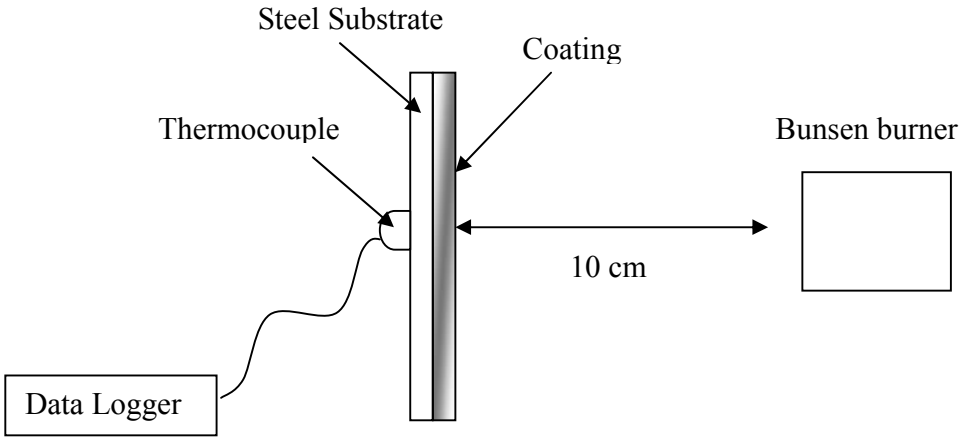


Figure 2.1.2: Bunsen Burner Diagram

Here, the steel substrate with coating will be exposed to the Bunsen burner about 1-h and the distance is about 10 cm. Hence, the temperature of the steel substrate was record and been interpret in a table. The burning characteristics are also been inspect using visual inspection. 100 mm x 100 mm test specimen will be used and been coated about 1 mm of thickness.

2.3 SHEAR TEST

Based on the American Standard Testing Method (ASTM), the shear test will be follow on ASTM D3136[18] where this standard is for determine strength of adhesively rigid plastic lap-shear joints in shear by tension loading. There are several items need to be considered. The items are scope of test, test procedure, specimen size, result and equipment used in order to run the test.

The scope of Lap Shear Test are determines the shear strength of adhesives for bonding materials when tested on a single-lap-joint specimen. The test is applicable for determining adhesive strengths, surface preparation parameters and adhesive environmental durability.

Figure 2.3.1: Shear Test Diagram

The test procedure based on ASTM D3163 for adhesively bonded rigid plastic, two specimens are bonded together with adhesive and cured as specified. The test specimens are placed in the grips of a universal testing machine and pulled at 1.3 mm/min (0.05 in/min) until failure.

The criteria of specimen is using two specimens, each 25.4 x 101.6 mm (1" x 4") are bonded together with adhesive so that the overlap is sufficient to provide failure in the adhesive, and not in the substrate. Typical overlaps are 12.7 mm and 25.4mm (0.5", 1").

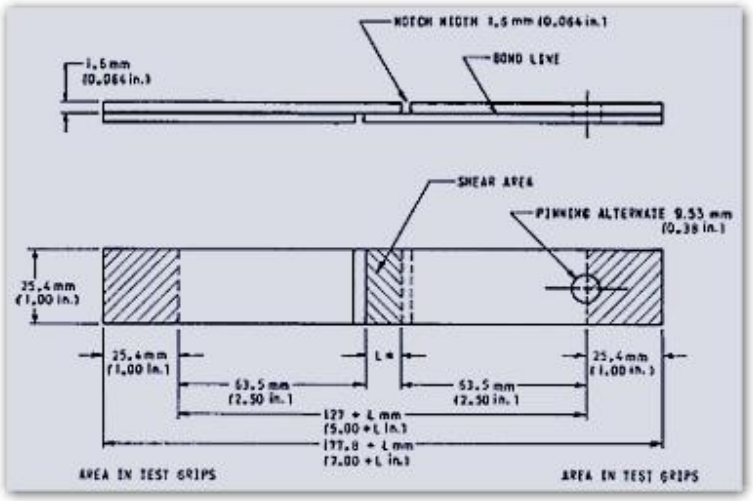


Figure 2.3.2: Test Specimen Diagram

There are several data collection will be obtained when running the test [12]. The data collection as shown below;

1. Maximum, minimum and average values of failure load
2. Failing Stress in Megapascals, MPa
3. Type of failure (cohesive, adhesive, or substrate)

The equipment that will be used in the test is Universe Tensile Machine, UTM

CHAPTER 3

METHODOLOGY

3.1 RESEARCH METHODOLOGY

Based on *Appendix I,* flow chart of the project methodology, shown that after preliminary research or literature review is Experimental Setup. At Experimental Setup stage, where, several of formulation had been made and the amounts of the additives were been variable. However, in order to make a formulation, several of procedures need to be follow. One of the procedures is sample preparation. The sample preparation is divided into 3 phase, which are Preparation of Expandable Graphite, Preparation of Steel Substrate and Preparation of Coating with additives. The detail of all this procedure will be covered on 3.2 Procedure of Sample Preparation

3.2 PROJECT ACTIVITIES

During, project activities are at sample preparation stage, which is the experimental setup before move to the Experiment Work. Sample preparation procedure are divided into 3 phase. The first phase is Expandable Graphite preparation, Steel Substrate preparation and Coating preparation.

3.2.1 Expandable Graphite Preparation Procedure

1. Graphite was been grinding into 300µmm size about 1 minute with a constant speed in grinding machine
2. Sieze the graphite in order to obtain 300µmm size.
3. Expandable graphite was prepared by mixing of graphite flake with H_2SO_4 with ratio 1:1 respectively in a conical flask
4. Stirred the mixture at 25^{o}C in a conical flask
5. Washed the mixture with distilled water and filter it.
6. Leave the Expandable Graphite to dry in the oven with 60^{o}C temperature

3.2.2 Steel Substrate Preparation Procedure

1. A large steel plate is prepared.
2. Steel plate was cut into 3 different sizes;
    a. 100 mm x 100 mm
    b. 50 mm x 50 mm
    c. 25.4 mm x 101.6mm
3. The steel plate was polished by using sand paper to remove rust on the steel
4. Steel plate was sand blast to remove contaminant on the steel.

3.2.3 Coating Preparation Procedure

1. Those materials were prepared
    a. Expandable Graphite (EG)
    b. Ammonium polyphosphate (APP)
    c. Boric Acid (BA)
    d. Melamine (Mel)
    e. Bisphenol a epoxy resin BE-188 (BPA)
    f. ACR Hardener H-2310 polyamide amine
2. APP, BA, Mel were weighted based on ratio using micro weighing scales, *refer Appendix II*
3. The ingredient was grinded for about 1 minute in order to obtain 300µm size using grinding machine, *Appendix III*
4. Mixture was mix with Expandable graphite according to the ratio in a plastic container
5. Epoxy and hardener were weighted according the ratio and been mix using high shear mixer until the color change yellow milk
6. Mix all chemical by using High Shear Mixer, *Appendix IV* about 20 minute with increment in speed of mixer until it reached 50 rpm.
7. Apply the coating on the steel, *refer appendix V*

Thus, the formulation of intumescent coating without inorganic filler as shown on the table below;

Table 3.2.3.1: Formulation of Intumescent Coating

Formulation	**EG** (g)	**APP** (g)	**MEL** (g)	**Boric Acid** (g)	**Fumed Silica** (g)	**Epoxy** (g)	**Hardener** (g)
O_1	5.88	11.76	5.88	11.76	0	47.05	23.52

Hence, this formulation will be the reference for both inorganic fillers that is selected. The inorganic fillers are Fumed Silica and Alumina Trihydrate, ATH. The formulation of intumescent coating with Fumed Silica shown below;

Table 3.2.3.2.: Formulation of Intumescent Coating with Fumed Silica

Formulation	**EG** (g)	**APP** (g)	**MEL** (g)	**Boric Acid** (g)	**Fumed Silica** (g)	**Epoxy** (g)	**Hardener** (g)
F_1	5.88	11.76	5.88	11.76	1	46.5	23
F_2	5.88	11.76	5.88	11.76	2	46	22.5
F_3	5.88	11.76	5.88	11.76	3	45.5	22
F_4	5.88	11.76	5.88	11.76	4	44	21.5
F_5	5.88	11.76	5.88	11.76	5	43.5	21
F_6	5.88	11.76	5.88	11.76	6	43	20.5
F_7	5.88	11.76	5.88	11.76	7	42.5	20
F_8	5.88	11.76	5.88	11.76	8	42	19

For the formulation of intumescent coating with Alumina Trihydrate is shown below;

Table 3.2.3.3: Formulation of Intumescent Coating with ATH

Formulation	**EG** (g)	**APP** (g)	**MEL** (g)	**Boric Acid** (g)	**ATH** (g)	**Epoxy** (g)	**Hardener** (g)
A_1	5.88	11.76	5.88	11.76	1	46.5	23
A_2	5.88	11.76	5.88	11.76	2	46	22.5
A_3	5.88	11.76	5.88	11.76	3	45.5	22
A_4	5.88	11.76	5.88	11.76	4	44	21.5
A_5	5.88	11.76	5.88	11.76	5	43.5	21
A_6	5.88	11.76	5.88	11.76	6	43	20.5
A_7	5.88	11.76	5.88	11.76	7	42.5	20
A_8	5.88	11.76	5.88	11.76	8	42	19

3.3 GANTT CHART

Table 3.3: Gantt Chart

Activity	FYP 1				FYP 2				Remark
	May	June	July	Aug	Sept	Oct	Nov	Dec	
Early Stage of Documentation									**Done**
Study of Intumescent Coating Formulation with inorganic Fillers. (Obj 1)									**Done**
Run Fire Test. (Obj 2)									**Done**
Conduct Shear Test (Obj 2)									**Done**
Analyses on different characteristic of bonding strength for each sample. (Obj 2)									**Done**
End Stage Documentation									**Done**

3.4 KEY MILESTONE

Table 3.4: Key Milestone Table

Activity	FYP 1				FYP 2				Remark
	May	June	July	Aug	Sept	Oct	Nov	Dec	
Determine the formulation of sample Intumescent Coating. (Obj 1)									**Done**
Completion of Shear Test. (Obj 2)									**Done**
Completion of Fire Test. (Obj 2)									**Done**
Conclude The Analyses (Obj 2)									**Done**

CHAPTER 4

RESULT AND DISCUSSION

4.1 RESULT

Below was the result for Furnace Test with purchase graphite and treated graphite, Fire Bunsen Burner Test and Shear Test.

4.1.1 Furnace Test – Purchase Graphite

On furnace test, in early stage of experimental work, the carbon source that be used was been purchase from graphite manufacture from China, thus the result as below:-

a. Without Inorganic Filler

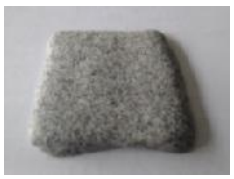
Table 4.1.1.1: Initial Thickness

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3	4		
O_1	6.00	6.76	5.76	6.16	6.17	4.67

Table 4.1.1.2: Final Thickness

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3	4		
O_1	24.22	22.00	25.60	27.92	24.935	23.435

Table 4.1.1.3: Comparison of Intumescent Coating without Inorganic Filler

Formulation	Initial	Final
O_1		

A visual inspection, the initial characteristic shown the surface of the coating is smoother and stick firmly on the steel substrate. On the other hand, final visual inspection shown that, the coating is fall off from the steel substrate and made the steel substrate is not protected.

b. With ATH

Table 4.1.1.4: Initial Thickness

Formulation	Thickness with Steel (mm)			Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3		
A_1	6.38	6.30	6.38	6.35	4.85
A_2	6.00	5.40	5.70	5.70	4.20
A_3	6.38	6.10	6.18	6.22	4.72
A_4	5.54	5.34	5.24	5.37	3.87
A_5	4.58	4.80	5.46	4.95	3.45
A_6	4.72	4.72	5.00	4.81	3.31
A_7	5.70	5.30	5.40	5.47	3.97
A_8	5.80	5.20	5.20	5.40	3.90

Table 4.1.1.5: Final Thickness

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3	4		
A_1	0.10	9.50	32.00	22.60	36.05	34.55
A_2	30.30	26.56	24.40	25.78	26.76	25.26
A_3	34.58	37.64	37.64	32.06	35.48	33.98
A_4	31.76	30.98	26.84	30.00	29.90	28.40
A_5	27.66	35.88	23.00	18.92	26.37	24.87
A_6	20.62	20.00	21.52	23.34	21.37	19.87
A_7	11.00	24.58	20.00	19.22	18.70	17.20
A_8	23.64	22.30	27.86	26.66	25.12	23.62

Table 4.1.1.6: Comparison Characteristic of ATH

Formulation	Initial	Final
A_1		
A_2		
A_3		
A_4		
A_5		
A_6		
A_7		
A_8		

As looking the initial coating, the surface of the coating is smoother, however, it become rough until formulation A_8. Due to the mixture become more viscous than the A_1 formulation. When, the percentage of inorganic filler is higher, it become more difficult to mix and place on steel. However, in final result, all the coating is split off from the steel substrate. The expansion of coating is shown on Table 4.2.1. Where, each formulation has their own expansion characteristic.

c. Fumed Silica

Table 4.1.1.7: Initial Thickness

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3	4		
F_1	7.00	7.00	7.10	7.20	7.075	5.575
F_2	6.30	6.48	6.00	6.68	6.365	4.865
F_3	6.60	6.18	6.68	7.00	6.615	5.115
F_4	7.96	6.92	6.22	6.32	6.855	5.355
F_5	5.82	5.40	6.40	6.00	5.905	4.405
F_6	7.20	6.92	6.62	6.92	6.915	5.415
F_7	6.50	6.68	6.58	6.92	6.670	5.170

Table 4.1.1.8: Final Thickness

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3	4		
F_1	22.72	24.00	21.92	21.60	38.31	22.56
F_2	17.26	17.38	21.68	17.92	18.56	17.06
F_3	21.18	15.38	23.92	17.16	20.87	19.37
F_4	21.48	27.86	23.94	23.00	24.07	22.57
F_5	21.28	23.00	17.32	17.66	19.82	18.32
F_6	20.22	23.22	23.94	23.00	22.60	21.10
F_7	16.18	17.00	20.60	16.52	17.58	16.08

Table 4.1.1.9: Comparison Characteristic of Fumed Silica

Formulation	Initial	Final
F_1		
F_2		
F_3		
F_4		
F_5		
F_6		
F_7		

As all the intumescent coating were burn inside furnace at 500^{o}C, shown that, the coating is not stick firmly on the steel substrate. However, each of formulation shows its own char expansion characteristic. The characteristic is shown on the Table 4.1.1.9. Besides that, the initial coating characteristic can be visualize clearly shown, the surface of the coating is roughly and become even more roughly as the increasing the percentage of Fumed silica. Hence, it creates difficulties in mix and applied on the steel substrate. Thus, formulation of F_8 could not be performed as such problems occur.

4.1.2 Furnace Test – Treated Graphite

As the purchase graphite is not shown a good result, thus the result is not fulfill the objectives, yet it consider a failure project. In order to ensure the project successful, the expandable graphite is be change, by using treated expandable graphite. The fabrication process is shown on Methodology chapter. Moreover, the sample of intumescent coating been selected to reduce the cost. Further experimental work is using this kind of carbon source. The result of furnace test is shown below;

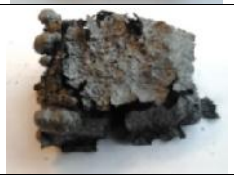
Table 4.1.2.1: Initial Thickness

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3	4		
O_1	4.82	5.00	4.22	4.22	4.565	3.065
A_1	4.42	4.32	4.52	4.92	4.545	3.045
A_2	5.32	4.42	4.22	4.94	4.725	3.225
A_3	4.62	5.62	5.54	5.84	5.405	3.905
F_1	5.18	5.10	5.42	5.50	5.300	3.800
F_2	4.62	4.98	5.02	5.26	4.970	3.470
F_3	4.82	4.88	4.52	5.00	4.805	3.305

Table 4.1.2.2: Final Thickness

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)
	1	2	3	4		
O_1	4.2	4.7	4.5	5.2	4.7	3.2
A_1	2.3	2.0	2.6	2.3	2.3	0.8
A_2	5.05	5.6	5.5	5.65	5.45	3.95
A_3	3.0	3.3	3.5	3.3	3.5	2.0
F_1	2.35	2.4	2.4	2.7	2.45	0.95
F_2	4.3	4.6	4.05	4.8	4.45	2.95
F_3	1.8	1.75	1.95	1.85	1.85	0.35

Table 4.1.2.3: Comparison Characteristic of Treated Graphite

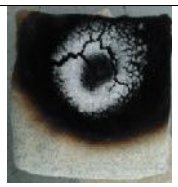
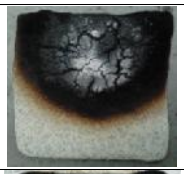
Formulation	Initial	Final
O_1		
A_1		
A_2		
A_3		
F_1		
F_2		
F_3		

Based on the visual inspection, the formulation of O_1, A_1, A_2, F_1 have smooth surface than A_3, F_2, F_3. As they have less percentage of inorganic filler than A_3, F_2, F_3. However, on the final result shown the reference coating which are O_1 is well attached on the steel substrate but there is slightly crack on the coating surface. On the other hand, the formulation of A_1, A_3, F_1 and F_3 are not well attached on the steel

substrate and yet they have plenty of cracks on the surface. As, the crack is not prefer for the swelling process. For the F_2 and A_2 formulation shown good bonding strength as the reference, however, F_2 has more crack on the coating than A_2. Besides that, each of the formulation has their own char expansion characteristic, the char expansion is shown on Table 4.2.2

4.1.3 Fire Bunsen Burner Test

Table 4.1.3.1: Comparison Characteristic of Fire Bunsen Burner Test

Formulation	Initial	Final
O_1		
A_1		
A_2		
A_3		
F_1		
F_2		
F_3		
Ref		

Based on the table above shown the characteristic of intumescent coating before and after the test. One of the picture which is ref is represent reference parameter, in order to find the temperature of the substrate steel without intumescent coating. Here, some of the result shown, there are liquid flowing out from the sample during the test. The liquid is mixture of hardener and epoxy. This gives an indicator that the mixture are not well mix with other material. Moreover, the obvious characteristic that could be observed during the test is the detachment of coating from the steel substrate. This failure were occur on samples F_2 and F_3, where F_2 is detached partial, mostly at the burning side and for F_3 fully detached at 20 minute after test been conducted. Detail picture shown below;

Table 4.1.3.2: Visual Inspection on F_2 and F_3

Formulation	**Visual Observation**		
	Above	**Side**	**Bottom**
F_2			
F_3			

Besides that, inspections on unaffected area, mainly below side of the sample were done, in order to check whether the sample is affected by direct fire or not. The thicknesses initial and final were measured. The result was shown below;

Table 4.1.3.3: Initial Measurement of Fire Bunsen Burner Test

Formulation	Trial Measurement, mm				Average Thickness with substrate steel, mm
	1	2	3	4	
O_1	4.22	4.44	4.40	4.34	4.35
A_1	5.98	5.90	6.20	5.89	5.99
A_2	4.8	5.0	4.9	4.42	4.78
A_3	4.96	4.36	4.72	4.00	4.51
F_1	4.66	5.36	4.92	5.40	5.09
F_2	5.24	5.28	5.44	5.96	5.48
F_3	3.82	3.64	4.42	4.04	3.97
Ref	1.5	1.5	1.45	1.55	1.50

Table 4.1.3.4: Final Measurement of Fire Bunsen Burner Test

Formulation	Trial Measurement, mm				Average Thickness with substrate steel, mm
	1	2	3	4	
O_1	4.54	4.34	4.20	4.44	4.38
A_1	5.96	5.89	6.15	5.92	5.98
A_2	4.42	5.0	4.9	4.72	4.96
A_3	4.92	4.15	4.52	4.50	4.41
F_1	5.35	5.85	5.00	5..00	5.30
F_2	7.60	8.46	4.22	5.68	6.49
F_3	-	-	-	-	-
Ref	1.45	1.55	1.49	1.58	1.52

Based on the result, there was measurement that could not been calculated, as the sample was detached after 20 minutes test been conducted. The sample was F_3. Hence the discussion of the thickness was on chapter 4: Discussion.

Moreover, the heat shielding effect data were been collected, by collecting the change of temperature in every 5 minute for 1 hour. Thus, the thermocouple had been place on the back of substrate steel. The individual heat shielding effect shown on the graph below;

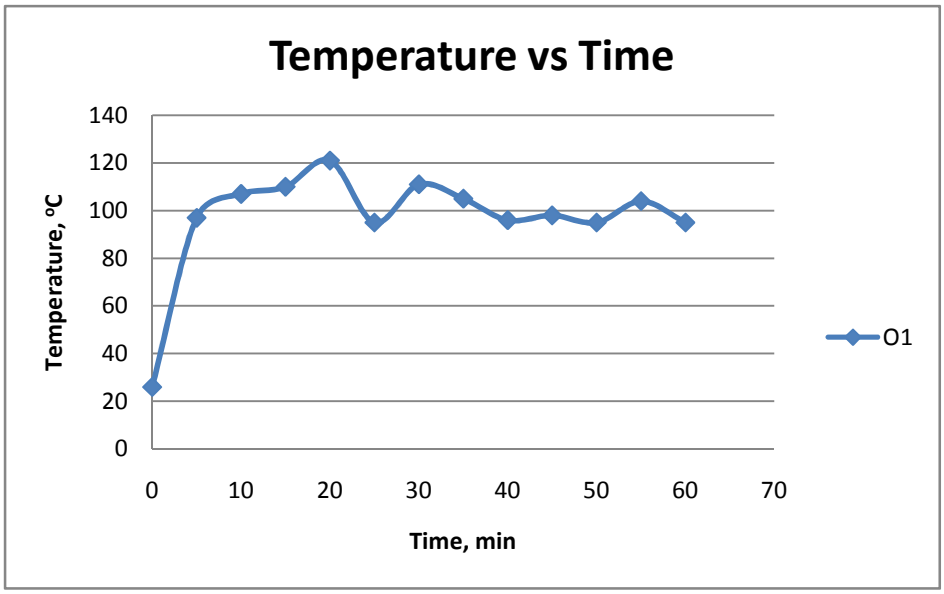


Figure 4.1.3.1: Graph of Intumescent Coating without Fillers

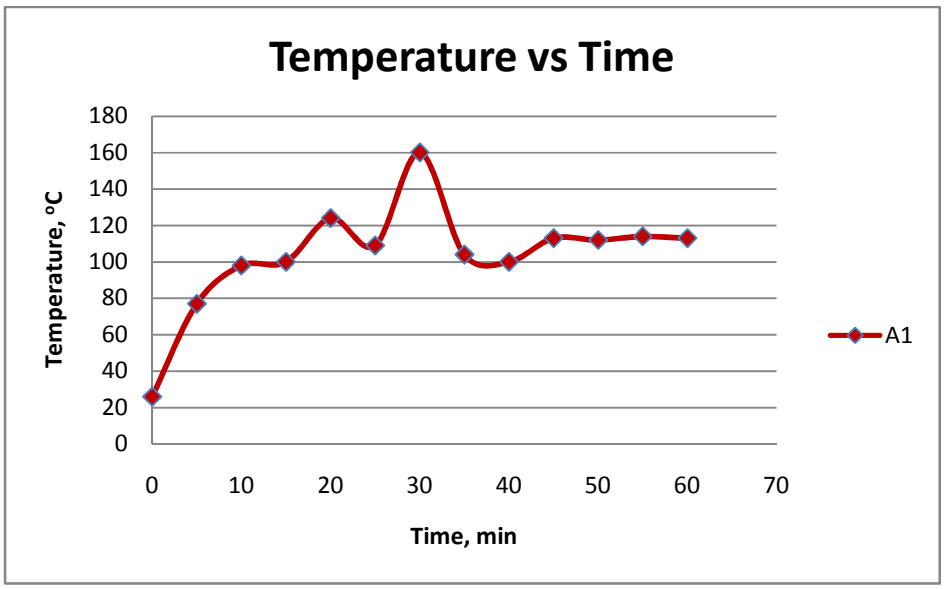


Figure 4.1.3.2: Graph of Intumescent Coating with 2% of ATH

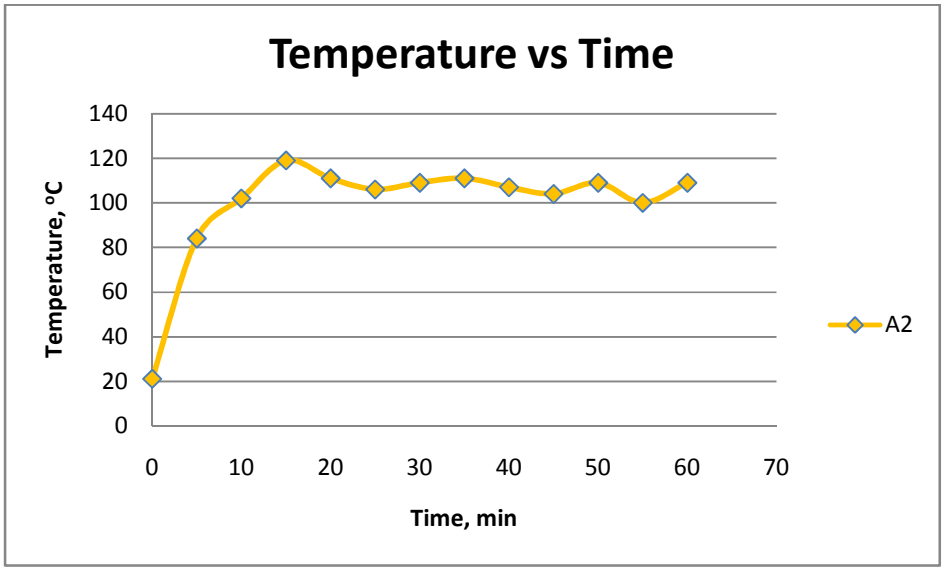


Figure 4.1.3.3: Graph of Intumescent Coating with 4% of ATH

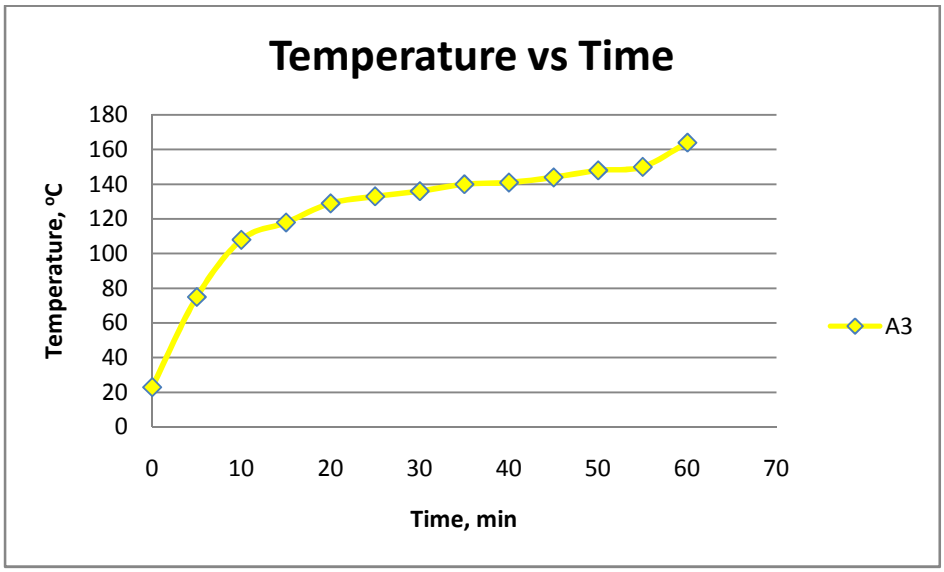


Figure 4.1.3.4: Graph of Intumescent Coating with 6% of ATH

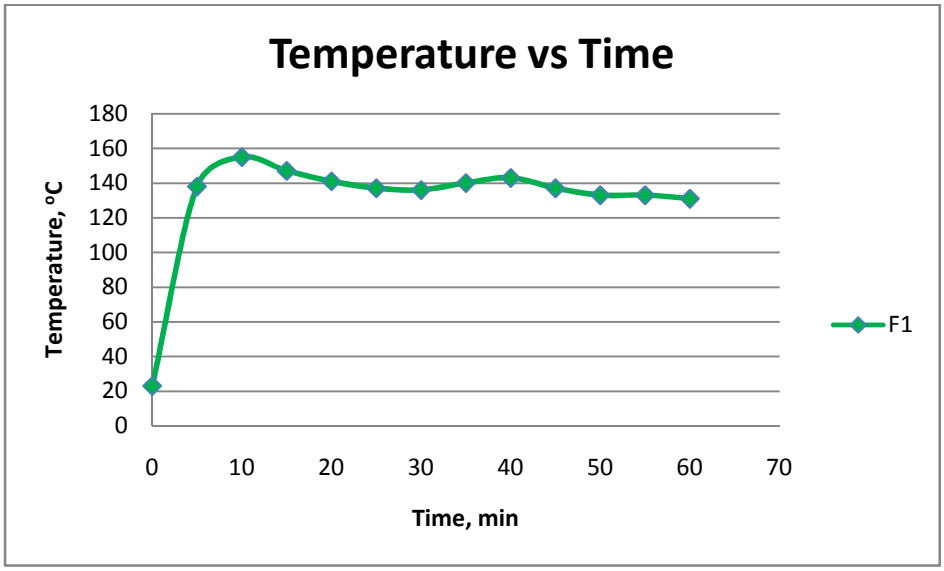


Figure 4.1.3.5: Graph of Intumecsent Coating with 2% of Fumed Silica

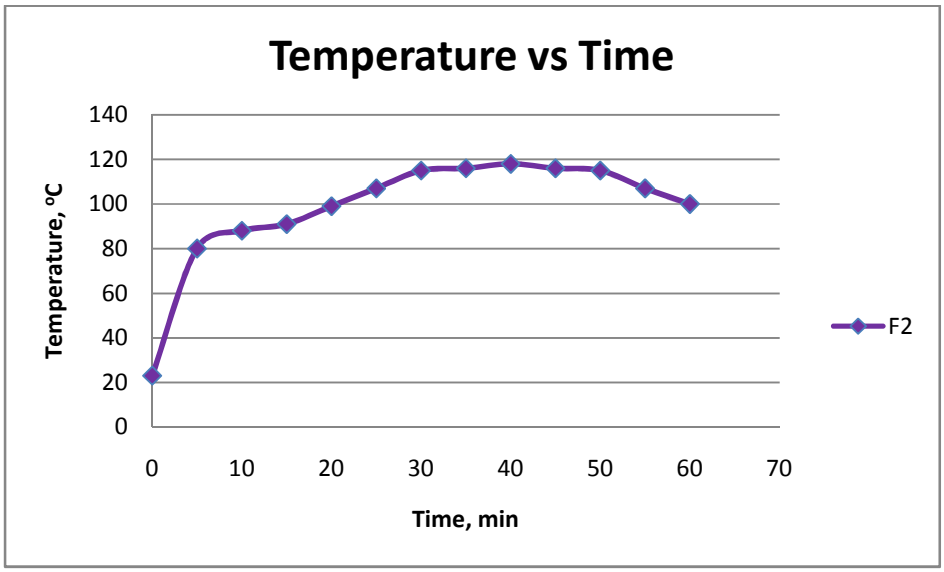


Figure 4.1.3.6: Graph of Intumescent Coating with 4 % of Fumed Silica

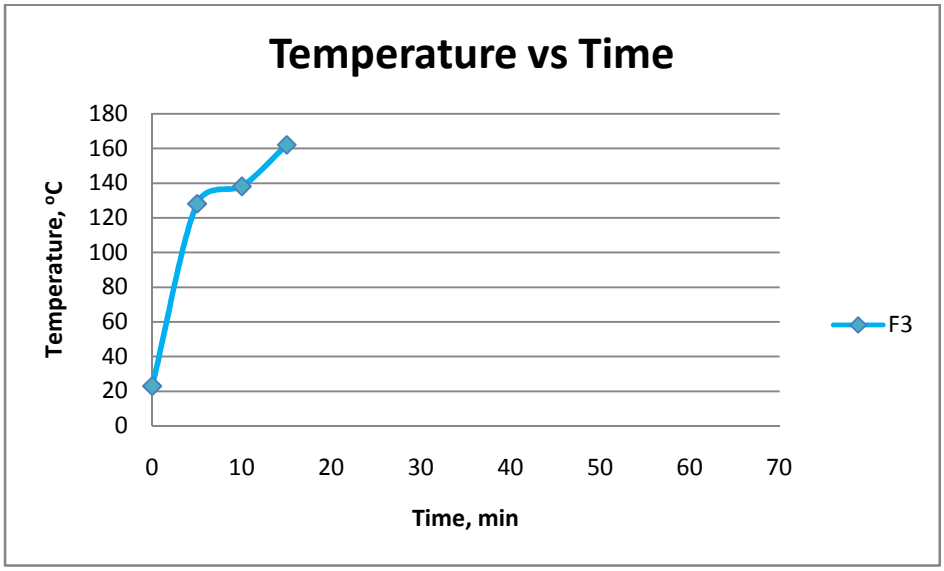


Figure 4.1.3.7: Graph of Intumescent Coating with 6% of Fumed Silica

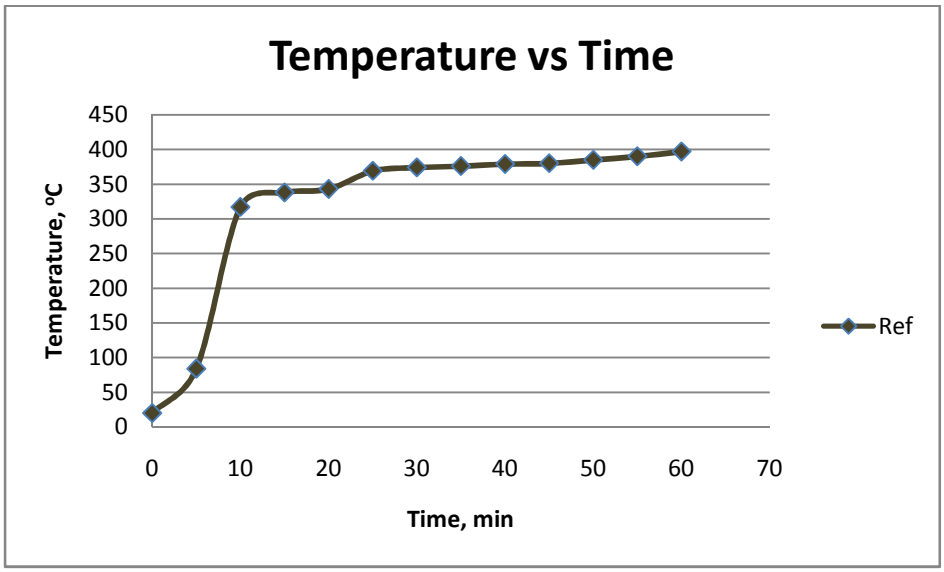


Figure 4.1.3.8: Graph of Substrate Steel without Intumescent Coating

Hence, the characteristic of heat shielding for each coating is maximum at certain time, then temperature will drop at certain time and become a constant approximately. However, these theory is not acceptable for some sample, example A_3 where the temperature is keep rising until 60 minute. Thus, the high temperature for each of sample as shown on the table below;

Table 4.1.3.5: Heat Shielding for Each Sample

Formulation	**The Highest Heat Shielding Temperature, ^{o}C**	**Time, min**
O_1	121	20
A_1	124	20
A_2	119	15
A_3	164	60
F_1	147	15
F_2	118	40
F_3	162	15
Ref	397	60

4.1.4 Shear Test

Table 4.1.4.1: Initial Data for Sample Shear Test

Formulation	Thickness with Steel (mm)				Average Thickness with Steel (mm)	Average Thickness without Steel (mm)	Length sample on the steel (mm)
	1	2	3	4			
O_1	4.42	4.44	4.12	4.10	4.27	1.27	37.0
A_1	4.92	5.70	5.40	4.78	5.20	2.20	39.0
A_2	4.80	5.00	5.18	5.12	5.03	2.03	36.0
A_3	5.90	5.52	5.44	5.70	5.64	2.64	40.0
F_1	5.70	6.08	5.62	6.30	5.93	2.93	40.5
F_2	6.18	6.78	6.64	6.08	6.42	3.42	37.0
F_3	6.28	5.92	6.00	6.48	6.17	3.17	37.5

***Note**: Overall length of the specimens is 237.4 mm, width is 28.0 mm

Table 4.1.4.2: Final Visual Inspection of Shear Test

Formulation	Visual	Characteristic
O_1		The coating attach well on both side of the steel substrate
A_1		The coating is attach heavily only at one side of steel substrate.
A_2		The coating is attach partial on both side side of steel substrate. Due to fracture on the coating or vacancy of molecule in the coating
A_3		The coating is not attach on the other side of steel substrate
F_1		The coating is not attach on the other side of steel substrate
F_2		The coating is attach partial on both side side of steel substrate. Due to fracture on the coating or vacancy of molecule in the coating
F_3		The coating is not attach on the other side of steel substrate

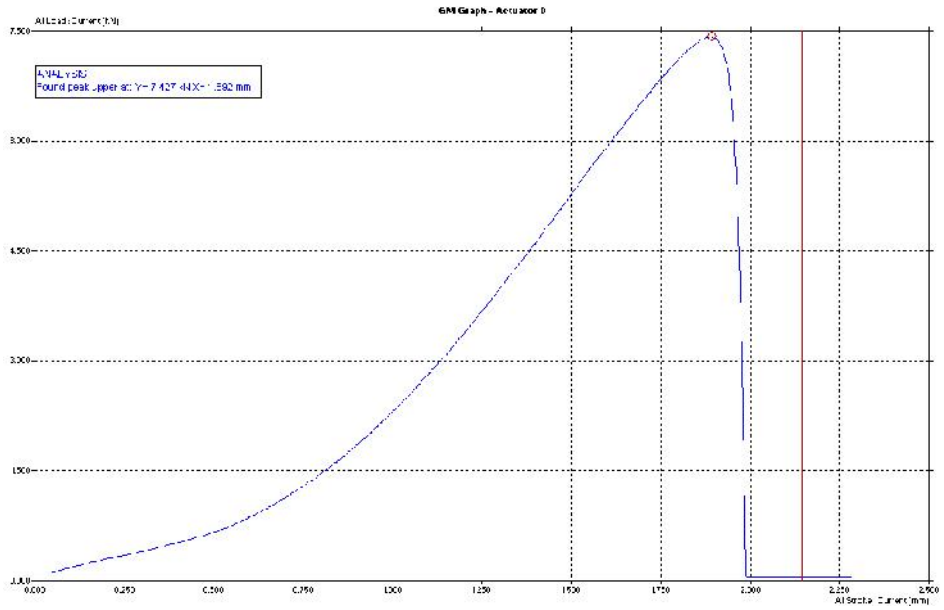


Figure 4.1.4.1: Graph of Intumescent Coating without Inorganic Filler

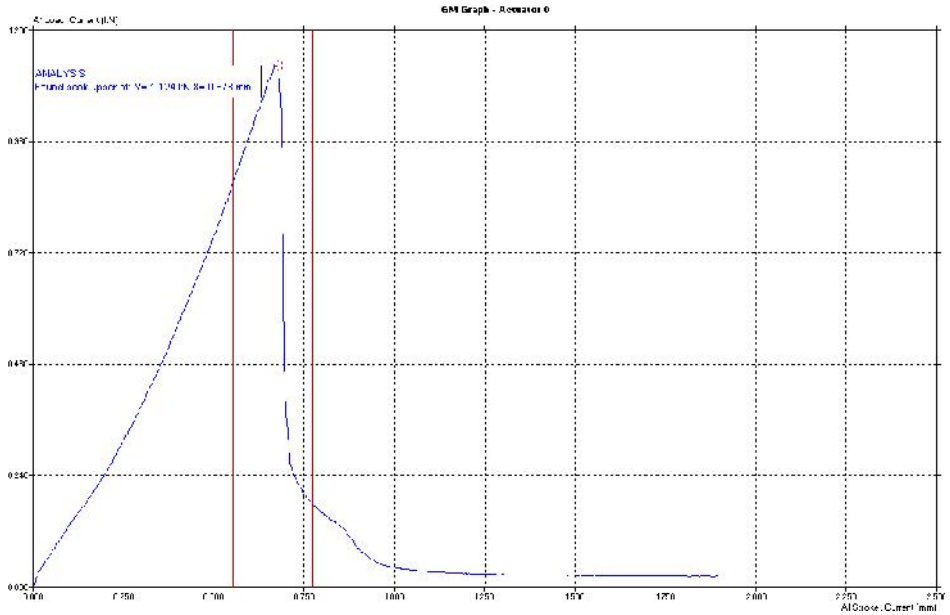


Figure 4.1.4.2: Graph of Intumescent Coating with ATH 2%

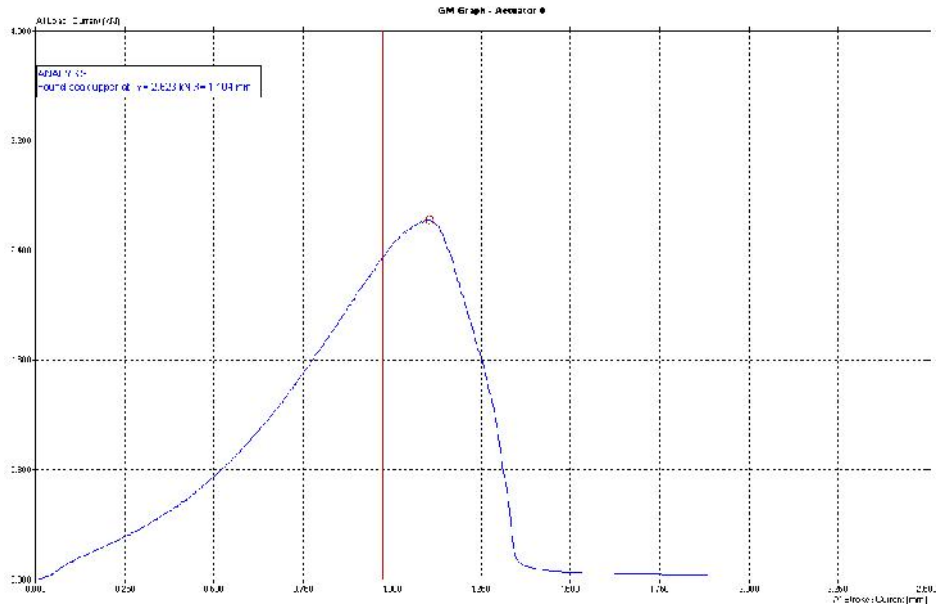


Figure 4.1.4.3: Graph of Intumescent Coating with ATH 4%

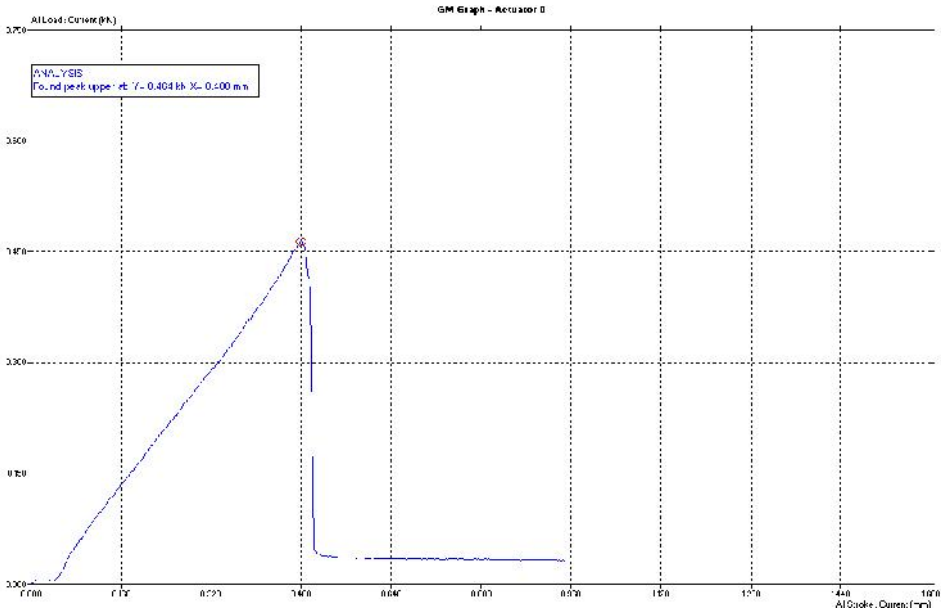


Figure 4.1.4.4: Graph of Intumescent Coating with ATH 6%

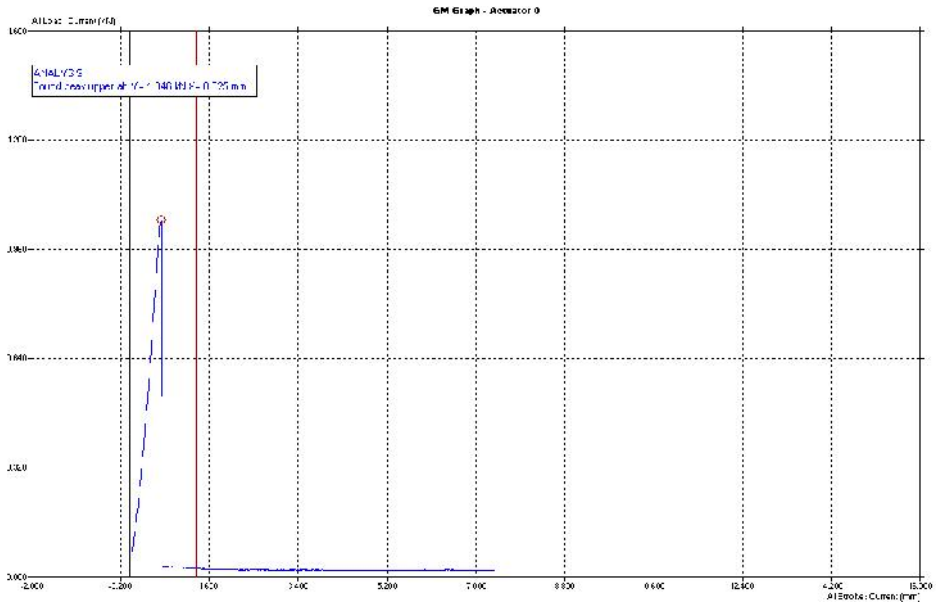


Figure 4.1.4.5: Graph of Intumescent Coating with Fumed Silica 2%

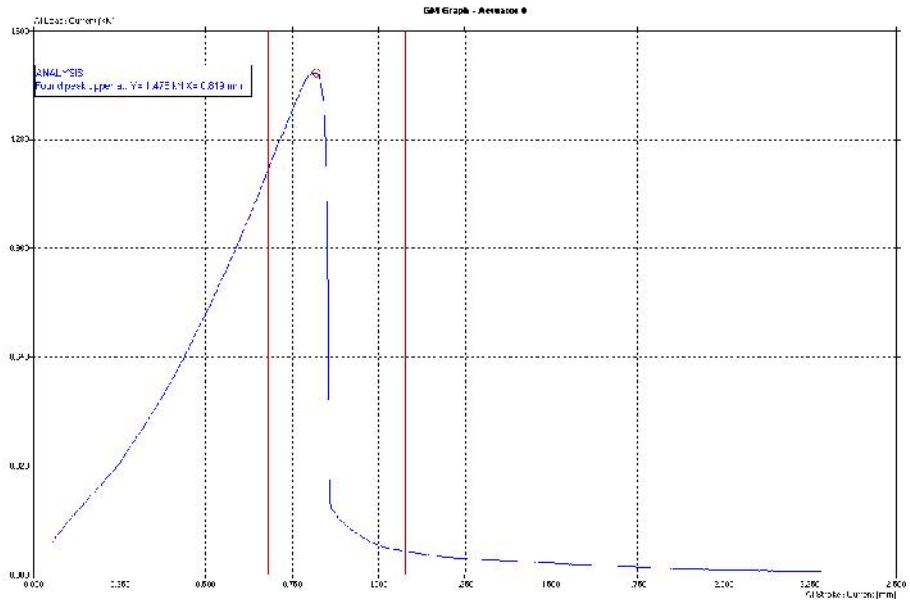


Figure 4.1.4.6: Graph of Intumescent Coating with Fumed Silica 4%

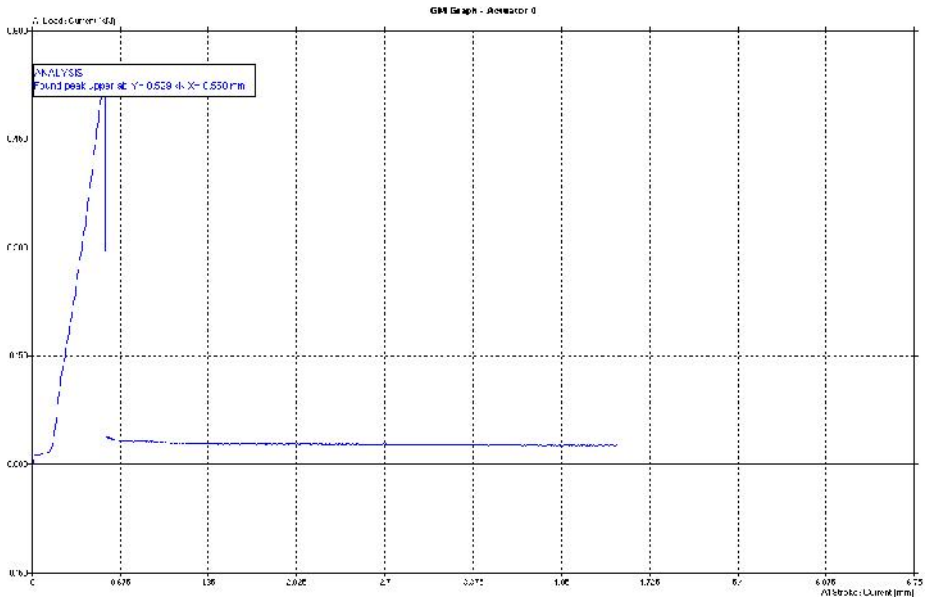


Figure 4.1.4.7: Graph of Intumescent Coating with Fumed Silica 6%

4.1.5 Thermal Gravimetric Analysis (TGA)

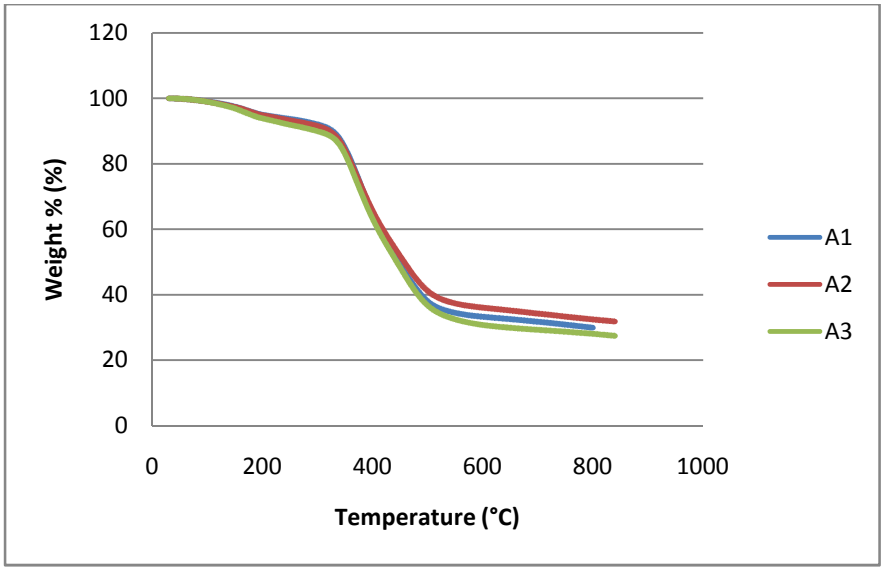


Figure 4.1.5.1: TGA Result for ATH

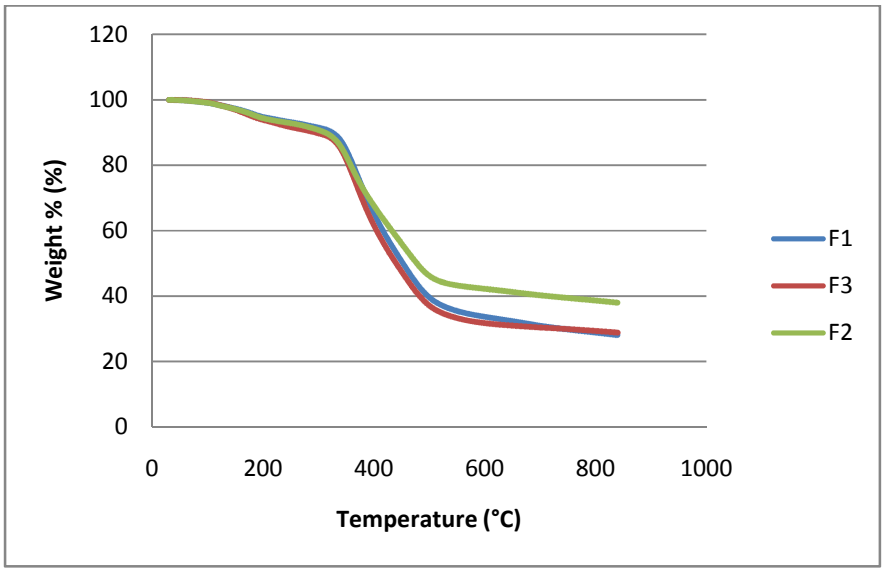


Figure 4.1.5.2: TGA Result for Fumed Silica

Based on two results from TGA, the intumescent coatings follow some kind of pattern before become residual. Thus, three phases can been see on figure above which are phase 1: between 30^{o}C until 300^{o}C, phase 2: between 301^{o}C until 500^{o}C and phase 3: between 501^{o}C until 840^{o}C. Those three phases are water elimination, degradation and residual process. The

highest residual weight have better characteristic in anti oxidation and thermal stability. A good intumescent coating must have a high residual weight. However, based on Figure 4.1.5.1, A_2 has better residual weight while on Fumed Silica TGA result, F_2 have better residual weight from other formulation. Thus, a comparison between A_2, F_2 and O_1 (reference) were done in order to find which formulation shown better in residual weight.

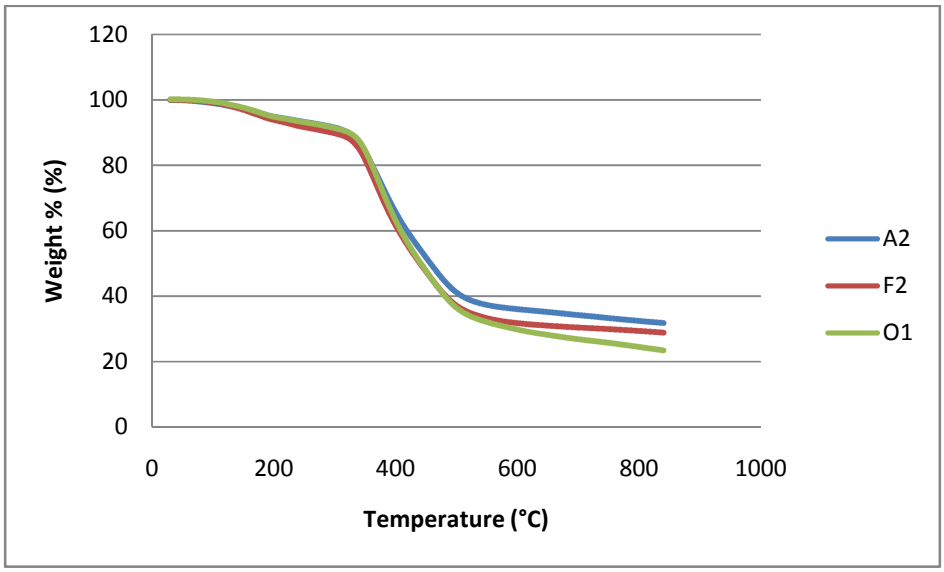


Figure 4.1.5.3: Comparison TGA Result between A_2, F_2 and O_1

Hence, A_2 have better residual weight from F_2 and O_1 based on figure above. Thus, A_2 is better in anti oxidation and thermal stability characteristic.

4.2 DISCUSSION

As the initial and final thickness of intumescent coating had been record, thus the char expansion of the intumescent coating were been found by dividing the final thickness without steel substrate with the initial thickness without steel substrate. The formula illustrate as below;

$$\text{Char expansion} = \frac{\text{Final Thickness without Steel Substrate}}{\text{Initial Thickness without Steel Substrate}} \quad (4.2.1)$$

Thus, the result of calculation for each inorganic fillers as shown in the table below;

Table 4.2.1: Char Expansion for Purchase Graphite

Formulation	Char Expansion
O_1	5.018
A_1	7.124
A_2	6.014
A_3	7.199
A_4	7.339
A_5	7.209
A_6	6.003
A_7	4.332
A_8	6.056
F_1	4.057
F_2	3.507
F_3	3.787
F_4	4.215
F_5	4.159
F_6	3.897
F_7	3.110

As the result been analyze, shown that there is a peak char expansion for the intumescent coating formulation. Thus, in order to see clearly, it had been interpreted into line graph. The green line is representing reference value which is O_1. The graph shown below;

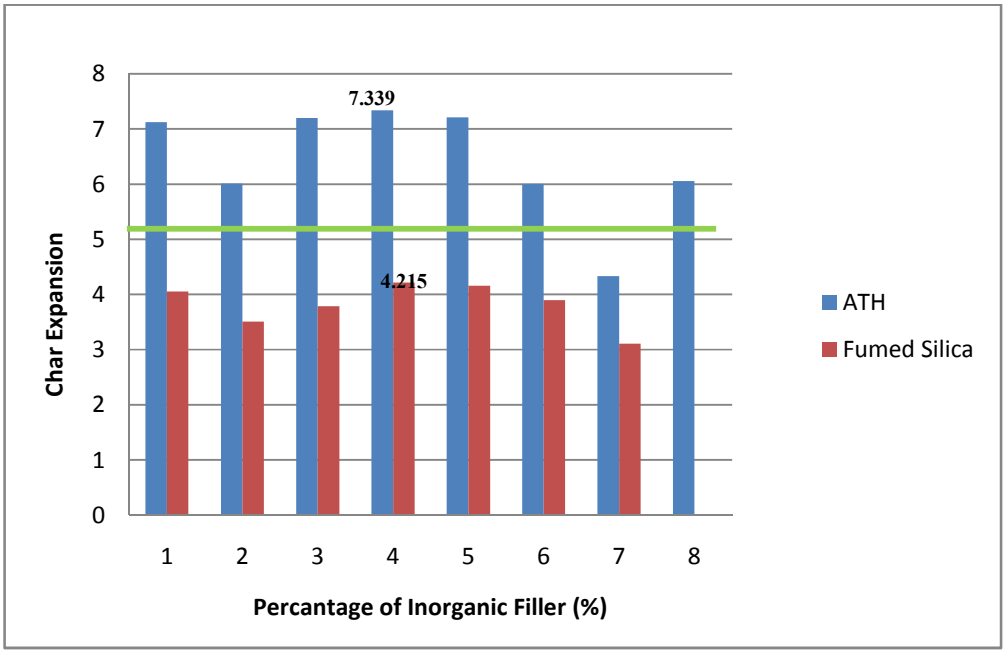


Figure 4.2.1: Comparison of reference, ATH and Fumed Silica of Purchase Graphite on Furnace Test

Based on the figure above, shown that the char expansion of fumed silica is below the reference value. On the other hand, the char expansion of ATH is highly above the reference value. However, both inorganic fillers shown adding 4% of inorganic filler give the highest value of char expansion. Hence, the next sample for treated graphite had been selected. The new samples were adding 2%, 4% and 6% of inorganic filler. Where, O_1 is as reference, A_1 until A_3 is intumescent coating with ATH and F_1 until F_3 is intumescent coating with Fumed Silica

Table 4.2.2: Char Expansion for Treated Graphite

Formulation	Amount of Inorganic Filler	Char Expansion
O_1	0%	1.044
A_1	2%	0.265
A_2	4%	1.225
A_3	6%	0.512
F_1	2%	0.250
F_2	4%	1.176
F_3	6%	0.106

In order to analysis the characteristic of char expansion of each formulation for treated graphite, a graph has been plot. The green line is representing reference value which is O_1. The graph is shown below

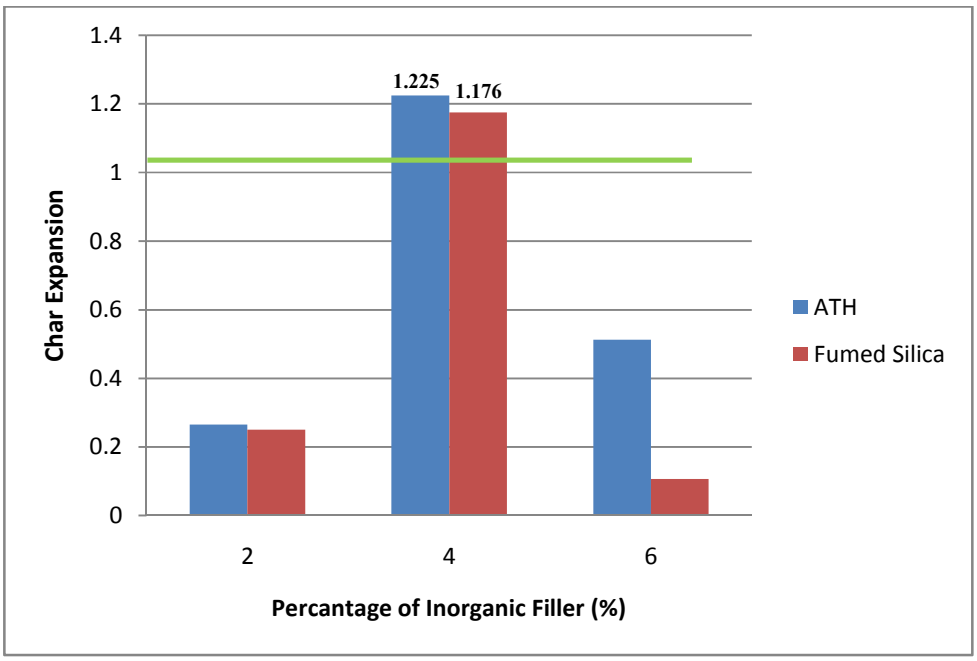
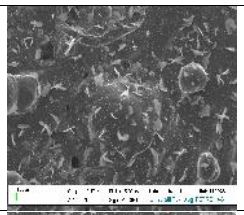
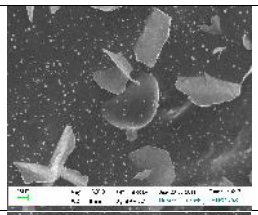
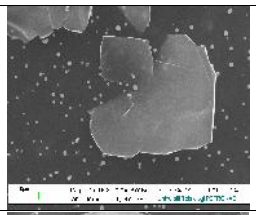
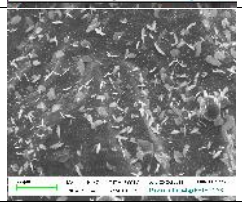
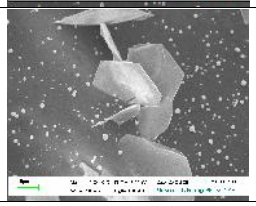


Figure 4.2.2: Comparison of Reference, ATH, Fumed Silica for Treated Graphite on Furnace Test

As shown on the graph above, the 4% of Fumed Silica and ATH is above on reference value. However, 2% and 6% of Fumed Silica and ATH is below than the reference value. This shown 4% of inorganic fillers has good char expansion.

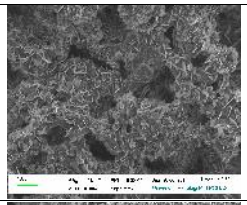
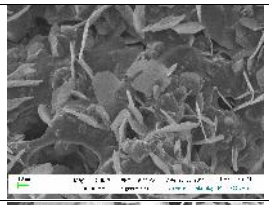
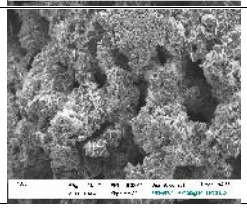
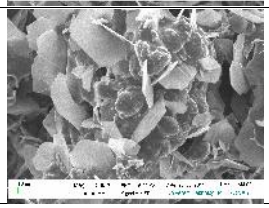
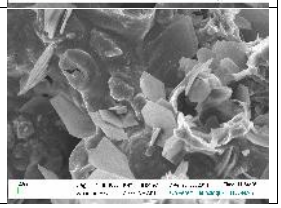
In order to know the how char expansion could take place, some samples were taken to the Scanning Electron Microscope, SEM to see the structure of the samples. Here, the best char expansion sample had been chosen. The sample are A_2 and F_2. Inner and outer layer of the sample had been magnified from 100 until 1000 magnification. The result shown below;

Table 4.2.3: Outer Layer of A_2 and F_2

Formulation	Magnification		
	100	500	1000
A_2			
F_2			

Here, as can been seen on table above, there are some flaw that occurs on both samples. On A_2, there is several hole at outer layer of coating as SEM magnified 100, meanwhile for F_2, there is internal crack occurs on the outer layer of coating, between the surface outer layer and carbon particle. Thus, those flaw had made the swelling process are not efficiently for both samples.

Table 4.2.4: Inner Layer of A_2 and F_2

Formulation	Magnification		
	100	500	1000
A_2			
F_2			

Based on the table above, has give a good indication for this project, where both sample poses a span structure. A span structure contains tiny hole that helps for char expansion and to be fulfilling by gases due to chemical reaction. On of the gases is Carbon Dioxide, which reacts with Expandable Graphite and Oxygen

during burning process. Hence, it is proven that, intumescent coating can act as thermal barrier. However, as the SEM magnified deeper into detail, there is an internal crack occurs on F_2 sample. Thus, this shown the reason why char expansion of Fumed Silica is much lower than ATH.

On the others hand, for the Fire Bunsen Burner Test result shown that A_2 and F_2 give a good characteristic, where the temperature are not exceed the temperature of O_1. However, when a visual inspection were done on both sample give a different characteristic, where on F_2, the coating was detached when fire reach at its highest temperature.

Moreover, the characteristic of shielding effect that have an approximately constant temperature after reach its highest temperature is due to the chemical reaction occurs during burning process. As the result, the Boron Oxide was form and Carbon Dioxide was released. Thus, it helps as a barrier for the coating and yet it saves the steel substrate from being exposed to the direct fire. Besides that, for non affected zone, the differences of initial and final were calculated. Thus, it can be summarized as below.

Table 4.2.5: Percentage of Difference in Thickness.

Formulation	**Percentage of Difference, %**
O_1	0.68
A_1	0.17
A_2	3.77
A_3	2.22
F_1	4.13
F_2	18.43
F_3	-
Ref	1.17

An assumption can be made for non affected zone is, it is can be negligible, since the percentage of difference is small and yet less than 5%. However, for F_2 is 18.43% and F_3 is unknown value because of, during the fire test, the coating were detached partially and fully. Thus, the measurement process cannot be taken perfectly.

In order to ensure, both elements which is Carbon Dioxide and Boron Oxide contain during fire test. Hence, XRD had been done in order to obtain composition in the coating. The sample A_2 and F_2 had been chosen as their shown better heat shielding than others formulation. The result of XRD shown below;

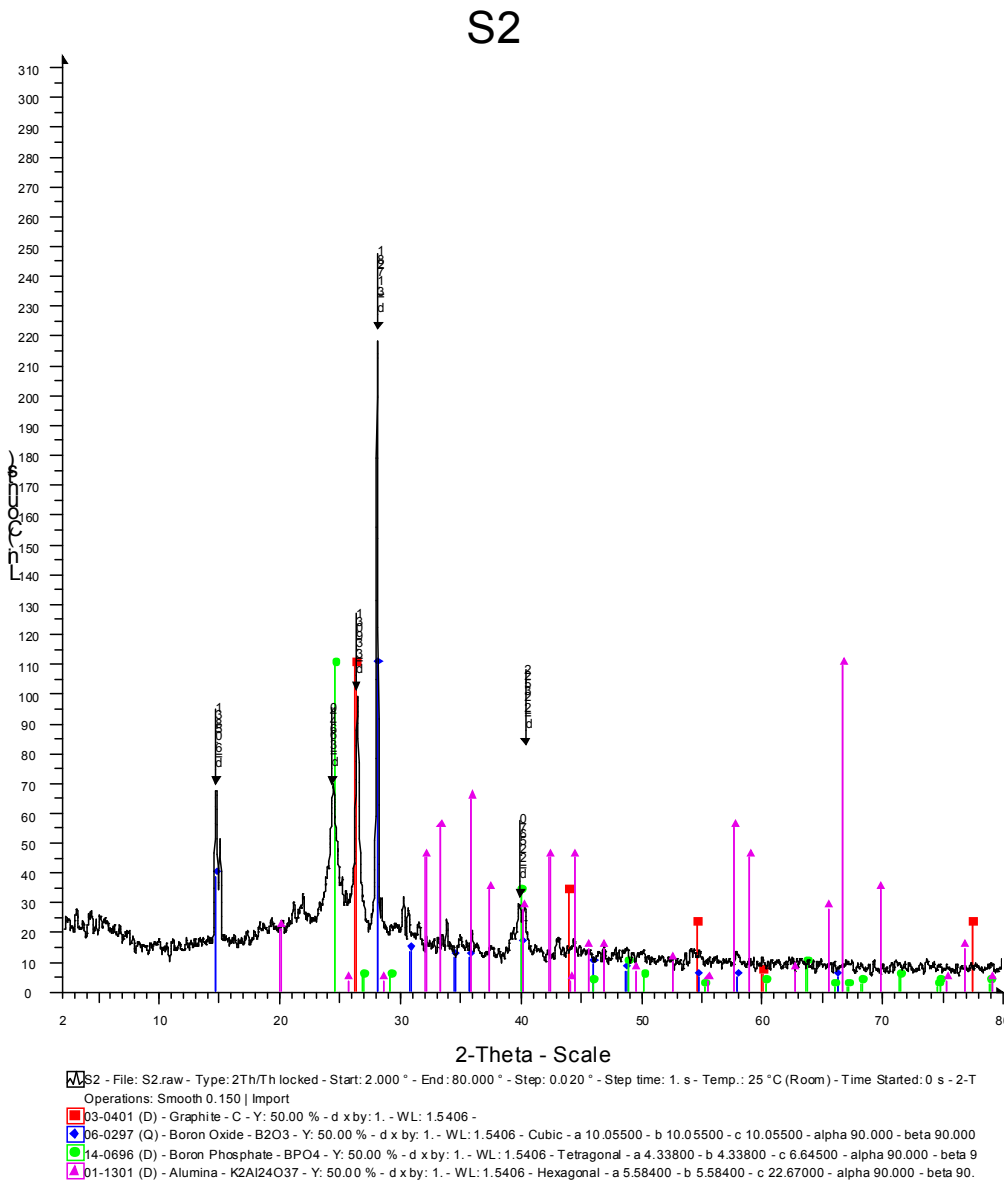


Figure 4.2.3: XRD Result for A_2

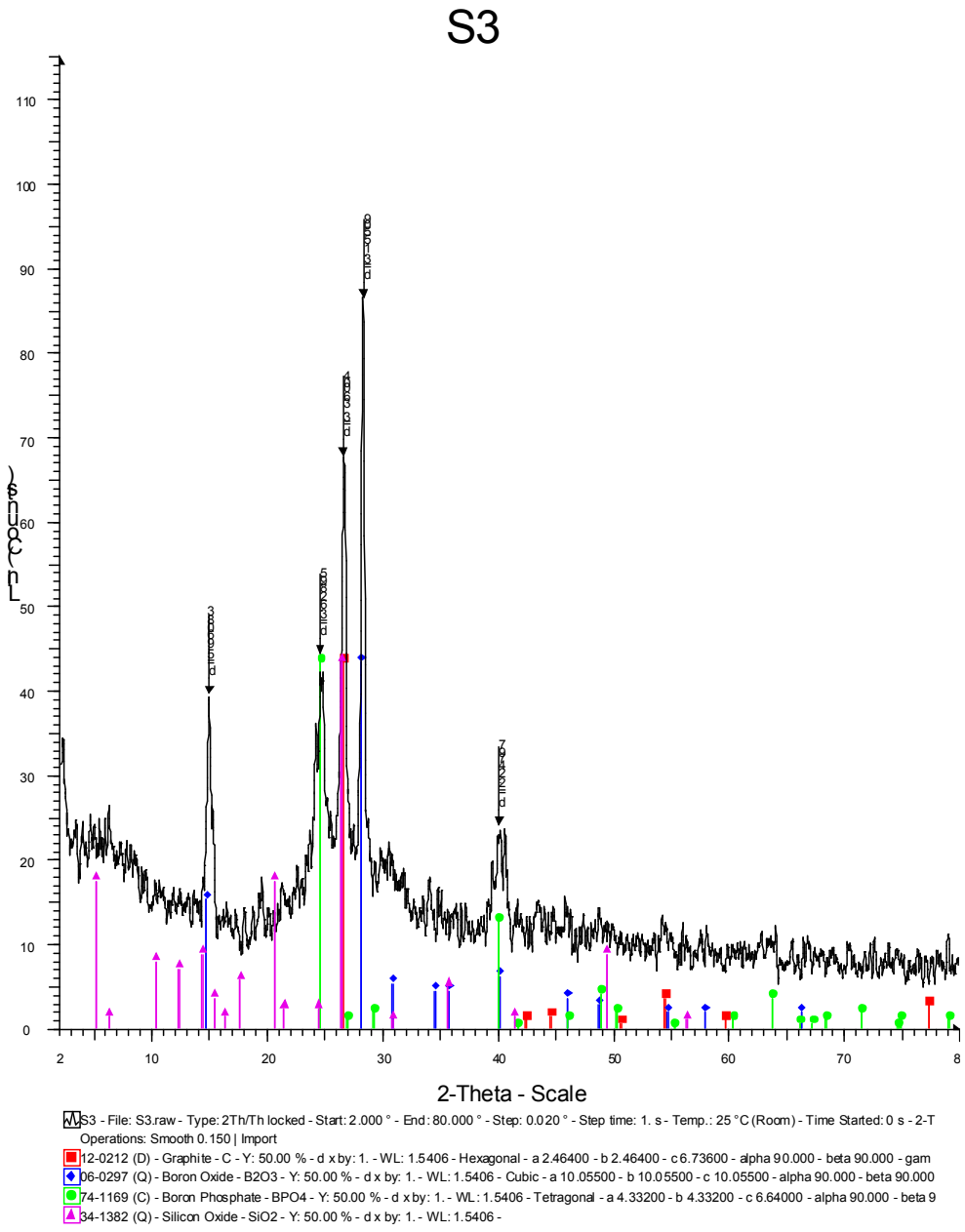


Figure 4.2.4: XRD Result for F_2

Based on two figure above, it is proven that both sample poses element of Boron Oxide, Carbon and yet Boron Phosphate that helps intumescent coating ac as thermal barrier.

For the Shear Test, there are different crack characteristics has been found from visual inspection. Thus, it gives a different load and elongation in the result. The final shear test result shown on the graph below;

Table 4.2.6: Final Result of Shear Test

Formulation	Load (kN)	Elongation (mm)
O_1	7.427	1.892
A_1	1.124	0.678
A_2	2.623	1.104
A_3	0.464	0.480
F_1	1.046	0.625
F_2	1.476	0.819
F_3	0.559	0.550

Based on the result, a graph could be plot in order to seen a comparison between two different inorganic fillers. The green line is representing reference value which is O_1. The graph as shown below;

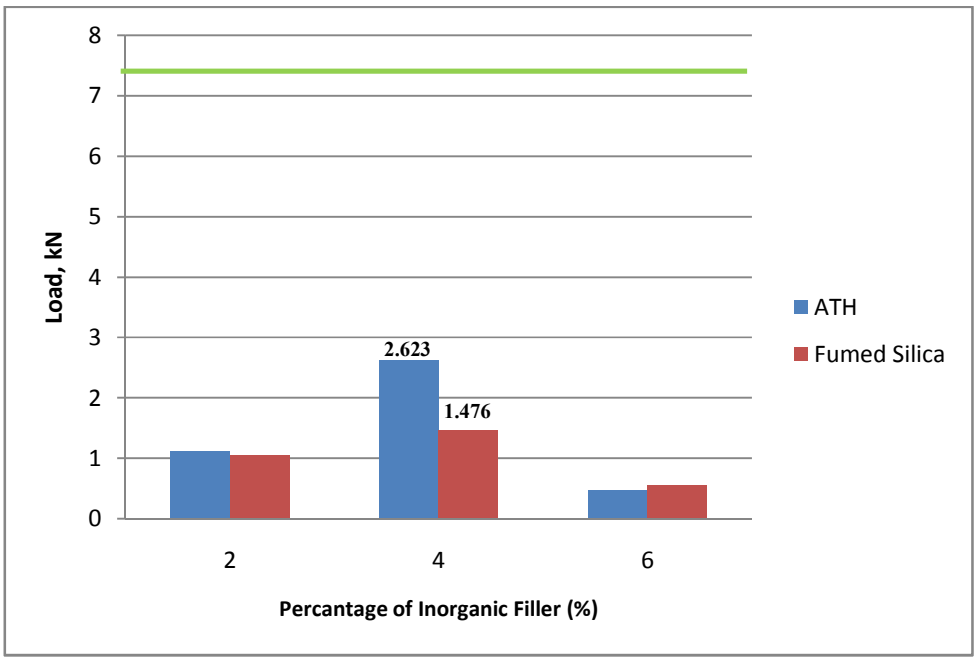


Figure 4.2.5: Comparison of Reference, ATH, Fumed Silica for Treated Graphite on Shear Test

Here, both of the inorganic fillers could not withstand and achieve the load been applied as the reference sample. The difference is about 5 kN for ATH and 6 kN for the Fumed Silica.

On the Figure 4.2.5 also, both of the inorganic fillers shown low result shear test than the reference value. This is because of, the mixture of epoxy and hardener is not well mix with the inorganic filler. Moreover, the final mixture samples of inorganic filler are not well dried as the reference sample. The mixture of reference need 3 weeks to completely, however, the inorganic filler mixture need more than that.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.0 CONCLUSION AND RECOMMENDATION

Based on the schedule and progress of the project, the project currently on the right track, although there are some activities are not meet the target of schedule and key milestone, due some internal and external problem. Moreover, the intumescent coating give a positive result as it is has gone through some test and analysis. Hence, a conclusion can be made, as all the activities are done and give a good feedback to do a conclusion. As the result, intumescent coating with ATH give a better performance than Fumed Silica and yet 4% of ATH gives the best performance than others formulation in term of bonding strength. This is proven by the test that been conducted, where on Furnace Test, give highest char expansion, Fire Bunsen Bunner Test gives the lowest heat shielding effect without detachment problem. On Shear Test, although it does not withstand higher load than reference value, yet it is the highest among others. Furthermore, on advanced analysis using SEM and TGA, yet 4% of ATH give good result where it has less internal crack inside the structure and have high residual weight for better in anti oxidation and thermal stability characteristic.

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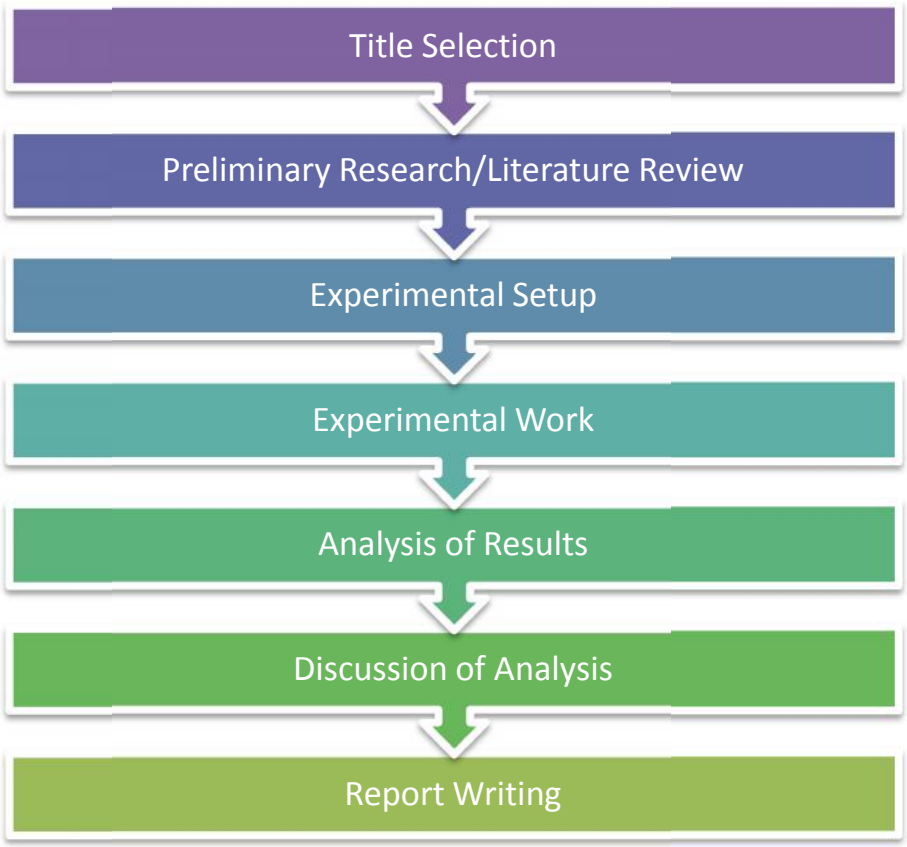
APPENDICES

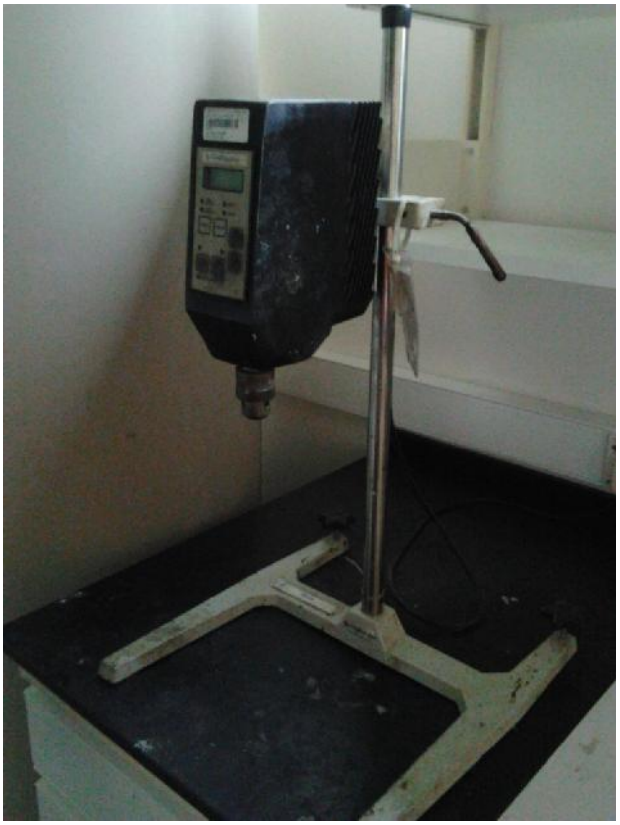
Appendix I: Flow Chart of the project



Appendix II: Micro Weighing Scale

Appendix III: Grinding Machine

Appendix IV: High Shear Mixter Machine



Appendix V: The Intumescent Coating with ATH