Effects of Ultraviolet and Rain on the performance of Fire Retardant Coating

By

Mohd Munir Effendy Bin Mohd Puad

14595

Dissertation submitted in partial fulfillment of

The requirements for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

SEPTEMBER 2013

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CERTIFICATION OF APPROVAL

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Approved by,

…….....

(A.P. Dr Faiz Ahmad)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

SEPTEMBER 2013

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 excepts as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

…….

MOHD MUNIR EFFENDY BIN MOHD PUAD

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ACKNOWLEDGEMENT

First of all, I would like to express my deepest gratitude to almighty Allah, the Most Merciful and Compassionate for blessing me strength, health and willingness to prevail and finish this project.

I would like to thank my supervisor Assoc. Prof. Dr. Faiz Ahmad for accepting me under his supervision and providing guidance, encouragement and ideas to assist me accomplish this project. It was Dr. Faiz who introduced me in the field of Intumescent Fire Retardant Coating and continued to help me in every stage to obtain deep knowledge in this field.

Furthermore, my utmost appreciation and gratitude is also extended to graduate assistance, Mr Hammad and Mr Zia for the dedication of their time and effort for teaching and guiding me despite their other commitments need to oblige.

A very special note of appreciation goes to my lovely family for their constant help, encouragement and supports throughout the period of performing this project. I would like to express my mechanical engineering colleagues for contributing and sharing ideas throughout the project been done in UTP.

Abstract

The fire protection of structural materials has become an important issue in the construction industry. The use of intumescent coating is one of the most effective ways to protect different types of substrate against fire including metals polymers, textiles and wood. This project is focusing on the performance of intumescent coating before and after being exposed to Ultraviolet and Rain. Intumescent coating is an insulating material designed to decrease heat transfer to a substrate structure. In the intumescent fire retardant (IFR) coating char thickness and its strength play a vital role to protect the base steel structure from the fire. The intumescent coatings were well formulated by using three 'active' ingredients which are ammonium polyphosphate (APP) as acid source, expandable graphite (EG) as carbon source, melamine as blowing agent, and epoxy as a binder. The coating were put in weathering chamber for Weathering Testing. A range of different formations were prepared to study the heat shielding effect and char expansion after fire test. The intumescent coating was tested at 800°C for one hour in the furnace and found to be very stable and well bonded with the steel substrate .The coating was characterized with thermogravimetric analysis (TGA) ,Fourier Transform infrared spectroscopy (FTIR),Gas chromatography mass spectrometry (GCMS) Scanning Electron Microscopy (SEM). The morphology of chars were studied by SEM on the coating after fire testing. The result showed that by exposure of intumescent coating to UV and rain will reduce its performance in char expansion as well as it degrades.

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CHAPTER 1

INTRODUCTION

1.0 Project background

Intumescent coatings represent an important class of passive-fire proofing materials which concern insulating systems designed to decrease heat transfer from a fire to the substrate being protected. It has been used in the fire protection of steel structure for more than 20 years [1]. Adding inorganic filler as one of the formula into an intumescent fire retardant has attracted more and more interests in both research and industry circles in recent years. Some intumescent, however, are susceptible to environmental influences such as Ultraviolet (UV), heat and humidity which can reduce or negate their ability to function. Intumescent fire retardant coating was found to lose the majority of their capability due to leaching out of fire active ingredients in reaction to weathering [2].

1.1 Problem statement

Intumescent fire retardant coating present as a mitigation to insulate and protect the substrate under the action of fire at high temperature. However, the issue of poor weathering properties of intumescent fire retardant coating has been raised recently. According to a research, the molecular structure of the coating can easily break on exposure to ultraviolet rays as well as in moisture condition [1]. Thus an assurance of intumescent fire retardant coating to provide the superior protection to the substrate, the coating must withstand to the challenging environment like exposed to UV rays and rain.

1.2 Objective

The objective of the project is to study the effect of UV and rain on the performance of fire retardant coating for on their structure characteristics. This project also aims to study the fire retardant performance of intumescent coating before and after exposure to UV and rain. Besides this project also aim to examine the changing in its color.

1.3 Scope of study

i) Preparation of intumescent coating without filler

ii) Preparation of intumescent coating with inorganic filler such as Mica

iii) Furnace test and Bunsen burner test before of intumescent coating before exposure to UV and rain

iv) Furnace test and Bunsen burner test before of intumescent coating after exposure to UV and rain

v) Scanning Electron Microscopy (SEM) technique for coating with and without filler.

vi) Determine residual net of the coating using TGA analysis

The effects of UV and rain on performance of the intumescent coating are studied in this project. Varieties percentage of filler was used in preparing the intumescent coating. The intumescent coating also was exposed to UV and rain by using weathering chamber for 2 weeks. Fire test such as furnace test for char expansion and Bunsen burner were conducted for the examination of heat insulation of the coating. For the char morphology, the char residue is examined by using Scanning Electron Microscopy (SEM).

CHAPTER 2

LITERATUR REVIEW

2.1 Intumescent coating

Intumescent coating represents an increasingly used way to provide passive fire protection to the structural steel in modern architectural design, whilst at the same time maintaining aesthetic appearance. Intumescent can be defined as the swelling of certain substances on exposure to heat [2]. Intumescent coating is also known an insulating system designed to decrease heat transfer from a fire to the structure that is being protected so that the structure can maintain its integrity within one to three hours for evacuation process [3] .It is also acts as 'thermal barrier' which effectively protects the substrate against high temperature [4]. A protective carbonaceous char is produced when intumescent coating is subjected to heat which acts as insulative barrier between the fire and substrate [5].

2.2 Main components of intumescent coating

There are four important components in preparing the intumescent coating [6]:

1) Acid source

   Normally acid source like Ammonium Polyphosphate (APP) is used as a source of phosphoric acid which acts a catalyst to speed up the formation of carbonaceous char and NH_3 which improves the swelling of char [7].

2) Carbon source

   Expendable graphite (EG) will actcas the carbon source as it has low weight percentage which makes the swelling of the char even better. Expandable Graphite (EG) is also a new invention of intumescent fire retardant coating additive. It can give the good fire retardancy to various materials [3].

3) Blowing agent

   Blowing agent like Melamine (MEL) is used in preparing the intumescent coating as it can improve the char strength and the adhesion of char to the steel substrate [3].

4) Binder

   An Epoxy resin acts as a binder to make the compounds contact each other [3].

The good combination between these components is very crucial in order to produce a very excellent fire retardant which will give a high expansion of char with good mechanical resistance in protecting the substrate from losing it structural properties.

2.3 Mechanism of char swelling

The mechanism of the char swelling begins with the breaks down of the acid source in which result into mineral acid. Next the process of dehydration of the carbonization agent to yield the carbon chars. Finally is the production of gaseous from the decomposition of the blowing agent. From the previous occurrence, it will then cause the char to swell in which later provide an insulating multi-cellular protective layer as shown in Figure 1. Consequently, this shield limits the heat transfer from the heat sources to the substrate and the mass transfer from the substrate to the heat source resulting in a conservation of the underlying materials. The flame-retardant coating fire resistance is mainly depends on the creation or formation of char which initiated by the reaction of Ammonium Polyphosphate (APP), pentaerythritol and melamine (MEL) as shown in Figure 2 [7].

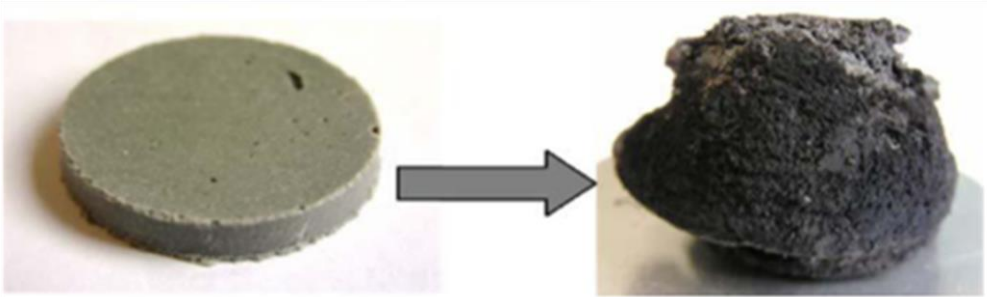


Figure 1: Swelling of intumescent coating [7].

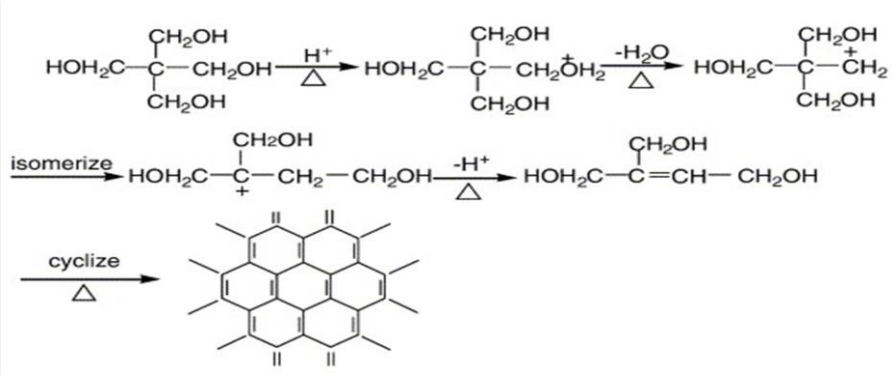


Figure 2: Reaction of char formation [1].

2.4 Filler

There is one of inorganic filler with different weight percentage that is used in preparing intumescent coatings, Mica.

2.4.1 Mica

Mica will be used as filler to prepare the intumescent coating. According to a study, the presence of mica in a polymer can increase the rigidity, heat resistance and dimension stability. Besides that, mica also has the capability to improve the stiffness to the mechanical properties of the structure. It has the following chemical formula, $H_2KAI_3(Si0_4)_3$. The density of Mica is 986 kg/m-cube. Mica also resistant to thermal decomposition at temperatures above 500° C. Below are the other characteristics of mica [10]:

Table 1: Properties of Mica [10]

Physically	Chemically
Mica has a transparent, optically flat, easily splittable into thin films along its cleavage, colourless in thin sheets, resilient and incompressible. They can remain tough and elastic even at high temperature when split int thin film	Mica is a complex hydrous silicate of aluminium containing potassium, magnesium, iron, sodium fluorine and or lithium and also traces of several other elements, It is also stable and completely inert when exposed to water, acids (except hydro-fluoric and concentrated sulphuric),

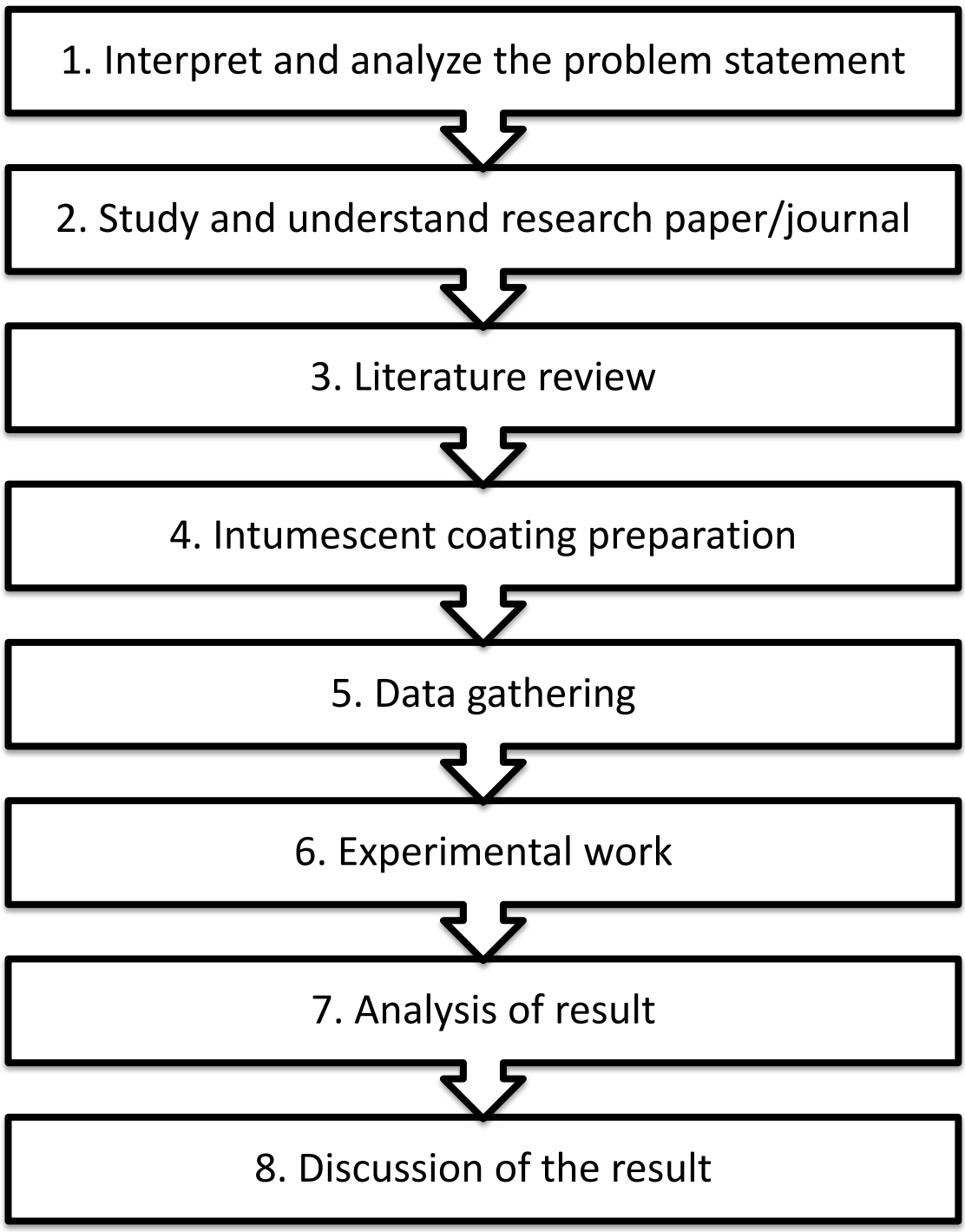
	alkalies, conventional solvents, oils and is virtually unaffected by atmospheric action
Thermally	**Mechanically**
Mica has properties of fireproof, infusable, incombustible and non-flammable and can withstand temperatures of 600° C to 900° C depending on the type of mica. It also has low heat conductivity, good thermal stability and could exposed to high temperatures without noticeable effect	Mica is soft and can be hand-cut, machined or die-punched. It is flexible, elastic and tough , it also has high tensile strength. It can withstand great mechanical.

Figure 3: Mica powder [10]

CHAPTER 3

METHODOLOGY

3.1 Project Flow Chart



3.2 Explanation on the project flow

1. Interpret and analyse the problem statement: This is the stage where understanding the problem statement as well as the title selected

2. Study and understand research paper/journal: Find journals and extract important things from it.

3. Literature review: Study on the intumescent coating into details in order to have a deep knowledge in the coating

4. Intumescent coating preparation: Formulate the intumescent coating with different ingredients percentage

5. Data gathering: Collect all the data based on formula of intumescent for result analysis

6. Experimental work: Several parameters are needed to conduct the experiment. The various substrates will be exposed to the different type of weather like UV and rain, then will undergo the furnace test

7. Analysis of result: Any changes in thickness of the coating, colour and composition will be analysed.

8. Discussion of the result Based on the result obtained further study on the intumescent will be investigated.

3.3 Intumescent coating preparation

3.3.1 Substrate preparation

The steel (carbon steel) substrate with size of 5cm x 5cm and 10cm x 10cm with the thickness of 2.5mm will be prepared by using shear cutting machine. The steel must be sand blasted in order to clean the corroded part as well as to improve the adhesion properties to the steel substrate.

Figure 4: Sand-blasted steel

3.3.2 Intumescent Formulation

The intumescent coating has a range of formula which comprises of **Ammonium Polyphosphate** (APP), **Expendable graphite** (EG), **Melamine** (MEL),Boric acid (BA) ,Fillers (Mica and Titanium dioxide), Binder (Epoxy) and Hardener(TETA)

Table 2: Intumescent coating formula

Ingredient	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
APP (%)	11.11	11.11	11.11	11.11	11.11	11.11
EG (%)	5.56	5.56	5.56	5.56	5.56	5.56
MEL (%)	5.56	5.56	5.56	5.56	5.56	5.56
BA (%)	11.11	11.11	11.11	11.11	11.11	11.11
FILLER (MICA) (%)	0	1	2	3	4	5
BINDER (%)	44.44	43.78	43.12	42.46	41.80	41.14
TETA (%)	22.22	21.89	21.56	21.23	20.90	20.57

APP, BA and melamine are weighed according to the formulation. All these materials are grinded to make sure they are homogenously mixed. EG is weighed then combined with the grinded material. This is called as ingredient A. TETA and BPA are weighed based on the formulation. By using mixer, the binder is mixed for about 5 minutes. Ingredient A is poured into the binder then they are mixed again homogenously. The coating is applied on the structural steel until it covered all the surface of the substrate. The coating is cured in the room temperature for 2 days. The thickness of each sample is measured and the data is recorded. For coating using filler, step number 1-9 is repeated but the filler is added in the first step. The step and formulation is same when use Titanium dioxide as filler.

3.3.3 Steps in preparing intumescent coating

In preparing the intumescent coating, usually there are three 'active ingredients are needed to be combined together. Acid source or mineral, **Ammonium Polyphosphate** (APP) will be one of the important ingredients. **Expendable graphite** will act as carbon source while **Melamine** (MEL) as the blowing agent and boric acid as additives. Two different of filler will be used which are **Titanium dioxide** and **Mica** in order to improve the intumescent coating performance. **Epoxy** will be used as binder. Below is the diagram that shows the step to prepare the intumescent coating [2].

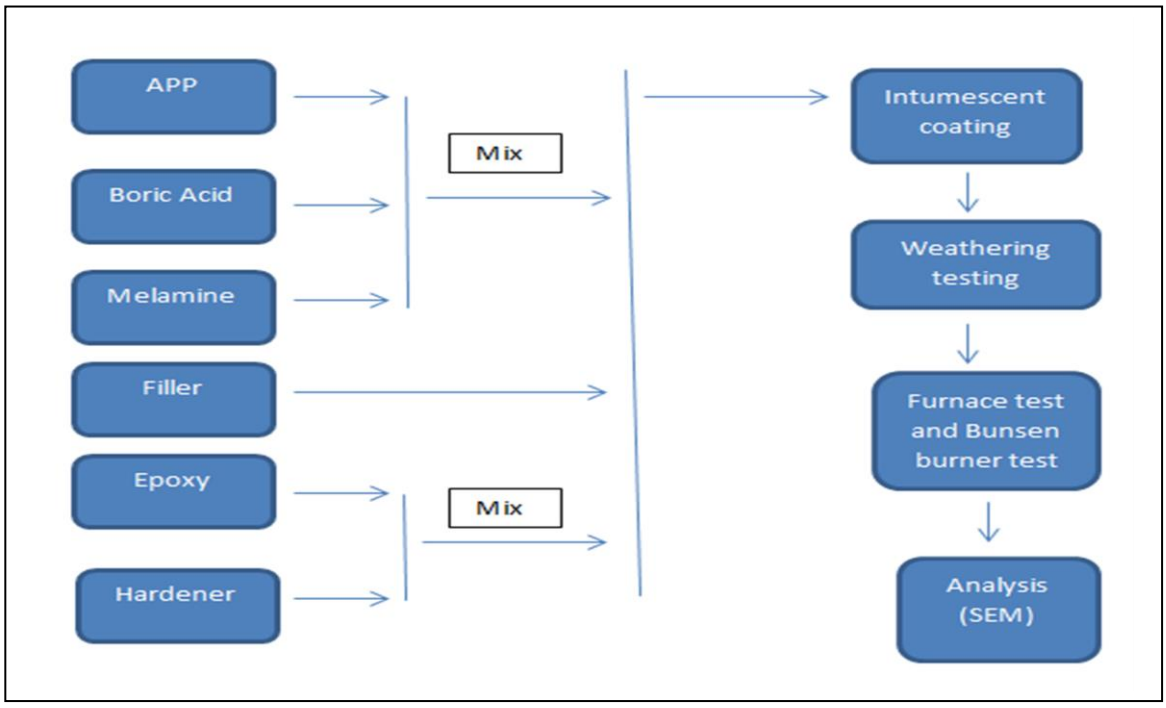


Figure 5: Coating preparation [2]

3.4 Weathering testing (Accelerated Testing)

The sample were put in the weathering chamber with two different environments. The substrate will be left in the weathering chamber and initial data will be recorded. The sample will be put in the weathering chamber and a test is based on **ASTM D 4587-91 Standard**.

- Cycles of water spray and drying (spraying water 18 min per 2h) with exposure UV radiation. Temperature and relative humidity are 50±5° c and 70±5 %.
- Light of xenon arc lamp used as UV rays with intensity of 550 W/m K.
- The substrate were exposed at a slope 18°.
- Test for 2 weeks (336h).

The sample will be monitored once a week and any change in colour, structure and weight will be recorded.

3.5 FURNACE TEST

The samples were tested by using furnace test for char morphology. The samples were left approximately two hours during this test. The chamber was set with temperature of 450°C to burn the samples. There were 3 phases of this process. For the phase 1, the temperature was rising about 30 minutes to reach 450°C. Then the process proceeded to the phase 2 which was the dwell of the temperature for about 30 minutes in order to burn the samples with perfect. The last phase was the decreasing of the temperature to the initial back which takes about 50 minutes for the chamber to cool down the sample to avoid cracking of char.

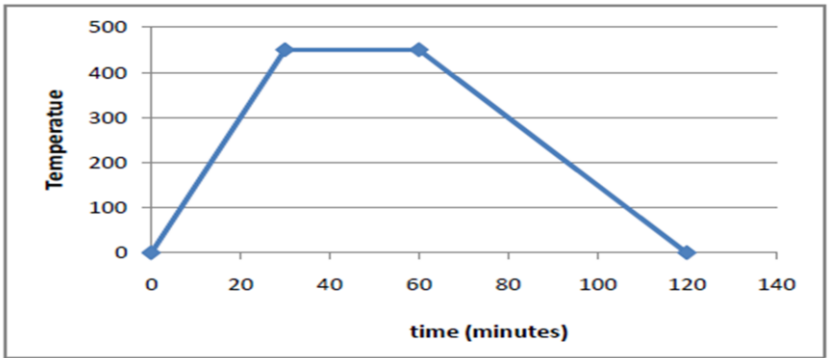


Figure 6: Temperature vs. Time Plot for sample

3.6 GCMS TEST

After the samples had dried completely, the residues of the coating at the edges of the samples had been cut off and grinded until it become powder. After that, the powder of the coating had been sent to the Gas Chromatography Mass Spectrometry (GCMS) laboratory to undergo the GCMS test using a PYRO-CHEM WILKS pyrolyser, Hewlett Packard 5890 Series II gas chromatograph and TRIO 1000 mass spectrometer. The sample with weight of 10 mg was placed in the pyrolyser. The pyrolysis process was carried out at 25oC to 800oC in 10 sec. Then the pyrolysis products in the gas phase were injected into ion detector column to characterize the gaseous products. The GCMS is shown in the figure below.

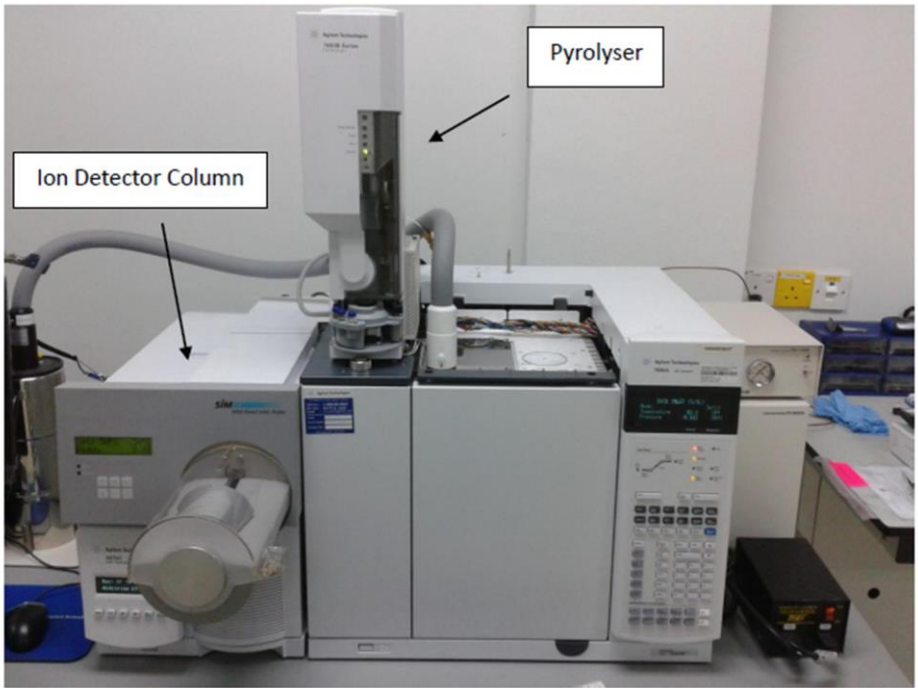
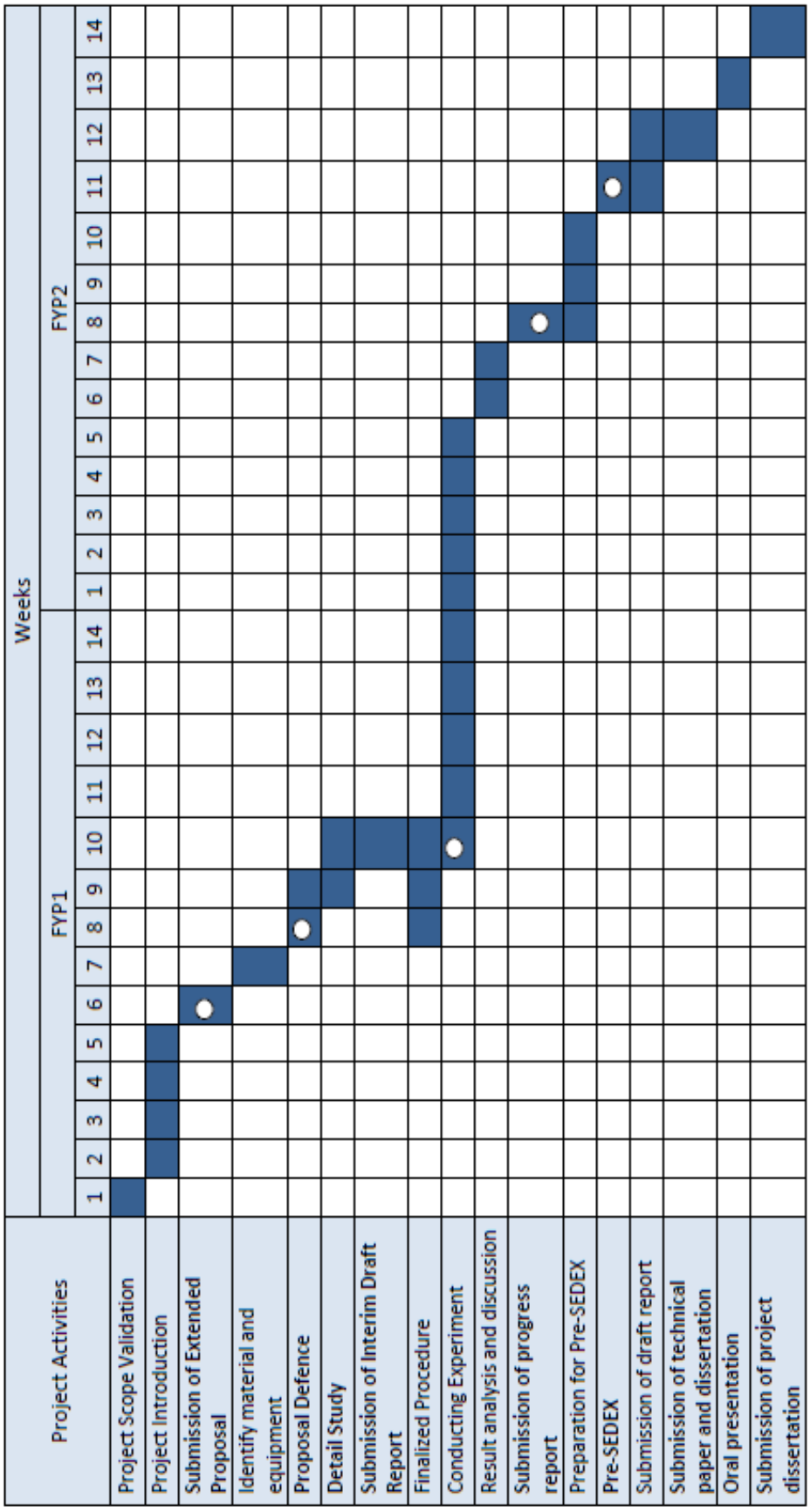


Figure 7: The Gas Chromatography Mass Spectrometry Machine

3.7 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity.

3.8 Gantt Chart



CHAPTER 4

RESULT AND DISCUSSION

4.1 Work Completed

All the process involved in completing the project has been carry out by the author. The coating was developed based on the formulation later applied to the steel substrate. The steel substrate were put in weathering chamber for Weathering testing. The coated steel then tested for fire retardant performance using Bunsen burner testing. The fire testing result was analysed by using Scanning Electron Microscope (SEM) and Fourier Transform infrared spectroscopy (FTIR).

4.2 Data recorded

4.2.1 Intumescent Coating Using Mica As Filler

Six formulations have been developed with different sizes. In the first stage, Mica is used as filler. The table below shows the details of the formulation

Sample 1

Ingredient	APP	EG	MEL	BA	FILLER (MICA)	BINDER	TETA
Percentage (%)	11.11	5.56	5.56	11.11	0	44.44	22.22

Sample 2

Ingredient	APP	EG	MEL	BA	FILLER (MICA)	BINDER	TETA
Percentage (%)	11.11	5.56	5.56	11.11	1	43.78	21.89

Sample 3

Ingredient	APP	EG	MEL	BA	FILLER (MICA)	BINDER	TETA
Percentage (%)	11.11	5.56	5.56	11.11	2	43.12	21.56

Sample 4

Ingredient	APP	EG	MEL	BA	FILLER (MICA)	BINDER	TETA
Percentage (%)	11.11	5.56	5.56	11.11	3	42.46	21.23

Sample 5

Ingredient	APP	EG	MEL	BA	FILLER (MICA)	BINDER	TETA
Percentage (%)	11.11	5.56	5.56	11.11	4	41.80	20.90

Sample 6

Ingredient	APP	EG	MEL	BA	FILLER (MICA)	BINDER	TETA
Percentage (%)	11.11	5.56	5.56	11.11	5	41.14	20.57

4.2.2 Furnace Fire Test

Below is the thickness of the coating before the fire test:

Table 4.1: Thickness of coating before fire test

SAMPLE	READING 1 (mm)	READING 2 (mm)	READING 3 (mm)	READING 4 (mm)	READING 5 (mm)	READING 6 (mm)	AVERAGE THICKNESS (mm)
S1	3.95	3.97	3.90	3.91	3.91	3.93	3.93
S2	3.72	3.75	3.71	3.83	3.79	3.80	3.77
S3	3.75	3.57	3.68	3.70	3.71	3.70	3.69
S4	3.83	3.76	3.65	3.70	3.77	3.75	3.74
S5	3.56	3.66	3.69	3.69	3.63	3.66	3.65
S6	3.52	3.40	3.61	3.33	3.49	3.55	3.48

After the test, the thickness of the coating is measured as in the table below:

Table 4.2: Thickness of coating after fire test

SAMPLE	READING 1 (mm)	READING 2 (mm)	READING 3 (mm)	READING 4 (mm)	READING 5 (mm)	READING 6 (mm)	AVERAGE THICKNESS (mm)
S1	10.81	11.24	11.08	10.95	11.09	10.94	11.02
S2	13.92	13.72	14.01	13.55	13.16	14.06	13.75
S3	14.27	13.82	14.09	13.95	13.79	14.03	13.99
S4	11.11	12.84	11.67	11.96	12.72	12.06	12.06
S5	14.01	14.50	14.33	14.56	14.25	14.77	14.03
S6	12.20	13.25	12.24	13.01	12.44	12.39	12.59

From the result obtained the analysis has been done to show how much differences occurred and percentage of the expansion in the data below:

Table 4.3: Percentage of coating expansion

SAMPLE	THICKNESS (mm)		EXPANSION	PERCENTAGE OF EXPANSION (%)
	BEFORE FIRE TEST	AFTER FIRE TEST		
S1	3.93	11.02	7.09	180.41
S2	3.77	13.75	9.98	264.72
S3	3.69	13.99	10.3	279.13
S4	3.74	12.06	8.32	222.46
S5	3.65	14.03	10.38	284.38
S6	3.48	12.59	9.11	261.78

From the table, most of the samples expand more than 100%.Below are the graphs show expansion of char as well as the percentage of char expansion for Furnace Test and Bunsen Burner Test.

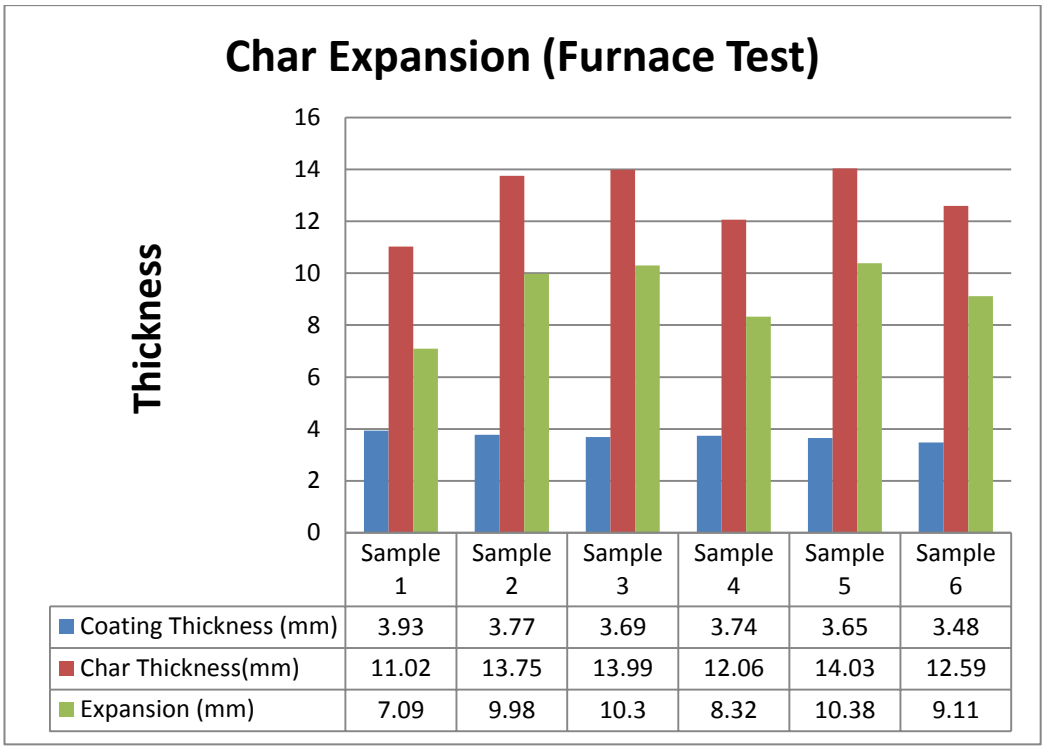


Figure 8: Char Expansion (Furnace Test)

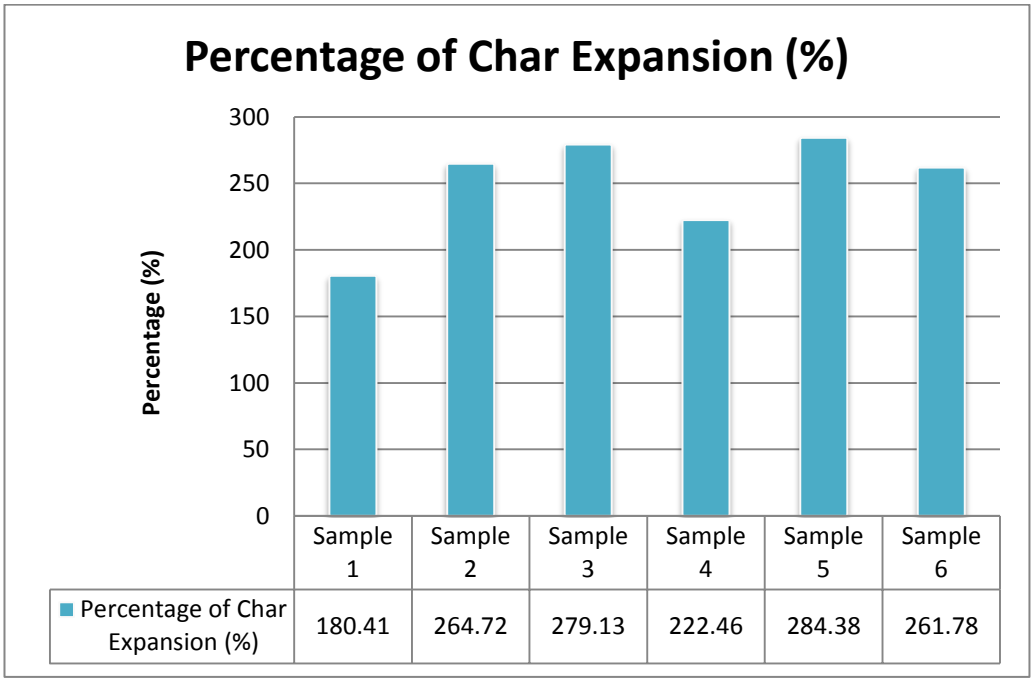
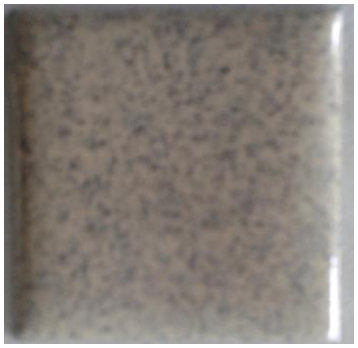
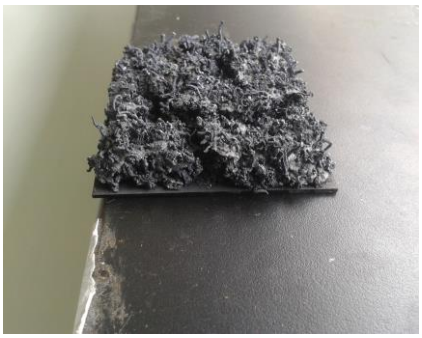


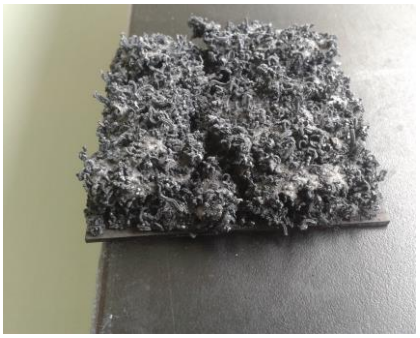
Figure 9: Percentage of Char Expansion (Furnace Test)

Observation to the coating surface has been done after the fire test. The differences before and after the fire test is shown as below:

Table 4.4: Differences of the samples before and after Fire Test

SAMPLE	BEFORE FIRE TEST	AFTER FIRE TEST
S1	 Thickness of the coating = 3.93mm	 Thickness of the coating = 11.02mm Percentage of expansion = 180.41% (2.8 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth
S2	 Thickness of the coating = 3.77mm	 Thickness of the coating = 13.75mm Percentage of expansion = 264.72% (3.6 times of expansion) The coating is detached from the steel. The surface is cracked

S3	 Thickness of the coating = 3.69mm	 Thickness of the coating = 13.99mm Percentage of expansion = 279.13 % (3.8 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth
S4	 Thickness of the coating = 3.74mm	 Thickness of the coating = 12.06mm Percentage of expansion = 222.46% (3.2 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth

S5	 Thickness of the coating = 3.65mm	 Thickness of the coating = 14.03mm Percentage of expansion = 284.38% (3.8 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth
S6	 Thickness of the coating = 3.48mm	 Thickness of the coating = 12.59mm Percentage of expansion = 261.78% (3.6 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth

4.2.3 Fire Testing (Bunsen Burner Test)

Below is the thickness of the coating before the fire test as shown in the Table:

Table 4.5: The thickness of char before Fire Testing (Bunsen Burner)

SAMPLE	READING 1 (mm)	READING 2 (mm)	READING 3 (mm)	READING 4 (mm)	READING 5 (mm)	READING 6 (mm)	AVERAGE THICKNESS (mm)
S1	4.10	4.21	3.88	3.92	4.02	3.90	4.01
S2	4.09	4.05	3.94	4.10	3.91	4.03	4.02
S3	3.69	3.84	3.80	3.72	3.68	3.82	3.76
S4	4.01	4.21	3.89	3.94	3.88	4.11	4.01
S5	3.41	3.43	3.51	3.55	3.45	3.50	3.48
S6	3.53	3.66	3.48	3.57	3.49	3.60	3.56

After the Fire Testing by using Bunsen Burner, the thickness of the coating is measured again as in the table below:

Table 4.6: Thickness of the coating after Bunsen Burner Test

SAMPLE	READING 1 (mm)	READING 2 (mm)	READING 3 (mm)	READING 4 (mm)	READING 5 (mm)	READING 6 (mm)	AVERAGE THICKNESS (mm)
S1	11.33	10.81	10.40	11.04	11.24	10.92	10.96
S2	12.40	13.01	12.55	12.34	12.99	12.78	12.68
S3	13.11	12.76	13.23	13.10	12.91	13.43	13.09
S4	12.67	11.67	11.82	11.49	12.05	12.79	12.09
S5	13.55	13.95	14.09	14.31	13.99	13.85	13.96
S6	11.89	12.78	12.54	13.01	12.44	12.65	12.56

Based on the result obtained from Fire Testing using Bunsen Burner, an analysis has been done to show how much differences occurred and percentage of the expansion in the data below:

Table 4.7: Percentage of Char Expansion

SAMPLE	THICKNESS (mm)		EXPANSION(mm)	PERCENTAGE OF EXPANSION (%)
	BEFORE FIRE TEST	AFTER FIRE TEST		
S1	4.01	10.96	6.95	173.31
S2	4.02	12.68	8.66	215.42
S3	3.76	13.09	9.33	248.14
S4	4.01	12.09	8.08	201.50
S5	3.48	13.96	10.48	301.15
S6	3.56	12.56	9.00	252.81

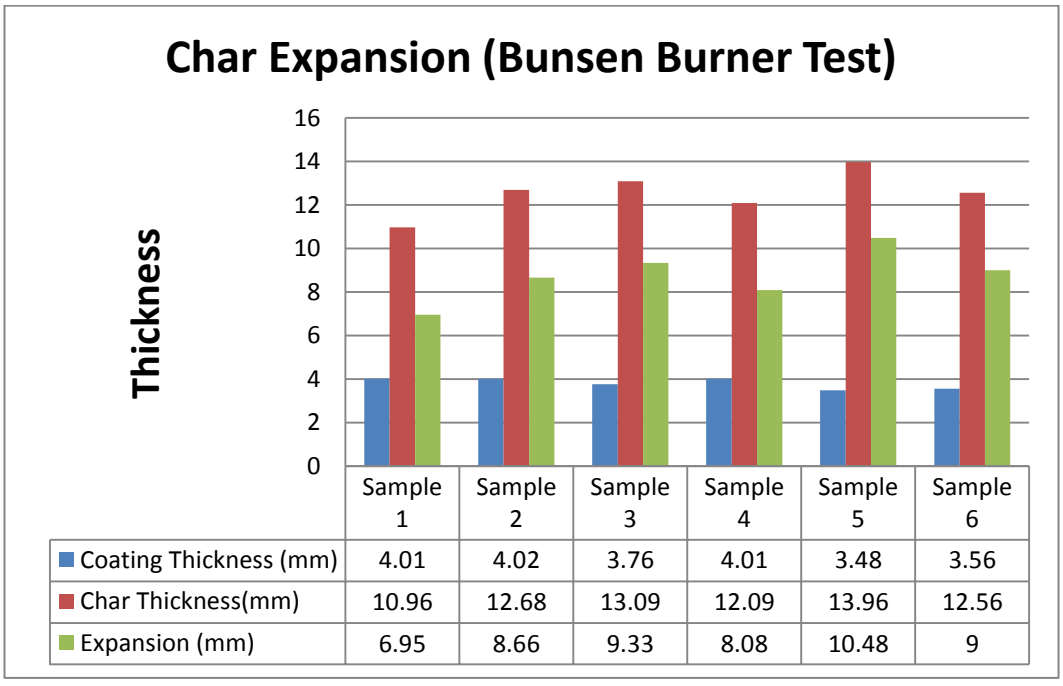


Figure 10: Char Expansion (Bunsen Burner Test)

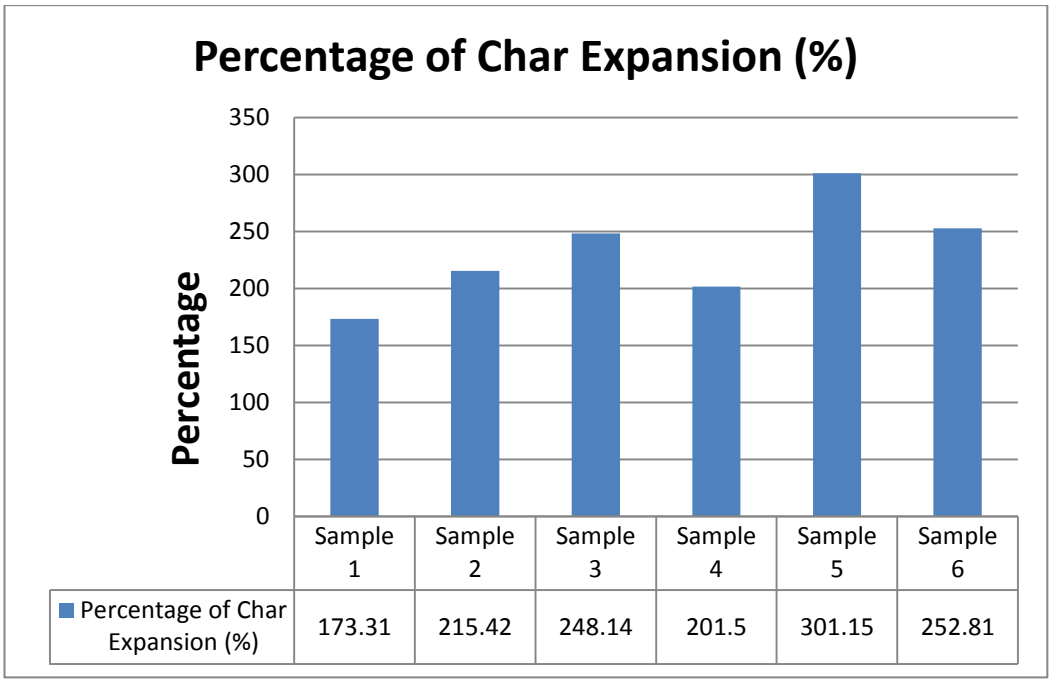


Figure 11: Percentage of Char Expansion (Bunsen Burner Test)

4.3 Heat Shielding Test

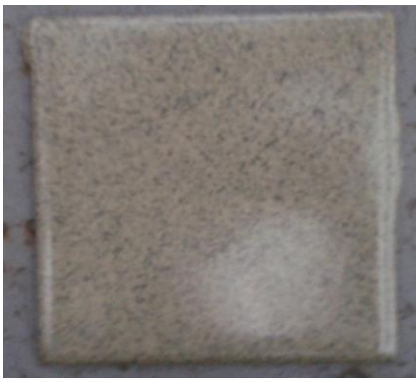
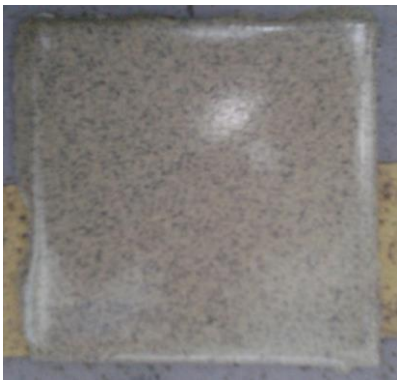
Temperature of the steel substrate is measured during the bunsen burner testing. During the testing, the temperature of the steel substrate is measured using digital thermo logger and thermo couple. Steel critical temperature is 600 °C before its lost the mechanical integrity. The temperature is recorded for every 5 minutes until 60 minutes of testing Observation to the coating has been done after the fire test. The differences before and after the fire test is shown as in Table below:

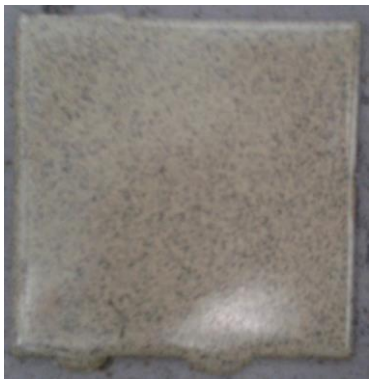
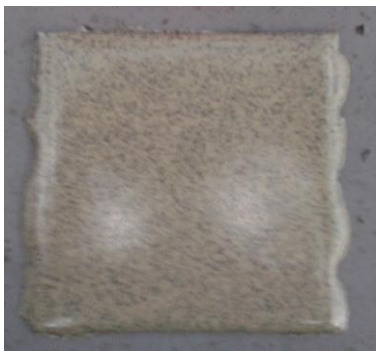
(see appendix A for graph and temperature)

Table 4.8: Final Temperature of the sample for Bunsen Burner Testing

Sample	Final Temperature (degree Celsius)
1	195.66
2	176.44
3	178.33
4	170.12
5	169.59
6	171.22

Table 4.9: Before and after Bunsen Burner Test

SAMPLE	BEFORE FIRE TEST	AFTER FIRE TEST
S1		
S2		

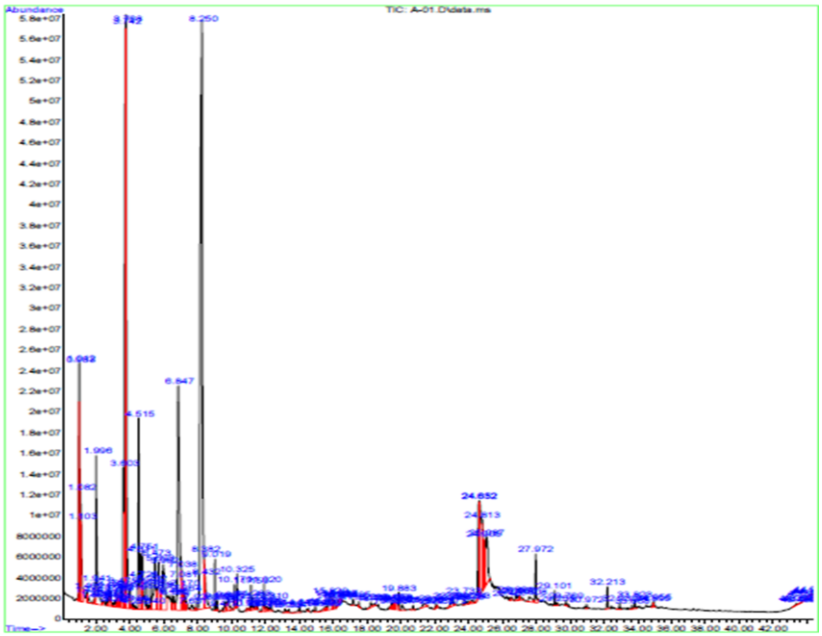
S3		
S4		
S5		

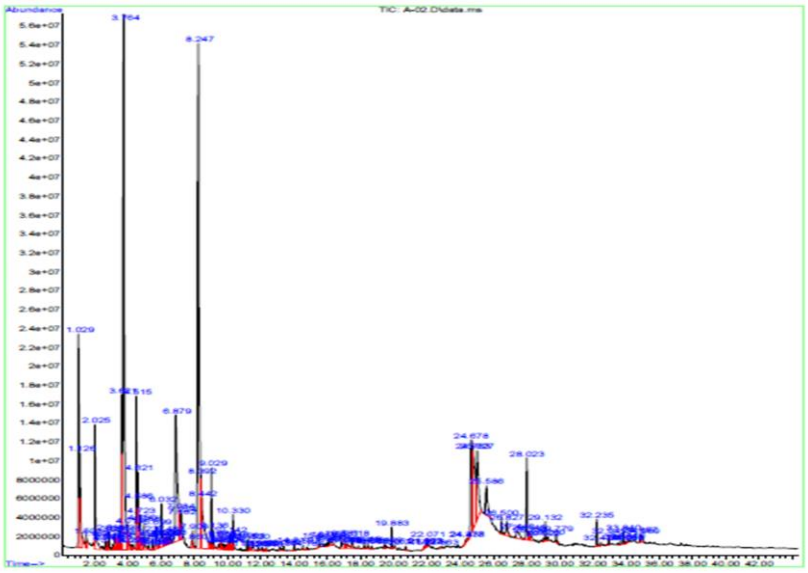
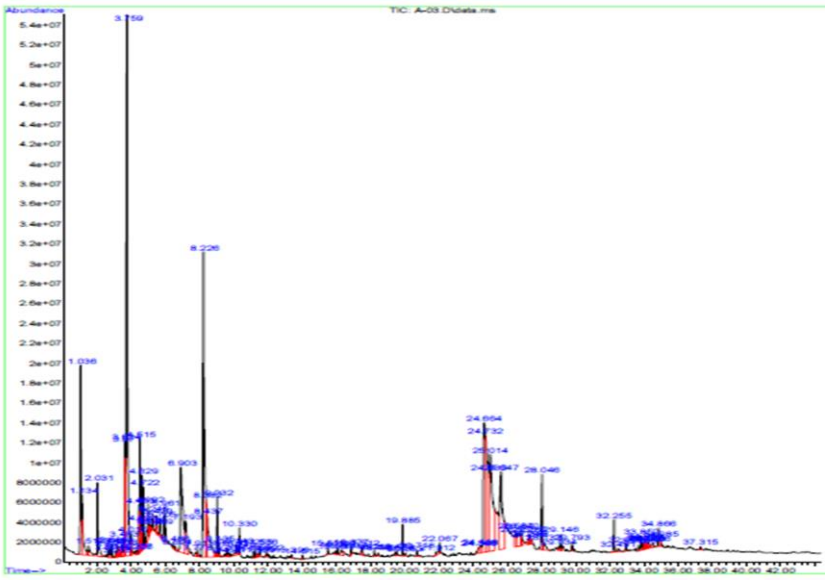
S6		

4.4 GCMS TEST

For GCMS TEST, 3 samples which are Sample 1 (0 % filler), Sample 4 (3 % filler) and Sample 5 (4 % filler) had been went through the testing. The result is as shown below.

Table 4.10: Sample 1 GCMS TEST

Sample	Result
1	

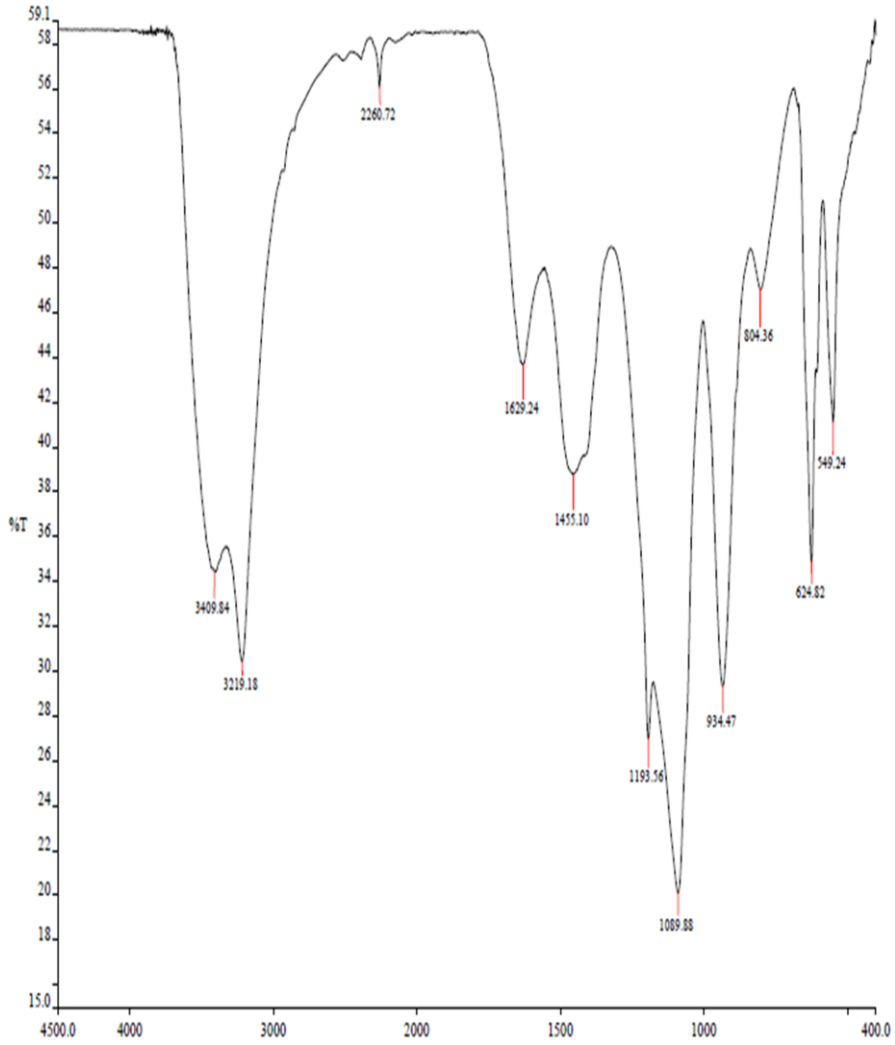
4	
5	

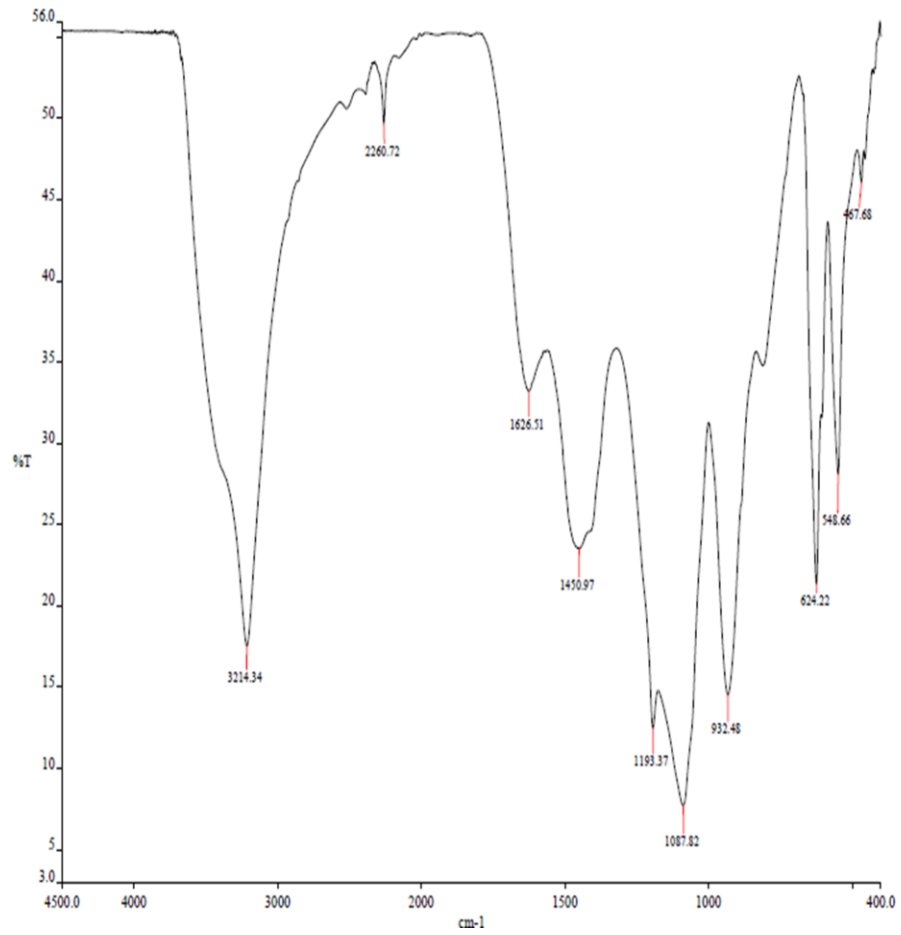
From the result obtained for Sample 1 the highest retention time occurred on 3.743 minute and the compound that exist is phenol. While for Sample 4, the highest retention time occurred on 3.764 where phenol exist in it. For Sample 5 the highest retention time occurred on 3.759 and the compound exist is also phenol

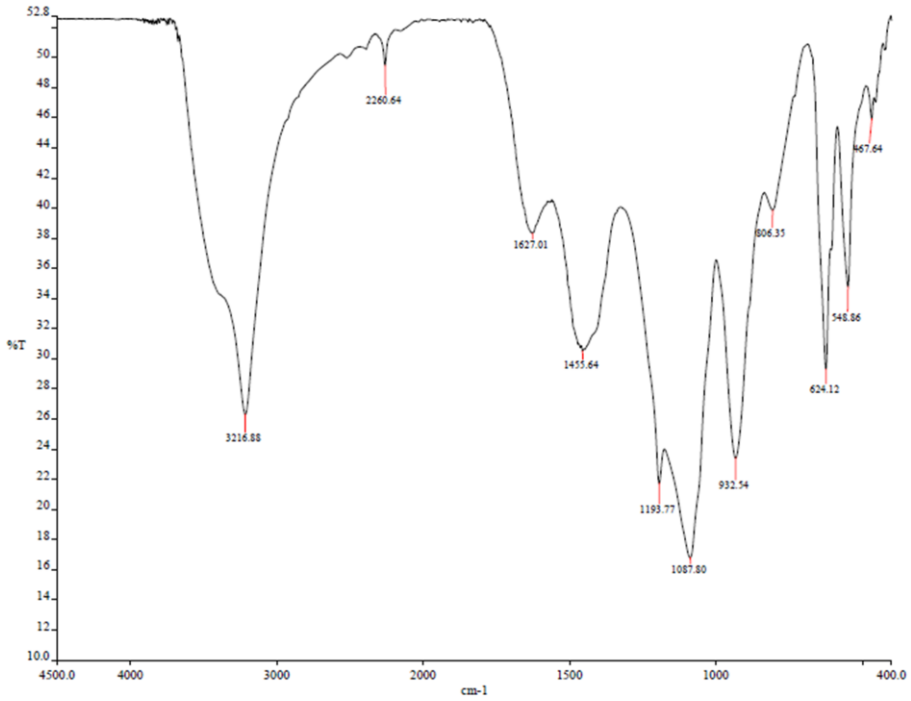
4.5 FTIR TEST

For FTIR TEST, 3 samples also been chosen which are Sample 1 (0 % filler), Sample 4 (3 % filler) and Sample 5 (4 % filler) had been went through the testing. The result is as shown below .

Table 4.11: Sample FTIR TEST

Sample	Result
1	

4	 56.0 50 45 40 35 30 25 20 15 10 5 3.0 %T 4500.0 4000 3000 2000 1500 1000 400.0 cm-1 3214.34 2260.72 1626.51 1450.97 1193.37 1087.82 932.48 624.22 548.66 467.68

5	 The reason why FTIR is conducted is purposed to see the structure of the molecule inside the char .Based on the result above ,for the range of the wavelength 3000-4000 the bonding exist is O-H bonding.

4.6 TGA Analysis

TG curve of the intumescent coating is presented in Figure below.

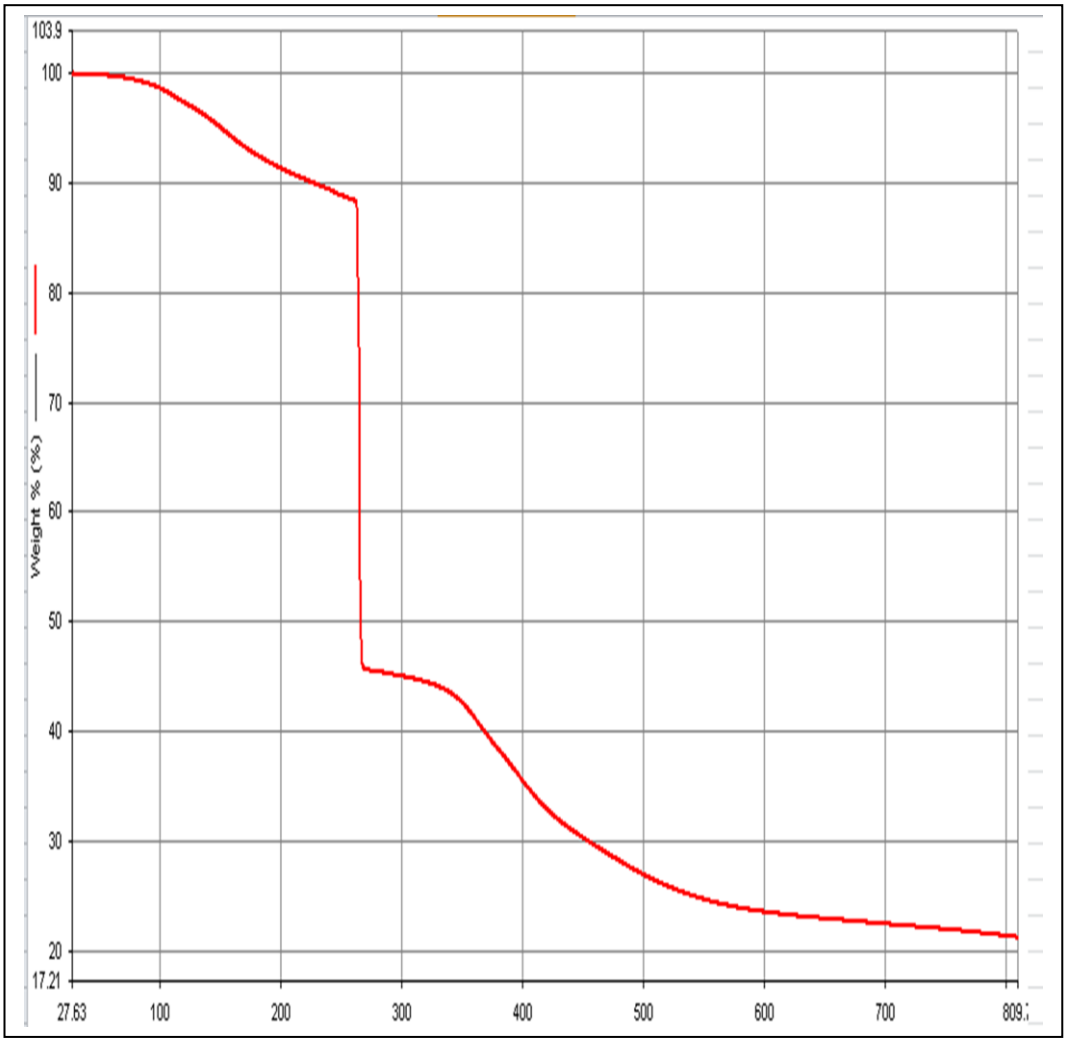


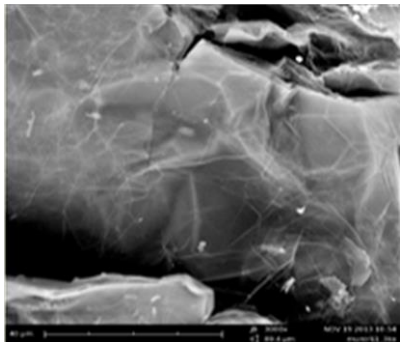
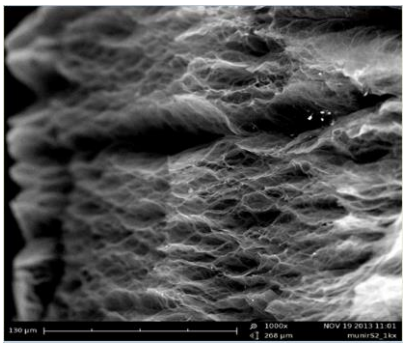
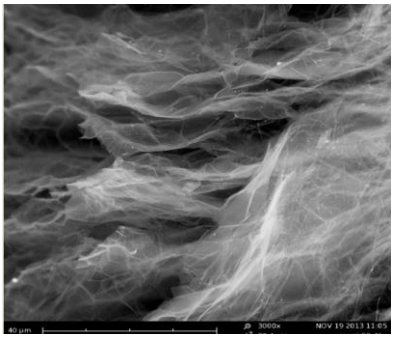
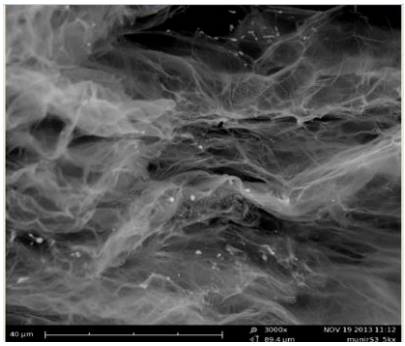
Figure 12: TG curve of the intumescent coating

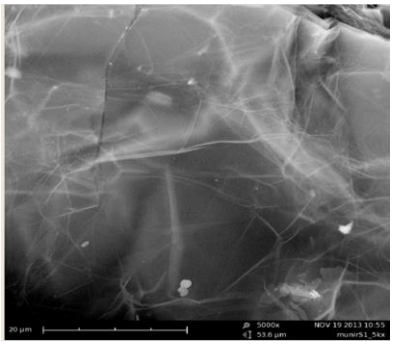
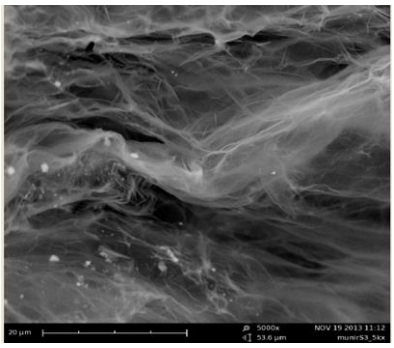
A two-step degradation mechanism is observed: first step between 260 °C and 270°C corresponding to a weigt loss of about 20 % and a second step in the temperature range 270 ° C- 285 °C leading thermally stable material (22 wt.% of the initial mass).

4.7 Scanning Electron-Microscope (SEM) Analysis

From the result obtain, 3 samples are analysed by using Scanning Electro-Magnetic (SEM) Machine with **Magnification 3000x**. The analysis is described below:

Table 4.12: SEM Analysis

Sample 1 (outer layer)  This picture shows the surface Is rough and bubbles present. There are also holes. No flake can be seen.	Sample 1 (inner layer)  This picture shows there is crack.No bubbles happened.
Sample 4 (outer layer)  This picture shows the surface Is rough.There are also holes. No flake can be seen.	Sample 4 (inner layer)  This picture shows there is crack. Little bubble detected

Sample 5 (outer layer)	**Sample 5 (inner layer)**
	
This shows the surface Is rough and bubbles present. There also holes. No flake can be seen.	This picture shows there is crack. No bubbles.

4.8 Weathering Test

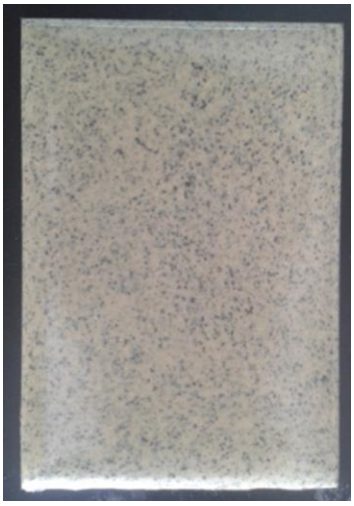
After all the samples have been taking out from the weathering chamber, some observation has been conducted. It shows that the surface colour of the coating is changed to yellowish. This is due to the explosion to the Ultra-Violet (UV) light and exposure to rain. The weight is also measured. Below is the weight of the sample before and after the weathering chamber test.

Table 4.13: Weight of samples before and after weathering test

Sample	**Weight**		**Percentage differences (%)**
	Before	**After**	
1	**66.99**	**66.90**	**0.13**
2	**63.62**	**63.55**	**0.11**
3	**62.83**	**62.79**	**0.05**
4	**61.67**	**61.60**	**0.10**
5	**63.09**	**63.04**	**0.08**
6	**60.23**	**60.19**	**0.06**

Based on the result obtained all the samples have lost the weight not more than 1%.

Table 4.14: Differences of the samples before and after went through Weathering Testing

Sample	Before	After
1		
2		

3		
4		

5		
6		

As usual, the thickness of the coating before and after fire test is measured. The data is presented in the tables below:

Table 4.15: Thickness of the coating before Fire Test (Furnace Test)

SAMPLE	READING 1 (mm)	READING 2 (mm)	READING 3 (mm)	READING 4 (mm)	READING 5 (mm)	READING 6 (mm)	AVERAGE THICKNESS (mm)
S1	4.16	3.93	3.87	4.17	4.09	3.99	4.04
S2	3.69	3.60	3.93	4.01	3.98	3.88	3.84
S3	3.55	3.41	3.44	3.72	3.54	3.75	3.57
S4	3.31	3.40	3.41	3.34	3.48	3.39	3.38
S5	3.34	3.49	3.52	3.39	3.44	3.50	3.45
S6	3.44	3.50	3.53	3.31	3.54	3.48	3.47

After the Fire Testing by using Furnace Test, the thickness of the coating is measured again as in the table below:

Table 4.16: Thickness of the coating after Fire Test (Furnace Test)

SAMPLE	READING 1 (mm)	READING 2 (mm)	READING 3 (mm)	READING 4 (mm)	READING 5 (mm)	READING 6 (mm)	AVERAGE THICKNESS (mm)
S1	6.84	7.21	7.01	6.99	6.85	7.88	7.13
S2	10.62	9.97	10.43	10.83	10.44	11.01	10.55
S3	10.58	10.33	10.11	10.66	10.87	9.67	10.37
S4	9.20	8.60	8.55	8.77	9.22	8.76	8.85
S5	9.51	9.56	10.11	9.95	9.77	10.20	9.85
S6	9.19	8.94	9.44	9.08	9.03	9.58	9.21

Therefore, the percentage of expansion can be summarised as follow:

Table 4.17: Percentage of char expansion

SAMPLE	THICKNESS (mm)		EXPANSION	PERCENTAGE OF EXPANSION (%)
	BEFORE FIRE TEST	AFTER FIRE TEST		
S1	4.04	7.13	3.09	177.81
S2	3.84	10.55	6.71	262.44
S3	3.57	10.37	6.80	275.79
S4	3.38	8.85	5.47	220.69
S5	3.45	9.85	6.40	283.05
S6	3.47	9.21	5.74	258.70

Table 4.18: Char Result after Weathering Testing

Sample	Before Fire Test	After Fire Test
1	 Thickness of the coating = 4.04mm	 Thickness of the coating = 7.13mm Percentage of expansion = 177.81% (1.76 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth
2	 Thickness of the coating = 3.84mm	 Thickness of the coating = 10.55mm Percentage of expansion = 262.44% (2.75 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth

3	 Thickness of the coating = 3.57mm	 Thickness of the coating = 10.37mm Percentage of expansion = 275.79% (2.90 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth
4	 Thickness of the coating = 3.38mm	 Thickness of the coating = 8.85mm Percentage of expansion = 220.69 (2.62 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth

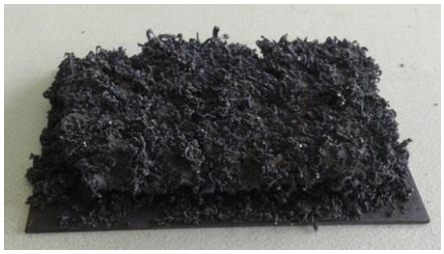
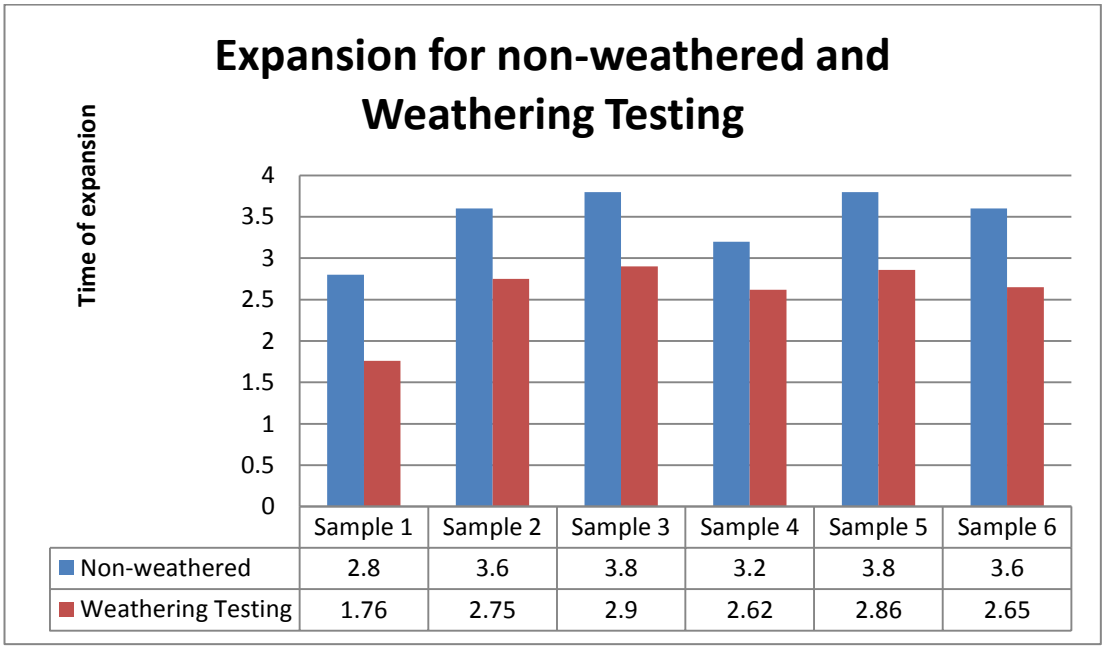
5	 Thickness of the coating =3.45mm	 Thickness of the coating =9.85mm Percentage of expansion = 283.05% (2.86 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth
6	 Thickness of the coating =3.47mm	 Thickness of the coating = 9.21mm Percentage of expansion = 258.70% (2.65 times of expansion) The coating is detached from the steel. The surface is cracked. The surface not so smooth

Table 4.19: Comparison percentage of char expansion before and after Weathering Testing

Sample	Percentage of char expansion before Weathering Testing (%)	Percentage of char expansion after Weathering Testing (%)	Differences
1	180.41	177.81	-2.6
2	264.72	262.44	-2.28
3	279.13	275.79	-3.34
4	222.46	220.69	-1.77
5	284.38	283.05	-1.33
6	261.78	258.70	-.3.08

Difference performance of expansion can be seen after the samples are exposed to the simulated weather. Figure 12 below shows that the expansion of coating that has never been exposed to weather are having greater performance where the highest expansion is up to 3.8 times from the initial thickness. Meanwhile, for the coating that has been put in the weathering chamber, the highest expansion is only 2.9 times from the initial thickness. The below graph is also shows that none of the samples after weathering test are having higher expansion compared to the non-weathered sample



	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Non-weathered	2.8	3.6	3.8	3.2	3.8	3.6
Weathering Testing	1.76	2.75	2.9	2.62	2.86	2.65

Figure 13: Expansion of coating for non-weathered and Weathering Testing

CHAPTER 5

CONCLUSION AND DISCUSSION

5.1 CONCLUSION

This project has revealed that weather affects the performance of intumescent fire retardant coating in term of its expansion. The usage of Mica as filler in the coating also gives different performance implication to the coating .The coating with different amount of filler percentage also affects the performance of the intumescent coating. From the sample that has been carried out filler with 4 % gives the highest char expansion. The char expansion and heat shielding effect shows the fire testing result where the intumescent coating really can protect steel from high temperature From the result obtained, it shows that the weathering effect which are UV and rain degrade the coating's expansion. This is due to the leaching out of its active ingredients. The coating is not detachable and remain at the substrate at the end of exposure period .The coating expands more when they do not expose to the simulated weather. Therefore, there is no doubt that the weathering has reduces the expansion of the fire retardant coating

5.2 RECOMMENDATION

It is recommended for the future improvement and development in the project, variety particle size of the filler is used to observe the performance of the intumescent coating. Other than that, another parameter of weathering properties are recommended to be studied such as oxidization and industrial atmosphere. Besides, the duration of time exposed to weather can be studied too for knowing the exact limit of the coating's endurance before it fails to protect the substrate.

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APPENDICES

Appendix A

Table and graph for Heat Shielding Test

Table A-1 temperature Heat Shielding Test

Time (Minute)	Temperature (° C)					
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
0	44.58	39.05	38.76	39.32	37.40	40.04
5	50.81	56.12	53.05	48.53	47.40	51.61
10	74.86	67.91	65.33	59.22	66.07	63.81
15	83.22	80.43	84.61	70.36	87.95	83.73
20	101.89	97.43	98.95	94.78	95.66	92.15
25	120.46	104.62	105.76	105.99	98.49	102.78
30	133.99	120.76	124.39	127.65	110.61	113.10
35	149.05	127.52	129.55	132.55	120.88	122.33
40	156.51	133.94	135.87	140.46	129.79	131.79
45	177.90	148.91	151.69	149.59	136.36	138.58
50	180.34	159.46	160.04	158.92	147.83	153.43
55	193.29	173.41	173.22	165.66	156.65	160.68
60	195.66	176.44	178.33	170.12	169.59	171.22