

# CHAPTER 1

## INTRODUCTION

### **1.1 Background of Study**

Intumescent coatings are increasingly used because they provide a high standard of finish, quality and reliability and can be used on buildings requiring high fire resistance. Most construction materials nowadays use intumescent coating to protect steel substrate. As rockwool and glasswool that is widely been used nowadays is having high thermal resistance, the author believed the composition of rockwool and glasswool with intumescent coating give wide spectrum of advantages, especially in construction field.

This research is to develop intumescent formulations reinforced with rockwool and compare with the formulations with glasswool. Therefore a better coating to enhance the thermal resistance of the coating will be determined. It is strongly believed that the addition of rockwool and glasswool will increase the thermal resistance of the intumescent coating.

Thin film Intumescent coatings are increasingly used because they provide a high standard of finish, quality and reliability. They can be applied on or off-site and can be used on structures to provide protection for the steel. These coatings are from organic materials and are inert at low temperatures[1]. They swell (or intumesce) to provide a charred layer of low conductivity foam when exposed to high temperatures[1]. This char layer reduces the rate of temperature rise in the steel and prolongs the steels load bearing capacity[1]. Intumescent coatings foam and bubble when subjected to high temperatures and create multicellular cushions which prevent (a) heat from penetrating into the substrate and (b) flames from spreading along the surface. [2]



Figure 1: Swollen intumescent coating[2]

### 1.1.1 GLASSWOOL

Glasswool is an insulating material, made from fibre glass, arranged into a texture similar to wool. Glass wool is produced in rolls or in slabs, with different thermal and mechanical properties. Glasswool is a thermal insulation that consists of intertwined and flexible glass fibres, which causes it to "package" air, resulting in a low density that can be varied through compression and binder content. It can be a loose fill material, blown into attics, or, together with an active binder sprayed on the underside of structures, sheets and panels that can be used to insulate flat surfaces such as cavity wall insulation, ceiling tiles, curtain walls as well as ducting. It is also used to insulate piping and for soundproofing[11].

### 1.1.2 ROCKWOOL

Rockwool products are robust insulation material suitable for hot surfaces up to 650°C. Rockwool are made from long fine fibres spun from natural rock bonded with a thermosetting resin. Rockwool products are easy to install and they come in the form of blankets, slabs and pipe sectionals.

## **1.2 Problem statement**

Most structures are steel-based structure. Intumescent coatings are used to protect these structures from the damage of fire. The basic intumescent coating is quite fragile and cannot stand high temperature. Rockwool and glasswool are two high temperature resistance materials. Incorporating these fibers into intumescent coating might improve thermal performance of the coating. However, appropriate fiber content is among the key element of an effective coating fiber.

## **1.3 Objectives and scope of study**

### OBJECTIVES

- 1) To prepare the intumescent formulation
- 2) To prepare intumescent formulation with rockwool
- 3) To prepare intumescent formulation with glasswool
- 4) Compare thermal effects between the coatings with different formulations

### SCOPE OF STUDY

- a) Research on the formulation of the intumescent coating, glasswool and rockwool
- b) Study on the composition of intumescent coating with glasswool and rockwool
- c) The samples will gone through laboratory test to obtain its thermal performance
- d) Analysis will be done due to the data attained
- e) Report of the project is then been established based on the result gained

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Literature Review

Intumescent coatings is a fire retardant coatigs that swell (or intumesce) to provide a charred layer of low conductivity foam when exposed to high temperatures. This char layer reduces the rate of temperature rise in the steel and prolongs the steels load bearing capacity.

##### 2.1.1 COMPOSITION OF INTUMESCENT COATING

The most commonly used intumescent coatings accordingly comprises a binder matrix and three to four basic components dispersed in the binder matrix. Said basic components include (1) an acid source comprising for instance an inorganic acid or a material yielding an acid at temperatures of typically 100-250 °C, such as ammonium polyphosphate and sodium borate yielding phosphoric and boric acid, (2) a carbon source, typically a polyhydric material rich in carbon, such as trimethylolthane, pentaerythritol and/or dipentaerythritol which normally are used as micronized products, (3) a spumific or blowing agent being for instance an organic amine or amide, such as a melamine and melamine derivatives, and optionally (4) a halogenated material releasing for instance hydrochloric acid gas on decomposition[8].

Components;

- Ammonium Polyphosphate (APP) as the acid source
- Melamine (MEL) as the blowing agent
- Pentaerythritol (PER) as the carbon agent
- Hardener
- Epoxy resin
- Boric acid

The mechanism of intumescence is usually described as follow;

- 1) Acid source breaks down to yield a mineral acid
  - 2) Then it takes part in the dehydration of the carbonization agent to yield the carbon char
  - 3) Blowing agent decomposes to yield gaseous products
  - 4) The latter causes the char to swell and hence provides an insulating multicellular protective layer
  - 5) This shield limit the heat transfer to substrate and the mass transfer from the substrate to the heat source resulting in a conservation of the underlying material
- [6]

The invention features a fire-retardant and heat-resistant coating applied by first coating the surface to be protected with a protective composition comprising (1) an intumescent paint consisting essentially of a volatile liquid vehicle, 35 to 150 parts by weight of a resinous film-forming binder, and 120 to 475 parts by weight of a solid spumific agent which when heated to 400°C causes a dried film of the paint to increase in thickness at least 4 times, and (2) 2 to 30% by weight (exclusive of the weight of any organic coating on the fibers), based on the remaining ingredients of the paint, of glass fibers; and overlaying this protective composition with a protective layer comprising a flexible, surface-conformable fabric sheet, e.g., a textile fabric, having sufficient strength and permeability to unite with a paint applied therewith. The coating may be dried either before or after applying the protective layer. In preferred embodiments, this protective layer may consist of an incombustible fabric, such as fiber glass cloth; a combustible fabric coated with an overcoating of intumescent paint; or an incombustible fabric coated with an overcoating of intumescent paint. The overcoating intumescent paint may, but need not also be combined with glass fibers. Also, 10% or more of the fibers in the protective composition may be glass fibers precoated with polyvinyl chloride. [3]

### 2.1.2 GLASSWOOL

Centrifugal glass wool is a silk-like material made by fiberization and spraying of thermosetting resin. Glass wool is non-combustible, non-toxic, and resistant to corrosion. It has low weight by volume, low thermal conductivity, stable chemical property, and low moisture absorption rate due to its excellent hydrophobicity. Due to its intertwined flexible fibers, glass wool is the best insulating material against noise, cold and heat and also offers excellent fire-resistant properties. The panel, felt, and pipe products made of glass wool have been used in the industries of construction, chemical, electronic, energy, metallurgy and communication[13].

The basic raw materials are natural sand, to which recycled glass and fluxing agents like soda ash are added. Use of recycled glass coming from buildings flat glass, car glass and containers, is steadily increasing. Its share may represent up to 80 % of the batch. Glass batch is made of ground particles with a very precise sieve analysis. In addition, the mix of components is quite accurate in order to get an homogeneous batch to be melted in an optimal way[14].

### 2.1.3 ROCKWOOL

In the other hand, one of the most vital properties of Rockwool is its ability to withstand temperatures of more than 1000°C. Properly used, Rockwool acts as a fire resistant barrier, which can provide those vital extra minutes for rescuing people.

This insulating material is made from waste products namely, bottom ash, cement kiln dust, slag, and waste from mineral wool production. These materials, along with a binder, are homogenized into a mixture. Thereafter, the process includes briquetting the mixture into agglomerated pieces. The agglomerates are then melted in a cupola furnace and the molten agglomerate is discharged into a receiver. Hot combustion gases are then passed into the melt or molten agglomerate to chemically homogenize the melt and heat the melt to a preselected temperature. Thereafter, the melt is converted into fibers using conventional practices[4]. The final product is a mass of fine, intertwined fibres with a typical diameter of 6 to 10 micrometers. Rockwool may contain a binder, often food

grade starch, and an oil to reduce dusting. Though the individual fibres conduct heat very well, when pressed into rolls and sheets their ability to partition air makes them excellent heat insulators and sound absorbers[11].

Table1: Thermal resistance & density of glasswool and rockwool [3]

| MATERIAL  | TEMPERATURE  | NOMINAL DENSITY               |
|-----------|--------------|-------------------------------|
| Glasswool | 230 – 250 °C | Less than 32kg/m <sup>3</sup> |
| Rockwool  | 700 – 850 °C | 40kg/m <sup>3</sup>           |

[6]

#### 2.1.4 LITERATURE ON THERMAL RESISTANCE TEST OF INTUMESCENT COATINGS

The paper demonstrates the benefits of Lapinus' fibres when used in simplified intumescent coating formulations and their effect on the performance and hence the protective efficiency of said coatings.

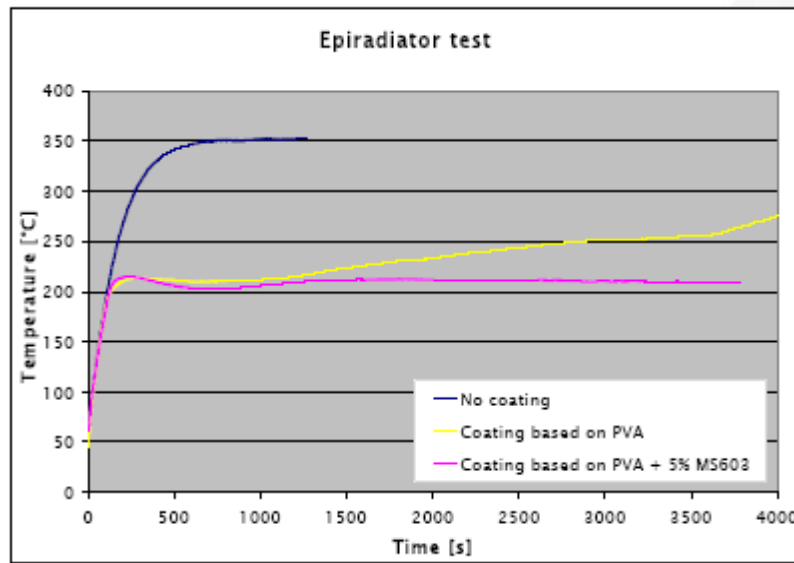


Figure 2: Result on Epiradiator Test (see Fig.10 in appendix)

\*Rockforce® MS603-Roxul®1000 is a high quality engineered mineral fibre.

\*Epiradiator test used to evaluate the performance of intumescent coating

The figures show the influence of the presence of Lapinus' fibres on the protective properties of the char layer of both PVA based system and a 2K epoxy system. Not only does the temperature of the substrate remain lower for a longer period of time (up to more than 120 min for the 2K epoxy) when fibres are used, but also the strength of the char layer is improved which is beneficial for the performance during raging fire. The results obtained with Lapinus' fibres are better than the results obtained with the competitor fibre type.

It is shown that the performance of both a waterborne PVA and 2K epoxy based simplified

intumescent coating can be improved using Lapinus' fibres:

- The temperature reached at the coating/ substrate interface is lower and remains lower when Lapinus' fibres are used. The most suitable fibre grade depends on the binder used;
- The strength of the char layer is increased, while the integrity remains. As a result, the char layer is more stable and longer lasting (> 120 min) compared to the other tested systems.

- From the research paper above, it is proven that the addition of fibre into the intumescent coating will improve the performance of intumescent coating in term of thermal performance. Briefly explaining, Lapinus Fibres is the world's largest producer of precision-engineered mineral fibre products.



## 2.1.5 THERMAL PERFORMANCE TEST STANDARD

### ASTM Standard for Proposed Thermal Measurements Test Method to Determine Effective Thermal Conductivity, WK12643:

The thermal performance test that will be going through to test the intumescent coatings resistance to fire resembles the standardized test from ASTM Standard.

The test will obey the Standard WK12643, Test Method for Slug Calorimetry. The proposed standard is under the jurisdiction of Subcommittee E37.05 on Thermophysical Properties.

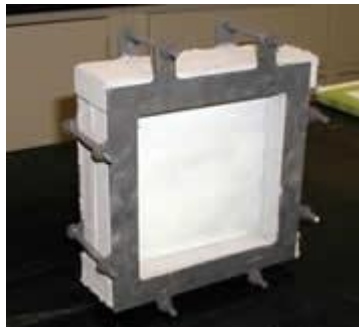


Figure 3: Slug calorimeter

From the above picture, assembled slug calorimeter specimen ready for testing in the furnace. The coating shown is an organic intumescent material, surrounded by a high temperature microsilica thermal insulation, with the assembly being held together by two retaining plates.

Below is the figure that shows the cone calorimeter. Cone calorimeter resembled the usage of burner burner for direct burning.

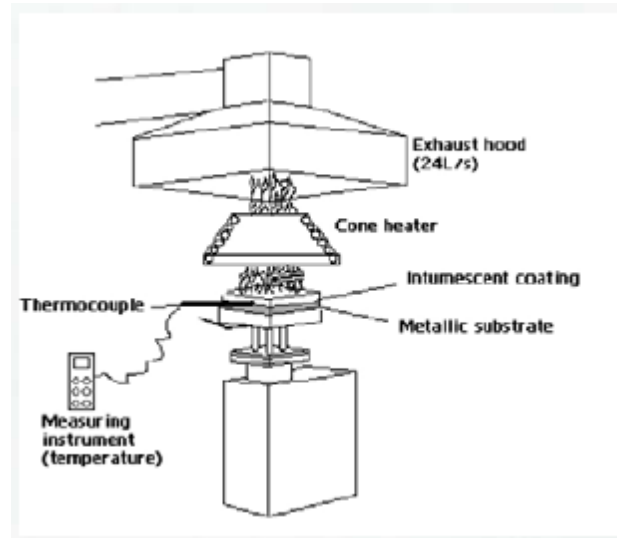
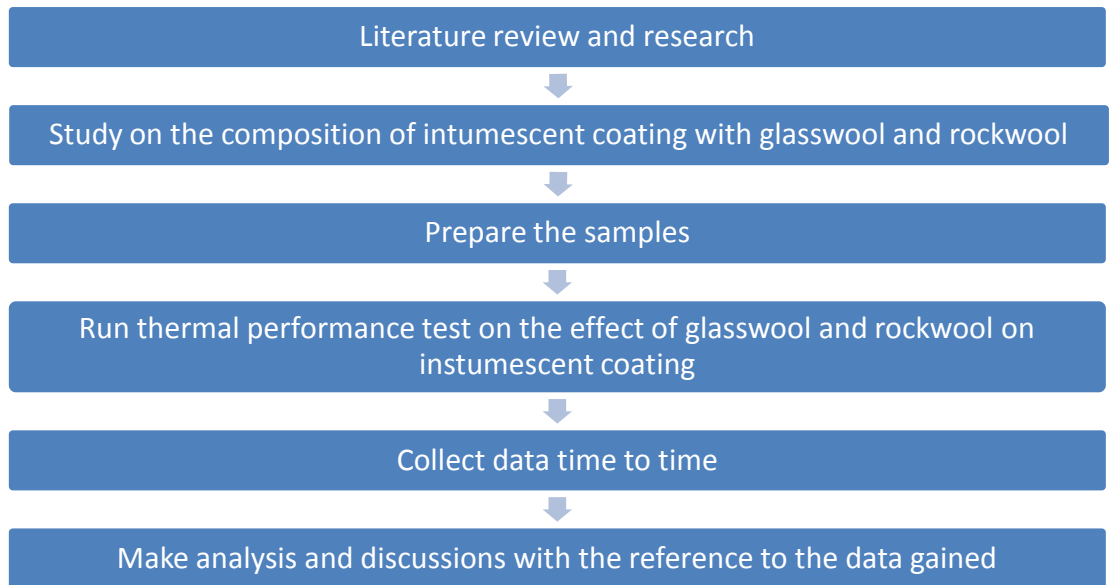


Figure 4: Cone calorimeter [7]

# CHAPTER 3

## METHODOLOGY

### 3.1 PROJECT FLOW



### 3.2 SPECIFIC ACTIVITIES

1) Research on the composition and behavior of intumescent coating, glasswool and rockwool

- this research consist of the study on the properties of the respective elements
- the study also cover on how the glasswool and rockwool is to be mixed with intumescent coating

2) Prepare the sample and run tests

- an amount of samples will be prepared for the thermal performance test of the material

3) Collect data and make analysis

### 3.3 SAMPLE PREPARATIONS

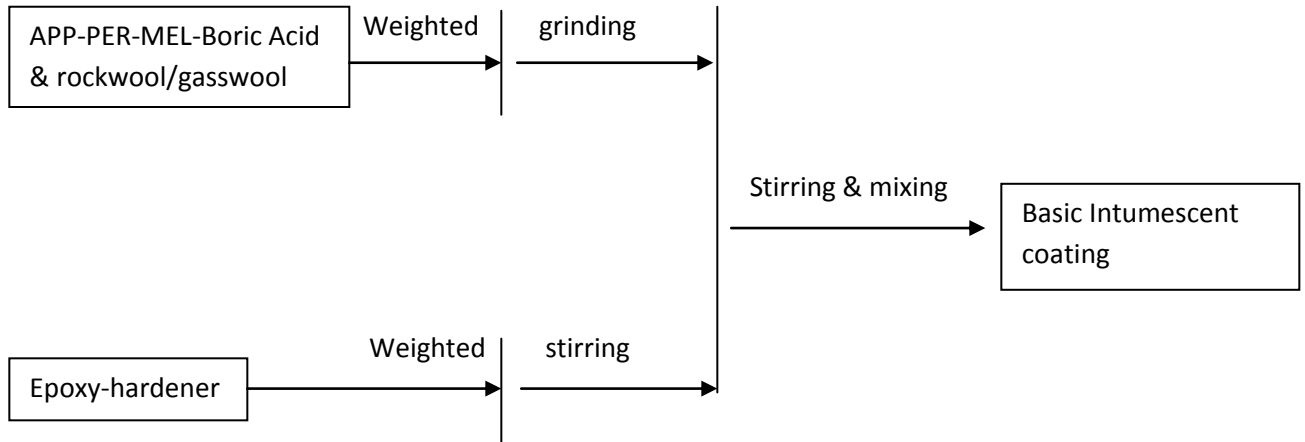


Figure 5: Intumescent coatings sample preparations

#### 1) Weight the components.

- the main components in making intumescent coating will be prepared and varied by weight
- the reinforced fiber will also be varied by weight when subjected to different sample of intumescent coating

#### 2) Mixing the components

- the main components; acid source, carbon source and the blowing agent will be grinded along with the fiber
- after the grinding process, epoxy and hardener will be filled into the beaker having the grinded components. Then, all the components will be mixed up using equipment.

#### 3) Coating and curing.

- After all the components being well mixed up, coating process of intumescent coating will be done onto the metal substrate, mild steel plate.
- The sample will cure in room temperature.

### **3.4 SAMPLE ANALYSIS**

#### **3.4.1 SCANNING ELECTRON MICROSCOPE**

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a light microscope.

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification. Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive. The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today.

The development of the Scanning Electron Microscope in the early 1950's brought with it new areas of study in the medical and physical sciences because it allowed examination of a great variety of specimens.

As in any microscope the main objective is for magnification and focus for clarity. An optical microscope uses lenses to bend the light waves and the lenses are adjusted for focus. In the SEM, electromagnets are used to bend an electron beam which is used to produce the image on a screen. By using electromagnets an observer can have more control in how much magnification he/she obtains. The electron beam also provides greater clarity in the image produced.

### 3.4.2 THERMAL TEST

To test the performance of the intumescent coating applied, the mild steel substrate coated with applied intumescent coating will be fired up using Bunsen burner. At the same time, a digital thermocouple will be attached at the opposite side of the substrate to take the value of temperature vs. time. Here is the setup:

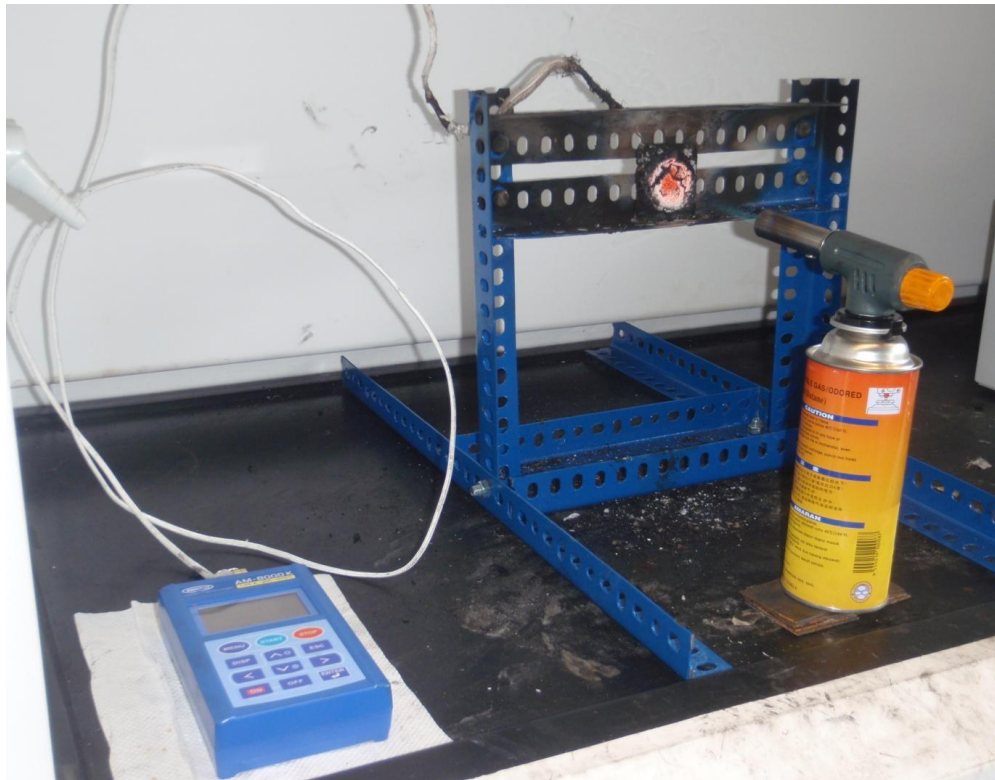


Figure 6: Direct burning setup for measuring the temperature of back steel

# CHAPTER 4

## RESULTS AND DISCUSSION

### 4.1 RESULTS & DISCUSSION

#### 4.1.1 NORMAL FORMULATION (WITHOUT REINFORCEMENT)

Attached in Table 2 is the basic intumescent coatings formulations. The formulations of the intumescent coatings are varied from the amount of the PER, APP, MEL and boric acid. The amount of epoxy and hardener remains the same with the ratio of 2:1.

Table 3 shows the results of the height before the fire burning test. While Table 3 shows the results of the samples height after the fire burning test. From the results gained, the best sample is determined.

**Table 2: Basic intumescent coating formulation**

| No. | Pentaerythrytone (PER) | APP   | Melamine (MEL) | Boric Acid | Total weight | Epoxy | Hardener |
|-----|------------------------|-------|----------------|------------|--------------|-------|----------|
| 1   | 20                     | 40    | 20             | 20         | 100          | 2     | 1        |
| 2   | 16.67                  | 33.33 | 25.00          | 25.00      | 100          | 2     | 1        |
| 3   | 16.67                  | 33.33 | 16.67          | 33.33      | 100          | 2     | 1        |
| 4   | 16.67                  | 25.00 | 33.33          | 25.00      | 100          | 2     | 1        |
| 5   | 16.67                  | 16.67 | 33.33          | 33.33      | 100          | 2     | 1        |
| 6   | 25.00                  | 33.33 | 25.00          | 16.67      | 100          | 2     | 1        |
| 7   | 33.33                  | 33.33 | 16.67          | 16.67      | 100          | 2     | 1        |
| 8   | 25.00                  | 25.00 | 33.33          | 16.67      | 100          | 2     | 1        |
| 9   | 16.67                  | 16.67 | 33.33          | 16.67      | 100          | 2     | 1        |
| 10  | 25.00                  | 25.00 | 25.00          | 25.00      | 100          | 2     | 1        |
| 11  | 16.67                  | 25.00 | 25.00          | 33.33      | 100          | 2     | 1        |
| 12  | 33.33                  | 25.00 | 25.00          | 16.67      | 100          | 2     | 1        |
| 13  | 33.33                  | 33.33 | 16.67          | 25.00      | 100          | 2     | 1        |

**Table 3: Height before burnt**

| Samples | h1 (mm) | h2 (mm) | h3 (mm) | h4 (mm) | h5 (mm) | h6 (mm) | Avg (mm) |
|---------|---------|---------|---------|---------|---------|---------|----------|
| 1       | 1.80    | 2.80    | 2.30    | 2.50    | 2.22    | 2.04    | 2.28     |
| 2       | 2.50    | 2.84    | 2.78    | 2.72    | 3.22    | 3.14    | 2.87     |
| 3       | 4.60    | 4.90    | 4.82    | 4.70    | 5.60    | 4.98    | 4.93     |
| 4       | 2.34    | 2.72    | 2.78    | 2.88    | 3.24    | 3.00    | 2.83     |
| 5       | 1.96    | 2.34    | 2.44    | 2.50    | 2.92    | 2.70    | 2.48     |
| 6       | 2.54    | 2.42    | 2.44    | 2.30    | 3.22    | 3.40    | 2.72     |
| 7       | 2.25    | 2.26    | 2.30    | 2.20    | 2.31    | 2.26    | 2.26     |
| 8       | 2.44    | 2.70    | 2.14    | 2.32    | 3.02    | 3.32    | 2.66     |
| 9       | 2.20    | 2.24    | 2.60    | 2.42    | 3.40    | 3.32    | 2.70     |
| 10      | 2.38    | 2.34    | 2.40    | 2.44    | 3.10    | 3.04    | 2.62     |
| 11      | 1.94    | 2.20    | 2.24    | 2.04    | 2.84    | 3.14    | 2.40     |
| 12      | 2.74    | 2.54    | 2.44    | 2.82    | 3.42    | 3.84    | 2.97     |
| 13      | 1.84    | 1.98    | 2.20    | 2.16    | 3.02    | 2.78    | 2.33     |



**Table 4: Height after burnt**

| Samples | h1 (mm) | h2 (mm) | h3 (mm) | h4 (mm) | h5 (mm) | h6 (mm) | Avg (mm) |
|---------|---------|---------|---------|---------|---------|---------|----------|
| 1       | 16.50   | 14.50   | 14.50   | 13.50   | 15.50   | 15.50   | 15.00    |
| 2       | 13.50   | 9.50    | 8.50    | 10.50   | 9.50    | 11.50   | 10.50    |
| 3       | 18.50   | 23.50   | 23.50   | 22.50   | 21.50   | 24.50   | 22.33    |
| 4       | 10.50   | 9.50    | 9.50    | 9.50    | 9.50    | 10.50   | 9.83     |
| 5       | 10.50   | 9.50    | 8.50    | 9.50    | 8.50    | 11.50   | 9.67     |
| 6       | 24.50   | 23.50   | 21.50   | 22.50   | 22.50   | 21.50   | 22.67    |
| 7       | 19.50   | 18.50   | 19.50   | 19.50   | 18.50   | 19.50   | 19.17    |
| 8       | 17.50   | 18.50   | 18.50   | 19.50   | 19.50   | 17.50   | 18.50    |
| 9       | 13.50   | 12.50   | 12.50   | 14.50   | 12.50   | 12.50   | 13.00    |
| 10      | 12.50   | 12.50   | 12.50   | 12.50   | 10.50   | 11.50   | 12.00    |
| 11      | 13.50   | 13.50   | 12.50   | 11.50   | 11.50   | 8.50    | 11.83    |
| 12      | 18.50   | 17.50   | 15.50   | 14.50   | 15.50   | 13.50   | 15.83    |
| 13      | 14.50   | 15.50   | 10.50   | 14.50   | 13.50   | 14.50   | 13.83    |

**Before burning test samples**








Fig7: before fire burning test samples









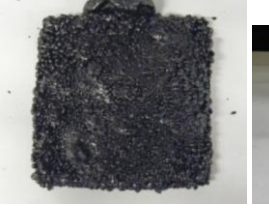

Note:

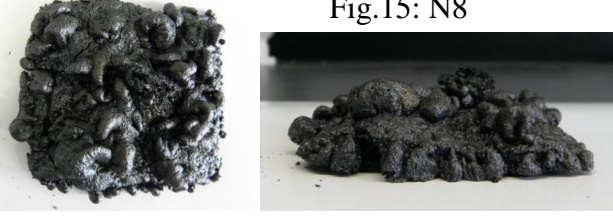
\*the dimension for the steel plate is 5mm x 5mm

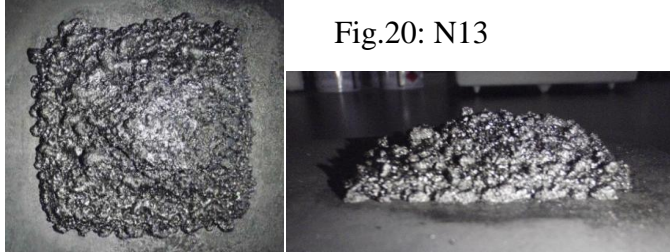
\*only for direct burnt test, 10mm x 10mm steel plate being used

**Table 5: After burnt samples**

|  |   |
|--|---|
| <p><b><u>FORMULATION 1</u></b></p> <p><b>Initial height: 2.28</b><br/><b>Final height: 15.00</b></p> | <div style="display: flex; justify-content: space-around;"> <div style="text-align: center;">  <p>Fig.8: N1</p> </div> <div style="text-align: center;">  </div> </div> <div style="text-align: center; margin-top: 10px;">  <p>Fig.9: N2</p> </div> |
| <p><b><u>FORMULATION 2</u></b></p> <p><b>Initial height: 2.87</b><br/><b>Final height: 10.50</b></p> | <div style="display: flex; justify-content: space-around;"> <div style="text-align: center;">  </div> <div style="text-align: center;">  </div> </div>   |

|  |   |  |
|--|---|--|
| <p><b><u>FORMULATION 3</u></b></p> <p><b>Initial height: 4.93</b><br/><b>Final height: 22.33</b></p> |    | <p>Fig.10: N3</p>    |
| <p><b><u>FORMULATION 4</u></b></p> <p><b>Initial height: 2.83</b><br/><b>Final height: 9.83</b></p>  |    | <p>Fig.11: N4</p>    |
| <p><b><u>FORMULATION 5</u></b></p> <p><b>Initial height: 2.48</b><br/><b>Final height: 9.67</b></p>  |   | <p>Fig.12: N5</p>   |
| <p><b><u>FORMULATION 6</u></b></p> <p><b>Initial height: 2.72</b><br/><b>Final height: 22.67</b></p> |  | <p>Fig.13: N6</p>  |
| <p><b><u>FORMULATION 7</u></b></p> <p><b>Initial height: 2.26</b><br/><b>Final height: 19.17</b></p> |  | <p>Fig.14: N7</p>  |

|   |   |
|---|---|
| <p><b><u>FORMULATION 8</u></b></p> <p><b>Initial height: 2.66</b><br/><b>Final height: 18.50</b></p>  |  <p>Fig.15: N8</p>    |
| <p><b><u>FORMULATION 9</u></b></p> <p><b>Initial height: 2.70</b><br/><b>Final height: 13.00</b></p>  |  <p>Fig.16: N9</p>    |
| <p><b><u>FORMULATION 10</u></b></p> <p><b>Initial height: 2.62</b><br/><b>Final height: 12.00</b></p> |  <p>Fig.17: N10</p>  |
| <p><b><u>FORMULATION 11</u></b></p> <p><b>Initial height: 2.40</b><br/><b>Final height: 11.83</b></p> |  <p>Fig.18: N11</p> |
| <p><b><u>FORMULATION 12</u></b></p> <p><b>Initial height: 2.97</b><br/><b>Final height: 15.83</b></p> |  <p>Fig.19: N12</p> |

|   |   |
|---|---|
| <p><b><u>FORMULATION 13</u></b></p> <p><b>Initial height: 2.33</b><br/><b>Final height: 13.83</b></p> |  <p>Fig.20: N13</p> |
|---|---|

**Table 6: 4 best formulations**

| SAMPLE         | DESCRIPTION   |
|----------------|---|
| Formulation 2  | <ul style="list-style-type: none"> <li>• small expansion</li> </ul>   |
| Formulation 4  | <ul style="list-style-type: none"> <li>• small expansion</li> <li>• empty space at expansion</li> </ul>           |
| Formulation 8  | <ul style="list-style-type: none"> <li>• bigger expansion</li> <li>• less empty space</li> </ul>                  |
| Formulation 12 | <ul style="list-style-type: none"> <li>• bigger expansion</li> <li>• empty space between the expansion</li> </ul> |

From visual analysis, Formulation 8 or N8 is chosen to be the best basic formulation as it has big expansion less empty space between char.

#### 4.1.2 SCANNING ELECTRON MICROSCOPE (SEM) FOR NORMAL FORMULATION

Fig. 21 and 22 are the SEM image of the normal formulation chosen, formulation N8. Fig.21(a) & (b) shows its outer image. There are some pores seen. Fig. 22(a) & (b) shows the inner image of N8 samples, which we can see there are many flakes there.

**N8**

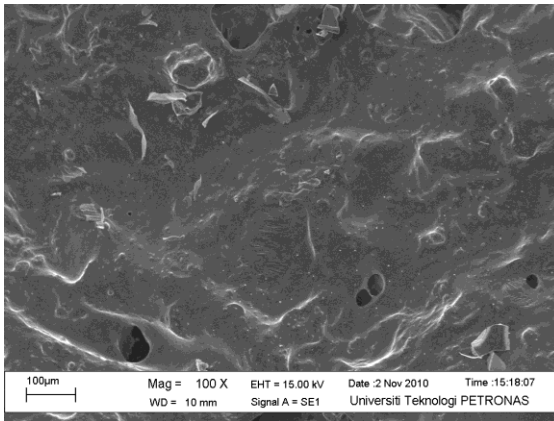


Fig. 21 (a)

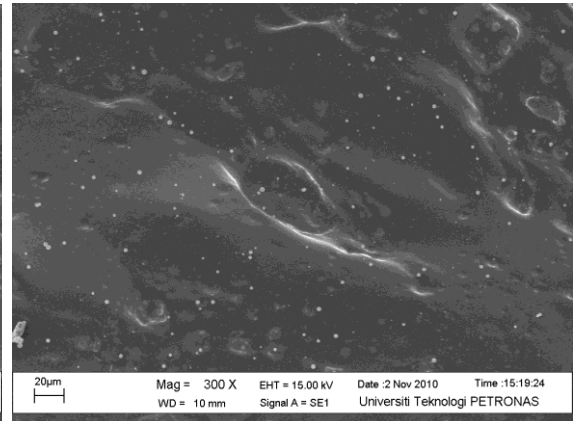


Fig. 21 (b)

Fig. 21: (a): Outer image-magnification 100; (b): Outer image-magnification 300

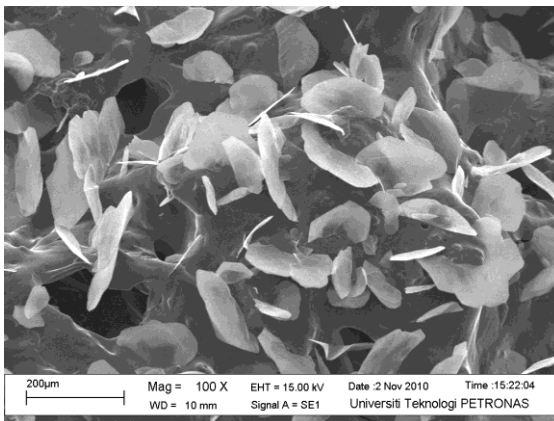


Fig. 22 (a)

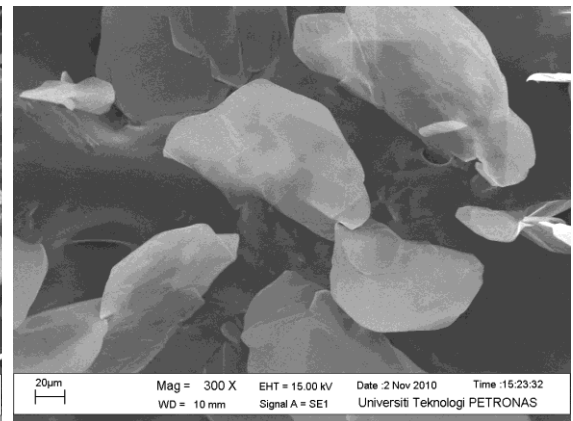



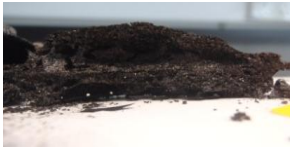




Fig. 22 (b)

Fig. 22: (a): Inner image-magnification 100; (b): Inner image-magnification 300







4.1.3 RESULTS & DISCUSSION OF FIBRE REINFORCED INTUMESCENT COATINGS

Table 6 and 7 shows the results of the samples reinforced glasswool and rockwool with its description. For glasswool samples, the swelling is quite good, yet some of the samples are not uniformly swell. In the other hand, for rockwool samples, the there are not much swell as compared with the glasswool samples, but the chat is quite hard and not easily broken.

**Table 7: Intumescent coating with reinforced glasswool**

| Formula<br>-tion | Fibre<br>percent-<br>age (%) | Height          |                | Description   | Top view  | Cross section  |
|------------------|------------------------------|-----------------|----------------|---|---|--|
|                  |                              | Before<br>burnt | After<br>burnt |   |   |  |
| G0.5             | 2.08                         | 3.63            | 17.8           | Good swell  |   | Fig.23: G0.5<br>  |
| G0.24            | 1                            | 2.73            | 10.33          | Not uniform swell, do not swell at the center. Yet not much empty space |  | Fig.24: G0.24<br> |
| G0.12            | 0.5                          | 2.37            | 8.5            | Not uniform swell, lesser expansion than G0.24                          |  | Fig.25: G0.12<br> |

**Table 8: Intumescent coating with reinforced rockwool**

| Formula<br>-tion | Fibre<br>percent-<br>age (%) | Height          |                | Description   | Top view   | Cross section   |
|------------------|------------------------------|-----------------|----------------|---|--|---|
|                  |                              | Before<br>burnt | After<br>burnt |   |  |   |
| R0.5             | 2.08                         | 2.35            | 6.63           | Not swell<br>much, hard,<br>not smooth<br>outer layer         |   | Fig.26: R0.5<br>   |
| R0.24            | 1                            | 2.41            | 7.02           | Swell a bit   |   | Fig.27: R0.24<br>  |
| R0.12            | 0.5                          | 2.72            | 8.66           | Swell more<br>than R0.24<br>and R0.5,<br>uniform<br>expansion |  | Fig.28: R0.12<br> |



#### 4.1.4 SCANNING ELECTRIC MICROSCOPE (SEM) FOR FIBRE REINFORCED INTUMESCENT COATINGS

As can be seen from the SEM results of the intumescent coatings reinforced fibres as compared to the normal formulations, the fibre are strongly attached with the intumescent formulation, resulting in a better coatings' char, providing a better layer to protects from fire attack.

##### **R0.5**

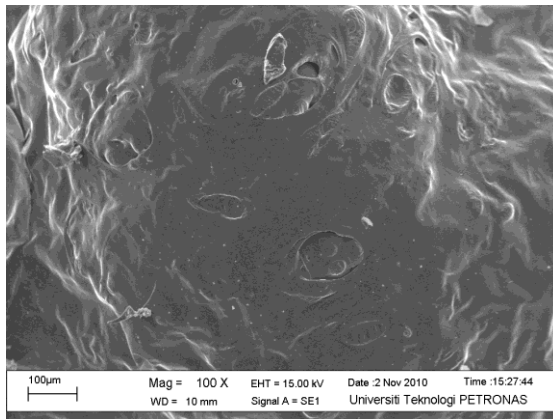


Fig. 29 (a)

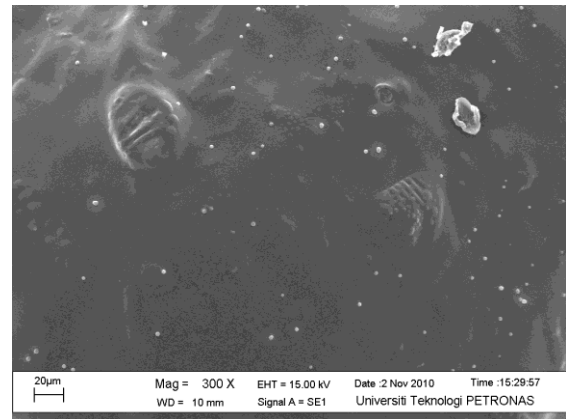


Fig. 29 (b)

Fig. 29: (a): Outer image-magnification 100; (b): Outer image-magnification 300

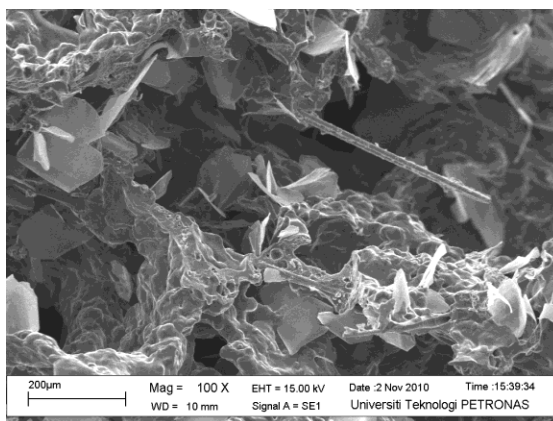


Fig. 30 (a)

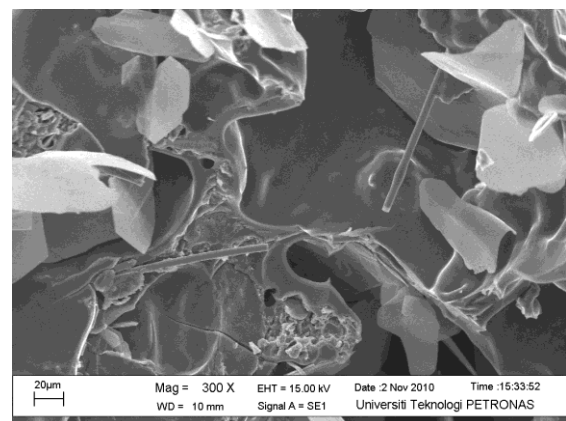


Fig. 30 (b)

Fig. 30: (a): Inner image-magnification 100; (b): Inner image-magnification 300

G0.5

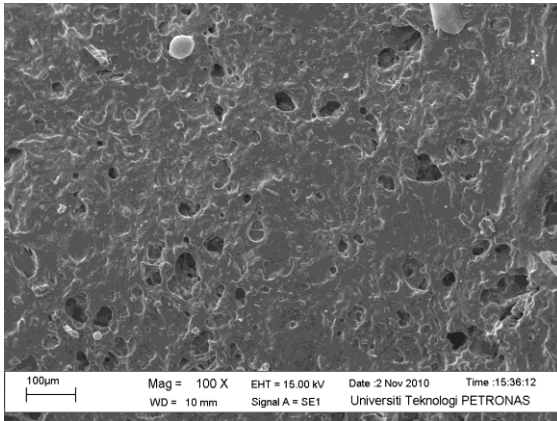


Fig. 31 (a)

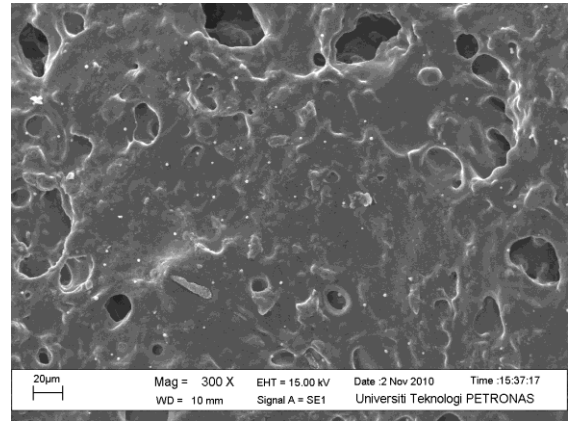


Fig. 31 (b)

Fig. 31: (a): Outer image-magnification 100; (b): Outer image-magnification 300

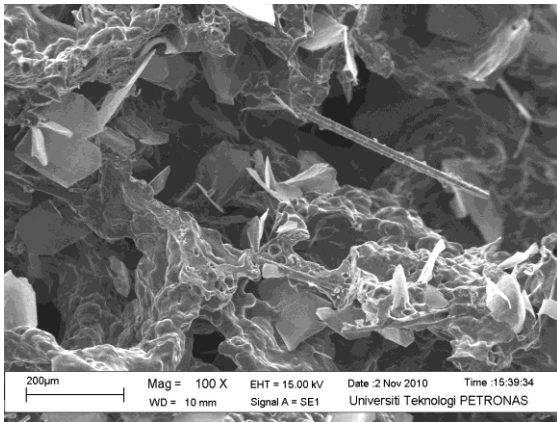


Fig. 32 (a)

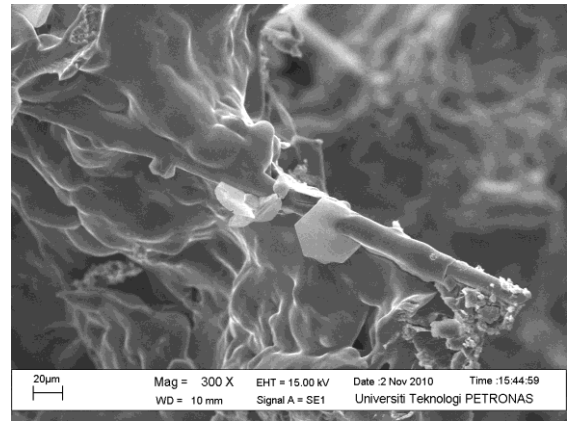


Fig. 32 (b)

Fig. 32: (a): Inner image-magnification 100; (b): Inner image-magnification 300

G0.24

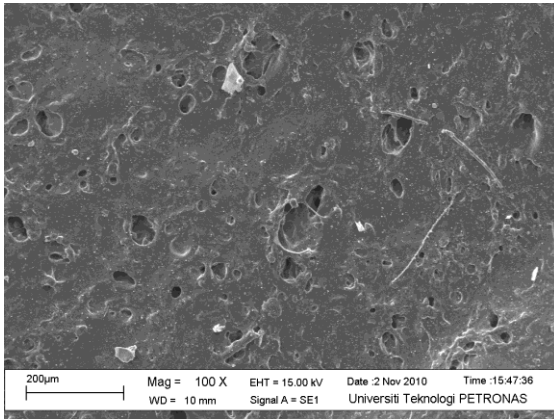


Fig. 33 (a)

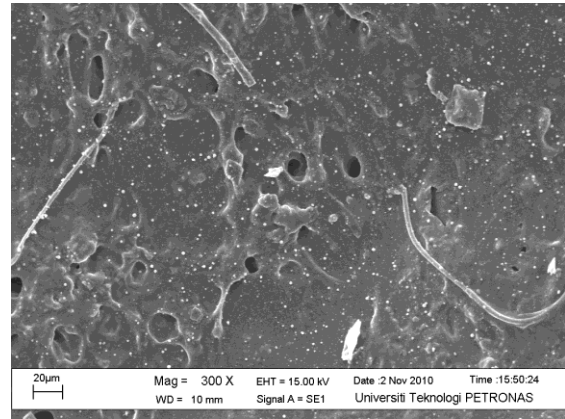


Fig. 33 (b)

Fig. 33: (a): Outer image-magnification 100; (b): Outer image-magnification 300

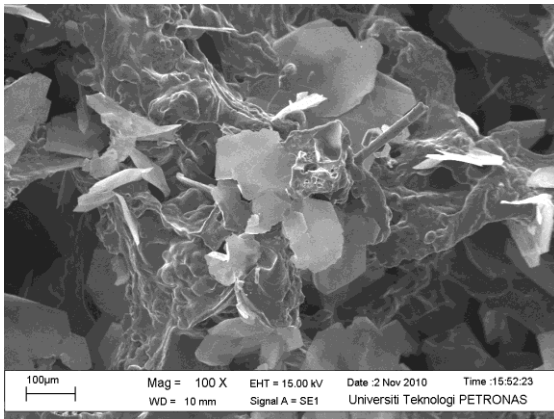


Fig. 34 (a)

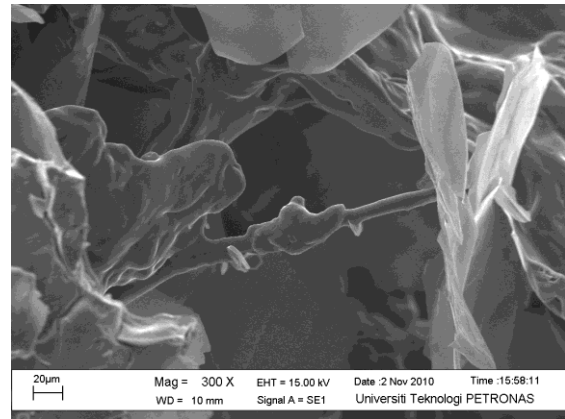


Fig. 34 (b)

Fig. 34: (a): Inner image-magnification 100; (b): Inner image-magnification 300

#### 4.1.4.1 ANALYSIS AND DISCUSSION FOR SEM

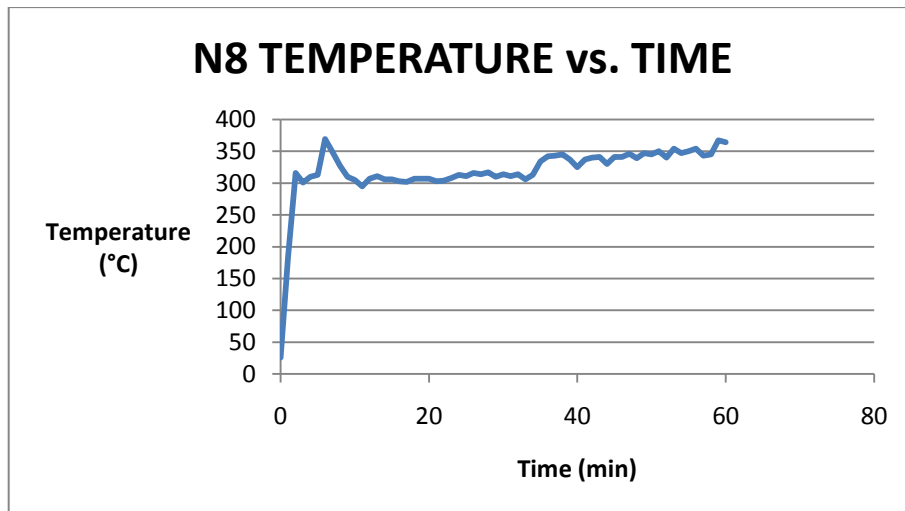
| Samples \ Characteristics | N8                                      | R0.5                                      | G0.5   | G0.24  |
|---------------------------|---|---|--|--|
| Outer char                | Smooth outer char layer, not much pores | Smooth outer char layer, not much pores   | A lot of pores                                     | A lot of pores and fibres                        |
| Inner Char                | A lot of flakes                         | Lesser flakes than N8 and a lot of fibres | Lesser flakes than N8 and a lot of fibres          | Much flakes than R0.5 and G0.5, but lesser fibre |
| Viscosity                 | Low viscosity, easy to apply            | Less than G0.5, not too hard to apply     | Very high viscosity, sticky and very hard to apply | Less than G0.5                                   |

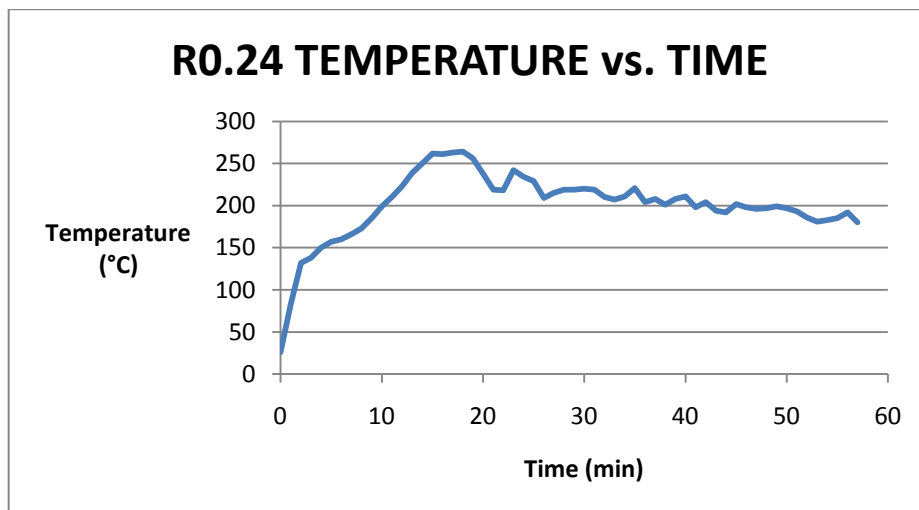
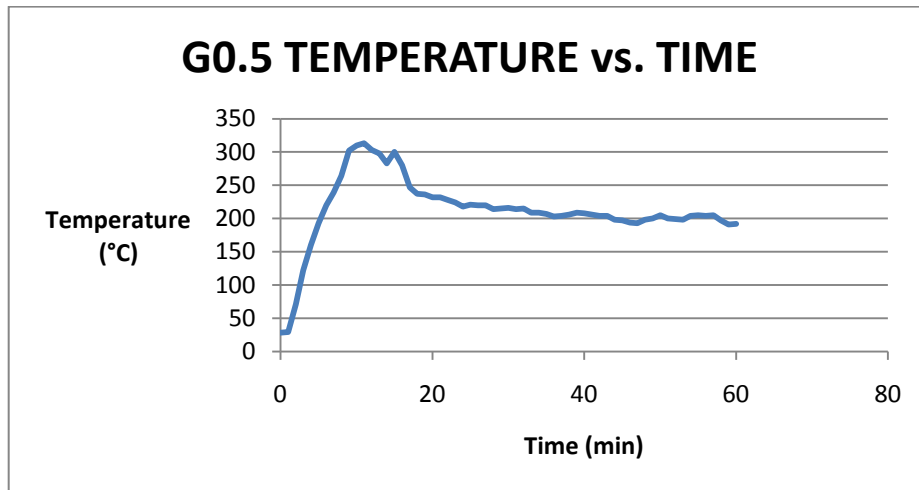
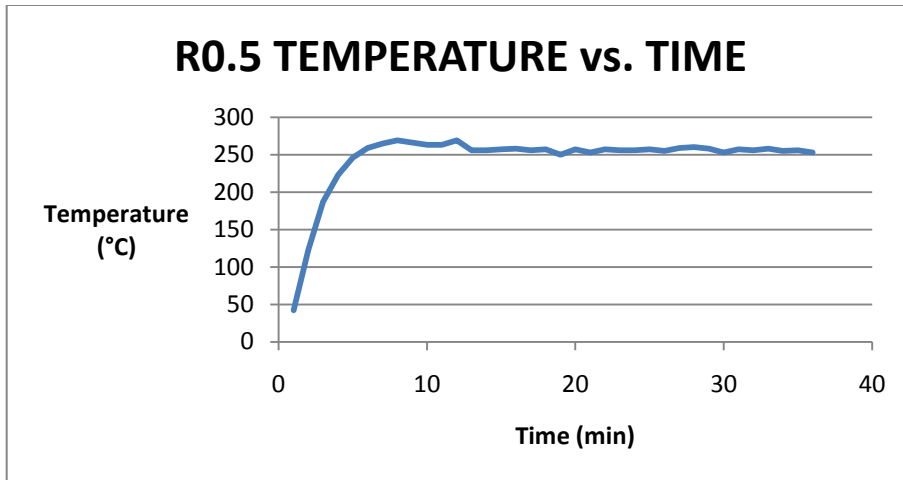
The charring layer protects the matrix materials, and its protective property depends on the physical and chemical structure of the charring layer. Researches indicate that there are other elements in the charring-layer structure. But the non-charring element is easy to be oxygenated and very unstable on chemistry aspects. There are two ideal typical charring structures listed as follows. There are a great deal of integrated closed honeycomb pores in Fig. 6(a); such structure can form adequate temperature grads in the charring layer and protect the molten mass and matrix below. But the other structure is nonideal; there are many channels and apertures, and the gas and molten mass of polymers can overflow to the entry of the flame-region. Thus the isolation effect of heat transfer is inferior [8]. The physical structure of the charring layer plays a very important role in the performance of the flame retardant [8]. The formation of the final charring layer and their morphological structures is studied by SEM. If the structure of the charring layer inclines to be more compact and more homogeneous, the intension of

charring layer improves largely, and the effect of the flame retardant is better. The irregular mini-pore structures of spongy foams in the charring layer explains the dehydration charring of PER and frothing of Mel proceeds in the range of rather appropriate temperature. Moreover, the intumescent layer is compact and spongy, and the heat insulation effect. The different aperture surface tensions in the course of gas cavities lead to the asymmetry of abscess, and the surface tension relies on the viscosity and symmetry of the coating. The intumescent charring layer with many mini-pores acts as the effect of the flame retardant, heat insulation and protecting inner matrix materials.

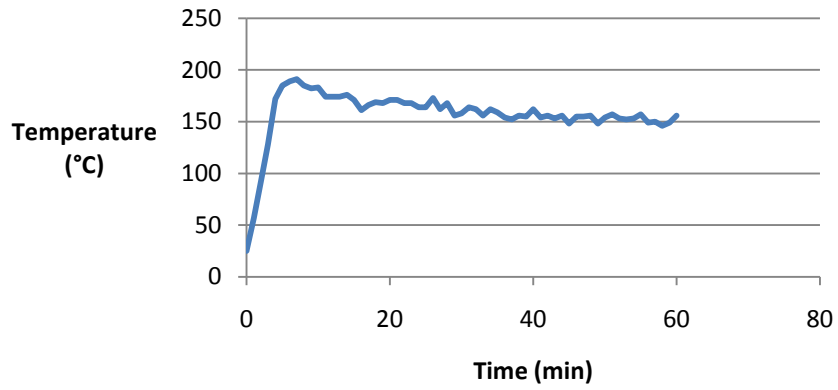
#### 4.1.5 DIRECT FIRE TEST (THERMAL TEST)

Direct fire test, using Bunsen burner is used to determine the value of back steel during the burning. The temperature data taken using digital thermocouple.

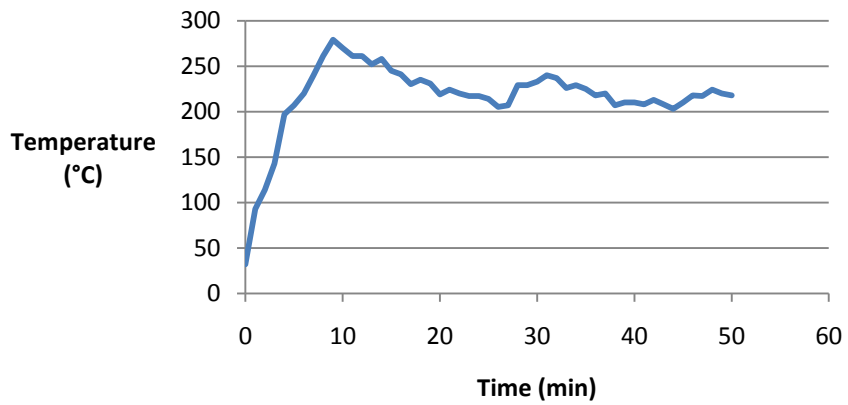




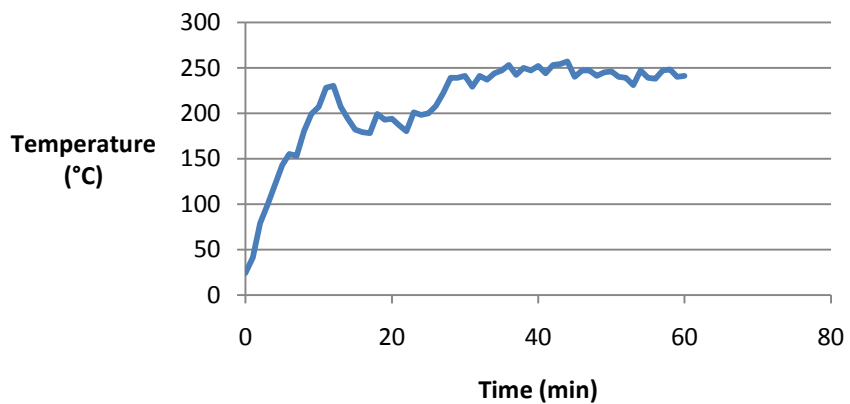
### GO.24 TEMPERATURE vs. TIME



### R0.12 TEMPERATURE vs. TIME



### GO.12 TEMPERATURE vs. TIME



#### 4.1.5.1 ANALYSIS AND DISCUSSION

From the direct fire test, the back steel temperature of sample N8 is proved to have the highest value of temperature when directed with fire. The intumescent coating reinforced rockwool and glasswool have a better resistance to fire, therefore protects the steel from high temperature. From the graph of sample N8, it is shown that the value of temperature of the back steel plate is higher than 300°C in average. As compared with reinforced intumescent coatings, those are having quite low value of back steel temperature which is mostly below 300°C. From the experiment done, the best formulation is formulation G0.24. It has good structure inside and out, and easier to apply as compared with G0.5 which is quite sticky.



## CHAPTER 5

### Conclusion and recommendation

#### 5.1 CONCLUSION

Intumescent coating consists of 3 main components which are acid source, carbon source and blowing agent. The right amount of those 3 components results in a good sample of intumescent coating. Other than that, fibre reinforcement is a good way in improving the intumescent coating. The fibre which is having high thermal resistance somewhat is a medium for the coatings components to attached and bonded with. The fibre makes the char structure tougher. Different fibre has different properties. In this project, two different fibres are used; rockwool and glasswool. Rockwool has a higher density value as compared to glasswool. So, it is quite hard to apply the intumescent coating reinforced glasswool of higher amount as compared to rockwool. The best formulation is formulation G0.24 as it has good structure inside and out, and easier to apply as compared with G0.5 which is quite sticky. In direct fire test also, it has quite good resistance to fire.

## 5.2 RECOMMENDATION

Intumescent coating is essential in protecting structures. As it is widely used nowadays, many research and improvements is made. Fibre is a good reinforcement in improving the ability of intumescent coating. Yet, much more can be done to obtain the best result. These are the recommendations:

- i. instead of using Bunsen burner which result in not uniform result, a proper calorimeter should be prepared to obtain the back steel temperature of the sample.
- ii. Other high resistance fibre could be used for reinforcement
- iii. APP, PER and MEL are the common basic components for acid source, carbon source and blowing agent. More research can be done to obtain a better component to replace those remaining components for a better improvement in intumescent coatings
- iv. Other tests like DSC and TGA can be done to study more on the thermal performance of the intumescent coatings.

## APEENDIXES



Figure 35: Glasswool used in study



Figure 36: Rockwool used in study

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