**FINAL YEAR PROJECT**

**REPORT**

**Title:**

# Glass Fiber Reinforced-Fire Retardant Composites For Offshore Platforms Application

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# CERTIFICATION OF APPROVAL

## GLASS-FIBER REINFORCED FIRE RETARDANT COMPOSITES FOR OFFSHORE PLATFORMS APPLICATION

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Approved

_____

(Associate. Prof. Dr Faiz Ahmad)

Project Supervisor

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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 except 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.

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# Contents

# ABSTRACT

Composite materials have been selected and introduced as one the main materials in all industries, especially construction of buildings and also for oil and gas industry applications for the past few decades. However, composite plastics are facing the same problem as most materials, especially regarding the fireproofing characteristics when caught in a fire. This report is focusing on preparing, developing and analyzing the performance of fire retardant composites, which is glass fiber reinforced fire retardant composites for potential application on offshore platforms by using fire retardant coating called Intumescent Fire Retardant (IFR) coating. IFR coating is an organic resin based product functioning as fire retardant coating where it can be applied to metallic materials, polymers, textiles, wood as well as structural steel in buildings, storage tank in order to protect them from weakening when encounter elevated temperature in a fire. In this paper, an Intumescent Fire Retardant (IFR) coating formulations which consist of three main agents; Acid Source; Ammonium Polyphosphate (APP), Carbon source; Expandable Graphite (EG) and blowing agent; Melamine (MEL) followed by epoxy, Boric Acid, Polyamide Hardener (TETA) and few additives are developed. The formulations were varied with the presence of alumina as fillers. The developed coatings later were applied to the glass fiber using impregnation method, in which the coatings act as the composites matrices and will be reinforced by Chopped Strand Mat Glass Fiber. The cured samples later were tested on fire retardant performance test using portable Bunsen burner. The char expansion as well as heat shielding will be thoroughly observed and the results obtained will be further studied using X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) techniques to observe the thermal analysis and char morphology of the formulations.

# List of Figures

## List of Tables

# List of Graphs

# CHAPTER 1

# PROJECT BACKGROUND

## 1.1 Background of Study

Over the past centuries, steel have been widely used and become an important structure-based material in many applications such as in the construction of roads, cars, railways, buildings, infrastructures and also offshore oil and gas application (oil and gas platform). [1] Steel is chosen for most applications because of its good fatigue strength, durability and flexibility and it is a non-combustible material which assembles a good ductility. Steel can undergo large plastic deformation before failure and hence providing large reserve strength. This property is referred to as ductility. A ductile structure has energy-absorbing capacity and will not incur sudden failure easily.

However, steel exhibits one major disadvantages; fireproofing. The strength of steel will be reduced substantially when heated at temperatures commonly observed in building fires. Besides, steel conducts and transmits heat from a burning portion of the building at a fast rate. [2,3] Steel begins to lose its structural properties between 470 and 500$^{o}$C [4]. Therefore, prevention of the structural collapse of the building, which can occur if steel elements reach a temperature above 550$^{o}$C, is supreme to ensuring the safe evacuation of people from the building, and is a main requirement of building regulations in many countries. For the past few years, researches have been made to protect steel with fire retardant elements to withstand the increase in temperature of the steel when exposed to fire [1, 26].

Through the advancement of materials engineering, in the past few decades, the field of composite materials has developed tremendously such as application in aircraft, spacecraft and even in oil and gas industry. The fiber reinforced composites are being used to reduce the use of steel for certain parts in the industries due to its cost-effective, light weight, high strength, and resistance to corrosion [5]. Composite can be characterized as a material composed of two or more different materials, with the properties of the resultant material that make up the composites [6]. The characters of composite materials which include low in density, high strength and stiffness, chemical

and corrosion resistance make it as one of the best alternative to replace the widely used metals and alloys.

However, the traits of composite materials especially concerning about its combustibility and reaction towards fire are sill the main concern. In order to increase the market penetrations and increasing the safety, improvements in fire retardant characteristics should be the main priority [7]. Therefore, the development of fire retardant plastic composites, specifically fiberglass, involving fire retardant intumescent coatings for offshore applications will be discussed later in this project. The use of fire retarded coatings such as intumescent coating is one of the easiest, oldest and one of the most efficient ways to protect a material against fire [9-10].

## 1.2 Problem Statement

The oil and gas industries has begun shifted to use composites materials especially glass-fiber reinforced plastics composites (GFRP) and fiber reinforced plastics (FRP) in variety of applications such as sea-water pipe system, storage tanks, offshore structures, tubing and shelters[10-11]. The main objective is to reduce the use of steel in offshore platforms. However, fiberglass are also facing problem regarding fire-proofing characteristics and their combustibility when exposed to fire [12]. Common fiberglass will soften between 800-900$^{o}$C when exposed to fire (Amir et. al, 2013).

As a solution, the fire retardant intumescent coatings should be applied to the material and samples to retard the spread of fire. In developed countries such as the United States and United Kingdom, intumescent fire protective coatings are widely used as passive fire protection in oil and gas industries, civil buildings, chemical plants and other facilities However, as in Malaysia there is still a shortage of wide-ranging research in the field of fire protective coatings and of the awareness of its role and ability in the inhibition of fires [8]. As the use of IFR coating in steel has been studied before, the impregnation method of glass fiber and IFR coating to form fire retardant glass fiber reinforced plastics are still under research and this scope of studies are really interesting especially for application in the offshore platforms.

## 1.3 Objective

The objective of this project is to prepare, develop and analyze the performance of glass-fiber reinforced fire retardant composites samples for potential application on offshore platforms, especially for the floor/deck parts of the platform as an alternative to reduce of the widely used steel frame works and alloys. Another objective is to study the effect of using inorganic fillers in the intumescent coating formulations in correspond to the fire retardant characteristics of the samples.

## 1.4 Scope of Study

The scope of study on this project is mainly focusing on using glass-fiber reinforced plastic impregnate with intumescent fire retardant coating in using different coating formulations and composition. Chopped Strand Mat E-Glass was reinforcing material while the intumescent coating which also consisting of Bisphenol A Epoxy resin with Polyamine Hardener (TETA) acted as the matrices. This project used impregnation method, in which the epoxy and intumescent coatings of certain formulation that have been developed acts as the resin while the reinforcing material consisted of the E-Glass glass fiber. The samples were prepared by using Hand Lay Up fiber glass construction technique. The fire retardant coatings were analyzed using Thermogravimetric Analysis (TGA). Fire performance test was then conducted by using portable Bunsen burner while the char produced from the test were finally tested with SEM (Scanning Electron Microscopy), and XRD (X-Ray Diffraction) techniques to observe the thermal analysis and char morphology of the coatings.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1. Composite Materials

A composite material is made when two or more materials – usually ones that have very different properties. The two combining materials work together to give the composite such unique properties [13]. However, within the composite you can easily tell the different materials apart as they do not dissolve or blend into each other.

Composite materials are not new, since materials are known to have been used by the ancient Egyptians, whom embedded straw in bricks to improve their structural capabilities [14]. Nowadays, the increases in numbers of the usage of composite materials are greatly seen. Nowadays, composites have been widely used for airframes, automobile components and even in the oil and gas industries, especially for the offshore applications.

Although composite materials seems to have more advantages for vast industrial applications and overcoming the usage of steel and alloys, there are still properties that needs further researches and investigations regarding the material; its fire-proofing characteristics and their reaction in the event of fire[12]. Therefore, in this chapter, related theories and literature review of previous work and research made on this field will be discussed.

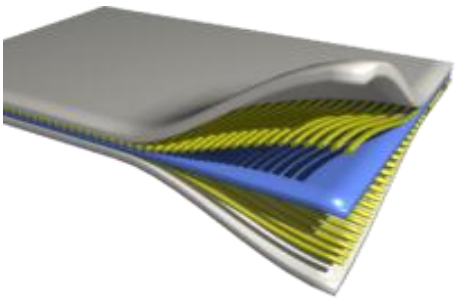


**Figure 1: Illustration of composite materials (reference from http://en.wikipedia.org/wiki/Composite_material)**

## 2.2 Glass-Fiber Reinforced Plastics (GFRP)

Fiberglass (also called glass-fiber reinforced plastic, GFRP) is a fiber reinforced polymer which is made from plastic matrix and reinforced by fine fiber of glass. Fiberglass is made of glass, similar to drinking glasses in the kitchen. Its major advantages are lightweight, extremely strong, and tough material [24]. When comparing with the carbon fiber, the material is far less brittle, and much cheaper. It can be easily formed using molding processes and its bulk strength and weight properties are also very advantageous when compared to metals.

### 2.2.1 Fiber Reinforcements

Fiber is a material consists of thousands and bunches of filaments, in which every filament is having a diameter between 5 to 15 micrometers allowing them to be produced by using textile machines [15]. Reinforcements can be continuous, woven or chopped fiber which can increase the mechanical properties of the composites. Continuity of the composites will specify the fiber length.

Glass, by far is the most widely used fiber in all applications due to its good characteristics which include high abundances, high tensile strength and cost-effective compared to other composites. Glass fibers are formed by 4 different forms; roving, woven roving, chopped strand and chopped strand mat. There are several grades of glass fiber that are produced commercially, for example E-glass, S-glass, R-glass, C-glass and Cemfil. The code "E" in E-glass fiber stands for electrical [16]. Almost 90% of the composites fabrication in aerospace industry used E-glass fiber as reinforcements. This is because of its high corrosion-resistence, high strength-to-weight ratio, low thermal conductivity, adequate optical properties, low electrical conductivity, dimensional stability, good energy savings and light in weight. Fiberglass cloth has a melting point of 1121˚C. Fiberglass has no true melting point, but it has a softening point of 846˚C which could be an advantage for its mechanical properties especially when it interacts with fire [25].

**Figure 2: Different physical forms supplied of glass reinforcements; fine ground, chopped, woven ( reference from http://en.wikipedia.org/wiki/Fiberglass)**

### 2.2.2 Chopped Strand Mat

Chopped strand mat or CSM is a form of reinforcement used in fiberglass. It consists of glass fibers laid randomly across each other and held together by a binder.It is typically processed using the hand lay-up technique, where sheets of material are placed in a mold and brushed with resin. Because the binder dissolves in resin, the material easily conforms to different shapes when wetted out. After the resin cures, the hardened product can be taken from the mold and finished. Using chopped strand mat gives a fiberglass with isotropic in-plane material properties. In this project, CSM will be used as the fiber reinforcement

### 2.2.3 Matrix Material

Besides having its own advantages, fiber has limits in its engineering applications, in which it cannot transmit load from one to another. The composite are consist of fiber and matrix material that are embedded together, where the matrix serves to bind and transfer load to the fiber and protect them again environmental attack and damage due to handling. In this research, epoxy resin is the type of matrix that is going to be used to fabricate fiber composites. Epoxy resin is almost totally transparent when cured. Epoxy is used as a structural matrix material or as structural glue in the aerospace industry.

Resin is a good resistance to most chemicals, good resistance to creep and fatigue, high strength and good electrical properties [18]. To fabricate the composites, fiberglass hand lay-up construction method will be used in this research. There are major advantages of using hand lay-up methods; low molding costs, it is widely and commonly used, it is possibility for large products small series products.

## 2.4 Fiberglass Hand Lay-Up Operation

Hand lay-up is one of the common methods of manufacturing fiberglass composites. It is the method of cutting lengths of fiber reinforcement off of rolls. The reinforcement most often comes in the form of chopped fiber, woven fiber, or stitched fiber. A release agent, usually in either wax or liquid form, is applied to the chosen mold. This will allow the finished product to be removed cleanly from the mold. Resin – typically a 2-part polyester, vinyl or epoxy – is mixed with its hardener and applied to the surface. Sheets of fiberglass matting are laid into the mold, then more resin mixture is added using a brush or roller.

The material must conform to the mold, and air must not be trapped between the fiberglass and the mold. Additional resin is applied and also possibly additional sheets of fiberglass.

Hand pressure, vacuum or rollers are used to make sure the resin saturates and fully wets all layers, and any air pockets are removed. The work must be done quickly enough to complete the job before the resin starts to cure, unless high temperature resins are used which will not cure until the part is warmed in an oven.[19] In some cases, the work is covered with plastic sheets and vacuum is drawn on the work to remove air bubbles and press the fiberglass to the shape of the mold.

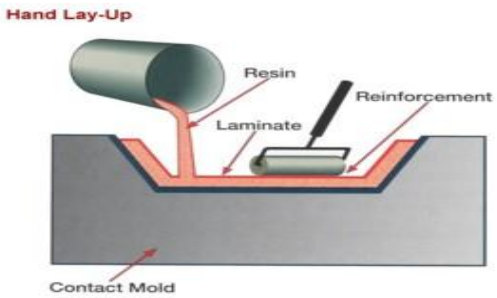



**Figure 3: Hand Lay-Up Method (Reference from http://maybach300c.blogspot.com/2012/08/manufacturing-processes.html)**

## 2.5 Fire Retardant Elements

### 2.5.1 Intumescent Fire Retardant Protective (IF) Coatings

Intumescent fire retardant (IFR) is a substance that swells as a result of heat exposure, thus increasing in volume and decreasing in density. IFR which act as protective coatings are widely used as passive fire protection in oil and gas industries, civil buildings, chemical plants and other facilities in developed countries.[8] Intumescences may be defined as —"thermally induced expansion of material". [21] In simple words, intumescences can also be defined as the ability of the coating, while being exposed to high temperature of fire, to swell or froth into a solid heat insulating layer and thus protecting the substrate from getting direct exposure to the flame which can damage the mechanical properties of the substrate. Intumescent fire protective coatings expand when exposed to a sufficiently high temperature.

When getting caught in a fire, the coating retains its expanded or foamed structure at high temperatures to provide a heat isolative layer which protects the substrate for a long period of time, approximately up to 3 hours to 4 hours. [22] This precious time may evacuate people from the building that is struck by the fire. The intumescent coating swells in size to form a char, which protects the steelwork for a specified period. [1]

. The char produced thermally insulates the underlying steel substrate and establishes a protective barrier against oxygen. This is important during fires because at temperatures above 500$^{o}$C steel loses its mechanical strength to a degree where collapse of the steel structure may occur with potential loss of lives or assets. [23]

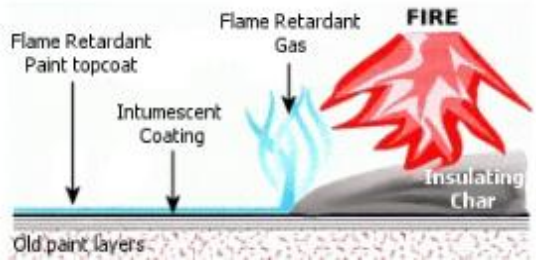



**Figure 4: Mechanism of intumescent coating (Reference from http://www.astroflame.com/intumescent-coating-walls.html)**

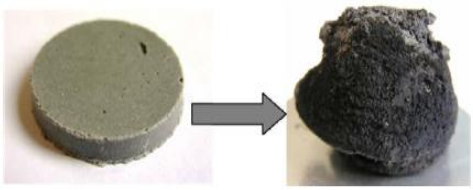


**Figure 5: Swelling of an intumescent coating (Reference from Jimezez et al. 2006)**

### 2.5.2 Basic Elements of Intumescent Coating

Intumescent coatings contain four basic elements: [24]:

- Acid source or catalyst; a dehydrating or carbonizing agent, such as Ammonium Polyphosphate (APP), which at temperatures above 2000$^{o}$C liberates polyphosphoric acid.
- Carbon source; organic substances which can be charred and turned into coal by pentaerythritol, dipentaerythritol and Expendable Graphite (EG).
- Blowing agent such as melamine, which under decomposition release gases (N2, NH3) and expands the char.
- Binder; such as epoxy resin makes the compounds contact each other.

### 2.5.3 Soft Char

These intumescent produce a light char, which is a poor conductor of heat, thus retarding heat transfer. Typically, these materials contain a significant amount of hydrates. As the hydrates are spent, water vapour is released, which has a cooling effect. Once the water is spent, the insulation characteristics of the char that remains can slow down heat transfer from the exposed side to the unexposed side of an assembly. Soft char producers are typically used in thin film intumescents for fireproofing structural steel as well as in firestop pillows. Typically, the expansion pressure that is created for these products is very low, because the soft carbonaceous char has little substance, which is beneficial if the aim is to produce a layer of insulation.

### 2.5.4 Mechanism of Intumescent Coating

The mechanism of intumescent is usually started with the acid source breaks down to yield a mineral acid, then it takes part in the dehydration of the carbonization agent to yield the carbon char, and finally the blowing agent decomposes to yield gaseous products. Then, the char will swell and this will provide an insulating multi-cellular protective layer. This shield limits at the same time the heat source to the substrate and the mass transfer from the substrate to the heat source resulting in a conservation of the underlying material [1].

Expandable graphite is formed by treating crystalline graphite, which is imposed of stack of parallel planes of carbon atoms, with intercalants such as sulphuric acid and/or nitric acid. When exposed to heat source, the intercalation compound ($H_2SO_4$) decomposes into gaseous product ($SO_2$ and $H_20$). The high layer pressure resulting from decomposition of intercalation compounds produces a strong push force between the graphite layers, so the graphite basal planes can be pushed apart . The decomposition of sulphuric acid and redox reaction between $H_2SO_4$ and carbon are responsible for most of the expansion process. The fused resin and carbonaceous compound can stick a large amount of expanded graphite in expanding process.

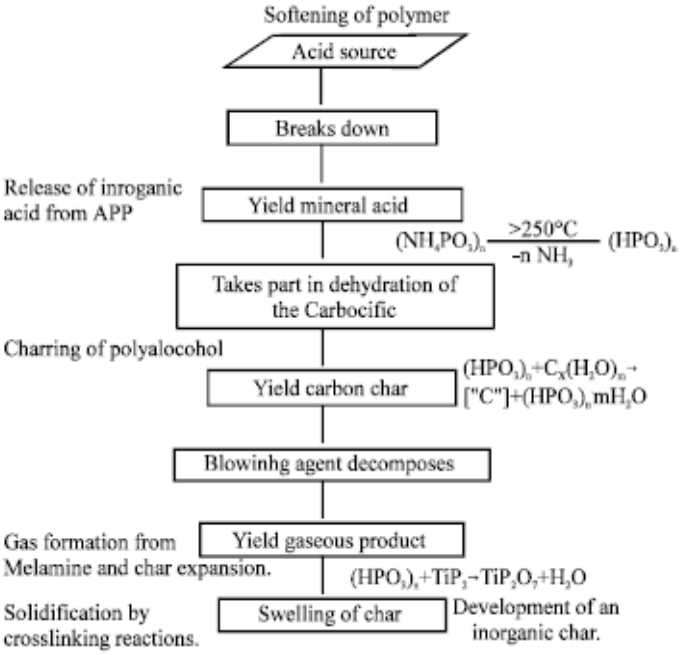



**Figure 6: Mechanism of intumescent coatings in a fire (Jimenez et. al, 2011)**

# CHAPTER 3

# METHODOLOGY

## 3.1 Research Methodology

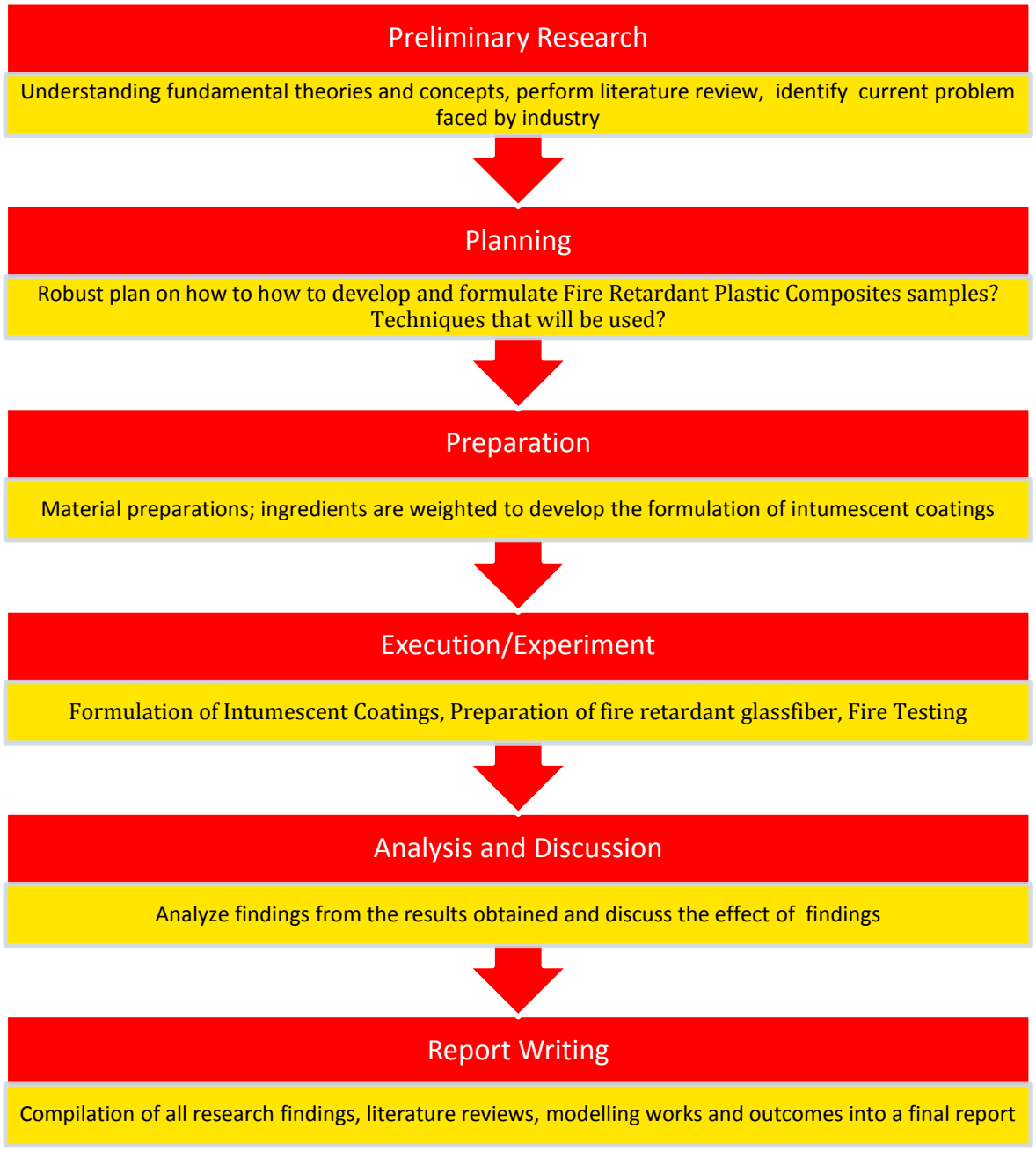



**Figure 7: Research Methodology**

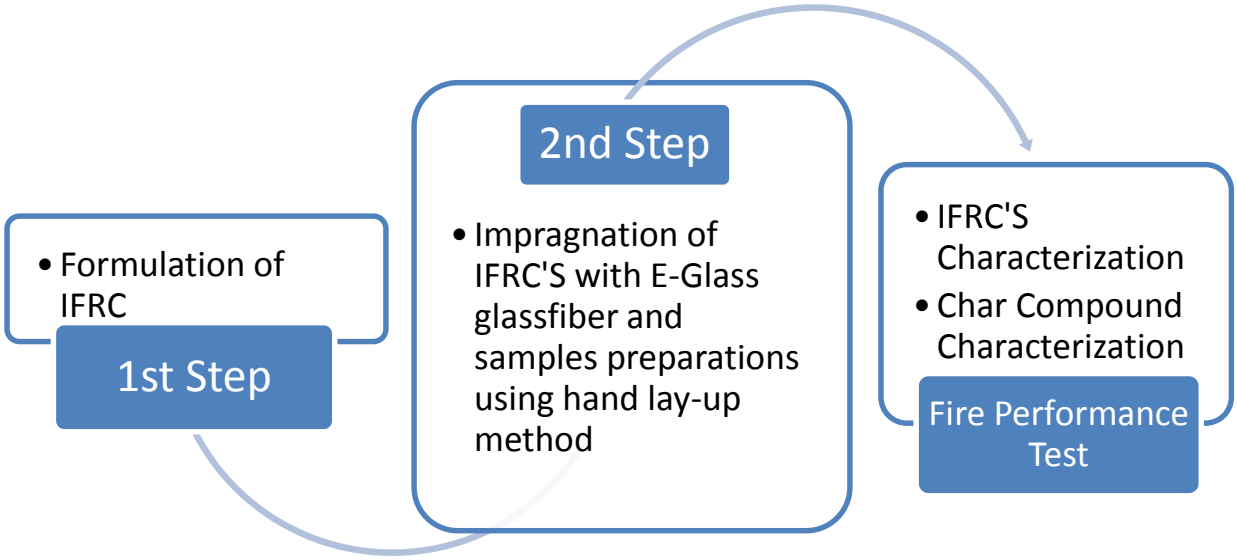



**Figure 8: Steps towards preparation of Fire Retardant Glass-Fiber Reinforced Plastics Samples**

## 3.2 Experimental Materials and Procedures

### 3.2.1 Intumescent Coating Materials

The main ingredient of Intumescent Coating to be used in this project are ammonium polyphosphate (APP) as acid source, expandable graphite (EG) as a carbon source, melamin (MEL) as blowing agent and boric acid (BA) as additive. Mineral filler which is alumina is selected in intumescent coating to improve the fire retardant performance. Bisphenol-A (BPA) and tertraethylene tetramine (TETA) are used to bind and harden all the intumescent ingredients to increase its physical properties and characteristics.

### 3.2.2 Intumescent Coating Formulation Preparation

A range of formulations containing IFR components (APP, EG, MEL, Boric Acid, Epoxy and hardener) were developed for this project. Based on the formulations, there are 6 different formulations that the author had developed. The main differences of all formulations are in terms of percentage of BPA (Epoxy resin), TETA (hardener) and alumina in weight percent. However, the author will try to find the relationship between the percentage of the alumina (filler) and its effect on the performance of Fire Retardant Glass fiber Reinforced Plastics.

**Table 1: Intumescent Coating Formulation**

| Sample | APP (%) | B.A (%) | E.G (%) | MEL (%) | BPA (%) | TETA (%) | Alumina (wt %) |
|---|---|---|---|---|---|---|---|
| **A0** | 11.11 | 11.11 | 5.56 | 5.56 | 44.92 | 21.71 | - |
| **A1** | 11.11 | 11.11 | 5.56 | 5.56 | 44.26 | 21.38 | 1 |
| **A2** | 11.11 | 11.11 | 5.56 | 5.56 | 43.60 | 21.05 | 2 |
| **A3** | 11.11 | 11.11 | 5.56 | 5.56 | 42.94 | 20.72 | 3 |
| **A4** | 11.11 | 11.11 | 5.56 | 5.56 | 42.28 | 20.39 | 4 |
| **A5** | 11.11 | 11.11 | 5.56 | 5.56 | 41.62 | 20.06 | 5 |

| | | |
|---|---|---|
| A0 = Alumina 0% | A1= Alumina 1% | A2= Alumina 2% |
| A3= Alumina 3% | A4= Alumina 4% | A5= Alumina 5 % |

**Steps involved in preparing IFRC'S** :

1) APP, MEL, BA and Alumina is mixed according to the specific ratio.

**Figure 9: APP, MEL, BA and Alumina (filllers) after weighted**

2) The mixed ingredients are then grind for 60 seconds using grinder.


**Figure 10: Mortar Grinder in Block 17, UTP**

3) Later the EG being added to the ground mixture.

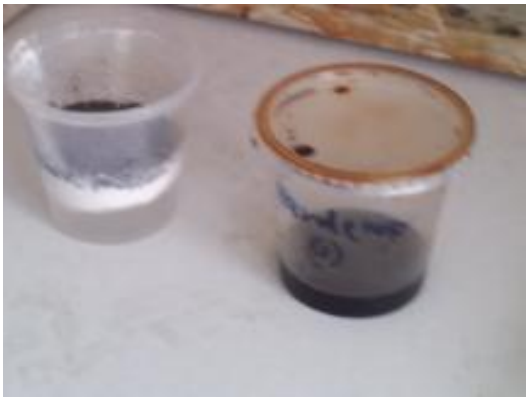


**Figure 11: EG was added into the mixed ingredients of APP, BA, and Alumina**

4) BPA and TETA were stirred in the mixer. Subsequently the mixture is added to BPA and TETA.
5) The mixture is mixed using a mixer in Block 17, Mechanical Engineering Department, Universiti Teknologi PETRONAS for 20 minutes.

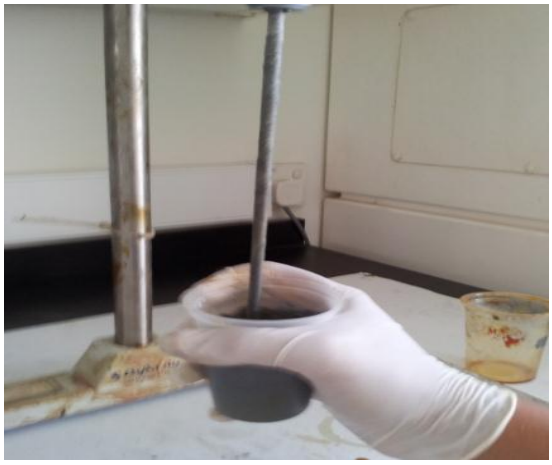


**Figure 12: Mixture is mixed using Mixer in Block 17, UTP**

### 3.2.3 Impregnation Method of Intumescent Coating with Glass Fiber

In this project**,** impregnation is one method that will be used to develop, formulate and produce the sample of fire retardant plastic composites. Kandola et. al (2002), stated that fabrics layers are impregnated with the resins in determining the effect of intumescence on the burning of polyester-resin-contain composites. Shaun et al. (2002), also has adopted this technique in which MP, PEPA are impregnated to the samples of flame retarded polypropylene(PP) to investigate the flame retardation and char formation mechanism of intumescent flame retarded polypropylene(PP) composites containing melamine phosphate(MP) and pentaerythritol phosphate (PEPA)

Epoxy resin is the type of matrix that is going to be used in this research to fabricate fiber composites. During the fabrication of the epoxy resin, the epoxy resin formulation will be mixed and impregnate with the intumescent based coating formulation which consists acid source; Ammonium Polyphosphate (APP), carbon source (EG), blowing agent (melamine), epoxy-resin as the binder and hardener which acts as the curing agent. Meanwhile, acid substance such as boric acid can be used as the additive for these formulations.

After the epoxy-resin intumescent coating formulation have been made and act as the matrix material, the glass-fiber will be constructed and reinforced using hand lay-up construction method. The sample produced, so-called fire retardant plastic composites will undergo fire testing procedures; using the Bunsen burner and also heated in the furnace.

The physical properties characteristics of the composites will be observed, especially the formation of the char and swelling of the coatings. The sample piece of the char formed tested will later be further tested using TGA Anlysis, X-ray Diffraction method (XRD) and Scaning Electron Microscope (SEM) to investigate the characteristics of the substances formed after the reaction mechanism during the fire in the char.

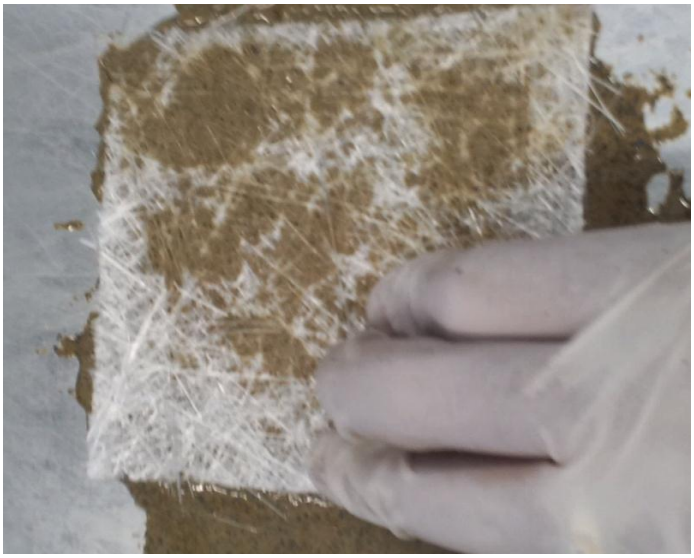


**Figure 13: Impregnation method to prepare the fire retardant glass-fiber samples**

### 3.2.4 Fire Retardant Glass Fiber Preparation

Glass wool with 2.0 mm thickness is used as the fiber. The glass wool substrate was cut using scissors into dimension 10cm x 10cm for the ease of fire testing purpose. After the cutting process is done, the glass wool will be reinforced and impregnated with the intumescent fire retardant coatings that have been prepared. Figure 14 shows the hand lay-up method on preparing the samples (Khairul, 2013).

**Figure 14: During hand lay-up process**

### 3.2.5 Fire Retardant Glass Fiber Reinforced Plastic Samples

Few fire retardant glass fiber reinforced plastics have been prepared by the author according to the IFR formulations that have been developed before. The samples are prepared by using hand lay-up reinforcement method. The samples produced are 10cm x 10cm in dimensions.

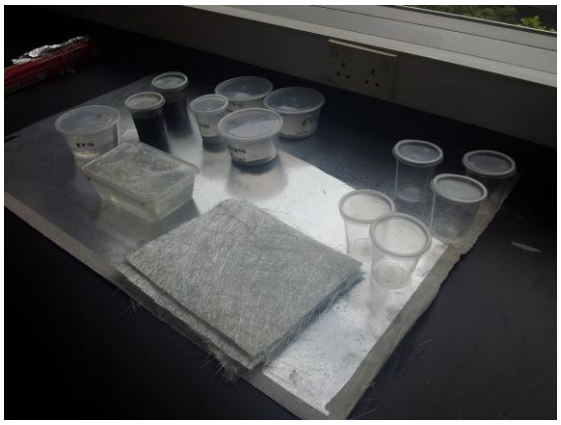


**Figure 15: Materials to prepare the samples**

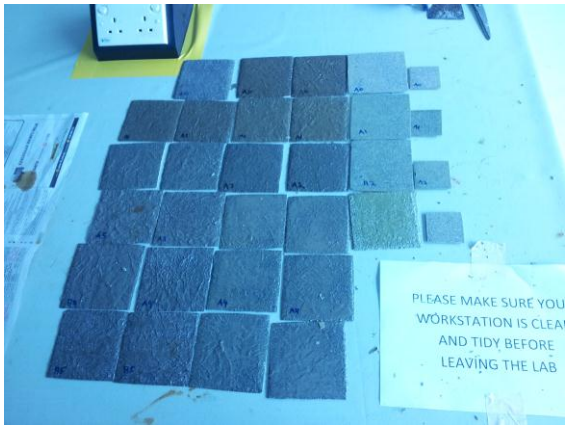



**Figure 16: Glass-Fiber Reinforced Fire Retardant Composites**

## 3.2 Fire Performance Test

### 3.3.1 Bunsen Burner Test

This test was used to examine thermal performance of the intumescent coating.

During the testing, the temperature profile of the fire retardant glass fiber samples are measured using digital thermo logger and thermocouple . In this experimental work, 800 ºC was chosen as the critical temperature for the glass fiber to ensure a high level of safety [25]. However, to ensure the data (temperature) recorded are of high level of accuracy, there are few steps and precautions that need to be taken care of as mentioned below.

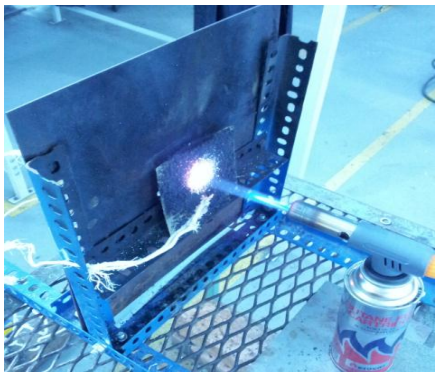


**Figure 17: Bunsen Burner Test**


**Figure 18: Data Logger 1**

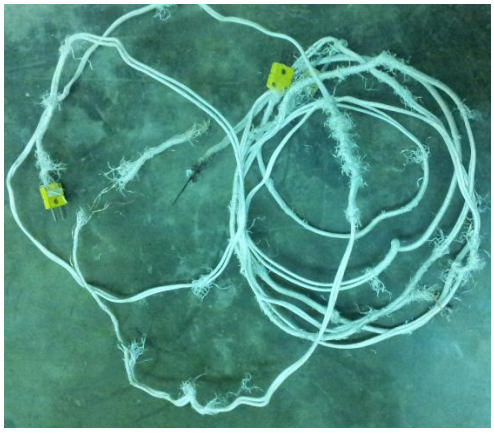


**Figure 19: Thermocouples used**

Steps Taken to ensure efficient results:

1) Make sure the samples are placed between the holes on the metal board to get a better temperature when doing fire testing.

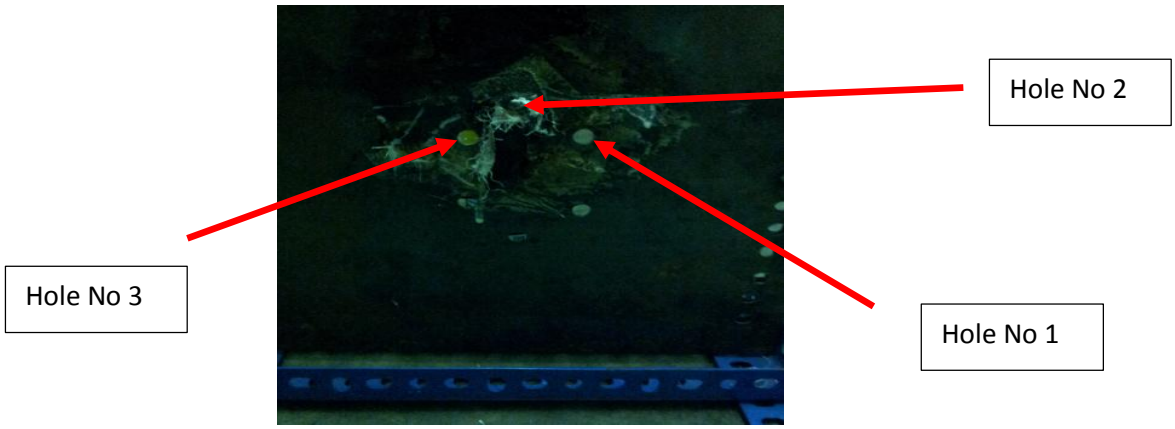



**Figure 20: Holes on the metal board**

2) Set a specific length between the source of fire and the point at which the fire will strike on the sample. The author used 9 cm lengths (own estimation).

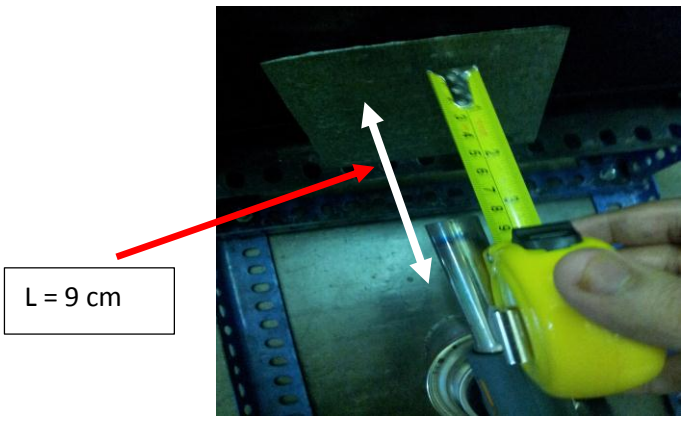



**Figure 21: Length between the source of fire and the samples**

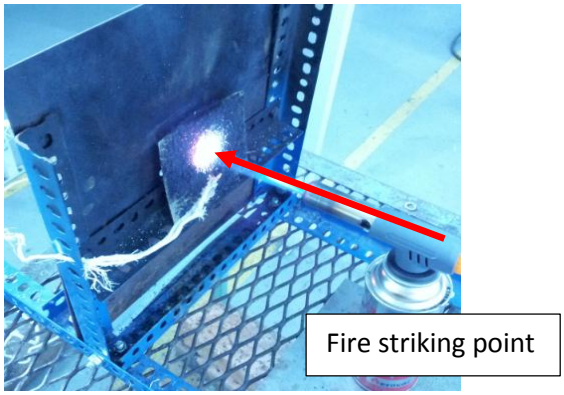



**Figure 22: Length between the source of fire and the samples**

3) Make sure the thermocouples really touch the samples to get accurate temperature.

**Table 2: Project Gantt Chart**

## 3.3 Gantt Chart of the Project

| Project Activities | Weeks | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | FYP1 | | | | | | | | | | | | | | FYP2 | | | | | | | | | | | | | | |
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Project Scope Validation | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Project Introduction | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Submission of Extended Proposal** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Identify material and equipment | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Proposal Defence** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Detail Study** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Submission of Interim Draft Report** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Finalized Procedure | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Conducting Experiment | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Result analysis and discussion | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Submission of progress report** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Preparation for Pre-SEDEX | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Pre-SEDEX** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Submission of draft report | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Submission of technical paper and dissertation** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Oral presentation** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| **Submission of project dissertation hardbound** | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Processes  Key Milestone

# CHAPTER 4

# RESULT AND DISCUSSION

## 4.1 Prepared Samples

All the process involved in preparing the samples of the project has been carry out by the author. The samples, so called Fire Retardant Glass fiber Reinforced Plastics have been successfully developed. The coating, as the resin which was developed based on the formulation later impregnated with the glass fiber which acts as the reinforcement. Pieces of the coating then was sent for Thermo gravity Analysis (TGA) to investigate the degradation temperature of the intumescent fire retardant coatings (IFR).

As the main objective of this project is concerning more on fire retardant characteristics, the samples produced then are tested for fire retardant performance using Bunsen burner testing. As for now, this is the author's main progress. Later, the fire testing result, which focus more on the formation of the char will be analyzed by using Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) if the time permits. Basically, the SEM will be useful to observe the morphology and structure of the char produced, meanwhile X-ray Diffraction (XRD) analysis will focus more on finding the composition of the char residue.

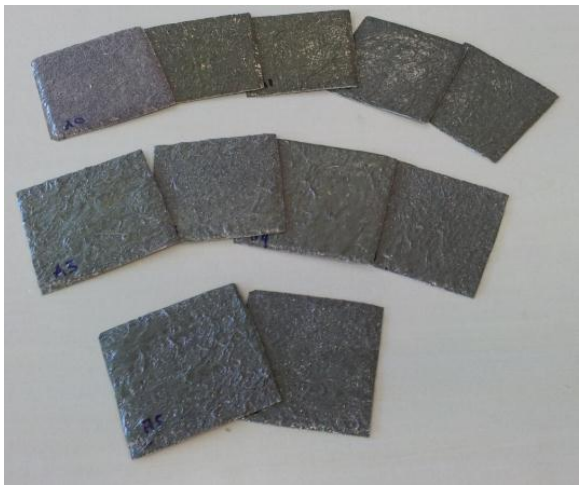


**Figure 23: Glass Fiber Reinforced Fire Retardant Composites**

## 4.2 Fire Test (Bunsen Burner Test)

The temperature of the fire retardant glass fiber reinforced plastics samples are measured during the Bunsen burner testing. The temperature is recorded for every 5 minutes until 60 minutes of testing. However, it is stressed here that the temperature that the author had measured and recorded using thermocouples and data logger are the back temperature of the samples at holes No.1 (refer Figure 20), as we are interested with the insulation effect of the char towards the back temperature of the samples which was formed during the fire test. For safety, the front temperature should not be exceeding 1000oC.

### 4.2.1 Heat Shielding Results

**Sample: A0**

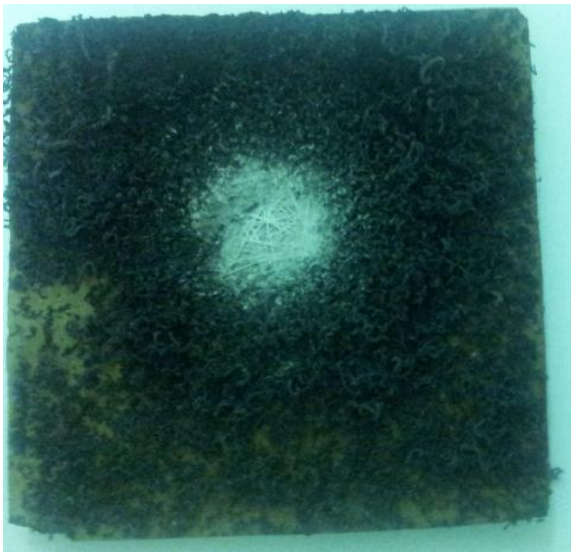


**Figure 24 After Fire Test (front)**

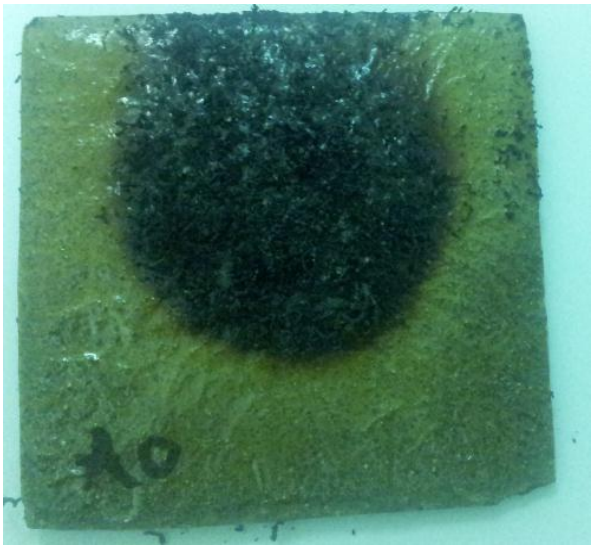



**Figure 25: After Fire Test (back)**

Observation for Sample A0:

1) Front: Soft char was formed mostly at the region the fire strike the sample after one hour
   : There is a "crack" region somehow after more than half an hour of heating

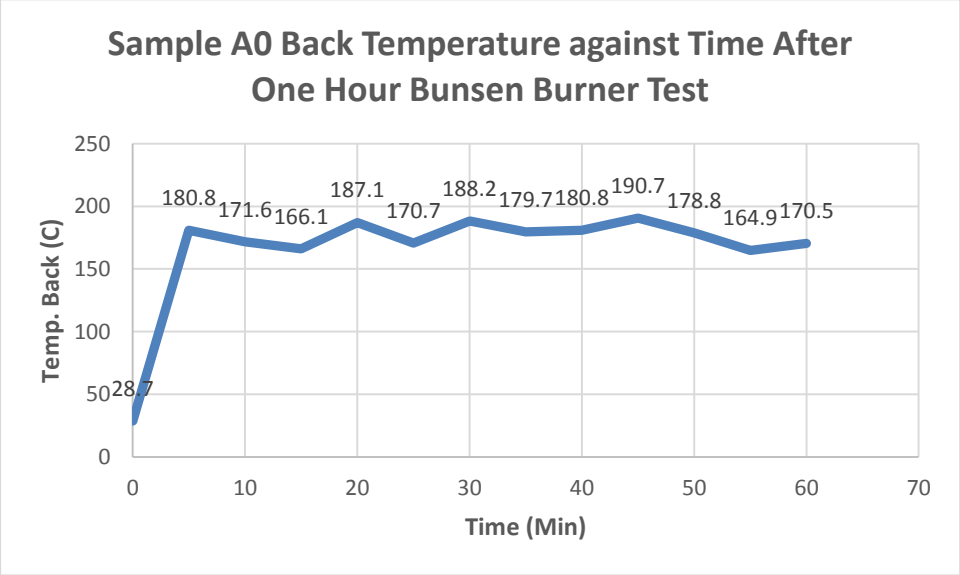
2) Back : Char was formed only at the region the fire strike the sample
   : Other region remains as usual

- The sample does not melt or soften after one hour of fire testing.

**Table 3: Sample A0**

| Time (min) | Temperature Back (°C) |
|---|---|
| 0 | 28.7 |
| 5 | 180.8 |
| 10 | 171.6 |
| 15 | 166.1 |
| 20 | 187.1 |
| 25 | 170.7 |
| 30 | 188.2 |
| 35 | 179.7 |
| 40 | 180.8 |
| 45 | 190.7 |
| 50 | 178.8 |
| 55 | 164.9 |
| **60** | **170.5** |

Final Temperature




**Graph 1: Time against Temperature for Sample A0**

**Sample: A1**

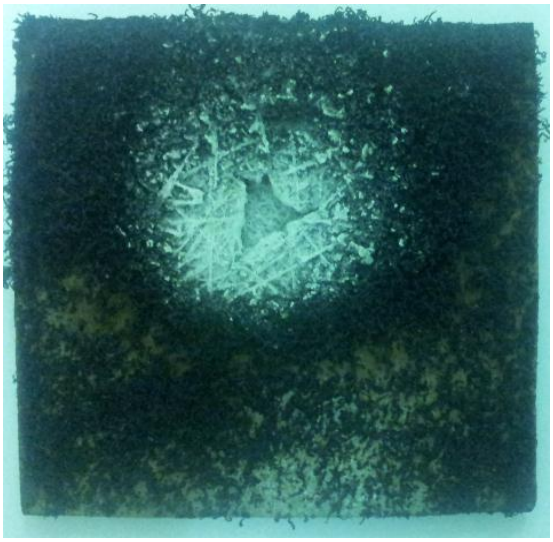


**Figure 26: After Fire Test (front)**

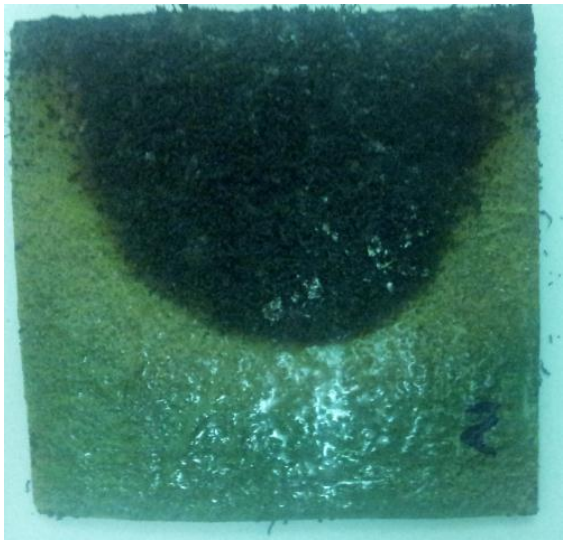


**Figure 28: After Fire Test (back)**

Observation for sample A1:

3) Front: Soft char was formed mostly at the region the fire strike the sample after one hour
: There is a "crack" region somehow after more than half an hour of heating

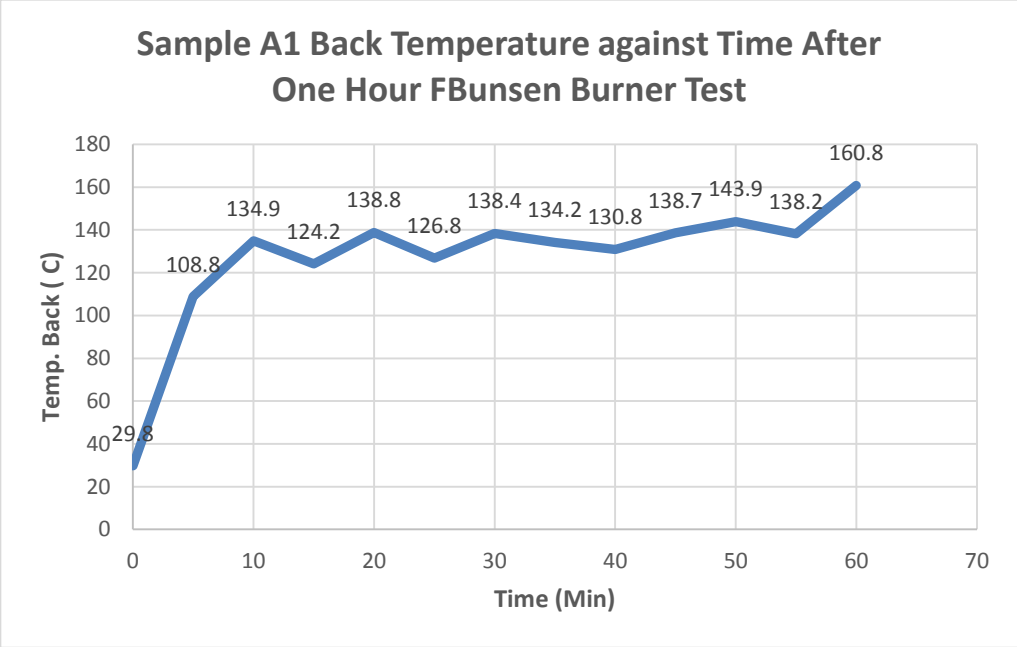
4) Back : Char was formed only at the region the fire strike the sample
: Other region remains as usual

- The sample does not melt or soften after one hour of fire testing.

**Table 4 : Sample A1**

| Time (min) | Temperature Back (°C) |
|---|---|
| 0 | 29.8 |
| 5 | 108.8 |
| 10 | 134.9 |
| 15 | 124.2 |
| 20 | 138.8 |
| 25 | 126.8 |
| 30 | 138.4 |
| 35 | 134.2 |
| 40 | 130.8 |
| 45 | 138.7 |
| 50 | 143.9 |
| 55 | 138.2 |
| **60** | **160.8** |

Final Temperature




**Graph 2: Temperature versus Time for Sample A1**

## Sample A2

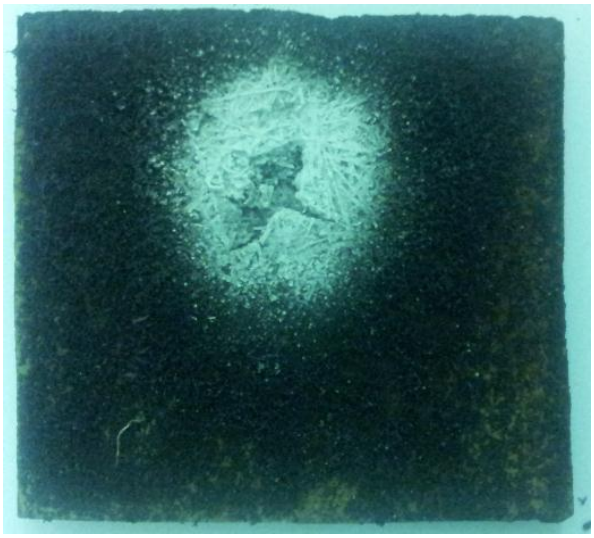


**Figure 29: After Fire Test (Front)**

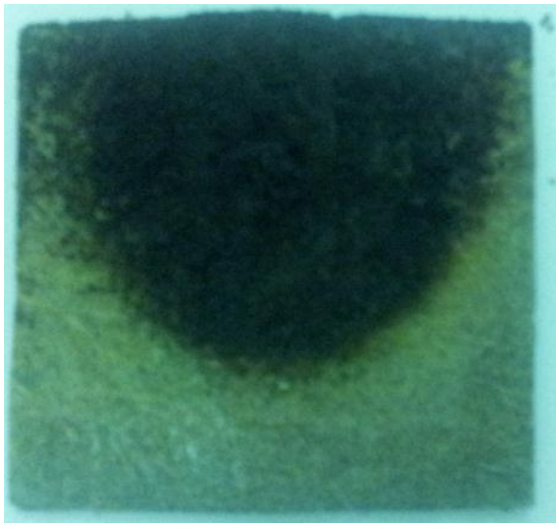


**Figure 30: After Fire Test (back)**

Observation for Sample A2:

5) Front: Soft char was formed mostly at the region the fire strike the sample after one hour
: There is a "crack" region somehow after more than half an hour of heating

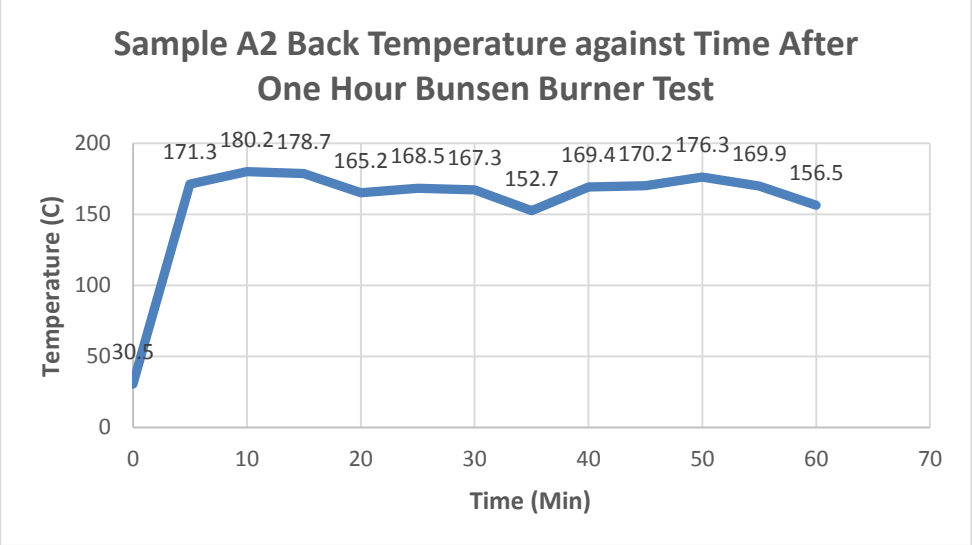
6) Back : Char was formed only at the region the fire strike the sample
: Other region remains as usual

- The sample does not melt or soften after one hour of fire testing.

**Table 5: Sample A2**

| Time (min) | Temperature Back (oC) |
|---|---|
| 0 | 30.5 |
| 5 | 171.3 |
| 10 | 180.2 |
| 15 | 178.7 |
| 20 | 165.2 |
| 25 | 168.5 |
| 30 | 167.3 |
| 35 | 152.7 |
| 40 | 169.4 |
| 45 | 170.2 |
| 50 | 176.3 |
| 55 | 169.9 |
| **60** | **156.5** |

Final Temperature




**Graph 3: Time versus temperature for Sample A2**

**Sample A3**

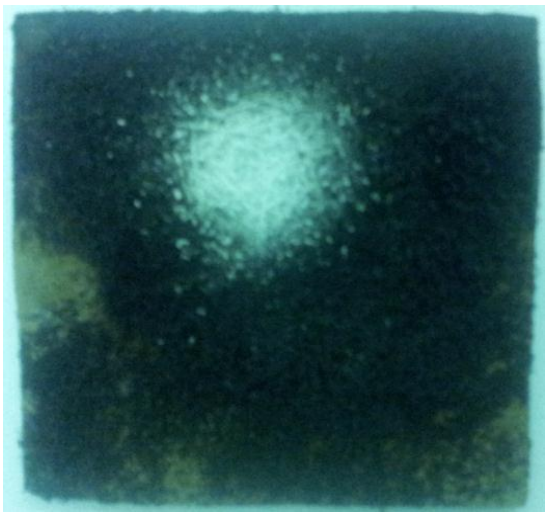


**Figure 31: After Fire Test (front)**

**Figure 32: After Fire Test (back) 1**

Observation for Sample A3:

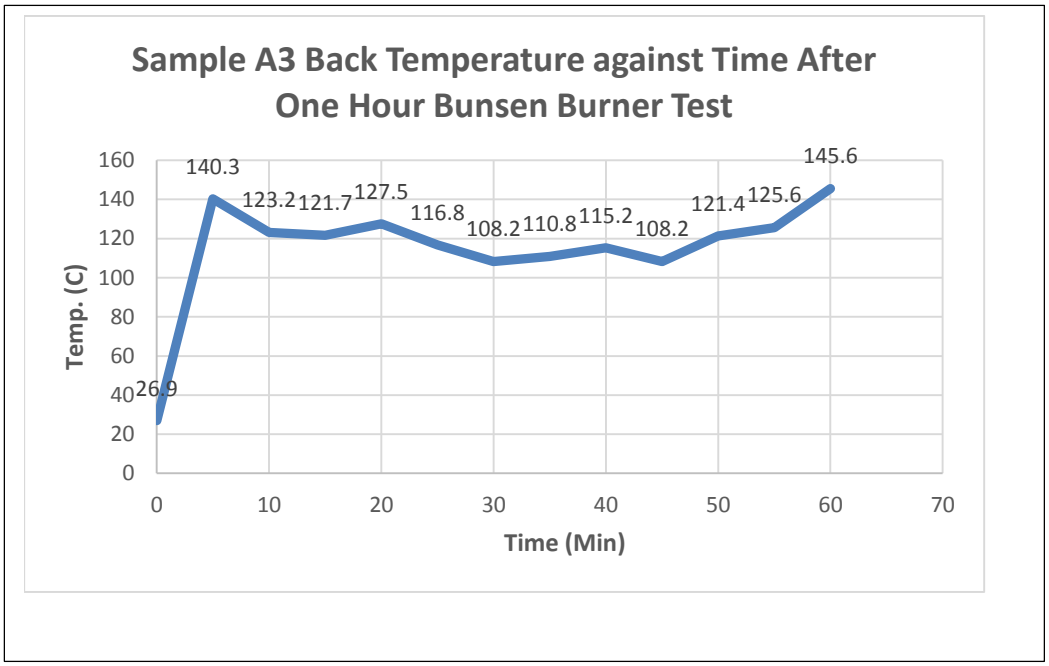
7) Front: Soft char was formed mostly at the region the fire strike the sample after one hour
: There is a no "crack" region s after more than half an hour of heating, only 'circle' of fibers seen

8) Back : Char was formed only at the region the fire strike the sample
: Other region remains as usual

**Table 6: Sample A3**

| Time (min) | Temperature Back (oC) |
|---|---|
| 0 | 26.9 |
| 5 | 140.3 |
| 10 | 123.2 |
| 15 | 121.7 |
| 20 | 127.5 |
| 25 | 116.8 |
| 30 | 108.2 |
| 35 | 110.8 |
| 40 | 115.2 |
| 45 | 108.2 |
| 50 | 121.4 |
| 55 | 125.6 |
| **60** | **145.6** |

Final Temperature




**Graph 4: Temperature versus Time for Sample A3**

## Sample A4

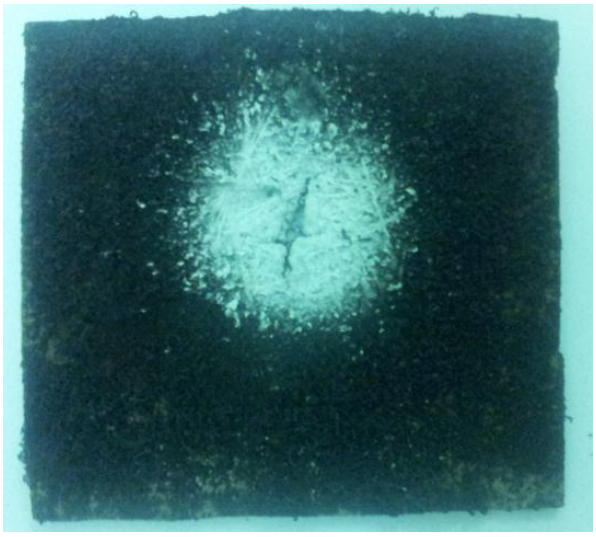


**Figure 33: After Fire Test (front)**

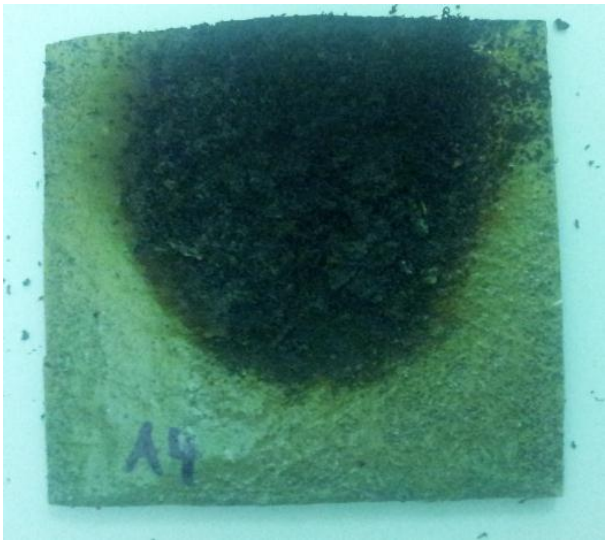



**Figure 34: After Fire Test (back)**

Observation for Sample A4:

9) Front: Soft char was formed mostly at the region the fire strike the sample after one hour
: There is a "crack" region somehow after more than half an hour of heating

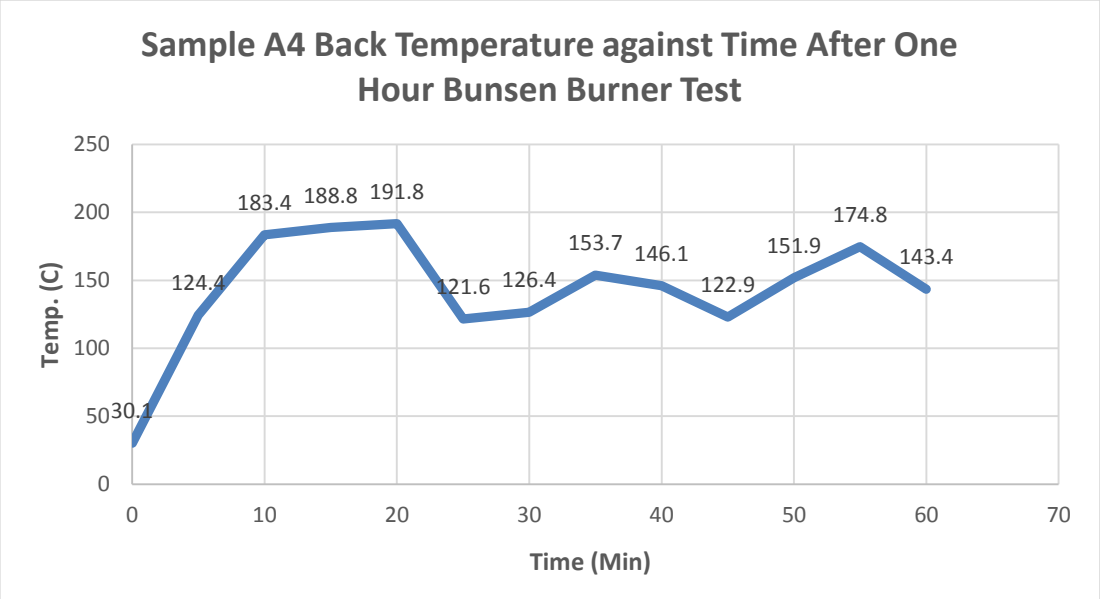
10) Back : Char was formed only at the region the fire strike the sample
: Other region remains as usual

- The sample does not melt or soften after one hour of fire testing.

**Table 6: Sample A4**

| Time (min) | Temperature Back (oC) |
|---|---|
| 0 | 30.1 |
| 5 | 124.4 |
| 10 | 183.4 |
| 15 | 188.8 |
| 20 | 191.8 |
| 25 | 121.6 |
| 30 | 126.4 |
| 35 | 153.7 |
| 40 | 146.1 |
| 45 | 122.9 |
| 50 | 151.9 |
| 55 | 174.8 |
| **60** | **143.4** |

Final Temperature




**Graph 5: Temperature versus Time for Sample A4**

**Sample A5**

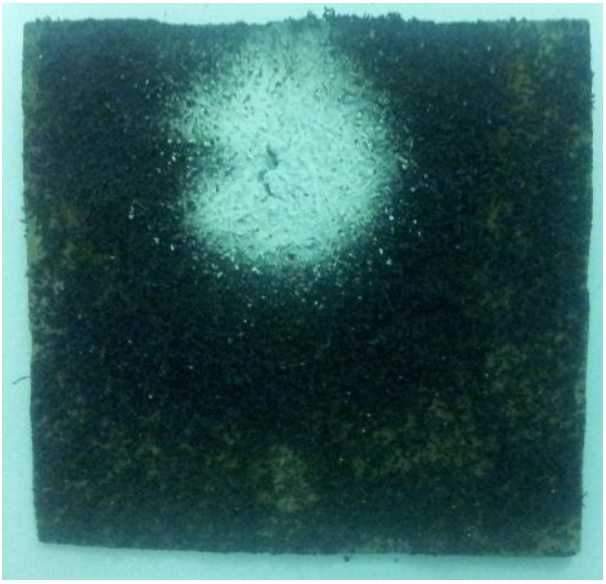


**Figure 34: After Fire Test (front)**

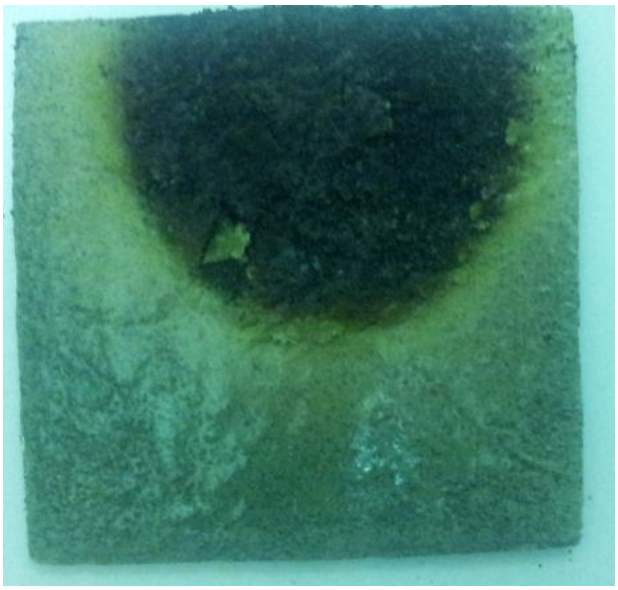


**Figure 35: After Fire Test (back)**

Observation for Sample A5:

11) Front: Soft char was formed mostly at the region the fire strike the sample after one hour
: There is a "crack" region somehow after more than half an hour of heating

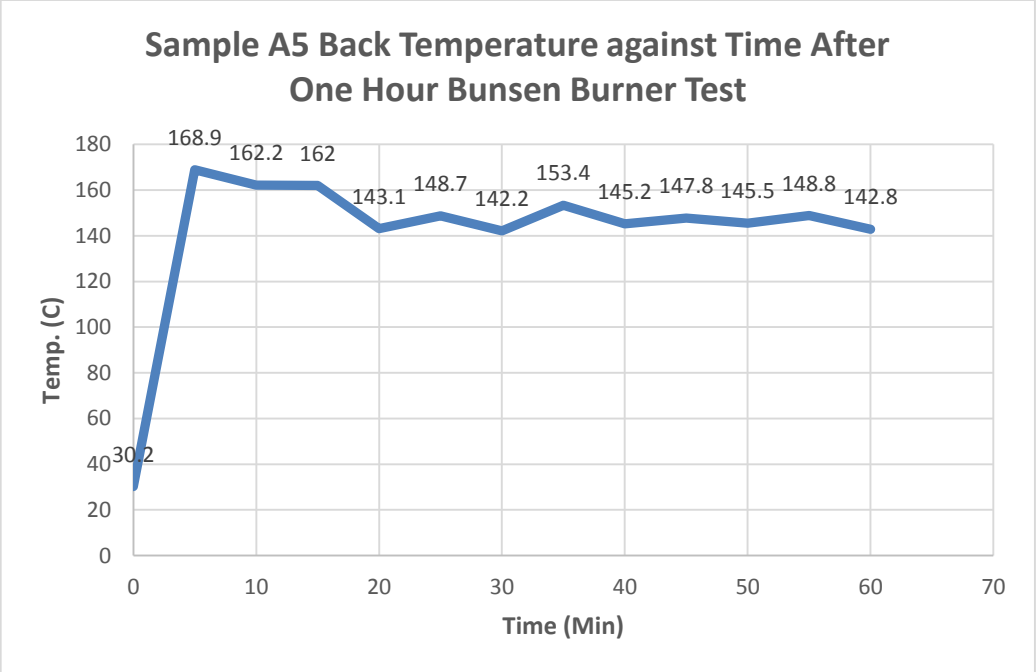
12) Back : Char was formed only at the region the fire strike the sample
: Other region remains as usual

- The sample does not melt or soften after one hour of fire testing.

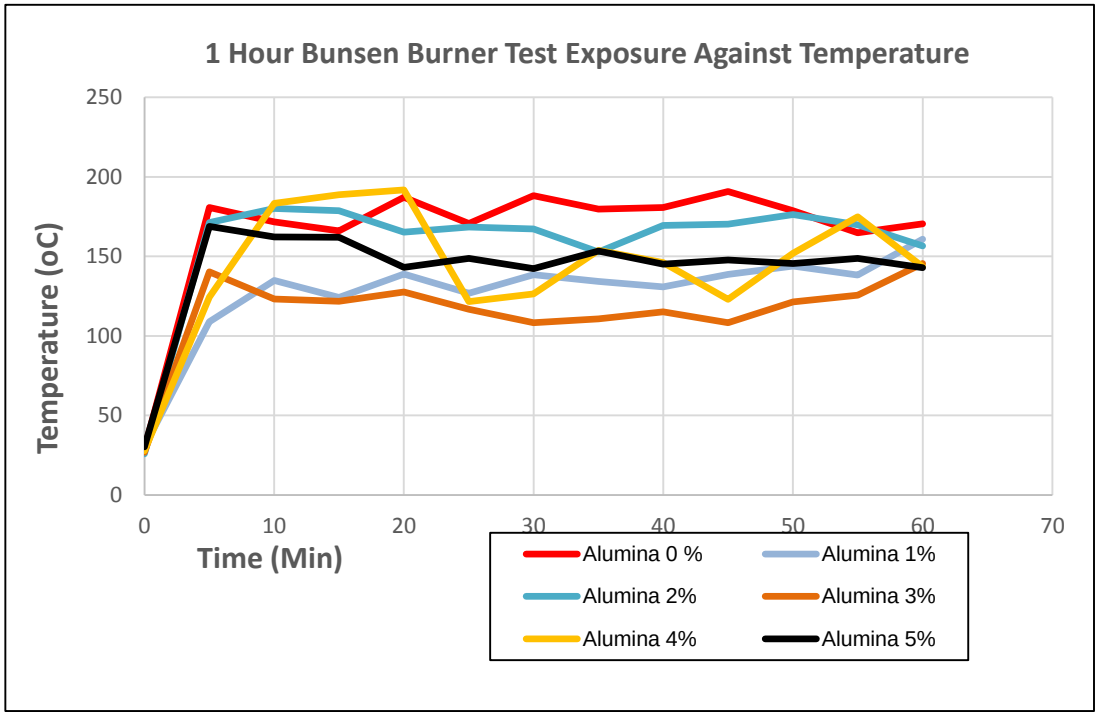
**Table 7: Sample A5**

| Time (min) | Temperature Back (oC) |
|---|---|
| 0 | 30.2 |
| 5 | 168.9 |
| 10 | 162.2 |
| 15 | 162 |
| 20 | 143.1 |
| 25 | 148.7 |
| 30 | 142.2 |
| 35 | 153.4 |
| 40 | 145.2 |
| 45 | 147.8 |
| 50 | 145.5 |
| 55 | 148.8 |
| **60** | **142.8** |

Final Temperature




**Graph 6: Temperature versus Time for Sample A5**




**Graph 7: Temperature against Time for sample A0-A5**

During the testing, the temperature profile of the glassfiber is measured using digital thermo logger (Figure 18) and thermo couple (Figure 19). Common fiberglass critical temperature is 800$^{o}$C before it lost the mechanical integrity [25].

Based on the above graphs and tables, it can be seen that sample A5 which contains alumina 5 wt% showed the lowest back temperature among all. Alumina of 5% in weight is the best formulation of all, as we are interested in determining the back temperature of the sample when exposed to fire after one hour. We can say that the presence of alumina as fillers enhance the fire retardant capabilities of the glass fiber. The higher the percentage of alumina as fillers, the better the fire retardant capability of the glass fiber However, the validity of the fire test are furthered proven by using Thermogravity analysis (TGA) technique which concerns on the final weight loss of the formulations. Another analysis that will be taking into considerations are X-Ray Diffraction Analysis (XRD) and Scanning Electron Microscope test (SEM).

Bunsen burner test performed on the fire retardant glass fiber samples have proven the fire-retardant characteristics of the samples as the samples did not melt or soften even if the temperature of the fire were recorded at almost 800-1000$^{O}$C. The back temperature of all samples are always lower than the front temperature. The only thing the author need to consider when constructing the samples using hand lay-up method is the edge of sample which can be seen sometimes catching the fire. The hypothesis accepted.

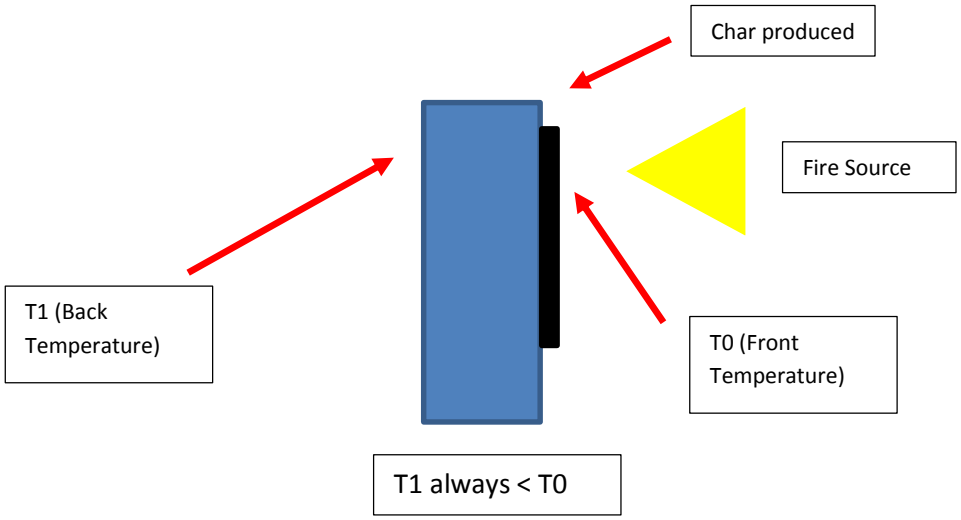


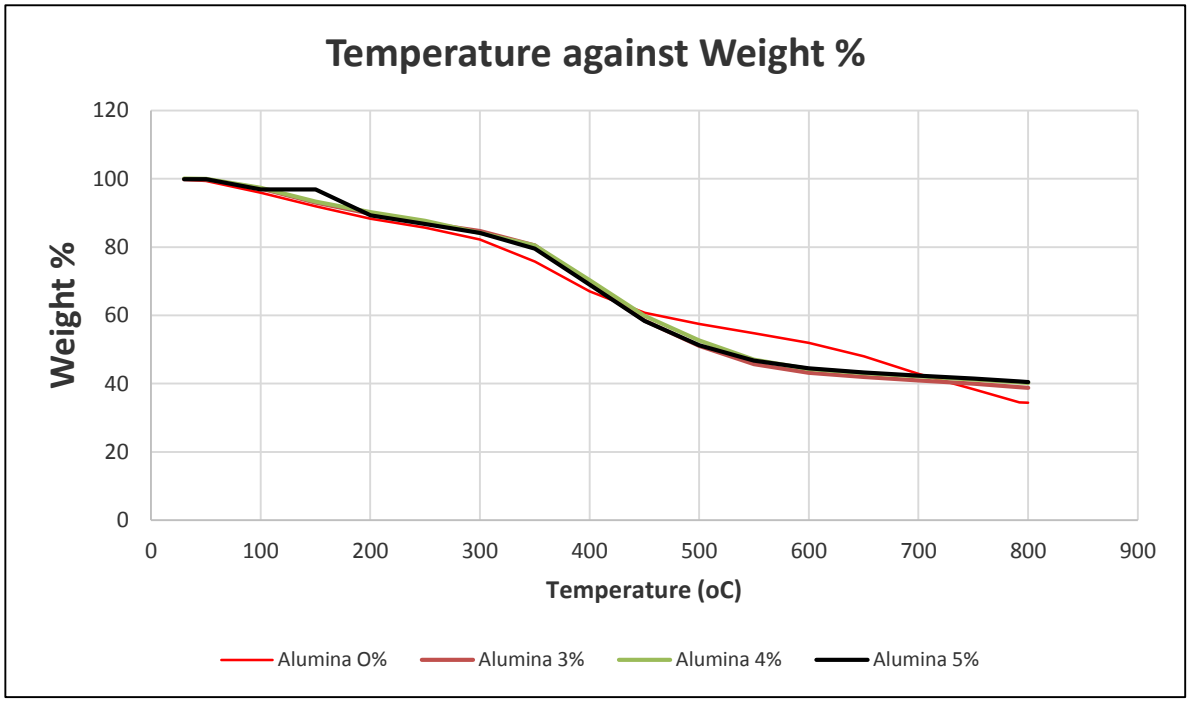

**Figure 36: Bunsen Burner Test schematic simulation**

## 4.3 Thermo gravity Analysis (TGA)

Thermogravimetric analyses (TGA) were carried out at 20$^{o}$C/min under air flow in the temperature range of 30–800 $^{o}$C using a TGA/SDTA851e model to investigate the thermal degradation of the samples. The parameter of interest of this test were the temperature against residual weight % of the samples. The samples tested under TGA were samples consisting of different percent of alumina as fillers.

| Temperature (°C) | Alumina O% | Alumina 3% | Alumina 4% | Alumina 5% |
|---|---|---|---|---|
| 30.1 | 99.76 | 99.934 | 100.072 | 99.854 |
| 50.31 | 99.385 | 99.766 | 99.975 | 99.854 |
| 100.25 | 95.918 | 97.154 | 97.387 | 96.932 |
| 150 | 91.944 | 93.013 | 93.365 | 96.932 |
| 200.31 | 88.307 | 89.741 | 90.26 | 89.345 |
| 250.08 | 85.703 | 87.022 | 87.731 | 86.739 |
| 300.05 | 82.195 | 84.718 | 84.188 | 84.178 |
| 350.14 | 75.803 | 80.505 | 80.5 | 79.578 |
| 400.22 | 67.03 | 69.243 | 70.283 | 68.978 |
| 450.22 | 60.715 | 58.495 | 59.846 | 58.344 |
| 500.22 | 57.464 | 51.038 | 52.668 | 51.211 |
| 550 | 54.754 | 45.756 | 47.06 | 46.721 |
| 600.03 | 51.977 | 43.223 | 44.338 | 44.452 |
| 650.24 | 48.051 | 41.941 | 43.046 | 43.27 |
| 700.23 | 42.857 | 40.961 | 42.138 | 42.331 |
| 750.23 | 38.334 | 39.971 | 41.247 | 41.453 |
| 792.07 | 34.534 | 38.998 | 40.335 | 40.622 |
| 800 | **34.424** | **38.796** | **40.147** | **40.468** |

**Table 8: Temperature against Weight % of different samples performed under TGA**




**Graph 8: Temperature versus Weight % for different samples performed under TGA**

Based on the table and graph above, four samples Intumescent Fire Retardant Coatings of different percentage of fillers were tested using TGA equipment in Block 17, Universiti Teknologi PETRONAS. The samples tested are consist of 0% Alumina, 3% Alumina, 4% Alumina and 5% Alumina. Before the test was carried out, the coatings must first be grinded to obtain the smallest particles it could be, especially in the form of powder.

The above table and graph show the results of the experiment after being heated up until 800 ºC. Based on the table and graph, it can be seen that the IFR coating consisting of the highest percentage of alumina (5%) has the lowest weight loss in percentage, which is 40,47% compared to others while the IFR coating without any percentage of alumina has the highest weight loss in percentage, which is 34.42%. The result shows that alumina as fillers enhanced the fire retardant capability of the IFR coatings. The percentage of weight loss is a very important parameter in protecting the sample when exposed to fire of high temperature (800-900ºC). When the samples of Fire Retardant Glass Fiber Reinforced Plastics are exposed to fire, the IFR coatings which impregnated with the glass fiber will form a 'char' layer which protects the samples. Thus, as we have a higher residual weight percentage, it produces a thick layer of protection which helps to reduce the heat transfer to the samples.

## 4.4 X-Ray Diffraction Analysis (XRD)

X-Ray Diffraction analysis (XRD) was done in Faculty of Science Physics, University Science Malaysia (USM) using XRD Equipment. This test was done outside from UTP due to the limitations of equipment in Universiti Teknologi PETRONAS. Due to the costs limitations for FYP, the author only managed to send two samples of residual char for this test, which is sample consisting 3% (A3) of 5% (A5) of alumina as fillers. The facial residue char of samples A3 and A5 tested at 800 $^{o}$C were analyzed by the XRD software.

### Sample A3

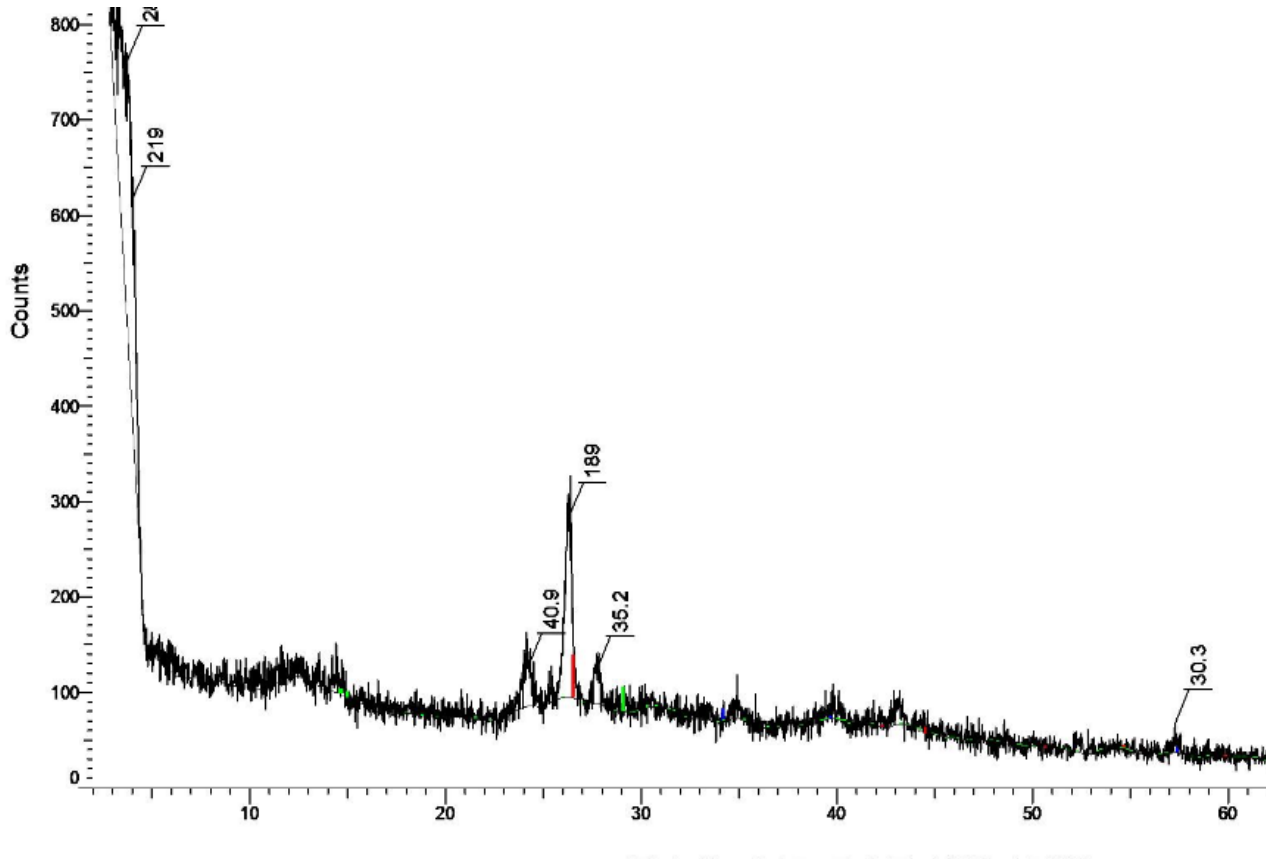
**Table 9: XRD Analysis for Sample of Alumina**

| Show | Icon | Color | Index | Name | Parent | Scan | Pattern # | Compound Name |
|---|---|---|---|---|---|---|---|---|
| Yes | | | 1 | COD 9011577 | Pattern List #8 | khairul01.raw #1 | COD 9011577 | Graphite |
| Yes | | | 2 | COD 9008835 | Pattern List #8 | khairul01.raw #1 | COD 9008835 | BP |
| Yes | | | 3 | COD 9007675 | Pattern List #8 | khairul01.raw #1 | COD 9007675 | Sassolite |

| Formula | Y-Scale | I/Ic DB | I/Ic User | S-Q | Added Reference | d x by | Scan WL | Wavelength |
|---|---|---|---|---|---|---|---|---|
| C | 15.38 % | 2.520 | | 46.69 % | | 1.0000 | Yes | 1.54060 |
| B P | 4.53 % | 3.940 | | 8.80 % | | 1.0000 | Yes | 1.54060 |
| B H3 O3 | 8.84 % | 1.520 | | 44.51 % | | 1.0000 | Yes | 1.54060 |

| System | Space Group | a | b | c | alpha | beta | gamma | Z | Volume | Density |
|---|---|---|---|---|---|---|---|---|---|---|
| Hexagonal | P 63/m m c (194) | 2.46400 | | 6.71100 | | | | | 35.29 | |
| Cubic | F -4 3 m (216) | 4.53800 | | | | | | | 93.45 | |
| Triclinic | P -1 (2) | 7.01870 | 7.03500 | 6.34720 | 92.490 | 101.460 | 119.760 | | 262.90 | |

| Cell Tuned | F (N) |
|---|---|
| No | |
| No | |
| No | |




**Graph 9: Peak graph for XRD Analysis for Sample A3**

## Sample A5

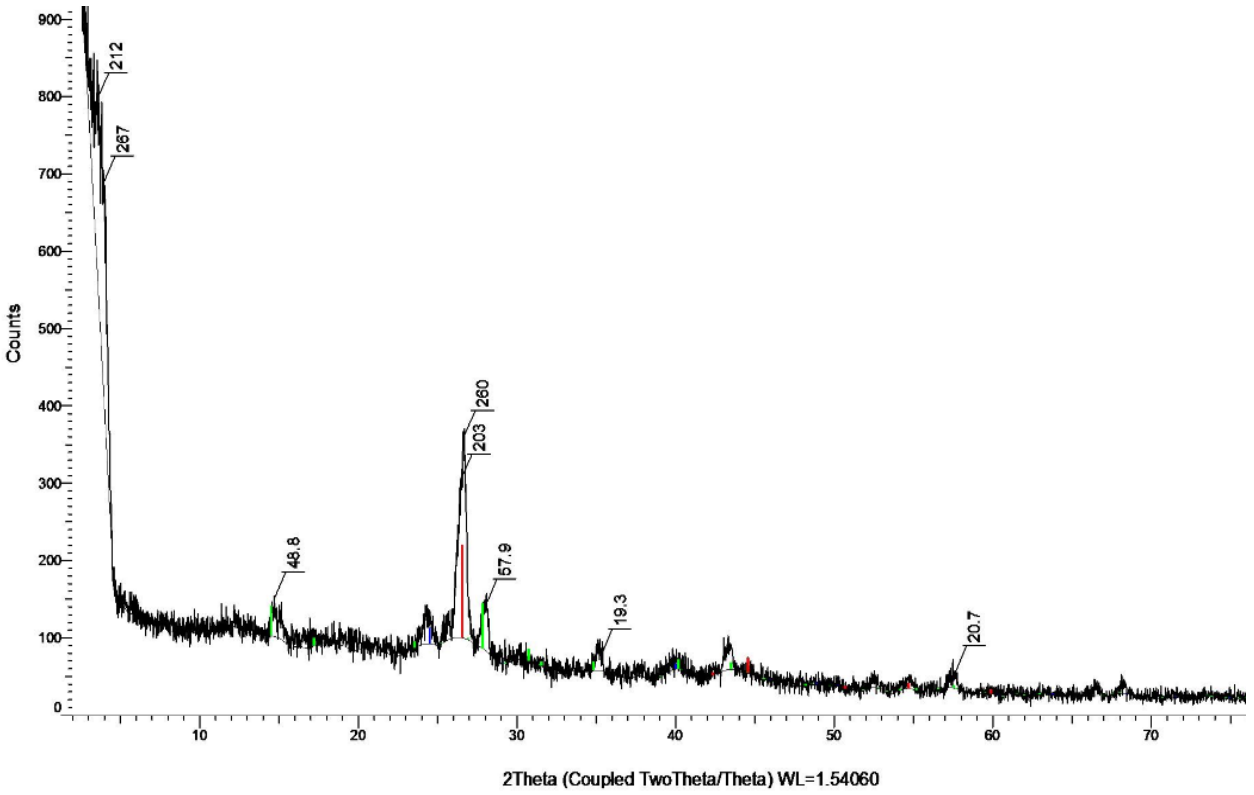
**Table 10: XRD Analysis for Sample with Alumina 5%**

| Show | Icon | Color | Index | Name | Parent | Scan | Pattern # |
|---|---|---|---|---|---|---|---|
| Yes | | | 1 | COD 9012230 | Pattern List #12 | khairul02.raw #1 | COD 9012230 |
| Yes | | | 2 | COD 1010299 | Pattern List #12 | khairul02.raw #1 | COD 1010299 |
| Yes | | | 3 | COD 2016172 | Pattern List #12 | khairul02.raw #1 | COD 2016172 |

| Compound Name | Formula | Y-Scale | I/Ic DB | I/Ic User | S-Q | Added Reference | d x by | Scan WL |
|---|---|---|---|---|---|---|---|---|
| Graphite | C | 36.78 % | 2.530 | | 51.43 % | | 1.0000 | Yes |
| | B O4 P | 6.29 % | 4.210 | | 5.29 % | | 1.0000 | Yes |
| | B O3 | 18.10 % | 1.480 | | 43.28 % | | 1.0000 | Yes |

| Wavelength | System | Space Group | a | b | c | alpha | beta | gamma | Z | Volume |
|---|---|---|---|---|---|---|---|---|---|---|
| 1.54060 | Hexagonal | P 63 m c (186) | 2.46100 | | 6.70800 | | | | | 35.18 |
| 1.54060 | Tetragonal | I -4 (82) | 4.33200 | | 6.64000 | | | | 2 | 124.61 |
| 1.54060 | Hexagonal | P 32 (145) | 7.04530 | | 9.60000 | | | | 6 | 412.67 |

| Density | Cell Tuned | F (N) |
|---|---|---|
| | No | |
| 2.758 | No | |
| | No | |




**Graph 10: Peak graph for XRD Analysis for Sample A5**

Table 9, 10 and Figure 36, 37 shows the XRD result of the residue char sample consisting alumina 3% and 5%. Several peak was assigned using XRD software. Those elements shown higher peak in XRD result and give indicator that, the elements are the dominant composition of intumescent coating during burning process. When the char layer continued to be oxidized at high temperatures, the carbon and inorganic materials originally present in the intumescent formulation barely remained in the carboneous char. However, the presence of inorganic materials in char is important, as they provide shielding in the later stages of fire when the temperature is higher than 600$^{o}$C.

For residual char sample consisting Alumina 3% (A3), the peak at blue color (3.940) assign to boron phosphate and the red peak (2.520) to graphite (carbon). The peak at green (1.520) assigned to sassolite. The reaction between APP and boron oxide yield some boron phosphate [27]. The sassolite (mineral acid of boric acid $H_3B0_3$) is due to the dehydration to support the formation of $B_20_3$, glass-like material which increase (Ahmad, Ullah, & Hamizol, 2012). Meanwhile, the S-Q scale shows that the percentage of graphite is 46.69%, boron phosphate 8.80%, and sassolite is 44.51% after the char produced. These compositions protect the sample and blocked heat from being transferred to the glass fiber.

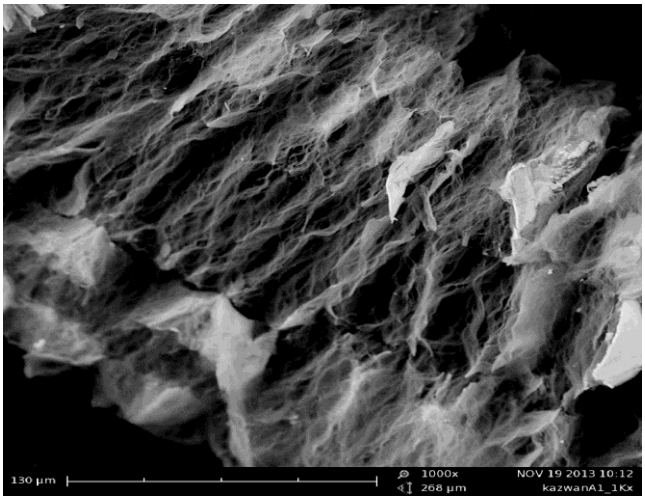
Meanwhile, for the sample consisting alumina 5% (A5), the peak at blue color (4.210) was assigned to boron phosphate and the red peak (2.520) was assigned to graphite (carbon). The peak at green color (1.450) again assigned to sassolite. The S-Q scale shows that the percentage of graphite is 51.43%, boron phosphate 5.29% and sassolite 43.28%. The compositions are slightly higher than sample A3 which proved that the higher the percentage of alumina presence, the better the 'char' residue in protecting the sample. The presence of these compounds in the residual char was considered helpful in minimizing heat flow to the substrate.

The XRD results showed that the reaction between boric acid and melamine, APP, and O2 enhanced the antioxidation performance of the intumescent coating by the formation of a protective char layer on the glass fiber samples. The XRD results also showed that boric acid in the char was converted into boron oxide and sassolite. In turn, the boron oxide reacted with APP to form borophosphate, which is a very stable compound up to 1200 C [26]. However, melamine or its derivate could not be detected from the XRD spectra; this could be explained as due to the reaction between melamine and APP to form melamine phosphate, which decomposed at temperatures above 350 C. Because the char was produced at 800$^{o}$C and above, the melamine probably decomposed completely.

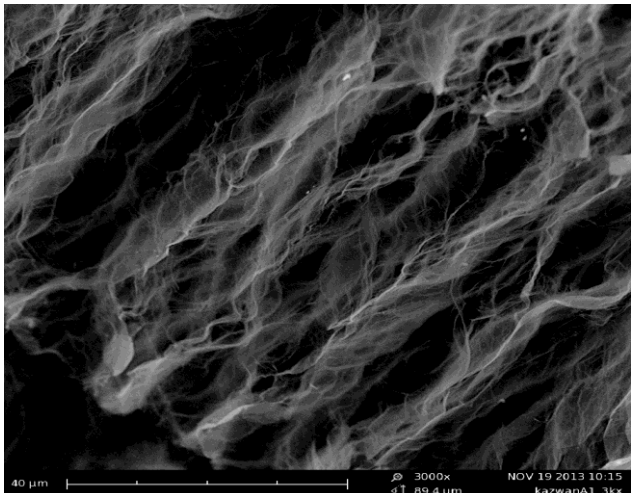
## 4.5 Scanning Electron Microscope (SEM) Analysis

### 4.5.1 Char Morphology

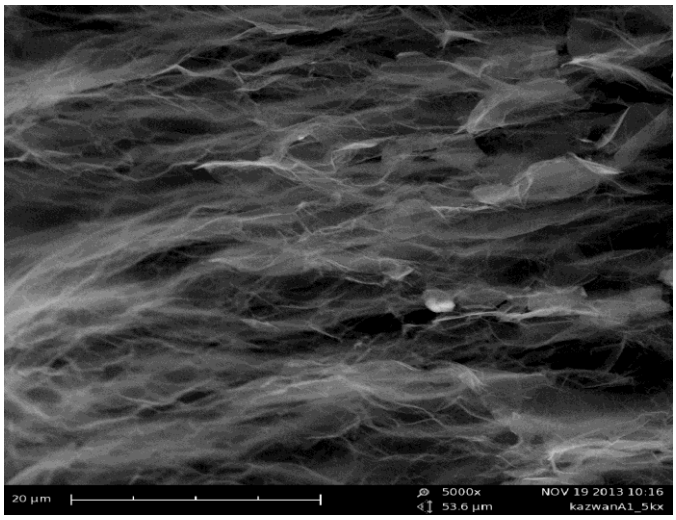
**Sample A0**




a)




b)




c)

**Figure 37(a-c) . Char Morphology of Intumescent Coating Formulations: A0 Outer Surface**

## Sample A3

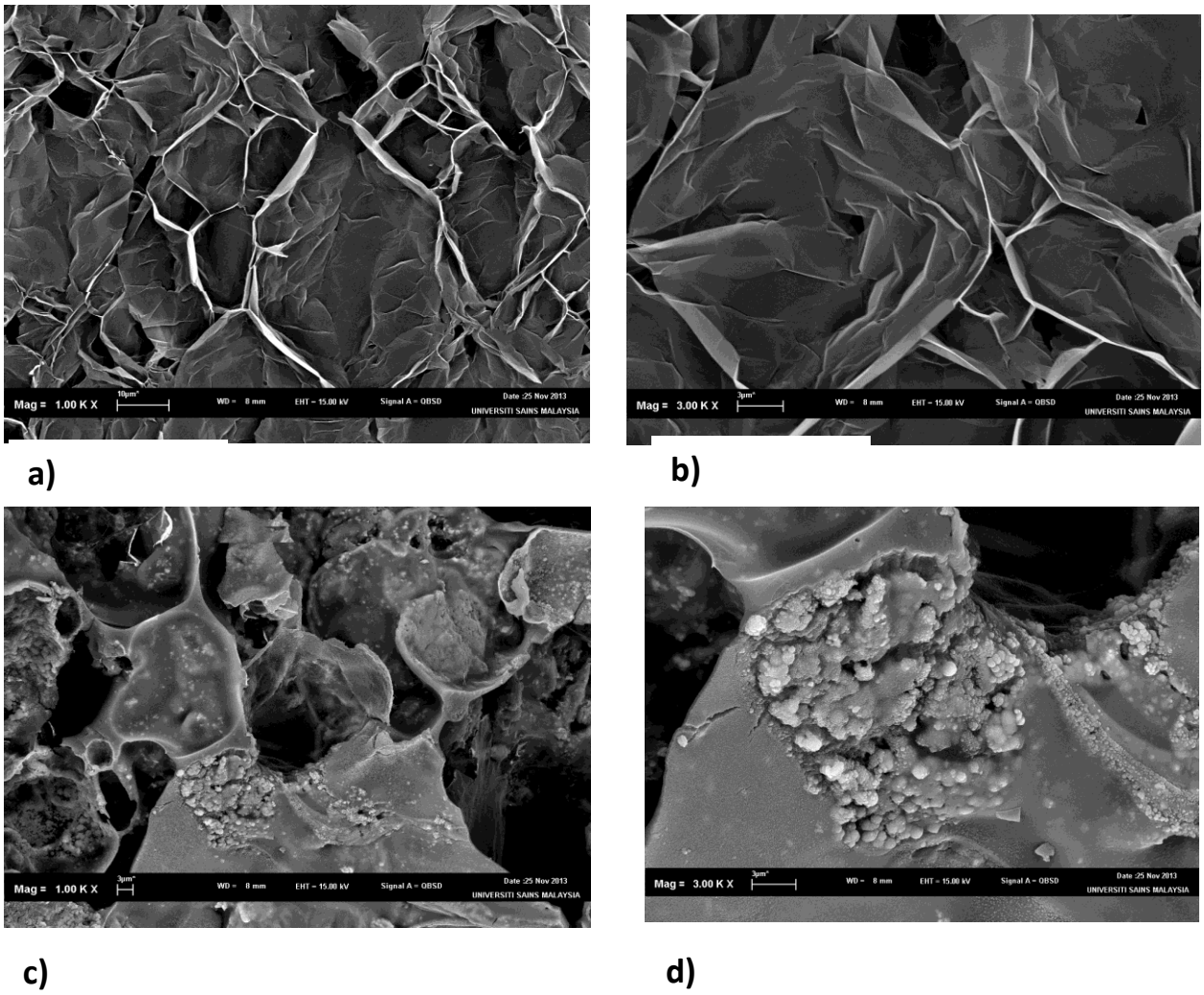



**Figure 38. Char Morphology of Intumescent Coating Formulations: a-b) A3 Outer Surface, c-d) A3 Inner Surface**

## Sample A5

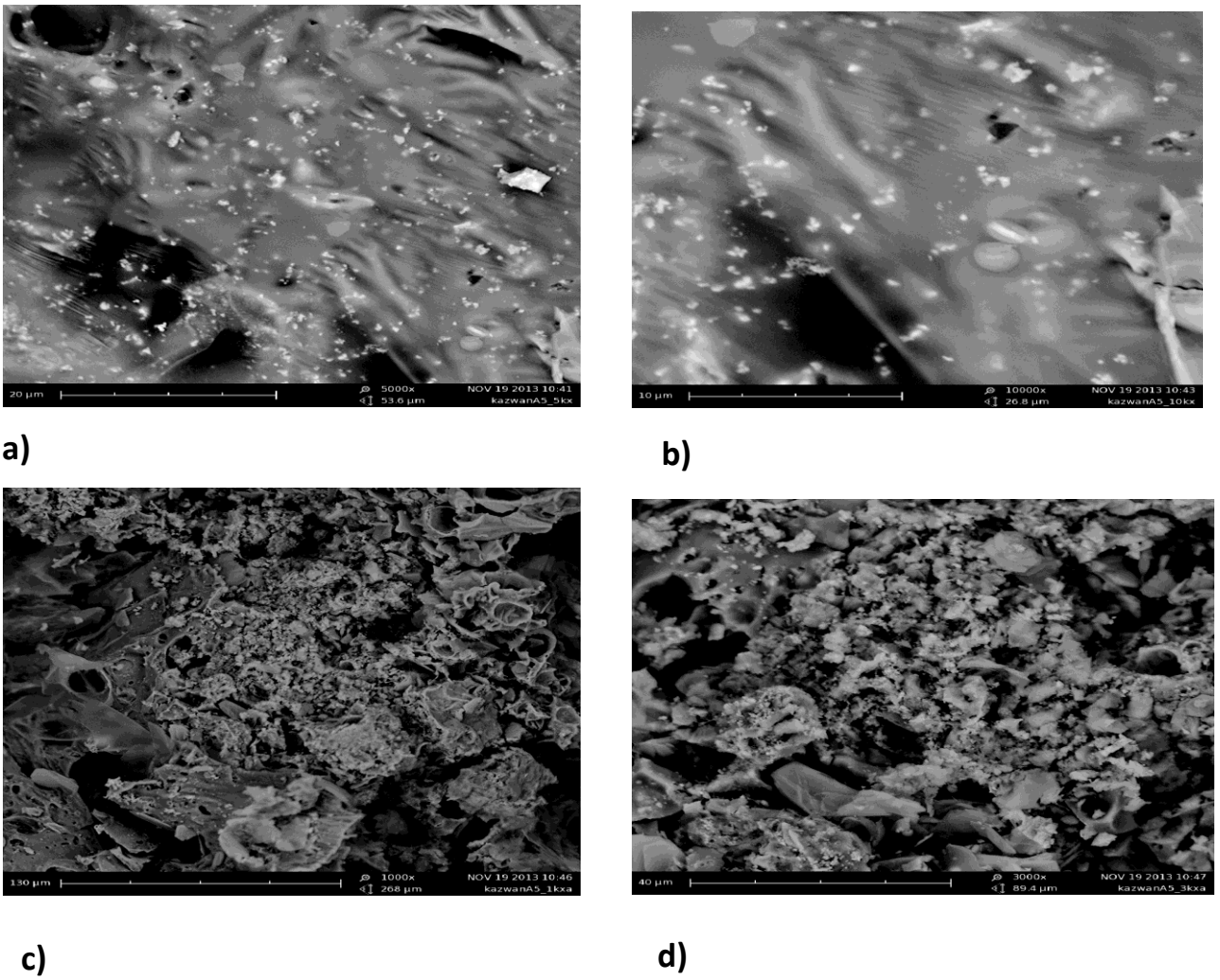



**Figure 39. Char Morphology of Intumescent Coating Formulations: a-b) A5 Outer Surface, c-d) A5 Inner Surface**

After fire test have been done, the samples are tested and observed for its morphology under Scanning Electron Microscope (SEM). As the equipment is limited in Universiti Teknologi PETRONAS campus, the author took an initiative to test the samples in Faculty of Science Physics, University Science Malaysia (USM). However, as the cost relocation for Final Year Project (FYP) is very limited, the author only managed to test 3 residual char samples under SEM; sample A0, A3, and A5.

The SEM micrograph of chars from the three coating formulations, A0, A3, and A5, are shown in Figure 38(a–c), Figure 39 (a-d), and Figure 40 (a-d). Figure 37(a-c) shows the outer surface of A0 char. From the figure, the outer surface was observed to have a smooth fibrous texture and with very thin and non-compact layer of char. The thin layers and tiny holes explained the results of the heat shielding effect, where the backside temperature increased to 175$^{o}$C after 60 min, which is the highest among all samples. Meanwhile, Figure 39 (a-b) shows the outer surface of A3 char. The surface observed to have more organized and compact fibrous structure. In Figure 39 (c-d), the inner surface morphology of A3 char shows a very thick surface, porous structure with holes which were produced as a result of the dehydration of the coating during fire testing [26]. The thick char layer reduced heat penetration in the fiber glass samples; the recorded backside temperature was 145.6 $^{o}$C, lower than sample A0 (without fillers).
Figure 40 (a-b) shows the outer surface morphology of A5 char. The fibrous structures are much more compact, with less holes observed. Meanwhile, Figure 40 (c-d) shows the morphology of inner surface structure of A5 char. With greater magnifications (3000x), it can be seen that the structures are thicker and more porous with only small holes observed compared to previous samples. Therefore, sample of A3 and A5 char morphologies proved that the existence of inorganic fillers hinder the heat transfer to the glass fiber samples with lower back temperature during fire test.

The multiporous arrangement of char can hinder heat relocation to the samples and insulate the samples from the adverse effects of heat. The relocation rate of heat through the charring layers depends on the expansion and structure of the residual char. The expansion and structure of the char are very important to common fire-retardant characteristics of coatings. (W.Farhana M, Faiz Ahmad, 2013).

# CHAPTER 5

# CONCLUSION

## 5.1 Conclusion

Samples of glass-fiber reinforced fire retardant composites have been successfully prepared using impregnation method of the Chopped Strand Mat E-Glass Fiber as the reinforcement with different formulations of Intumescent Fire Retardant Coating (IFR) as the plastic matrix. The manipulated variable in the formulations are the percentage of inorganic fillers that presence in the coatings. The sample later being tested to proof for fire retardant performance of intumescent coating by using portable Bunsen Burner. The char expansion and heat shielding effect shows the fire testing result.

From the result, it is shown that the coating which has been impregnated with the glass fiber protected the samples under the high temperature of fire (800-900 $^{o}$C). This can be seen as the samples of the fire retardant glass-fiber did not melt or burnt up on fire after one hour of fire test. The XRD and SEM result shows the presence of char element and morphology respectively in different samples of char. The author also proved that the presence of inorganic fillers, alumina in the highest weight percentage (5%) in the impregnated coating enhanced the fire retardant capability of the samples by having the lowest back temperature after one hour fire test, the highest residual weight percentage (%) after TGA test, the highest composition of elements in the char after XRD Test and the most compact with multiparous char morphology under SEM. Therefore, the existence of inorganic fillers like alumina in the coating formulations improved the fire retardant capability of the glass fiber samples with existence of protective layer called ‘char’ that insulates and hinder the heat from being transferred to the samples when exposed to fire.

## 5.2 Recommendations

It is recommended that in the future the samples of fire retardant glass-fiber reinforced plastics be analyzed further in terms of the mechanical properties of the samples, for example by performing tensile, hardness and impact test to see strength of the samples according to the American Society for Testing and Materials (ASTM). As we are concerning about the use of fire retardant fiberglass to reduce the use of steel in offshore platforms, these test are important parameters. From these te sts, we can investigate and study the effect of impregnation of intumescent fire retardant coating towards the hardness and strength of the fire retardant glass fiber samples.

Another important test that can be carry on is by performing fire test by using furnace. Furnace test is important as we can measure the thickness of the char after being exposed to heat provided by the furnace of 800-900 $^{o}$C in temperature. Thus, the results can be improved. The author was unfortunate because this test could not be done during the duration of this project as the furnace that is available in Universiti Teknologi PETRONAS is only for samples that do not produce smoke. The fire retardant glassfiber samples produced smoke and are not suitable to be tested using the available furnace. By performing furnace test, the thickness of the char produced can be measured and the results could be better. Further studies on the effect of different fillers used in the IFR Coating on the fire retardant capabilities of the fiberglass can also be done to get more validate and better results.

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