To Study the Effect of Waste Glass Powder and Zirconium Phosphate on the Properties of Intumescent Fire-Retardant Coating

by

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22712

Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Mechanical)

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Universiti Teknologi PETRONAS

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Perak Darul Ridzuan

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CERTIFICATION OF APPROVAL

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Approved by,

Faiz Ahmad

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Prof Dr Faiz Ahmad

UNIVERSITI TEKNOLOGI PETRONAS

BANDAR SERI ISKANDAR, PERAK

January 2020

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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.

Mohd Ameerul Haziq

MOHD AMEERUL HAZIQ BIN ABDULL HADI

ABSTRACT

Char is rather weak in mechanical strength, or if it cannot adhere to substrate, causing ineffectiveness of intumescent coating systems and make it unable to insulate a material. This study seeks to understand how adding waste glass powder and Zirconium Phosphate can strengthen the char in an epoxy-based intumescent fire-retardant coating and its effect on the based and fillers on thermal performance. The 5cm x 5cm coated samples was placed inside the furnace test machine to undergo furnace test, at 600°C by rate 20^{o}C/min for one hour to determine the physical properties of the char produced. The samples were then immersed in the seawater for 15 days to undergo immersion test to determine the effect of natural environment towards the thickness and the weight of the samples. Finally, the thermal performance of the coating was examined through a fire testing process wherein the coated samples of 10cm x 10cm were burned up to a high temperature and backside temperature of the substrates was recorded using a Data Logger.

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I would like to thank Prof Dr Faiz Ahmad, my project supervisor, for taking me into his supervision throughout the process of completing this project. My utmost appreciation and gratitude are also extended to post graduate student, Yasmin Azmi for her dedication in guiding me and providing all the necessary resources despite her many other obligations. Special thanks to my family for continuously giving moral support and for their sacrifices. Special gratitude for the lab technicians for their support in completing this project. My appreciation is also extended to everyone, especially my friends for their support from the beginning of the project.

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CHAPTER 1: INTRODUCTION

1.1 Background

The sector of infrastructure is one of the main important sectors for every country's economic growth. In Malaysia, the construction industry plays an important role to generate wealth for the country and develop social as well as economic infrastructures and buildings (Kamar & Hamid, 2011). There have been many incidents for years, causing fatalities, injuries and loss of properties due to fire. However, these kinds of incident could have been prevented, especially for the modern infrastructure. Either being used as structural material or will be enforeced with concrete, steel will always be used in constructing buildings. The carbon content and the process of heat treatment will decide the strength of the steel (quenching and tempering) (Mariappan, 2016). Fire can break rapidly to the unprotected structure since the structural steel can increase its temperature to 800 ^{o}C within 10 minutes or lower. The steel will start losing its strength and stiffness when exposed to the high temperature, as steel can only capability to withstand temperature up to 450–500 ^{o}C. Apart from that, the heat transfer and temperature profile will change because they were affected by the changes of thermal properties, such as value of specific heat, thermal expansion and thermal conductivity (Harper, 2007).

In the event of fire, time is such a vital element. With an addition of just 10 minutes of time, we can evacuate more people and prevent them from injuries and fatalities, save important documents and hazards can be prevented from be external surrounding. To protect substrates against fire, the best way is by using intumescent fire-retardant coating (Yan et al., 2017). Other than that, this coating can protect these structural steels when reaching high temperature, and to increase the time taken for the structure to be heated up. When heated, intumescent fire-retardant coatings will swell up as a char, that provides a physical barrier to protect the substrate against flame or heat. What contains in the regular intumescent polymer-based coatings include a carbon source (char former), an inorganic acid and a blowing agent (Alongi et al., 2015). In this case, the intumescence process is determined by the char formation (at the surface of substrate) and its foaming. The char layer represents a physical barrier

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reducing the heat transfer, therefore preventing the ignition and burning of substrate (Alongi et al., 2015). This paper explores the possibility to obtain a more efficient intumescent coatings, which is by adding waste glass powder and zirconium hydrogen phosphate. The study on the effect of the glass type will be very useful, especially on its reactivity at different temperatures through uniform composition, amorphous nature and the content of silica in glass (Mirzahosseini & Riding, 2014).

Every year, million tons of waste glass is generated. Waste glass will never decompose in the nature and will be disposed as landfills. In Malaysia, there is no recycling of glass products and this has led to almost 100 percent of glass ending up in dumpsters and, subsequently, in the landfills. Soda-lime glass is used to make window glass and glass bottle while lead-alkali-silicate glasses are used in manufacturing optical glassware and lightbulbs. In the manufacture of cooking and laboratory ware, borosilicate glasses were used since they have chemical resistance and high temperature softening point. Other than that, upon combination with oxygen, glass-formers have elements that can be changed into glass. So far, the most glass former is silicon dioxide (SiO2), usually used in the form of sand, consist about 70 percent of SiO2. In the early 1970s, there was a study conducted on the glass aggregate pavements and a conclusion came out stated that natural aggregates took lower time to cool down as compared to the glass aggregate due to the lower thermal conductivity of the glass (Siddique, 2008).

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1.2 Problem Statement

The current intumescent fire-retardant coatings mostly are lack of strength. This affects the efficiency of coating in protecting the base structure when exposed to high temperature. At high temperature, intumescent coating expands and forms a char layer. A char layer is a poor conductor which prevent heat transfer, thus avoid damage to a protected material. However, char layer can be depleted by physical erosion and chemical processes such as oxidation, thus its protection capability is reduced. The depletion causes it to crumble and without enough strength, a char layer will easily be falling off and fail to protect a material. The weakness of current coating strength has led to recent interest in developing a better coating formulation that improves the strength.

In this paper, waste glass powder was used as filler to improve the existing traditional intumescent fire-retardant coating. Due to the poor thermal properties of intumescent fire-retardant coating, the coating oxidizes at high temperature and loses their fire protection properties (Wang et al., 2007). For this paper, the waste glass powder comes from waste glass bottle. So, the composition of waste glass powder was discussed, and how it can help to improve the existing traditional intumescent fire-retardant coating. It can be concluded that intumescent silicone-based coatings are the best material for protecting carbon fiber reinforced polymer in the case of jet fuel fire (Bourbigot et al., 2014).

Since waste glass powder was used in this intumescent coating, environmentally friendly version of intumescent coating was produced since glass will never decompose when it becomes a waste (Islam et al., 2017). Glass is a non-biodegradable material. Finely grinded glass has been used as a pozzolanic material because the main component of the glass is amorphous silica (Carsana et al., 2014). Most of the previous studies used waste glass powder in making concrete to improve their thermal properties. So, one of the purposes of this project is to implement the usage of waste glass powder into fire intumescent coating to improve its performance.

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1.3 Objective and Scope of Study

1.3.1 Objective

i. To develop the intumescent fire-retardant coating by using waste glass powder and Zirconium Phosphate, both as fillers.
ii. To study the composition of waste glass powder on thermal performance.
iii. To investigate the effect of composition of waste glass powder on thermal performance.

1.3.2 Scope of Study

The scope of study on this project is mainly focusing on using waste glass powder mainly from waste glass bottle, which acts as a filler, and was mixed into the intumescent fire-retardant coating. Zirconium phosphate was then added to the fire-retardant coating, which also acts as filler. The coating then coated on carbon steel plate structure, with thickness of 1.5mm, before testing. Each of the samples was undergo immersion test to observe any changes on the coating when exposed to natural environment. The char produced from the furnace test was analysed and intumescent factor was calculated to observe the expansion of the char. Fire performance test was then has been conducted to determine the best formulation of intumescent fire-retardant coating that gives the best fire protection towards the structure.

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CHAPTER 2: LITERATURE REVIEW

2.1 Method of Fire Protection

There are two methods of fire protection, which are passive fire protection and active fire protection. Both are used to protect the steel structures to collapse upon exposure towards heat from the fire. In the event of fire, active fire protection needs some motion or action to works efficiently. The purpose of a fire protection system may be to extinguish and control the spread of fire or provide protection against domino effects. Taking an instance, fixed water monitors or foam pourers are better choices to deliver protection than sprinklers or sprays. Active fire protection system can either be manual, like a fire extinguisher or automatic, such as a sprinkler system. Apart from that, active fire protection systems consist of smoke detectors, fire alarms and even firefighters.

On the contrary, the basic component of structural fire protection of a building is the passive fire protection. With the same function as active fire protection, both of them are used to stop fires, which allow people to evacuate in short period of time and minimizing the damage of important documents of equipment. Passive fire protection can prevent the spread of fire using fire-resistance floors and walls by breaking the building into “compartments”. To help with the compartmentalization of the structures and dampers, the fire doors will also be utilized to prevent the spread of fire and smoke.

Passive fire protection includes fire/smoke dampers, fire doors, and fire walls/floors. Dampers are used to avert the spread of fire/smoke by its ductwork while fire doors help to sectionalize a building. To prevent the exposure of adjacent equipment to thermal radiation and the spread of fire, fire walls are another form of passive fire protection that are used. It is crucial to consider the duration of the fire exposure to decide which system is most suitable for fire protection because the effectiveness of passive fire protection is only for a short duration exposure.

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2.2 Intumescent Fire Coating

Intumescent coatings are also known as intumescent paint and it is used in buildings as a passive fire resistant. They are basically deemed as a convenient fireproofing product. Intumescent paints are formulated specially as a reactive coating that swell or intumesce into thick foamed char whenever exposed to heat. It is to protect substrates against fire by insulation and becoming barrier against heat and mass transfer (Mariappan, 2016). 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. Therefore, it works by protecting the substrate from getting direct exposure to the flame which can damage the mechanical properties of the substrate (Shahbudin, 2013). Figure 1 below shows a simple model of intumescent fire coating system.

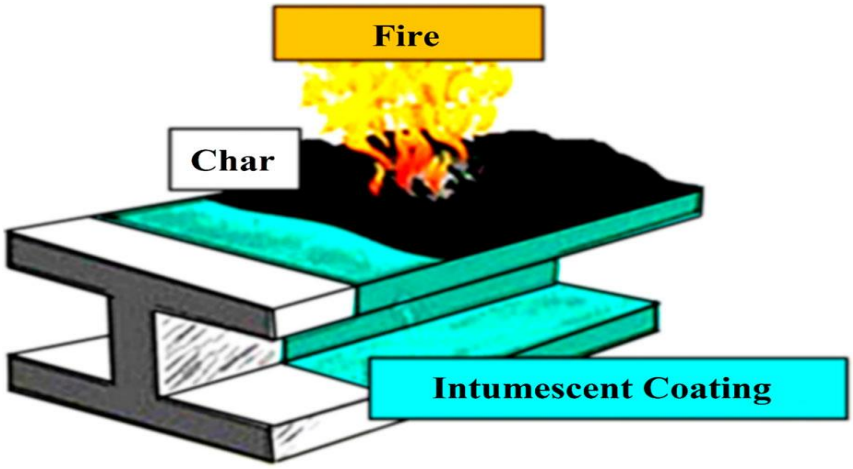

Figure 1: Steel coated with Intumescent Coating (Yasir et al., 2019)

Intumescent coating usually consists of acid, blowing agent and a carbon source. Carbon source is the material that produced char in the intumescent system (Othman, 2016). These three sources are linked together with a binder. When the source is exposed to thermal behaviour, the blowing agent will cause the carbon source to solidify into a form of cellular foam (Othman, 2016). The thickness of these coatings range between 5 and 25 mm, depending on the required fire resistance, the steel section type and section factor (the ratio of heated surface area to volume of steel) and the limiting temperature adopted in design, typically based on design code requirements. Intumescent coatings are generally categorized as thin film (dry film thickness of 1–3 mm) and thick film/mastic (3–30 mm) coating (Mariappan, 2016). When getting caught in a fire, the coating retains its expanded or foamed structure at high

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temperatures to provide a heat isolative layer which protects the substrate for a longer period, approximately up to 3 hours to 4 hours (Hao & Chow, 2003).

2.3 Char Formation

The charred layer formed will act as physical barrier for the substrate that are protected by intumescent systems, which slow down the mass and heat transfer between gas and condensed phase. The char yield is known as the yield of residue that is obtained by heating the char forming additives, either alone or in mixture with APP at 400°C in air, to constant weight loss (Bertelli et al., 1991). When heating large amounts of thermally stable carbonaceous residue, intumescent systems will be decomposed and formed. This intumesced char can increase up to 50 times the original thickness of the applied coating and it is used to insulate the steel, thus giving additional time before the steel reaches a critical temperature (550^{o}C) at which it loses load bearing capacity (Mariappan, 2016). The main purpose of intumescent materials is the protection against temperature, whereby the intumescent shield will reduce the amount of heat transfer. Deep understanding of the expansion of char and its mechanisms are important and fire protective capabilities depend on the swelling.

In the intumescent behaviour, the heat transfer and temperature gradients are very important. To make the intumescent flame-retardant efficient, a well-selected component is vital. Char formers, carbonising, dehydrating substances and modifiers lead to obtaining a maximum degree of carbonization and thus bringing about the efficiency of the protective char (Yew, 2011). Furthermore, it is very important to select proper binder resins. Binders are either polymers or resins that bind together the ingredients of a coating since they control the main degradation step of the coating when subjected to high temperatures, which means they control the char formation temperature (Yasir et al., 2019). During the degradation stage, char is formed, and it contained chain of carbon along the way to graphitization. Fire resistance of intumescent fire-retardant coating leans on the char formation proposed by the reaction of APP, pentaerythritol (PER) and melamine (Othman, 2016).

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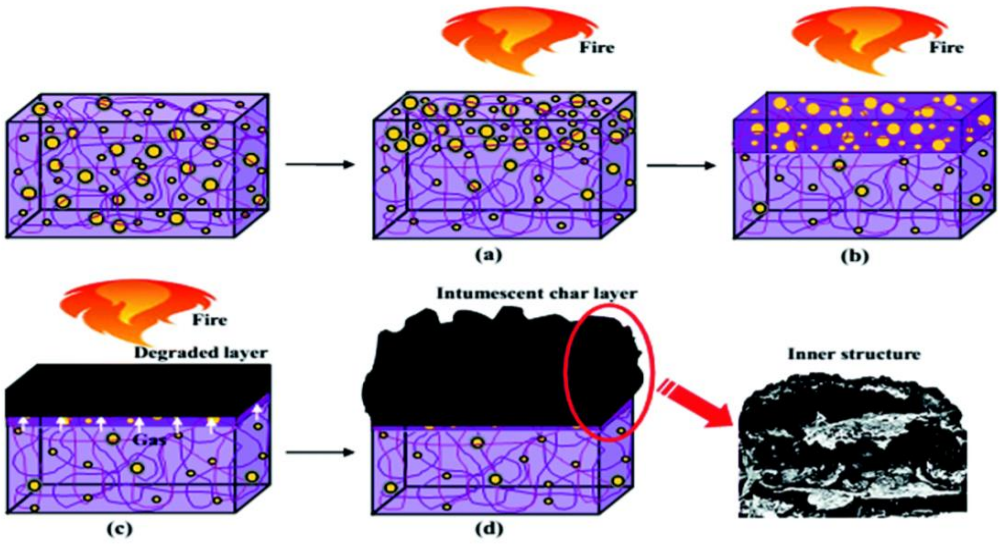

Figure 2: Ideal Char Formation (Othman, 2016)

The mechanism of intumescent usually starts with the acid source breaking 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 (Shahbudin, 2013). The char will then swell and insulating multi-cellular protective layer will be provided. 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 (Jimenez et al., 2006).

2.4 Fillers in Fire Intumescent Coating

Fillers is defined as a substance or material added to compounds to decrease usage of expensive binder and to improve the mechanical properties of the compounds. In this system, adding a certain amount of filler can enhance the flame retardant. Besides that, addition of fillers will decrease the amount of smoke produced during heating process and reducing the time to retard flame (Othman, 2016). Inert filler basically reduces the flammability and smoke by diluting the combustible substrate and absorb heat to decrease the combustibility reaction occurring onto substrate produced by a few mechanisms. Typical inert filler used in intumescent systems are silica, calcium carbonate, pumice, talc, calcium sulphate.

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2.4.1 Waste Glass Powder

Since glass waste occupies a major part of the wastes and by-products, it is a serious problem towards the environment. According on the research, the usage of waste glass powder in intumescent coating is very rare to find. So, in this project, waste glass powder was used as a filler. It is reported that the usage of glass powder reduces the $Ca(OH)_2$ amount and the thermal conductivity (Pan et al., 2017). Furthermore, the utilization of the glass powder can help reducing the spalling and degradation of the high-strength concretes when exposed to high temperatures (Ali et al., 2017).

2.4.2 Zirconium Phosphate (ZrP)

Zirconium phosphates (zirconium hydrogen phosphate) are known to be acidic, and it is an inorganic cation exchange material of a layered structure (Clearfield & Stynes, 1964). Zirconium phosphates have the character of high thermal and chemical stability and solid-state ion conductivity. It is also known for its resistance to ionizing radiation, and the capacity to fuse different types of molecules of different sizes between their layers. Furthermore, zirconium phosphate was also been used in several applications such as catalysis, nanocomposite and clinical dialyze.

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CHAPTER 3: METHODOLOGY

This chapter will briefly discuss the details of the laboratory equipment which will be used to test and conduct this project as well as the experimental set-up. Other than that, the planning chart, flow chart, Gantt chart and key milestone of this study will also be presented in this chapter.

3.1 Materials Used

No	Materials	Function	Sources
1	Ammonium Phosphate (APP)	Inorganic salt that contain polyphosphoric acid and ammonia chains. The physical property is colourless and incombustible. In the intumescent system, the role of APP as an acid source which then reacts with the carbon source to form ester.	Clariant (Malaysia) Sdn Bhd
2	Melamine (MEL)	An organic base compound that contain abundant of nitrogen molecule. It will degrade to commute gaseous such as ammonia gas and nitrogen gas when scorched. Thus, it was used as a gas source in intumescent coating system.	Sigma-Aldrich (M) Sdn Bhd, Malaysia
3	Boric Acid (BA)	Boric acid is an occurring material containing boron, oxygen and hydrogen. Its crystalline structure is white and inodorous. Boric acid can impede the movement of flammable gaseous and able to release chemically bonded water which can reduce the combustion rate in coating system.	Sigma-Aldrich (M) Sdn Bhd, Malaysia
4	Expandable Graphite (EG)	Expandable graphite is a composition of graphite that gets enlarged when exposed to flame. The thickness of	Sigma-Aldrich (M) Sdn Bhd, Malaysia

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		graphite is 100 times larger of its original thickness retaining the superior heat resistance properties of graphite. These properties are vital in increasing the efficiency of the intumescent coating system. The role of EG acts as both blowing agent and carbon source in the intumescent coating formulation.	
5	Epoxy Resin and Hardener	For this project, epoxy resin Bisphenol A BE-188 (DGEBA) is used as binding agent and TETA polyamide amine as curing agent. The binder can bind the additives and give out adhesiveness to the coated substrate.	Mc-Growth Chemical Sdn Bhd, Malaysia
6	Zirconium Phosphate (ZrP)	Zirconium Phosphate (ZrP) was used as filler in this experiment. Other than that, it can act as flame retardancy as ZrP is applicable in high temperature environments	China
7	Waste Glass Powder	Waste glass powder was used as filler in this experiment. Based on previous study, glass powder can improve the thermal properties.	Glass & Plastic Packaging (Ipoh) Sdn Bhd, Malaysia

Table 1: List of materials used

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3.2 Equipment and Tools Used

1. Ball Mill Machine

Figure 3: Ball mill machine

Ball mill machine was used to break the waste glass bottle into smaller pieces before it will be taken into the grinding machine. This process took up to 4 hours.

2. Grinding Machine (Tencan GJ-2)

Figure 4: Inside the grinding machine

Figure 5: Grinding machine

This grinding machine was used to grind the smaller pieces of waste glass bottle after the ball mill process. This process took 2 minutes to grind.

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3. Mixer Machine (Caframo BDC6015-220)

Figure 6: Mixer

This mixture machine was used to mix the colourless mixture with EG and Epoxy. This mixture machine was set at 40rpm for its rotational speed and this mixing process was carried out for 25 minutes.

4. Furnace test machine (Carbolite Gero)

Figure 7: Furnace test machine

The char expansion was tested, when expose to high temperature, by using furnace test machine. Better fire resistance performance was produced when the expansion is higher.

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5. Sieving Machine (Endecotts EFL2000)

Figure 8: Sieving machine

The grinded waste glass bottle then was sieved by using this sieving machine. The waste glass powder was sieved and retained at 212µm sieve.

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3.3 Project Activity

3.3.1 Producing Waste Glass Powder

i. The waste glass was break into small pieces so it can be easily to undergo ball mill process to make it smaller pieces.
ii. Then, it was undergoing ball mill process for 3 hours using ball mill machine.
iii. The smaller pieces of the waste glass were then undergoing grinding process using the grinding machine to get powder. The process took 2 minutes.
iv. The waste glass powder then was sieved using sieving machine and retained at 212µm sieve.

Figure 9: Process to produce waste glass powder

3.3.2 Experimental Formulation

The formulation used in the experiment is different to be applied onto substrate. The overall weight percentage is 100% for all samples but different weight percentage is used for the usage of hardener and epoxy resin. The difference is varying with the weight percentage of waste glass powder and Zirconium Phosphate used in the formulation. Tables below show all the samples' formulation:

Component (wt. %)	APP	MEL	BA	EG	Epoxy Resin	Hardener
Control Formulation (CF)	11.23	5.5	11.11	5.5	44.44	22.22

Table 2: Control formulation

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Component (wt. %)	S001	S002	S003	S004	S005
APP	11.23	11.23	11.23	11.23	11.23
MEL	5.5	5.5	5.5	5.5	5.5
BA	11.11	11.11	11.11	11.11	11.11
EG	5.5	5.5	5.5	5.5	5.5
Epoxy Resin (BPA)	43.77	21.55	42.44	41.77	41.11
Hardener (PA)	21.89	21.55	21.22	20.89	20.55
Filler					
Waste Glass Powder	1	2	3	4	5

Table 3: First formulation

Component (wt. %)	ZS091	ZS092	ZS093	ZS094	ZS095
APP	11.23	11.23	11.23	11.23	11.23
MEL	5.5	5.5	5.5	5.5	5.5
BA	11.11	11.11	11.11	11.11	11.11
EG	5.5	5.5	5.5	5.5	5.5
Epoxy Resin (BPA)	43.17	42.51	41.84	41.17	40.51
Hardener (PA)	21.59	21.25	20.92	20.59	20.25
Fillers					
Waste Glass Powder	1	2	3	4	5
Zirconium Phosphate	0.9	0.9	0.9	0.9	0.9

Table 4: Second formulation

3.3.3 Coating Application

The control basic formulation will be prepared as reference. The control formulation based on formulation provided by author's supervisor. The coating is prepared following procedure:

i. Mix all formulation and grind for 1 minute using mixer grinder except for expandable graphite (EG).
ii. Mix the grinded mixture with epoxy using mixer with at 30 rpm for 15 minutes, the hardener will be added.
iii. Continue mixing with the same rpm for 10 minutes before increasing the speed to 60 rpm for another 5 minutes.

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iv. Rest the mixture for 5 minutes before spreading it to the two substrate of 5 cm x 5 cm sample and on one substrate of 10 cm x 10 cm sample. Ensure the steel is covered with tape before the experiment.

3.3.4 Fire Test

To determine the thermal performance of the intumescent coating, fire test need to be carried out onto the 10cm x 10cm samples. During this test, the temperature of the backside of each sample were recorded. For each of the samples, the backside temperature for each of the specimen has been recorded every 30 minutes interval for one hour during the fire test. Thermocouples have been attached at the backside of the specimen during the test and the temperature was recorded using Thermo Logger

Figure 10: Fire performance test setup

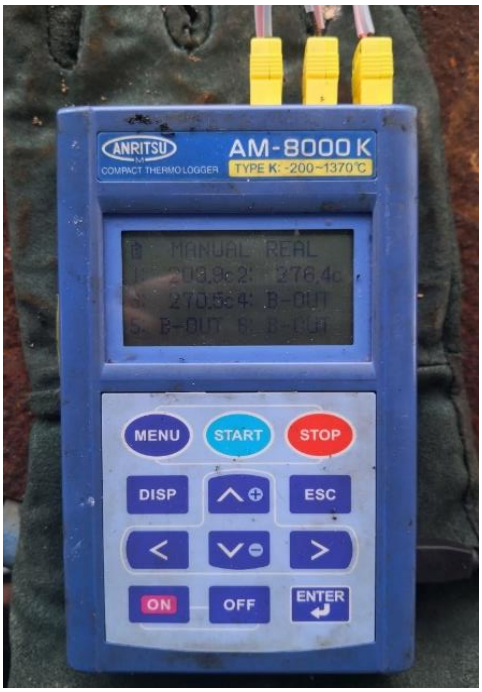

Figure 11: Thermo Logger

i. The experiment was set up as shown in figure above.
ii. Thermocouples were attached at the backside of the sample. The thermocouples needed to touch the backside of the sample to get accurate results.
iii. The distance between the source of fire and the sample was set to be 7cm.
iv. The temperature was recorded every 30 seconds for one hour.

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3.3.5 Furnace Test

This test was carried out to observe the char expansion and the characterization of the char of each of the 5cm x 5cm sample when they were exposed to high temperature. This test was carried out inside the furnace test machine.

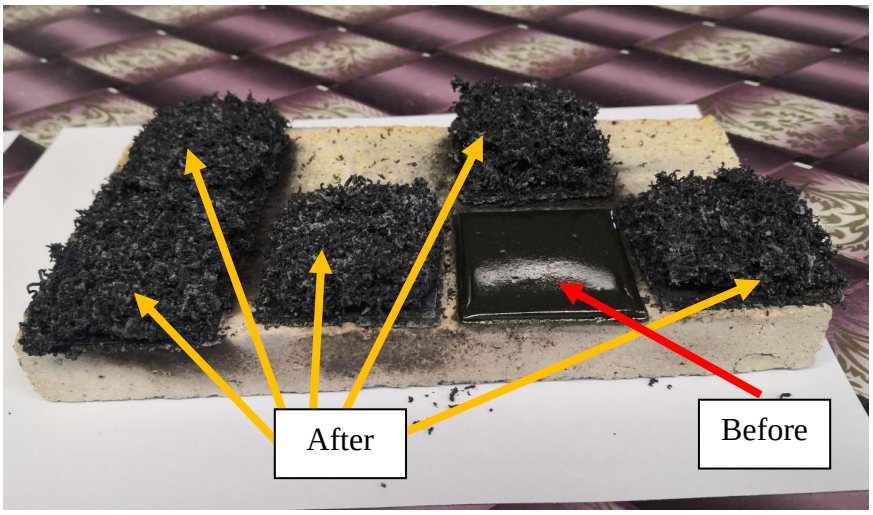

Figure 12: Samples before and after undergo furnace test

i. The samples are put into the furnace and the cover is closed.

ii. The temperature is raised to 600°C by rate 20°C/min and the temperature inside the furnace maintain for one hour.

iii. The sample is the cool down to room temperature without removing it from the furnace and the expansion of each sample is then taken.

iv. The sample thickness before and after the test will be recorded and an intumescent factor (IF) will be calculated from the thickness obtain. The formulation involved as followed:

$$\text{Intumescent Factor (IF)} = \frac{(d2-d0)}{(d1-d0)}$$

Equation 1: Intumescent factor (IF)

Where:

d_2 = expanded char thickness

d_1 = coating thickness

d_0 = substrate thickness

`

3.3.6 Immersion Test

For immersion test, 5cm x 5cm samples has been immersed into seawater. The changes of the samples have been recorded for every 3 days for 15 days. Then, the samples were let dry for 24 hours before the last measurement have been recorded. The purpose of this test is to check the changes of each sample when exposed to natural environment, which is seawater.

Figure 13: Immersion test

3.4 Flow Chart

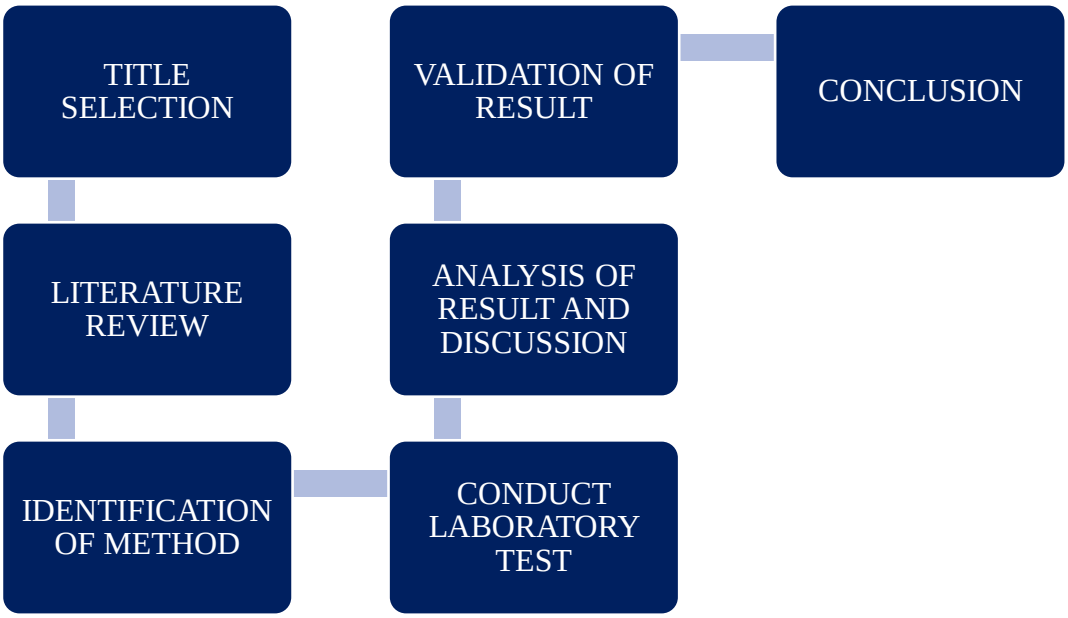

Figure 14: Flow chart

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3.5 Gantt Chart

NO	ACTIVITY	WEEK													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
FYP 1															
1	Selection of Project Topic														
2	Literature Review Analysis														
3	Methodology Identification														
4	Submission of Extended Proposal														
5	Formulation Preparation														
6	Proposal Defence														
7	Submission of Interim Report														
FYP 2															
1	Substrate Preparation														
2	Samples Preparation														
3	Furnace Test														
4	Immersion Test														
5	Fire testing														
6	Data gathering														
7	Final report preparation														
8	VIVA presentation														
9	Submission of Project Dissertation														

Table 5: Gantt chart

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CHAPTER 4: RESULT AND DISCUSSION

4.1 Waste Glass Powder

Before the waste glass powder being used for making the coating, it has been sieved and retained at 212µm sieve. The sieved waste glass powder has been sent for Thermogravimetric Analysis (TGA) to investigate the degradation temperature of the waste glass powder. Then, the sieved waste glass powder has been sent for X-Ray Fluorescence (XRF) Analysis to determine the elemental composition of the waste glass powder. The result is shown below:

4.1.1 Thermogravimetric Analysis (TGA)

By using TGA, the residual weight of the waste glass powder was analysed. Under a controlled temperature and air condition, this analysis has been carried out to give an overview of the degradation process of the waste glass powder. Figure below shown the graph of residual weight (%) against temperature for sieved waste glass powder under this analysis:

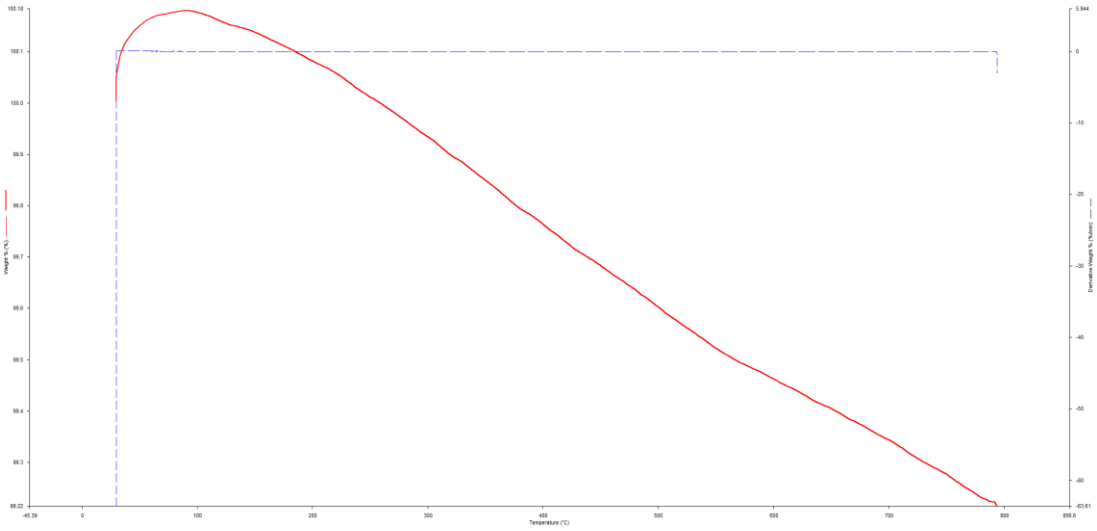

Figure 15: Residual weight (%) against temperature graph for waste glass powder under TGA

Based on the graph above, at the starting point of this analysis, there was 100% residual weight of the waste glass powder. The residual weight increased a bit for the first 100^{o}C before it starts to reduce and at approximately 200^{o}C, the residual weight of the waste glass powder was back to 100% before continuing to reduce. At approximately 800^{o}C, the residual weight of the waste glass powder was 99.22%.

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The residual weight of the waste glass powder only drops less than 1% even after being heat up with the temperature up to 800ºC. The result shows that the waste glass powder has the potential to be used as filler and can enhanced the fire-retardant capability of the intumescent coating since the intumescent coating will be exposed to high temperature fire, up to 800ºC to 900ºC.

4.1.2 X-Ray Fluorescence (XRF) Analysis

To determine the elemental composition of material, in this case is sieved waste glass powder, XRF analysis was carried out. This analysis is a non-destructive analytical technique. Table below shows the elemental composition of the waste glass powder:

Formula	Concentration (%)	Formula	Concentration (%)
Si	33.00	**S**	0.12
Ca	13.50	**Ti**	0.12
Na	3.75	**Cu**	0.07
Al	0.78	**Cl**	0.07
Mg	0.56	**Sb**	0.06
P	0.45	**Zr**	0.04
W	0.44	**Co**	0.03
Fe	0.27	**Mn**	0.02
K	0.13	**Sr**	0.01

Table 6: XRF analysis result for waste glass powder

From the table above, most of the element present in the waste glass powder is silica (Si) which is 33% concentration, followed by 13.50% concentration of calcium (Ca) and sodium (Na) which is 3.75% concentration.

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4.2 First Formulation Samples

There are several tests that has been carried out on the samples containing waste glass powder as filler and the samples' name are S001, S002, S003, S004 and S005 based on the first formulation. The tests are as following:

4.2.1 Immersion Test

Tables and graphs below show the average thickness (mm) and average weight (g) of each sample during the 15 days of immersion test and 24 hours after the immersion test:

	Average Thickness (mm)						
	Day 0	**Day 3**	**Day 6**	**Day 9**	**Day 12**	**Day 15**	**Day 16 (24 hours test)**
S001	3.17	3.19	3.20	3.21	3.21	3.24	3.18
S002	3.58	3.57	3.59	3.60	3.59	3.61	3.61
S003	3.58	3.57	3.55	3.58	3.60	3.58	3.59
S004	3.47	3.51	3.51	3.52	3.51	3.52	3.51
S005	3.90	3.92	3.92	3.93	3.94	3.95	3.91
CF	3.36	3.37	3.40	3.42	3.40	3.40	3.43

Table 7: Average thickness of first formulation samples

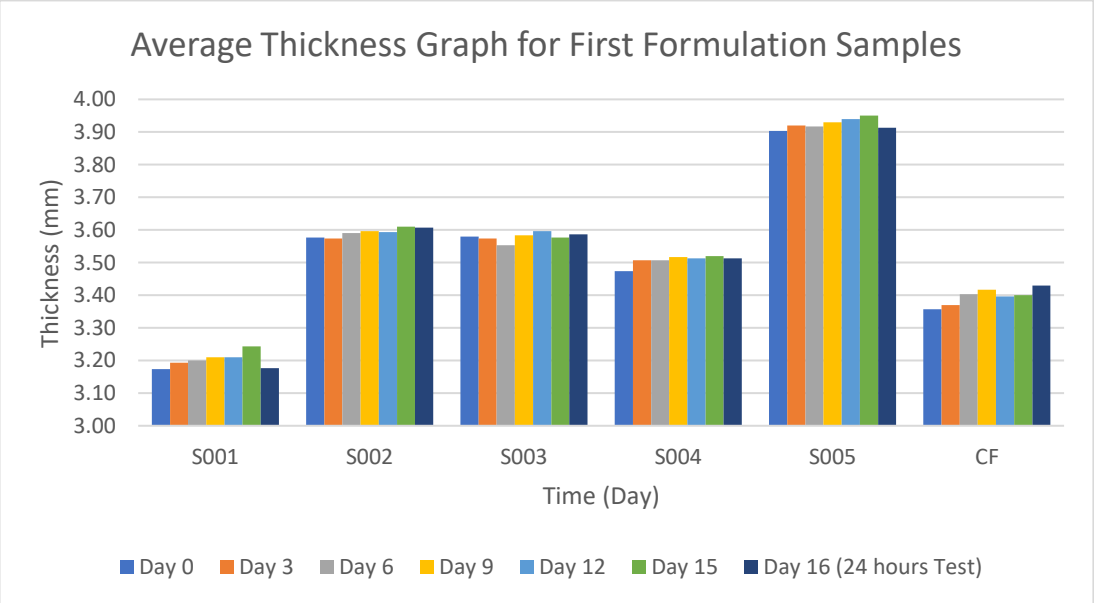

Figure 16: Average thickness graph for first formulation samples

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	Average Weight (g)						
	Day 0	**Day 3**	**Day 6**	**Day 9**	**Day 12**	**Day 15**	**Day 16 (24 hours test)**
S001	38.2012	38.2074	38.2201	38.2265	38.2189	38.2284	38.1619
S002	42.9969	42.9967	43.0010	43.0051	43.0027	42.9965	42.9585
S003	36.2659	36.2619	36.2625	36.2630	36.2550	36.2862	36.2193
S004	40.2618	40.2592	40.2638	40.2672	40.2639	40.2603	40.2304
S005	36.8715	36.8910	36.9139	36.9359	36.7861	36.9546	36.8845
CF	34.3834	34.3805	34.3809	34.3843	34.3876	34.3717	34.3182

Table 8: Average weight of first formulation samples

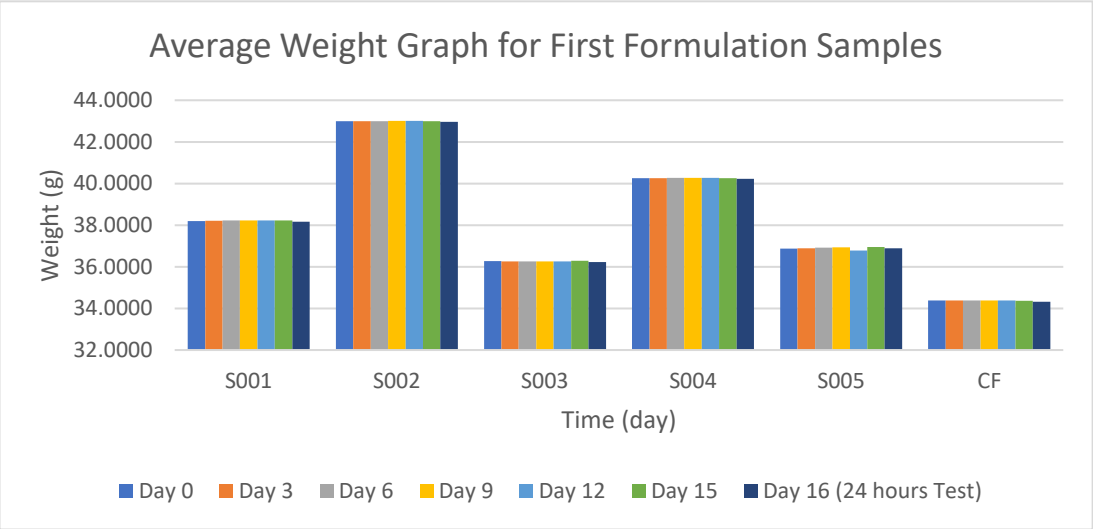

Figure 17:Average weight graph for first formulation samples

It can be observed that there is not much change from the beginning of the immersion test, which was on day 0 to day 15. Most of the samples might have increased slightly, for both of their average thickness and average weight, due to some corrosion occur at the backside of the sample that make the samples to become slightly thicker. This is because there was no coating covered at the backside of each samples, which is the carbon steel structure, so they were exposed directly to the natural environment, which was seawater. From the results recorded, it showed that there will be no effect for each of the samples whenever they were exposed towards seawater. The coating will not become thinner and will not peeled off from the carbon steel structure. So, this coating can be used for the offshore platform structure for example, since it can withstand the natural environment.

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4.2.2 Furnace Test

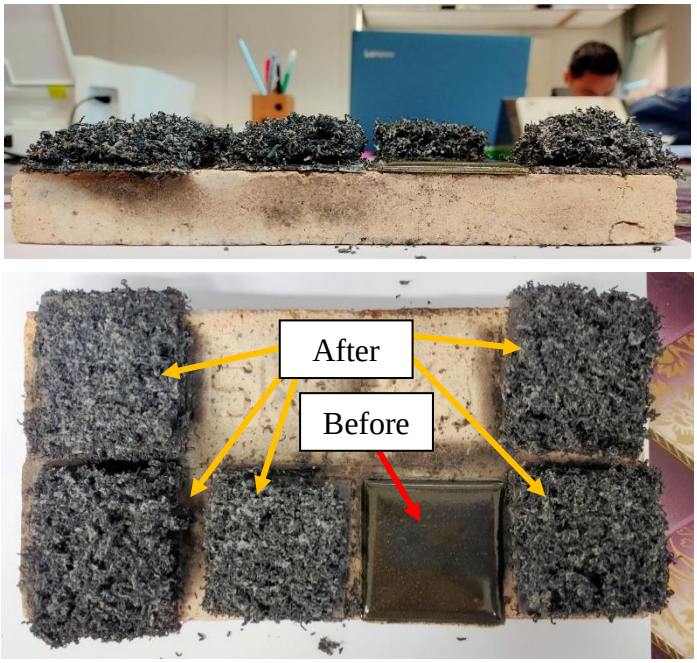

Figure 18: First formulation samples after furnace test

Figures above shown the first formulation samples after furnace test. From the observation, there are two samples that shows shrinkage, which are sample S001 and S004. Without enough adhesion strength, the char produced were not properly attached to the carbon steel structure and easily peeled off from the carbon steel structure. For this kind of conditon, both of the samples were rejected since the char formed will failed to protect the structure. Each of the samples of first formluation is shown below:

Figure 19: S001 after furnace test

Figure 20: S002 after furnace test

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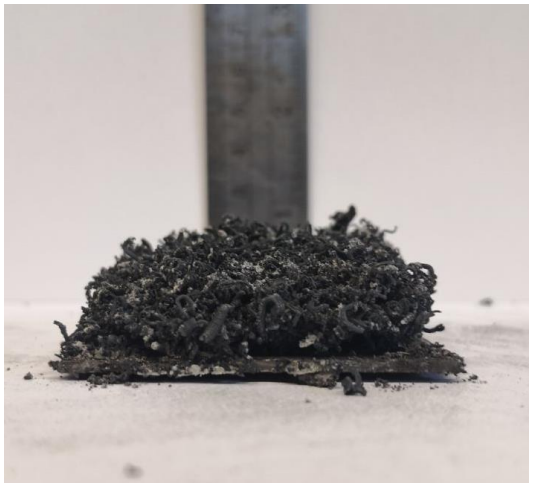

Figure 21: S003 after furnace test

Figure 22: S004 after furnace test

Figure 23: S005 after furnace test

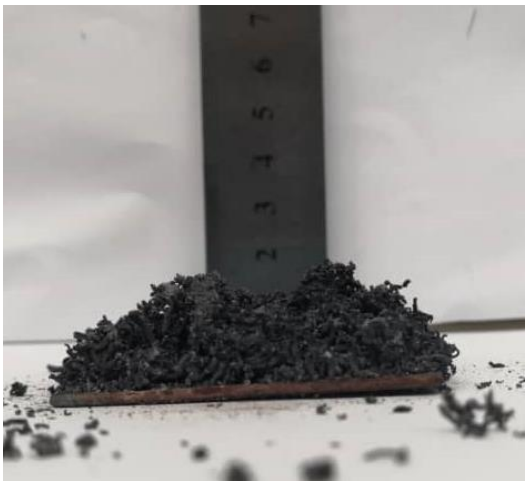

Figure 24: CF after furnace test

Table and graph below show the expansion of first formulation samples after the furnace test:

	Thickness Before (mm)	Thickness After (mm)	Expansion (mm)
S001	3.27	14.85	11.58
S002	3.65	16.89	13.24
S003	3.66	17.25	13.59
S004	3.66	18.65	14.99
S005	3.90	20.33	16.43
CF	2.73	14.34	11.61

Table 9: Expansion of first formulation samples

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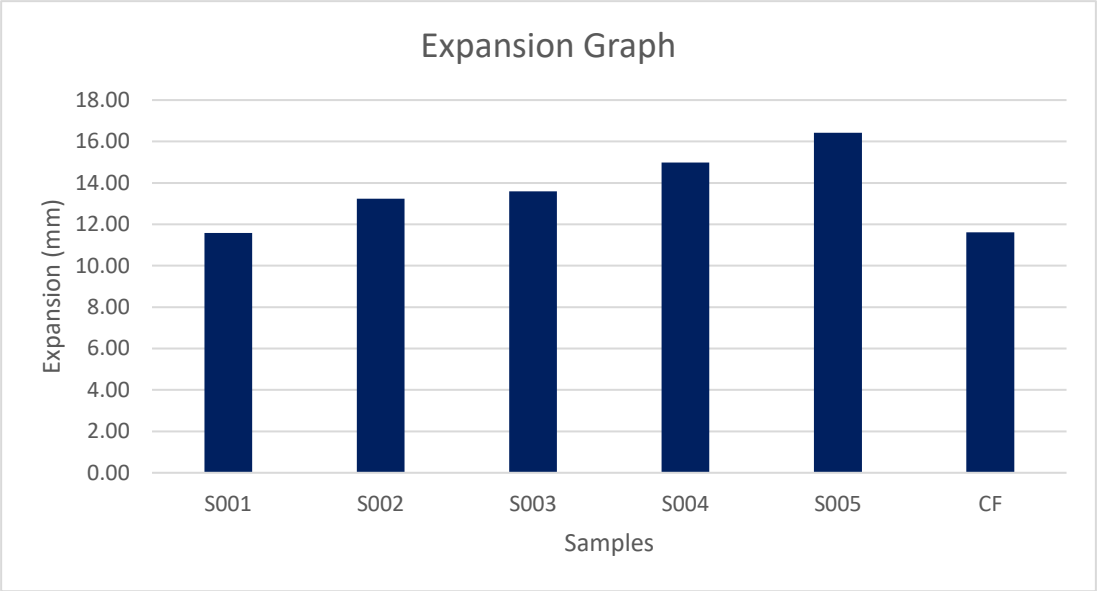

Figure 25: Graph of expansion for first formulation samples

From the graph above, the highest char expansion is the formulation that contains the most wt.% of waste glass powder, which is S005. The formulation contains of 5 wt.% of glass powder, with expansion of 16.43mm. Then, followed by sample which contain 4 wt.% of waste glass powder and sample which contain 3 wt.% of waste glass powder, with expansion of 14.99mm and 13.59mm respectively. The least expansion is the sample that contain 1 wt.% of glass powder. So, the expansion increases as the wt.% of waste glass powder in the formulation is increases.

4.2.3 Intumescent Factor

To determine the expansion of the coating, intumescent factor need to be calculated. The expansion needs to be compared to the control initial thickness. Intumescent factor can be calculated using Eq. 1 stated earlier:

	Coating thickness (mm)	Expansion (mm)	Intumescent Factor
S001	3.27	11.58	5.71
S002	3.65	13.24	5.47
S003	3.66	13.59	5.59
S004	3.66	14.99	6.24
S005	3.90	16.43	6.21
CF	2.73	11.61	8.22

Table 10: Intumescent factor of first formulation samples

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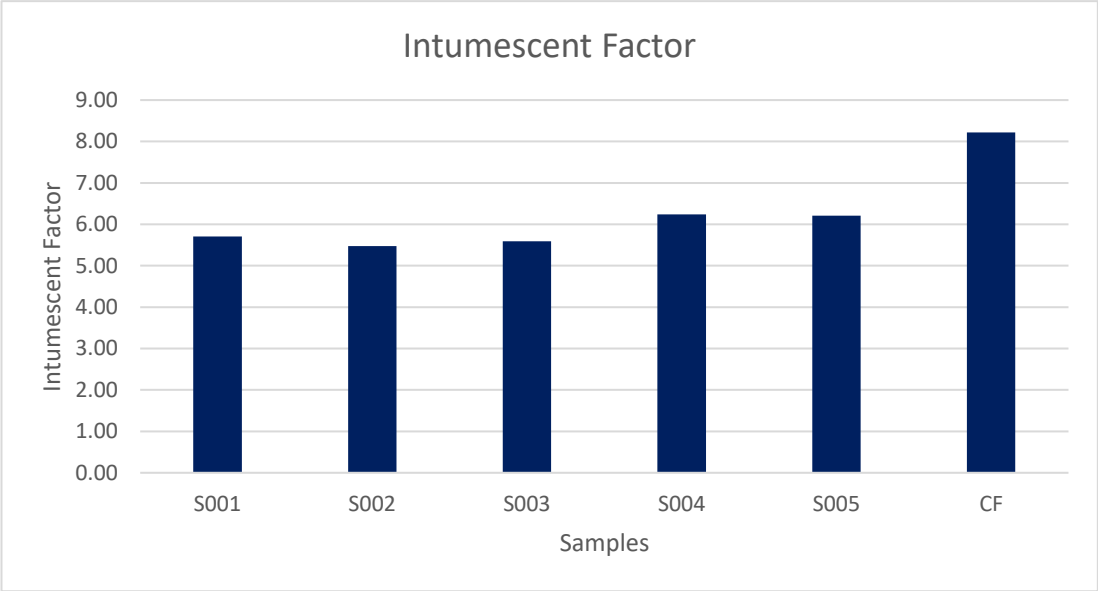

Figure 26: Intumescent factor graph for first formulation samples

Based on the figure above, the highest intumescent factor achieved by sample CF, which has the intumescent factor of 8.22. Then, it followed by sample S004, which contain 4 wt.% of waste glass powder, and sample S005, which contain 5 wt.% of waste glass powder, with slight different in intumescent factor which is 0.03 between both of the samples.

Since sample S001 experienced shrinkage of the char produced, from the figure above, it can be concluded that as the wt.% of waste glass powder increased, the intumescent factor will also increased. However, 4 wt.% of waste glass powder is the limit since the intumescent factor dropped with the usage of 5 wt.% of waste glass powder.

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4.2.4 Fire Test

Time (min)	Temperature (°C)					
	S001	S002	S003	S004	S005	CF
0	28.6	31.5	34.9	38.8	33.0	38.5
5	237	186.2	214	238.1	208.7	140.0
10	254.9	174.3	242.7	277.7	229.3	193.0
15	244.6	170.2	236.4	301.2	231.8	220.0
20	247.3	175.7	239.5	296.5	236.5	222.0
25	247.9	168.8	239.8	304.9	253.2	224.0
30	239.1	173.2	266.2	289.6	276.1	241.0
35	247	172.1	251.2	283.5	301.2	244.0
40	236.7	165.1	258.3	268.4	343.8	249.0
45	240.7	165.4	264	256.6	376.7	255.0
50	237.5	155.6	249.3	257.5	362.7	267.0
55	229.3	159.3	254.3	256.1	379.9	269.0
60	230.4	154.6	257.1	259.7	370.3	270.0

Table 11: Backside temperature of first formulation samples

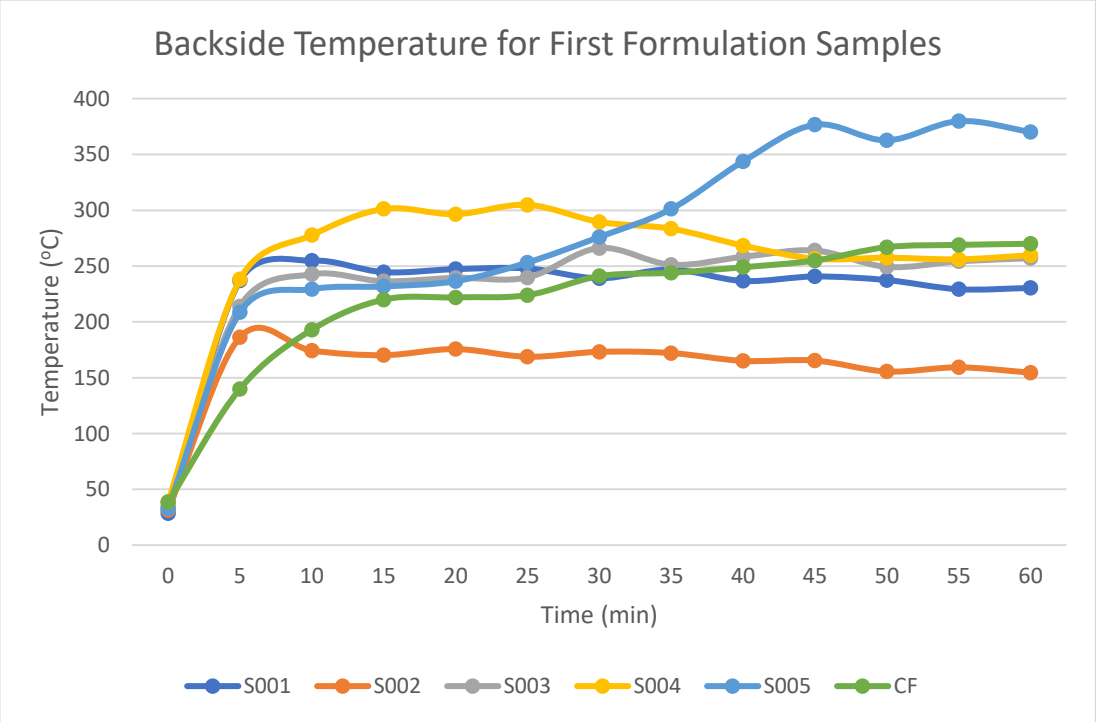

Figure 27: Backside temperature graph for first formulation samples

Based on the graph and table above, the backside temperature for sample S002, which contain 2 wt.% of waste glass powder, is the lowest among all the samples. The highest temperature recorded for sample S002 was 186.2°C which was lower

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compared to other samples' highest temperature recorded. The highest backside temperature recorded was 379.9^{o}C, which was for sample S005, which contains 5 wt.% of waste glass powder. It can be concluded that sample S002 is the best formulation among all the samples that using waste glass powder only as filler.

4.2.5 Discussion

Based on the furnace test that has been carried out, the sample CF showed that the char produced was not uniform and there were cracks and small holes ware observed on the char produced. However, the intumescent factor of sample CF is the highest. So, theoretically, the sample CF was the best formulation compared to other samples since it has the highest intumescent factor, but it was only the early indicator.

After fire test has been carried out, the result shows that the highest backside temperature recorded for sample S002 was better than sample CF, which was 186.2^{o}C and 270^{o}C respectively. For sample S002, the backside temperature recorded was constant during the last 10 minutes, and this due to the development of the intumescent effect. However, for sample CF, the backside temperature was increasing from the beginning of the test until the end, and this due to no development of intumescent effect from the sample.

With good adhesion of the char to the carbon steel structure, the best formulation was sample S002 since it has the lower highest backside temperature recorded compared to others. Despite the highest intumescent factor obtained by sample CF, the char produced was not dense, since the highest backside temperature recorded is higher than sample S002.

For the immersion test, sample S002 did not had any notable change in both weight and thickness after the test. So, it can be exposed to the natural environment, which is seawater and still the coating will not become thinner.

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4.3 Second Formulation Samples

There are several tests that has been carried out on the samples containing waste glass powder and Zirconium Phosphate as fillers and the samples' name are ZS091, ZS092, ZS093, ZS094 and ZS095 based on second formulation. The tests are as following:

4.3.1 Immersion Test

Tables and graphs below show the average thickness (mm) and average weight (g) of each sample during the 15 days of immersion test and 24 hours after the immersion test:

	Average Thickness (mm)						
	Day 0	**Day 3**	**Day 6**	**Day 9**	**Day 12**	**Day 15**	**Day 16 (24 hours test)**
ZS091	3.70	3.68	3.68	3.70	3.69	3.70	3.70
ZS092	3.84	3.84	3.83	3.80	3.86	3.82	3.84
ZS093	4.02	4.01	4.02	4.03	4.02	4.02	4.02
ZS094	4.11	4.09	4.12	4.07	4.12	4.08	4.09
ZS095	4.02	4.02	4.02	4.01	4.03	4.00	4.02
CF	3.36	3.37	3.40	3.42	3.40	3.40	3.43

Table 12: Average thickness of second formulation samples

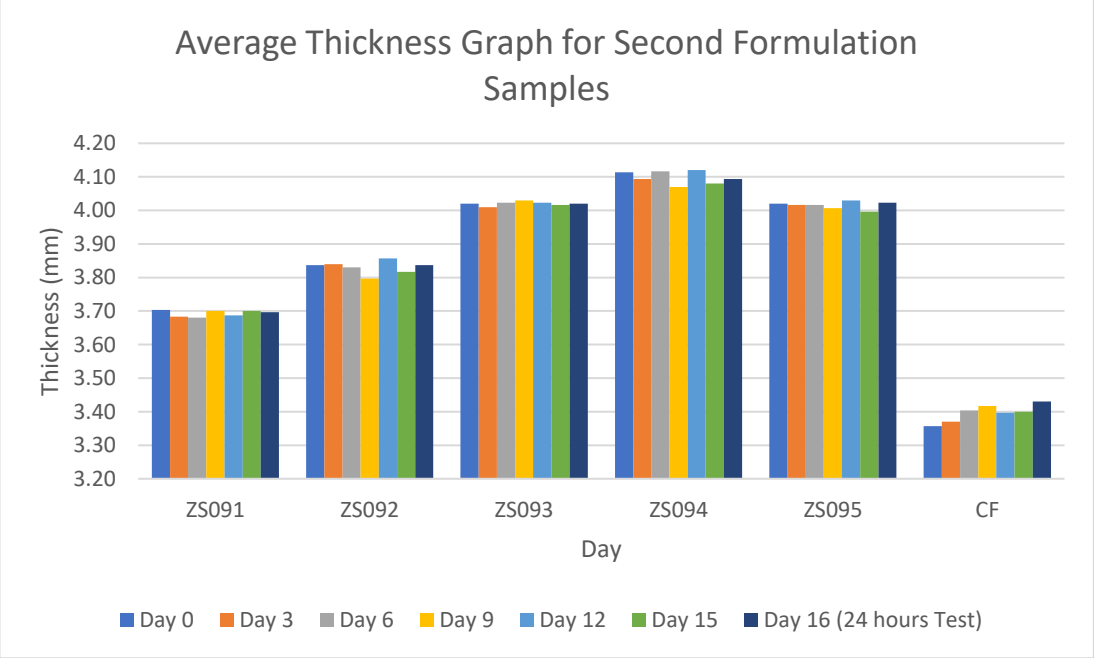

Figure 28: Average thickness graph for second formulation samples

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	Average Weight (g)						
	Day 0	**Day 3**	**Day 6**	**Day 9**	**Day 12**	**Day 15**	**Day 16 (24 hours test)**
ZS091	36.5258	36.5214	36.5212	36.5214	36.5052	36.4982	36.4748
ZS092	36.0039	36.0037	36.0006	35.9999	35.9990	35.9914	35.9454
ZS093	38.1918	38.1936	38.1904	38.1934	38.1804	38.1769	38.1465
ZS094	38.4090	38.4111	38.4081	38.3746	38.3509	38.3134	38.2841
ZS095	38.0195	38.0285	38.0300	38.0187	38.0130	38.0238	37.9630
CF	34.3834	34.3805	34.3809	34.3843	34.3876	34.3717	34.3182

Table 13: Average weight of second formulation samples

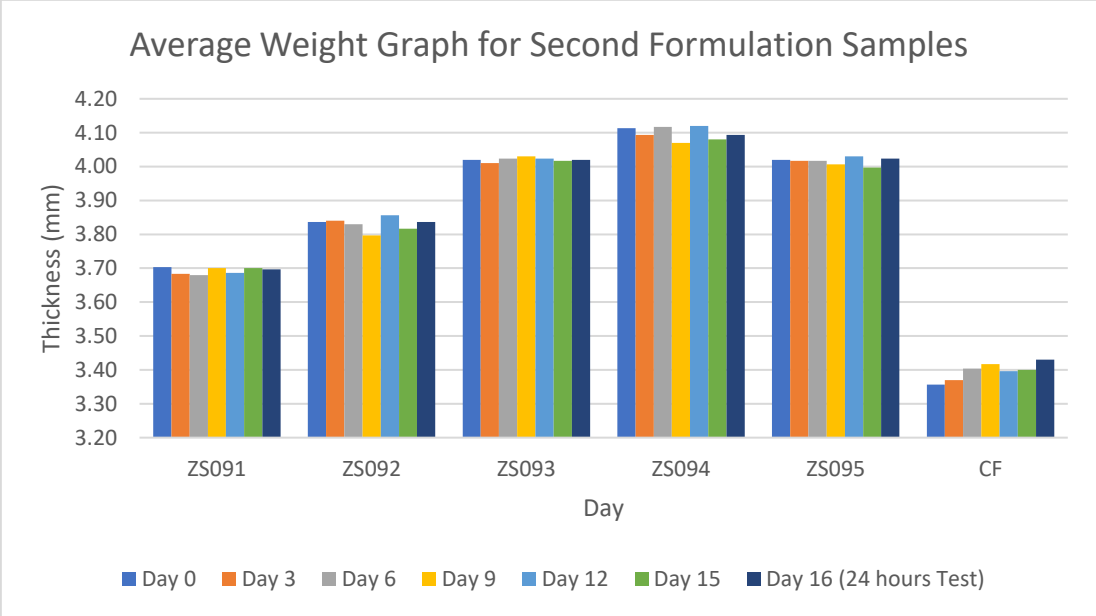

Figure 29: Average weight graph for second formulation samples

Based on the graphs and tables shown above, the result for immersion test of the samples using waste glass powder and Zirconium Phosphate as fillers are the same as the samples that only use waste glass powder as filler. There was not much change, in both weight and thickness of the samples, from the beginning of the test until the end. From the results recorded, it showed that there will be no effect for each of the samples whenever they were exposed towards naturel environment, which was seawater. The coating will not become thinner and will not peeled off from the carbon steel structure. So, this coating can be used for the offshore platform structure for example, since it can withstand the natural environment.

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4.3.2 Furnace Test

Figure 30: Second formulation samples after furnace test

Figure shown above are second formulation samples, which are ZS091, ZS092, ZS093, ZS094 and ZS095 after furnace test. From the observation, samples ZS091 and ZS095 showed shrinkage after the test, which means that the samples did not have strong adhesiveness, thus cannot protect the structure. Each of the samples of second formluation is shown below:

Figure 31: ZS091 after furnace test

Figure 32: ZS092 after furnace test

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Figure 33: ZS093 after furnace test

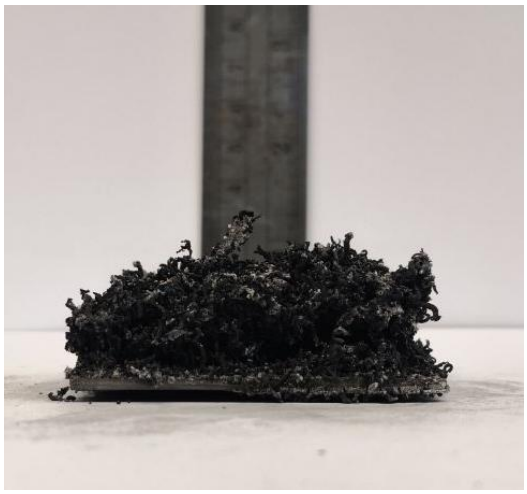

Figure 34: ZS094 after furnace test

Figure 35: ZS095 after furnace test

Figure 36: CF after furnace test

Table and graph below show the expansion of first formulation samples after the furnace test:

	Thickness Before (mm)	Thickness After (mm)	Expansion (mm)
ZS091	3.90	20.57	16.67
ZS092	3.96	19.02	15.06
ZS093	3.61	16.65	13.03
ZS094	3.84	16.87	13.04
ZS095	4.17	24.24	20.07
CF	2.73	14.34	11.61

Table 14: Expansion of second formulation samples

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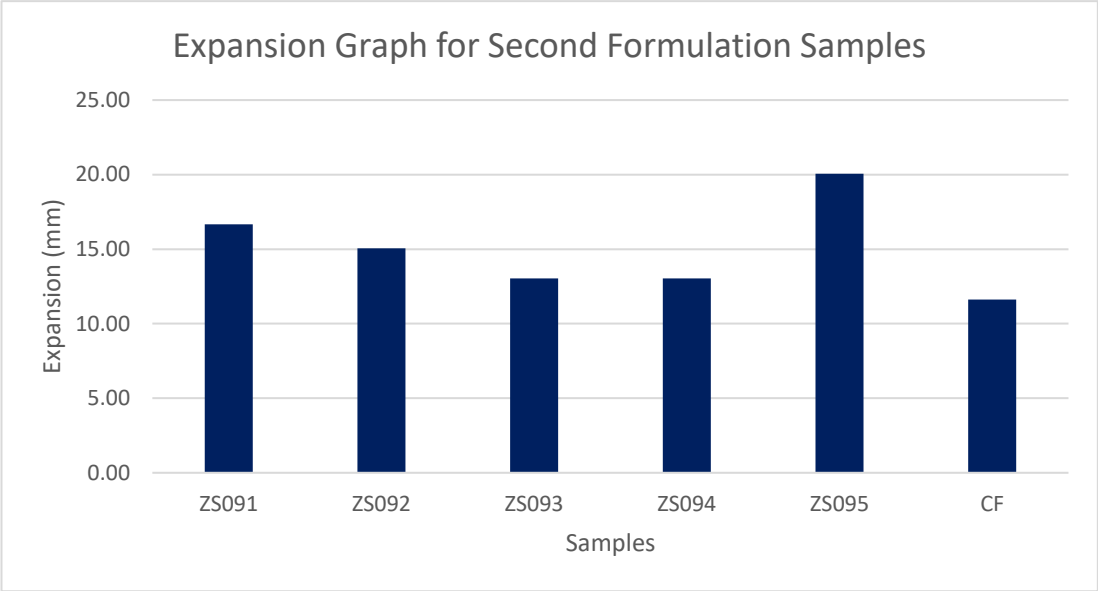

Figure 37: Graph of expansion for second formulation samples

Based on the table and figure above, the highest expansion sample is sample ZS095, which contains 5 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate, with expansion of 20.07mm. Then followed by sample ZS091, which contains 1 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate, and sample ZS092, which contains 2 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate, with expansion of 16.67mm and 15.06mm respectively. So, by ignoring sample ZS091 and ZS095 due to shrinkage of the char produced, the expansion of the char decreases as the wt.% of the fillers increases.

4.3.3 Intumescent Factor

The intumescent factor can be calculated using Eq. 1 stated earlier:

	Coating thickness (mm)	Expansion (mm)	Intumescent Factor
ZS091	3.90	16.67	6.32
ZS092	3.96	15.06	5.51
ZS093	3.61	13.03	5.46
ZS094	3.84	13.04	4.94
ZS095	4.17	20.07	6.96
CF	2.73	11.61	8.22

Table 15: Intumescent factor of second formulation samples

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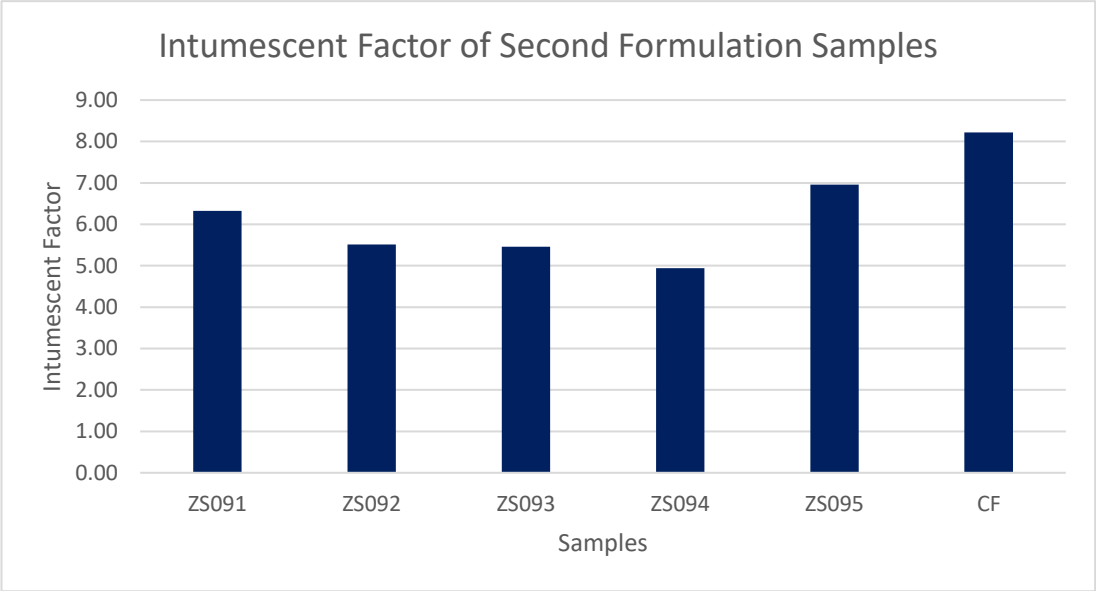

Figure 38: Intumescent factor graph for second formulation samples

Based on the figure above, sample CF has the highest intumescent factor which is 8.22. Then followed by sample ZS095, which contains 5 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate, and sample ZS091, which contains 1 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate. The lowest intumescent factor achieved by sample ZS094, which contains 4 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate.

Since sample ZS091 and ZS095 both experienced shrinkage of their char produced, from the figure above, it can be concluded that as the wt.% of fillers used increased, the intumescent factor were decreased.

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4.3.4 Fire Test

Time (min)	Temperature (°C)					
	ZS091	ZS092	ZS093	ZS094	ZS095	CF
0	27.4	31.1	31.9	37.1	35.6	38.5
5	235.2	214.3	236.1	236	225.1	140.0
10	224.7	227.6	327.4	243.7	266	193.0
15	234	226.3	423.8	244.1	292.9	220.0
20	252.6	220.5	397.6	251.4	341.7	222.0
25	230.8	210.5	384.6	251.6	333.9	224.0
30	224.1	197	369.9	254.9	325.4	241.0
35	206.2	192.4	373.2	255.3	319.3	244.0
40	210	211.5	374.1	262.2	311.2	249.0
45	215.9	241.8	374.4	267.4	305.2	255.0
50	227.7	258.8	363.1	271.8	307.2	267.0
55	236.8	267.3	355.9	285.4	303.1	269.0
60	238.9	266.7	345.3	289.5	306.9	270.0

Table 16: Backside temperature of second formulation samples

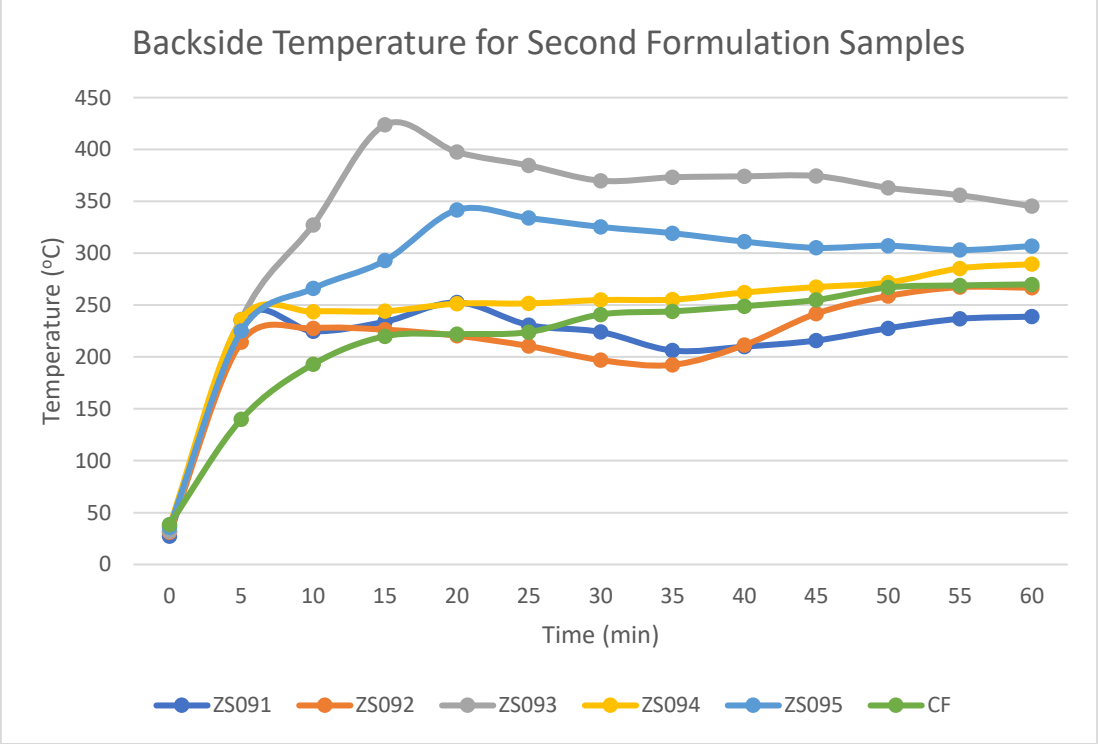

Figure 39: Backside temperature graph for second formulation samples

Based on the graph and table above, the highest backside temperature recorded for sample ZS091, which contain 1 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate, is the lowest among all the samples, which was 252.6°C. Then,

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followed by sample ZS092, which contain 2 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate, with highest backside temperature recorded was 267.3^{o}C. The highest backside temperature recorded was 423.8^{o}C, which was for sample ZS093, which contains 3 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate. Since sample ZS091 and ZS095 both experienced shrinkage of their char produced, it can be concluded that sample ZS092 is the best formulation among all the samples that using waste glass powder and Zirconium Phosphate as fillers.

4.3.5 Discussion

Based on the tests that has been carried out, it can be concluded that the best formulation is ZS092, which contain 2 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate, even the intumescent factor so samples ZS091, ZS095 and CF are higher. This is because during the furnace test, there were shrinkage observed on sample ZS091 and ZS095, and the char produced by sample CF was not uniform and there were cracks and small holes ware observed on the char produced. So, sample ZS092 is chosen since the adhesion of the char is good.

For fire test, the highest backside temperature recorded for sample ZS092 was 267.3^{o}C which is lowest among other samples. Despite sample ZS091, ZS095 and CF has higher intumescent factor, the char produced was not dense, since the highest backside temperature recorded is higher than sample ZS092. So, with denser char produced by sample ZS092, a better fire portection of intumescent coating was provided towards the carbon steel structure.

For the immersion test, sample Z092 did not had any notable change in both weight and thickness after the test. So, it can be exposed to the natural environment, which is seawater and still the coating will not become thinner.

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CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This study found only a few researchers had used waste glass powder as a filler in the fire intumescent coating. But many researchers used waste glass powder as one of the mixtures to make a new version of concrete. Most of them reported that under high temperature, the spalling and degradation of the concrete will be reduced by using waste glass powder, and some stated that the production of high-temperature resistant concretes is possible. Furthermore, the uses of glass powder cement replacement can help to reduce density, absorption, tensile strength and compressive strength of the concrete (Aliabdo et al., 2016). Therefore, this project uses waste glass powder as the filler for fire intumescent coating as its properties and the results of past researchers were outstanding.

From the results, it shows that from the intumescent factor, sample CF is the best formulation since the increment of the char height is highest after undergo furnace test. However, the char was not uniform, crack and there were several holes observed on the char produced. Furthermore, the backside temperature of simple CF was high and increase continuedly during the fire test since the absence of intumescent effect. Despite having highest intumescent factor compared to other samples, sample CF still has higher backside temperature recorded since the char produced by sample CF has lack of strength and not dense. So, the best formulation cannot be decided based on intumescent factor alone but still depend on the backside temperature of the samples through fire test.

The formulation was improved by adding fillers, which are waste glass powder and Zirconium Phosphate. At first, only waste glass powder was added into the formulation before the addition of 0.9 wt.% into each of the formulation containing waste glass powder. By adding the waste glass powder, the thermal behaviour of the samples was improved. The highest backside temperature recorded for sample CF was 270^{o}C while for sample S002, with formulation containing 2 wt.% of waste glass powder was 186.2 ^{o}C. However, the highest backside temperature recorded for sample ZS092, which contain 2 wt.% of waste glass powder and 0.9 wt.% of Zirconium Phosphate was 267.3^{o}C, which is as high as sample CF. So, by adding Zirconium

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Phosphate into the formulation can reduce the fire protection towards the carbon steel structure.

With good adhesion strength of char produced towards the carbon steel structure, sample S002 has the best formulation. From the above conclusions, this project has achieved its objectives since intumescent fire-retardant coating by using waste glass powder was successfully developed and the effect of composition of waste glass powder on thermal performance has been studied.

5.2 Recommendation

There are several recommendations that can be considered to improve the project outcome in the future such as:

1. Char morphology of the samples can be analysed using Field Emission Scanning Electron Microscope (FESEM).
2. Performance of the samples can be studied further in different conditions and environments.
3. Adhesion test can be performed on the samples to determine the minimum tensile stress needed to detach the coating from the carbon steel structure, perpendicularly.
4. The result of the fire test is not stable for each of the samples due to the windy surrounding of the place where the test was conducted. Thus, the fire test could be done in an enclosed place.

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