HEAT SHIELDING PERFORMANCE OF MINERAL BASED INTUMESCENT FIRE RETARDANT COATING

by

MOHD FARITH BIN SHARIFF

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A project dissertation submitted to the

Mechanical Engineering Programme

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In partial fulfillment of the requirement for the

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CERTIFICATION OF APPROVAL

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Approved by,

(AP Dr. Faiz Ahmad)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September, 2012

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 FARITH BIN SHARIFF

ABSTRACT

The objective of the paper is to develop intumescent fire retardant coatings with additives of talc and alumina and study the thermal performance. Intumescent coating is a fire protection system designed to reduce heat transfer from a fire to substrate to maintain its integrity. The intumescent coatings are based from three main intumescent components of Expandable Graphite (EG), Ammonium Poly Phosphate (APP) and Melamine (MEL). Epoxy resin of Bisphenol-A (BPA) and hardener Triethylene-tetramine (TETA) were used as curing agent. 11 samples were developed with varying filler percentage of talc and alumina. All 11 formulations were burned in furnace oven at maximum temperature of 450°C and expansion of char was observed. Formulation with 3% filled alumina and 2% filled talc produces the highest char expansion of 1874.78% and 1633.33% of its original thickness respectively. The results show that the char expansion percentage was improved with added filler of talc and alumina.

Heat shielding test was conducted to all 11 samples for 60 minutes each and the backside temperature of the steel substrate was logged for every two minutes. The results show dramatic improvement in intumescent effect with added filler of talc and alumina. The best intumescent effect was achieved with formulation A5 and T3 with maximum temperature of 154.6°C and 163.6°C respectively after reaching equilibrium.

A study on the effect of water resistance was conducted on all eleven samples using the gravimetric method. The coatings were immersed in distilled water and weighed every 24 hours for 168 hours. The weight changes were measured. It is found that small pores and surface damages appeared on alumina filled coating and no defect was found on talc filled coating.

ACKNOWLEDGEMENT

First and foremost, I would like to express my praises to ALLAH for his blessing.

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CHAPTER 1
INTRODUCTION

1.1 Background Study

Steel is a commonly used material in the construction of our regular infrastructures almost existing in all construction of buildings, vehicles, airplanes and oil platforms. Steel acquire good strengths and ductility while also being incombustible, however steel loses its physical properties when subjected to heat at high temperature such as in the case of fire. Heat treated carbon steel begins to lose its mechanical properties at temperatures above 400°C and continues to reduce in strength at steady rate up to temperature of 800°C [1]. Therefore it has become an important priority to develop a fire protection system for the construction industry.

Intumescent coating is a type of passive fire protection system which commonly used to protect steel against fire. The mechanism behind intumescent coatings lies in the ability of to reduce the rate of heat transfer from the fire to the protected material. The coating comprises of three active ingredients, usually ammonium polyphosphate-APP, pentaerythritol-PER and melamine-MEL [2]. The three main ingredients are linked together with a binder which then reacts together at higher temperatures. The result of the reaction is the expansion of the coating and the formation of a foamed char. The resulting foamed char acts as a thermal barrier and help reduces heat transfer to the coated material. Typical intumescent coating materials also contain a significant amount of hydrates which are converted to water vapour when subjected to heat. The water vapour released acts a cooling effect until it is completely spent. The binders for intumescent coatings mainly based from organic binders which have good expanding effect and char structure. Although having a good expanding effect and char structure, organic binders release solvent, toxic gas and smoke in a fire [2]. Comparing with inorganic intumescent coatings, the amount of toxic gas emission and smoke upon heating is relatively low [2]. However, the inorganic intumescent coating only protects against fire at low temperature and are vulnerable to water and moisture [3].

1.2 Problem Statement

In recent years, halogenated flame retardant materials have been widely used to coat and protect steel structures from fire. The performance of halogenated flame retardant materials is relatively high however the release of toxic gases and smoke during the burning has brought serious concern to health and the environment [4]. Alternatives such as inorganic intumescent coatings have been considered to replace halogenated flame retardant materials. However inorganic intumescent coatings are very vulnerable to water or moisture therefore the use of mineral based filler in intumescent coating is deemed as one of the bright solution for the situation.

1.3 Objectives

- To develop an intumescent fire retardant coating with added fillers of Talc and Alumina and study the thermal performance

1.4 Significant of the project

Modern construction industry has been relying heavily on halogenated intumescent coatings in recent years to enhance the structure capability in handling extreme heat in the case of fire. However concern rises from the release of toxic gases and smoke produced by the burning of the coatings. Therefore it is very important to look for other alternative to replace the halogenated flame retardant material. The usage of mineral based fillers is inferred to enhance the properties and thermal performance of the coating.

1.5 Scope of Study

The scope of the project focuses on studying the performance of mineral based intumescent coating with Talc and Alumina as the fillers. The tests conducted to study the thermal performance are fire test to study thermal degradation performance and furnace test to study the char formation. Static immersion test are conducted to evaluate water resistance of coating under gravimetric method. FESEM test is used to observe and study the microstructure of the char formation.

1.6 Feasibility of the Project

The time limit for the project is 14 weeks therefore all of the tests and results must be accomplished in time with respect to the planned Gantt chart. All of the objectives can be accomplished provided the procedures are closely followed.

CHAPTER 2
THEORY AND LITERATURE REVIEW

2.1 Intumescent Coating

Intumescent coating is a type of passive fire protection which utilizes the properties of inherently flame-retardant materials to insulate the steel thus giving the structure ‘buying time’ to evacuate personnel or event extending the time before the structure fail due to high temperature. Alternatives to fire protection system has been developed over the years and it has been found that intumescent flame-retardant coating is one of the easiest, economical and the most efficient ways to protect materials against fire. Other advantages of using intumescent coating as the fire protection system are the ability to prevent flames from spreading or penetrating and the unneeded modification to the intrinsic properties of the materials [5].

An intumescent coating comprises of three major components which are ammonium polyphosphate (APP) as acid source, melamine (MEL) as the blowing agent and pentaerythritol (PER) as carbon agent. In addition to that, various materials have been tested as an additive to further extend the fire protective time in a fire. Boric acid is an example of additive which is tested to improve the performance of the coating by providing longer thermal protection (highest expansion), better adhesion and better mechanical resistance [6].

2.2 Mechanism of Intumescent Coating

The mechanism of intumescent is characterized by expansion and mass loss of the coating to produce a foam char which expand above twice the original thickness [7] where its strength plays a vital role to protect the base from fire attack. A study performed by Anderson et al shows that the efficiency of the insulation of the char was contributed from the pockets of trapped gas within the porous char which act as a blowing agent to the solid material [8]. Throughout the years, there is not so much change in intumescent technology. Almost all coatings are based on the combination of the typical three main components: a dehydrating agent, a carbonaceous source, a spumific and a binder resin.

Through the combination of APP, PER and MEL, the first reaction of all three when subjected to heat flux is the decomposition of the dehydrating agent, ammonium polyphosphate into ammonia and phosphoric acid. At higher temperatures (200 – 300 ^{o}C) the acid will reacts with the carboniferous agent. The formed gases will expand thus initiating the intumescence process in the form of a carbonaceous char. The performance of the coating is measured based on the time or temperature at which the intumescence process begins and how long it acts as a thermal barrier.

The process of intumescence can be summarized briefly by the following figures:

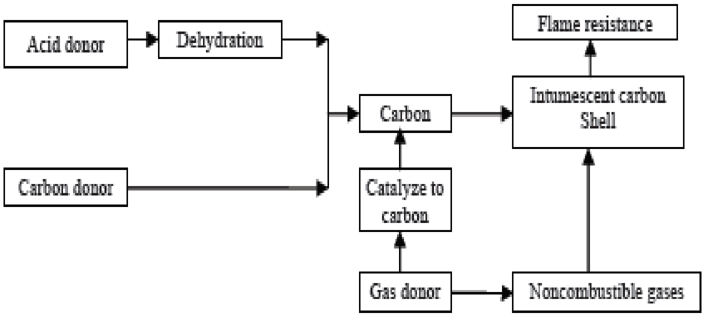


Figure 2-1: Intumescent flow diagram [8]

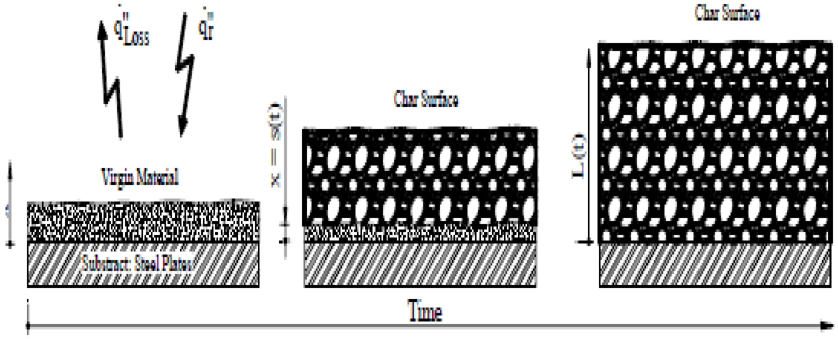


Figure 2-2: Char formation process [8]

2.3 Fillers in Intumescent Coating

Fillers are material added to a polymer to improve processing behavior and to modify the final properties of the product [9]. According to encyclopedia, fillers do not just modify the properties of a product, nevertheless can also be used to improve the cost [10,11]. Based on ASTM C859, [12] fillers are termed for a material that is inert and used to occupy space thus improving physical properties.

For a successful commercial use, a fire retardant fillers need to accomplish several characteristics to be deemed ideal for application. The characteristics are as follows [13]:

i. Ready availability and low cost
ii. Low toxicity
iii. Available in small sized particle, well defined morphology, low surface area and ability to be used at higher filler loadings
iv. Acquire significant endothermic decomposition in temperature range of 100°C - 300°C with at least 25% release of water and/or carbon dioxide by weight
v. Colourless
vi. Low levels of solubility, extractable salts and of potentially detrimental impurities

2.4 Effect of Fillers on Intumescent Performance

Sami Ullah et al performed a study on the effect of boric acid with kaolin clay on thermal degradation of intumescent fire retardant coating. In the experiment, the coating was based on expandable graphite (EG), ammonium polyphosphate (APP), melamine (MEL), kaolin clay, bisphenol-A epoxy resin BE-188 (BPA) and ACR hardener H-2310 [1]. Sami et al found that by addition modification of 3 to 5 wt% of Kaolin clay, the fireproofing times increased dramatically [1]. Sami found that the addition of Kaolin clay as reinforcement material will create a ceramic like protective barrier on the surface of insulation thus giving better intumescent effect [1].

A study performed by Kirschbaum [14] on the effect of alumina trihydrate (ATH) and magnesium hydroxide $Mg(OH)_2$ found that the combination of both filler give great intumescent performance when used together over wide range of weight percentages. The effect was down to the release of water vapour during vapour phase. The resulting combination of the oxides also contributes to the increased performance.

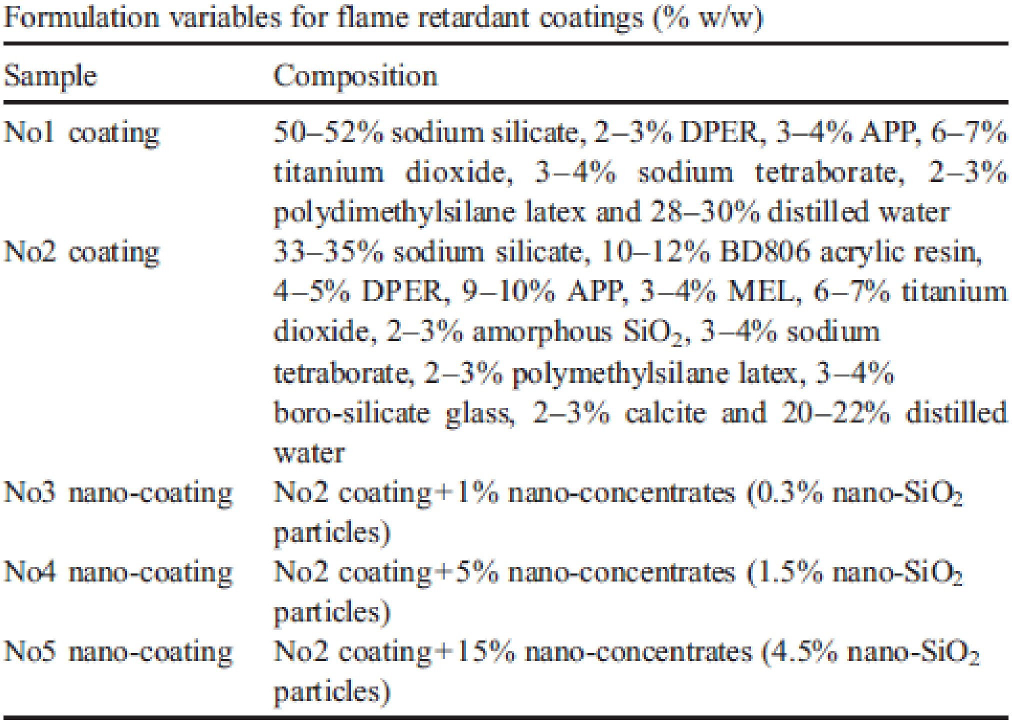
Duquesne et al. [15] studied on the development of intumescent coating with variable talc fillers. The study found that talc reinforces the mechanical properties of polymer and subsequently modifies the mechanical stability of the intumescent protective shield.

A study on the addition of nano-sized fumed SiO_2 and alumina Al_2O_3 has been conducted by Chou et al [16]. Chou reported that without additives or reinforcement, the traditional intumescent coatings of APP/PER/MEL produce a fluffier barrier of fire retardant which can be easily penetrated by fire. After addition of high temperature fillers of SiO_2 and Al_2O_3, a compact microstructure is formed in the charred layer which contributes to better intumescent effect.

2.5 Effect of Fillers on Water Resistivity

Hannatul et al [23] performed immersion test on talc filled intumescent coating and found that no weight loss occurred and improvement in water resistance with increasing content of talc filler due to platy structure and hydrophobicity properties of talc.

Zhenyu Wang et al [17] performed static immersion test in his study of water resistance of intumescent nano-coatings. The samples used are as follows:

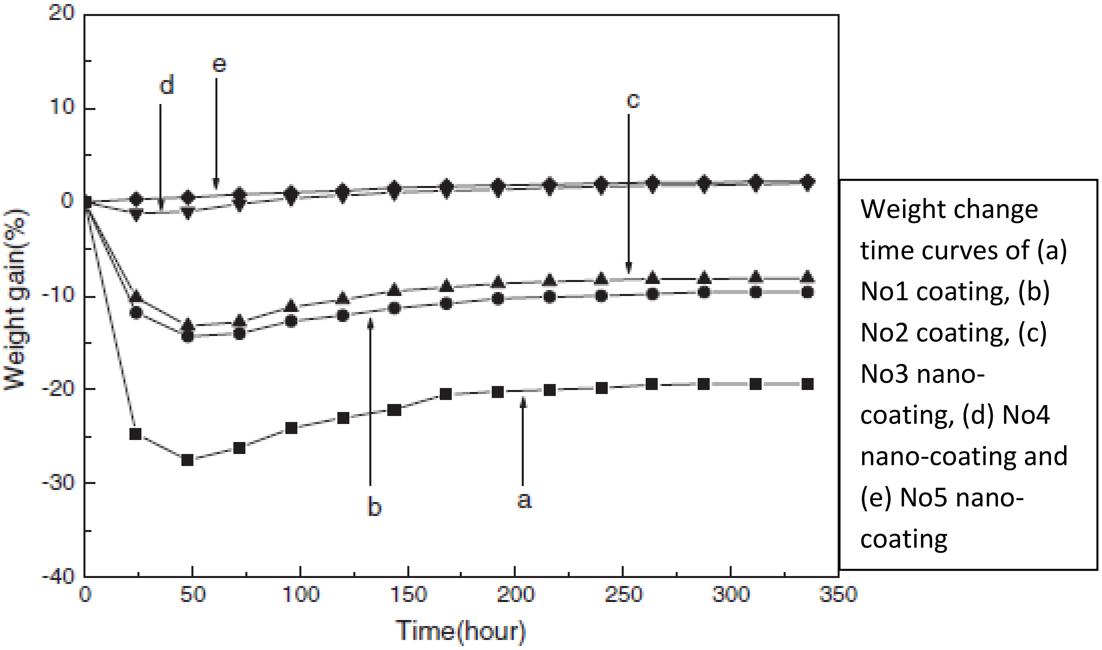


Formulation variables for flame retardant coatings (% w/w)

Sample	Composition
No1 coating	50–52% sodium silicate, 2–3% DPER, 3–4% APP, 6–7% titanium dioxide, 3–4% sodium tetraborate, 2–3% polydimethylsilane latex and 28–30% distilled water
No2 coating	33–35% sodium silicate, 10–12% BD806 acrylic resin, 4–5% DPER, 9–10% APP, 3–4% MEL, 6–7% titanium dioxide, 2–3% amorphous SiO_2, 3–4% sodium tetraborate, 2–3% polymethylsilane latex, 3–4% boro-silicate glass, 2–3% calcite and 20–22% distilled water
No3 nano-coating	No2 coating+1% nano-concentrates (0.3% nano-SiO_2 particles)
No4 nano-coating	No2 coating+5% nano-concentrates (1.5% nano-SiO_2 particles)
No5 nano-coating	No2 coating+15% nano-concentrates (4.5% nano-SiO_2 particles)

Figure 2-3: Water resistivity samples [17]

In the study, the samples are immersed in distilled water at 25 Deg C and weighed every 24 h. The samples are then dried for 1 h in a furnace and the weight change was calculated using the formula:



Weight change time curves of (a) No1 coating, (b) No2 coating, (c) No3 nano-coating, (d) No4 nano-coating and (e) No5 nano-coating

Figure 2-4: Weight change time curves [17]

From the result, Wang deduced that binders, flame-retardant additives and fillers affect the water resistance of the coatings [17]. Wang observed that No1 coating's result labeled as 'a', the film forming material (sodium silicate) and flame retardant additive (APP and DPER) are easily destroyed by water. The amount of weight loss caused by dissolution of sodium silicate is also higher than that of weight gain caused by water permeation of the initial immersion stage [17]. The trend of dissolution and weight loss of No1 weakened after 48 h of water immersion but the initial immersion stage produces pores and defects in the coating structure which contributed to 7% weight increase by the means of permeation of small molecules and ions.

Compare to No2 coating labeled as 'b', the coating comprises of waterborne acrylic resin, sodium silicate, flame retardant additives and fillers. The waterborne acrylic resin is known to form good continuous film that enhances the resistance to water permeation [17]. Compared to No1 coating maximum weight loss of 27.5% after 48 h of water immersion, No2 coating maximum weight loss is at 14.3% after 48 h water immersion. Wang conclude that there is appreciable improvement of water resistance in No2 coating.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

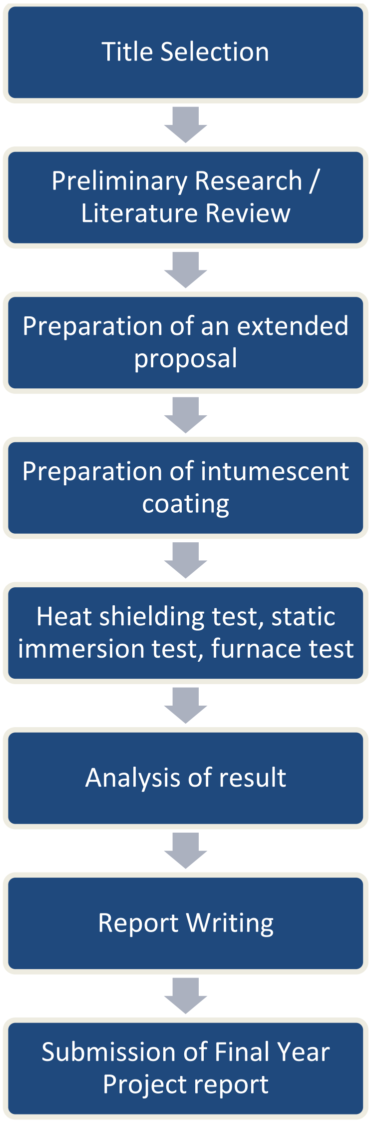


Figure 3-1: Methodology flowchart

3.2 Samples Preparation

The intumescent ingredients were mixed according to their composition measured from their individual weight percentage. The ingredients were mixed and grinded using Rocklabs grinding machine. The detailed formulation (EG, MEL, ZB, APP, epoxy, hardener and fillers) is as described in the following table.

Table 3-1: Sample formulations

Sample	**BPA (g)**	**TETA (g)**	**APP (g)**	**EG (g)**	**Mel (g)**	**ZB (g)**	**Filler (Talc/Alumina), (g)**
NF	44.44	22.22	11.11	5.56	5.56	11.11	0
T1	43.94	21.72	11.11	5.56	5.56	11.11	1 (Talc)
T2	43.44	21.22	11.11	5.56	5.56	11.11	2 (Talc)
T3	42.94	20.72	11.11	5.56	5.56	11.11	3 (Talc)
T4	42.44	20.22	11.11	5.56	5.56	11.11	4 (Talc)
T5	41.94	19.72	11.11	5.56	5.56	11.11	5 (Talc)
A1	43.94	22.22	11.11	5.56	5.56	11.11	1 (Alumina)
A2	43.44	21.72	11.11	5.56	5.56	11.11	2 (Alumina)
A3	42.94	21.22	11.11	5.56	5.56	11.11	3 (Alumina)
A4	42.44	20.72	11.11	5.56	5.56	11.11	4 (Alumina)
A5	41.94	19.72	11.11	5.56	5.56	11.11	5 (Alumina)

3.2.1 Weighing

The ingredients were weighted with respect to their weight percentage and mixed together to produce one sample. The sample can be applied to four substrates each of size 50 x 50 mm.

Figure 3-2: Ingredients weighing

3.2.2 Grinding

The measured samples were then grinded together using the Rocklabs grinding machine to mix the ingredients evenly and reduce the size into finer quality.

Figure 3-3: Grinding process

3.2.3 Mixing and Application to Substrate

After grinding, the samples were mixed with epoxy resin, Bisphenol-A (BPA) and Triethylenetetramine (TETA) before being applied to the steel substrate. The steel substrates were cut using a shear cutter to a size of 50 x 50 mm. Image of the samples after application to the substrates is as follow.

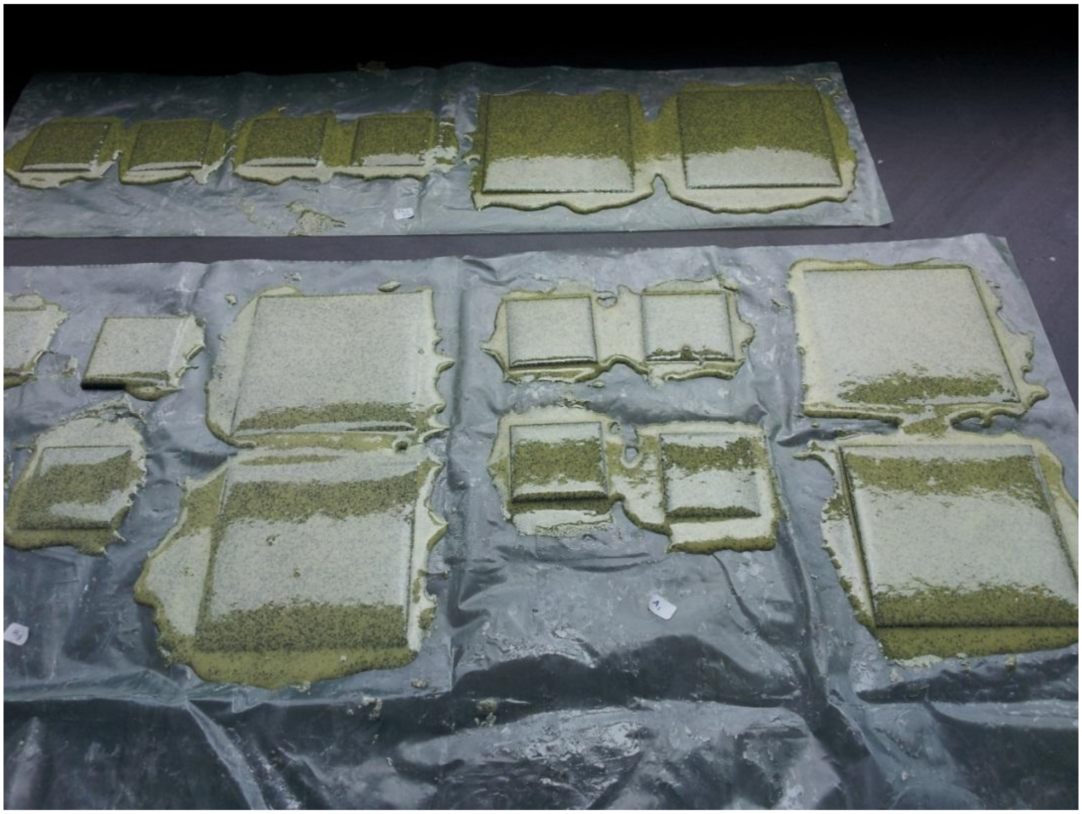


Figure 3-1: Coating curing on steel substrate

3.3 Furnace Test

The furnace test was conducted to study the char expansion of the samples. All of the samples were placed on a steel plate and fire brick and heated progressively in Carbolite electric furnace. The samples were heated from ambient temperature to 450°C in 20 min. The temperature was allowed to dwell at 450°C for 30 min before being cooled to room temperature of 30°C in 30 min. Physical properties of the char were observed and the final thickness of the char is compared with the original thickness of the coating to obtain the char expansion percentage.

Figure 3-2: Samples burning in furnace

Figure 3-3: Carbolite furnace

3.4 Heat Shielding Effect

Bunsen burner was used as a fire test source to study the heat shielding effect of intumescent fire retardant coating. The temperature logging was measured by using Anritsu AM-8000K data logger with two thermocouples measuring the surface temperature and the back side of the substrate. The temperature readings were taken every 2 min each for all samples with dimensions of 100x100 mm and burning time of 60 min. When the temperature of the backside reached 300°C, the time was defined as fire-resistant time.

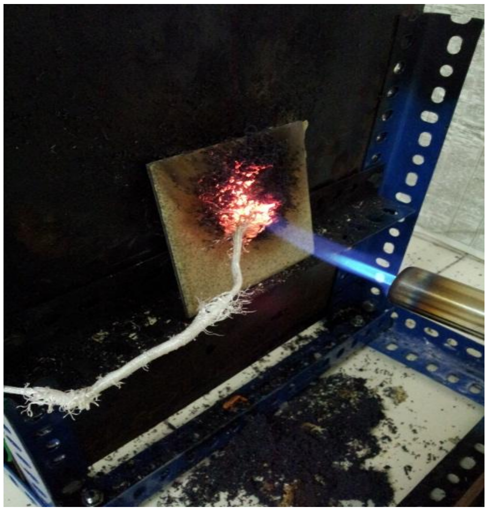


Figure 3-4: Heat shielding test

3.5 Static Immersion Test

Static immersion test is a standard method to evaluate water resistance of coatings based on gravimetric method. In this test, samples were immersed in distilled water at room temperature and weighed every 24 h. The samples are then dried at 40°C for 1 h in a furnace oven. The weight change of the samples were calculated using the equation:

$$\Delta W = \frac{W_2 - W_1}{W_1} \times 100\% \qquad (3\text{-}1)$$

ΔW	weight loss, %;
W_1	sample weight before water immersion, g;
W_2	sample weight after water immersion, g.

Equation 3-1: Weight loss equation

The resulting percentage of weight loss was expressed as a function of time to study the water resistivity of the coatings.

Figure 3-5: Samples immersed in distilled water

Figure 3-6: Samples drying in oven

3.6 Field Emission Scanning Electron Microscope (FESEM)

Char samples for FESEM analysis were prepared by heating coating samples at temperature of 450°C for 30 min in a furnace oven. The char samples are then examined using JSM-7100F FE-SEM machine.

3.7 Gantt Chart

Table 3-2: FYP I & II Gantt chart

Activity	**FYP 1**				**FYP 2**			
	May	**June**	**July**	**Aug**	**Sept**	**Oct**	**Nov**	**Dec**
Research on the topic assigned	🟥	🟥	🟥	🟥				
Interim report and oral presentation		🟥	🟥					
Preparation of samples				🟥				
Furnace test					🟥	🟥	🟥	
Static immersion test							🟥	
Heat shielding test							🟥	
FESEM								🟥
Report writing						🟥	🟥	🟥

Table 3-3: FYP I & FYP II key milestones

Activity	**FYP 1**				**FYP 2**			
	May	**June**	**July**	**Aug**	**Sept**	**Oct**	**Nov**	**Dec**
Develop Intumescent Coating				🟦				
Submission of Interim Report				🟦				
Completion of Furnace test							🟦	🟦
Completion of Static Immersion Test								🟦
Completion of Heat Shielding								🟦
Report Submission								🟦

3.8 Tools

Equipments:

- Bunsen burner
- Thermal logging device
- Furnace oven
- Digital weighing scale
- Shear mixer
- Shear cutter
- Grinding machine
- Weighing scale

Materials:

- Steel
- Expandable graphite (EG)
- Melamine (MEL)
- Zinc Borates (ZB)
- Ammonium Polyphosphate (APP)
- Epoxy Bisphenol A (BPA)
- Triethylene-tetramine resin (TETA)
- Distilled water

Inorganic filler – Talc and Alumina

CHAPTER 4
RESULTS AND DISCUSSIONS

4.1 Furnace Test

The maximum temperature of the furnace was set at 450°C as steels physical strength decreases as the temperature increases. The strength of steel decreases rapidly at temperature above 400°C therefore the critical temperature at which the material fail lies between 400 and 500°C. Table 2 summarizes the result of char expansion for all samples tested.

Table 4-1: Samples formulation

Sample	Coating Thickness (mm)	Final Thickness (mm)	Expansion Percentage (%)
No Filler (NF)	1.50	17.00	1033.33
Talc 1% (T1)	1.35	15.50	1048.15
Talc 2% (T2)	1.05	18.20	1633.33
Talc 3% (T3)	1.35	17.40	1188.89
Talc 4% (T4)	1.20	18.02	1401.67
Talc 5% (T5)	1.24	17.26	1291.94
Alumina 1% (A1)	1.38	17.50	1168.12
Alumina 2% (A2)	1.31	18.69	1326.72
Alumina 3% (A3)	1.15	22.71	1874.78
Alumina 4% (A4)	1.59	23.50	1377.99
Alumina 5% (A5)	1.37	19.20	1301.46

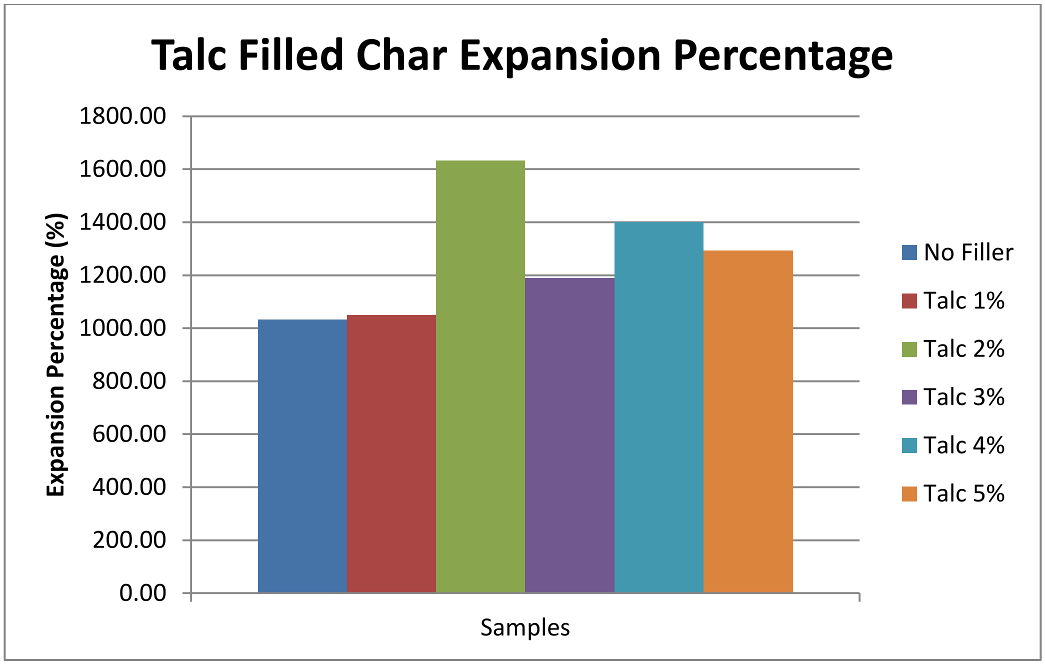


Figure 4-1: Talc filled char expansion chart

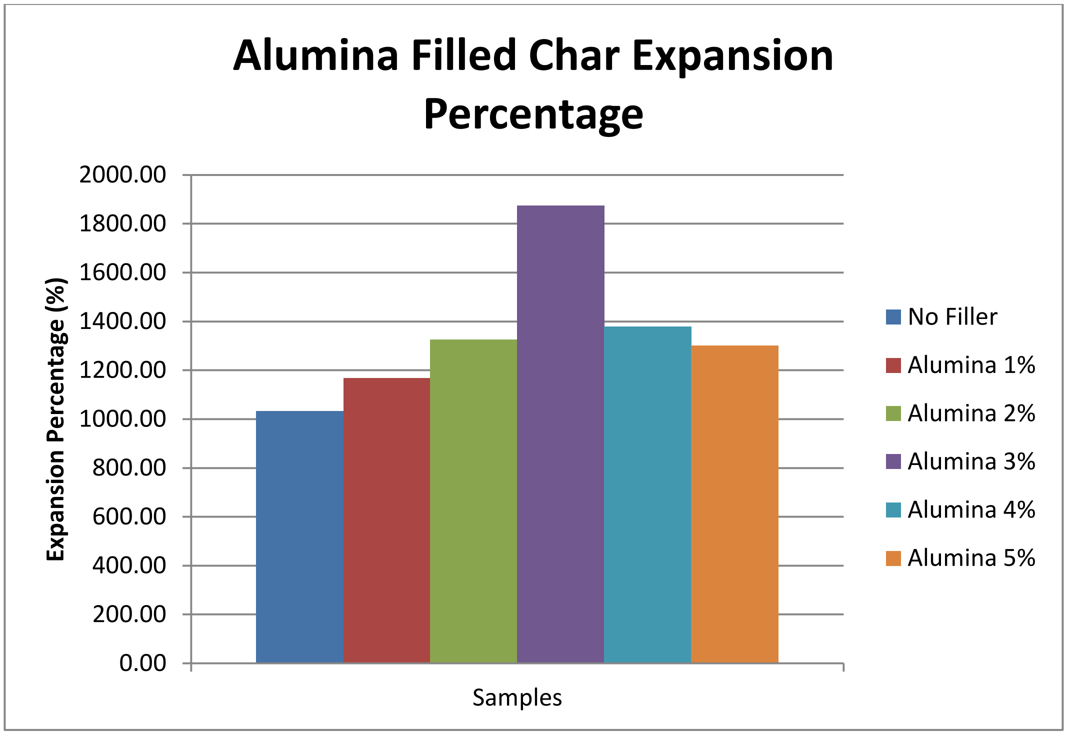


Figure 4-2: Alumina filled char expansion chart

4.2 Heat Shielding Effect

In this study, 11 samples with different compositions of intumescent ingredients were tested. The result after testing shows that formulation A5 which contains 5% of alumina fillers gives the best intumescent effect.

The temperature time curves and data for the fire test are illustrated in figure 4-3 and 4-4. The formulation with no filler has the highest temperature reached of 373.2°C. For talc filled intumescent coatings, the formulation 3% percentage has the lowest maximum backside substrate temperature of 163.6°C and for alumina, 5% alumina filled coating has the lowest maximum backside substrate temperature of 154.6°C. From the result, formulation A5 gives the best intumescent effect as it contains the highest percentage of alumina. Higher percentage of alumina gives better strength to the char formation thus giving the better intumescent effect. Alumina have outstanding physical ability such as high mechanical strength at room temperature and high melting point of 2050°C [18] hence higher percentage of alumina presence would enhance the heat shielding capability of the coating.

3% filled talc intumescent coating produces the best intumescent effect after A5 formulation. Presence of talc reinforces the mechanical stability of the intumescent protective shield [15]. A study conducted by Almeras et al. on the effect of talc fillers suggested a chemical reactivity between talc and intumescent ingredients that may affect the performance of the formulation [19]. The study demonstrates that the ratio percentage of talc and intumescent ingredients must be optimized to produce the best intumescent effect. This explains why 3% filled talc performed better than the higher filled talc formulation.

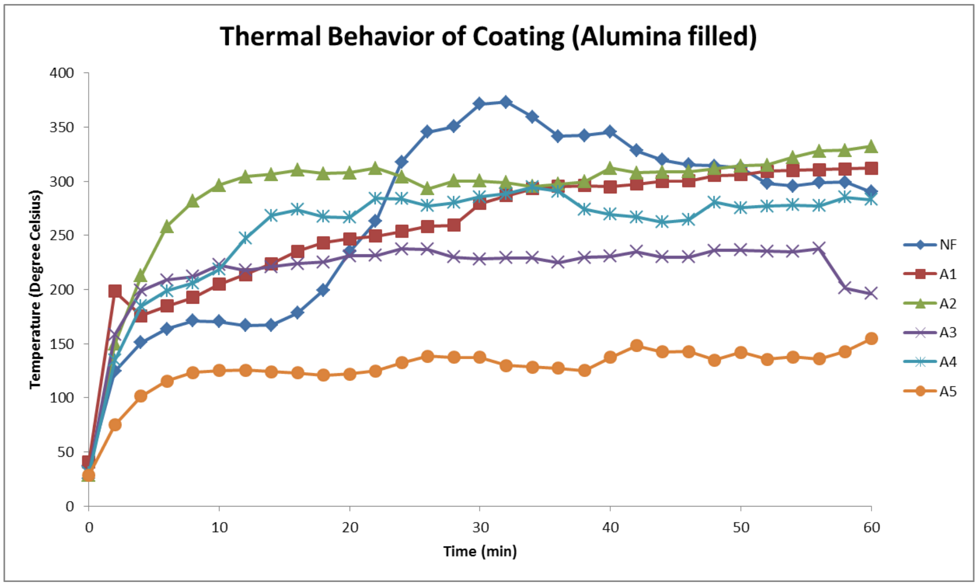


Figure 4-3: Thermal behavior of alumina filled coating

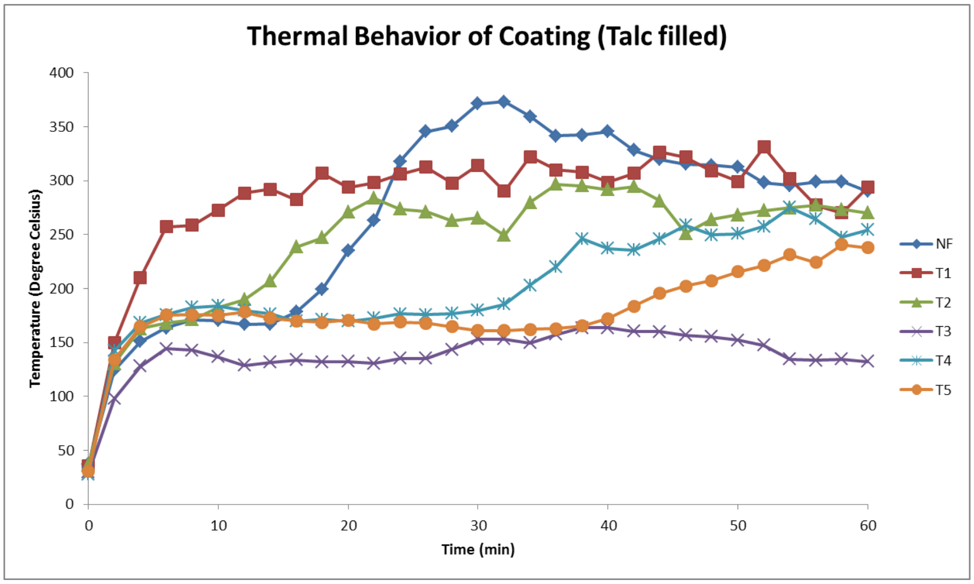


Figure 4-4: Thermal behavior of talc filled coating

4.3 Static Immersion Test

Weight change – time curves of all formulations are shown in Figure 4-5 and 4-6. Looking at the graph of alumina filled coating, weight change percentage of all the coating has increased dramatically from the first day until the fourth day of immersion where the percentage declined slightly. Equilibrium is not achieved in the initial stage of immersion therefore coatings continue to gain weight [20]. The increased weight change percentage is due to the absorption of water by the coating and the slight declination is due to the weight loss of the coating from formation of pores and damages to the surface [17]. The formation of pores allowed more water to be absorbed and the trend continue to increase until it reached physical and chemical equilibrium. Formulation A2 shows the highest water absorption rate of 0.167% and formulation NF has the lowest rate of absorption of 0.032%.

Based on the weight change time curves of talc filled intumescent coating, the trend of the curves are almost similar with alumina filled coating but with more settled curves. Formulation T1 has the highest water absorption of 0.170%. The talc filled intumescent coating does not produce pores and surface defects as alumina filled coating thus reaching physical and chemical equilibrium after 48 hours of testing.

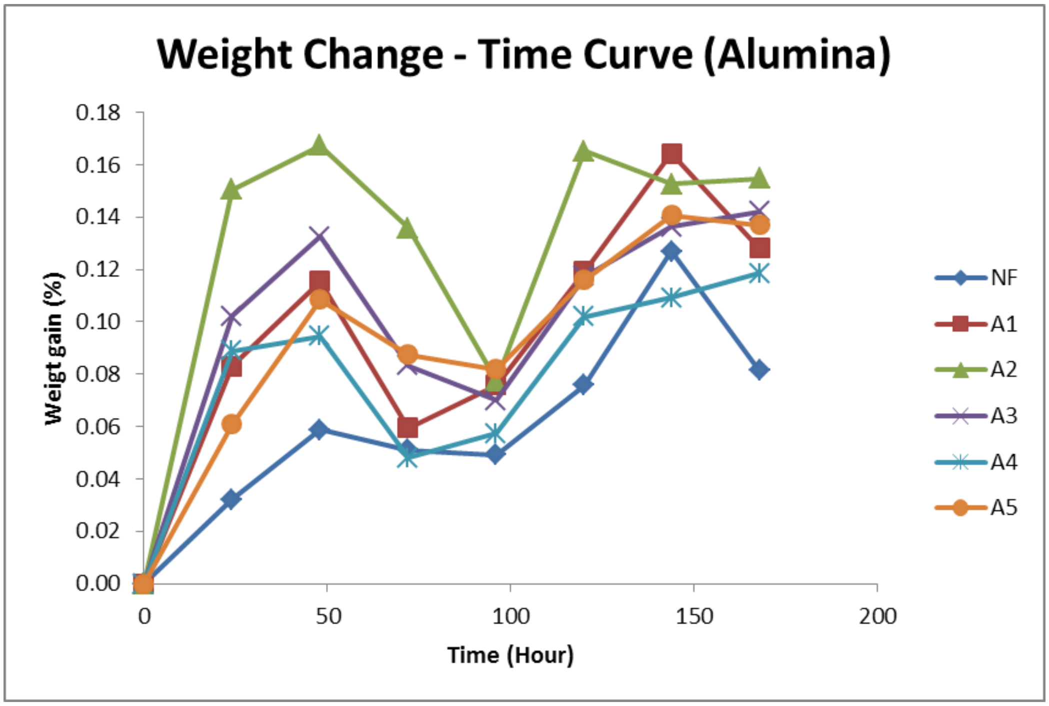


Figure 4-5: Weight change time curves of alumina filled coating

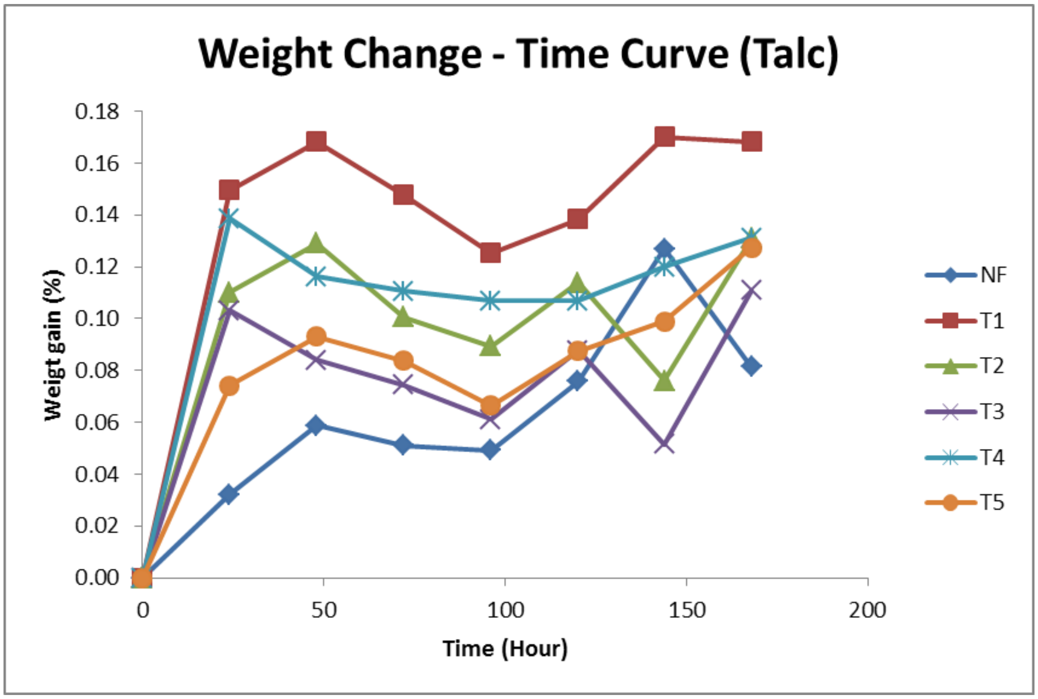


Figure 4-6: Weight change time curve of talc filled coating

4.4 Field Emission Scanning Electron Microscopy

Figure 4-7, 4-8 and 4-10 shows the morphology of char after burning at 450°C in a furnace oven. From Fig 4-7(b) no voids were observed due to the absence of filler which subsequently reduce the strength of the char. For both alumina and talc filled coating, voids can be observed in Fig 4-8(b) and 4-9(b). Voids were formed from evolution of trapped gas by blowing agent when the coating is subjected to fire. The voids act as a thermal barrier which prevent heat transmission to the substrate [21]. Voids also prevent diffusion of gaseous degradation products to the combustion zone and prevent oxygen diffusion to the surface polymer [21]. Fillers were believed to improve the efficiency of intumescent coating. This is down to the plate like microstructure of the fillers which consequently provide stabilizing effect on the cell structure of char foam [22].

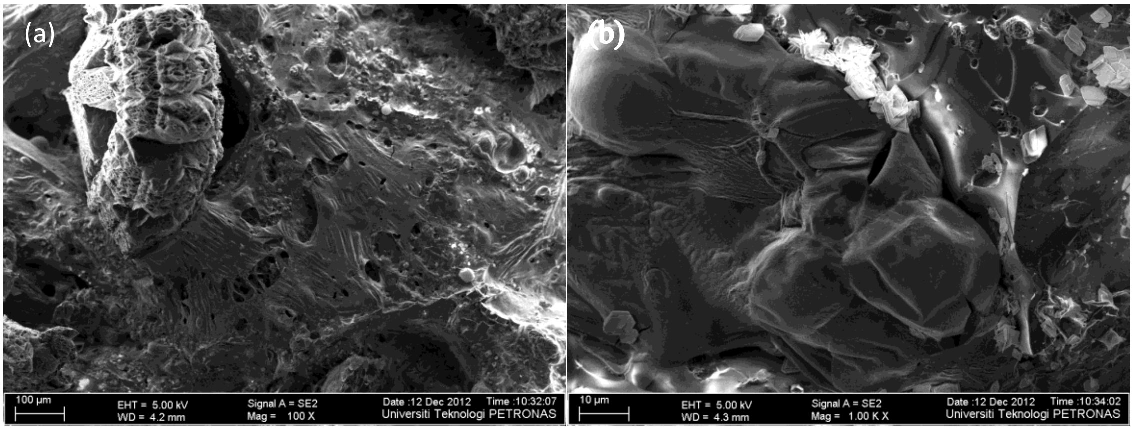


Figure 4-7: SEM micrographs (a) NF 450°C char at 100X mag (b) NF 450°C char at 1000X mag

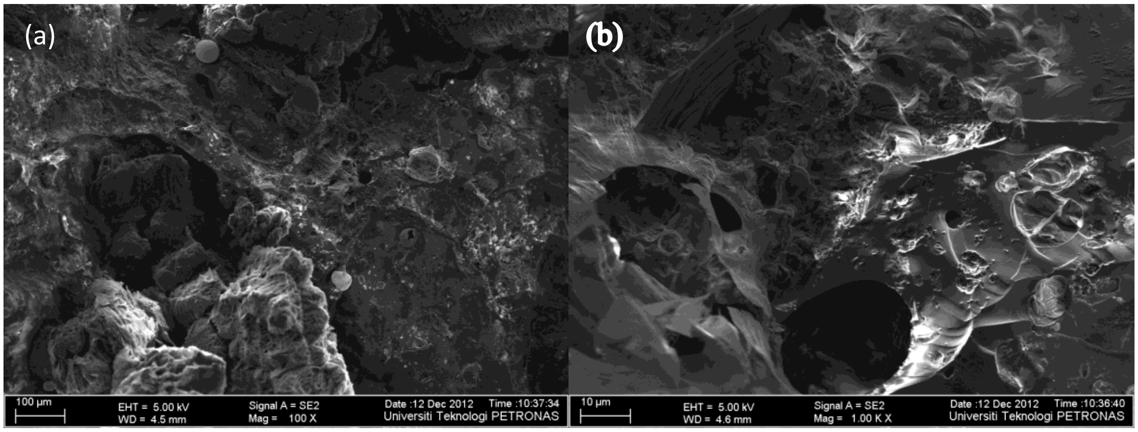


Figure 4-8: SEM micrographs (a) A5 450°C char at 100X mag (b) A5 450°C char at 1000X mag

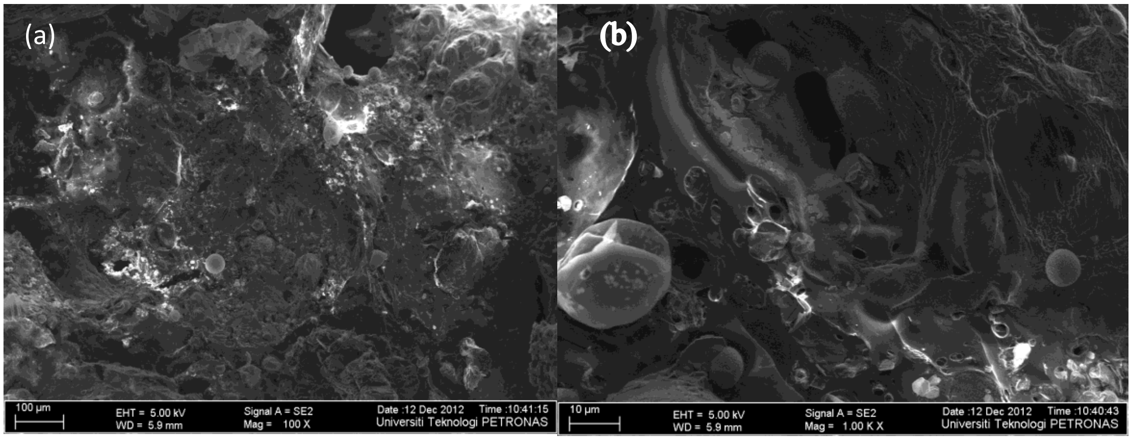


Figure 4-9: SEM micrographs (a) T5 450°C char at 100X mag (b) T5 450°C char at 1000X mag

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The study was taken with objectives to develop mineral based intumescent fire retardant coating and study the thermal performance. Based on the results and analysis done, it can be concluded that the objective of the study was achieved.

From overall result, incorporating alumina and talc into coating formulation showed good thermal properties. The higher expansion of char and better intumescent effect indicate that good thermal performance has been achieved. The water resistance of coating does not improve with added alumina filler but showed significant improvement due to talc insolubility in water.

5.2 Recommendation

Several improvements can be made for future project work. Fire test for after immersed intumescent coating can be performed to study the performance of the coating after subjected to the damage by water. The duration of the immersion test also can be extended to observe further the damage afflicted by water to the coating. Thermogravimetric analysis can be performed to study the changes in physical and chemical properties of the coating.

APPENDIX A

Heat Shielding Data for Talc Filled Coating

Time (min)\Temperature (Deg C)	NF	T1	T2	T3	T4	T5
0	36.9	35.5	36.6	29.7	28.0	30.6
2	124.4	149.6	130.4	97.9	142.3	134.0
4	150.9	210.1	162.6	127.9	168.1	164.4
6	163.3	257.3	167.7	144.2	175.8	175.4
8	170.9	258.6	170.9	142.5	182.6	175.6
10	170.3	272.4	182.1	136.5	183.8	175.1
12	166.8	288.4	189.6	128.6	179.5	178.3
14	167.2	291.8	206.8	131.5	176.3	172.6
16	178.1	282.2	238.4	133.7	169.7	169.8
18	199.5	306.6	247.2	132.0	171.2	168.5
20	234.8	293.6	270.6	132.4	169.6	170.7
22	263.0	298.1	283.8	130.6	172.3	166.9
24	317.7	305.9	273.8	135.2	176.4	168.8
26	345.5	312.3	271.1	135.3	175.6	167.8
28	350.4	297.5	262.9	143.4	176.6	164.7
30	371.3	314.0	265.5	153.1	179.6	160.8
32	373.2	290.2	249.2	152.9	185.5	161.1
34	359.4	322.1	279.6	149.4	202.9	162.1
36	341.4	309.8	296.7	157.4	220.0	162.9
38	342.0	307.7	295.3	163.3	246.0	165.3
40	345.5	298.3	291.6	163.6	236.9	172.2
42	328.0	306.6	294.4	160.4	235.7	183.4
44	319.6	326.2	281.3	159.8	246.0	195.3
46	315.1	321.7	250.9	156.7	258.6	202.3
48	314.3	308.8	263.9	155.2	249.6	207.2
50	312.4	299.0	268.2	152.1	250.7	215.6
52	298.1	330.9	272.3	147.2	257.6	221.1
54	295.4	301.7	274.8	134.4	275.1	231.2
56	298.9	277.2	277.1	133.4	264.2	224.0
58	299.2	270.1	273.2	134.5	247.3	240.7
60	290.0	293.9	270.1	132.4	254.2	237.7

Heat Shielding Data for Alumina Filled Coating

Time (min)\Temperature (Deg C)	NF	A1	A2	A3	A4	A5
0	36.9	40.8	28.1	30.8	30.2	28.6
2	124.4	198.4	149.4	158.0	134.4	75.0
4	150.9	175.6	212.6	198.9	184.7	101.3
6	163.3	184.6	258.2	208.6	198.7	115.3
8	170.9	192.7	281.6	212.0	205.8	123.4
10	170.3	204.6	296.1	222.6	218.2	124.9
12	166.8	213.8	304.3	217.8	247.1	125.6
14	167.2	223.9	306.2	220.9	268.4	123.9
16	178.1	235.2	310.1	223.9	273.7	122.9
18	199.5	243.3	307.0	225.2	267.3	120.9
20	234.8	246.7	307.8	230.9	266.6	121.8
22	263.0	249.3	312.1	231.2	284.2	124.7
24	317.7	253.6	304.0	237.3	283.7	132.4
26	345.5	258.3	293.0	236.9	277.3	138.3
28	350.4	259.3	300.3	230.0	280.0	137.3
30	371.3	279.4	300.3	227.9	285.4	137.2
32	373.2	286.6	298.7	229.2	288.2	129.7
34	359.4	293.1	295.2	229.2	294.4	128.2
36	341.4	295.2	297.3	225.0	290.4	127.4
38	342.0	295.8	299.6	229.5	274.1	125.2
40	345.5	294.8	311.9	230.7	269.3	137.4
42	328.0	297.3	307.8	234.9	266.9	147.9
44	319.6	299.7	308.3	230.0	262.2	142.2
46	315.1	300.3	308.7	229.9	264.4	142.6
48	314.3	305.1	311.6	235.8	280.4	134.8
50	312.4	306.0	314.2	236.2	275.5	141.8
52	298.1	309.1	314.8	235.1	276.8	135.6
54	295.4	310.0	321.9	234.9	277.9	137.8
56	298.9	310.5	327.7	237.7	277.3	136.0
58	299.2	311.2	328.4	201.2	285.2	142.8
60	290.0	312.2	332.0	196.0	282.8	154.6

Static Immersion Test Data Alumina Filled Coating

NF/time(h)	24	48	72	96	120	144	168
Wi (g)	52.743	52.731	52.742	52.727	52.734	52.732	52.736
Wf(g)	52.760	52.762	52.769	52.753	52.774	52.799	52.779
W%	0.032	0.059	0.051	0.049	0.076	0.127	0.082
A1/time(h)	24	48	72	96	120	144	168
Wi (g)	55.404	55.390	55.415	55.387	55.389	55.382	55.394
Wf(g)	55.450	55.454	55.448	55.429	55.455	55.473	55.465
W%	0.083	0.116	0.060	0.076	0.119	0.164	0.128
A2/time(h)	24	48	72	96	120	144	168
Wi (g)	47.824	47.812	47.825	47.810	47.818	47.818	47.819
Wf(g)	47.896	47.892	47.890	47.847	47.897	47.891	47.893
W%	0.151	0.167	0.136	0.077	0.165	0.153	0.155
A3/time(h)	24	48	72	96	120	144	168
Wi (g)	52.808	52.800	52.807	52.796	52.803	52.803	52.803
Wf(g)	52.862	52.870	52.851	52.833	52.865	52.875	52.878
W%	0.102	0.133	0.083	0.070	0.117	0.136	0.142
A4/time(h)	24	48	72	96	120	144	168
Wi (g)	53.967	53.964	53.985	53.961	53.967	53.968	53.969
Wf(g)	54.015	54.015	54.011	53.992	54.022	54.027	54.033
W%	0.089	0.095	0.048	0.057	0.102	0.109	0.119
A5/time(h)	24	48	72	96	120	144	168
Wi (g)	52.573	52.560	52.576	52.554	52.568	52.566	52.569
Wf(g)	52.605	52.617	52.622	52.597	52.629	52.640	52.641
W%	0.061	0.108	0.087	0.082	0.116	0.141	0.137

Static Immersion Test Data for Talc Filled Coating

T1/time(h)	24	48	72	96	120	144	168
Wi (g)	53.484	53.474	53.477	53.470	53.478	53.478	53.481
Wf(g)	53.564	53.564	53.556	53.537	53.552	53.569	53.571
W%	0.150	0.168	0.148	0.125	0.138	0.170	0.168
T2/time(h)	24	48	72	96	120	144	168
Wi (g)	52.666	52.654	52.669	52.651	52.664	52.663	52.662
Wf(g)	52.724	52.722	52.722	52.698	52.724	52.703	52.731
W%	0.110	0.129	0.101	0.089	0.114	0.076	0.131
T3/time(h)	24	48	72	96	120	144	168
Wi (g)	52.371	52.370	52.376	52.365	52.372	52.372	52.372
Wf(g)	52.425	52.414	52.415	52.397	52.418	52.399	52.430
W%	0.103	0.084	0.074	0.061	0.088	0.052	0.111
T4/time(h)	24	48	72	96	120	144	168
Wi (g)	53.341	53.338	53.341	53.323	53.334	53.338	53.334
Wf(g)	53.415	53.400	53.400	53.380	53.391	53.402	53.404
W%	0.139	0.116	0.111	0.107	0.107	0.120	0.131
T5/time(h)	24	48	72	96	120	144	168
Wi (g)	52.603	52.592	52.595	52.581	52.590	52.593	52.593
Wf(g)	52.642	52.641	52.639	52.616	52.636	52.645	52.660
W%	0.074	0.093	0.084	0.067	0.087	0.099	0.127

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