STUDY OF WEATHER RESISTANCE OF INORGANIC FILLERS BASED INTUMESCENT COATING

BY

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10937

Dissertation submitted in partial fulfillment of

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(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

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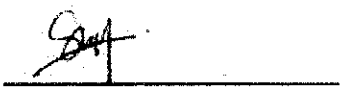
TRONOH, PERAK

September 2011

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.



NUR SYAFIQAH BINTI MD RAZIB

ABSTRACT

Intumescent is a substance which swells when it is exposed to heat. The use of intumescent coating is one of the easiest and efficient ways to protect the substrate against fire. It has been used in firestopping, fireproofing and gasketing applications as well as closure components. By insulating or coating the steel, the rate of heat transfer can be reduced. It will extend the time taken to reach the structural failure temperature and 'buying time' for the evacuation of persons. To improve the performance of intumescent coating, fillers have been added in the coating. The objective of this study is to produce an intumescent coating formulation with inorganic fillers to get the optimal performance in char formation. Apart from that, the other objectives is to study the weather resistance of inorganic filled intumescent coating since the performance of intumescent coating may be deteriorated because of weathering issues. Generally known ultraviolet light, heat, moisture (humidity) and pollution will contribute to the degradation of the material and this will affect the performance of the coating. The study will be focusing on improving the intumescent coating by adding the inorganic fillers. The organic fillers which will be used are fumed silica and Alumina Trihydrate (ATH). Fumed silica has the ability to act as the thermal insulator and as for ATH, it is an effective flame retardant due to its thermodynamic properties. This study will also include the performance of intumescent coating in terms of weather resistance. The performance will be evaluated based on the expansion of char and physical properties of intumescent coatings.

ACKNOWLEDGEMENT

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

My deepest appreciation and gratitude is extended to my supervisor, AP Dr. Faiz Ahmad for all his teachings, guidance, supervision and supports from the preliminary to the final report enable me to develop an understanding of the subject. It has been a hardship for you, sorry and thank you so much.

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LIST OF ABBREVIATIONS

APP	Ammonium Polyphosphate
ATH	Alumina Trihydrate
ATR	Attenuated Total Reflectance
EG	Expandable Graphite
FTIR	Fourier Transform Infrared
MEL	Melamine
PER	Pentaerythritol
PFP	Passive Fire Protection
UK	United Kingdom
UV	Ultraviolet

CHAPTER 1

INTRODUCTION

1.1 Background Of Study

Steel is a material usually used in the construction of bridges, building, boats, cars, and also offshore platforms. Steel is a non-combustible material or in other words it does not burn and it exhibits a good ductility but it does have a serious weakness as it begins to lose its structural strength at a high temperature [1]. Because of this advantage, the protection of metallic materials against fire has become an important issue in the construction industry.

The fire protection industry has divided the methods of fire protection into two big categories which are active fire protection and passive fire protection. The active fire protection uses systems like deluge, sprinklers, inert gases etc. While the passive fire protection uses insulation or coating. This kind of protection does not attack the fire itself but insulate the substrate from the heat effects of the fire *[2]*.

In this study we will only be focusing on the passive fire protection which is widely practiced by using intumescent coating. Intumescent is a substance which swells when it is exposed to heat. The use of intumescent coating is one of the easiest and efficient ways to protect the substrate against fire. It has been used in firestopping, fireproofing and gasketing applications as well as closure components [3]. By insulating or coating the steel, the rate of heat transfer can be reduced. It will extend the time taken to reach the structural failure temperature and 'buying time' for the evacuation of persons [2].

Intumescent has several advantages as it does not modify the mechanical properties of the material. It is easily processed and may also be used onto several materials including metallic materials, polymers, textiles and woods. Other advantages of intumescent are it has resistance to blast or explosion and to pressurized inventory fires (jet fire), it has

very high levels of long term durability, it is light in weight and low film thickness and also it does not contain water so resistant to freeze or thaw deterioration [2].

1.2 Problem Statement

Addition of additives to the intumescent coating formulation may be seen as the best solution to improve its performance to protect from weather. However there are concerns that the performance of intumescent coating may be deteriorated because of weathering issues. Ultraviolet light, heat, moisture (humidity) and pollution will contribute to the degradation of the material and this will affect the performance of the coating.

1.3 Objectives

1) To produce an intumescent coating formulation with inorganic fillers (ATH or fumed silica) to get the optimal performance in char formation.
2) To study the weather resistance (effect of humidity) of inorganic filled intumescent coating.

1.4 Scope Of Study

The study was mainly focusing on improving the intumescent coating by adding the inorganic fillers. The organic fillers used were Fumed Silica and Alumina Trihydrate (ATH). Fumed silica has the ability to act as the thermal insulator and as for ATH, it is an effective flame retardant due to its thermodynamic properties. This study also includes the performance of intumescent coating in terms of weather resistance. The performance was evaluated based on the expansion of char and also the physical properties of the intumescent coatings.

1.5 Significance Of Study

The use of inorganic fillers can improve the performance in intumescent coating as it improves the mechanical properties of the coating. This includes the performance in fire and smoke as the inorganic fillers do not send out organic solvent in application and have little toxic gas emissions and smoke output on heating.

1.6 Feasibility Of Project

This project required some experimental works in producing the intumescent coating with inorganic fillers and to study its performance especially in terms of weather resistance. This project was done within the allocated time given and everything went fine as planned. All of the objectives were achieved since all procedures were followed accordingly.

CHAPTER 2

LITERATURE REVIEW

2.1 Intumescent Coating

Intumescent coatings are designed to perform under severe conditions and to maintain the steel integrity for one up to three hours when the temperature of the surroundings is in excess of 1100 ℃ [1]. Intumescent is defined as the swelling of substances when they are heated. Intumescent coatings form on heating an expanded multicellular layer, which acts as a thermal barrier that effectively protects the substrate against rapid increase of temperature, thereby maintaining the structural integrity of the building [4].

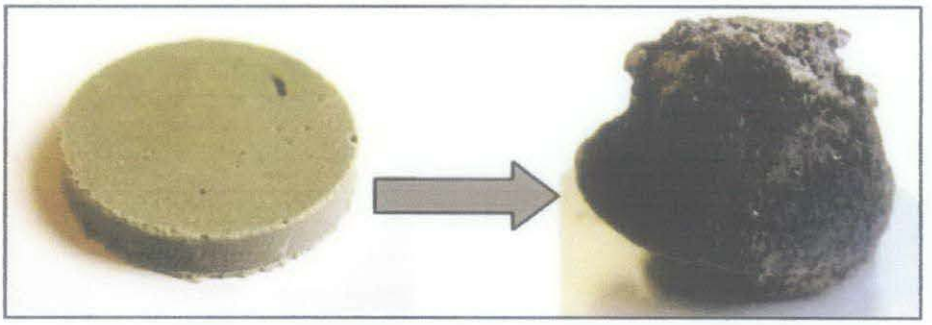


Figure 1: Swelling of an intumescent coating

2.2 Mechanisms of Intumescent

Intumescent concept allows a balance between the fire properties and the level of additives in the material. The mechanism of intumescent is usually started with the acid source breaks down to yield a mineral acid, then it takes part in the dehydration of the carbonization agent to yield the carbon char, and finally the blowing agent decomposes to yield gaseous products. Then, the char will swell and this will provide an insulating multi-cellular protective layer. This shield limits at the same time the heat transfer from

the heat source to the substrate and the mass transfer from the substrate to the heat source resulting in a conservation of the underlying material [1].

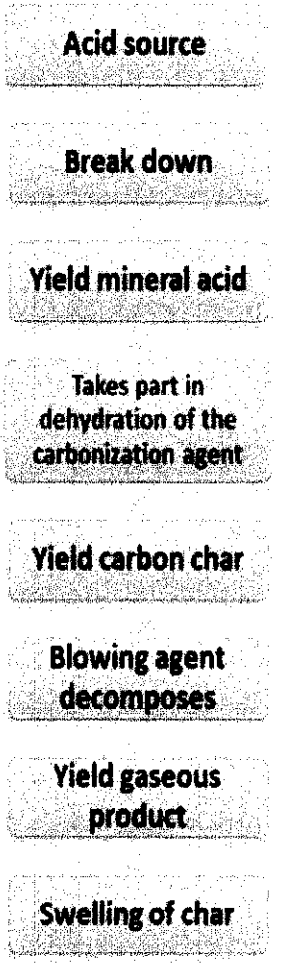


Figure 2: Mechanisms of intumescent

Intumescent coatings basically contain ingredients which are bound together by a binder. Generally, three main ingredients are used: an acid source (normally ammonium polyphosphate, APP), a carbon source (such as pentaerythritol, PER) and a blowing agent (e.g. melamine, MEL) [4]. During the intumescent process, the binder became important due to two effects which it contributed to the char layer expansion and

ensured the formation of uniform foam structure. However the fire retardant additives (APP and PER) in the coatings were very sensitive to corrosive substances, such as water, acid and alkali. They could easily migrate to the surface of the coatings in corrosive environment. This would significantly depress the expected effect of the intumescent coatings. The binder could prevent or remarkably reduce migration of fire retardant additives and access of the corrosive substances [5].

2.3 Inorganic Fillers

To improve the performance of intumescent coatings, inorganic fillers will be used in this project as they do not send out organic solvent in application and have little toxic gas emissions and smoke output on heating compared to the organic fillers even though they have good expanding effect [6]. Two types of inorganic fillers will be used: Alumina Trihydrate (ATH) and fumed silica. Alumina Trihydrate (ATH) is a non-toxic, non-corrosive, flame retardant and smoke suppressant. It is the most frequently used flame retardant in the world. ATH is a very effective flame retardant due to its thermodynamic properties which absorb heat and release water vapour. ATH releases its 35% water of crystallization as water vapour when heated above 205 °C. The resulting endothermic reaction cools the product below flash point, reducing the risk of fire and acts as a vapour barier to prevent oxygen from reaching the flame [7]. As for fumed silica it has potential to reduce heat release and burning rates [8]. Thus it reduces the flammability properties of intumescent coating. Both characteristics of inorganic fillers inside intumescent coating formula will be compared in terms of weather resistance.

2.4 Weather Resistance

However, there are also concerns that the performance of the intumescent coatings in a fire may be deteriorated because of weathering issues. Degradation may be provoked or accelerated, by conventional elements of the weather such as ultraviolet light, heat, moisture, and pollution [9]. According to passive fire protection (PFP) weathering

programme by Shell UK in 1987, the topcoat (coating) had an important role in determining the longevity of the bulk fire protective coating. Failure of the topcoat by erosion or cracking leads to ultra-violet (UV) attack of the bulk coating in the case of intumescents and erosion and water ingress into the materials [10]. Based on a result obtained after 10 years of natural exposure in Hendaye (south of France) it indeed appears that one of the main defects for the prepainted panels exposed at 5° or 45° South, is their blistering on flat faces[11]. A lot of little blisters start appearing on defective systems after about 6 years exposure. These blisters can degrade the overall appearances of panels as shown in figure below [11].

Figure 3: Example of blistering on a red sample exposed for 10 years on natural exposure in Hendaye.

The basic requirements for real-time weathering are direct and continuous exposure to the natural environment. For the organic coatings, it was considered that exposure could cause degradation of the coating by one or more of the mechanisms like water uptake into the coating, ionic uptake into the coating or breakdown of the coating resin or

pigments/additives releasing ionic species[10]. To successfully resist weathering, most fire protective coatings require [10]:

a) Proper preparation (cleaning, priming, key coat, etc.) of the substrate.
b) Closely controlled application within the specified environmental range (temperature, humidity) with as smooth a finish as possible.
c) A resilient coating suitable for exposed duty.
d) Proper treatment of edge features to prevent the interface becoming a corrosion site and, when necessary, to provide resistance to jet fires.
e) Adequate inspection, maintenance and repair.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

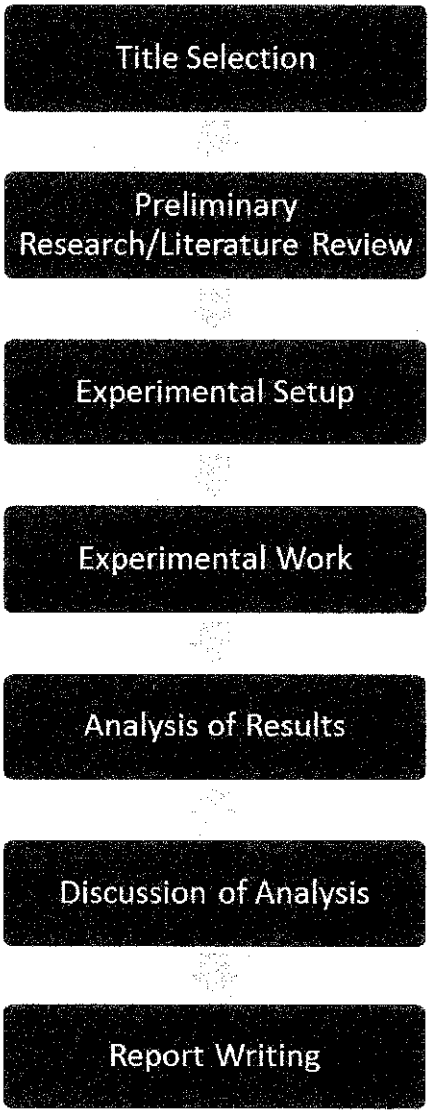


Figure 4: Flow Chart of the research methodology

3.2 Project Activities

Details of Flow Chart

i. **Title Selection** – An appropriate title was selected for Final Year Project.

ii. **Preliminary Research/Literature Review** – Studies on related journal on Intumescent Coating was conducted for better conceptual understanding.

iii. **Experimental Setup** – The formulation and materials of intumescent coating with inorganic fillers were prepared. The percentage of inorganic filler of Fumed Silica and ATH were determined.

iv. **Experimental Work** – Experimental works are conducted to get the results based on the initial properties and the variables set. The tests that were used are weathering test, furnace test and surface roughness test. Weathering chamber was used to see the weathering effects on the samples while the furnace test was run in the furnace and surface roughness tester was used for surface roughness test for this experiment.

v. **Analysis of Results** – Results such as discolouration of samples, volume (thickness) of samples, weight of samples, char height and the surface roughness of coatings were analysed.

vi. **Discussion of Analysis** – The results obtained by using two different inorganic fillers were compared. Recommendation was made from the results which meet the objectives.

vii. **Report Writing** – The final stage of the study was the compilation of all research findings, literature reviews, experimental works and outcomes into the final report.

3.3 Experimental Methodology

3.3.1 Samples Preparation

The intumescent ingredients were mixed with their weight percentage composition homogeneously using high shear mixer. The detailed formulation is (EG, MEL, BA, APP, epoxy, hardener and fillers) as described in the Table 1.

Sample	BPA (g)	TETA (g)	APP (g)	EG (g)	MEL (g)	BA (g)	Filler (ATH/FS), (g)
A	44.44	22.22	11.11	5.56	5.56	11.11	0
B	43.94	21.72	11.11	5.56	5.56	11.11	1 (FS)
C	43.44	21.22	11.11	5.56	5.56	11.11	2 (FS)
D	42.94	20.72	11.11	5.56	5.56	11.11	3 (FS)
E	42.44	20.22	11.11	5.56	5.56	11.11	4 (FS)
F	41.94	19.72	11.11	5.56	5.56	11.11	5 (FS)
G	43.94	22.22	11.11	5.56	5.56	11.11	1 (ATH)
H	43.44	21.72	11.11	5.56	5.56	11.11	2 (ATH)
I	42.94	21.22	11.11	5.56	5.56	11.11	3 (ATH)
J	42.44	20.72	11.11	5.56	5.56	11.11	4 (ATH)
K	41.94	19.72	11.11	5.56	5.56	11.11	5 (ATH)

Table 1: Formulation Mixing Ratio

The formulations were coated manually on the steel substrate. The coated substrates were cured at room temperature for two weeks. After the curing process, thickness and weight of the samples were measured using the vernier caliper for thickness and digital weighing scale for weight and recorded to get the value of the thickness and weight before weathering test. Surface roughness testing was also conducted using the surface roughness tester to get the roughness before the samples were placed in the weathering machine.

Figure 5: Sample of coated substrate

Figure 6: Weight of sample measured using Digital Weighing Scale

3.3.2 Weathering Test

The coated samples were placed in the weathering chamber to test the weathering effects. The samples were supposed to be tested for one month under the following conditions; the weather ability of specimens will be assessed in an accelerated weathering chamber according to JIS K5600-7-7 (xenon lamp method) for up to 720 hours. The accelerated weathering regime involves continuous exposure to xenon arc UV radiation (0.35 ± W/m² at 340 nm = 41.5 W/m² for 300-400 nm), 18 minutes light and water spray, 6 hours dark using 95% relative humidity (no water spray) with a black panel temperature of 65° ± 2 °C and a chamber temperature of 35 °C ± 2 °C. Since the available weathering chamber was having some problems, some changes were made on the conditions and the samples were only managed to be tested under the condition of 95% relative humidity with 45 minutes dark and 5 minutes of water spray and a chamber temperature of 30 °C ± 2 °C to give the humidity effect.

Figure 7: Samples in weathering chamber

The samples were placed in the weathering chamber for four weeks (28 days) with everyday check to make sure the machine was operating accordingly.

3.3.3 Discolouration

After four weeks, the samples were then taken out and colour change was observed by comparing the colour of the non-weathered coatings and weathered coatings. This was to see whether the weather could affect the colour of the coatings and to see the intensity of the discolouration.

Figure 8: Example of colour comparison between non-weathered and weathered coatings

3.3.4 Change in Thickness and Weight

Thickness and weight of the weathered coatings were also measured and recorded. The values that obtained were then compared with the previous data which had been taken before the coatings were placed in the weathering chamber to see how the weather had affected the coatings. The increment in thickness and weight were calculated as well. From weight data also, water absorption percentage was calculated to see how much water had been absorbed by the samples. The increment in thickness and weight described the permeability of the coatings.

3.3.5 Surface Roughness Test

After that, the weathered coatings were tested with surface roughness tester to see the roughness of the surface after the weathering test. The values obtained were compared

with the values from the previous surface roughness test which was conducted before the coatings were placed in the weathering chamber. Coating with higher roughness value had a rougher surface.

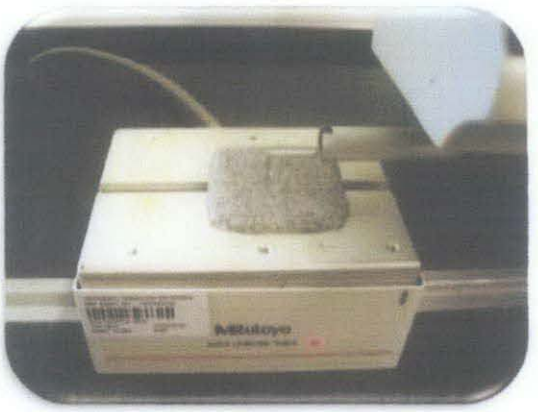


Figure 9: Surface roughness testing

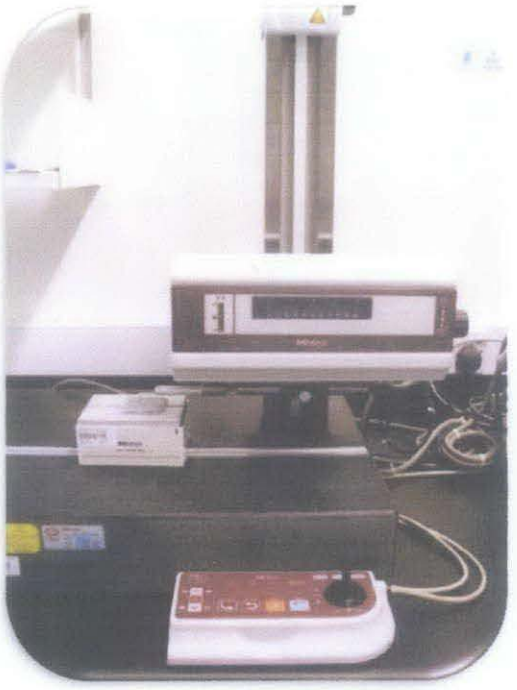


Figure 10: Surface roughness tester

3.3.6 Furnace Test

The last test was on char expansion. The samples were burnt in a furnace with a temperature of 500 °C for 20 minutes. The samples were then left to cool down until the temperature reached 30 °C before they were taken out from the furnace to see the expansion of the char. The char thickness was measured for each sample and the expansion was calculated using the initial thickness and the thickness after the samples were burnt to see the difference between char expansion of non-weathered coatings and weathered coatings.

Figure 11: Samples in furnace

Figure 12: Example of char expansion

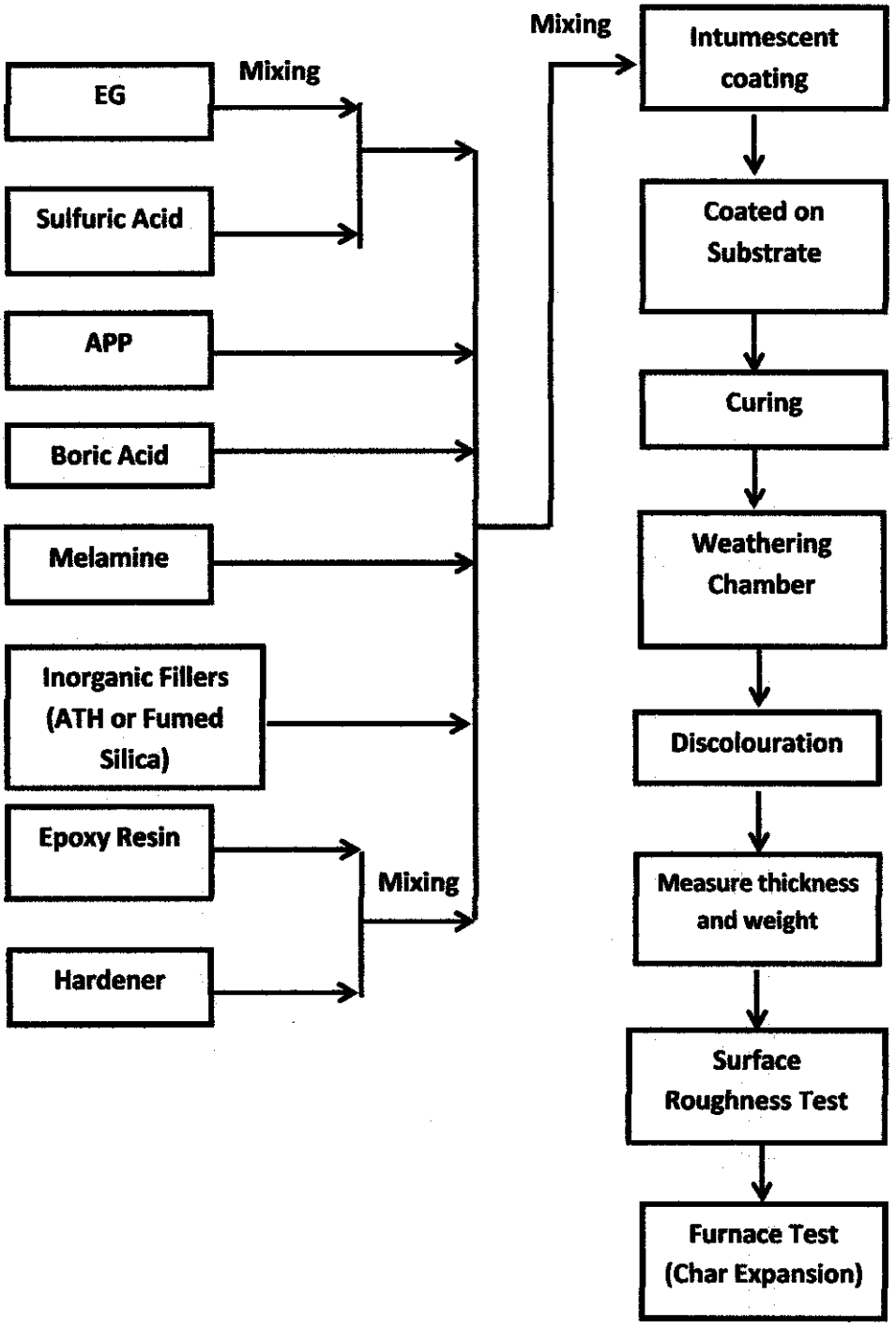


Figure 13: Experimental flow chart

3.4 Equipment and Tools

Materials:

- Stainless steel
- Expandable graphite (EG)
- Melamine (MEL)
- Boric Acid (BA)
- Ammonium Polyphosphate (APP)
- Epoxy Bisphenol A (BPA)
- Triethylene-tetramine resin (TETA)
- Inorganic filler - Alumina Trihydrate (ATH) and Fumed Silica

Equipments:

- Shear mixer
- Weathering chamber
- Vernier caliper
- Digital weighing scale
- Surface roughness tester
- Furnace oven
- Ruler

3.5 Gantt Chart

Activity	FYP 1				FYP 2				
	May	June	July	Aug	Sept	Oct	Nov	Dec	Jan
Early Stage of Documentation									
Studies on Intumescent Coating Formulation with inorganic fillers.									
Sample preparation.									
Expose to weathering chamber									
Record data of the samples									
Run Fire Test in Furnace.									
Analyses on different char expansion for each sample.									
Report documentation.									

Table 2: Gant Chart

3.6 Key Milestone

Activity	FYP 1				FYP 2				
	May	June	July	Aug	Sept	Oct	Nov	Dec	Jan
Determine the formulation of the Intumescent Coating.									
Completion of sample.									
Completion of exposing samples to outdoor surrounding and weathering chamber.									
Completion of Fire Test.									
Conclude The Analyses and report documentation									

Table 3: Key Milestone

CHAPTER 4

RESULT AND DISCUSSION

4.1 Discolouration

Sample	Discolouration (Colour Intensity)
A (0%)	Low
B (FS 1%)	Low
C (FS 2%)	Low
D (FS 3%)	Low
E (FS 4%)	Low
F (FS 5%)	Low
G (ATH 1%)	Low
H (ATH 2%)	Low
I (ATH 3%)	Low
J (ATH 4%)	Low
K (ATH 5%)	Low

Table 4: Discoloration of coatings

The change in colour for both Fumed Silica and ATH coatings were observed by comparing the colour of non-weathered with weathered samples. Although the change of colour was not that significant and the colour intensity was low, it had proved that weathering had affected the colour of the coatings. The findings are as in the Table 4 above.

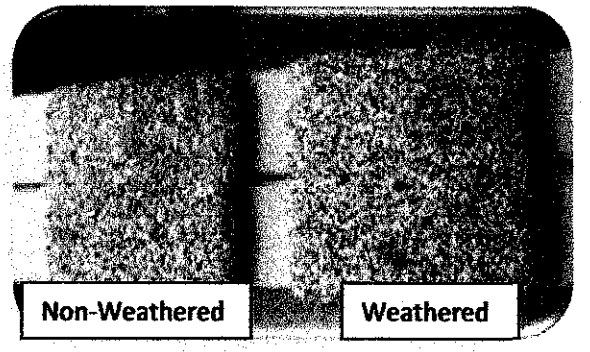


Figure 14: Discoloration for 3% of Fumed Silica coatings

4.2 Sample Volume (Thickness)

Thickness of the samples was measured to see the changes in thickness of the coatings. The Thickness before and after weathering test was measured using the vernier caliper. Average thickness of each sample was recorded in the table below.

Thickness (mm)				
Sample	**1**	**2**	**3**	**4**
A (0%)	4.71	4.64	4.77	4.81
B (FS 1%)	4.86	4.69	4.83	4.87
C (FS 2%)	5.32	5.12	4.95	4.98
D (FS 3%)	5.08	4.73	5.07	4.83
E (FS 4%)	5.55	5.41	5.48	4.96
F (FS 5%)	4.66	5.00	4.76	4.84
G (ATH 1%)	4.67	5.42	5.34	5.26
H (ATH 2%)	4.83	5.99	5.67	5.51
I (ATH 3%)	5.07	4.77	5.17	4.45
J (ATH 4%)	4.62	4.62	4.18	5.16
K (ATH 5%)	5.42	5.57	4.46	4.91

Table 5: Average thickness of samples (mm) before weathering test

The thickness of the samples after they had been taken out from the weathering chamber was measured again and the data was tabulated as shown in the table below.

Sample	Thickness (mm)				
	Duration (days)				
	0	7	14	21	28
A (0%)	4.71	4.73	4.78	4.80	4.82
B (FS 1%)	4.86	4.92	4.96	4.98	5.00
C (FS 2%)	5.12	5.14	5.18	5.20	5.22
D (FS 3%)	5.08	5.12	5.16	5.16	5.17
E (FS 4%)	5.55	5.60	5.66	5.69	5.71
F (FS 5%)	4.66	4.73	4.78	4.80	4.82
G (ATH 1%)	4.67	4.69	4.71	4.73	4.75
H (ATH 2%)	4.83	4.89	4.95	4.98	5.00
I (ATH 3%)	5.16	5.17	5.19	5.20	5.21
J (ATH 4%)	4.62	4.64	4.66	4.67	4.68
K (ATH 5%)	4.91	4.95	5.00	5.01	5.03

Table 6: Average thickness of samples (mm) after weathering test

The percentage of thickness increment was calculated using the equation below and the values were tabulated as shown in Table 7.

$$\frac{Thickness\ after - Thickness\ initial}{Thickness\ initial} \times 100$$

Sample	Thickness Increment Percentage (%)				
	Duration (days)				
	0	7	14	21	28
A (0%)	0	0.42	1.49	1.91	2.34
B (FS 1%)	0	1.23	2.06	2.47	2.88
C (FS 2%)	0	0.39	1.17	1.56	1.95
D (FS 3%)	0	0.79	1.57	1.57	1.77
E (FS 4%)	0	0.90	1.98	2.52	2.88
F (FS 5%)	0	1.50	2.58	3.00	3.43
G (ATH 1%)	0	0.43	0.86	1.28	1.71
H (ATH 2%)	0	1.24	2.48	3.11	3.52
I (ATH 3%)	0	0.19	0.58	0.78	0.97
J (ATH 4%)	0	0.43	0.87	1.08	1.30
K (ATH 5%)	0	0.81	1.83	2.04	2.44

Table 7: Thickness increment percentage (%)

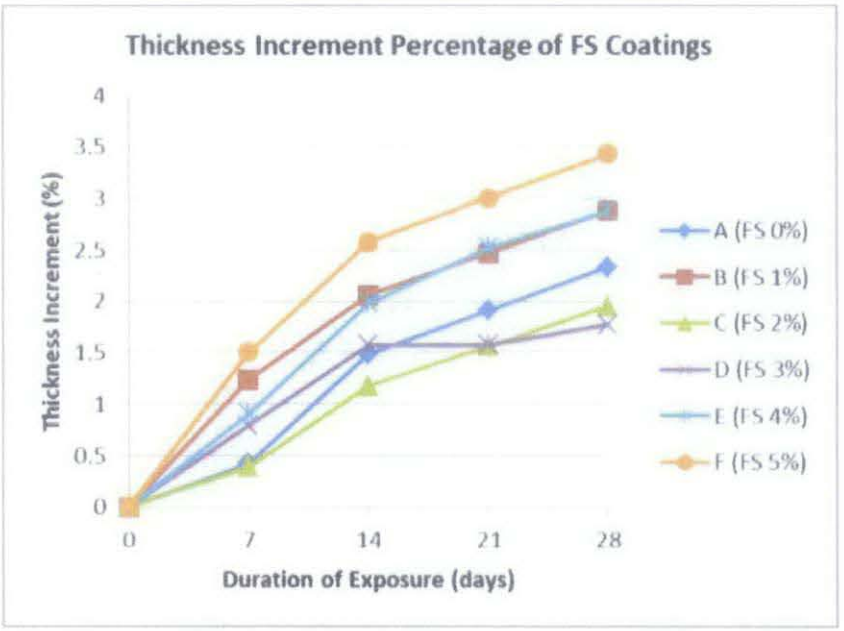


Figure 15: Thickness increment in percentage of Fumed Silica coatings vs. the duration of exposure in days

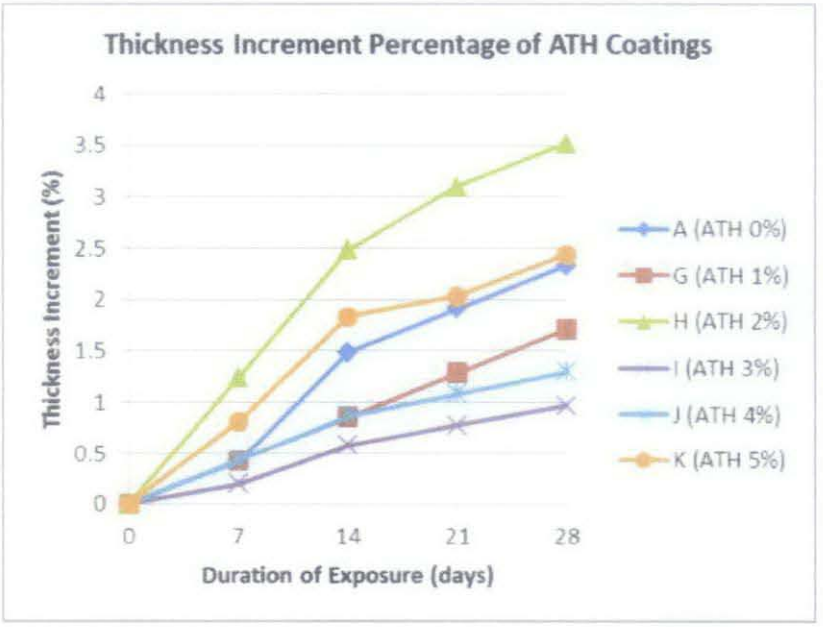


Figure 16: Thickness increment in percentage of ATH coatings vs. the duration of exposure in days

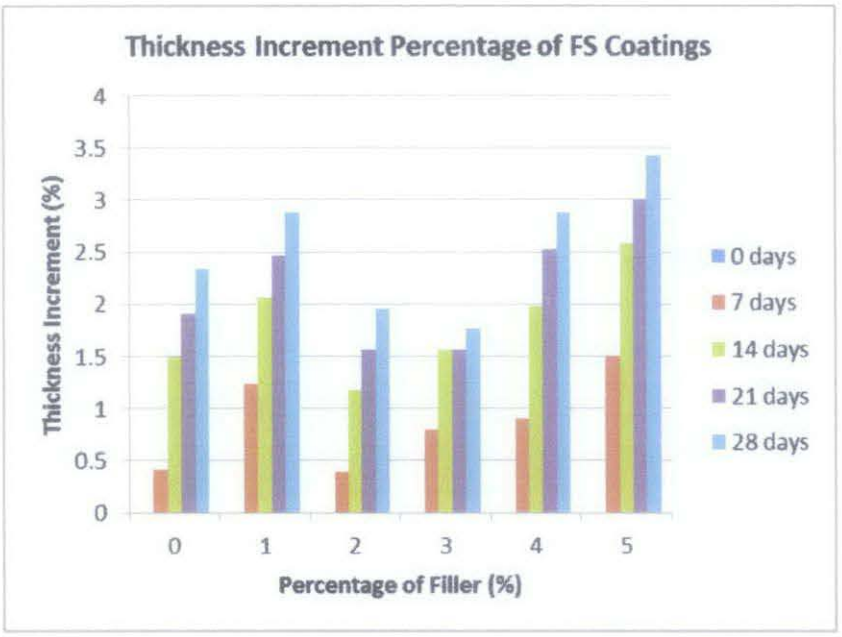


Figure 17: Thickness increment in percentage of Fumed Silica coatings vs. percentage of filler in each sample

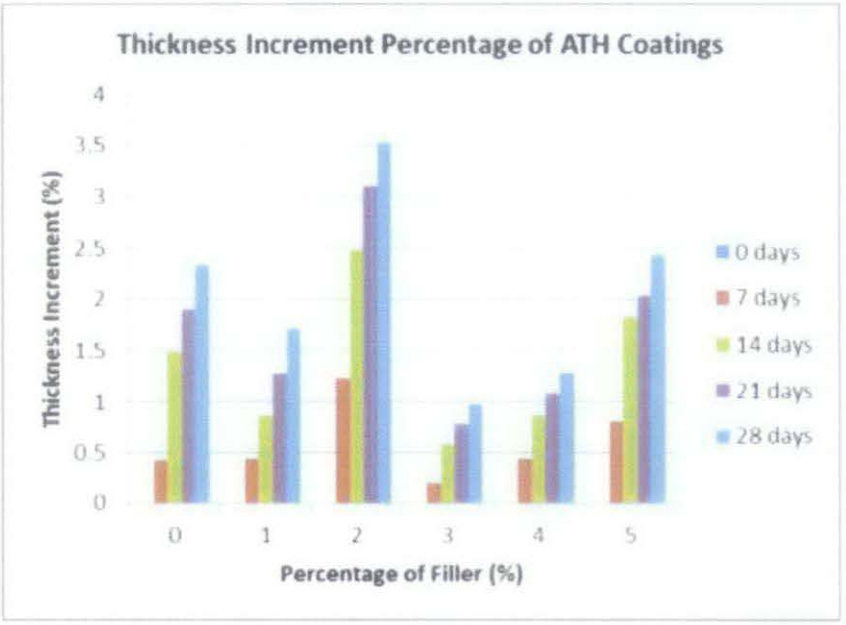


Figure 18: Thickness increment in percentage of ATH coatings vs. percentage of filler in each sample

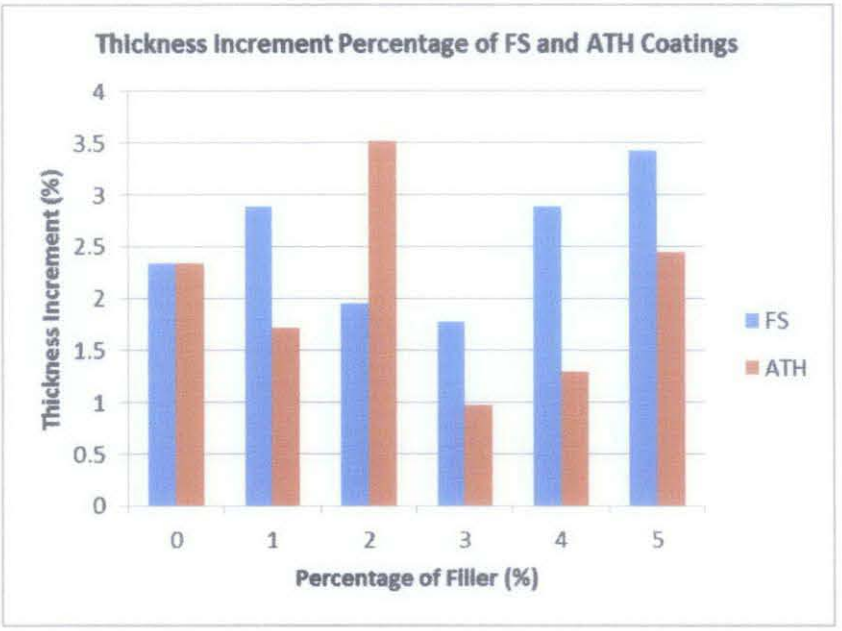


Figure 19: Thickness increment in percentage for both Fumed Silica and ATH coatings vs. the percentage of filler in each sample

Based on the graphs above (Figure 15 and Figure 16), the thickness of the samples is increasing gradually for both of Fumed Silica and ATH coatings. The thickness has been seen increasing from 0 day to 28 days of experiment (Figure 17 and Figure 18). This has shown that weathering did affect the physical properties of the coatings in terms of their volume which has been measured from their thickness. As the duration of exposure to humidity increase, the volume of coatings will also increase. This is due to the presence of water that has been absorbed by the coatings. Comparing between the Fumed Silica coatings and ATH coatings (Figure 19), the thickness increment in Fumed Silica coatings is higher than the thickness increment in ATH coatings.

4.3 Char Expansion

Thickness of char after the samples were burnt up to 500 °C in furnace was measured and recorded in the tables below.

Sample	Thickness (mm)	
	Before	***After***
A (0%)	4.64	21.25
B (FS 1%)	4.69	25.25
C (FS 2%)	4.95	25.75
D (FS 3%)	4.73	23.50
E (FS 4%)	4.96	24.75
F (FS 5%)	5.00	26.50
G (ATH 1%)	5.42	27.25
H (ATH 2%)	5.99	33.00
I (ATH 3%)	5.17	23.00
J (ATH 4%)	4.18	22.50
K (ATH 5%)	5.42	32.00

Table 8: Thickness before and after burning for non-weathered coatings

Sample	Thickness (mm)	
	Before	**After**
A (0%)	4.82	21.75
B (FS 1%)	5.00	25.50
C (FS 2%)	5.22	23.75
D (FS 3%)	5.17	21.50
E (FS 4%)	5.71	23.25
F (FS 5%)	4.82	19.00
G (ATH 1%)	4.75	21.75
H (ATH 2%)	5.00	24.00
I (ATH 3%)	5.21	25.75
J (ATH 4%)	4.68	26.00
K (ATH 5%)	5.03	27.75

Table 9: Thickness before and after burning for weathered coatings

To be able to see the effect of weathering on the char expansion of coatings, the expansion was calculated using the equation below and the findings were tabulated in the Table 10. The data are then presented in graphs form to see the correlation.

$$\frac{Thickness\ after(mm) - 1.5(mm)}{Thickness\ before\ (mm) - 1.5\ (mm)}$$

Sample	Expansion (...×)		Percentage of Reduction in Char Expansion (%)
	Non-weathered	Weathered	
A (0%)	6.29 ×	6.10 ×	3.02
B (FS 1%)	7.45 ×	6.86 ×	7.92
C (FS 2%)	7.03 ×	5.98 ×	14.94
D (FS 3%)	6.81 ×	5.45 ×	19.97
E (FS 4%)	6.72 ×	5.17 ×	23.07
F (FS 5%)	7.14 ×	5.27 ×	26.19
G (ATH 1%)	6.67 ×	6.23 ×	6.60
H (ATH 2%)	7.02 ×	6.43 ×	8.40
I (ATH 3%)	5.86 ×	6.54 ×	-10.40
J (ATH 4%)	7.84 ×	7.70 ×	1.82
K (ATH 5%)	7.78 ×	7.43 ×	4.50

Table 10: Char expansion for Fumed Silica and ATH coatings and their percentage of reduction in char expansion

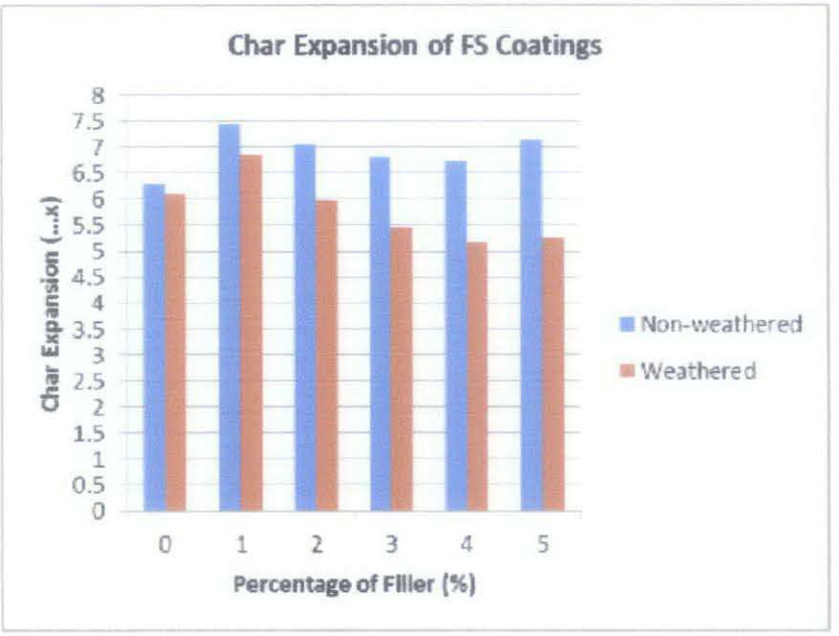


Figure 20: Char expansion for Fumed Silica coatings vs. the percentage of filler in coatings

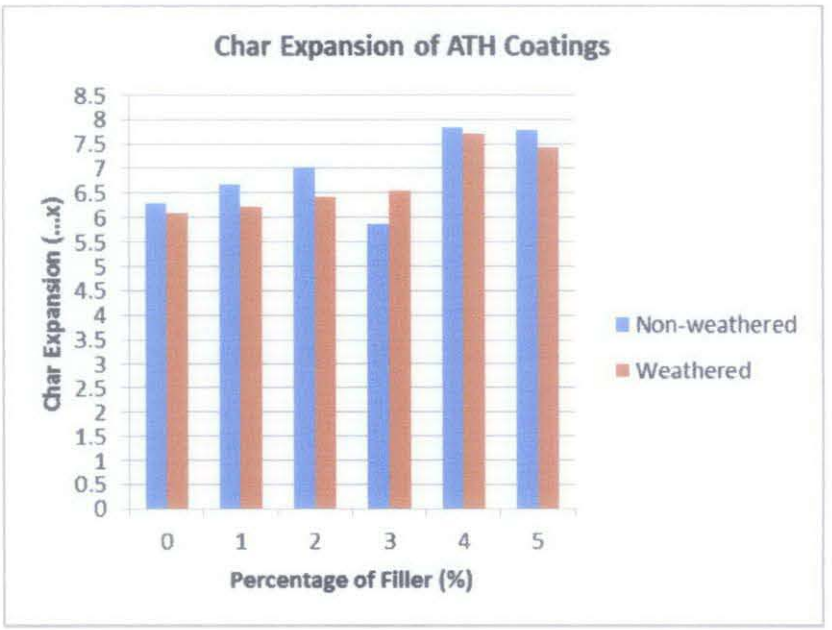


Figure 21: Char expansion for ATH coatings vs. the percentage of filler in coatings

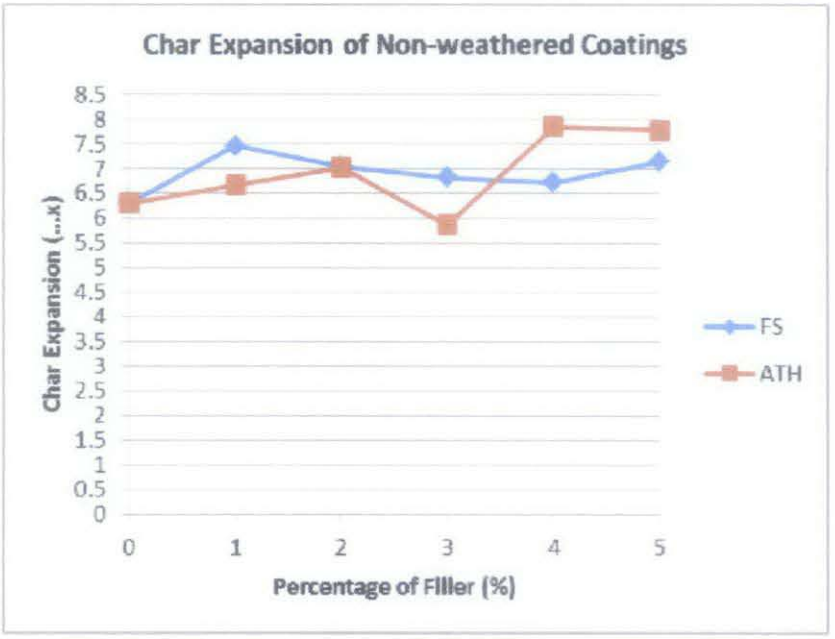


Figure 22: Char Expansion for non-weathered coatings vs. the percentage of fillers in coatings

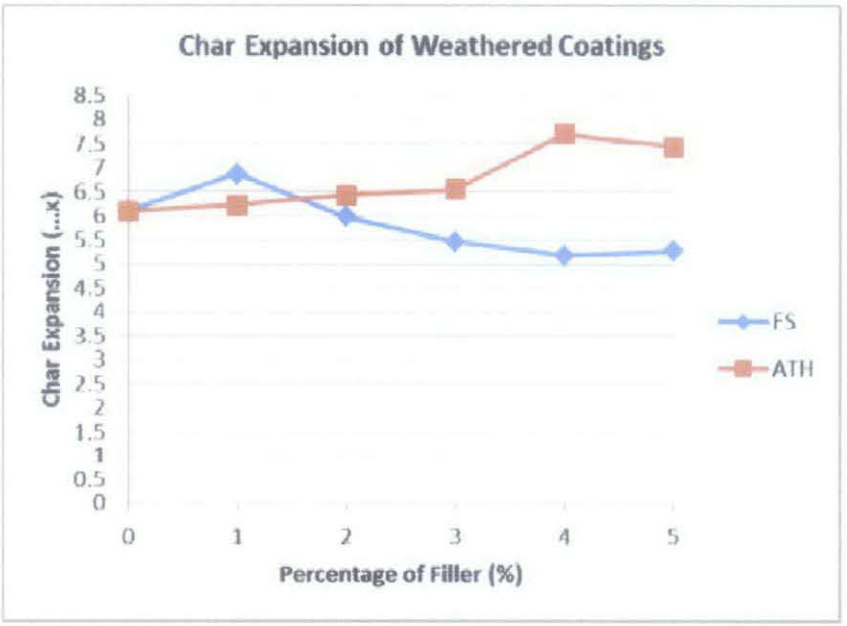


Figure 23: Char Expansion for weathered coatings vs. the percentage of fillers in coatings

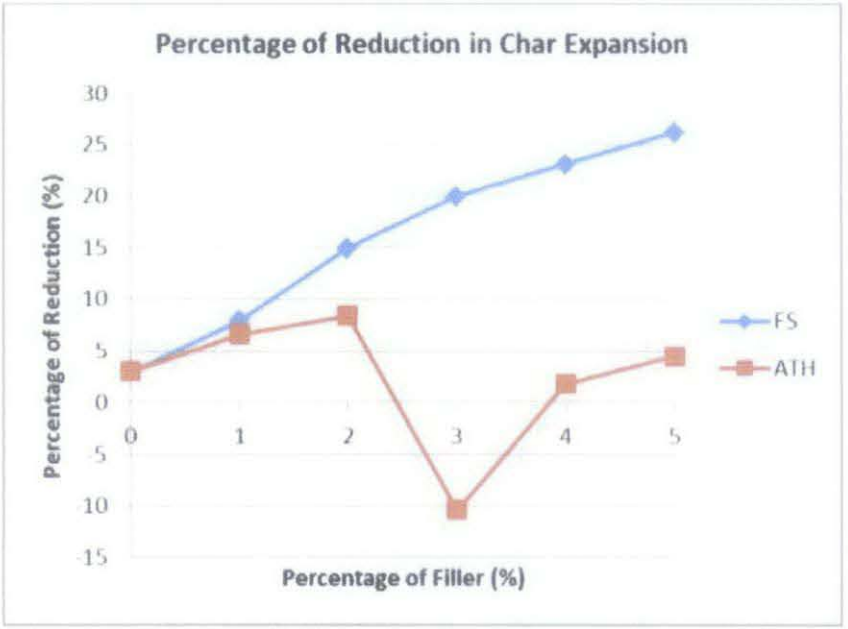


Figure 24: Reduction in percentage of char expansion vs. the percentage of fillers in coatings

From the graphs above (Figure 20 and Figure 21), the non-weathered coatings for both Fumed Silica and ATH have more expansion compared to the weathered coatings. Looking at the graph in Figure 23, ATH weathered coatings have more expansion than Fumed Silica weathered coatings. The graph in Figure 24 shows that the expansion for ATH weathered coatings is increases unlike the expansion for Fumed Silica weathered coatings as it gradually decreases as the percentage of filler increase. It shows that the reduction in char expansion for Fumed Silica weathered coatings is greater than the ATH weathered coatings. This indicates that ATH coatings have better performance in char formation than Fumed Silica coatings.

4.4 Water Absorption Percentage (%)

Water absorbed by the coatings can be determined using the weight data. Since the machine to calculate the volume of water that has been absorbed was not available, the water absorption percentage is calculated manually using equation below:

$$\frac{(Weight\ of\ wet\ sample - Weight\ of\ dried\ sample)}{Weight\ of\ dried\ sample} \times 100$$

Samples weight was measured by using the digital weighing scale. Changes in weight of the samples were recorded. The weight was gradually increasing due to the absorption of water by the coating. The changes in weight in each sample are recorded in the tables below.

Sample	Weight of Samples (g)			
	1	2	3	4
A (0%)	39.71	37.84	39.78	39.50
B (FS 1%)	41.27	38.73	39.30	39.29
C (FS 2%)	41.83	41.16	40.25	40.00
D (FS 3%)	41.97	38.86	39.83	40.32
E (FS 4%)	42.40	42.44	41.48	39.92
F (FS 5%)	40.60	39.78	38.96	40.49
G (ATH 1%)	39.38	39.56	42.02	39.15
H (ATH 2%)	37.28	40.45	42.40	41.41
I (ATH 3%)	41.49	42.81	37.71	39.68
J (ATH 4%)	39.32	39.45	37.89	41.81
K (ATH 5%)	41.52	41.45	38.84	39.84

Table 11: Weights of samples (g) before weathering test

Sample	Weight of Samples (g)				
	Duration of Exposure (days)				
	0	7	14	21	28
A (0%)	39.71	39.81	39.93	40.01	40.11
B (FS 1%)	41.27	41.41	41.52	41.64	41.75
C (FS 2%)	41.16	41.35	41.50	41.63	41.75
D (FS 3%)	41.97	42.12	42.29	42.42	42.55
E (FS 4%)	42.40	42.49	42.62	42.74	42.87
F (FS 5%)	40.60	40.69	40.79	40.88	40.98
G (ATH 1%)	39.38	39.48	39.60	39.71	39.83
H (ATH 2%)	37.28	37.37	37.47	37.58	37.70
I (ATH 3%)	39.68	39.74	39.81	39.89	39.98
J (ATH 4%)	39.45	39.53	39.59	39.66	39.74
K (ATH 5%)	39.84	39.90	39.99	40.10	40.22

Table 12: Weight of samples (g) after weathering test

The results of water absorption percentage for each sample are recorded in the table below.

Sample	Water Absorption Percentage (%)				
	Duration of Exposure (days)				
	0	7	14	21	28
A (0%)	0	0.25	0.55	0.75	1.00
B (FS 1%)	0	0.34	0.60	0.89	1.15
C (FS 2%)	0	0.46	0.82	1.13	1.41
D (FS 3%)	0	0.36	0.76	1.06	1.36
E (FS 4%)	0	0.21	0.52	0.80	1.10
F (FS 5%)	0	0.22	0.47	0.68	0.92
G (ATH 1%)	0	0.25	0.56	0.83	1.13
H (ATH 2%)	0	0.24	0.51	0.80	1.11
I (ATH 3%)	0	0.15	0.33	0.53	0.75
J (ATH 4%)	0	0.20	0.35	0.53	0.73
K (ATH 5%)	0	0.15	0.38	0.65	0.94

Table 13: Increment in weight percentage (%) after weathering test

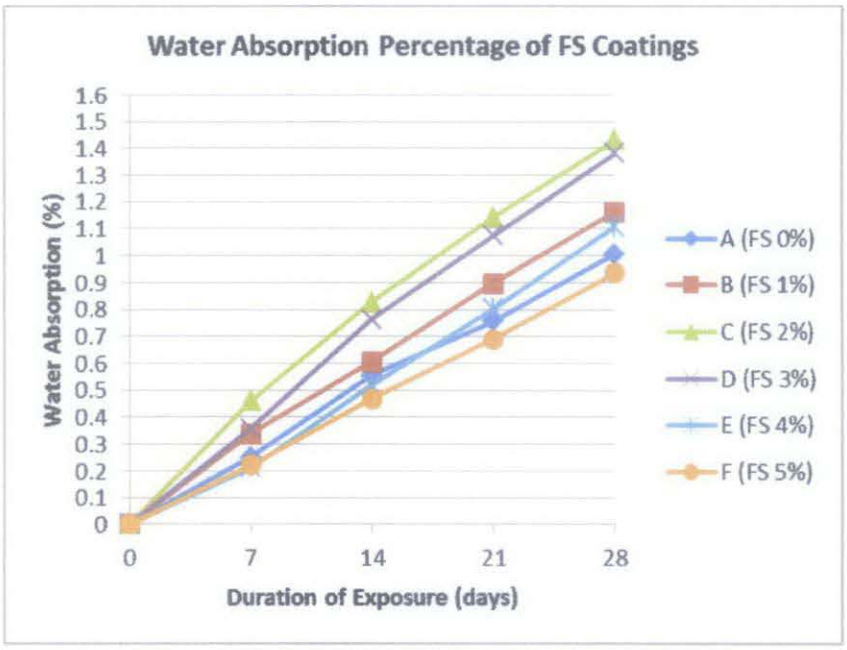


Figure 25: Water absorption percentage for Fumed Silica coatings vs. duration of exposure in days

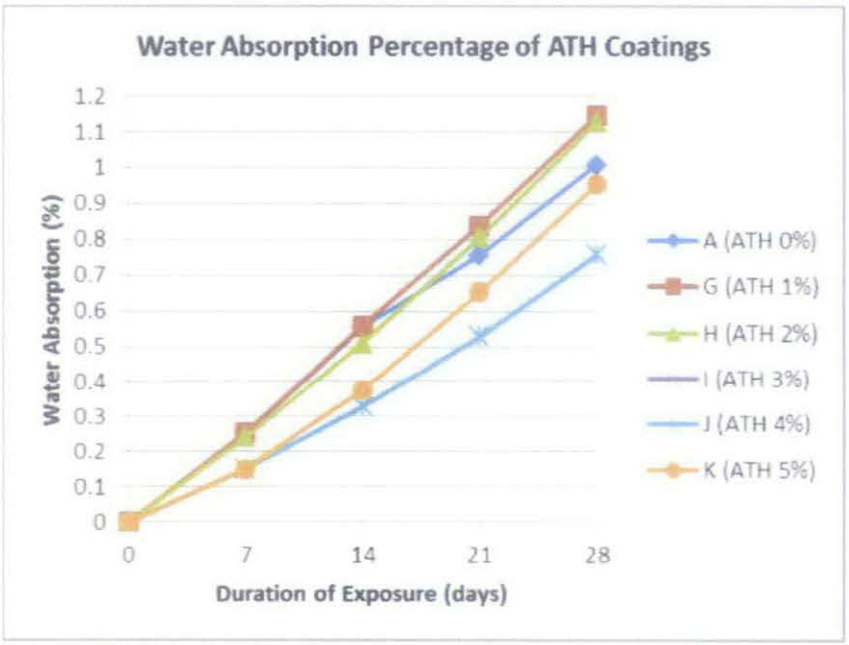


Figure 26: Water absorption percentage for ATH coatings vs. duration of exposure in days

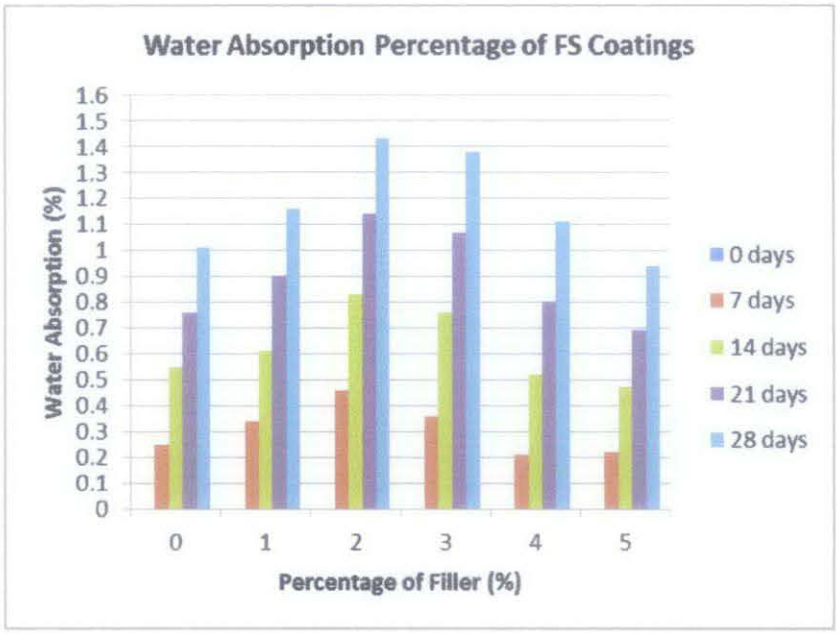


Figure 27: Water absorption percentage for Fumed Silica coatings vs. percentage of filler in coatings

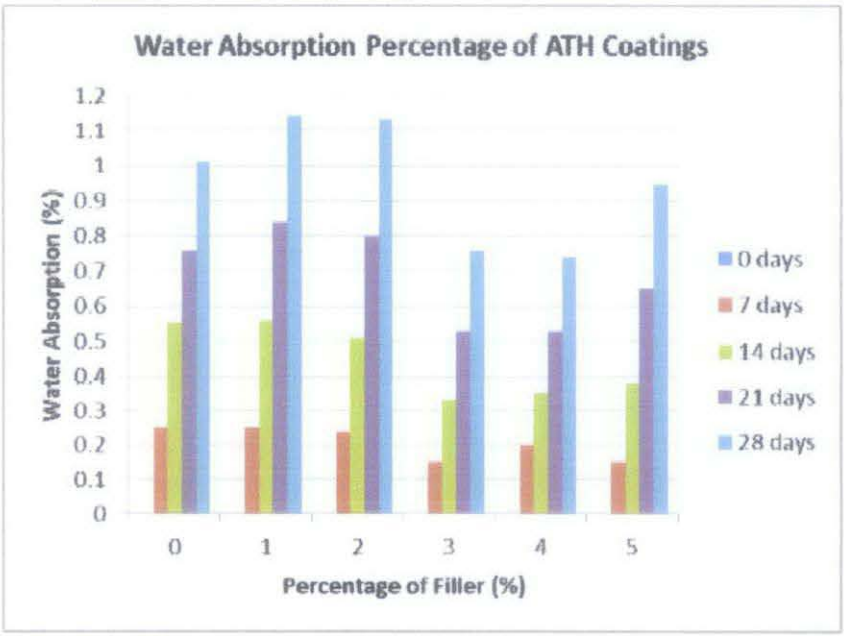


Figure 28: Water absorption percentage for ATH coatings vs. percentage of filler in coatings

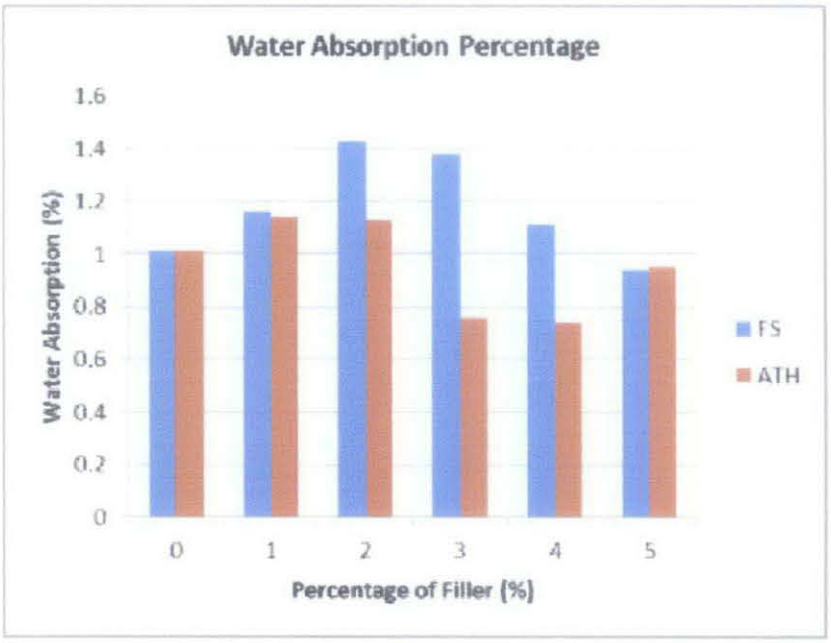


Figure 29: Water absorption percentage for both Fumed Silica and ATH weathered coatings vs. percentage of fillers in coatings

Based on the graphs in Figure 25 and Figure 26 the percentage of water absorbed increases as the duration of exposure is increasing. This shows that the coatings have become more permeable as the duration of exposure is increasing where the coatings absorbed more and more water. Comparing the Fumed Silica coatings and ATH coatings, the Fumed Silica coatings were tend to absorb more water than the ATH coatings. This can be seen in graph that is shown in Figure 29.

4.5 Surface Roughness

For surface roughness test, the roughness values before and after weathering test were measured and recorded in Table #. The percentage of surface roughness increment was then calculated using equation below.

$$\frac{(Final\ roughness - Initial\ roughness)}{Initial\ roughness} \times 100\%$$

Samples	Surface Roughness (μm)		Surface Roughness Increment (%)
	Before Weathering	After Weathering	
A (0%)	9.259	12.879	39.10
B (FS 1%)	9.395	10.527	12.05
C (FS 2%)	9.604	10.631	10.69
D (FS 3%)	9.334	10.314	10.50
E (FS 4%)	7.746	8.481	9.49
F (FS 5%)	7.046	7.334	4.09
G (ATH 1%)	10.402	12.669	21.79
H (ATH 2%)	8.287	11.060	33.46
I (ATH 3%)	10.539	13.018	23.52
J (ATH 4%)	7.635	8.784	15.05
K (ATH 5%)	10.826	11.757	8.60

Table 14: Comparison of surface roughness for Fumed Silica and ATH coatings before and after weathering and the increment in surface roughness

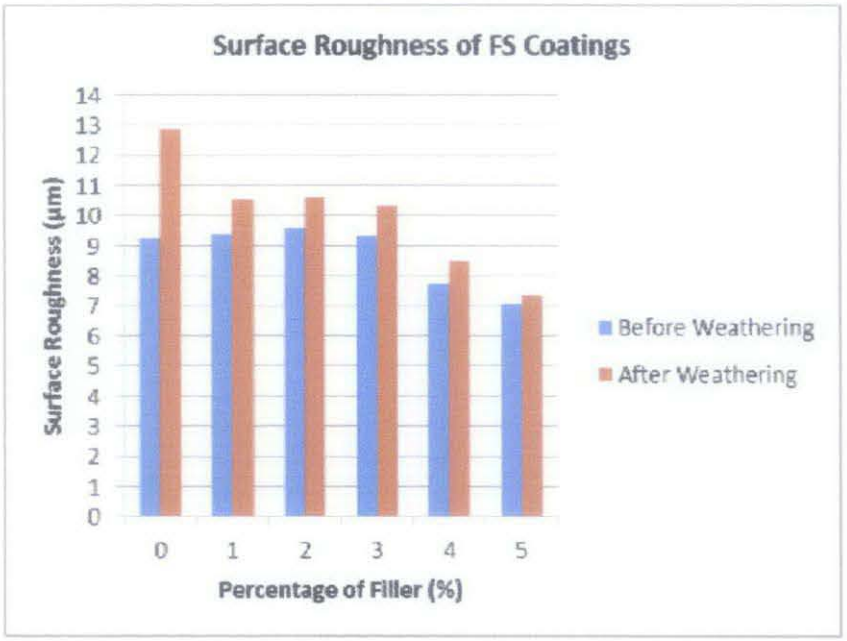


Figure 30: Surface roughness before and after weathering for Fumed Silica coatings vs. percentage of filler in coatings

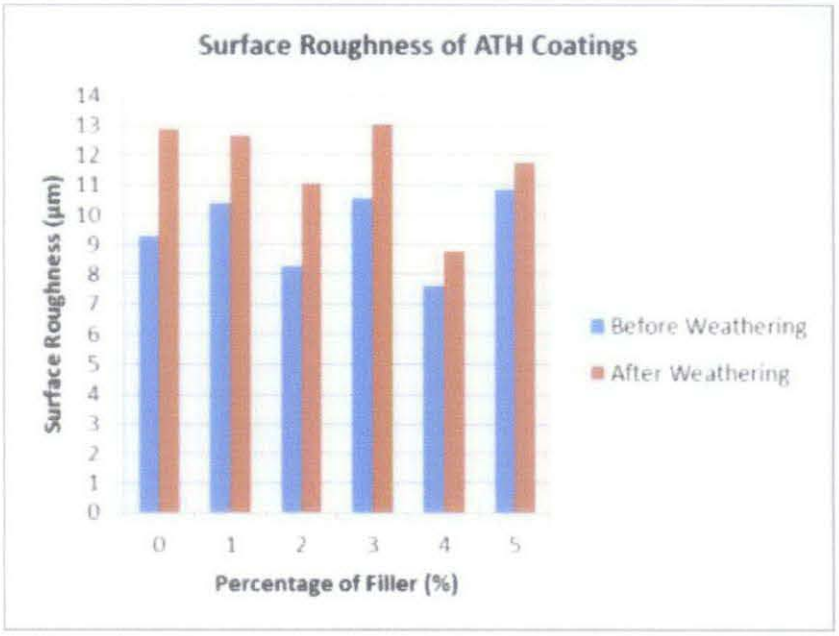


Figure 31: Surface roughness before and after weathering for ATH coatings vs. percentage of filler in coatings

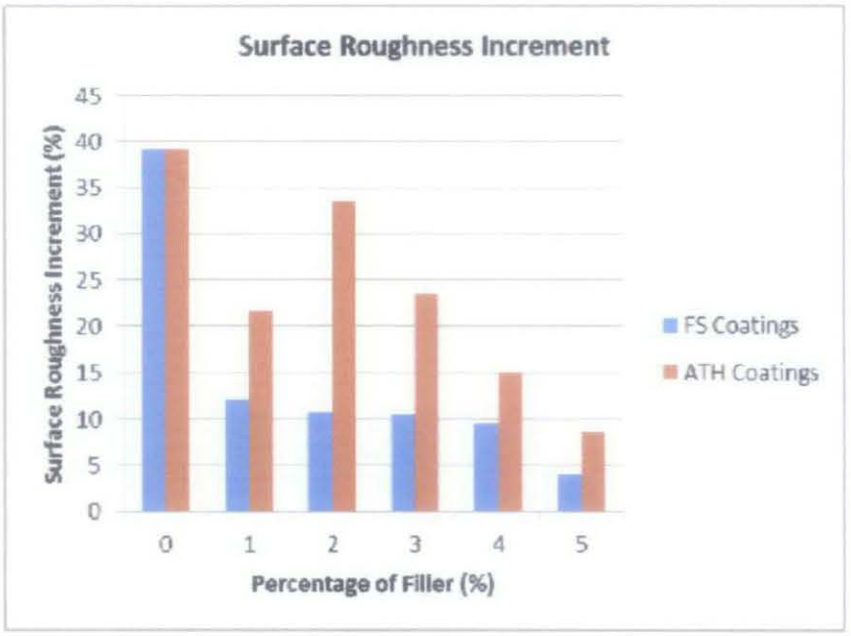


Figure 32: Surface roughness increment for both Fumed Silica and ATH coatings vs. percentage of filler in coatings

Looking at the graph in Figure 30 and Figure 31, the values of surface roughness after weathering for both Fumed Silica and ATH coatings are greater than the values before weathering. This is because the coatings were degraded due to the weathering. The surface has become uneven as the coatings were exposed to weathering issue which in this case is humidity. Based on the graph in Figure 32, the surface roughness for ATH coatings is greater than the Fumes Silica coatings after weathering. This has shown that the ATH coatings tend to degrade more than the Fumed Silica coatings.

4.6 Problem Faced

The problem that was faced in completing the project was the weathering chamber which was very important in the project had technical problem. The weathering chamber was supposed to test the effect of UV exposure on the samples but because of the xenon lamps were broken, the UV test cannot be done. The only parameter that could be tested is the effect of humidity on the samples. Due to the broken xenon lamps, the temperature inside the weathering camber (air temperature) could not be controlled and this lead to the failure of the machine. The machine stopped automatically when the temperature was too low, as low as 25 °C. When this happened, nothing could be done except to restart the machine manually and this had given problem to the author as the author has to restart the machine everyday.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The project has been successfully done eventhough there were problems occurred in the middle of the progress. The project is a success and it has met its objectives. The project is to study the weathering effects (humidity) on the intumescent coatings formulated with inorganic fillers. Few factors must be taken into consideration since they might be the potential influence of physical aging on coating performance such as volume, toughness and permeability of the coatings. Several tests were conducted on the coated samples such as weathering test, furnace test and also surface roughness test.

The weathering issue which in this case is humidity did affect the performance in char formation and also the physical properties of intumescent coating with inorganic fillers. The changes in colour, decreasing of char expansion and increasing volume of water absorbed by the coatings had proved the theory. ATH coatings have better performance in terms of char formation and they absorb less water eventhough the coatings are rougher compared to Fumed Silica coatings.

5.2 Recommendation

For future purpose, it is recommended for the next research to use Scanning Electron Microscopy (SEM) to study the relative physical changes on the intumescent coatings like roughness, micro-cracks and erosion etc. The author also recommends that the experiment to be able to be tested in a good condition weathering chamber so that the effect of ultraviolet light can be studied on the coatings. The coatings also need to be tested in the chamber for at least three months to be able to see the further changes in their physical properties.

REFERENCES

[1]M. Jimenez, S. Dubuquesne, S. Bourbigot (2006). Intumescent fire protective coating: Toward a better understanding of their mechanism of action. *ScienceDirect Thermochimica Acta 449 (2006) 16-26*

[2] B. Graham, Protecting Steel in Fire. Retrieved from http://www.leighspaints.com/me/tASteelinFire.aspx

[3] Intumescents: What they are and what they do. Retrieved from http://ahering.webs.com/intumescents.htm

[4] M. Jimenez, S. Dubuquesne, S. Bourbigot (2006). Characterization of the performance of an intumescent fire protective coating. *ScienceDirect Surface & Coatings Technology 201 (2006) 979-987.*

[5] W. Guojian, Y. Jiayun (2010). Influences of binder on fire protection and anticorrosion properties of intumescent fire resistive coating for steel structure. *ScienceDirect Surface & Coatings Technology 204 (2010) 1186-1192*

[6] W. Zhenyu, H. Enhou, K. Wei (2006). An investigation into fire protection and water resistance of intumescent nano-coatings. *ScienceDirect Surface & Coatings Technology 201 (2006) 1528-1535.*

[7] Alumina Trihydrate. Akrochem Corporation. Retrieved from http://www.akrochem.com/pdfs/fire/X-alumina%20trihydrate.pdf

[8] Takashi Kashiwagi, John R. Shields, Richard H. Harris, Jr., Rick D. Davis (2002). *Flame-Retardant Mechanism of Silica: Effect of Resin Molecular Weight, 2002*

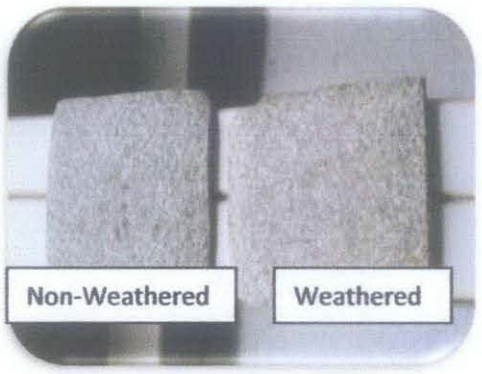
[9] C. Stuart, H. Brian (2008). A framework for predicting the service lifetime of composite polymeric composite polymeric coatings. *Springer,J Mater Sci (2008) 43:6630-6641.*

[10] T.A. Roberts, L.C. Shirvill, K. Waterton, I.Buckland (2009). Fire resistance of passive fire protection coatings after long-term weathering. *ScienceDirect Process Safety and Environmental Protection 88 (2010) 1-19.*

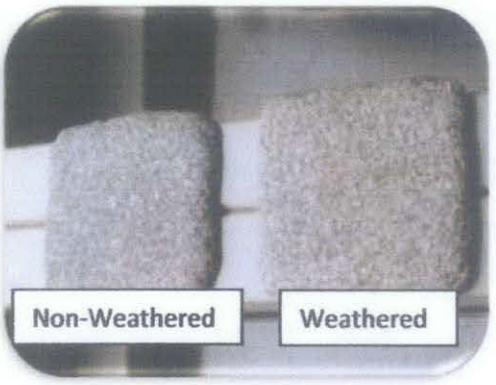
[11] J. Mallegol, M. Poelman, M.G. Oliver (2007). Influence of UV weathering on corrosion resistance of prepainted steel. *ScienceDirect Progress in Organic Coatings 61 (2008) 126-13*

APPENDICES

Appendix A - Colour Comparison between Non-weathered and Weathered Samples



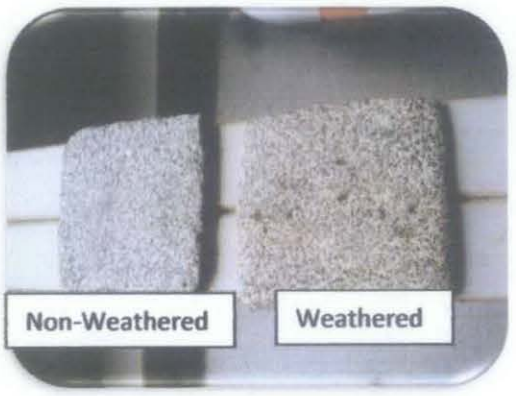
Sample A (0%)



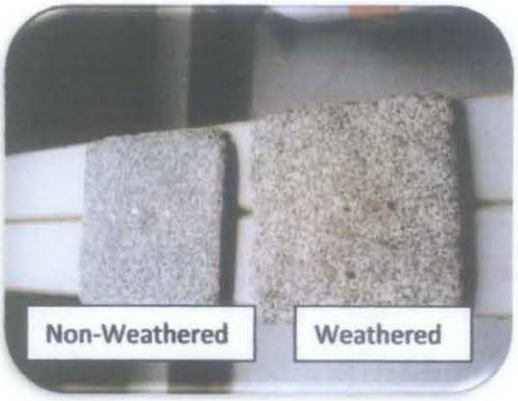
Sample B (FS 1%)

Sample C (FS 2%)

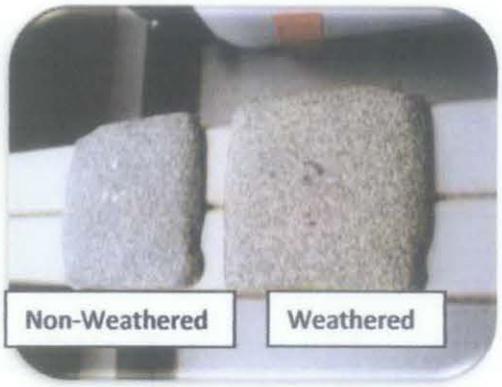
Sample D (FS 3%)



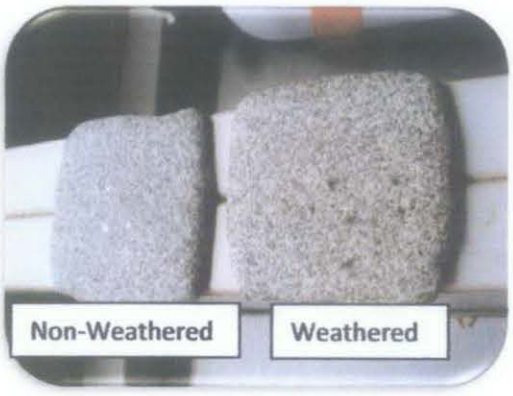
Sample E (FS 4%)



Sample F (FS 5%)



Sample G (ATH 1%)



Sample H (ATH 2%)

Sample I (ATH 3%)

Sample J (ATH 4%)

Sample K (ATH 5%)

Appendix B - Char Expansion for Non-weathered Samples

Sample A (0%)

Sample B (FS 1%)

Sample C (FS 2%)

Sample D (FS 3%)

Sample E (FS 4%)

Sample F (FS 5%)

Sample G (ATH 1%)

Sample H (ATH 2%)

Sample I (ATH 3%)

Sample J (ATH 4%)

Sample K (ATH 5%)

Appendix C - Char Expansion for Weathered Samples

Sample A (0%)

Sample B (FS 1%)

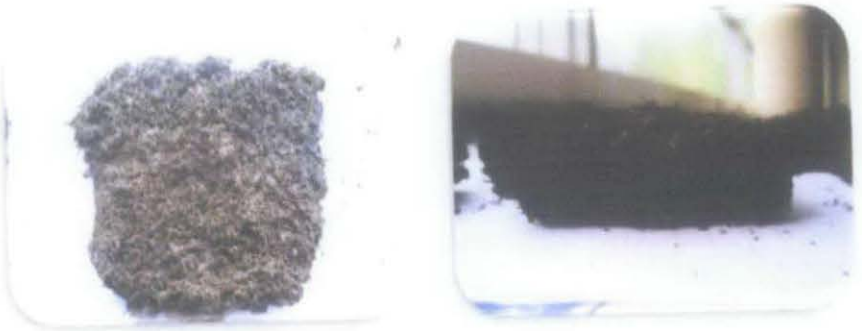
Sample C (FS 2%)

Sample D (FS 3%)

Sample E (FS 4%)

Sample F (FS 5%)

Sample G (ATH 1%)



Sample H (ATH 2%)

Sample I (ATH 3%)

Sample J (ATH 4%)

Sample K (ATH 5%)