FINAL YEAR PROJECT REPORT

Corrosion Behavior of Intumescent Coated Steel In Seawater Environment

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CERTIFICATION OF APPROVAL

CORROSION BEHAVIOR OF INTUMESCENT COATED STEEL IN SEAWATER ENVIRONMENT

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Approved

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(Asooc. Prof. Dr Faiz Ahmad)

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May 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(MOHD SHAHRUL EZWAN BIN ISMAIL)

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ABSTRACT

Intumescent coating is a mineral based or organic resin based product functioning as fire retardant coating where it can be applied to metallic materials, polymers, textiles, wood as well as structural steel in buildings, storage tank in order to protect them from weakening when encounter elevated temperature in a fire. Most of the offshore and marine structure are heavily exposed to the marine environment mainly seawater which is one of the corrosion medium. Coating protect steels through barrier layer action of the coating, secondary barrier action of corrosion product layer. The presence of the mechanical damage allows the access of corrodents to the substrate, eventually resulting in destruction of the coating by the growth of corrosion products. Researcher will develop an intumescent coating formulation which consist of three agents mainly Acid Source (AAP, Polyphosphate), Carbon source (EG, Expandable Graphite) and blowing agent (MEL, Melamin) followed by epoxy, Boric Acid, Polyamide Hardner and etc to the steel and exposed it to the seawater environment. Various percentage of coated area will be applied to the substrate as manipulated variable. Corrosion effect will be evaluated using visual inspection and microscopic view. The substrates later will be testing on fire retardant performance by bunsen burner test. The char expansion as well as heat shielding will be thoroughly observed and result will be obtained and further studied.

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CHAPTER 1

INTRODUCTION

1.0 Problem Statement

The presence of Intumescent coating is the mitigation action to protect the substrate from high temperature as well as corrosion. However, exposure of the intumescent coating in sea water will reduce the its function. The presence of defect in the coating allow access of corrodents to the substrate eventually resulting in the destruction of the coating by the growth of corrosion products [18]. The destruction of the intumescent coating giving the impact in substrate protection against corrosion and fire protection properties.

1.1 Objective

The objective of the project is to study the corrosion effect on intumescent coated steel when expose to the seawater environment for the certain period of time. It is also to test the fire retardant performance after the exposure in sea water environment.

1.2 Scope of Study

There are some parameters and limitations in this project. The main focus of studies will be on the effect of seawater towards the intumescent coating for certain period of exposure. Area of steel that being coated vary from one another for comparison. Salt spray (fog) testing which one of the accelerated testing will be used to simulate the seawater environment. Visual inspection will be done after the coated steel being exposed to the sea water in the salt spray chamber. The intumescent coated steel will be tested on fire retardant performance by fire test which is conducted using bunsen burner test. The sample produced will be tested in the laboratory by using Field Emission Scanning Electron Microscopy (FESEM) and XRD (X-Ray Diffraction) technique. An analysis will be done based on the result collected from the test.

CHAPTER 2

LITERATURE REVIEW

2.1 Intumescent Coating

Intumescent fire protective coatings are widely used as passive fire protection in oil and gas industries, civil buildings, chemical plants and other facilities in developed countries [1]. Intumescences may be defined as "thermally induced expansion of material" [2]. Intumescences also defined as the ability of the coating, upon exposure to high temperature flame, to swell or foam into a solid heat insulating layer while protecting the substrate from direct exposure to the flame. The coating retains its expanded or foamed structure at high temperatures to provide a heat isolative layer which protects the substrate for a prolonged period [3]. The coating swells in size to form a char, which protects the steelwork for a specified period [4].

2.2 Basic Elements In The Intumescent Coating

Intumescent coatings contain four basic elements which are [5]:

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

2.3 Mechanism of Intumescent Coating

The mechanism of intumescent is usually started with the acid source breaks down to yield a mineral acid, then it takes part in the dehydration of the carbonization agent to yield the carbon char, and finally the blowing agent decomposes to yield gaseous products. Then, the char will swell and this will provide an insulating multi-cellular protective layer. This shield limits at the same time the heat 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 [6].

Expandable graphite is formed by treating crystalline graphite, which is imposed of stack of parallel planes of carbon atoms, with intercalants such as sulphuric acid and/or nitric acid. When exposed to heat source, the intercalation compound (H2SO4) decompose into gaseous product (SO2 and H20). The high layer pressure resulting from decomposition of intercalation compounds produces a strong push force between the graphite layers, so the graphite basal planes can be pushed apart [5]. The decomposition of sulphuric acid and redox reaction between H2SO4 and carbon are responsible for most of the expansion process. The fused resin and carbonaceous compound can stick a large amount of expanded graphite in expanding process.

2.4 Coated steel in Sea Water

Intumescent coatings were composed of three fire retardant additives: an acidsource (such asammoniumpolyphosphate, APP), a carbon source (such as pentaerythritol, PER) and a blowing agent (such as melamine, MEL) bound together by a binder. During the intumescent process, the binder became important due to two effects: it contributed to the char layer expansion and ensured the formation of uniform foam structure [7-10]. However, hydrophilic 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 [10]. This would significantly depress the expected effect of intumescent coatings. The binder as a film-forming component could prevent or remarkably reduce migration of fire retardant additives and access of the corrosive substances [10]. An offshore or a marine structure is located in the corrosive environment containing abundant sea salt, sea wave, and sunlight. To prevent the corrosion of the structural steel under such a severe condition, various methods are applied corresponding to each site of the structure. Under the sea, the electrical protection using the sacrificial anode is effective. In the mild tidal zone, the steel is covered with the insulating varnish or paint. In the severe tidal and the splash zones, the coating requires the sufficient physical strength in addition to corrosion resistance. Therefore, cladding of the anti-corrosion metal and alloy is adopted in these zones [11].

There are major concern on being given on deterioration of equipment and infrastructure exposed to actual hostile marine environments mainly sea water. As a result structural engineers and naval architects are increasingly interested in the rate of loss of strength of steel hence in the loss of material even short-term exposures is important in part because protective measures are not always wholly effective [14]. Sea water is one of the corrosive medium.

2.5 Corrosion of Coated Steel

Corrosion is the deterioration of the properties, mechanical, aspect of a material due to the surrounding environment. It means a loss of an electron of metals reacting with water and oxygen. That is why coating is important to solve for corrosion problem. Coatings are used to prevent or control corrosion so know the basic concept of corrosion will help in coatings technology [15]. When coatings break down, then the steel will corrode where the corrosion of steel arises from its environment of exposure. The chemical reaction between the exposed steel with moisture and oxygen as below:

$$\underset{\text{(iron)}}{Fe} + O_2 + H_2O \longrightarrow \underset{\text{(rust)}}{Fe_2O_3.H_2O}$$

Steel is the most commonly used metals, corrode in many media including most outdoor atmospheres. Usually they are selected not for their corrosion resistance but for such properties as strength, ease of fabrication, and cost. These differences show up in the rate of metal lost due to rusting. All steels and low-alloy steels rust in moist atmospheres [16]. Few method to protect steel from corrosion. One of the way is passive coating. Passive barrier protection works by coating the steel with a protective coating system that forms a tight barrier to prevent exposure to oxygen, water and salt (ions). The lower the permeability of the coating system to water, the better the protection provided. Two-pack epoxy coatings and chlorinated rubbers applied at sufficiently high film builds offer the most successful corrosion protection through passive barrier protection [17].

Zinc-containing paints are widely used in coating applications throughout the world because of their high corrosion protection performance. The mechanism by which zinc-containing paints protect steel has been of interest since the early 1940s. (MinYoung Shon, 2010). Subsequently, several researchers examined the corrosion protection properties of zinc-containing paints and discovered the cathodic protection mechanism of zinc-containing paints on metals which is zinc particles in galvanic contact with the steel substrate will contribute to this effect. As the zinc particles corrode, the contact to the steel will gradually be lost, and at a certain time the potential of the steel exceeds the protection potential. [18]

The use of the epoxy giving many positive impact. The epoxy binder is electrically insulating and protects metallic surfaces against corrosion. The zinc particles in a zinc epoxy primer may then be protected by the epoxy and insulated from the steel surface. (Ole Øystein Knudsen, Unni Steinsmo, Marit Bjordal,2005). An experiment conducted at Snorre. Snorre is an offshore oil production platform located in the Norwegian sector, west of the Norwegian coast. The field test was started in March 1995 and terminated in September 2000. The samples had then been exposed for 5½ years.

The results is the film thickness of the zinc epoxy seems to be important for the performance of the coating system. The system with the thickest zinc epoxy performed best in the field test, and there seemed to be a correlation between film thickness of the zinc epoxy and electrochemical properties [19].

Another experiment conducted to simulated the ballast tank that being used in oil tanker. The ballast tank is completely exposed to the sea water directly whenever the biodiesel of the tanker ship being used. The electrochemical corrosion of carbon steel exposed to a mixture of biodiesel and 3.5% NaCl solution simulated seawater was characterized using wire beam electrode (WBE) technique. (Wei Wang, Peter E. Jenkins, Zhiyong Ren, 2012). The result is the carbon steel material corrodes quickly when exposed to a mixture of biodiesel and 3.5% NaCl solution simulated seawater. [20]

2.6 Seawater

The dominant chemical characteristic of seawater is its dissolved salts, which typically constitutes 3.5% of its composition. This means that every 1 kg of seawater has approximately 35 grams of dissolved salts [21]. The average density of seawater at the surface of the ocean is 1.025 g/ml; seawater is denser than fresh water because of the added weight of the salts and electrostriction. The principle ions are oxygen, sodium, magnesium, chloride, and sulfate as tabulate in Table 2.1. Entrapped bubbles of seawater vapor, as in foam, may collapse suddenly be able to lead to corrosion of offshore structure.

Element	**Percent %**
Oxygen	85.84
Hydrogen	10.82
Chloride	1.94
Sodium	1.08
Magnesium	0.1292
Sulfur	0.091
Calcium	0.04
Potassium	0.04
Bromine	0.0067
Carbon	0.0028

Table 1: Seawater composition (by mass) [22]

2.7 Structural Steel

Steel is a noncombustible material and available in various type of products such as structural, reinforcing, prestressing or cold formed. Similar to other materials, exposure to elevated temperature leads to a temporary decrease in the strength and stiffness of steel. Such prolonged degradation adversely affects the resulting deformations and load carrying capabilities of steel during the fire exposure. For example, the deformations are increased while the strength and stiffness is reduced. Thermal properties of steel are also affected such as the coefficient of thermal expansion, specific heat and conductivity [23]

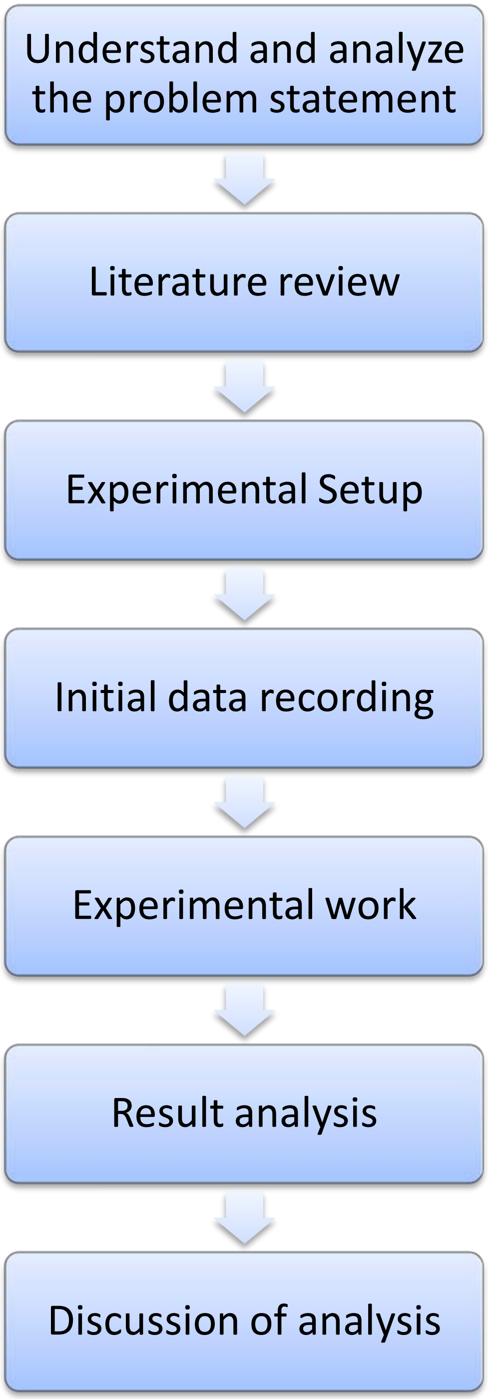
Although steel does not burn, it can be severely weakened when exposed to high temperatures for extended periods, such as in a building fire. It is believed that such damage led to the collapse of the World Trade Centre towers Since the attacks of 9/11, greater attention has been focused on the development and application of better and more durable intumescent coatings

In exposures to a temperature excess of $600^{O}C$ for more than 15 minutes, unprotected steel will tend to deform, twist and buckle. At and above such high temperatures, the crystalline and metallurgical structure of typical carbon based steels use for buildings also undergoes a transformation Structural steel loses its load carrying ability when temperature of the fire reaches $500^{O}C$. The prime requirement is to maintain the steel integrity between 1-3 hours when the temperature of the surroundings is excess of $1100^{O}C$. The structural steel begins to lose its structural properties above $500^{O}C$ or even as low as $450^{O}C$ in case of fire and tend to distort, leading to the collapse of building structures [24].

CHAPTER 3

METHODOLOGY

3.1 Project Flow Chart



3.2 Details of Flow Chart

I. **Understand and analyze the problem statement-** Problem analyzing and clear understanding on the title select

II. **Literature review-** Studies on related journal on Intumescent Coating and related information for better understanding

III. **Experimental Setup-** The formulation of intumescent coating prepared with various application to the substrate

IV. **Initial data recording-** All the initial data recorded for result analysis and calculation

V. **Experimental work-** Experimental works are conducted to get the results based on the initial properties and variable set. The test that will be used are salt spray (fog) test and furnace test.

VI. **Result analysis-** Results such as weight loss, thickness change of the coated substrate is analyze. The surface of the coating is analyze using microscopic view.

VII. **Discussion of Analysis**- The result obtained is compared for fully coated and one side coated intumescent steel.

3.3 Experimental Materials Used

3.3.1 Intumescent Coating

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

3.3.2 Intumescent Formulation

A range of formulations containing IFR components (APP, EG, MEL, Boric Acid, Epoxy and hardener) were developed.

Sample	APP (%)	EG (%)	MEL (%)	BA (%)	BPA (%)	TETA (%)	Alumina
A11	11.11	5.56	5.56	11.11	42.94	20.72	3
A12	11.11	5.56	5.56	11.11	42.94	20.72	3
A13	11.11	5.56	5.56	11.11	42.94	20.72	3
A21	11.11	5.56	5.56	11.11	42.94	20.72	3
A22	11.11	5.56	5.56	11.11	42.94	20.72	3
A23	11.11	5.56	5.56	11.11	42.94	20.72	3
A31	11.11	5.56	5.56	11.11	42.94	20.72	3
A32	11.11	5.56	5.56	11.11	42.94	20.72	3
A33	11.11	5.56	5.56	11.11	42.94	20.72	3
B11	11.11	5.56	5.56	11.11	42.94	20.72	3
B12	11.11	5.56	5.56	11.11	42.94	20.72	3
B13	11.11	5.56	5.56	11.11	42.94	20.72	3
B21	11.11	5.56	5.56	11.11	42.94	20.72	3
B22	11.11	5.56	5.56	11.11	42.94	20.72	3
B23	11.11	5.56	5.56	11.11	42.94	20.72	3
B31	11.11	5.56	5.56	11.11	42.94	20.72	3
B32	11.11	5.56	5.56	11.11	42.94	20.72	3
B33	11.11	5.56	5.56	11.11	42.94	20.72	3
S11	11.11	5.56	5.56	11.11	42.94	20.72	3
S12	11.11	5.56	5.56	11.11	42.94	20.72	3
S13	11.11	5.56	5.56	11.11	42.94	20.72	3
S21	11.11	5.56	5.56	11.11	42.94	20.72	3
S22	11.11	5.56	5.56	11.11	42.94	20.72	3
S23	11.11	5.56	5.56	11.11	42.94	20.72	3
S31	11.11	5.56	5.56	11.11	42.94	20.72	3
S32	11.11	5.56	5.56	11.11	42.94	20.72	3
S33	11.11	5.56	5.56	11.11	42.94	20.72	3

Table 2 Intumescent Coating Formulation

APP, MEL, BA and Alumina is mixed according to the ratio. The mixed then grind for 60 seconds using grinder. Later the EG being added to the grind mixture. BPA and TETA was stirred in the mixer. Subsequently the mixture is added to BPA and TETA.

3.4 Steel Substrate Preparation

Structural steel A36M with 2.5mm thickness was using. Steel substrate was cut using shear cutter machine into dimension 10cm x 10cm for fire testing purpose. After cutting process, the steel plate was sand blasted to remove dirt and corroded part also improve the adhesion of coating on the steel substrate. Figure 1(a) and 1(b) show the steel substrate before and after sand blasting.

Figure 4(a) Before Sand Blasting

Figure 1(b) After Sand Blasting

3.5 Application of Intumescent Coating To Steel Substrate

After the coating being formulated and prepared, it have to be immediately applied to the steel surface. The Application to the steel is divided in three categories; fully coated, one side coated in one side and one side coated in two side.

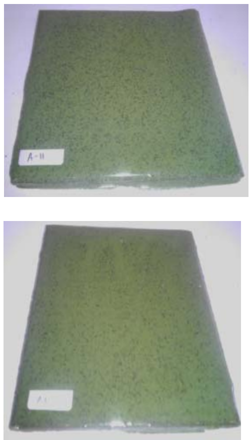


Figure 2(a) Fully Coated

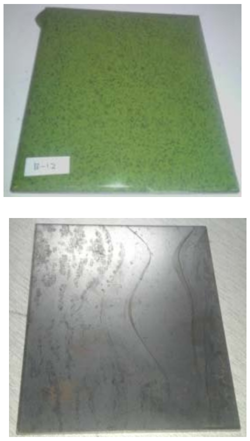


Figure 2(b) One side coated

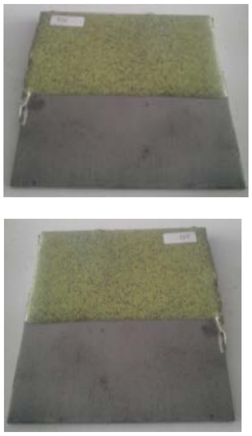


Figure 5(c) Two side coated (partially)

3.6 Salt Spray Testing (ASTM B117)

Salt spray test is an accelerated corrosion test that been conduct to determine the corrosion resistance of specimens against exposure of various environment type such as seawater environment.

Salt spray testing carried out as per standard ASTM B117 Standard Practice for Operating Salt Spray (Fog)

Figure 3(a) Salt Spray Chamber

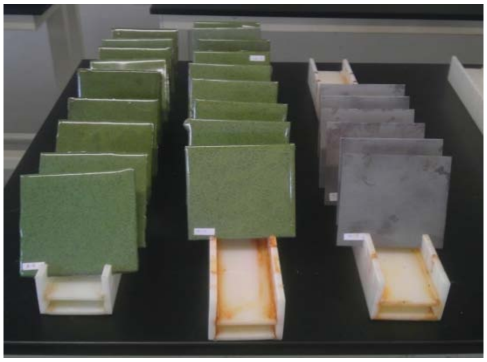


Figure 3(b) Sample Position in Salt Spray Chamber

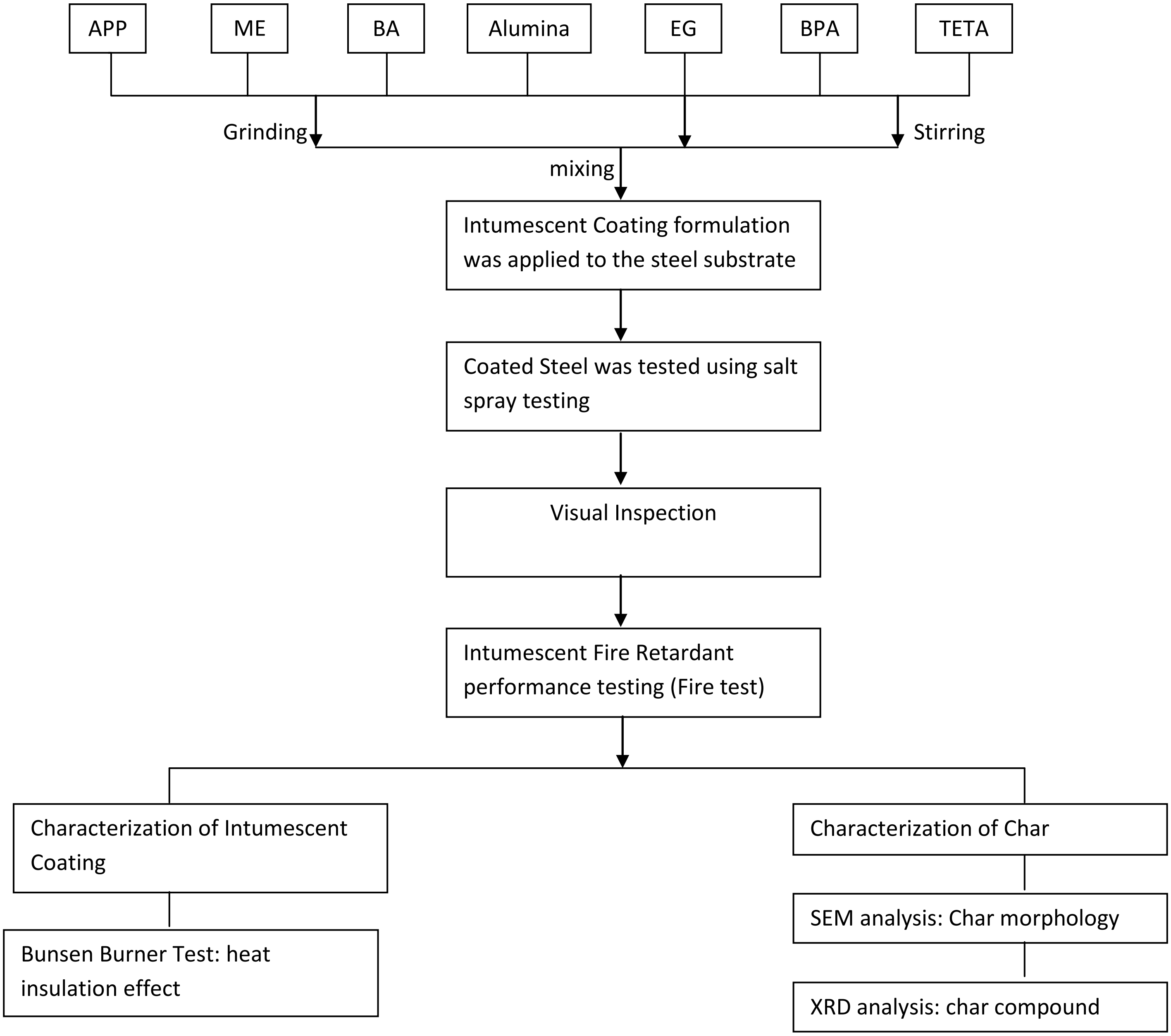
3.7 Bunsen Burner Test

This test was used to characterized the formation of the char and reaction of the intumescent coating.. During the testing, the temperature profile of the steel substrate is measured using digital thermo logger (Figure 5) and thermo couple. In this experimental work, 400^{o}C was chosen as the critical temperature for steel to ensure a high level of safety [25].

Figure 6 Bunsen Burner Test

Figure 5 Thermo Logger

3.8 The flow chart of the project activities is shown



3.9 Gantt Chart

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

■ Processes

○ Milestone

CHAPTER 4

RESULT AND DISCUSSION

4.1 Work Completed

All the process involved in completing the project has been carry out by the author. The coating was developed based on the formulation later applied to the steel substrate. The steel substrate was applied the intumescent coating in 3 different area which is fully coated area and one side coated area. Then, the coated steel being tested in salt spray chamber for corrosion testing. The result was analyzed by visual and weight loss calculation. The coated steel then tested for fire retardant performance using Bunsen burner testing testing. The fire testing result was analyzed by using Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD).

4.2 Data Recorded

4.2.1 Intumescent Coating

27 Formulations have been developed with alumina as a filler. The table below shows the detail of the formulation.

Sample	**APP (%)**	**EG (%)**	**MEL (%)**	**BA (%)**	**BPA (%)**	**TETA (%)**	**Alumina**
A11	11.11	5.56	5.56	11.11	42.94	20.72	3
A12	11.11	5.56	5.56	11.11	42.94	20.72	3
A13	11.11	5.56	5.56	11.11	42.94	20.72	3
A21	11.11	5.56	5.56	11.11	42.94	20.72	3
A22	11.11	5.56	5.56	11.11	42.94	20.72	3
A23	11.11	5.56	5.56	11.11	42.94	20.72	3
A31	11.11	5.56	5.56	11.11	42.94	20.72	3
A32	11.11	5.56	5.56	11.11	42.94	20.72	3
A33	11.11	5.56	5.56	11.11	42.94	20.72	3
B11	11.11	5.56	5.56	11.11	42.94	20.72	3
B12	11.11	5.56	5.56	11.11	42.94	20.72	3
B13	11.11	5.56	5.56	11.11	42.94	20.72	3
B21	11.11	5.56	5.56	11.11	42.94	20.72	3
B22	11.11	5.56	5.56	11.11	42.94	20.72	3
B23	11.11	5.56	5.56	11.11	42.94	20.72	3

B31	11.11	5.56	5.56	11.11	42.94	20.72	3
B32	11.11	5.56	5.56	11.11	42.94	20.72	3
B33	11.11	5.56	5.56	11.11	42.94	20.72	3
S11	11.11	5.56	5.56	11.11	42.94	20.72	3
S12	11.11	5.56	5.56	11.11	42.94	20.72	3
S13	11.11	5.56	5.56	11.11	42.94	20.72	3
S21	11.11	5.56	5.56	11.11	42.94	20.72	3
S22	11.11	5.56	5.56	11.11	42.94	20.72	3
S23	11.11	5.56	5.56	11.11	42.94	20.72	3
S31	11.11	5.56	5.56	11.11	42.94	20.72	3
S32	11.11	5.56	5.56	11.11	42.94	20.72	3
S33	11.11	5.56	5.56	11.11	42.94	20.72	3

Table 3 Intumescent Coating Formulation

A= Fully coated B= One side coated (one surface) S= One side coated

4.2.1 Thickness of The Intumescent Coat Applied to Steel Substrate

Sample	Reading1 (mm)	Reading2 (mm)	Reading 3 (mm)	Reading 4 (mm)	Reading 5 (mm)	Reading 6 (mm)	Average (mm)
A-11	1.82	1.85	1.92	2.01	1.82	2.08	1.92
A-12	1.8	2.21	2.23	1.87	1.64	1.77	1.92
A-13	1.85	1.98	1.84	1.85	2.25	2.06	1.97
B-11	1.52	1.56	2.2	1.94	2.1	1.98	1.88
B-12	1.66	1.4	1.54	1.74	1.76	1.82	1.65
B-13	1.2	2.06	1.54	1.6	1.42	1.32	1.52
S-11	1.45	1.7	1.6	1.65	1.75	1.7	1.64
S-12	1.6	1.6	1.49	1.62	1.7	1.7	1.62
S-13	1.6	1.35	1.45	1.4	1.65	1.7	1.53
A-21	1.77	2.02	2.11	1.97	2.13	1.96	1.99
A-22	1.87	2	1.87	1.88	1.85	2.02	1.92
A-23	1.84	1.9	1.82	2.02	2.07	1.98	1.94
B-21	1.5	1.9	1.96	1.72	2.32	1.54	1.82
B-22	1.76	1.76	1.76	1.54	1.56	1.66	1.67
B-23	1.64	2.06	2.5	1.8	1.98	1.8	1.96
S-21	1.91	2	2.05	1.95	1.97	2	1.98
S-22	1.92	1.8	2.03	2	1.97	1.98	1.95
S-23	1.85	1.9	1.89	1.77	1.77	1.87	1.84
A-31	2.02	1.93	1.87	1.78	2.02	1.98	1.93
A-32	1.75	1.77	1.91	1.9	2.15	2.12	1.93
A-33	1.82	1.88	2.12	1.96	1.9	1.93	1.94
B-31	1.8	1.84	1.82	1.6	2	1.9	1.83

B-32	1.74	1.94	1.98	1.84	1.7	1.52	1.79
B-33	1.96	1.94	1.52	1.64	2.14	2.1	1.88
S-31	2.04	1.89	1.87	1.9	1.95	1.92	1.93
S-32	1.85	1.87	2.02	1.9	1.95	1.93	1.92
S-33	1.95	1.95	1.9	1.93	1.95	1.92	1.93

Table 4 Thickness of The Insumescent Coating Applied to The Steel

4.2.2 Mass of The Intumescent Coated Steel

Sample	Mass Before (g)	Mass After (g)	Mass Loss (g)
A-11	231.23	231.1	0.13
A-12	229.909	229.81	0.099
A-13	236.715	236.713	0.002
B-11	213.665	210.211	3.454
B-12	212.143	207.331	4.812
B-13	208.15	204.224	3.926
S-11	215.172	211.204	3.968
S-12	213.767	210.113	3.654
S-13	214.875	209.375	5.5
UC11	195.44	184.22	11.22
UC21	196.11	186.39	6.3
A-21	235.426	235.389	0.037
A-22	236.503	236.49	0.013
A-23	231.885	231.711	0.174
B-21	214.187	207.687	6.5
B-22	213.825	206.837	6.988
B-23	216.746	210.977	5.769
S-21	219.631	213.231	6.4
S-22	214.829	207.922	3.44
S-23	216.225	210.245	5.63
UC21	196.55	182.572	13.978
UC22	195.73	180.007	15.723
A-31	235.794	235.621	0.173
A-32	232.17	231.982	0.188
A-33	234.802	233.25	1.552
B-31	215.721	203.721	7.2
B-32	213.75	202.3	6.91
B-33	215.855	204.325	3.01
S-31	216.64	206.38	6.91
S-32	218.467	206.137	4.55
S-33	217.428	207.19	6.03
UC31	195.23	184.577	14.51

UC32	196.313	178.871	16.49

Table 5 Mass of Intumescent Coated Steel

4.3 Exposure to Salt Spray Chamber

Sample	Exposure (hour)
A-11	720
A-12	
A-13	
A-21	1440
A-22	
A-23	
A-31	2160
A-32	
A-33	

Table 6Fully Coated Steel

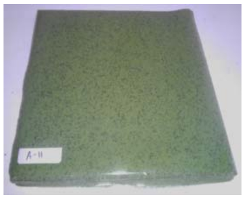
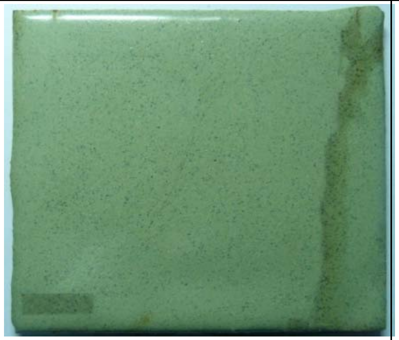
Sample	Exposure (day)
B-11	720
B-12	
B-13	
B-21	1440
B-22	
B-23	
B-31	2160
B-32	
B-33	

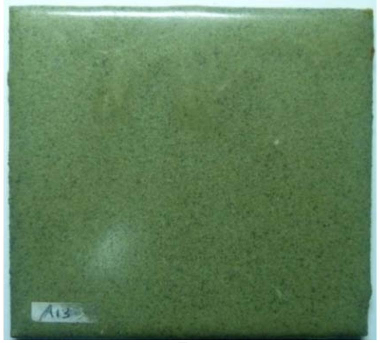
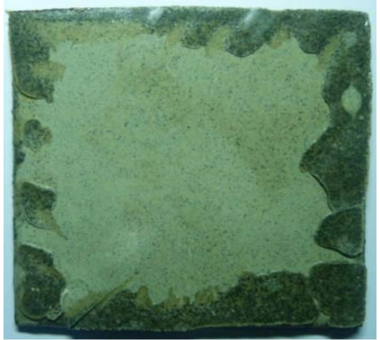
Table 7One side coated Steel

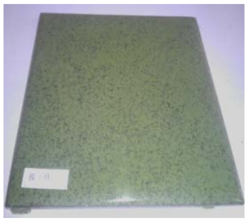
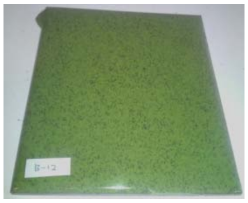
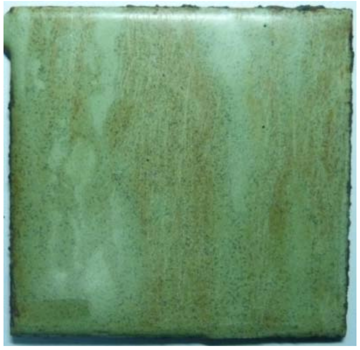
Sample	Exposure (day)
S-11	720
S-12	
S-13	
S-21	1440
S-22	
S-23	
S-31	2160
S-32	
S-33	

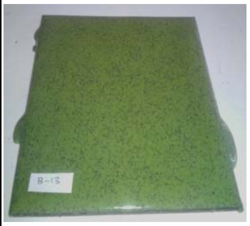
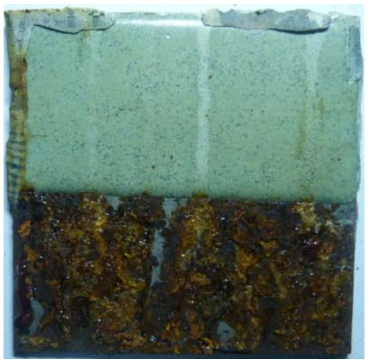
Table 8 Two side coated

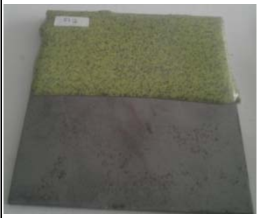
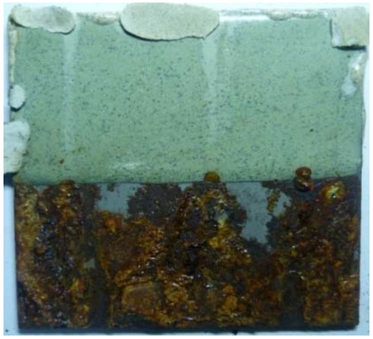
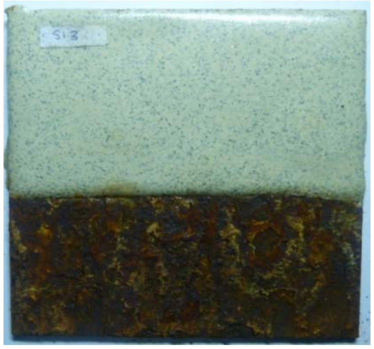
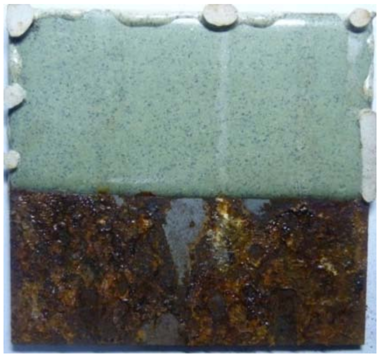
4.3.1 Exposure to Salt Spray Chamber (720 hours)

Sample	Before Exposure in Salt Spray Chamber	After Exposure in Salt Spray Chamber	Comments
A11		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached

A12		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached
A13		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached

B11		 Front  Back	• Thin Corrosion product forms at the uncoated side at 144 hours (6 days) • The coating is not detached
B12		 Front  Back	• Thin Corrosion product forms at the uncoated side at 144 hours (6 days) • The coating is not detached

B13		 Front  Back	• Thin Corrosion product forms at the uncoated side at 144 hours (6 days) • The coating is not detached
S11		 Front  Back	• Thin Corrosion product forms at the uncoated side at 144 hours (6 days) • The coating is not detached

S12		 Front  Back	• Thin Corrosion product forms at the uncoated side at 144 hours (6 days) • The coating is not detached
S13		 Front  Back	• Thin Corrosion product forms at the uncoated side at 144 hours (6 days) • The coating is not detached

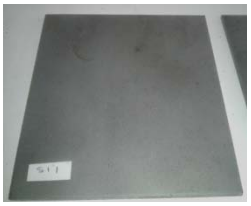
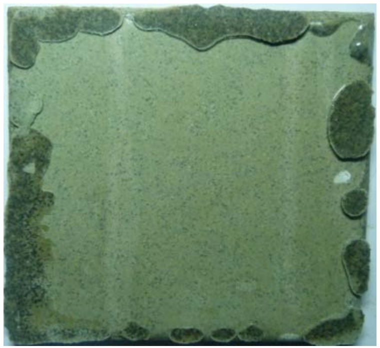
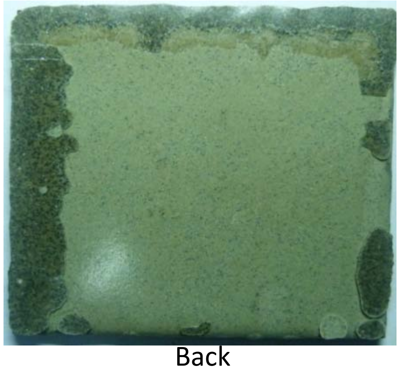
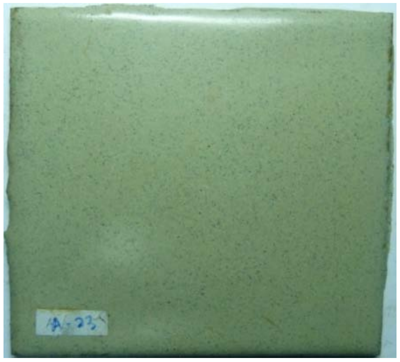
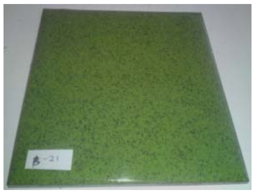
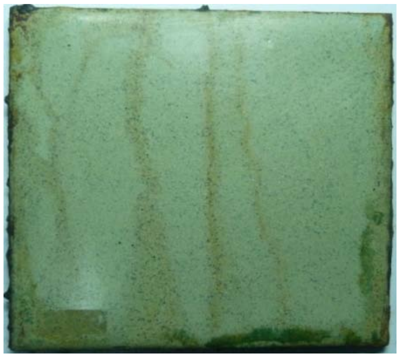
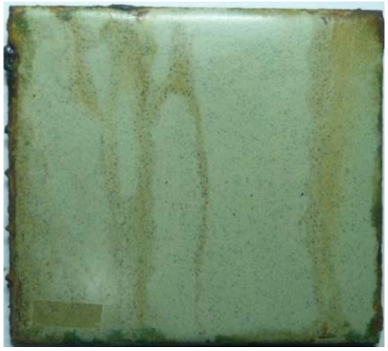
Steel without coating1		 Front  Back	- Thin corrosion Products form at both sides
Steel without coating2		 Front  Back	- Thin corrosion Products form at both sides - Uneven Corrosion products found at the back side of the uncoated steel

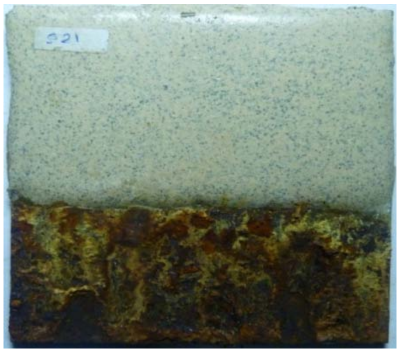
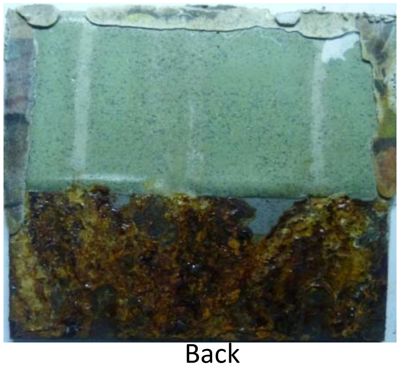
Table 9 Exposure to Salt Chamber in 720 Hours

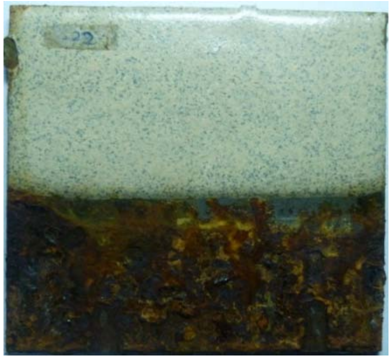
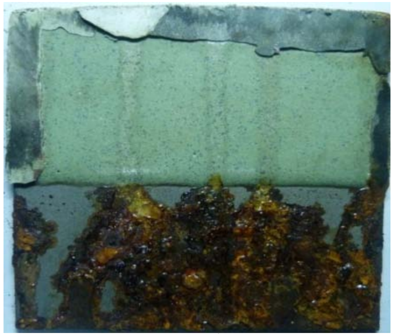
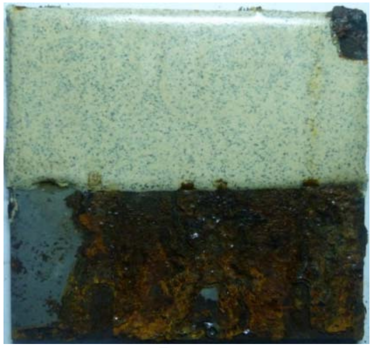
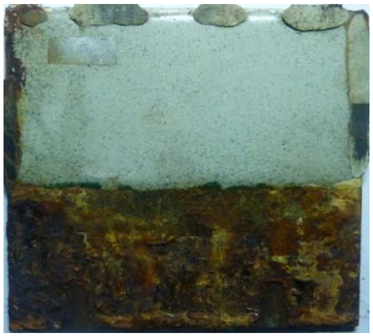
4.3.2 Exposure to Salt Spray Chamber (1440 hours)

Sample	**Before Exposure in Salt Spray Chamber**	**After Exposure in Salt Spray Chamber**	**Comments**
A21		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached

A22		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached
A23		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached

B21		 Front  Back	- Thick Corrosion product forms at the uncoated side - The coating is not detached
B22		 Front  Back	- Thick Corrosion product forms at the uncoated side - The coating is not detached

B23		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached
S21		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • Uneven corrosion found at one side of the coating

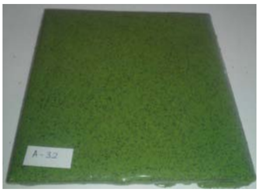
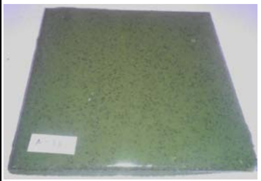
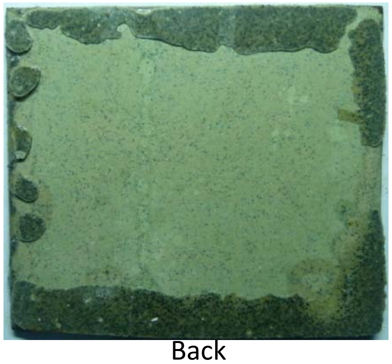
S22		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • Uneven corrosion found at one side of the coating
S23		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • Uneven corrosion found at one side of the coating

Steel without coating1		 Front  Back	- Thick corrosion Products form at both sides
Steel without coating2		 Front  Back	- Thick corrosion Products form at both sides

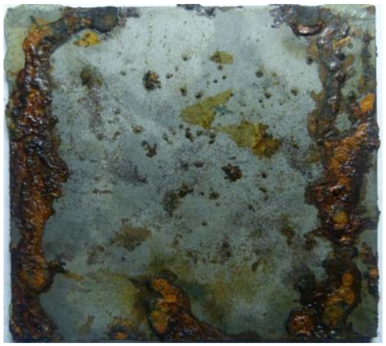
Table 10 Exposure to Salt Chamber in 1440 Hours

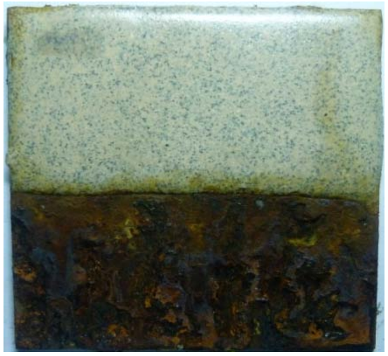
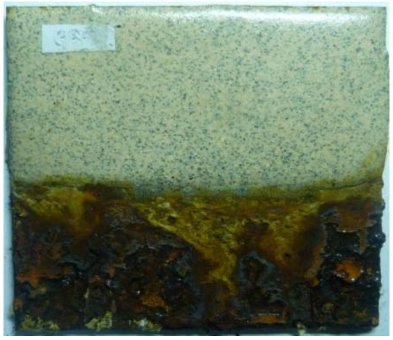
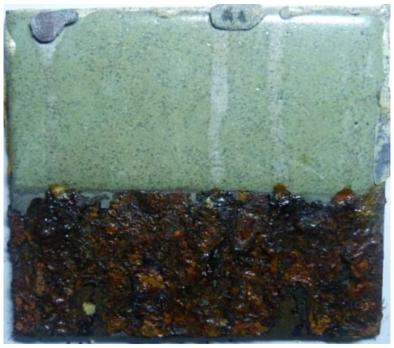
4.3.3 Exposure to Salt Spray Chamber (2160 hours)

Sample	Before Exposure in Salt Spray Chamber	After Exposure in Salt Spray Chamber	Comments
A31		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached

A32		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached
A33		 Front  Back	• No corrosion product form • Both side not corroded • The coating is not detached

B31		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • The corrosion product's fluid found at the coated side.
B32		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • The corrosion product's fluid found at the coated side.

B33		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • The corrosion product's fluid found at the coated side. • Uneven corrosion product found at the uncoated side.
S31		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached

S32		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • Uneven corrosion found at one side of the coating
S33		 Front  Back	• Thick Corrosion product forms at the uncoated side • The coating is not detached • Uneven corrosion found at one side of the coating

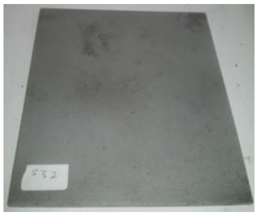
Steel without coating1		 Front  Back	- Thick corrosion Products form at both sides
Steel without coating2		 Front  Back	- Thick corrosion Products form at both sides

Table 11 Exposure to Salt Chamber in 2160 Hours

Table 9, Table 10 and Table 11 show the result of the sample exposure in salt spray chamber for duration of 720 hours, 1440 hours and 2160 hours respectively.

From Table 9, the thin corrosion product start to form in 6 days (144 hours). The corrosion product form at the uncoated side. The part that being coated observed unchanged. At the end of the 1440 hours exposure, the corrosion product forms at the entire of the uncoated side.

In Table 10, all the uncoated side is cover with corrosion product except some of the sample show uneven corrosion development. This is due to the position during exposure in salt spray chamber. The efficiency of salt water spray is not 100%. During the exposure, there are parts, that are not extremely exposed to salt water. As a result of the corrosion product form not even in a particular sample.

In Table 11, the corrosion product form is not much different with the exposure in previous period (1440 hours). Some of the sample show uneven corrosion development at the uncoated area.

4.4 Fire Testing (Bunsen Burner Test)

4.4.1 Thickness of The Expansion Char

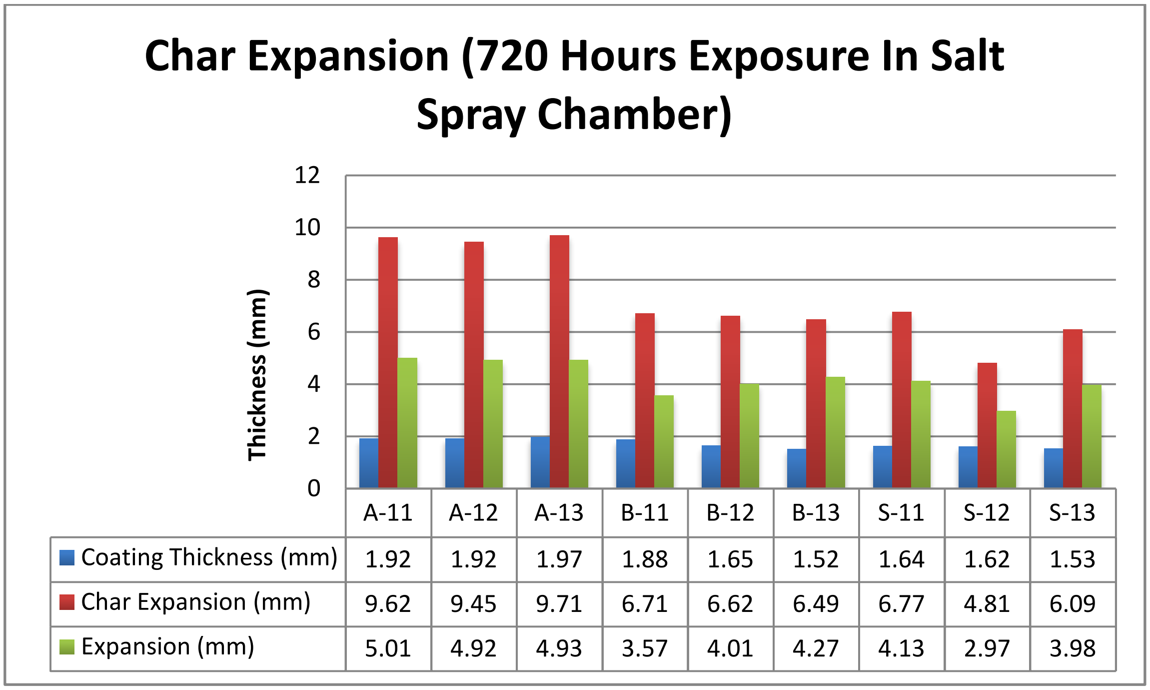
After fire test being carry out, the expansion of the char is measured as figure 6 below.

Figure 6 (a) Measuring The Thickness of The Expansion Char

Sample	Thickness (mm)		Expansion
	Before Fire Test (Coating Thickness)	After Fire Test (Char Expansion)	
A-11	1.92	11.00	5.73
A-12	1.92	10.48	5.46
A-13	1.97	9.71	4.93
B-11	1.88	6.71	3.57
B-12	1.65	6.01	3.64
B-13	1.52	6.49	4.27
S-11	1.64	6.77	4.13
S-12	1.62	4.81	2.97
S-13	1.53	5.81	3.8
A-21	1.99	12.34	6.2
A-22	1.92	11.46	5.97
A-23	1.94	12.28	6.33
B-21	1.82	9.83	5.4
B-22	1.67	8.27	4.95
B-23	1.96	9.21	4.7
S-21	1.98	9.70	4.9
S-22	1.95	11.31	5.8
S-23	1.84	7.99	4.34
A-31	1.93	10.23	5.3
A-32	1.93	8.70	4.51
A-33	1.94	12.18	6.28
B-31	1.83	10.83	5.92
B-32	1.79	9.13	5.1

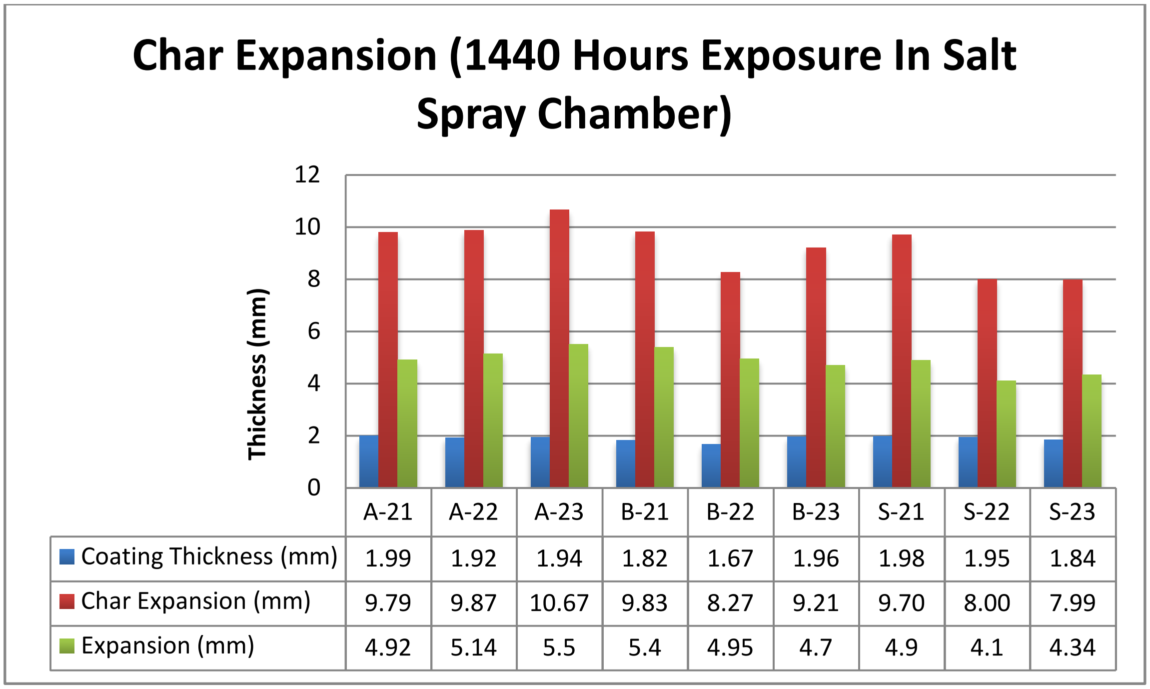
B-33	1.88	8.95	4.76
S-21	1.93	8.14	4.22
S-22	1.92	10.31	5.37
S-23	1.93	7.04	3.65

Table 4 Percentage of Char Expansion



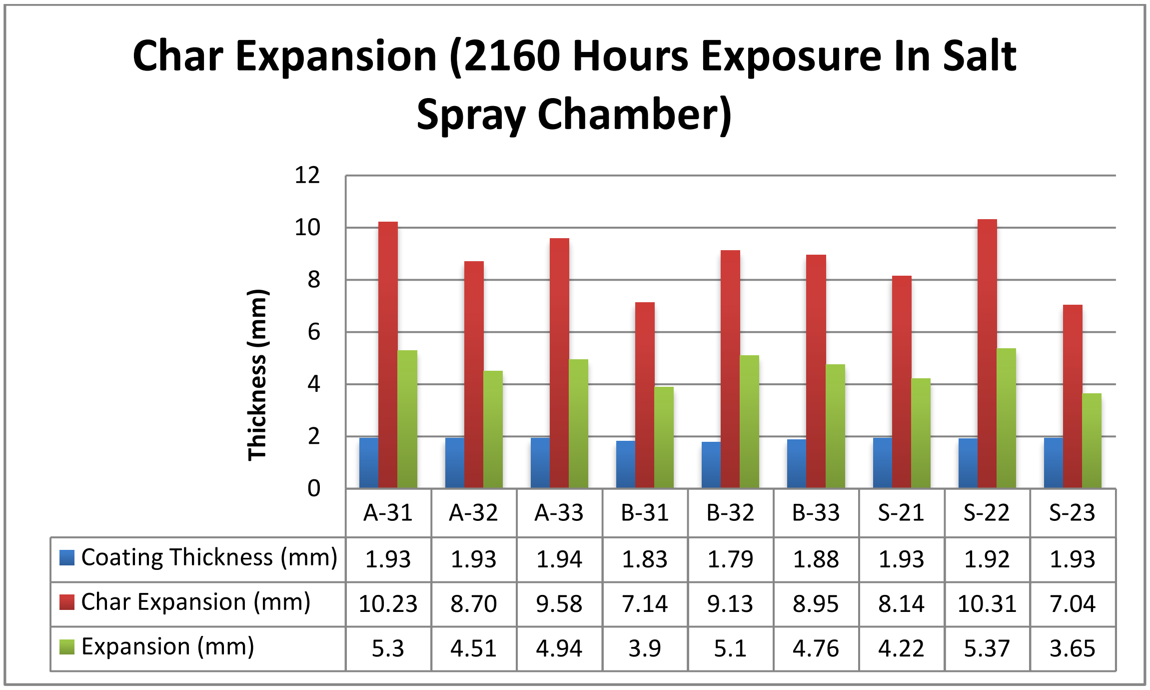
	A-11	A-12	A-13	B-11	B-12	B-13	S-11	S-12	S-13
🟦 Coating Thickness (mm)	1.92	1.92	1.97	1.88	1.65	1.52	1.64	1.62	1.53
🟥 Char Expansion (mm)	9.62	9.45	9.71	6.71	6.62	6.49	6.77	4.81	6.09
🟩 Expansion (mm)	5.01	4.92	4.93	3.57	4.01	4.27	4.13	2.97	3.98

Graph 1 Char Expansion (720 Hours Exposure In Salt Spray Chamber)



	A-21	A-22	A-23	B-21	B-22	B-23	S-21	S-22	S-23
🟦 Coating Thickness (mm)	1.99	1.92	1.94	1.82	1.67	1.96	1.98	1.95	1.84
🟥 Char Expansion (mm)	9.79	9.87	10.67	9.83	8.27	9.21	9.70	8.00	7.99
🟩 Expansion (mm)	4.92	5.14	5.5	5.4	4.95	4.7	4.9	4.1	4.34

Graph 2 Char Expansion (1440 Hours Exposure In Salt Spray Chamber)



	A-31	A-32	A-33	B-31	B-32	B-33	S-21	S-22	S-23
🟦 Coating Thickness (mm)	1.93	1.93	1.94	1.83	1.79	1.88	1.93	1.92	1.93
🟥 Char Expansion (mm)	10.23	8.70	9.58	7.14	9.13	8.95	8.14	10.31	7.04
🟩 Expansion (mm)	5.3	4.51	4.94	3.9	5.1	4.76	4.22	5.37	3.65

Graph 3 Char Expansion (2160 Hours Exposure In Salt Spray Chamber)

After fire test being carry out, the char formation thickness is measured as shown in Figure 6. Graph 1 shows the expansion of the char for the samples that being exposed in salt spray chamber for duration of 720 hours. Average expansion is 4.2 with lowest expansion of 2.97 and the highest expansion was 5.01

Graph 2 shows the char expansion for the samples that being exposed for 1440 hours in salt spray chamber. Average expansion was 4.88. Graph 3 shows the char expansion for the samples that being exposed in salt spray chamber for duration of 2160 hours)

Graph 3 shows the char expansion for the samples that being exposed for 2160 hours in salt spray chamber. Average expansion was 4.64.

The expansion of the char is very important in intumescent coating since it provides a shield that insulate the steel substrate from radiant heat and direct contact with the flame (Guoxin *et al*, 2007)

4.4.2 Heat Shielding Results

Temperature of the steel substrate is measured during the bunsen burner testing. The temperature is recorded for every 1 minutes until 60 minutes of testing. (see appendix)

4.4.2.1 Summary of Heat Shielding Effect Data

720 Hour Exposure to Salt Spray Test

	Sample	**Final Temperature (degree Celsius)**
Fully coated	A-11	164.11
	A-12	156.4
	A-13	160.25
One side coated	B-11	173.4
	B-12	211.63
	B-13	169.77
Two side coated (partially)	S-11	631.52
	S-12	551.3
	S-13	558.0
Uncoated	UC1	779.21
	UC2	783.5

Table 13 720 hours exposure

1440 Hours Exposure to Salt Spray Test

	Sample	**Final Temperature (degree celcius)**
Fully coated	A-21	155.43
	A-22	155.77
	A-23	158.67
One side coated	B-21	220.14
	B-22	181.5
	B-23	174.77
Two side coated (partially)	S-21	672.77
	S-22	665.3
	S-23	668.22
Uncoated	UC1	819.3
	UC2	821.56

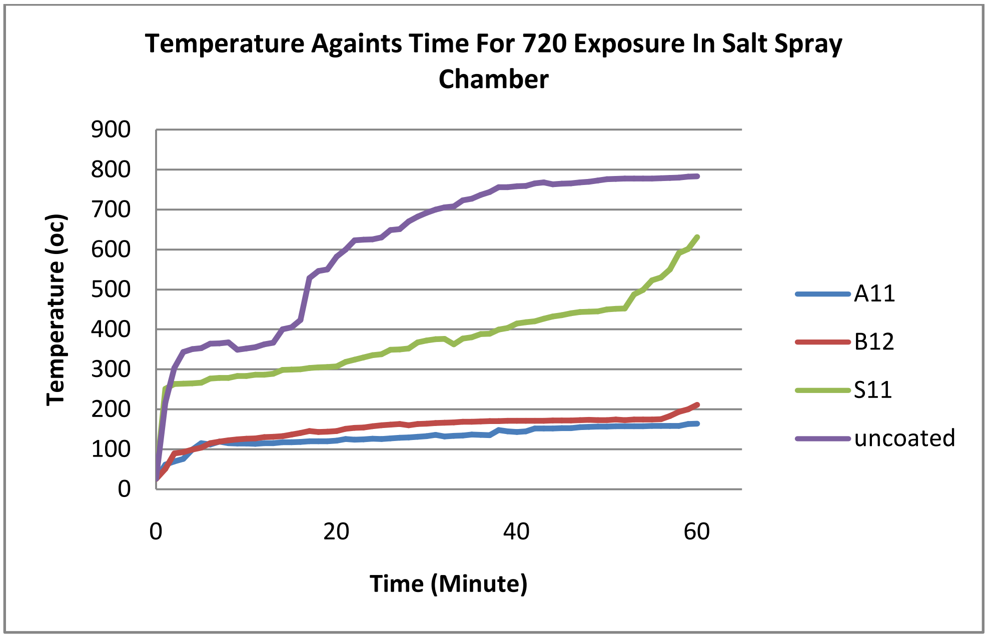
Table 5 1440 hours exposure

2160 Hours Exposure to Salt Spray Test

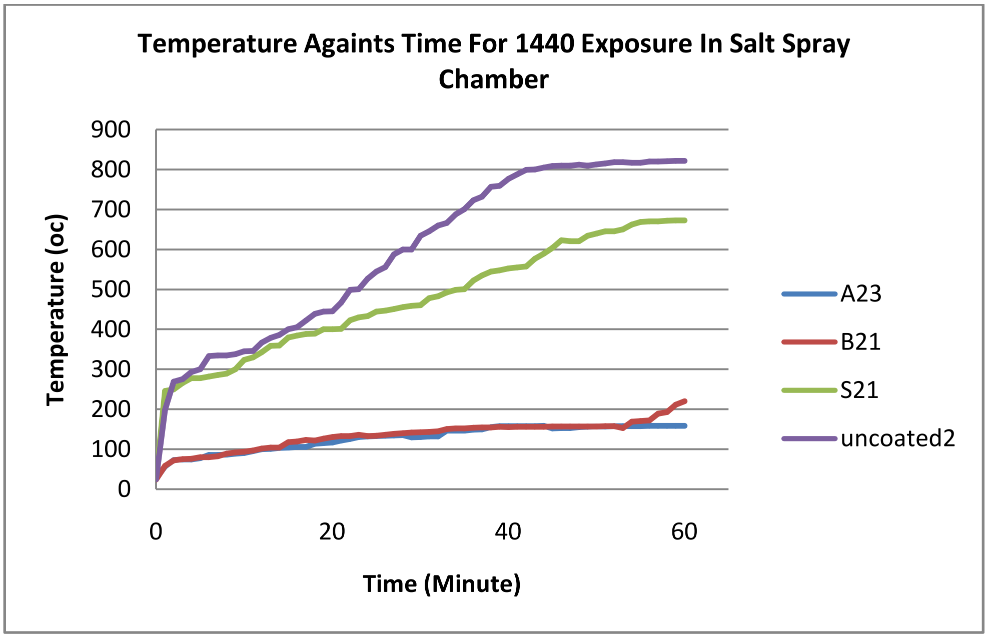
	Sample	**Final Temperature (degree celcius)**
Fully coated	A-31	157.1
	A-32	160.22
	A-33	161.50
One side coated	B-31	220.04
	B-32	187.51
	B-33	183.5
Two side coated (partially)	S-31	680.92
	S-32	676.33
	S-33	683.45
Uncoated	UC1	835.7
	UC2	768.9

Table 6 2160 hours exposure

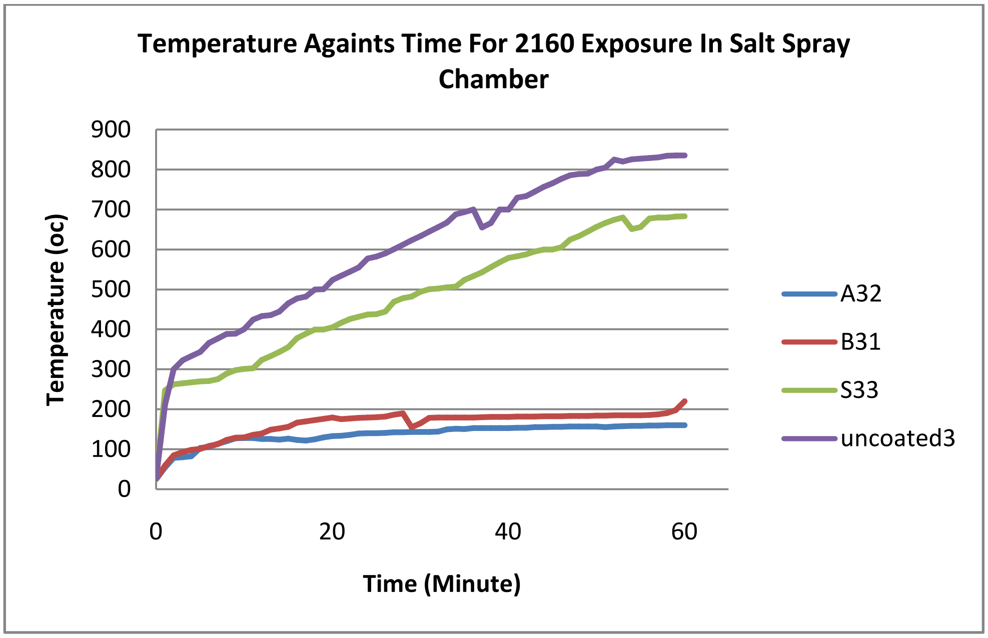
From the data collected in Table 9, Table 10 and Table 11, the highest temperature for each category and duration is taken for comparison. The graphs below show the comparison:



Graph 4 Temperature Againts Time For 720 Exposure In Salt Spray Chamber



Graph 5 Temperature Againts Time For 1440 Exposure In Salt Spray Chamber



Graph 6 Temperature Againts Time For 2160 Exposure In Salt Spray Chamber

During the testing, the temperature profile of the steel substrate is measured using digital thermo logger (Figure 5) and thermo couple. Steel critical temperature is 600^{o}C before its lost the mechanical integrity

From Graph 4, Graph 5 and Graph 6, it can be seen that the intumescent coated steel with fully coated show the lowest final temperature after 60 minutes of fire testing followed by one side coated steel. The fully coated shows the lowest final temperature because the backside of the steel is still protected with the intumescent coating during fire testing.

Steel with two side coated (partially) shows the final temperature exceeding the critical temperature. This is due to exposure of the steel as Figure 6 (b) below.

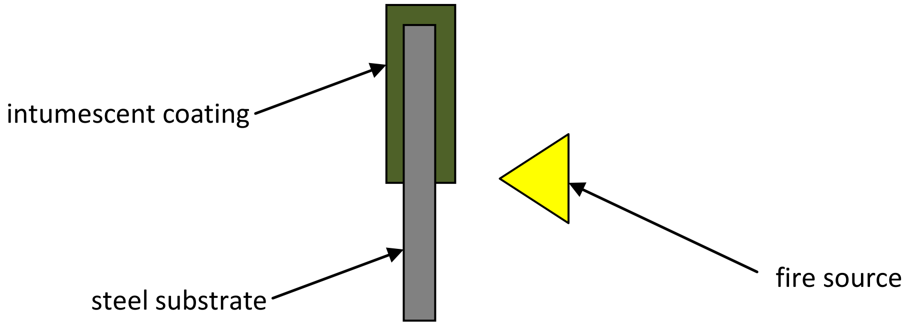


Figure 6 (b) Two side coated (partially) fire testing

Steel which at the front side is still exposed to the fire source. It lead to the temperature rise exceeding 600^{o}C. The uncoated steel shows the highest final temperature as it is not being protected with intumescent coating.

The temperature of the steel faced sudden decreased in value for couple of minutes. This is due to the char formation and the heat shielding effect. The experiment also conducted with the controlled air flow. Smoke suction chamber is installed during the fire testing to comply with HSE building regulation. Due to this suction effect, the temperature varies at the beginning of the fire testing.

4.5 X-Ray Diffraction Analysis

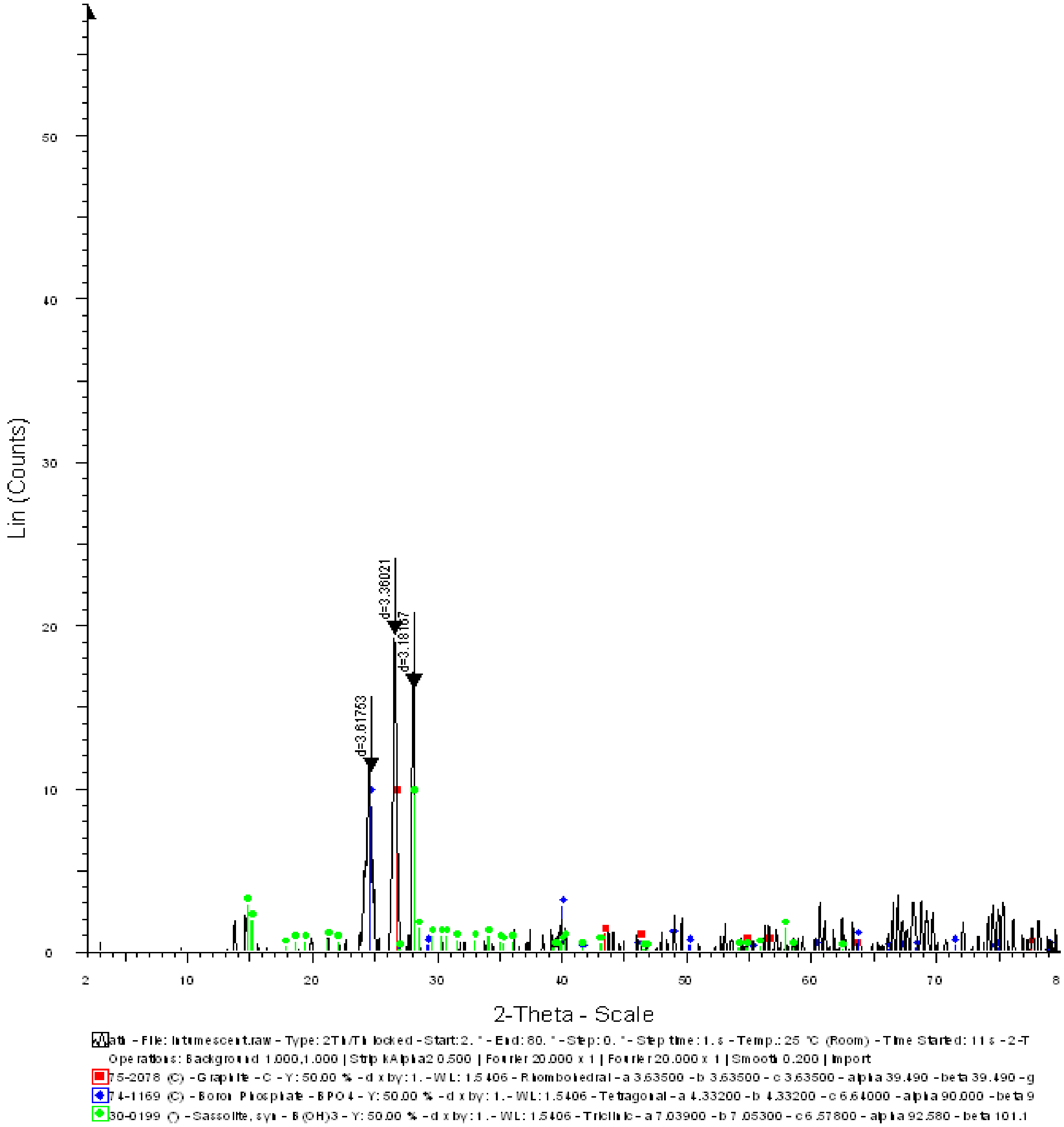


Figure 7 XRD Result of Char Formation

Figure 3 shows the XRD result of the residue char sample. Several peak was assigned using XRD software. Those elements shown higher peak in XRD result and give indicator that, the elements is dominant the composition of intumescent coating during burning process. The peak at 3.61753 assign to boron phosphate and 3.6021 to graphite (carbon). The peak at 3.318167 assigned to sassolite. The reaction between APP and boron oxide yield some boron phosphate [23]. The sassolite (mineral acid of boric acid H3B03) is due to the dehydration to support the formation of B_20_3, glass-like material which increase (Ahmad, Ullah, & Hamizol, 2012).

4.6 Scanning Electron Microscope (SEM) Analysis

4.6.1 Corrosion Product

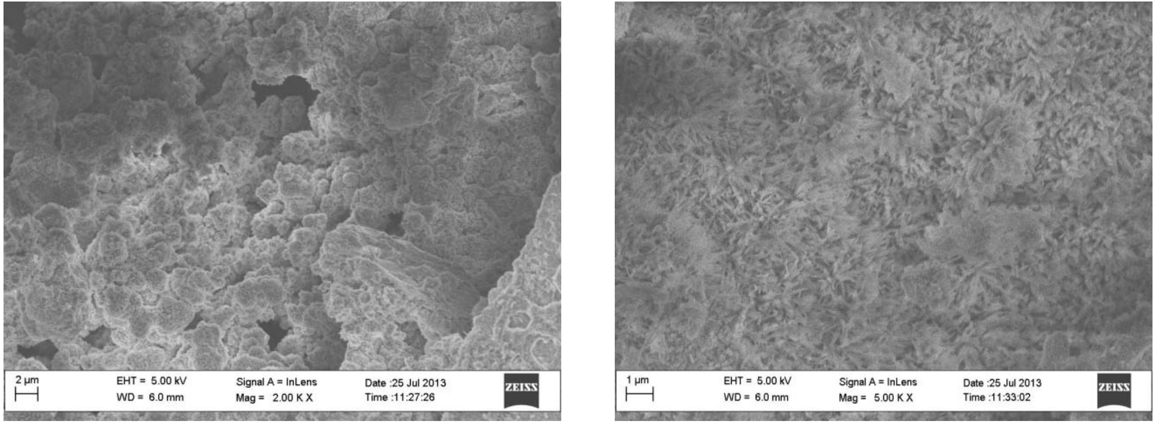


Figure 8 Electron Image of Corrosion Product After Exposure in Salt Spray Chamber (2000 & 5000 Magnification)

4.6.2 Char Morphology

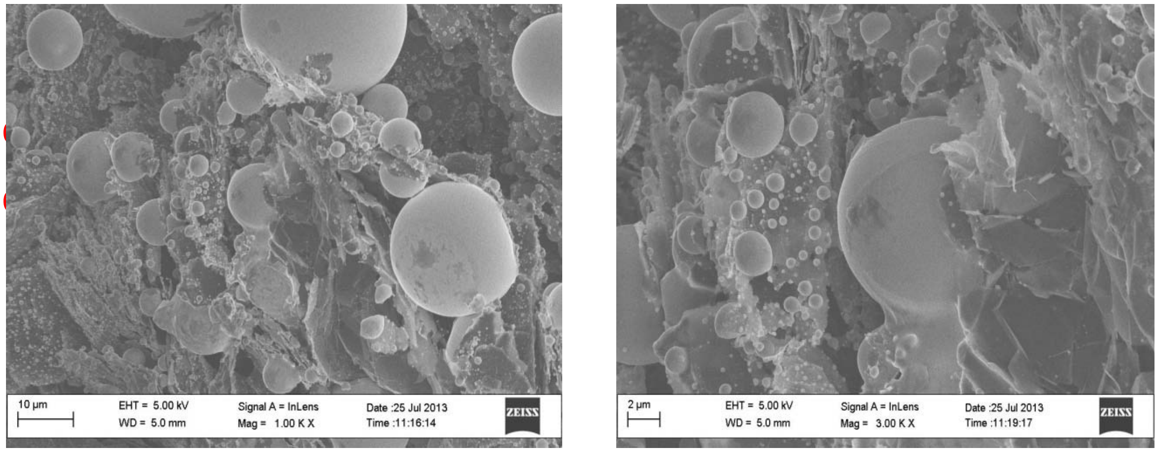


Figure 9 Electron Image of Char Formation (1000 & 3000 magnification)

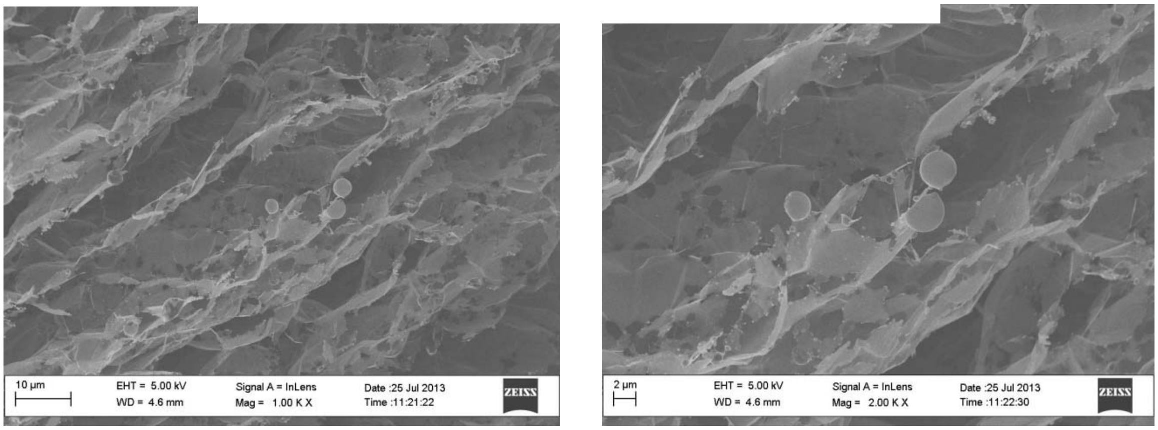


Figure 10 Electron Image of Char Formation (1000 & 2000 magnification)

From picture 10, it can be seen there is a lot of bubbles formation. The formation of bubbles is due to immersion of nitrogen, ammonia and carbon dioxide gas during exposure to fire. With higher magnification in Figure 5, the outer surface showed compact foam of char structure contain flakes and produce the heat barrier to protect steel (substrate). The multiporous char structure can block heat transferring to the steel substrate and protect the substrate from fire. (W.Farhana M, Faiz Ahmad, 2013). The expansion effect and char formation is very important to the fire resistance properties. (W.Farhana M, Faiz Ahmad, 2013).

However, in Figure 5 (b) , there are holes formation. The hole formation affect the cell structure where the heat transfer to the substrate will be increase.

CHAPTER 5

CONCLUSION AND DISCUSSION

5.1 Conclusion

The corrosion product growth at the uncoated part of the steel does not give any effect towards the fire retardant performance of intumescent coating. The steel is intentionally being not fully coated to represent the defect in coating. Thus to see the corrosion product spread toward the intumescent coating. The coating is exposed in salt spray chamber to simulate the sea water environment. The corrosion product only growth at the uncoated area of the steel samples. The coating is not detachable and remain at the substrate at the end of exposure period. It is shows that the corrosion and the sea water environment didnot give any effect towards the adhesion of the intumescent coated steel. The sample later being tested to proof for fire retardant performance of intumescent coating. The char expansion and heat shielding effect shows the fire testing result. From the result, it is shows that the coating is able to protect the steel under the high temperature although it being exposed to the salt water and corrosion product growth at the uncoated area. The XRD and FESEM result shows the presence of char element and morphology respectively. Therefore, there is no doubt that the corrosion growth at the uncoated or coating defect area didnot give any effect to the coating and not reducing the fire retardant performance of the intumescent coating.

5.2 Recommendation

It is recommended for the future improvement, the corrosion under insulation of the coated substrate being analyze to observe the under insulation effect. Other than that, the intumescent coating also should be tested in other area such as industrial area and the exposure towards sunlight and rain. The duration of the exposure can be increased so that the performance of intumescent coating can be evaluated better.

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