The Effect of Gypsum on the Performance of Intumescent Coating

by

Ahmad Syafiq Bin Md Suhaimy

Dissertation submitted in partial fulfilment of the requirements for the Bachelor of Engineering (Hons) (Mechanical Engineering)

JANUARY 2009

Universiti Teknologi PETRONAS Bandar Seri Iskandar 31750 Tronoh Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

The Effect of Gypsum on the Performance of Intumescent Coating

by

Ahmad Syafiq Bin Md Suhaimy

A project dissertation submitted to the Mechanical Engineering Programme Universiti Teknologi PETRONAS in partial fulfilment of the requirement for the BACHELOR OF ENGINEERING (Hons) (MECHANICAL ENGINEERING)

Approved by (AP Dr. Bambang Ariwahjoedi)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2009

t TH 1074.5 A 250 20009 i) Fire resistant polymens >) Intumescent 3) ME - Theses

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.

AHMAD SYAFIQ BIN MD SUHAIMY

ABSTRACT

This study investigates the effect of gypsum to the performance of intumescent coating. Intumescent or fire retardant coatings are one of the most efficient ways to protect materials against fire. In fact, it is generally known that acid source, carbon source, binding agent and blowing agent are the main ingredients of such a paint. In this project work, effect of using gypsum as additive in intumescent coating were investigated by performing fire test to sample coated with certain intumescent coating composition and by using Scanning Electron Microscope (SEM). All the results indicated that gypsum can increase fire retardation time which means intumescent coating modified with gypsum can withstand higher temperature and can last for longer time. This study and improvement of intumescent coating.

ACKNOWLEDGEMENT

The author wishes to take the opportunity to express her utmost gratitude to the individual that have taken the time and effort to assist the author in completing the project. Without the cooperation of these individuals, no doubt the author would have faced some minor complications through out the course.

First and foremost the author's utmost gratitude goes to the author's supervisor, AP Dr. Bambang Ariwahjoedi. Without his guidance and patience, the author would not be succeeded to complete the project.

To all the technician in Mechanical Engineering, Civil Engineering and Chemical Engineering Department, thank you for assisting the author in completing her project.

To all individuals that has helped the author in any way, but whose name is not mentioned here, the author thank you all.

TABLE OF CONTENTS

CERTIFICATIO	N OF AP	PROVAL	• .	•	-	•	i
CERTIFICATIO	N OF OR	RIGINALITY	ζ.	•	•	•	ii
ABSTRACT .	•			•	•	•	iii
ACKNOWLEDG	EMENT		•	•	•	•	iv
CHAPTER 1:	INTR	ODUCTION	Ι.	•	•	•	1
	1.1	Background	l Project	•	•	•	1
	1.2	Problem Sta	atement	•	•	•	2
	1.3	Objectives		•	•	•	2
	1.4	Scope of St	udy	•	•	•	2
CHAPTER 2:	LITE	RATURE R	EVIEW	· •	•	٠	3
	2.1	Intumescent	t.	-	•	•	4
	2.2	Intumescen	t Coating	g.	•	•	6
	2.3	Mechanism	of Intur	nescen	t Coatir	ng.	7
	2.4	Gypsum	•	•	•	•	9
	2.5	Dehydration	n Reactio	ons	•	-	11
	2.6	Pentaerythr	itol.	•	•	•	13
	2.7	Boric Acid	•	•	•	•	14
	2.8	Melamine	•	•	•	•	15
	2.9	Epoxy Resi	n.	•	•	•	15
CHAPTER 3:	MET	HODOLOG	Υ.	•		•	16
	3.1	Project Flow	<i>N</i> .		•	•	16
	3.2	Preparation	of Trial	Formu	la.	•	17
	3.3	Fire Test		•	•	•	19
	3.4	Scanning E	lectron M	Microso	cope	•	20
	3.5	Tools .	•	•	•	•	22
CHAPTER 4:	RESU	ULTS AND I	DISCUS	SION	•	•	23
	4.1	Height of C	har Proc	luced	•	•	23
	4.2	Upper Surfa	ace Tem	peratur	e Meas	urement	25
	4.3	SEM Analy	rsis	•	•	•	26
CHAPTER 5:	CON	CLUSION A	ND RE	COMN	/IEND/	ATIONS	S 28
	5.1	Conclusion	•	•	•	•	28
	5.2	Recommen	dations	•	•	•	29

REFERENCES	•	•	•	•	•	•	•	30
APPENDIX .	•	•	•	•	•	•	•	32

. .

LIST OF FIGURES

Figure 1	Soft char producers	5
Figure 2	Hard expanding char producers	5
Figure 3	Mechanism of intumescent coating	7
Figure 4	Intumescence process	8
Figure 5	Project flow	16
Figure 6	Bunsen burner test	19
Figure 7	Scanning Electron Microscopy	20
Figure 8	Structures of charring layer	21
Figure 9	Graph on height of char produced vs. percentage of gypsum	24
Figure 10	SEM micrographs of residue chars from Sample A	26
Figure 11	SEM micrographs of residue chars from Sample E	22

LIST OF TABLES

Table 1	Components of trial formula	17
Table 2	Coating composition for sample A, B, C, D, and E	18
Table 3	Project tools	22
Table 4	Height of char produced	23
Table 5	Upper surface temperature of each sample	25
Table 6	Gantt chart for semester 1	32
Table 7	Gantt chart for semester 2	32

LIST OF ABBREVIATIONS

- APP Ammonium Polyphosphate
- PETN Pentaerythritol tetranitrate
- PVC Polyvinyl chloride
- PCB Polychlorobiphenyl
- SEM Scanning Electron Microscope
- XRD X-Ray Diffraction

.

TGA Thermogravimetric Analysis

CHAPTER 1 INTRODUCTION

1.1 Background Project

The use of fire retardant coatings is one of the easiest, one of the oldest and one of the most efficient ways to protect materials against fire. Indeed, it presents several advantages which it does not modify the intrinsic properties of the materials such as the mechanical properties; it is easily processed and may be used onto several materials such as metallic materials, polymers, textiles and wood. Moreover, since ignition occurs usually on the surface of a material it is important to concentrate the protective action at this place.

The protection of metallic materials against fire has become an important issue in the construction industry. Indeed, in case of fire such materials distort leading to the collapse of building structures, and as a consequence, to dramatic human and economical losses.

Intumescent paints are a way to achieve such protection. Fire retardant coatings, acting by the phenomena of intumescence, form on heating an expanded multicellular layer, which acts as thermal barrier. It prevents heat from penetrating and flames from spreading. As a consequence, this insulative barrier makes intumescent coatings particularly suitable for the protection of structural steelwork.

1.2 Problem Statement

Intumescent coatings are typically used in passive fire protection, firestopping and gasketting applications. However some intumescents are susceptible to environmental influences such as humidity which can reduce or negate their ability to function. So, this means that some improvement must be made to this existing intumescent coating. Although there are many type of intumescent coating already available at the market, there is still no intumescent coating which adopts gypsum as additive. Since the physical properties of gypsum that has a very low thermal conductivity and other unique characteristic of gypsum which is its dehydration reaction, it is believe that gypsum can be an additive to intumescent coating to improve its performance. Furthermore, gypsum is common mineral and easy to find. This finding would be useful in intumescent coating industry.

1.3 Objective

The main objectives of this research are:

- Prepare trial formula of typical intumescent coating.
- Establish the fire test on the trial formula
- Study the effect of gypsum as an additive

1.4 Scope Of Study

The scope of work of this project is to study the effect of gypsum as an additive to the performance of intumescent coating. Trial formulation will be prepared by imitating the formula of existing intumescent coating which can be found in the literature. Fire test will be conducted on the trial formulation to investigate the height of char produced after fire testing. Result from this fire test will be analyzed to study the effect of gypsum as an additive to the performance of intumescent coating.

CHAPTER 2 LITERATURE REVIEW

2.1 Intumescent

An intumescent is a substance which swells as a result of heat exposure, thus increasing in volume, and decreasing in density. Intumescents are typically used in passive fire protection and, in America, require listing and approval use and compliance in their installed configurations in order to comply with the law [1]. There are two types of intumescent coating which are soft char producers and hard expanding char.

Soft char producers:

These intumescents produce a light char, which is a poor conductor of heat, thus retarding heat transfer. Typically, these materials also contain a significant amount of hydrates. As the hydrates are spent, water vapour is released, which has a cooling effect. Once the water is spent, it is only the insulation characteristics of the char that was produced, which can slow down heat transfer from the exposed side to the unexposed side of an assembly. Soft char producers are typically used in thin film intumescents for fireproofing of structural steel as well as firestop pillows. Typically, the expansion pressure that is created for these products is very low, because the soft carbonaceous char has little substance, which is beneficial if the aim is to produce a layer of insulation. [1]. Picture of soft char produced is shown in Figure 1.

• Hard expanding char producers:

Harder chars are produced with sodium silicates and graphite. These products are suitable for use in plastic pipe firestops as well as exterior steel fireproofing. In those applications, it is necessary to produce a more substantial char, with a quantifiable expansion pressure. In the case of the firestops, a melting, burning plastic pipe must be squeezed together and shut so that there will be no hole for fire to go through an opening in an otherwise fire-resistance rated wall or floor assembly. In the case of the exterior fireproofing, a hydrocarbon fire must be held off with quite potentially more kinetic energy than a house fire. Intumescents that produce hard chars are not typically used for interior spray fireproofing as they are not suitable for that application [1]. Picture of hard expanding char produced is shown in Figure 2.

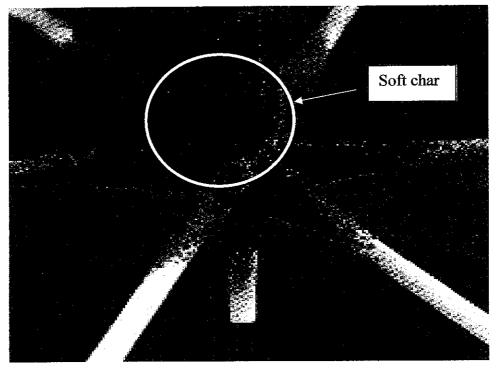


Figure 1: Soft char producers [1]

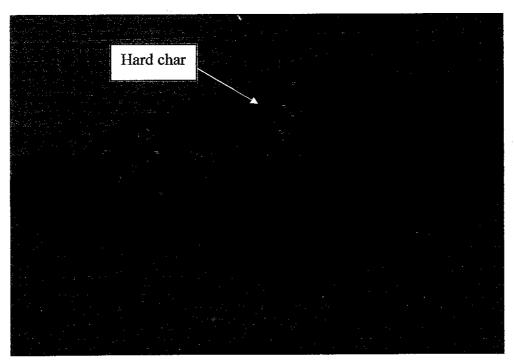


Figure 2: Hard expanding char producers [1]

2.2 Intumescent Coating

Intumescent coatings provide an appearance similar to that of a paint finish, and remain stable at ambient temperatures. However, in a fire situation the increase in temperature causes a chemical reaction, and the intumescent coating expands to many times its original thickness. This provides an insulating foam-like coating or 'char' which protects the substrate. Intumescent coating used generally in protecting steelwork where the intumescent coating is designed to insulate the steel, and prevent the temperature of the steel from rising above a certain point. Second intumescent also used in protecting timber structures where intumescents are designed to reduce heat propagation, and reduce the spread of flame because of timber structures are more susceptible to the surface spread of flame and heat propagation. This is important because in case of fire, such materials distort leading to the collapse of building structures, and as a consequence, to dramatic human and economical losses [2]. The expansion process is caused by the interaction of three precisely formulated components:

- Carbon Supplier: Such as (di) pentaerythritol
- Acid Source: Generally ammonium polyphosphate (APP)
- Expanding/Blowing Agent: Most often melamine

All this three components are link together by a binder.

2.3 Mechanism of Intumescent Coating

As heat is applied the chemical reaction begins, the heat begins to soften the polymeric binder. The heat also causes a release of an organic acid from the Ammonia Polyphosphate. Then Carbonisation of the polyols begins to take place. Gas is then produced through the decomposition of the blowing agent (melamine), swelling the molten mixrure. The final stage is solidification of the foamed char, through a cross-linking reaction, to maintain the insulation [2].

In an ideal situation the intumescent can expand some 100 times its original thickness. This requires the precise matching of various processes, which is achieved through careful selection of the formulation components [2]. The mechanism of intumescent coating is shown in Figure 3.

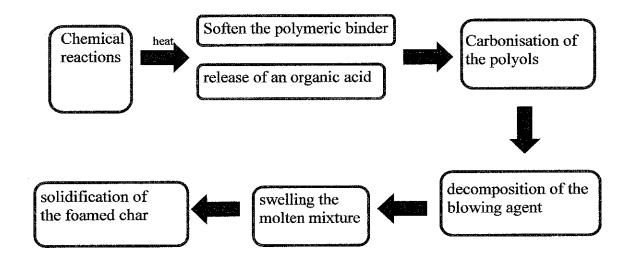


Figure 3: Mechanism of intumescent coating

The formation of the effective char occurs via a semi-liquid phase, which coincides with gas formation and expansion of the surface. Gases released from the degradation of the intumescent material, and in particular of the blowing agent, have to be trapped and to diffuse slowly in the highly viscous melt degraded material in order to create a layer with appropriate morphological properties as shown in Figure 4 [2].

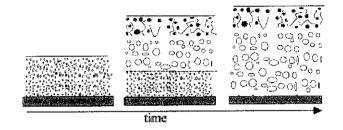


Figure 4: Intumescence process [2]

2.4 Gypsum

Gypsum is a very soft mineral composed of calcium sulfate dihydrate, with the chemical formula $CaSO_4 \cdot 2H_2O$. Gypsum, the most common of the sulphate minerals, is also known as satin spar, alabaster, or selenite, which is a colourless and transparent variety of gypsum. In the gypsum crystal structure, calciums are coordinated by six oxygens from sulphate (SO₄), and by two oxygens from water (H₂O). Two sheets of sulphates are bound together by calciums forming double sheet layers. At each side of these layers are water molecules, which form weak hydrogen bonds to the next layer in the structure [11].

There is a considerable research interest in the study of structure and coordination behaviour of water molecules in gypsum (calcium sulphate dihydrate, $CaSO_4 \cdot 2H_2O$) and hemihydrate (plaster of Paris, $CaSO_4 \cdot 1/2H_2O$). A wide range of opinions concerning the dehydration of gypsum and various ideas relating to its mechanism are well known [11].

The chemistry of the gypsum transformation products on heating is still, despite extensive work, not entirely agreed upon. It is claimed that heating gypsum below 100 °C gives rise to limiting value of weight loss around that of the hemihydrate formation indifferent of the length of heating time. Calcination above 100 °C causes a continuous loss in weight as the time is prolonged, leading to anhydrite (CaSO₄). Hamad [13] has reported the presence of molecular hydrogen and hydrogen-bonded water molecules in the gypsum and hemihydrate structures. There has been considerable controversy on the bonding of water in gypsum and hemihydrate. The crystal structure of gypsum was determined by X-ray data by several workers and it was suggested that the water molecules in gypsum are asymmetrical [13]. Some suggested that gypsum has a layer structure [13]. However, the presence of anion water in gypsum has not been discussed so far. The water, which is coordinated through anion of a compound via hydrogen bonding, is called anion water [13].

Due to presence of anion water, the water molecule is held strongly and difficult to remove from the parent compound due to which more heat energy is required to release this water molecule. In case of gypsum, the anion water is removed through heating nearly by 350 °C.

The reason why gypsum is chosen in this project is because of its dehydration reactions. The dehydration (specifically known as calcination) begins at approximately 80 °C (176 °F), although in dry air, some dehydration will take place already at 50 °C. The heat energy delivered to the gypsum at this time (the heat of hydration) tends to go into driving off water (as water vapor) rather than increasing the temperature of the mineral, which rises slowly until the water is gone, then increases more rapidly[4].

The endothermic property of this reaction is exploited by drywall to confer fire resistance to residential and other structures. In contrast to most minerals, which when rehydrated simply form liquid or semi-liquid pastes, or remain powdery, calcined gypsum has an unusual property: when mixed with water at normal (ambient) temperatures, it quickly reverts chemically to the preferred dihydrate form [11].

2.5 Dehydration reactions

Heating gypsum to between 100 °C and 150 °C (302 °F) partially dehydrates the mineral by driving off approximately 75% of the water contained in its chemical structure. The temperature and time needed depend on ambient partial pressure of H₂O. Temperatures as high as 170 °C are used in industrial calcination, but at these temperatures γ -anhydrite begins to form [6]. The reaction for the partial dehydration is:

$$CaSO_4 \cdot 2H_2O + heat \rightarrow CaSO_4 \cdot \frac{1}{2}H_2O + \frac{1}{2}H_2O \text{ (steam)}$$
[6]

The partially dehydrated mineral is called calcium sulfate hemihydrate or calcined gypsum (commonly known as plaster of Paris) (CaSO₄·nH₂O), where n is in the range 0.5 to 0.8 [6].

The dehydration (specifically known as calcination) begins at approximately 80 °C (176 °F), although in dry air, some dehydration will take place already at 50 °C. The heat energy delivered to the gypsum at this time (the heat of hydration) tends to go into driving off water (as water vapor) rather than increasing the temperature of the mineral, which rises slowly until the water is gone, then increases more rapidly [6].

The endothermic property of this reaction is exploited by drywall to confer fire resistance to residential and other structures. In a fire, the structure behind a sheet of drywall will remain relatively cool as water is lost from the gypsum, thus preventing (or substantially retarding) damage to the framing (through combustion of wood members or loss of strength of steel at high temperatures) and consequent structural collapse [6].

In contrast to most minerals, which when rehydrated simply form liquid or semi-liquid pastes, or remain powdery, calcined gypsum has an unusual property: when mixed with water at normal (ambient) temperatures, it quickly reverts chemically to the preferred dihydrate form, while physically "setting" to form a rigid and relatively strong gypsum crystal lattice:

$$CaSO_4 \cdot \frac{1}{2}H_2O + \frac{1}{2}H_2O \rightarrow CaSO_4 \cdot 2H_2O$$
[6]

This reaction is exothermic and is responsible for the ease with which gypsum can be cast into various shapes including sheets (for drywall), sticks (for blackboard chalk), and molds (to immobilize broken bones, or for metal casting). Mixed with polymers, it has been used as a bone repair cement. Small amounts of calcined gypsum are added to earth to create strong structures directly from cast earth, an alternative to adobe (which loses its strength when wet). The conditions of dehydration can be changed to adjust the porosity of the hemihydrate, resulting in the so-called alpha and beta hemihydrates (which are more or less chemically identical) [6].

On heating to 180 °C, the nearly water-free form, called γ -anhydrite (CaSO₄.nH₂O where n=0 to 0.05) is produced. γ -Anhydrite reacts slowly with water to return to the dihydrate state, a property exploited in some commercial desiccants. On heating above 250 °C, the completely anhydrous form called β -anhydrite or "natural" anhydrite is formed. Natural anhydrite does not react with water, even over geological timescales, unless very finely ground [6].

The variable composition of the hemihydrate and γ -anhydrite, and their easy interconversion, is due to their possessing nearly-identical crystal structures, containing "channels" that can accommodate variable amounts of water, or other small molecules such as methanol [6].

2.6 Pentaerythritol

Pentaerythritol is the organic compound with the formula $C(CH_2OH)_4$. This white, crystalline polyol is a versatile building block for the preparation of many polyfunctionalized compounds such as the explosive PETN and pentaerythritol triacrylate^[1]. Derivatives of pentaerythritol are components of alkyd resins, varnishes, PVC stabilizers, tall oil esters, and olefin antioxidants [8].

PVC stabilizers, tall oil esters, and olefin antioxidants.Halogen-free pentaerythritol esters are also environmentally friendly alternative to conventional electrical transformer fluids, being both readily biodegradable and non-hazardous in water. They advantageously replace polychlorobiphenyl (PCB), and even silicone-based or fluorinated hydrocarbons, as dielectric fluid in transformers. Their low volatility and high flash point give them an excellent resistance to ignition in case of major electrical failure and transformer rupture [8].

2.7 Boric Acid

Boric acid, also called boracic acid or orthoboric acid or Acidum Boricum, is a weak acid often used as an antiseptic, insecticide, flame retardant, in nuclear power plants to control the fission rate of uranium, and as a precursor of other chemical compounds. It exists in the form of colorless crystals or a white powder and dissolves in water. It has the chemical formula H_3BO_3 , sometimes written $B(OH)_3$. When occurring as a mineral, it is called sassolite [9].

Boric acid was first prepared by Wilhelm Homberg (1652-1715) from borax, by the action of mineral acids, and was given the name sal sedativum Hombergi ("sedative salt of Homberg"). The presence of boric acid or its salts has been noted in seawater. It is also said to exist in plants and especially in almost all fruits.^[2] The free acid is found native in certain volcanic districts such as Tuscany, the Lipari Islands and Nevada, issuing mixed with steam from fissures in the ground; it is also found as a constituent of many minerals (borax, boracite, boronatrocaicite and colemanite). Boric acid is soluble in boiling water. When heated above 170 °C it dehydrates, forming metaboric acid HBO₂. Metaboric acid is a white, cubic crystalline solid and is only slightly soluble in water. It melts at about 236 °C, and when heated above about 300 °C further dehydrates, forming tetraboric acid or pyroboric acid, H₂B₄O₇. Boric acid can refer to any of these compounds. Further heating leads to boron trioxide [9].

2.8 Melamine

Melamine is an organic base and a trimer of cyanamide, with a 1,3,5-triazine skeleton. Like cyanamide, it contains 66% nitrogen by mass and, if mixed with resins, has fire retardant properties due to its release of nitrogen gas when burned or charred, and has several other industrial uses. Melamine is also a metabolite of cyromazine, a pesticide. It is formed in the body of mammals who have ingested cyromazine . It has been reported that cyromazine can also be converted to melamine in plants [7].

Melamine combines with cyanuric acid to form melamine cyanurate, which has been implicated in the Chinese protein export contaminations [7].

2.9 Epoxy resin

In chemistry, epoxy or polyepoxide is a thermosetting epoxide polymer that cures (polymerizes and crosslinks) when mixed with a catalyzing agent or hardener [10]. Epoxy resins used as binder agent in the intumescent coating.

CHAPTER 3 METHODOLOGY

3.1 Project Flow

This project can be divided into two parts, part one and part two. The first part of this project is to gather all information and data that are related to the project. Information such as basic knowledge of intumescent coating, preparation of trial formula and components in intumescent coating are important because this trial formula will be use for fire test and further study on the effect if gypsum as additive. The flow of the project is shown in Figure 5.

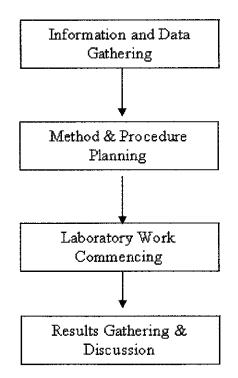


Figure 5: Project flow

3.2 Preparation of Trial Formulation

Trial formula for this project was prepared by imitating it from journal. Various journals have been viewed in order to come out with the most suitable and available components to produce the coating with correct concentration. Table 1 shows the components of trial formulation with its specific percentage:

12.20
12.20
28.40
18.70
31.70
9.00 max
-

Table 1: Components of trial formula

- Pentaerythritol used as carbon supplier.
- Boric acid used as acid source.
- Melamine used as blowing agent.
- Epoxy resin used as binder.
- Gypsum used as additive.

All the components are mixed together in a mixer.

5 samples which are Sample A, B, C, D and E were prepared. 5 steel plates with 150mm x 150mm and 3mm thickness was prepared. These samples then were coated and let to dry for 3 days. The composition of the coating for each sample is shown in Table 2.

Sample Coating Components							
Sample A	Epoxy resin						
	Boric acid						
	Melamine						
	• Pentaerythritol						
Sample B	Epoxy resin						
	Boric acid						
	Melamine						
	Pentaerythritol						
	• 1 % Gypsum						
Sample C	Epoxy resin						
	Boric acid						
	Melamine						
	• Pentaerythritol						
	• 3% Gypsum						
Sample D	Epoxy resin						
	Boric acid						
	Melamine						
	Pentaerythritol						
	• 6 % Gypsum						
Sample E	Epoxy resin						
	Boric acid						
	Melamine						
	• Pentaerythritol						
	• 9% Gypsum						

Table 2: Coating composition for sample A, B, C, D and E

3.3 Fire Test

Effect of gypsum as additive to performance of intumescent coating can be investigated by performing fire test. Bunsen burner test was carried out on these 5 samples, sample A, B, C, D, and E. The intumescent performance of the coating were determined by exposing the coated side of 150×150 mm steel plate to the effect of a gas burner with a flame temperature of 1000 °C for 25 minutes. The steel plate was hold by a holder to make it hanged. The visual aid of the process is shown in Figure 6.

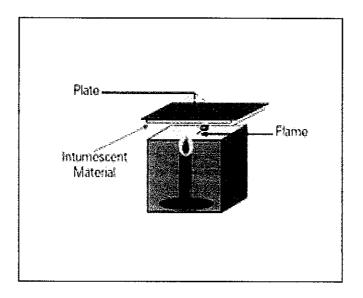


Figure 6: Bunsen burner test [3]

3.4 Scanning Electron Microscope (SEM)

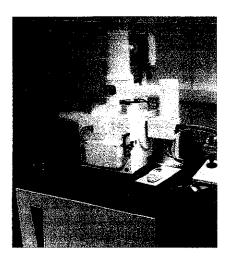


Figure 7: Scanning Electron Microscope

Scanning Electron Microscope (SEM) used to images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. SEM is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a light microscope. Scanning Electron Microscopy is shown in Figure 7.

The charring layer protects the steel materials, and its protective property depends on the physical and chemical structure of the charring layer. Researches indicate that there are other elements in the charring-layer structure. But the non-charring element is easy to be oxygenated and very unstable on chemistry aspects. There are two ideal typical charring structures which are shown in Figure 8.

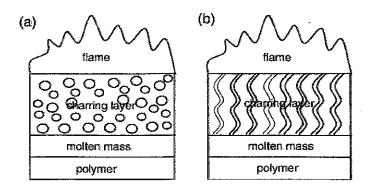


Figure 8: Structures of charring layer: (a) uniformity and (b) asymmetry [3]

There are a great deal of integrated closed honeycomb pores in Figure 8(a); such structure can form adequate temperature grads in the charring layer and protect the molten mass and matrix below. But the other structure is nonideal; there are many channels and apertures, and the gas and molten mass of polymers can overflow to the entry of the flame-region. Thus the isolation effect of heat transfer is inferior. The physical structure of the charring layer plays a very important role in the performance of the flame retardant.

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification. Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive. The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today.

3.5 Tools

Table 3 shows the equipment and tools required for project work. All the equipments are available in the laboratory.

Tools	Description
Small mixer	Equipment used to mix the components.
Thermometer	Used to measure temperature of upper surface of the samples.
Bunsen Burner	Equipment used as heat source for fire test.
Caliper	Used to measured thickness of samples.
Optical Microscope	Magnifies an image.
Scanning Electron	Images the sample surface by scanning it
Microscope (SEM)	with a high-energy beam of electrons in a raster scan pattern.

Table 3: Project tools

CHAPTER 4 RESULTS AND DISCUSSION

4.1 Height of Char Produced

Height of Sample A, Sample B, Sample C, Sample D and Sample E were measured before fire test being applied to them. The height measured is the total height of steel plate with coating. The height of samples before fire test is about 4mm. This means the coating thickness before the fire test is 1mm approximately because the thickness of metal plate is 3mm. After exposed to Bunsen burner flame for 25 minutes, thicknesses of the samples were measured once again to determine the new thickness which is the thickness of samples with char layer produced as a result of fire test. Height of char produced can be measured by measuring the thickness of samples. Result is shown in Table 4:

Sample	Height (mm)
Sample A	7.2
Sample B	5.7
Sample C	5.0
Sample D	4.5
Sample E	3.8

Table 4:	Height	of char	produced
----------	--------	---------	----------

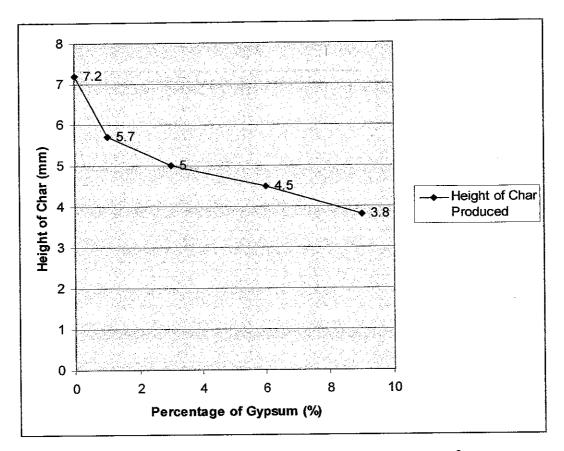


Figure 9: Graph on height of char produced vs. percentage of gypsum

From Figure 9, it can be seen that increasing in percentage of gypsum result in decreasing of thickness of samples. This means that gypsum delayed the time for intumescences reaction. The reason is because of its dehydration reactions. The heat energy delivered to the gypsum at this time tends to go into driving off water vapor rather than increasing the temperature of the mineral, which rises slowly until the water is gone, and then increases more rapidly. In other word, Sample E need more time to produce same height of char as Sample A.

4.2 Upper Surface Temperature Measurement

The surface of the upper size or the uncoated side of each sample was measured each time before and after the fire test. The readings are as shown in the Table 5. From Table 5, it shows that intumescent coating retard the temperature increasing in steel plate due to heat exposure as a result of fire test. This is important to make sure that the structure of steel plate is still stable.

Samples	Temp. before fire test	Temp. After 25 minutes
	(°C)	(°C)
Sample A	24	41
Sample B	25	39
Sample C	24	38
Sample D	24	40
Sample E	25	38

Table 5:	Upper surface	temperature	of each	sample

4.3 SEM Analysis

The formation of the final charring layer and their morphological structures is studied by SEM and the charring-layer SEM of the charring layer for Sample A and Sample E are presented in Figure 10 and Figure 11.

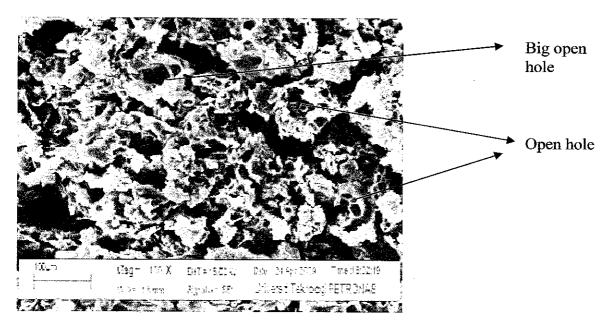


Figure 10: SEM micrographs of residue chars from coating without gypsum (Sample A)

Figure 10 indicate that surface of intumescent char layer of Sample A coating was compact with some big open holes. When the coating was burned on the second stage, gas with high pressure charged the char layer formed on the first stage. Because the thermal stability of Sample A coating was worse, the char layer was degraded quickly and the little residue char form impact structure with big holes at outer surface.

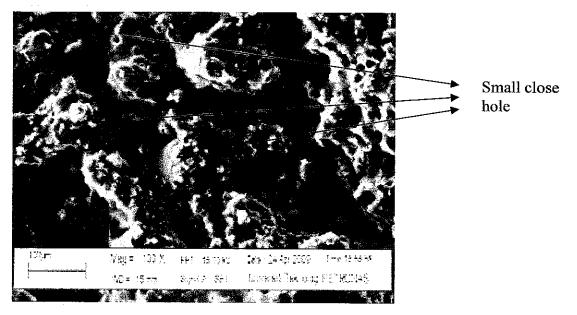


Figure 11: SEM micrographs of residue chars from coating modified with 9% gypsum (Sample E)

Figure 11 indicates that surface of intumescent char layer of Sample E coating formed well-distributed small close holes. Air in small hole can lower efficiency of heat transference, but air convection increases efficiency of heat transference if volume of small hole is too big. Increase of heat transference can accelerate heat degradation of resin and flameretardant, which harms expansion process. The intumescent charring layer with many mini-pores acts as the effect of the flame retardant, heat insulation and protecting inner matrix materials.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The aim of this work was to study the effect of gypsum as additive in intumescent coating. Gypsum composed of calcium sulfate dehydrates and due to presence of anion water in gypsum, the water molecule is held strongly and difficult to remove from the parent compound due to which more heat energy is required to release this water molecule. This concept can be adopted if using gypsum been used as additive for intumescent coating. The reaction time for intumescence process can be delayed which mean that coating can withstand higher temperature. Based on the result of the fire test, it can be conclude that gypsum can increase fire retardation time for intumescent coating.

5.2 Recommendations

Further study can be done to produce better analysis and better intumescent product. These are the analysis that is recommended:

- Thermogravimetric analysis (TGA) testing that is performed on samples to determine changes in weight in relation to change in temperature.
- X-Ray Diffraction analysis (XRD) powerful method by which X-Rays of a known wavelength are passed through a sample to be identified in order to identify the crystal structure.

REFERENCES

- [1] Intumescent, <<u>http://en.wikipedia.org/wiki/Intumescent</u>> (accessed September 4 2008)
- [2] S. Duquesne, C. Jama, R Delobel, S. Magnet (2003), Intumescent paints: fire protective coating for metallic substrates, <<u>http://www.sciencedirect.com</u>> (accessed February 5 2009)
- [3] Jun-wei Gu, Guang-cheng Zhang, Shan-lai Dong, Qiu-yu Zhang, Jie Kong, (May 2006), Study on preparation and fire retardant mechanism analysis of intumescent flame retardant coating, <<u>http://www.sciencedirect.com</u>> (accessed February 17 2009)
- [4] Pradip K. Mandal, Tanuj K. Mandal (March 2001), Anion water in gypsum and hemihydrate, <<u>http://www.sciencedirect.com</u>> (accessed February 17 2009)
- [5] C. A. Strydom, D. L. Hudson-Lamb, J. H. Potgieter, E. Dagg (September 1994), *The thermal dehydration of synthetic gypsum*, <<u>http://www.sciencedirect.com</u>> (accessed March 5 2009)
- [6] Dehydration Reactions, <<u>http://en.wikipedia.org/wiki/Calcium_sulfate</u>> (accessed April 4 2009)
- [7] Melamine, <<u>http://en.wikipedia.org/wiki/Melamine</u>> (accessed April 4 2009)

- [8] Pentaerythritol, <<u>http://en.wikipedia.org/wiki/Pentaerythritol</u>> (accessed March 24 2009)
- [9] Boric Acid, <<u>http://en.wikipedia.org/wiki/Boric_acid</u>> (accessed March 24 2009)
- [10] Epoxy Resin, <<u>http://en.wikipedia.org/wiki/Epoxy_resin</u>> (accessed February 12 2009)
- [11] Gypsum, <<u>http://en.wikipedia.org/wiki/Gypsum</u>> (accessed February 12 2009)
- [12] J. H. Khoo (1998), Thermal Characterization of a Ceramic Intumescent Material, <<u>http://www.sciencedirect.com</u>> (accessed February 25 2009)
- [13] S. El. D. Hamad (1981), A study of solid phases in the system CaSO₄-H₂O: II. Infrared spectra of the hemihydrates and soluble anhydrides.
 <<u>http://www.sciencedirect.com</u>> (accessed February 17 2009)

APPENDIX A PROJECT GANTT CHART

No	Activities / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic														
2	Information & Data Gathering on Topic														ĺ
3	Submission of Preliminary Report			1											
4	Further Information & Data Gathering														
6	Submission of Progress Report														
9	Method & Procedure Planning														
10	Results Gathering														
11	Submission of Interim Report														
12	Oral Presentation				1										

Table 6: Gantt chart for Semester 1

Table 7: Gantt chart for Semester 2

No	Activities / Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Materials & Tools Purchasing			-										 	
2	Laboratory Work														
3	Submission of Progress Report 1														
4	Result Gathering & Discussion									1					
5	Submission of Preliminary Report 2				1										
6	Seminar										1. 4				
7	Conclusion & Recommendation														
8	Poster Exhibition													 	
9	Submission of Dissertation (softbound)							<u> </u>							
10	Oral Presentation														
11	Submission of Dissertation (hardbound)		• •												

Process

Milestone