DEVELOPMENT OF INTUMESCENT FIRE RETARDANT COATING FOR PROTECTION OF WOODS

By

Abdul Halim Bin Ab Hamid

Dissertation submitted in partial fulfillment
of the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)
September 2012

Universiti Teknologi PETRONAS
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31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by:

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CERTIFICATION OF ORIGINALITY

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

Signature:

……....

Name: Abdul Halim Bin Ab Hamid

I/C No: 900811-03-5551

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ABSTRACT

Intumescent coatings make the most efficient way of fire retarding of flammable materials. The coatings swell under the influence of heat and form a thick porous charred layer which acts as thermal barrier that effectively protects the wood substrate against rapid increase of temperature and thereby maintaining the integrity of the structure. The aim of this study was to develop the intumescent coating formulation for wood and to determine the performance of the coating. Bunsen burner test, Field Emission Scanning Electron Microscope (FESEM) and Thermogravimetric Analysis (TGA) were conducted on samples to study the heat shielding effect, morphology and the residual weight of the coatings.In the end, this project was able to present the composition of the coating formulation for wood and the results of the analysis done on the coating.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Growing concerns over fire along with the development of urban market are focusing new attention on the benefits of specially treated timber, plywood and engineered wood. As wood has many good properties (from environmental and aesthetic aspects, physical and mechanical properties, easily processed, etc.), it is widely being used in building industry as a construction material [1]. Nowadays, artificial wood or engineered wood is also increasingly being exploited. Engineered wood like Medium Density Fibreboard (MDF) is a composite material consisting of wood and adhesives formed from the breaking down of hardwood or softwood residuals into wood fibres. MDF can be used as a building material similar in application to plywood. Like plywood, MDF can also be used in furniture application [2].

With the depleted natural resources crisis and environmental problems becoming serious all over the world, wood is being paid more-than-ever attention due to its being a unique renewable and environmental friendly material. However, the flammability of wood limits its wide applicability. Wood catches fire easily and burns vigorously with flame and thermally degraded with great full of ignitable gas above 300^{o}C [3].Therefore, improving the capability of fireproof of the wooden material becomes one of the important issues and has attracted more attentions than ever before.

Some additives to polymers have been known to increase the material resistance to fire ignition, retard the fire stress and rate of combustion of the materials and prevent sustained burning [3-6]. These additives that are incorporated in polymeric materials are called fire retardant. In the previous studies, the use of fire retarded coatings is one of the easiest, one of the oldest and one of the most efficient ways to protect a substrate against fire [4].It is efficient, environmental friendly and economical method of fire protection. Indeed, it presents several advantages: it can prevent heat from penetrating and flames from spreading, it does not modify the intrinsic properties of the material; it is easily processed and may also be used onto several materials including metallic materials, polymers, textiles and woods [3-5].

Fire retardant in the form of foaming char is called intumescent and it is known as "passive fireproofing materials", which means insulating systems designed to decrease heat transfer from a fire to the structure being protected [4, 5]. Intumescent is accomplished with a minimum of three general components: a source of mineral acid (typically ammonium phosphate), a source of carbon (typically pentaerythritol), a blowing agent (typically melamine) and bound together by a resin binder [3, 5-6].

1.2 Problem Statement

1.2.1 Problem Identification

There are growing concerns regarding the use of intumescent fire retardant coating for wood. Several studies had shown the commonly used intumescent coating for protecting steel substrate [4, 5] for up to two hours, so that firefighter are able to safely evacuate people from the building. Likewise, intumescent application on wood also has been studied [6, 7], and some commercial products intended for wood are available. However, the limitation associated with wood based structure is that when temperature rises more than 300°C, the wood structure tends to fall. Therefore, there is a need to protect the wood by using fire retardant intumescent coating. As for this reason, we will develop an intumscent coating formulation for wood substrate and explains the effectiveness of the coating using Bunsen burner test, Field Emission Scanning Electron Microscopy and Thermogravimetric Analysis [8].

1.2.2 Significant of the project

The project was focused on the development of intumescent coating formulation for the wood and the performance of the coating according to ASTM E119 standard. The detail on the selection and preparation of the material is crucial because it will be used in the future by the researchers in order to enhance the coating effectiveness. This project will be the reference for them to follow or modify so that it will meet their objectives of experiment. Besides that, this project will provide the guidelines in term of testing methodology that commonly used by certain researchers. According to recent research [8], fire test, thermal insulation test, FESEM, and TGA are used for coating analysis.

1.3 Objective and Scope of Study

The aim of this research is to develop an intumescent coating formulation for the wood. It will focus on the ingredient's composition and the procedure to prepare the coating.

Second, to determine the coating performance on the heat shielding effect, morphology, and the residual weight of the coatings according to BS standard test methodology.

1.4 The Relevancy of the Project

The intumescent coating for wood is crucial as to protect the wood for safe evacuation of people from the building. Several studies have showed the coating formulation for wood [1, 7, 9], but didn't put a clear finish on the particular constituents used for the coating. As for this study, each coating constituents will be discussed. Method of preparations and the test methodology also will be discussed for further improvement by other researchers.

1.5 Feasibility of the Project within the Scope and Time Frame

The following are the goals to be achieved for the project during the first four months (FYP1) period;

- Review of literatures related to the study.
- Selection of coating ingredients and preparation on coating.

While during four months of the project (FYP 2), the coating formulation and testing of the coating was carried out. Basically, the project is feasible within the scope and time frame if proper planning is done. The equipment needed for the testing is available in the Mechanical Laboratory.

CHAPTER 2

LITERATURE REVIEW

2.1 Intumescent Coating for Wood

Wood catches fire easily and burns vigorously with flame and thermally degraded with great full of ignitable gas above 300^{o}C [3].Therefore, improving the capability of fireproof of the wood material becomes one of the important issues and has attracted more attentions than ever before. This “intumescent concept” allows the balance between the fire properties and level of additives in the material. Generally, three intumescent constituents are used for wood coating: an acid source, a carbon source and a blowing agent [5, 6]. In addition to the basic constituents, other substances such as expandable graphite flakes can be included [10]. To form an efficient protective char in terms of their physical and chemical properties, formulation of these ingredients has to be adapted. Intumescent coatings are inert at low temperature. When exposed to high temperature, the coating swells and produces a porous char layer of low thermal conductivity. The char layer protects the underlying material by delaying its temperature rise and hindering the transport of oxygen to and inert gases from the surface [4-6, 10]. According to recent researches [7, 9, 12], three basic ingredient used in the intumescent coating for wood protection are ammonium polyphosphates (APP), melamine (MEL) and pentaerythritol (PER). The specific functions of each ingredient used: an acid source like ammonium polyphosphates (APP) to catalyses the cross linking during the char process. The presence of the blowing agent like melamine (MEL) in intumscent coating ensures that the coating, when dried on a surface, is clear and transparent. The blowing agent produces gas and foaming in the char during the intumescent process. The charring agent typically pentaerythritol (PER) ensures that the char, when produced, has the required consistency to prevent or retard the onset of flames. The Bisphenol A (BPA) acts as a binder and aids charring of the coating in the event that the surface to which it is applied is subjected to excessive heat.

Lastly, the presence of Alumina trihydrate (ATH) as filler or pigments in the coating of wood also have been identified [7].

The research's summary from different research papers (by years) were summarized as below:

Title of research paper	Publisher's name	Research's summary
[9] Fire Retardant Surface Coating for Cellulosic Materials.	Jain, J. P., Saxena, N. K., Ilam Singh, & Gupta, D. R. (1985).	Fire retardant intumescent coating based on copolymer of vinyl acetate and 2-ethyl hexyl acrylate resin emulsion.
[1] Flame retardant treated plywood.	Grexa, O., Horváthová, E., Bešinová, O., & Lehocký, P. (1999).	Flame retardant of Magnesium hydroxide, polyphosphate, diammonium hydrogen phosphate and glue mixture were used.
[12] Study on preparation and fire-retardant mechanism analysis of intumescent flame-retardant coatings.	Gu, J., Zhang, G., Dong, S., Zhang, Q., & Kong, J. (2007).	Preparation of intumescent coating based on Pentaerythritol, expendable graphite, ammonium polyphosphate, melamine, titanium dioxide.
[13] New Fire Protective Intumescent Coating for Wood.	Hassan, M. A., Kozlowski, R., & Obidzinski, B. (2008).	Increase compatibility of polyurethane coating by mixing intumescent additives with butyl acrylate polymer
[11] Fire Protection & Retardant Coating of Wood.	Ward., Afred D., kent D. (2009)	Provide a coating formulation with a clear and transparent finish for the treated surface.
[3] Effect of nitrogen phosphorus flame retardants on thermal degradation of wood.	Jiang, J., Li, J., Hu, J., & Fan, D. (2010).	Study the effect of coating obtained from Phosphoric acid, pentaeryrthritol, urea and trolamine in water solution.
[7] Hybrid Intumescent Coatings for Wood Protection against Fire Action.	Canosa, G., Alfieri, P. V., & Giudice, C. A. (2011).	Improve efficiency by using reinforcing fiber. Hybrid coating consist of chlorinated rubber, phenolic resin, epoxy resin and alumina .
[4] A study of bonding mechanism of expandable graphite based intumescent coating on steel substrate.	Ullah, S., Ahmad, F., Megat-Yusoff, P. S. M., & Azm, N. H. B. (2011).	Study the bonding mechanism of expendable graphite in the intumescent coating.

2.2 Intumescent Mechanism

Intumescent can be described as fire retardant technology, which causes another wise flammable material to foam, forming a physical barrier when exposed to heat which slow down heat and mass transfer between the gas and condensed phases. When the intumescent coating subjected to heat and reaches certain temperature, the coating surface begins to melt and is converted into highly viscous liquid and reactions are initiated that result in the release of low thermal conductivity inert gases. These gases are trapped inside the viscous fluid (formation of bubbles).The result is the expansion or foaming of the coating, sometimes up to several times its original thickness, to form a protective carbonaceous char that acts as an insulative barrier between the fire and the substrate [4-6]. The mechanism of intumescence is usually described as follows: first, a mineral acid is produced from the breaking down of acid source, then it yield carbon char from the dehydration of carbonization agent. Finally the blowing agent decomposes to yield gases products. The result is the swelling of the char which provides an insulating protective barrier on substrate. This barrier slows down 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 [5, 6].

CHAPTER 3

METHODOLOGY/ PROCESS WORK

3.1 Materials and Coating Formulations for Wood Substrate

Materials: Ammonium polyphosphate (APP), Pentaerythritol (PER), Expendable Graphite (EG), Melamine (MEL), Boric acid (BA), Alumina trihydrate (ATH), Bisphenol A epoxy resin BE-188 (BPA) and Tetraethylene tetramine harderner (TETA). Meranti timber (Shorea pauciflora), Plywood and Medium Density Fibre (MDF).

The formulations of intumescent ingredients for wood (in grams) were shown as below:

Sample No	Components (wt %)						
	BPA	TETA	APP	PER	MEL	Boric Acid	Alumina Trihydrate
F0_PER	44.44	22.22	11.11	5.56	5.56	11.11	0
F1_PER	43.94	21.72	11.11	5.56	5.56	11.11	1
F2_PER	43.44	21.22	11.11	5.56	5.56	11.11	2
F3_PER	42.94	20.72	11.11	5.56	5.56	11.11	3
F4_PER	42.44	20.22	11.11	5.56	5.56	11.11	4
F5_PER	41.94	19.72	11.11	5.56	5.56	11.11	5

Table1: Formulations of intumescent ingredients using PER as carbon source (in grams)

The formulations of intumescent ingredients were repeated by replacing PER with Expendable Graphite (EG) and shown as below:

Sample No	Components (wt %)						
	BPA	TETA	APP	EG	MEL	Boric Acid	Alumina Trihydrate
F0_EG	44.44	22.22	11.11	5.56	5.56	11.11	0
F1_EG	43.94	21.72	11.11	5.56	5.56	11.11	1
F2_EG	43.44	21.22	11.11	5.56	5.56	11.11	2
F3_EG	42.94	20.72	11.11	5.56	5.56	11.11	3
F4_EG	42.44	20.22	11.11	5.56	5.56	11.11	4
F5_EG	41.94	19.72	11.11	5.56	5.56	11.11	5

Table2: Formulations of intumescent ingredients using Expendable Graphite (EG) as carbon source (in grams)

3.2 Preparation of Intumescent Formulations

Fig.1 below showed the experimental flow chart of coating preparation. The intumescent ingredients were mixed with their weight percentage composition homogeneously for 30min using Ultra Turrax mixer. F0, F1, F2, F3, F4 and F5 were controlled formulations with PER-APP-Mel-Boric acid-epoxy-hardener. All formulation was then prepared again using expendable graphite (EG) replacing the pertaerythritol (PER) as the carbon source.

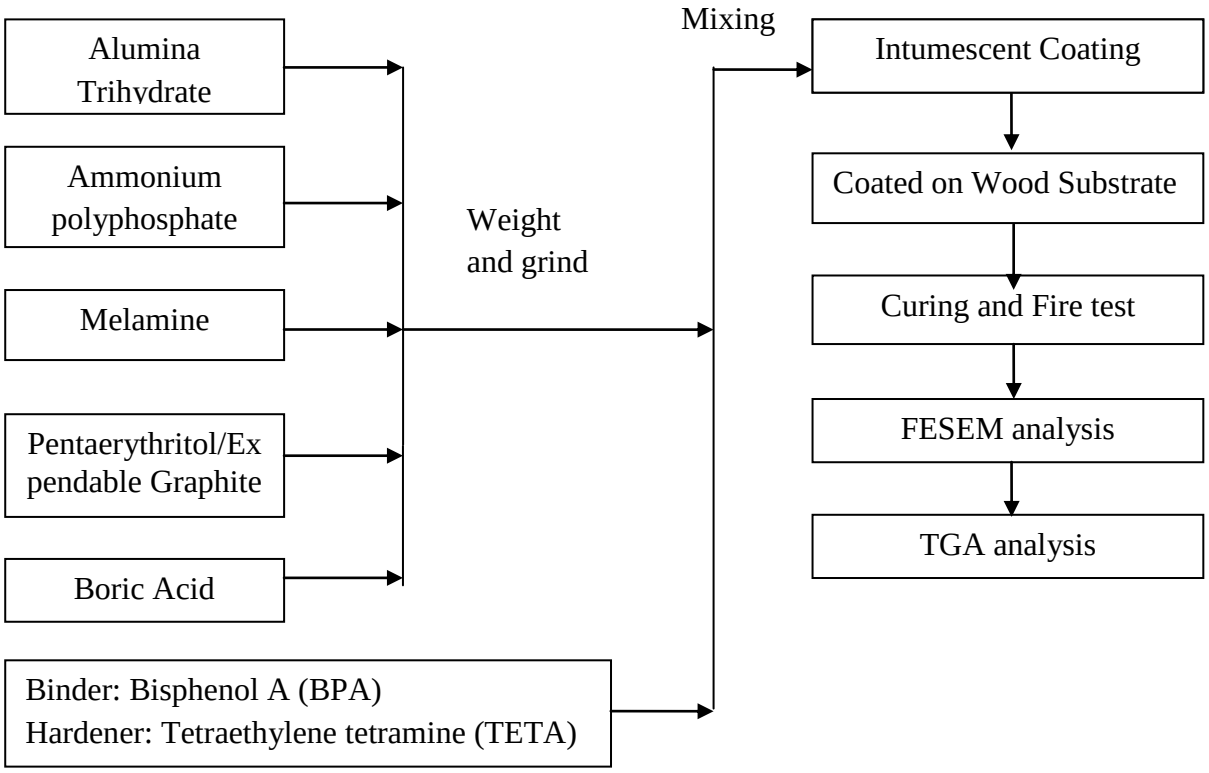


Figure 1: Experimental flow chart

3.3 Characterization of Coating

3.3.1 Bunsen Burner Test

In every sample, the coated wood was wired with a digital thermocouple for heating record. Portable Bunsen burner was used to burn the coating and the distance of the Bunsen form the coating was set at 7cm according to UL 94 standard. Two K-type thermocouple (-200~1370°C) were connected to AMS-850 data logger to measure the temperature. The other ends of the two thermocouples were connected to the coated surface. The flame temperature of Bunsen burner is 800^{o}C. The sample with the lowest back wood temperature should be the expected result as it will indicate the good thermal insulation properties of the coating [8].

Figure 2: Vertical Fire Test UL 94 standard arrangement

Fig 2 shows the Vertical Fire Test for the burning of the wood. Temperature was recorded using AMS-850 data logger.

3.3.2 Field Emission Scanning Electron Microscopy (FESEM)

The charring layer and the morphological structures of the inside and outside of the char after the burning were observed and analyzed using SUPRA Instrument FESEM. The FESEM samples were prepared by sticking char to a double-coated tape fixed onto an aluminum specimen mount stub, and the samples were coated with an ultra thin film as an electrically conducting material which was deposited on the sample by using a low vacuum sputter coater. The formation of bubbles, holes and cracks will be observed and outer layer which is smooth demonstrates a good intumescent behavior [8].

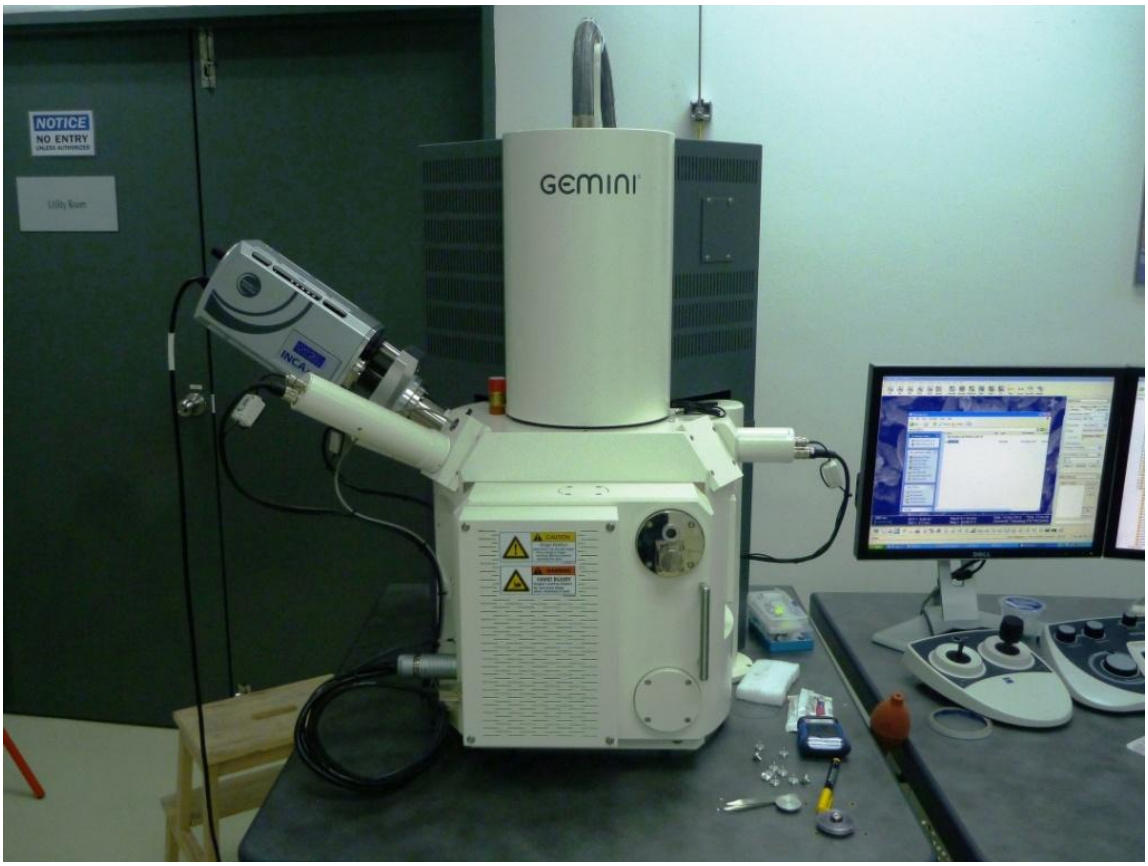


Figure 3: SUPRA Field Emission Scanning Electron Microscopy (FESEM) Instrument

Fig 3 shows the FESEM instrument used to study the morphological structure of the char after the burning.

3.3.3 Thermogravimetric Analysis (TGA)

The residual weight of intumescent coatings will be analyzed using TGA. Thermogravimetric Analysis (TGA) measures the amount and rate of change in the weight of an intumescent coating as a function of temperature or time in a controlled atmosphere. Measurements are used primarily to determine the decomposition of intumescent ingredients and to predict their thermal stability up to 800°C. This technique can characterize materials that exhibit residual weight (weight loss or gain) due to decomposition, oxidation, or dehydration. The residual weight versus temperature will be plotted to examine the effectiveness of intumescent coating. The aim is to obtain a high level of homogenous char at the end of the experiment with high amount of residue weight [8].

CHAPTER 4

RESULTS AND DISCUSSION

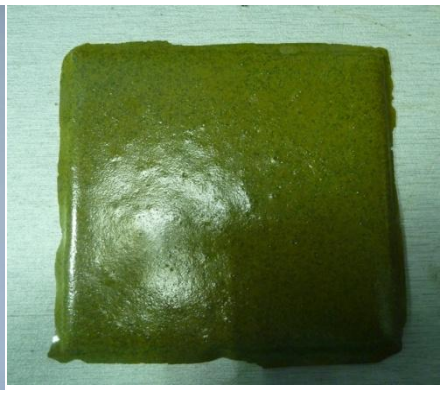
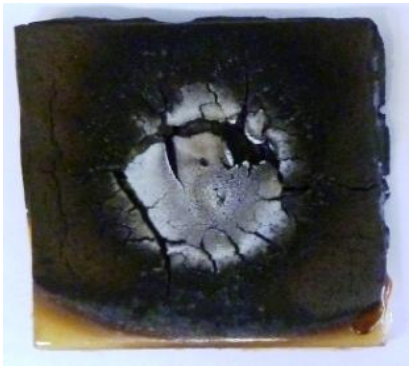
4.1 Intumescent coating thickness in mm

Table 3: Thickness of Intumescent Coating

Materials	T_0 (mm)	T_1 (mm)	T_2 (mm)	T_3 (mm)	T_4 (mm)	T_5 (mm)
Timber (Meranti)						
Without coating	5.19	5.04	5.10	5.07	5.12	5.14
With coating (PER)	7.20	6.29	6.41	6.95	6.63	6.67
Coating Thickness	***2.10***	***1.25***	***1.31***	***1.88***	***1.51***	***1.53***
Without coating	5.41	5.85	5.90	5.75	5.78	5.68
With coating (EG)	7.48	8.65	8.77	7.88	7.69	7.80
Coating Thickness	***2.07***	***2.80***	***2.87***	***2.13***	***1.91***	***2.12***
Plywood						
Without coating	3.16	3.09	3.13	3.10	3.03	3.13
With coating (PER)	4.79	4.68	4.72	4.83	4.73	5.12
Coating Thickness	***1.63***	***1.59***	***1.59***	***1.73***	***1.70***	***1.99***
Without coating	3.46	3.51	3.58	3.49	3.46	3.55
With coating (EG)	5.37	6.52	6.83	6.09	5.24	5.70
Coating Thickness	***1.91***	***3.01***	***3.25***	***2.6***	***1.78***	***2.15***
MDF						
Without coating	2.90	3.11	3.01	2.97	3.02	3.09
With coating (PER)	4.40	5.30	5.02	4.38	5.28	4.92
Coating Thickness	***1.50***	***2.19***	***2.10***	***1.41***	***2.26***	***1.83***
Without coating	3.04	3.20	3.41	3.19	3.15	3.42
With coating (EG)	4.81	5.40	5.94	5.77	5.09	5.42
Coating Thickness	***1.76***	***2.2***	***2.53***	***2.58***	***1.94***	***2.00***

4.2 Observations of Samples Before and After Fire Test

Table 4 : Observations of Samples Before and After Fire Test

Formulations	F4_PER	F4_EG
Physical Appearance Before Fire test		
Observations	- Smooth and clear surface - Slightly yellowish - Touch dry after 2 days	- Irregularities on surface - Slightly darker when exposed to sun - Touch dry after 2 days
Physical Appearance after Fire test		
Observations	- Coating did not expand - No formations of bubbles - Cracked surface	- Coating expands - Formations of char dust

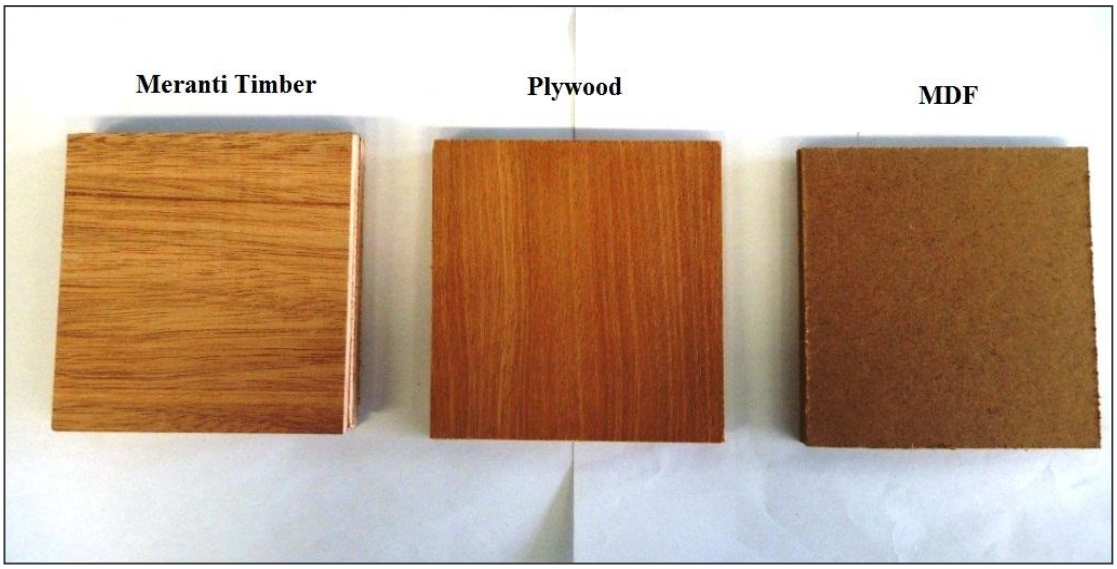


Figure 4: Meranti timber, plywood and MDF samples.

Fig 4 shows the three types of wood used in the experiment. The purpose was to determine coating performance on different woods.

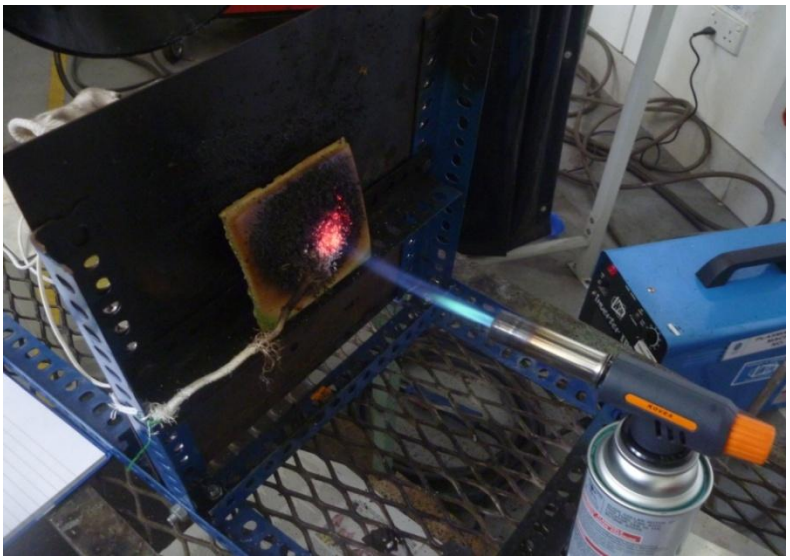


Figure 5: Fire Test on Wood samples.

Fig 5 shows the setup of Bunsen burner test on wood samples. One thermocouple is placed on char and one more on the backside of the wood. The other ends of each thermocouple are connected to datalogger.

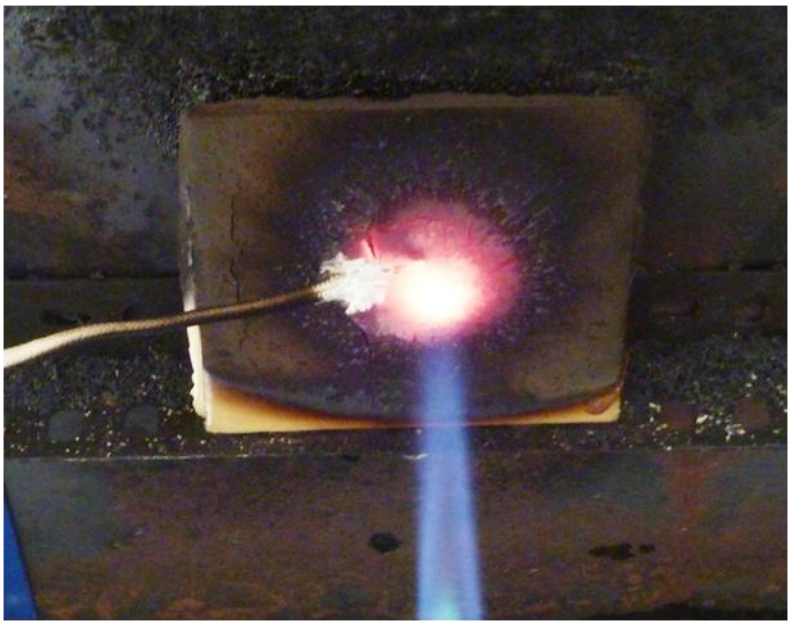


Figure 6: Burning on sample F4 using PER carbon source

Fig 6 above shows the burning on sample F4 using PER carbon source with no indications of char expansion.

Figure 7: Burning on sample F4 using EG carbon source

Fig 7 above shows the burning on sample F4 using Expendable Graphite (EG) replacing the PER. Burning resulted in char expansion and formations of char dust.

Figure 8: PER formulation coating before burning

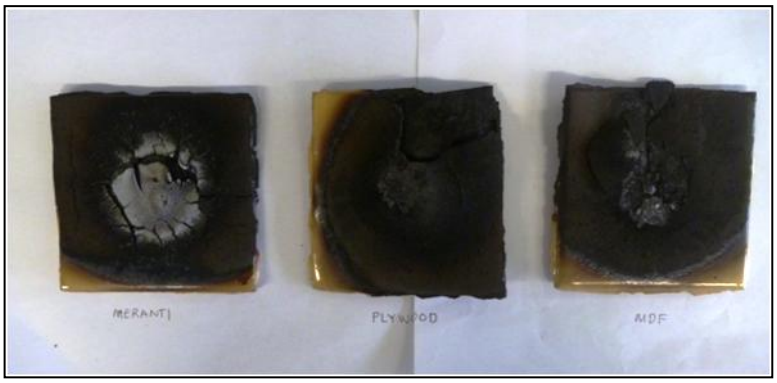


Figure 9: PER formulation coating after burning at 30min

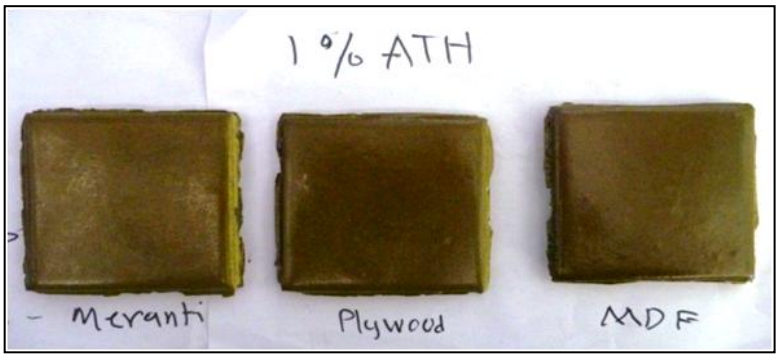


Figure 10: EG formulation coating before burning

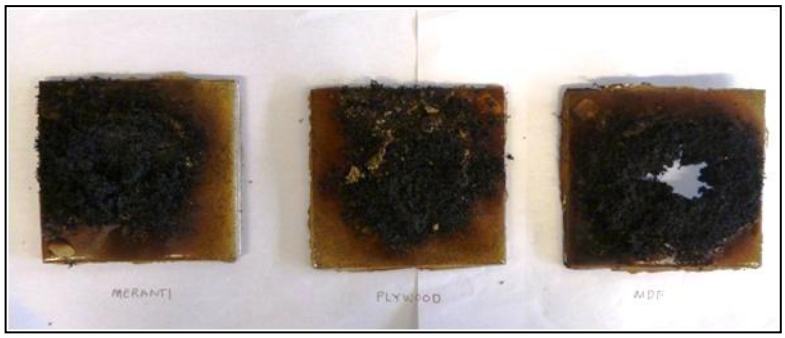


Figure 11: EG formulation coating after burning at 30min

4.3 Heat shielding Effect

In this research, six formulations with different compositions of intumescent ingredients were prepared. After the fire testing was done, it was found that formulation F4 that contain 5.56% of pentaerythritol, 11.11% of ammonium polyphosphate, 5.56% of melamine, 11.11% of boric acid, 4% of alumina trihydrate and 62.66% of epoxy-hardener mixture gives the best intumescent effect after fire testing being performed on the wood. Then, all six formulations were prepared again by replacing the pentaerythritol with expendable graphite.

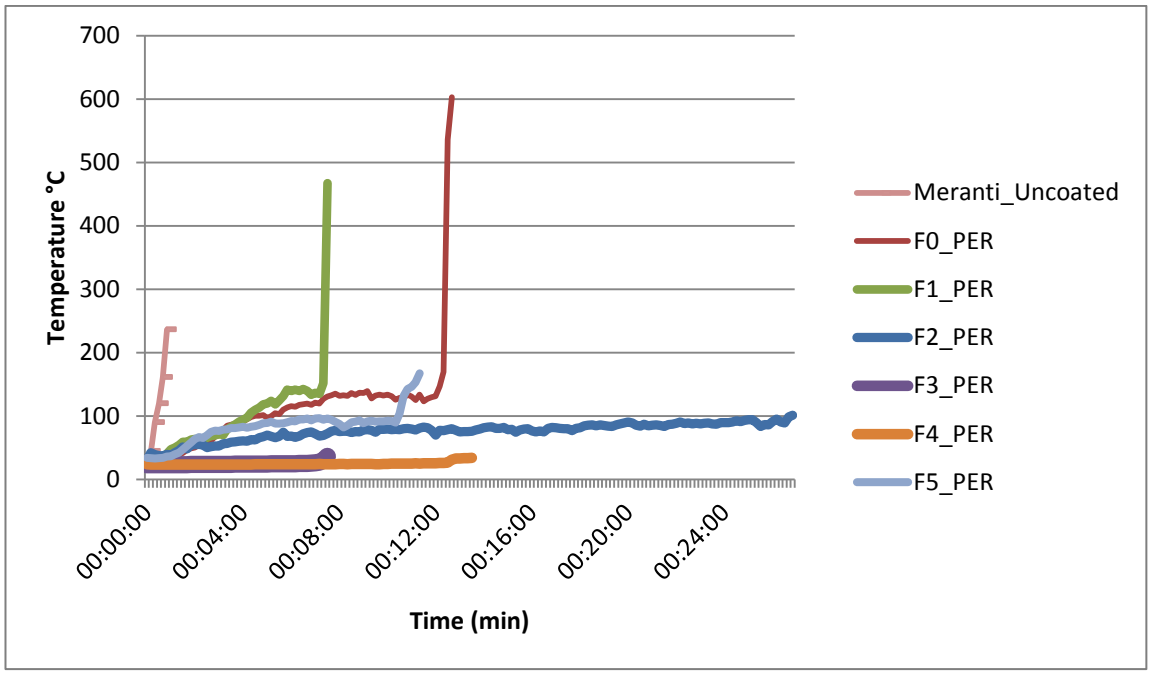


Figure 12: Thermal Behaviour of Coating PER-APP-Mel-BA-ATH-Epoxy and hardener on Timber (Meranti) wood.

Fig 12 above shows the plotted graph of Temperature vs. Time for Meranti wood. The temperature was taken from the wood backside temperature.

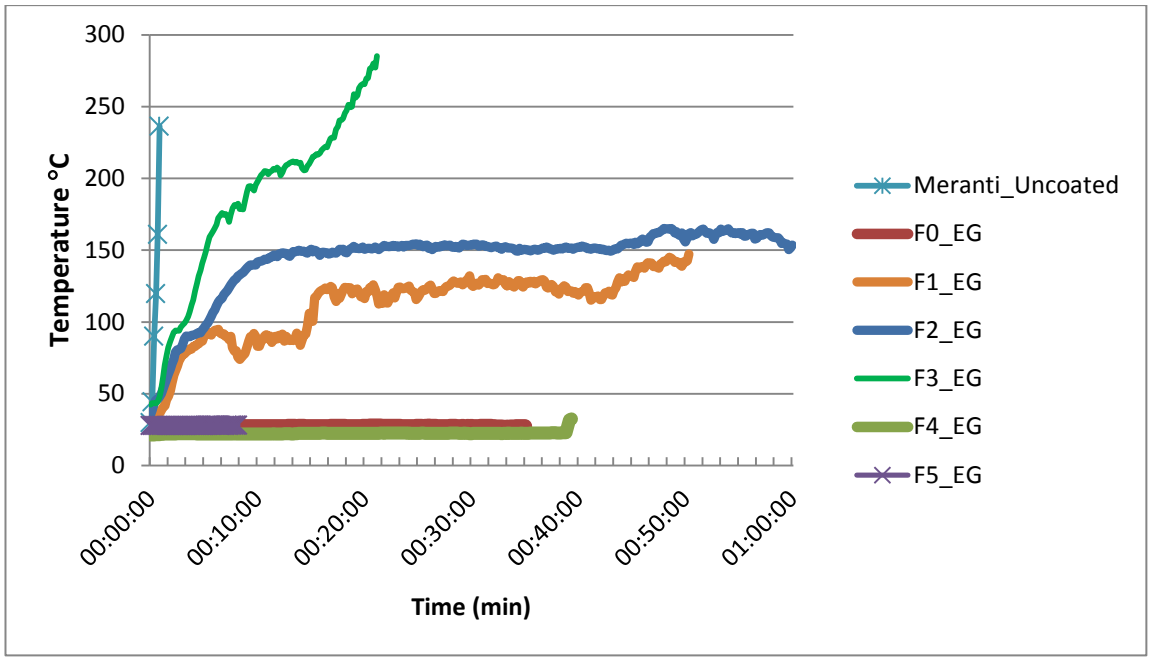


Figure 13: Thermal Behaviour of formulations replaced with Expendable Graphite (EG) on Timber (Meranti) wood.

The temperature time curves and data for fire proofing time of fire retardant coating for timber (meranti) wood are illustrated in Figs. 12 and 13 and Table 1. The uncoated timber sample can only sustain its integrity for about 3 minutes at 240°C after the fire. Fig 12, formulation 0, it has the back side wood temperature 121°C after 7 min. Formulation 1 has the back side wood temperature of 136°C, formulation 2 has back side temperature of 72°C, formulation 3 has back side temperature of 26°C while formulation F4 and F5 has the back side temperature of 24°C and 96°C after 7 min respectively. From the result obtained, formulation F4 has the lowest backside wood temperature followed by formulation F3, F2, F5, F0 and F1.This is because of the difference weight percentage of every component for each formulation and the thickness of coating. As shown in the table, by comparing the formulation F3 and F5 when amount of alumina trihydate is increased, the backside temperature of formulation F3 is lower than formulation F5; this is because amount of alumina trihydrate added no longer can increase the fire retardant properties of the coating.

The weight percentage of each material in the intumescent coating formulations has its own limit where it will gives optimum results by playing individual role as fire retardant which maintained the structural integrity of the char.

As the pentaerythritol carbon source in the formulations was replaced with expendable graphite, the fireproofing time of the coating was increased dramatically. Fig 13, the result showed that backside wood temperature was lowered to 27, 90, 120,175, 22 and 28°C after 7 min of burning of coating F0, F1, F2, F3, F4 and F5 respectively. Furthermore, the formulation F4 gives the best intumescent effect as the temperature of the wood backside is 22°C after 7 min and even reaching 40 min of burning, the temperature is still at 35°C.

This proved that expendable graphite as the carbon source showed a better fire retardant effect. Also, the increased amount of alumina trihydrate up to 4% can increase the intumescent effect of the coating. While for pentaerythritol, no carbon char is produced thus any trapping of gases didn't occur. The expendable graphite produced the carbon char during the burning process thus trapping the escaped gases and resulted in the swelling of the char which provides an insulating protective barrier on wood. Thus, it will give a better intumescent effect.

Figures below show the thermal behavior of the coating on plywood samples.

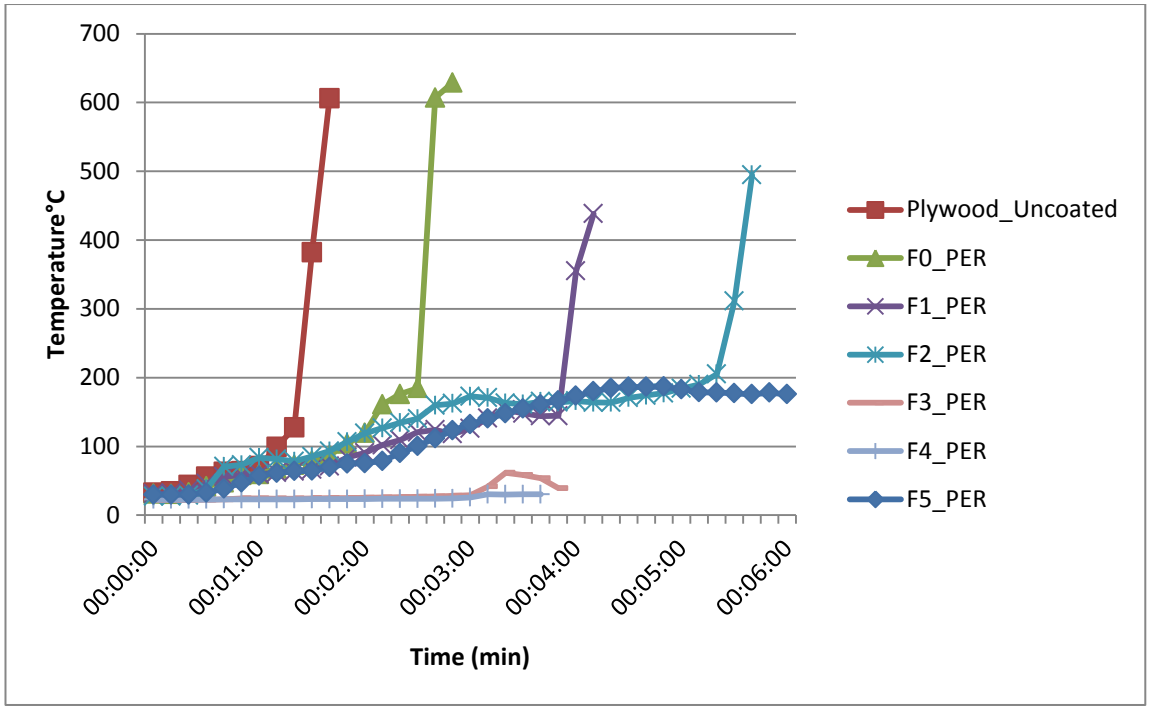


Figure 14: Thermal Behaviour of Coating PER-APP-Mel-BA-ATH-Epoxy and hardener on Plywood.

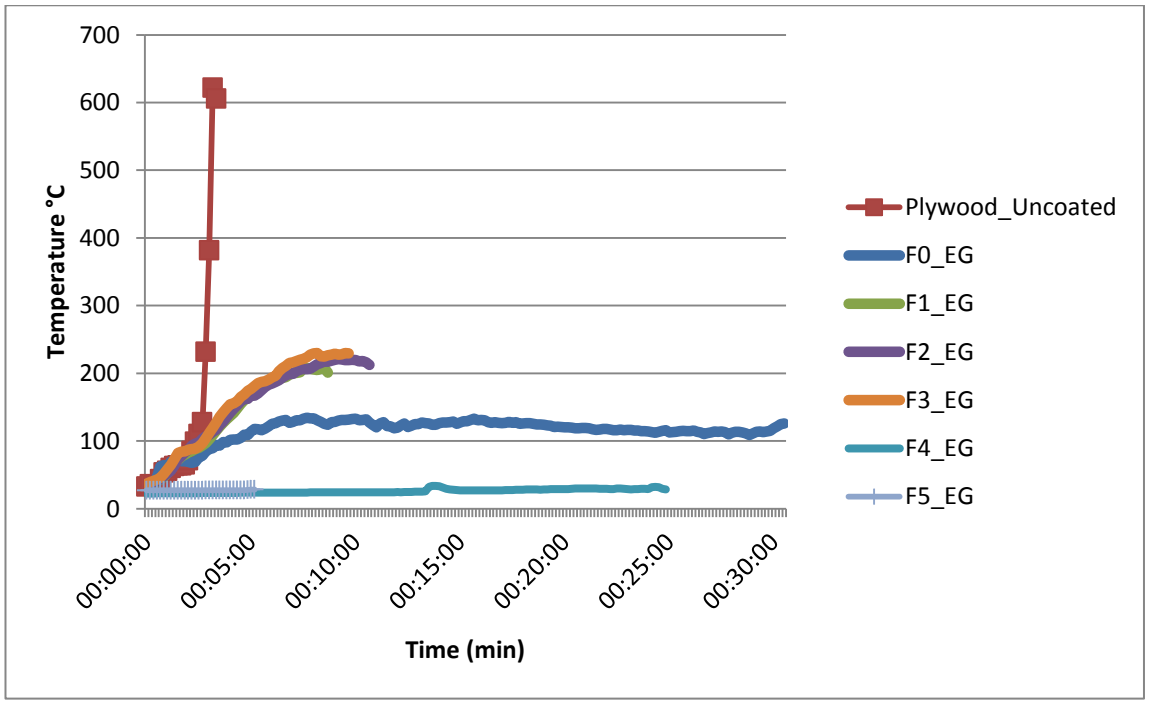


Figure 15: Thermal Behaviour of formulations replaced with Expendable Graphite (EG) on on Plywood.

The temperature time curves and data for fire proofing time of fire retardant coating for plywood are illustrated in Figs. 14 and 15. The uncoated plywood sample can only sustain its integrity for about 2 minutes after the fire with temperature exceeding 200°C.

Fig 14, formulation 0, it has the back side plywood temperature of 120°C after 2 min. Formulation 1 has the back side plywood temperature of 91°C, formulation 2 has back side temperature of 119°C, formulation 3 has back side temperature of 25°C while formulation F4 and F5 has the back side temperature of 24°C and 76°C after 2 min respectively. From the result obtained, formulation F4 has the lowest backside plywood temperature followed by formulation F3, F5, F1, F2 and F0.

The fire retardant effect of coating increased dramatically when the pentaerythritol carbon source is replaced with expendable graphite. Fig 15, the result showed that plywood backside temperature is lowered to of 69, 83, 86.9, 87, 23 and 27°C after 2 min of burning of coating F0, F1, F2, F3, F4 and F5 respectively. Furthermore, formulation F4 gives the best intumescent effect as the plywood backside temperature still below 30°C after 25 min of burning.

For the purpose of comparing the data of using pentaerythritol and expendable graphite carbon source, minimum time that the coating can maintain the properties of the plywood is taken for 2 minutes. Then, for rest of the coating, the burning is continued to the maximum time possible it can protect the plywood.

The result proved that the coating of formulation F4 which using expendable graphite instead of pentaerythritol as carbon source will provide a good insulating effect on the plywood.

Figures below show the thermal behavior of the coating on Medium Density Fibre (MDF) samples.

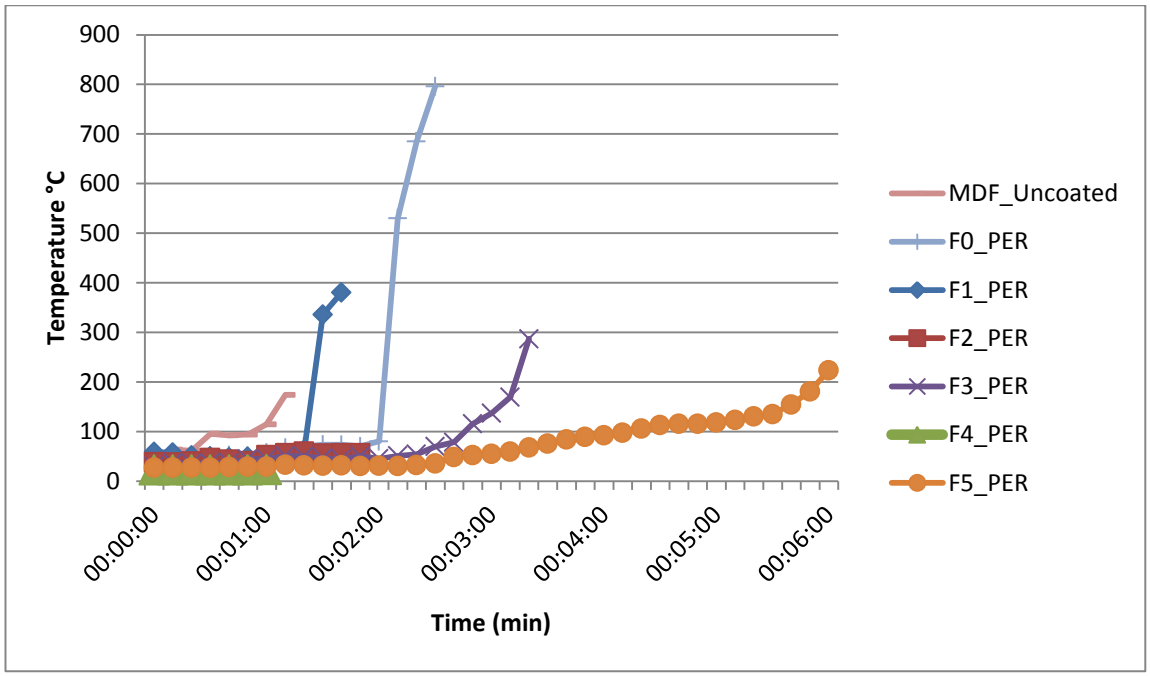


Figure 16: Thermal Behaviour of Coating PER-APP-Mel-BA-ATH-Epoxy and hardener on MDF samples.

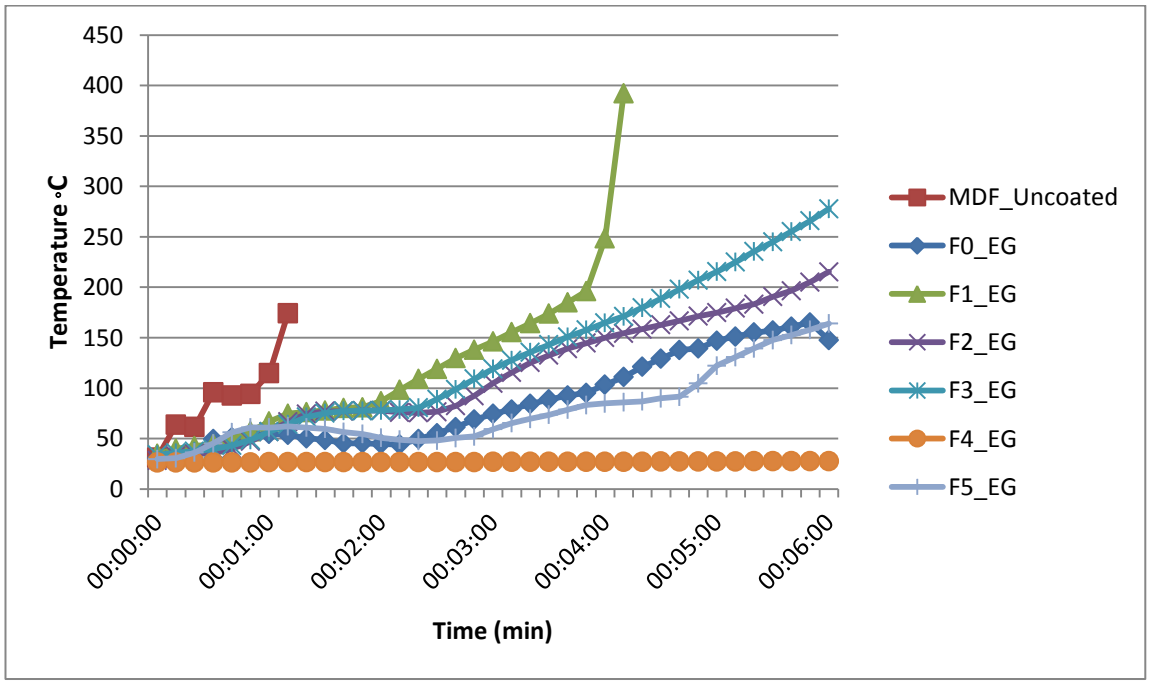


Figure 17: Thermal Behaviour of formulations replaced with Expendable Graphite (EG) on MDF samples.

The temperature time curves and data for fire proofing time of fire retardant coating for medium density fibre (MDF) are illustrated in Figs. 16 and 17. The uncoated MDF samples can only sustain its integrity for about 30 seconds after the fire with temperature at 96 °C and the MDF already started to catch fire.

Fig 16, formulation 0, it has the back side MDF temperature 58°C after 1 min. MDF back side temperature of 47, 53, 45, 24 and 30°C for formulations F1, F2, F3, F4 and F5 respectively after 1 min of burning. From the result obtained, formulation F4 has the lowest backside MDF temperature followed by formulation F5, F3, F1, F2 and F0.

The fire retardant effect of coating is lowered when the pentaerythritol carbon source is replaced with expendable graphite. Fig 17, the result showed that MDF backside temperature is increased to of 55, 67, 57, 57, 27 and 61°C after 1 min of burning of coating F0, F1, F2, F3, F4 and F5 respectively. Moreover, formulation F4 gives the best intumescent effect as the MDF backside temperature still at 28°C after 6 min of burning. The data obtained showed the opposite result as compared to fire test done on meranti timber and plywood. One of the possible reasons is because the coating using PER detached quickly from the MDF sample after the burning as the MDF sample appeared to be left open to constantly changing humid/dry environment of the wood factory which is affecting the properties of the MDF itself. Yet, the coating using expendable graphite still showed a good fire retardant effect as it take a longer time to detach from MDF sample and can maintain the integrity of the MDF. Furthermore, for normal MDF sample without being exposed to humid environment, both coating that using PER and EG is still attached to the sample after the burning.

For the purpose of comparing the data of using pentaerythritol and expendable graphite carbon source, minimum time that the coating can maintain the properties of the MDF is taken for 1 minute. Then, for rest of the coating, the burning is continued to the maximum time possible it can protect the MDF. The result proved that the coating of formulation F4 which using expendable graphite instead of pentaerythritol as carbon source still can provide a good intumescent effect on the MDF sample.

4.4 Field Emission Scanning Electron Microscopic (FESEM) analysis

The FESEM micrograph images of char for outer and inner surface for sample F4_PER and F4_EG were obtained. Formulation F4 was selected to be tested with FESEM as it gives the best fire retardant effect from the earlier Bunsen burner test.

4.4.1 Char morphology on Sample F4_PER

As can be observed from Table 4, F4_PER did not expand. There are holes and cracks observed on the surface of char: these are considered as due to decomposition of melamine into its derivates after 200°C, releasing ammonia gas which produces holes on the surface of char. These holes and cracks allow direct penetration of heat to the surface of wood. Fig 18 showed the holes and crack in FESEM image of F4_PER.

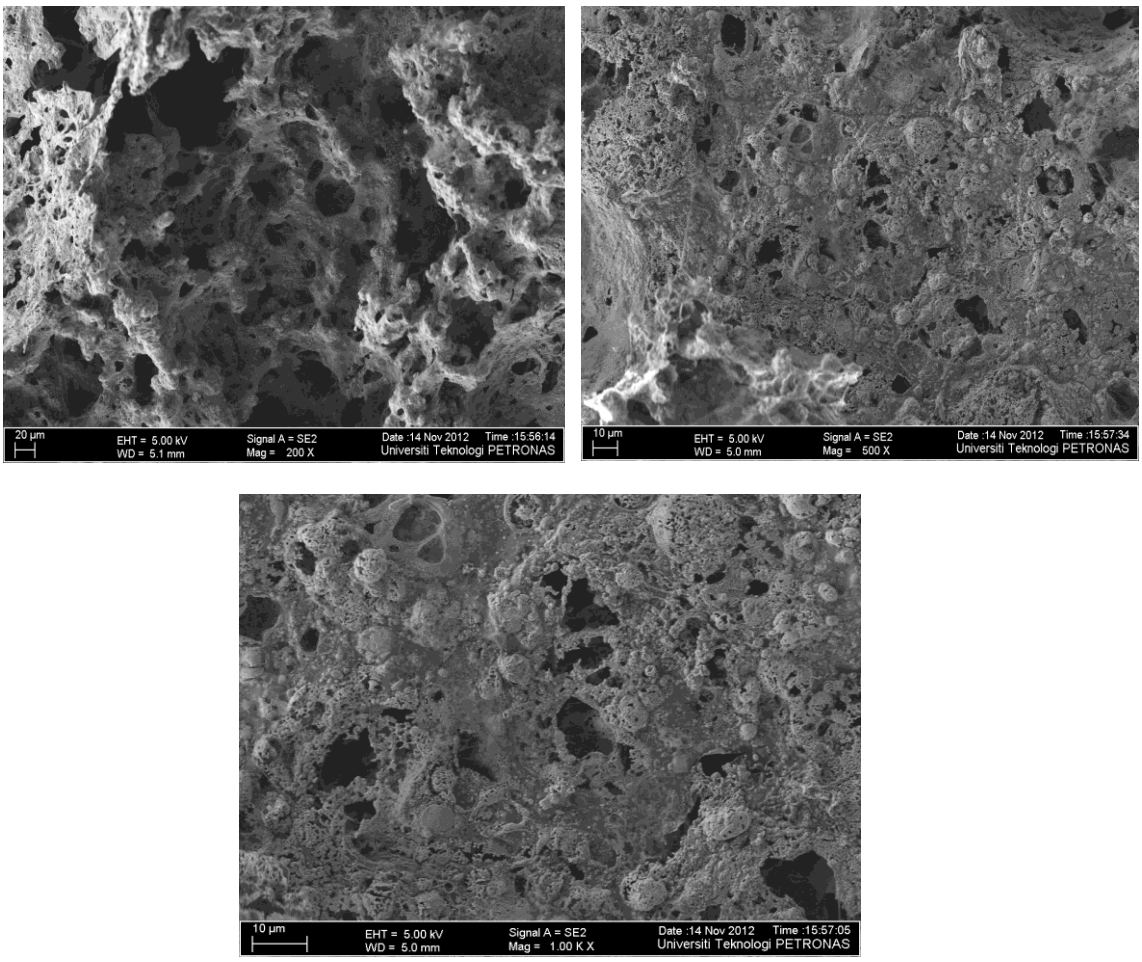


Figure 18: FESEM Micrograph showing holes and cracks on F4_PER

4.4.2 Char morphology on Sample F4_EG

There were small bubbles, holes and cracks observed on the charring layer of samples F4_EG. The outer layer was smooth and demonstrates a good intumescent behavior. Bubbles are formed due to emission of N_2 and ammonia gases during burning process. F4_EG coating swells nicely since there are balance emission of N_2, NH_3 and CO_2 gas and dehydration of water inside the coating. Small holes observed are due to the heat dissipation that prevents heat from transferring to the surface. Folding structures also can be observed on inner surface which demonstrate a good fire retardant effect.

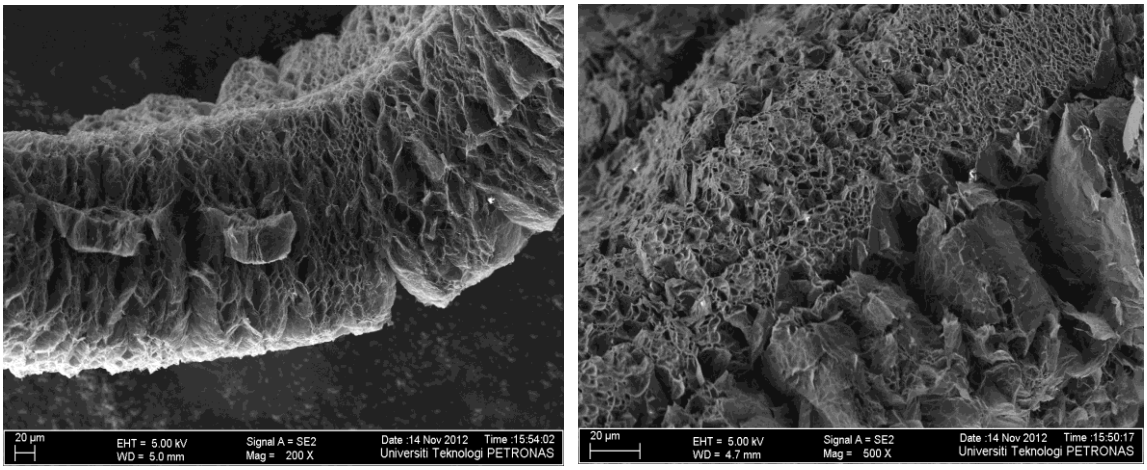


Figure 19: FESEM micrograph of F4_EG coating for outer surface: 200 and 500 X

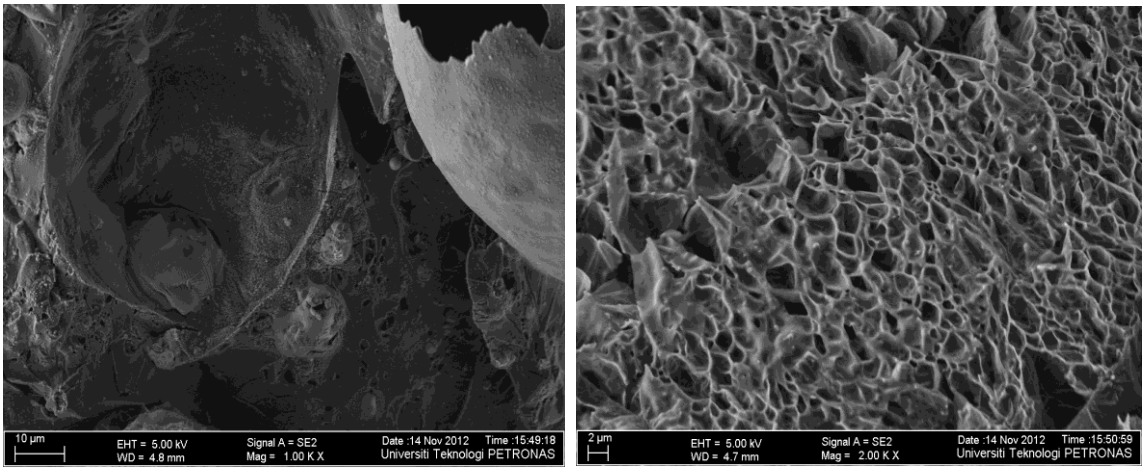


Figure 20: FESEM micrograph of F4_EG coating for inner surface: 1.0 and 2.0 KX

4.5 Thermogravimetric Analysis (TGA)

The thermogravimetric analyses of samples were carried out under controlled air and temperature conditions gives an overview of the degradation process of the coating. The residual weight is plotted against temperature to examine the effectiveness of intumescent coating. The aim is to obtain a high level of homogenous char at the end of the experiment with high amount of residue weight. This residue will limit the heat transfer to the wood and eventually limit the gases feeding combustion process. A slow degradation rate will lead to a more homogenous char at the end of burning.

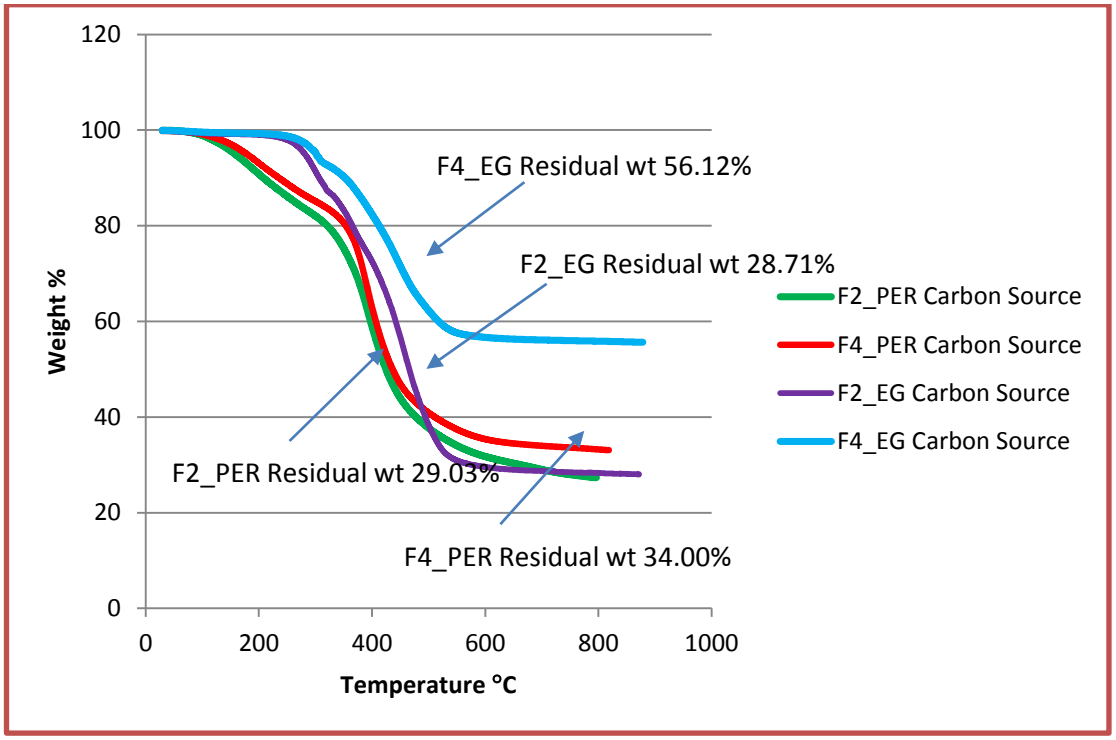


Figure 21: TGA curves of samples F2 and F4

As illustrated in Fig.21 the TGA curves of sample F2_PER, F4_PER, F2_EG and F4_EG showed three steps of thermal degradation. In the range of 0-200°C, the residual weight was 99-90%: this occurred because H_2O was being released from epoxy resin and boric acid is decomposed into meta boric acid.

Then, the residual weight was 90-60% in the range of 200-400°C due to decomposition of melamine and APP releasing N_2, H_2O and NH_3 gas. Later stage, APP is decomposed into polyphosphoric acid and meta phosphoric acid, epoxy and hardener decomposed into CO2 and H2O resulting in reduction weight to 60-30% in the range of 400-600°C. All four residues were observed after degradation over 700°C. The sample F2_PER and sample F4_PER contained residual weight of 29.03 and 34.00% respectively. Sample F2_EG with expendable graphite left 28.71% and F4_EG left with 56.12% residues at 700°C over. The summarized result of TGA was tabulated and shown as below:

F2_PER		**F4_PER**	
Temperature (°C)	Weight (%)	Temperature (°C)	Weight (%)
100.15	98.871	100.14	99.136
200	90.826	200.33	93.056
300.05	82.04	300.24	85.109
400.1	58.873	400.05	62.969
500.33	37.685	500.29	40.869
600.2	31.75	600.34	35.384
700.04	29.031	700.07	33.995
F2_EG		**F4_EG**	
Temperature (°C)	Weight (%)	Temperature (°C)	Weight (%)
100.19	99.323	100.03	99.579
200.09	98.992	200.2	99.264
300.15	91.363	300.28	95.286
400.2	72.495	400.23	82.346
500.32	38.263	500.31	62.262
600.21	29.551	600.19	56.662
700.35	28.714	700.11	56.116

Table 5 : Summarized result of Thermogravimetric Analysis (TGA)

It shows that the final weight percentage for samples with Expendable Graphite is slightly higher than samples without Expendable Graphite because the expendable graphite will only oxidize when the temperature reach 350°C. Thus, expendable graphite will remain in the residual char. Based on these TGA result, it was observed that F4_EG has the highest percentage of residual weight at the temperature over 700 °C with 95 percent higher than F2_EG, 93.3 percent higher than F2_PER and 65 percent higher than F4_PER. This shows that, sample F4_EG gives the best effectiveness of intumescent coating.

4.6 Recommendations

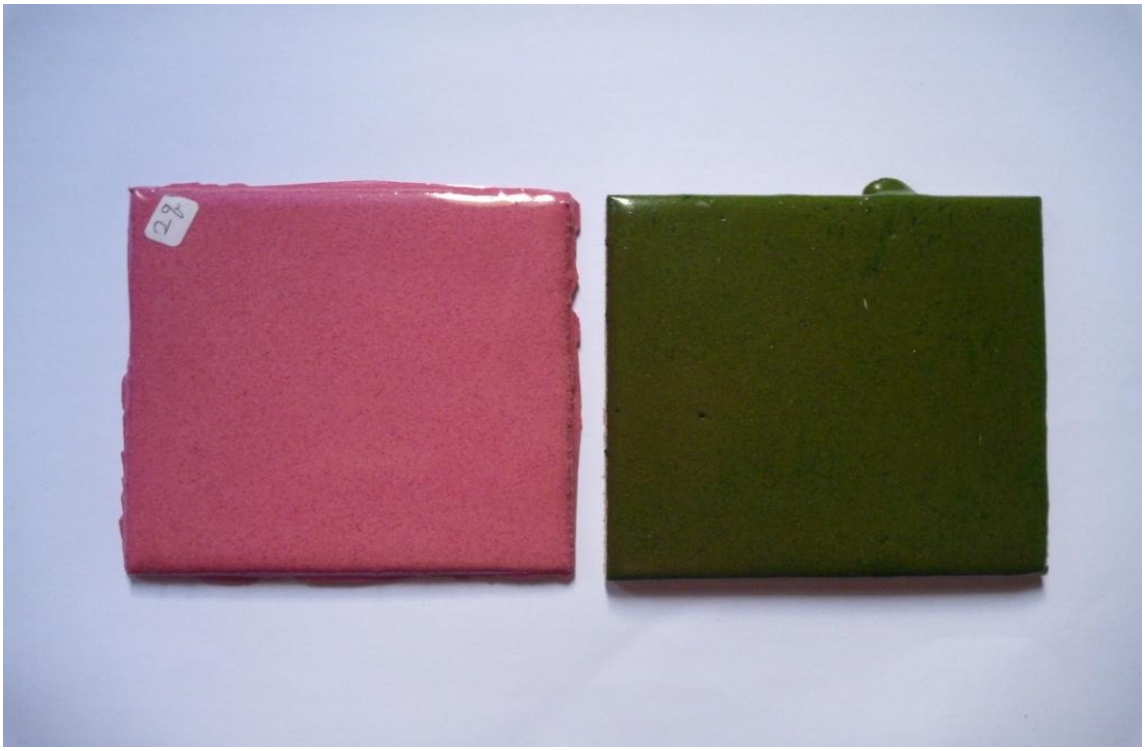


Figure 22: Sample of Wood with Colors

Fig.22 showed the sample of wood with color powder added into the formulations. Physical appearance shows the surface of coating was smooth, shiny and color became darker when exposed to the sun. The coating dried after 2 days.

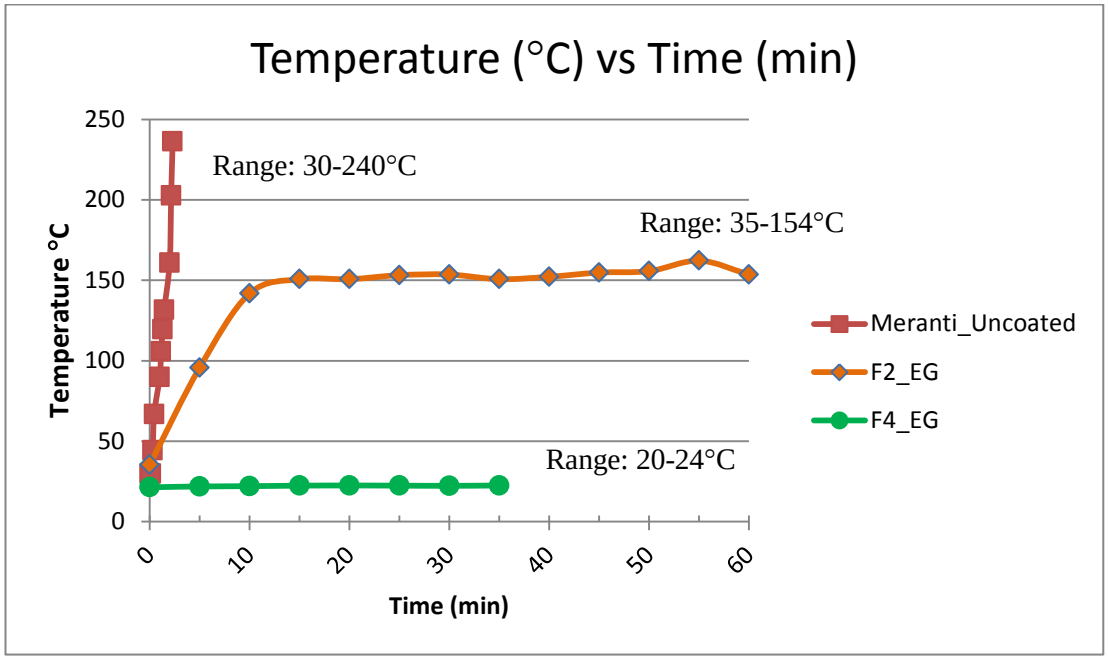


Figure 23: Thermal Behaviour of Wood samples with color

The samples with color of sample F2_EG and F4_EG were burned to analyze the performance of coating on heat shielding effect. Fig.23 showed the result of thermal behavior of the coating. The lower the temperature on the backside of wood, the better heat shielding effect which indicates a better intumescent properties. Sample F4_EG with colors exhibit a good properties of heat shielding effect with wood backside temperature of 24°C at 30min of burning.

It is recommended that the coating with color powder to be further experimented with:

(i) Weather reliability: Study the effect of weather on the performance of coloured coating.

(ii) Gas Chromatograph Mass Spectrometer (GCMS): Study the gaseous product from the coloured coating.

CONCLUSION

From the analysis, sample F4_EG consist of EG-APP-Mel-BA-ATH-epoxy and hardener mixtures gives the best result with a good heat shielding effect on the wood samples. The temperature of wood backside was lowered to 22°C after 7min and burning was continued till 40min with temperature of wood backside at 35°C. Besides, FESEM analysis of F4_EG on wood samples also showed the morphology of residue char which is improved by using the expendable graphite. Formations of small cracks, holes and folding structures show a good morphology of fire retardant properties. Last but not least, the TGA result showed that expendable graphite increased the amount of residual weight at the end of experiment and sample F4_EG contain the highest amount of residual weight of 56.12% at 700°C temperature. A high amount of residue will limit more heat transfer to the wood thus leads to a better effectiveness of the coating.

PROJECT ACTIVITIES AND KEY MILESTONES

Several targets have been set for the FYP I and FYP II. Table 6 and Table 7 show the project activities and key milestones for FYP I and FYP II respectively.

Table 6: Project Activities and Key Milestones for FYP I

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic	■	■													
									M							
2	Preliminary Research Work / Literatures Review		■	■	■	■			I							
									D							
3	Submission of Extended Proposal						○									
									S							
4	Extensive Research on Intumescent Formulations							■	E	■						
									M							
5	Proposal Defense									■	○					
									B							
6	Selection and preparation of intumscent formulations for wood								R			■	■	■	■	
									E							
7	Submission of Interim Draft Report								A						○	
									K							
8	Submission of Interim Report															○

Legends:

■ Project Activity ○ Key Milestone

Table 7: Project Activities and Key Milestones for FYP II

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Preparation of Coating and Analysis	■	■	■	■	■	■	■		■	■	■	■				
2	Submission of Progress Report								M	○							
									I								
4	Evaluation of Coating Analysis								D			■	■	■			
5	Pre-EDX								S				○				
									E								
6	Submission of Draft Report								M					○			
7	Submission of Dissertation (soft bound)								B						○		
									R								
8	Submission of Technical Paper								E						○		
									A								
9	Oral Presentation								K							○	
10	Submission of Project Dissertation (Hard Bound)																○

Legends:

■ Project Activity ○ Key Milestone

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APPENDICES

The fire test temperatures for sample using Expendable Graphite (EG) were shown as below:

Table 8: Sample F0_EG (0% ATH filler)

Time (min)	Wood Backside Tempt °C	Char Tempt °C
0	26.7	507
2	26.8	121.2
4	27.1	885
6	27.2	895
8	27.4	718
10	27.4	896
12	27.3	737
14	27.6	808
16	27.4	796
18	27.6	712
20	27.6	694
22	27.6	714
24	27.4	710
26	27.7	703
28	27.5	725
30	27.5	789
32	27.3	811
34	27.2	796

Table 9: Sample F1_EG (1% ATH filler)

Time (min)	Wood Backside Tempt °C	Char Tempt °C
0	35.6	448.6
2	57.5	706
4	83.1	776
6	94.2	732
8	79.9	838
10	83.1	772
12	90.6	734
14	83.7	829
16	122	764
18	122.3	681
20	116.2	639
22	113.3	621
24	124.4	645
26	124.7	681
28	125.7	641
30	125.6	655
32	125.9	612
34	124.4	643
36	126.6	536
38	121.2	631
40	118.7	555
42	115.6	863
44	127.9	801
46	138.5	803
48	142.4	851
50	141.7	789

Table 10: Sample F2_EG (2% ATH filler)

Time (min)	Wood Backside Tempt °C	Char Tempt °C
0	35.4	30.6
2	70.9	665
4	90.8	725
6	108.6	687
8	130.2	803
10	141.8	854
12	147.4	761
14	149.3	800
16	148.3	798
18	150.6	883
20	150.7	739
22	152.7	780
24	153.3	905
26	151.3	855
28	153.6	852
30	153.6	933
32	152.2	868
34	150.9	878
36	150.6	873
38	151.1	850
40	152.2	809
42	151.1	794
44	153.6	927
46	157.8	816
48	165.1	942
50	155.7	910

Table 11: Sample F3_EG (3% ATH filler)

Time (min)	Wood Backside Tempt °C	Char Tempt °C
0	42.5	31.3
2	89.9	752
4	115.3	802
6	165.2	828
8	181.1	893
10	197.8	918
12	206.2	945
14	210.9	945
16	219.7	956
18	241.6	974
20	265.6	953

Table 12: Sample F4_EG (4% ATH filler)

Time (min)	Wood Backside Tempt °C	Char Tempt °C
0	21.4	982
2	22.1	877
4	22.2	840
6	22.1	844
8	22.1	769
10	22.1	807
12	22.1	842
14	22.4	733
16	22.6	789
18	22.5	707
20	22.5	808
22	22.6	722
24	22.5	949
26	22.4	934
28	22.6	761
30	22.3	892
32	22.4	920
34	22.4	838
36	22.6	805
38	22.7	821

Table 13: Sample F5_EG (5% ATH filler)

Time (min)	Wood Backside Tempt °C	Char Tempt °C
0	27.7	710
2	27.8	855
4	27.9	779
6	28	757
8	28	900
10	28.1	698
12	28	664
14	28.1	633
16	28.1	605
18	28.1	580
20	28.1	558
22	28.1	538

* Burning time for each sample differs because the wood sample with a low percentage of alumina trihydrate filler burned earlier than the others.