



UNIVERSITI PUTRA MALAYSIA

***ENHANCING RICE HUSK ASH-BASED ALUMINOSILICATE FIRE
RETARDANT ADDITIVE AS A PASSIVE FIRE PROTECTION
MATERIAL***

MOHD NA'IM BIN ABDULLAH

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By

MOHD NA'IM BIN ABDULLAH

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
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Philosophy**

June 2021

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

ENHANCING RICE HUSK ASH-BASED ALUMINOSILICATE FIRE RETARDANT ADDITIVE AS A PASSIVE FIRE PROTECTION MATERIAL

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MOHD NA'IM BIN ABDULLAH

June 2021

Chairman : Faizal bin Mustapha, PhD, PEng
Faculty : Engineering

Several major research studies have concluded that most fire retardants (FR) are associated with numerous environmental and human health concerns, which led them to be replaced by more eco-friendly alternatives. Since Malaysia has been producing abundant of rice husk (RH), the utilisation of rice husk ash (RHA) as an aluminosilicate source in fire-resistant coating could reduce the environmental pollution and can turn agricultural waste into industrial wealth. The main objective of this research is to develop an eco-friendly RHA-based aluminosilicate FR additive. Four experiments were conducted; namely fire resistant, tensile, adhesion, and water absorption tests to assess the microstructure behavior of the developed RHA-based aluminosilicate FR additive. Response surface methodology (RSM) was used to design the experiments incorporating two factors; namely ratio of FR additive to paint and their interrelationship and effect on FR coating properties were analysed using analysis of variance (ANOVA). The relationship of different pre-treatment on the production of silica content from RH were also studied to understand the relationship of each pre-treatment on the produced silica content from RHA. From the fire resistance test and scanning electron microscopy images, higher ratio of RHA-based aluminosilicate contributed a better fire protection efficiency due to the formation of thicker char layer thickness which affected the equilibrium temperature. For both tensile and adhesion strength, the addition of RHA-based aluminosilicate FR improved the ultimate tensile strength and adhesion strength due to dense gel phase which has a well-connected structure and less unreacted particles on the surface coating. However, major drawback on the addition of RHA-based aluminosilicate is the enhancement of water permeation which led to the deterioration of the coating samples. Thus, it can be summarized that silica efficiently improved the strength and compactness of the char layer, which resulted in a relatively higher fire retardant efficiency. In this research, RHA proved to be a good aluminosilicate source alternative for fire retardant additive due to its intumescent process, which can potentially improve building fire safety through passive fire protection.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENINGKATAN ADITIF TAHAN API SEKAM PADI BAKAR BERASASKAN ALUMINOSILIKAT SEBAGAI BAHAN PERLINDUNGAN KEBAKARAN PASIF

Oleh

MOHD NA'IM BIN ABDULLAH

Jun 2021

Pengerusi : Faizal bin Mustapha, PhD, PEng
Fakulti : Kejuruteraan

Beberapa penyelidikan utama telah menyimpulkan bahawa kebanyakan bahan tahan api (FR) dikaitkan dengan pelbagai masalah alam sekitar dan kemudaratan kepada kesihatan manusia, menyebabkan ia digantikan dengan sumber yang lebih mesra alam. Oleh kerana Malaysia menghasilkan sekam padi (RH) yang berlebihan, penggunaan abu sekam padi (RHA) sebagai sumber aluminosilikat dalam salutan tahan api dapat mengurangkan pencemaran alam sekitar dan dapat mengubah bahan buang pertanian kepada kekayaan industri. Objektif utama penyelidikan ini adalah untuk mengembangkan aditif FR aluminosilikat berasaskan RHA yang mesra alam. Empat eksperimen dijalankan; iaitu ujian tahan api, tegangan, lekatan, dan penyerapan air untuk menilai tingkah laku mikro struktur aditif aluminosilikat FR berasaskan RHA yang dibangunkan. Kaedah tindak balas permukaan (RSM) telah digunakan untuk merancang eksperimen yang merangkumi dua faktor; iaitu nisbah aditif FR kepada cat dan hubungannya serta kesan terhadap sifat salutan FR dianalisis menggunakan analisis varians (ANOVA). Hubungan pra-rawatan yang berbeza terhadap pengeluaran kandungan silika dari RH juga dikaji bagi memahami hubungan setiap pra-rawatan terhadap kandungan silika yang dihasilkan dari RHA. Dari ujian ketahanan api dan imbasan mikroskop elektron, nisbah aluminosilikat berasaskan RHA yang lebih tinggi menyumbang kepada kecekapan perlindungan kebakaran yang lebih baik kerana pembentukan ketebalan lapisan arang yang lebih tebal yang mempengaruhi suhu keseimbangan. Untuk kekuatan tegangan dan lekatan, penambahan aluminosilikat FR berasaskan RHA meningkatkan kekuatan tegangan dan kekuatan lekatan utama kerana fasa gel yang padat yang mempunyai struktur yang bersambung dengan baik dan zarah yang kurang bereaksi pada lapisan permukaan. Walau bagaimanapun, kelemahan utama penambahan aluminosilikat berasaskan RHA adalah peningkatan penyerapan air yang menyebabkan kemerosotan sampel salutan. Oleh itu, dapat disimpulkan bahawa silika secara efisien meningkatkan kekuatan dan kekompakan lapisan char, yang menghasilkan kecekapan tahan api yang lebih tinggi. Dalam

penyelidikan ini, RHA terbukti menjadi sumber alternatif aluminosilikat yang baik untuk bahan tambahan tahan api kerana proses intumensinya, ia juga berpotensi untuk meningkatkan keselamatan kebakaran bangunan melalui perlindungan kebakaran pasif.



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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

Faizal bin Mustapha, PhD, PEng

Professor Ir.
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Mohd Halim Shah bin Ismail, PhD

Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Khamirul Amin bin Matori, PhD

Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

ZALILAH MOHD SHARIFF, PhD

Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

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Supervisory
Committee: _____

Signature: _____
Name of Member of
Supervisory
Committee: _____

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LIST OF ABBREVIATIONS

AA	Activated Alkaline
ANOVA	Analysis of Variant
APP	Ammonium Polyphosphate
ASTM	American Society for Testing and Materials
CCD	Central Composite Design
CFD	Computational Fluid Dynamic
DAQ	Data Acquisition
DF	Degree of Freedom
EDX	Energy Dispersive X-Ray Spectroscopy
FAO	Food and Agriculture Organization
FEM	Finite Element Method
FFD	Full Factorial Design
FR	Fire Retardant
GB	Geopolymer Binder
HCL	Hydrochloric Acid
ISO	International Organization for Standardization
LOI	Loss Of Ignition
LPG	Liquid Petroleum Gas
Na	Sodium
Na ₂ SiO ₃	Sodium Silicate
NaOH	Sodium Hydroxide
OH	Hydroxide
OSHA	Occupational Health and Safety Association
POFA	Palm Oil Fuel Ash

PVA/MMT	Poly Montmorillonite
PVC	Pigment Volume Concentrate
RH	Rice Husk
RHA	Rice Husk Ash
RSM	Response Surface Method
SEM	Scanning Electron Microscopy
Si	Silica
TAE	Temperature at Equilibrium
TEOS	Tetraethylorthosilicate
TGA	Thermogravimetry Analysis
TT200	Time Taken to Reach 200°C
UTS	Ultimate Tensile Strength
Wt.	Weight (g)
VOC	Volatile Organic Compounds
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

This chapter describes the research background, problem statement, objectives, scope of the research work and the importance of the study to the engineering community in general and to researchers in particular.

1.1 Research Background

Materials flammability is one of the most important elements that require strict measures and precautions necessary to maintain fire safety, especially for building and construction products. This is due to the growing loss of life because of the spread of fire. Thus, comprehensive research on this issue is the main focus of this study. In order to overcome this consequence, various measuring techniques of fire properties have been developed and improved such as lowering the heat release, controlling ignitability, or improving the extent of flame spread across the surface of flammable materials. Therefore, these factors allow a greater time for people to evacuate to safe areas before the fire takes hold and thus, saving more lives. To ensure the safe use of materials for the end-use environment, the selection of materials is critical for fire behaviour. For example, in order to prevent fire, protection is often needed for cellulosic materials, such as textiles, plastics and wood, whereas protection for steel structures is required to avoid catastrophic failure in the event of a fire.

In the case of fire incident at the World Trade Centre on September 11, it was reported that the collapse of the tower mainly due to the structure and rigidity of structural steel under heat and pressure, not because of flammability (Kiakojouri et al., 2020). Hence, this well-known accident has received attention on the use of fire protection materials in constructions and buildings. Without the fire protection material, people inside the building are at high danger during a building fire. The United States fire department reported that an estimated annual average of 4,100 structure fires in dormitories, fraternities, sororities, and barracks from 2011 to 2015. These fires caused annual averages of 35 civilian injuries and cost \$14 million in direct property damage (Campbell, 2017). Therefore, the fire protection systems of active and passive are advised to be installed in the building for human safety. Fire protection material is one of the passive building fire protection systems which is crucial in delaying the spread of fire, and thus increase the time for human lives to escape the building (Kodur et al., 2019).

The 'classic' process of acid source + char former + blowing agent + binder has been used in the vast majority of intumescent coatings in recent years (Mazela et al., 2020). It contains a large amount of organic compounds, and the foam that develops during intumescence is mainly carbonaceous char. Silicon-based

coatings containing expandable graphite and organoclay particles are among the more recent approaches. These systems have the advantage of producing a more mechanically and thermally stable residue after foaming, making them ideal to be applied at a higher temperatures areas or areas with higher concentrations of abrasive particles or corroding gases (Krueger et al., 2016). Fire-protective coatings for extreme conditions will continue to evolve as a result of increased demands, leading to the production of new materials.

Geopolymers have emerged as one of the most exciting materials in recent years. Geopolymer Binder (GB) is known for its outstanding thermal properties, so it has been used in many industrial applications and has drawn global marketing investments in many categories, such as resin, paint, binder, grout, cement, concrete, ceramic, panels and fiber reinforced composites. (Lahoti et al., 2019). GB material has been verified to demonstrate outstanding fire resistance properties (Basri et al., 2016), high mechanical strength (Neupane et al. 2018), high durability (Mohseni, 2018) and numerous aluminosilicates sources have been used such as fly ash, mekaolin, Palm Oil Fuel Ash (POFA) and dolomite (Zain et al., 2017). Geopolymers possessed high thermal stability and fire resistance properties and would be an ideal alternative to conventional intumescent paint due to their unique properties. However, there is still not as yet well establish research about the foaming process or intumescent of geopolymers.

Furthermore, the progressive global switch from non-renewable (fossil-based) to renewable (plant-based) raw materials has stepped up the hunt for alternative industrial raw materials. The paint and coating industry has also not excluded in this growing demand for renewable materials because several plant-based materials have been introduced, particularly as fillers (Bayer, 2020). Since these products are readily available, silica flour, kaolin, and calcium carbonate are probably the most commonly used fillers in the paint industry (Civancik-Uslu et al., 2018). Rice Husk Ash (RHA) with its known high silica content has a vast potential in offering an alternative to commercial paint as a filler or an additive. In recent studies, RHA has been shown to be ideal used as a filler. It is inexpensive and reusable, and certain mechanical properties of paints can be improved most significantly (Azadi et al., 2011).

Due to potential from previous studies, RHA could be next generation of geopolymer technology and environmentally friendly sources for paint filler or additive. The effective use of RHA-based aluminosilicate as a fire retardant (FR) additive therefore need to be studied so that an efficient coating produced can have maximum advantage. In this study, a novel formulations of RHA-based aluminosilicate FR additive for alkyd paint was developed. This coating is expected to contribute high mechanical properties and auspicious fire resistance properties. Thus, an eco-friendly, efficient, and green product can be produced and has extensive potential application in industry.

1.2 Problem Statement

The rising number of fire incidents in Malaysia has never stopped. From statistics in 2017 alone, 49 cases were reported and half of them involved fire fatalities and losses. On top of that, RM5 billion in losses nationwide was recorded by The Malaysian Fire and Rescue Department because of the fire incidents. According to the Malaysian Fire and Rescue Department deputy director-general (Operations) Datuk Soiman Jahid the losses amount increase up to RM1 billion compared to RM4 billion of the previous year (Bakar, 2018).

Moreover, the deadly fire incident at a religious school in Kampung Datuk Keramat shocked the nation that year. 23 people were killed in the fire at the Darul Quran Ittifaqiyah Tahfiz School (Sahalani, 2019). The incident has been dubbed the worst fire tragedy in the country in 20 years. Several fire incidents have occurred in Malaysia over the last five years, with the most notable cases depicted in Figure 1.1. The most recent incident, which occurred in 2020 at Sultanah Aminah Hospital Johor, demonstrates that Malaysians' awareness on fire safety is still lacking. Despite numerous fire outbreaks, Sultanah Aminah Hospital (HSA) still does not have a fire certificate issued by the Malaysian Fire and Rescue Department (Su-Lyn, 2020).

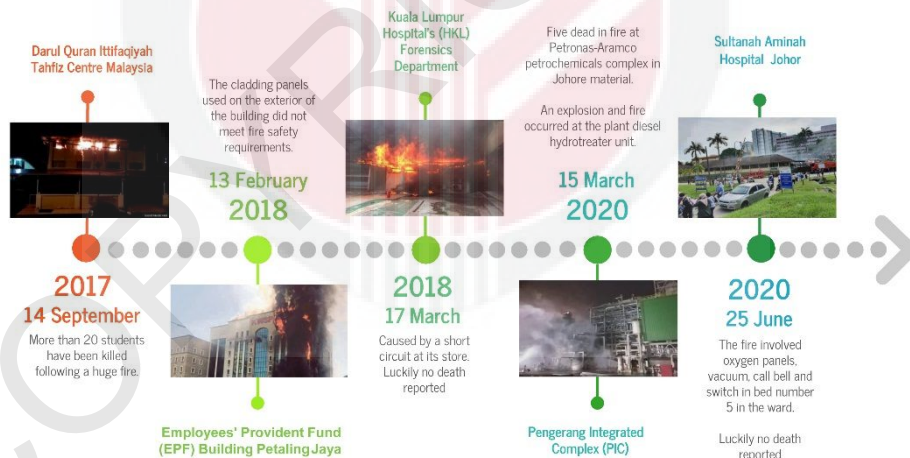


Figure 1.1: Notable cases of fire incident in Malaysia over the past five years.

Fire incidents occurred all over the world not only in Malaysia. One of the worst modern fire tragedies was Grenfell Tower in June 2017. A small refrigerator fire ravaged the 24-story Grenfell Tower apartment building in London. Seventy-one people were killed and was dubbed as Great Britain's deadliest fire incident in decades. An initial key finding of the inquiry's first report in 2019 was that the

external cladding that surrounded Grenfell Tower was largely responsible for the fire spreading so quickly (McKenna et al., 2019). Similar fire incidents also happened in Dubai Torch Tower that year, which involved highly-combustible external cladding panels. This incident demonstrated that the use of highly combustible exterior material leads to rapid fire spread and propagation along the building. More than 40 floors were burning on one side of the building and large quantities of flaming materials fell from the high-level fire which started a secondary fire at lower levels (Chet et al., 2019). Several fire incidents have occurred globally over the last five years, with the most notable cases depicted in Figure 1.2. Another infamous fire incidents was the Notre Dame Cathedral in which destroyed the symbol of the beauty and history of Paris. Approximately 400 firefighters battled the blaze for two days, saving the structure of the gothic cathedral after a fire gutted the roof and destroyed the spire. The blaze, which broke out in the early evening of April 15, 2019, stunned France and others from around the world (Gloennec et al., 2021).

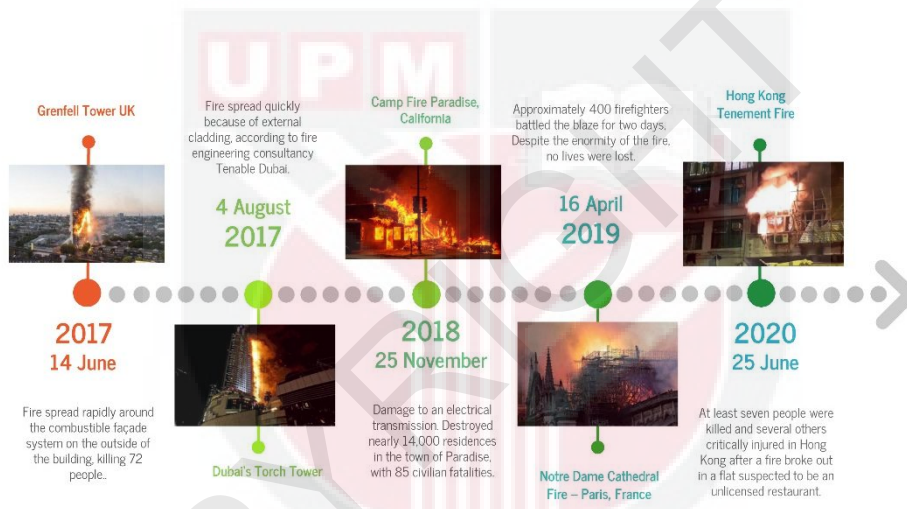


Figure 1.2: Notable cases of fire incident globally over the past five years.

From the data obtained from the Malaysian Fire and Rescue Department for 2017, 96.47% of 5,485 major incidents reported that structural or building fires were caused by accident. Meanwhile, the increase in total fire cases is reported in housing areas. Thus, the numbers highlighted the crucial fact of avoiding structural fires with high fire safety awareness within the community in residential buildings. This is because of the majority of structural fire incidents involved residential properties such as landed, high-rise, squatter and longhouse/traditional house with 60% cases, and followed by shop lots with 16% cases, stores with 12% cases, academic institution with 7% cases and public places with 5% cases as in Figure 1.3. Electrical, cooking gas, and unknown causes were reported as the top causes of residential fires (Chew, 2017).

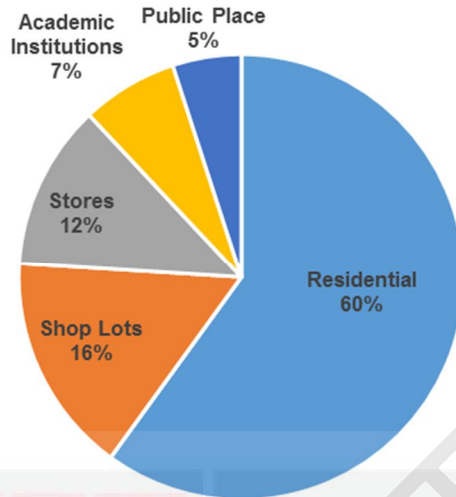


Figure 1.3: Fire incidents by premise types 2016. (Source: Chew, 2017)

In order to avoid a rise in the risk of fire, today's daily products including building materials, furniture, and clothing most made of FRs materials. However, some of these compounds present in the materials had adverse and harmful effects on the environment, hence leading to change to more eco-friendly alternatives in recent years. In the study by Chen et al. (2013), heat or ultraviolet light could decompose FR into potentially harmful compounds (Chen et al., 2013). In the past decades, several major research studies concluded that most FRs, especially halogenated FRs and Organophosphorus FRs are highlighted as major impacts towards environmental and health (Ali et al., 2018). Some of these products have been related to cancer and fertility issues. Moreover, during combustion, they induce an increase in smoke and corrosive gases.

Fire retardant systems that promote the formation of an insulating char layer on the surface of the burning sample are among the most promising environmentally friendly strategies for replacing halogenated fire retardants. In recent years, there has been an increasing interest on the development of a bio-based FRs. Researchers have discovered that Lignocellulosic Plant Fibers (LPF) have a natural defence behaviours against the aggression of fire. Rice husk (RH) which is categorized as a Lignocellulosic Plant Fibers, consist 35% cellulose, 25% hemicellulose, 20% lignin, 3% crude protein and 17% ash. In Malaysia, the Food and Agriculture Organization (FAO) reported that rice paddy production is estimated to increase 0.1 tons every year (Leong, 2015). The growing demand for rice paddy produced about 0.52 tons of RH annually (Macleon et al., 2002). While about 20% of the total grain weight obtained from the average husk weight. Therefore approximately 200 kilograms of husk produced from a ton of rough rice, which is considered as the biodegradable waste product in the rice mill industry and is commonly burned in the open area or dumped in landfills. Rice husk is known as a good source of renewable energy but if improperly incinerated could potentially lead to environmental pollution.

Furthermore, the utilisation of rice husk ash as an aluminosilicate source in fire-resistant coating could reduce the environmental pollution and can turn agricultural waste into industrial wealth. Therefore, an investigation on the addition of RHA-based aluminosilicate in alkyd paint as a FR additive is needed, with the attention of assessing the thermal and mechanical properties of the coating. In summary, below are the research questions formulated from the problem statement mentioned above, and these need to be answered by the end of this thesis;

- i. In terms of thermal and mechanical properties, how synergistic is the use of RHA-based aluminosilicate addition in alkyd paint as a FR additive?
- ii. What are the factors that will influence the ability of the RHA-based FR additive in thermal and mechanical test?
- iii. What is the optimal formulation for RHA-based aluminosilicate FR additive in order to produce the best thermal and mechanical performance?
- iv. Does the silica content in RHA-based aluminosilicate FR additive affects the insulating char layer on the surface of burning sample?

Despite the growing popularity of RHA in various applications, limited research has been devoted to identify the influence of several factors on the properties of RHA-based aluminosilicate. The effective use of RHA-based aluminosilicate as a fire retardant (FR) additive therefore need to be studied so that an efficient coating with good mechanical and auspicious fire properties can be produced. Rather than relying on the classic intumescent process for fire retardant material, an eco-friendly, efficient, and green product can be produced to replace the current existing halogen based fire retardant materials, which proved to be harmful to the environment and health. Ultimately, this research is targeted to enhance and fully utilized the usage of bio waste in FR coating applications.

1.3 Research Objectives

The overall objective of this research is to provide the necessary understanding for the development of an eco-friendly FR additive. Various parameters will be investigated to establish the effect on burning behaviour and the mechanical properties of the coating.

The specific objectives are:

- i. To design experimental works using response surface methodology (RSM) on the thermal and mechanical properties of the addition of RHA-based FR additive in alkyd paint.
- ii. To assess the microstructure behavior and material characterization of the RHA-based FR additive performance under thermal and mechanical test.
- iii. To evaluate the optimum range for RHA-based FR additive, which produced the best thermal and mechanical performance.

- iv. To compare the thermal performance of the RHA-based FR additive using commercial RHA and high purity RHA by investigating the relationship of different pre-treatment on the production of silica content from RH.

1.4 Scope and Limitation of Work

In achieving the objectives of this study, a series of experimental work were conducted. First, response surface methodology (RSM) were employed in experimental design and analysis. A different ratio of RHA-based aluminosilicate FR additive to alkyd paint were prepared. Then, the coating samples were then characterized into four main tests:

- i. Thermal properties (Fire resistant test UL-1709)
- ii. Thermal analysis by using thermogravimetry analysis (TGA)
- iii. Morphological analysis by using scanning electron microscopy (SEM)
- iv. Mechanical properties test (Tensile test, pull-off adhesion test, crosshatch adhesion test, water absorption test)

RH morphological analysis were studied. The parameters of the study are the different pre-treatment through chemical pre-treatment and combustion process on the production of high purity, RHA composition. The collected rice husk was pre-treated with distilled water and hydrochloric acid (HCl) separately. Then, RH are burned in furnace with a temperature of 600°C and 1000°C for duration of one hour and two hours. Subsequently, the RHA are characterize into three main analysis.

- i. Analysis of high-energy radiation by using X-ray fluorescence (XRF) is involved for element characterization.
- ii. Microscopy analysis by using scanning electron microscope (SEM).
- iii. Phase identification analysis by using X-ray Powder Diffraction (XRD).

The major limitations in this study that could be addressed in future research is this study only focused on using RHA to act as a thermal insulation layer source in paint. Other agricultural waste with high silica content could be an alternative resource to replace RHA. Moreover, the development of RHA-based aluminosilicate FR was focused to be incorporated with oil based paint. Further development of formulations should consider the use of a different type of paint such as latex paint. Further research on incorporating the FR additive with latex paint would be useful in order to broaden the potential application of the FR additive

1.5 Thesis Outline

This thesis consists of six chapters. Chapter one discusses the background of the research and problem statement on the current fire retardant materials. The research objectives, scope, limitation of work and outline of the dissertation.

Chapter two presents the extensive review related to the knowledge of this study including of the composition of paints, composition of RHA, factors that influence RHA properties, effect of pre-treatment on RH which affects the mechanical properties of RHA-based paint. The properties of intumescent coating and intumescent phenomenon, as well as the preparation methods are also presented. Literature review on the properties of intumescent coating related to the current study are presented to gauge the development of intumescent coating research. Review on related studies that have been carried out and reported previously for geopolymer, properties of geopolymer were also presented in this chapter. Statistical design method including response surface methodology (RSM) were also reviewed.

Chapter three illustrates the experiment work, materials used, equipment required and sample preparation of the coating samples are described. The mechanical properties, thermal testing and morphological study involved are reported in detail.

Results and discussions are presented in chapter four that includes the thermal properties test, mechanical test, microstructure analysis and fire resistance test. The results are shown in graph and table and supported by statistical data through ANOVA technique. Afterwards, the mathematical model is developed and compared with experimental results. The thermal properties comparison for commercially available RHA and optimum RHA is presented in chapter five. Eight different samples of RHA were analysed to obtain optimal silica content. Samples were analysed through element characterization surface characterization and phase identification.

Finally in chapter six, the conclusions on the mechanical properties and thermal properties of RHA-based aluminosilicate FR on steel substrate are drawn. The effects silica content on the formation of char layer are also summarized. Finally, contribution, and recommendations for future research are also presented.

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