UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF RICE HUSK ASH-BASED GEOPOLYMER BINDER FOR FIRE RESISTANT COATING

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By

MOHD SALAHUDDIN BIN MOHD BASRI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

To Allah SWT, my beloved parents, sisters, and brothers for supporting and encouraging me with affections, love, and their denoted contribution towards success in my life

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

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December 2016

Chair: Faizal Mustapha, PhD, PEng
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Despite the growing popularity of geopolymer in various industrial applications, the literature showed gaps on understanding of factors influential on the properties of geopolymer binder (GB). The main objective of this research is to develop an eco-friendly rice husk ash (RHA)-based GB material suitable as fire resistant coating and panel materials for building construction. Four experiments were conducted; namely fire resistant, compression, flexural, and adhesion tests. Fractional factorial design (FrFD) and response surface methodology (RSM) were used to design the experiments incorporating five factors; namely ratio of activated alkaline (AA) solution, RHA to AA (RHA/AA) ratio, curing temperature, curing time, and sodium hydroxide concentration and analyze their interrelationship and effect on GB properties. The optimum range for GB composition was accordingly determined using superimposed contour plotting. Results showed that RHA/AA ratio was the most influential factor on all GB properties (thermal, mechanical, and physical properties). RHA provided effective fire resistant properties since its GB coating exhibited intumescent-like expansion. The newly developed formulation of GB which exhibited semi-brittle behavior was able to produce good fire resistance and compressive strength properties. In addition, good adhesion and flexural properties were important in providing coating with good fire resistant properties. Optimal GB panels were formulated at W/S and Si/Al ratios of 1.05 to 1.13 and 95 to 110, respectively while optimal GB coating formulated at W/S and Si/Al ratios of 110 to 130 and 1.20 to 1.33, respectively. RHA proved to be the best alternative aluminosilicate source and the RHA-based GB, when used as panel or coating materials, can potentially improve fire safety in building construction.

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

PEMBANGUNAN PENGIKAT GEOPOLIMER BERASASKAN ABU SEKAM PADI SEBAGAI SALUTAN TAHAN API

By

MOHD SALAHUDDIN BIN MOHD BASRI

Disember 2016

Pengerusi: Faizal Mustapha, PhD, PEng
Fakulti: Kejuruteraan

Walaupun populariti geopolimer semakin berkembang dalam pelbagai aplikasi industri, tinjauan literatur mengenalpasti jurang dalam memahami faktor-faktor yang mempengaruhi sifat-sifat pengikat geopolimer *(GB).* Objektif utama penyelidikan ini adalah untuk membangunkan bahan GB berasaskan abu sekam padi yang mesra alam sesuai digunakan sebagai bahan salutan tahan api dan bahan panel untuk pembinaan bangunan. Empat eksperimen telah dijalankan iaitu ujian ketahanan api, mampatan, lekatan dan lenturan. Rekabentuk faktoran pecahan (FFD) dan kaedah tindakbalas permukaan (RSM) telah digunakan untuk menyediakan rekabentuk eksperimen yang melibatkan lima faktor iaitu nisbah larutan aktivasi alkali (AA), nisbah RHA kepada AA (RHA/AA), suhu pengawetan, masa pengawetan, dan kepekatan natrium hidroksida (NaOH) dan mengkaji hubungkait dan kesannya terhadap sifat-sifat GB. Julat optimum bagi komposisi GB dikenalpasti menggunakan plot kontur bertindan. Hasil kajian mendapati bahawa nisbah RHA/AA adalah faktor yang paling mempengaruhi semua sifat-sifat GB (sifat haba, mekanikal, dan fizikal). RHA memberi sifat tahan api yang efektif oleh kerana salutan GB itu mempamerkan kembangan seakan mengembung. Formulasi GB yang baru dibangunkan yang mempamerkan sifat separa rapuh mampu menghasilkan sifat ketahanan api dan kekuatan mampatan yang baik. Tambahan pula, sifat lekatan dan sifat lenturan yang baik adalah penting untuk menyediakan salutan yang mempunyai sifat ketahanan api yang baik. Panel GB yang optimum telah dirumuskan pada nisbah W/S dan Si/Al masing-masing 1.05 ke 1.13 dan 95 ke 110, manakala salutan GB yang optimum dirumuskan pada nisbah W/S dan Si/Al masing-masing 110 ke 130 dan 1.20 ke 1.33. RHA terbukti sebagai sumber bahan alumino-silikat alternatif terbaik dan GB berasaskan RHA berpotensi untuk meningkatkan faktor keselamatan dalam pembinaan bangunan apabila digunakan sebagai bahan panel atau salutan.

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I certify that a Thesis Examination Committee has met on 27 December 2016 to conduct the final examination of Mohd Salahuddin bin Mohd Basri on his thesis entitled "Development of Rice Husk Ash-Based Geopolymer Binder for Fire Resistant Coating" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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

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LIST OF ABBREVIATIONS

2-FrFD	2-level Fractional Factorial Design
AA	Activated Alkaline
Adj MS	Adjusted Mean Square Value
Adj SS	Adjusted Sum of Errors
Al	Alumina
ANOVA	Analysis of Variant
AS	Area of the Inverted Peak
ASTM	American Society for Testing and Materials
BFS	Blast Furnace Slag
CCD	Central Composite Design
CTE	Coefficient of Thermal Expansion
DF	Degree of Freedom
DSC	Differential Scanning Calorimetry
EDX	Energy Dispersive X-Ray Spectroscopy
EEC	Estimated Effect and Coefficient
FA	Fly Ash
Fe	Iron
FFD	Full Factorial Design
FrFD	Fractional Factorial Design
FTIR	Fourier Transform Infrared Spectroscopy
GB	Geopolymer Binder
GBC	Geopolymer Binder Coating
KOH	Potassium Hydroxide
N	Notation
Na	Sodium
Na_2SiO_3	Sodium Silicate
NaOH	Sodium Hydroxide
OH	Hydroxide
OPC	Ordinary Portland Cement
PSD	Particle Size Distribution
RH	Rice Husk
RHA	Rice Husk Ash
RHBA	Rice Husk Bark Ash
RSM	Response Surface Methodology
S/L	Solid-to-Liquid
SE	Standardized Effect
SEC	Standard Error of Coefficient
SEM	Scanning Electron Microscopy
Si	Silica
SS	Sequential Sum of Errors
TAE	Temperature at Equilibrium
TGA	Thermogravimetry Analysis
TT300	Time Taken to Reach 300°C (°C)
Vs	Versus
W/S	Water-to-Solid
Wt.	Weight (g)
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

LIST OF SYMBOLS

%	Percent
$\bar{x}$	Average
°C	Degree Celcius
μm	Micrometer
cm	Centimeter
E	Slope of Proportional Limit (Young's Modulus)
H	Inverted Peak Height
M	Molar
mm	Millimeter
MPa	Mega Pascal
V_1	Ratio of AA Solution
V_2	RHA/AA ratio
V_3	Curing Temperature
V_4	NaOH Concentration
V_5	Curing Time
ε	Compressive Strain
ε_e	Engineering Strain

CHAPTER 1

INTRODUCTION

1.1 Research Background

The collapse of World Trade Center in the United States of America is a turning point in research intensification of fire protection materials. One of the most vital aspects of building regulations is fire protection. Structural integrity, rapid flame spread, smoke and toxic fume emissions are normally the problems caused by fire in buildings. Buildings must be installed with active and passive fire protection systems. Active fire protection systems such as extinguishers, water sprinklers, and fire detectors work to effectively combat fire at the beginning of fire outbreak. At the same time, passive systems act as a sub-defense unit which is extremely important to control rapid fire spread and protect the structural integrity of the buildings.

Coatings and panels are passive fire protection systems. Steel, which is largely used in building construction, normally is unable to withstand high temperatures between 470°C and 500°C causing it slowly to lose its strength. Without coating, the temperature of unprotected steel increases rapidly due to high thermal conductivity of steel. Therefore, unprotected steel structures do not have desirable resistance against fire resulting in rapid creeping, buckling, collapse or other failure. For safety reason, fire resistant coating materials are necessary to keep the temperature of steel structures below the critical temperature during fire incidents. On the other hand, core panel such as in sandwich wall panel or fire door is unduly important to prevent fire from spreading to other side of the wall or door.

New fire resistant building materials are required for higher sustainability and durability. Since overall cost components in building constructions come from building materials and labor, the need for lower-cost and time-efficient technology becomes urgent. ‘Geopolymer’, a term coined by a French scientist named Joseph Davidovits, is the best alternative which is environmentally friendly and possesses excellent mechanical and thermal properties including good fire resistance and insulation properties (Davidovits, 1989).

For the last 30 years, geopolymers were applied in various field of applications. One of the first objective in geopolymer development is to be an alternative to existing fire and heat resistance organic thermosetting polymer which were insufficient as passive fire protection during a tragic fire in France around 1970 (Davidovits, 2002). Geopolymers or activated-alkaline aluminosilicate are prepared by synthesizing activated alkaline solution with aluminosilicate sources such as waste sources including fly ash, metakaolin, kaolin, blast furnace slag, palm oil fuel ash and rice husk ash (RHA).

According to Food and Agriculture Organization of the United Nations (2015), global annual rice paddy production between 2014 and 2015 was around 745 million tons. In Malaysia, rice paddy production in the same period was approximately 2.6 million tons. In general, more than 149 million tons of rice husks are produced annually. RH is one of the largest available biomass resources and the low cost of RH may potentially reduce the costs of geopolymer through lowered cost of production compared to other resin systems in the market such as polyimides, cyanide esters, silicone, epoxy, polyurethane and acrylic resins (Han *et al*., 2008) (Oyetola and Abdullahi, 2006).

1.2 Problem Statement

Coatings which are widely used for protective purpose contain combustible material that will easily burn during a fire event. Although the coating may have effective fire resistance properties, if it is burnt and emit smoke, especially toxic smoke, the emission of burnt steel smoke may pose a high risk to the community in case of fire events. Current coatings mainly available in the market are epoxy resin, whilst intumescent coating materials are less used due to lack of information.

A building structure reinforced with steel but with poor or insufficient coating may fail to protect its function in load-bearing resulting in building collapse. In order to prevent such catastrophic event, the coating must be able to withstand bending moment before the event thus preventing cracks development in the coating. Eco-friendly coating should possess good fire resistance properties in order to function as an effective shield to protect steel from early degradation during fire event. With the use of fire resistant coating, fire rate can be reduced, allowing longer opportunity time for escape and more lives could be saved in fire catastrophes. Initiative must be taken as well in providing not only better durability and longevity of the whole coating system, but also coating with effective good bonding with the steel surface before and during the fire event.

Existing wall panels in the market have poor fire resistance properties in some of the materials which are widely used such as resins, polystyrene, and polyurethane (Zhang *et al*., 2014). Therefore, environmental-friendly and energy consumption factors have to be crucially considered as alternatives to current wall panels. Although some of the panel structures, used as insulation materials, should possess high fire resistance characteristic, their mechanical requisites including compressive strength for walls and doors must not be unduly compromised. In addition, intensive research studies on the relationship between compressive strength and fire resistance properties of fire resistant materials, using statistical analysis approach, are not as yet well established.

Geopolymer binder has been proven to possess excellent fire resistance properties and various waste materials have been used in producing geopolymer binder including fly ash, metakaolin, kaolin, and blast furnace slag. Although RHA was found to contain the highest silica to alumina ratio among agricultural wastes, the utilization of this resource is still low due probably to several reasons including; (i) low awareness of RH potential among farmers and industrial communities, (ii) information on the material and its utilization was insufficiently known to the public, (iii) socio-economic problems, (iv) low penetration of technology, (v) lack of interest and (vi) lack of awareness about environmental problems (Omatola and Onojah, 2012).

In Malaysia, rice husk is considered biodegradable waste product in the rice mill industry and is burned in open area or dumped in landfills. Rice husk is a good source of renewable energy but if improperly incinerated, this vast quantity of waste may potentially pollute the environment including the air which through open burning, soil and water. Through large-scale industrial utilization of RHA as an aluminosilicate source in fire resistant geopolymer material, the environmental pollution can be reduced through the application of green technology in the manufacture of more sustainable eco-friendly and cost effective geopolymer binder (GB) material.

Despite the growing popularity of geopolymer in various applications, limited research has been devoted to identify the influence of several factors on the properties of RHA-based geopolymer binder. In this study, a new and novel type of geopolymer binder based on RHA was developed for coating and panel applications, particularly as a construction building material. Using statistical approach, this novel formulations of RHA-based geopolymer binder are expected to possess high compressive strength, good adhesion bonding, promising fire resistance properties, and good flexural strength and flexural modulus. The new product, based on RHA, should have efficient and cost-effective properties, with wide potential applications in industry.

1.3 Research Objectives

The main objective of this study is to design experimental work using suitable statistical method and develop a RHA-based geopolymer binder product which is suitable for fire resistant coating and panel materials for various applications, particularly in the construction building industry.

Along with the main objective, there are several specific objectives including:

i. To design experimental works using fractional factorial design (FrFD) and response surface methodology (RSM) and conduct experiments on fire resistant, compressive strength, flexural strength, flexural modulus, and adhesion strength properties of RHA-based geopolymer binder.

ii. To statistically analyze the experimental data to determine the effect of each factor, including ratio of activated alkaline (AA) solution, RHA/AA ratio, curing temperature, sodium hydroxide (NaOH) concentration, and curing time, and its interactions on the properties of the RHA-based geopolymer binder and respectively conduct their optimization tests.
iii. To investigate and characterize the microstructural and element characteristics of RHA-based geopolymer binder.
iv. To identify the optimum range for silica to alumina (Si/Al) ratio and water to solid (W/S) ratio in RHA-based geopolymer binder compositions which produced the best mechanical, thermal, and physical properties of RHA-based geopolymer binder.

Although the best binder may be discovered during the research process, it should be noted that the purpose of this study is not to solely develop the best or strongest possible binder material for coating and panel applications, but rather to study and identify the characteristics of RHA-based geopolymer binder that can be the best alternative as fire resistant binder material in the present market.

1.4 Research Scope

In achieving the objectives of this study, experimental works consisting of fire resistant, compressive, three-point bending (flexural), and adhesion tests were conducted based on three main properties; namely, thermal, mechanical, and physical properties. For this purpose, five factors were considered, namely; (i) ratio of activated alkaline (AA) solution, (ii) rice husk ash to activated alkaline (RHA/AA) ratio, (iii) curing temperature, (iv) sodium hydroxide (NaOH) concentration, and (v) curing time. RHA was used as aluminosilicate source whereas sodium silicate and NaOH solution as the AA solution. The geopolymer binder (GB) used in this study was fabricated into two main types of material; coating and panel. Mild steel plate was used as a substrate throughout this study. Statistical methods, including fractional factorial design (FrFD) and response surface methodology (RSM) were employed in experimental design and analysis.

The geopolymer binder materials were characterized into seven main tests including (i) mechanical properties test by using three-point bending (flexural) test, and compressive test, (ii) thermal properties test by using fire resistant test, (iii) physical properties test by using pull-off adhesion test, (iv) thermal analysis by using thermogravimetry analysis (TGA), differential scanning calorimetric (DSC), and coefficient of thermal expansion (CTE), (v) microscopy analysis by using scanning electron microscopy (SEM), (vi) element characterization by using energy dispersive X-ray spectroscopy (EDX), fourier transform infrared spectroscopy (FTIR), x-ray fluorescence (XRF), x-ray diffraction (XRD), differential scanning calorimetric (DSC), and (vii) particle size analysis by using particle size distribution (PSD).

1.5 Thesis Organization

This thesis consists of eight chapters. Chapter one discusses the background of the research and problem statement, followed by an outline of the research objectives, the scope and organization of the dissertation.

Chapter two presents the literature review for related studies that have been carried out and reported previously including rice husk ash, geopolymer, properties of geopolymer, influential factors, and statistical design method including fractional factorial design (FrFD) and response surface methodology (RSM). Chapter three describes the methods in conducting experiments, analyzing data and results on the preliminary findings of rice husk ash.

Results and discussions are presented in chapter four until seven. Chapter four presented the results and discussions of thermal properties (fire resistant) of RHA-based geopolymer binder coating (GBC). Chapter five discussed the properties of the binder under mechanical properties including compressive strength, flexural modulus, flexural strength. Chapter six discusses the results for physical properties (adhesion strength) of RHA-based GBC. Chapter seven discusses the optimum range of GB compositions namely Si/Al ratio and W/S ratio which produced the best properties in terms of thermal, mechanical, and physical properties. Finally, the conclusion, contribution, and recommendations for future research is given in Chapter eight.

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LIST OF PUBLICATIONS

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