THE MECHANICAL PROPERTIES OF TREATED RICE HUSK ASH (TRHA)MORTAR

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A project report submitted in partial fulfilment of the requirements for the award of Bachelor of Science (Hons.) Construction Management

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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

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ABSTRACT

Cement is an important material in construction industry which widely use nowadays. However, it is a non-environmental friendly product. One tonne of cement produce same amount of carbon dioxide (CO_2). Besides that, the calcium hydroxide $Ca(OH)_2$ contained in cement product will susceptible to chemical attack when react with CO_2 which turn the cement product become acidic. The $Ca(OH)_2$ will dissolved and escaped from concrete after contacted with water hence pores were formed inside the concrete. Hence supplementary cementitious materials (SCM) should be investigated to lower down the usage of cement and increase the concrete performances. The rice husk (RH) is an agriculture waste which was by-product of rice production. RH typically send to burn or landfill and release harmful gaseous which pollute the environment and harm to people. A large amount of amorphous silica (SiO_2) is produced when the RH was undergone a control incineration. However the SiO_2 content can be increased by giving an acid pre-treatment for RH to remove the metallic impurities on RH. The treated rice hush ash (TRHA) which contained high amount of SiO_2 is suitable to be used as SCM to substitute some of the cement in concrete. This study was determined the treatment method for rice husk and optimum replacement ratio of (TRHA) in mortar. Some tests such as workability test, compressive strength test, porosity test and gas permeability test was carried out to determine the performances of TRHA mortar. The results showed the 5% of TRHA where RH undergone citric acid treatment with 80 ℃ gave the highest strength and 5% to 15% of TRHA as partial replacement material for cement has lower porosity and permeability than conventional mortar. Further study on acid treatment of rice husk with different molarity of citric acid as well as the period of treatment without extra heat can be carried out.

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$Ca(OH)_2$	Calcium Hydroxide
CH_4	Methane Gas
CSH	Calcium Silicate Hydrate
CO	Carbon Monoxide
CO_2	Carbon Dioxide
H_2SO_4	Sulphuric Acid
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
K	Potassium
Na	Sodium
NaOH	Sodium Hydroxide
NH_4OH	Ammonium Hydroxide
NO	Nitrogen Oxides
SiO_2	Silica
SO_2	Sulphur Dioxide

kg	Kilogramme
kg/m^3	Kilogramme per Cubic Metre
kW	Kilowatt
M	Mol
N	Normality
ml	Millilitre
mm	Millimetre
cm	Centimetre
m^2	Square Metre
cm^3	Cubic Centimetre

μm	Micrometre
Nmm^{-2}	Newton per Square Millimetre
cm^3/s	Cubic Centimetre per Second
s	Second
RH	Rice Husk
RHA	Rice Husk Ash
RRH	Raw Rice Husk
RRHA	Raw Rice Husk Ash
TRH	Treated Rice Husk
TRHA	Treated Rice Husk Ash
SCM	Supplementary Cementitious Materials
SEM	Scanning Electron Microscope
VRC	Vibrated Reinforced Concrete
w/c	Water : Cement Ratio
XRF	X-ray Fluorescent
ASTM	American Society of the International Association for Testing and Materials
BS	British Standard
IS	Indian Standard
RILEM	International Union of Laboratories and Experts in Construction Materials, Systems and Structures
D_1	Diameter of flow (mm)
d	Original diameter (mm)
K	Intrinsic Permeability (m^2)
P_1	Absolute Applied Pressure Bars (Atmosphere Pressure) Usually 2 Bars
P_2	Pressure at which the Flow Rate is Measured (Atmosphere Pressure) Usually 1 Bar
A	Cross Sectional Areas of Specimen (m^2)
L	Length of Specimen (m)
V	Flow Rate (cm^3/s)

D	Flowmeter Diameter (cm)
H	Length Read on Flowmeter (cm)
T	Average Time (s)
P	Porosity (%)
W_1	Weight of Specimen in Air after Passed through the Porosity Test (kg)
W_2	Weight of Dry Specimen (kg)
W_3	Weight of Specimen in Water after Passed through the Porosity Test (kg)

LIST OF SYMBOLS

%	Percentage
wt. %	Percentage by Mass (Weight)
^{o}C	Degree Celsius

CHAPTER 1

INTRODUCTION

1.1 Background

Nowadays, the construction sector becomesthe vital part in enhancing the economy of a country. Construction is defined as the process to create and build the building or infrastructure such as shopping mall, school, hospital, bridge, road, semi-detached house, factory etc. The population of people is the most important factor that affects the number construction projects. The prime purpose of construction is fulfill the need of people, as the number of people increase, the number of buildings or construction projects also increase.

At the ancient time, human was lived near to the river or in the caves. The knowledge of humans was increased as time increased. People tried to use stones or timbers as the materials to construct their houses or purpose buildings. Near 1840, Portland cement was introduced and being used as the main material to replace the timber and stone (Nawy, 2008). It is because the cement's product- concrete was more flexible to use compare to timbers and stones. One of the examples is: not all the timbers are suitable to use in construction for the structural support and those timbers sometimes are also hard to find. Therefore, concrete was brings many advantages to people in construction industry.

Concrete is a composite material that strong in compression but weak in tension. The concrete will crack when there is not enough strength to overcome the force. Therefore reinforcement bars are added into concrete to increase the tensile strength. This concrete is so called vibrated reinforced concrete (VRC).

Cement is playing the role as the agent to bind the aggregate and sand which contribute strength to the hardened concrete. Cement will has chemical reaction become hardened when contact to water (Nawy, 2008).On the construction site, the cement’s quality can be guaranteed by the supplier like other materials. A good quality of cement will give the concrete structure of good performances in terms of durability, strength and etc. Cement in concrete can be said like flour in the sponge cake, and the cook is manipulating the quality of the sponge cake (Neville and Brooks, 2010).Hence a good quality cement not necessary will produced a good quality concrete if the procedures of mixing concrete, ratio of concrete mixture, other factors such as curing are not take in consideration.

According to previous research in United States, there was 70 percent of the total cement (83.3 million tonnes) produced approximate 58.31 million tonnescontributed to ready mixed concrete producers, concrete product manufacturers used 11 percent (9.163 million tonnes), 9 percent (7.497 million tonnes) for contractors used especially road paving, oil and gas well drillers and building material dealers each used 4 percent (3.332 million tonnes), lastly the remaining 2 percent (1.666 million tonnes) was other uses(Van Oss, 2015).

Table 1.1 showed the quantity of cement production (milliontonnes) was increased from 2013 to 2014 due to high spending levels for new non-residential and residential constructions (Van Oss, 2015).

Table 1.1 World Production and Capacity (Van Oss, 2015)

	Cement Production, million tonnes		**Clinker Capacity, million tonnes**	
	2013	2014	2013	2014
U.S.	77.4	83.3	104.3	104.3
Brazil	70	72	60	60
China	2,420	2,500	1,900	2,000
Egypt	50	50	46	46
Germany	31.3	31	31	31
India	280	280	280	280
Indonesia	56	60	51	50
Iran	72	75	80	80
Italy	22	22	46	46
Japan	57.4	58	55	55
Korea, Republic	47.3	47.7	50	50
Mexico	34.6	35	42	42
Pakistan	31	32	43.4	44
Russia	66.4	69	80	80
Saudi Arabia	57	63	55	55
Thailand	42	42	50	50
Turkey	71.3	75	68.6	69
Vietnam	58	60	80	80
Other Counties (rounded)	536	525	348	349
Total	4,080	4,180	3,470	3,570

Nowadays people are tried to use some supplementary cementitious materials (SCM)to modify the concrete by substitute some portion of Portland cement. The main reason to use SCM in concrete is SCM believed can contribute to lower the carbon dioxide (CO_2) in concrete compared to pure Portland cement concrete (Lothenbacha, Scrivenerb and Hooton, 2011). The combination of SCM with cement produces a more complication reaction. Portland cement's hydration and hydraulic reaction of SCM can occur at the same time and they may influence each other unlike Portland cement which only has simple reaction.The advantages of use SCM in concrete mix are improve the concrete durability, lower the greenhouse gaseous emission, increase concrete strength and etc.

Recently people found that the rice husk ash (RHA) which pass through a pre-treatment process can produce higher content of silica (SiO_2) compare to untreated RHA. There are few methods that generally use by people to produce higher purity silica in RHA such as acid leaching, basic pre-treatment. However acid leaching showed the better result compare to basic pre-treatment in getting the high purity silica (Ugheoke and Mamat, 2012). Acid leaching is actually a treatment for rice husk before it sends to incinerate. The rice husk (RH) was soaked in the acid to remove the metallic impurities and sent to incinerate after washed few times until there was no acid in the RH. Many researches are focused on this direction as the worthiness and feasibility of RHA as SCM in concrete was high.

1.2 Problem Statement

Due to the development of a country, many construction activities are rise day by day and it directly increase the production of cement. However the cement is not an environmental friendly material, it will release a lot of CO_2 and other greenhouse gasses. One tonne of cement production produced the same amount of CO_2(Gautam, Krishna and Srivastava, 2014).Some research showed that concrete industry was the world largest user of natural resources, most of the raw materials used each year produced large quantity of CO_2 (Mehta, 2002). The global CO_2 gas for cement production was emitted almost 829 million metric tons in 2000 (Hu and Kavan, 2014). It was a serious case in the global warming issue. The production of cement increased due to increase of construction activities (Utusan online, 2009). Figure 1.1 showed the global CO_2 emission in percent. In the cement production, clinker is the used the most energy and release most CO_2 (Zhu, 2011). It is a necessary to replace the clinker by other SCMs to reduce the CO_2 emissions.

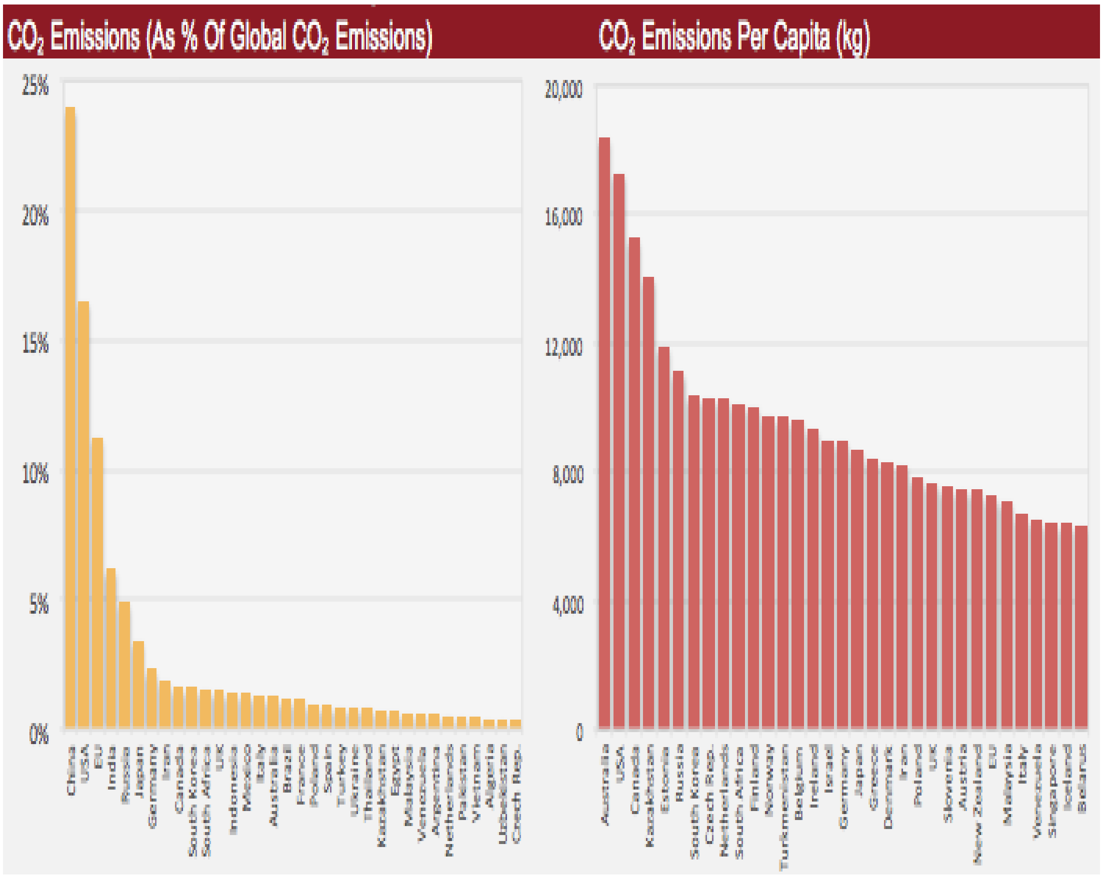

Figure 1.1 The Dirtiest Countries by CO_2 Emissions in 2009 (Helgi Library, n.d.)

The concrete contain of 25 wt.% to 50 wt.% calcium hydroxide ($Ca(OH)_2$) which susceptible to the chemical attack. It will react with the CO_2 in the air to have another reaction called carbonation. Carbonation of concrete reduces the pH value of the concrete towards acidic which will harm to the reinforcement bar inside the concrete. Besides that, when $Ca(OH)_2$met calcium chloride, it will change it chemical reaction and cause the bonding become weaker and reduce the strength. Hence concrete need SiO_2 which can form a continuous connection within the cement particles to increase its bonding force so that its strength is also increases.

There was calcium hydroxide in the mortar and concrete. According to Thomas and Jennings (n.d.), calcium hydroxide is found and appeared or formed at the capillary pores inside the OPC paste. It has a weak bonding with the hardened concrete due to it is a soluble substance. When the concrete contact to the water, the calcium hydroxide will dissolve and escape from the hardened concrete.Thus pores formed and directly affect the durability of concrete and lead it weaker for chemical attacks.This problem can be solved by calcium-silicate-hydrate (C-S-H) gel which is smaller and bind the cement particles into a continuous form. Besides that, the C-S-H gel also can fill up the pores inside the concrete hence the durability of concrete will increase. The concrete has low amount of C-S-H gel hence it is necessary to add on SCM to insert the amount of silica to produce the C-S-H gel in the concrete to increase its mechanical properties and durability.

As the population of human being increases, the demand for the food especially rice also increases. This situation directly contributed large quantity of waste (rice husk) in our world. Around 649.7 million tonnes rice produced every year worldwide (Habeeb and Mahmud, 2010) and the rice husk was 20 % (Northoff, 2008)of it about 130 million tonnes. That means there will almost 130 million tonnes of waste is producing annually. Table 1.2 showed the paddy and the RH production in Malaysia. It can be seen that the by-product from rice was increasing year by year.

Table 1.2 Paddy Production, Rice Production and Rice Husk Production in Malaysia by Year (Department of Agriculture, 2014)

Year	Paddy Production, metric tonnes	Rice Production, metric tonnes	Rice Husk Production, metric tonnes
2004	2,291,352	1,467,052	824,300
2005	2,314,378	1,490,015	824,363
2006	2,187,519	1,407,221	780,298
2007	2,375,604	1,530,971	844,633
2008	2,353,036	1,516,474	836,562
2009	2,511,043	1,620,256	890,787
2010	2,464,831	1,588,456	876,375
2011	2,578,519	1,661,260	917,259
2012	2,599,382	1,674,981	924,401
2013	2,615,845	1,685,236	930,609

Due to the protein contain in RH is very low and RH also a low digestible substance hence it is not suitable for human to eat. Typically RH will be solving by two ways: combustion and landfill. During the combustion process, the harmful gaseous been released such as CO_2, carbon monoxide (CO), nitrogen oxides (NO), sulphur dioxide (SO_2) and etc. These gaseous will seriously affect human being's health and environment to global warming, increase the earth temperature. The sulphur dioxide gas will contribute to the acid rain which can harm the natural environment and built environment (National Park Service, 2013). The aquatic life will die due to the pH value of the water decrease become acidic, the trees will die too because they can't survive in acidic environment. For sulphur dioxide in acid rain will corrode the steel structure of the building, the building will become weaker and may tend to failure.

Besides that, rice husk need large area for landfill and when the rice husk is being decomposed, it will release heat, methane gas (CH_4) and CO_2 which cause the global warming. CH_4 is a gas that can trap heat 21 times more than CO_2 (Mark, 2012).

Send the RH to landfill is meanings to increase the global warming rate. Moreover the landfill will release the smell which caused by the ammonia and hydrogen sulphites during the RH being breakdown by microorganism (New York State Department of Health, 2012).Other than that, the time for RH to decompose completely is very long because RH is non-biodegradable material, it hard to decompose compare to biodegradable material. Landfill also will cause the water pollution. The water pollution formed when there is water pass through the RH which undergoing decomposes process. This liquid contains of virus and toxic which is very danger and harmful to human when it penetrate to nearby water supply, groundwater or river. Most of the ways to solve the problem of rice husk are non-environmental friendly.

The RHA has promising high SiO_2that can improve concrete performance. It contain of 87-92 wt.% of SiO_2 (Ugheoke and Mamat, 2012). The SiO_2 form a stronger bonding and give higher compressive strength and durability of concrete(Nair et al., 2008).However, SiO_2 content of RHA can be lifted up by pre-treatment to remove the metallic impurities on the RH. Some of the studies found that the bonding between the oxides especially potassium (K) and sodium (Na) affect the percentage of SiO_2 in RHA. If want to obtain the higher percentage of $SiO_{2,}$ there must has some treatment for RH.

Besides that, the pre-treatment for rice husk ash can further separate into few methods: acid leaching, basic pre-treatment and carbonisation. Many studies found that acid leaching pre-treatment for rice husk ash required certain temperature and time to effectively decompose the metallic impurities inside the rice husk. When thetemperature needs to produce treated rice husk ash (TRHA) increase, the cost of TRHA also will increase. There is necessary to investigate a more convenient and cheaper RHA treated method. The most preferable way is acid leaching at room temperature, short period and with low concentrated acid.

1.3 Aim and Objectives

To determine the treatment method for rice husk and optimum replacement ratio of TRHA in mortar. In this study, the following objectives been formulated in order to achieve the aim of this study.
Objective 1
To investigate the mechanical properties of TRHA mortar
Objective 2
To determine the durability of TRHA mortar

1.4 Significant of Study

This study can bring a lot of benefits to human and the environment. First of all, it can reduce the environment impact that brings from cement and RH such as the waste produce from them and CO_2 emission especially during cement production and its outcome. This study is tried to use the agriculture waste to replace construction material whichis not environment friendly to reduce the emission of CO_2 besides reduce the agriculture waste.

The previous researches showed that it is possible to insert the RHA into the concrete and mortar which can improve the performance such as compressive strength and durability (Sata, Jaturapitakkul and Kiattikomol, 2007; Reddy and Alvarez, 2006).Yet there are still many researches to modify the rice husk ash by giving it some treatment to increase its effectiveness in concrete compare to raw rice husk ash and it is succeed.

This study is very important to human because this technology shows that the rice husk ash which originally was a waste and hazardous material to environment can convert into useful material. For the experimental benefit is maybe can develop an acid leaching method without insert additional heat the acid and something different from previous studies. Moreover, this technology also can turn the rubbish into money.

1.5 Scope of Study

This study is carry out within some scopes due to there are some limitation during this research process. This study is more focus on the laboratory test to analyse and investigate the workability, compressive strength of TRHA mortar compare to control mortar.The limitation for this research is the flexural test will not be carry out due to the material, TRHA not sufficient to carry out the specimens to do the test. Apart from that, the compressive test for hardenedmortar will only conduct until 28 days testing age due to limitation of time. Each compressive strength test for mortar will used three mortar cubes for each group at 3 days, 7 days and 28 days of mortar testing age.

The TRHA is produced at 800^{o}C. While the percentage of TRHA adds into the mortar mixture is 5% which means the mortar cube contain of 5% volume TRHA and 95 % volume cement binder. The mortarsare cast with the size of 50mm x 50mm x 50mm.

Table 1.3 Details of the Mortar Mix Proportion

Mix code	Ordinary Portland Cement, kg/m^3	Sand, kg/m^3	Water Ratio		TRHA 1, kg/m^3	TRHA 2, kg/m^3	TRHA 3, kg/m^3	Super plasticizer, %
			W/C	Sand Weight, %				
CTRL	733.33	1466.67	0.38	3.00	-	-	-	1.00
H 5	696.66	1466.67	0.38	3.00	26.77	-	-	1.00
H10	659.99	1466.67	0.38	3.00	53.54	-	-	1.00
H 15	623.33	1466.67	0.38	3.00	80.32	-	-	1.00
TD 5	696.66	1466.67	0.38	3.00	-	26.77	-	1.00
TD 10	659.99	1466.67	0.38	3.00	-	53.54	-	1.00
TD 15	623.33	1466.67	0.38	3.00	-	80.32	-	1.00
FD 5	696.66	1466.67	0.38	3.00	-	-	26.77	1.00
FD 10	659.99	1466.67	0.38	3.00	-	-	53.54	1.00
FD 15	623.33	1466.67	0.38	3.00	-	-	80.32	1.00

The TRHA 1 is the RH that treated by citric acid at 80^{o}C for 15 minutes meanwhile the TRHA 2 is RH treated by citric acid at room temperature for 72 hours and TRHA 3 is citric acid treated RH at room temperature for 96 hours. All of the citric acid used for leach the RH is 5 % concentration and they will dry at furnace with 100 ^{o}C for 1 hour after washed three times and it is then send to burned at 800 ^{o}C for 3 hours.

The flowtable test is use to carry out the workability of the TRHA mortar which follow the standard of ASTM C 1437 and the compressive strength determine by destructive compressive strength test.

Table 1.4 Tests with the Standard to Investigate the Mechanical Properties and Durability of TRHA Mortar

Test	Standard	Type	Size
Flow Table	ASTM C 1437	Fresh Mortar	-
Compressive Strength	BS EN 12390-4 (2000)	Cube	50mm x50mm x50mm
Porosity	RILEM CP 11.3	Cylinder	45mm diameter x 40mm height
Gas Permeability	Lynsdale Cabrera 1984	Cylinder	45mm diameter x 40mm height

1.6 Chapter Outline

Chapter 1: Introduction

Cement and RHA are discussed in this chapter to have a clear mind-set about them. In this chapter also discuss the problems that bring by cement and RHA either toward concrete or environment, aim and objectives of this study, significant and scope of this study.

Chapter 2: Literature Review

This chapter will discuss the experiments or studies done by previous researchers to collect some data make this research become successful. Previous researchers' experiences and conclusion are very useful for a new study.

Chapter 3: Research Methodology

The materials use in this study and tests that conduct to achieve the objectives will be discuss and the whole process of the experiment can be more understand at this chapter.

Chapter 4: Results and Discussion

The result collected from the tests will be record and analyse in this chapter.

Chapter 5: Conclusion and Recommendations

The recommendations and the conclusion of this research will be state in this chapter to let the next researcher won't repeat the mistake or get better result.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will discuss about the concrete and the materials used to form the concrete, the RHA and some previous research about RHA in concrete. This is very important to make sure the study that will carry out later be successful.

2.2 Concrete

Concrete is the mixture of cement, supplementary cementitious material, water, aggregates, and chemical mixtures in suitable proportion to obtain a better result in setting time, workability, strength, time to gain strength etc. This type of concrete was the normal concrete that for normal use, it is insufficient for the high quality concrete production. For the concrete that use at the special area such as jetty should be modified by add some other materials. A hardened concrete been defined as brittle material and has low tensile strength, to make it achieve the higher level tensile strength, the fibre or reinforcement bar was added in. When casting the concrete, the proportion must be taking correctly because the ratio will affect the hardened concrete strength. Besides that, the concrete making process must be look seriously and carefully which the concrete should be mixed, placed and cured properly (Nawy, 2008).

Concrete has different grades which represent different compressive strength gain by the hardened concrete at the first 28 days. For example concrete grade 30 produced 30 Newton per square millimetre at 28 days. The concrete been designed with different grades is because different grades of concrete have different applications.

Table 2.1 Concrete Application Grades (Expertsmind.com, n.d.)

Grade	**Mix proportion**	**Compressive strength on 28days, Nmm^{-2}**	**Group (IS: 456-2000)**
M5	1:5:10	5	Lean mix
M7.5	1:4:8	7.5	
M10	1:3:6	10	Ordinary concrete
M15	1:2:4	15	
M20	1:1.5:3	20	
M25	1:1:2	25	Standard concrete
M30	designed	30	
M35		35	
M40		40	
M45		45	
M50		50	
M55		55	
M60		60	High strength concrete

2.2.1 Portland cement

Portland cement act as a binding agent or instant glue when water is added to bonds the coarse aggregate, fine aggregate and sometimes the other materials such as steel fibre to form a concrete (Mamlouk and Zaniewski, 2006). The cement can fill up the voids that formed by coarse and fine aggregates and produce the strength to the concrete when the concrete is setting and hardened due to its reaction with water.

2.3 Rice Husk

RH contains of high quantity of amorphous silica and carbon. In India, around 125,000 small rural villages are lack of electricity supply. People tried to use RH to solve the problem and husk power system was formed which the RH acted as fuel to generate power. First of all, RH is heated by high temperature until it decomposes and release a type of combustible gas. After that, the power will be generated by the turbine which burned the combustible gas released from RH to produce steam or heat (Opic.gov, n.d.). The power generated from it around 25kW- 100kW (Santiaguel, n.d.). The product from heated RH will be sending for other uses such as construction industry.

Figure 2.1 Rice Husk

Table 2.2 Composition of Rice Husk (Bogeshwaran et al., 2014).

Composition	Percentage (%)
Cellulose	31.12
Hemi Cellulose	22.48
Lignin	22.34
Mineral Ash	13.87
Water	7.86
Extractives	2.33

2.4 Rice Husk Ash

RHA obtained from incineration of RH. In every incineration of RH, there will produce 20-22 % of RHA and silica was consists of more than 75 % of the RHA total weight (Ramezanianpour, Mahdi Khani and Hmadibeni, 2009). RHA can be categories as a renewable resource that can produce for annually while natural pozzolan is not. Most of the study were carried out prove that 5-10 % of RHA replaced the cement in the concrete succeed increased the strength of the concrete(Taylor, 2004).

Many benefits can get by insert the RHA into concrete mixture as natural admixture. Besides the strength can be improve, the problem and cost use to deal the RHA also can solved.

Figure 2.2 Rice Husk Ash

2.4.1 Properties of Rice Husk Ash

Once the RH is completely burnt, the cellulose and lignin will be completely removed and remained silica ash. RHA at certain control temperatures have the chemical and physical properties that met American Society for Testing and Materials (ASTM) Standard C618-94a. The percentage of amorphous SiO_2 in RHA depends on the temperature, period of burning and thermal treatment's environment. Typically the higher the thermal treatment temperature, the higher the SiO_2 can obtain in the RHA. The percentage of SiO_2 normally is between 87-92 % of the RHA (Ugheoke and Mamat, 2012). When the temperature is within 550-800 ^oC, the higher the temperature, the more crystalline silica produced (Reddy and Alvarez, 2006).The temperature also will affect the colour of RHA; RHA produced between 300-450 ^oC was carbonised husk while those heated 500-650 ^oC was grey-coloured ash (Kapur, 1985).

Figure 2.3 Carbonised RH and RHA

2.5 Pre-treatment of Rice Husk Ash

Although the SiO_2 obtained from burned raw rice husk (RRH) was around 92 % but there were still have many researchers carry out studies try to improve the SiO_2 in RH. The SiO_2 will react with calcium hydroxide during hydration of cement. Once the reaction completely done, it will produce calcium silicate hydrate which is a substance that can improve the concrete's mechanical properties and it durability.

Researchers found that by applying some treatment for RH before thermal treatment can improve the SiO_2 content in RHA. Therefore many studies were carried out to investigate the most effective way to increase the volume of SiO_2 in RHA.

Typically there are three types of treatment generally used by people to increase the content of SiO_2 in RHA, for example acid leaching, basic pre-treatment and microbiological pre-treatment (Ugheoke and Mamat, 2012). Acid leaching actually is a pre-treatment for RH. Firstly the RH will be send to do a treatment which soak in the acid with certain temperature and period. After that soaking process, the RH will be washing few times until there is totally free from acid and will send for thermal treatment with certain temperature. While the alkali pre-treatment also has the same procedures as acid leaching, it just change the acid into alkali. From the result, researchers found that acid leaching pre-treatment was shown a better result than alkali pre-treatment which used sodium hydroxide (NaOH) and ammonium hydroxide (NH_4OH) as the leaching agent. The acid is more effective in removing the metallic impurities in RH than alkali. Some of the researchers tried the post-treatment for RHA which use the acid and alkali to remove the metallic impurities in raw RHA. However the result obtained was shown negative (Ugheoke and Mamat, 2012). Moreover, although there is a similarity shown in the result of using the microbiological pre-treatment and acid leaching but the time to complete the microbiological pre-treatment is longer than acid leaching. Hence researchers concluded that pre-treatment is the effective and appropriate way to remove the metallic impurities in RH.

There were some studies about acid leaching pre-treatment had been carried out and shown that hydrochloric acid (HCl) can effectively remove the metallic impurities (Kurama and Kurama, 2003; Ugheoke and Mamat, 2012; Salas et al., 2009). One of the researches was shown that HCl more effective than sulphuric acid (H_2SO_4)and followed by nitric acid (HNO_3) in removing metallic impurities in RH, the SiO_2 can be obtain approximately 95 % by using HCl (Ugheoke and Mamat, 2012).

2.5.1 Hydrochloric Acid as Acid Leaching Agent

As many studies shown that HCl was a more effective acid leaching agent, hence there was a study carried out to determine the optimum concentration of HCl, time of contact between HCl and RH and it leaching temperature to get better result. The pre-treatment's procedures in this study are similar to other acid leaching pre-treatment. Firstly RH was washed to remove the impurities by water to get the pure RH for the test. After that they were weighted and put into HCl solution with 100ml. This experiment was carried out by 1, 2, 3 M of HCl at the temperature of 25 ^{o}C, 60 ^{o}C and 90 ^{o}C with different contact times. All RH were burned at 700 ^{o}C which been defined as the optimum thermal treatment (Kula et al., 1996).

The result shown that the SiO_2 was rose from 88 % to 97 % when the time for RH contacted to 1 M acid at 25 ^{o}C increased 2 hours and it remained constant at 97 % as the contacting period increased every 2 hours (Kurama and Kurama, 2003). In this experiment, the concentration of acid did not showed any linear relationship to volume of SiO_2 in RHA. It showed a fluctuating however 2 M HCl had 99.50 % of SiO_2.

The temperature was showed that 60 ^{o}C is the appropriate leaching temperature to obtain maximum content of SiO_2. Table 2.3 shows the RH reacted with 1 and 2 M HCl at 25 ^{o}C, 60 ^{o}C and 90 ^{o}C.

Table 2.3 Relationship between Leaching Agent's Temperature and the Different Concentration of HCl (Kurama and Kurama, 2003)

Temperature, ^{o}C	**SiO_2, %**	
	1 M HCl	2 M HCl
25	97.63	99.50
60	98.44	99.74
90	98.44	99.74

Other study was carried out by Krishnarao, Subrahmanyam and Jagadish Kumar on 2001 to investigate the relationship on the SiO_2 obtained from TRHA and the methods of thermal treatment. The RRH were separated into three groups where two groups leached by 3 N HCl (TRH1) and 5.6 N HCl (TRH2) for 1 hour in boiling condition and another group without any treatment. The treated rice husk (TRH) were washed to remove acid and dried at 110 ^oC before sent to burn at 400 ^oC, 500 ^oC, 600 ^oC and 700 ^oC. However this study used two types of thermal treatment: normal heating and sudden heating. Normal heating was the TRH put into the furnace at room temperature and heated to the required temperature. Meanwhile the sudden heating was direct put the TRH into the furnace at required temperature.

The Table 2.4 shows that the SiO_2 obtained from TRHA were more than raw rice husk ash (RRHA) however there hadnot much different for the SiO_2 obtained from RRHA by normal heating and sudden heating.

Table 2.4 SiO_2 (wt.%) in RHA Burned at 700 ^oC (Krishnarao, Subrahmanyam and Jagadish Kumar, 2001)

	RRH normal heating	**TRH1 normal heating**	**TRH2 normal heating**	**RRH Sudden heating**
SiO_2, wt.%	90.20	92.20	92.30	89.80

Moreover, an experiment was conducted to investigate the SiO_2 content in RHA when it leached with acid at room temperature. This experiment carried out by Salas et al. to compare the SiO_2 obtained from RRH and TRH where the TRH was used HCl as acid leaching agent with concentration of 1 N and the RH was soaking in HCl for 1 day at room temperature. The TRH was washed to remove the acid completely until TRH shows neutral pH value. Both RRH and TRH were sent to burn at temperature of 600 ^oC for 3 hours.

The result showed that TRHA with HCl provided higher SiO_2 compared to RRHA. TRHA obtained 99.0 wt.% of SiO_2while RRHA only 90.0 wt.% SiO_2.

Table 2.5 Chemical and Physical Properties of RRHA and TRHA(Salas et al., 2009)

Chemical analysis, wt.%	TRHA	RRHA
SiO_2	99.0	90.0
Physical properties		
Specific surface BET, m^2/kg	274,000	24,000

Many studies showed HCl acid leaching pre-treatment RH more effectively than RRH in obtaining high volume of SiO_2. This statement is proven by the images taken by Scanning Electron Microscope (SEM). One of the pre-treatment studies about different concentration of HCl in leaching treatment had shown three SEM micrographs: Figure 2.4, 2.5 and 2.6 which each of them was leaching in different concentration of HCl. The silica in RH is mostly focus in the inner epidermis and also at protuberances and trichomes (hairs) which on the outer epidermis (Krishnarao, Subrahmanyam and Jagadish Kumar, 2001). The Figure 2.4 and Figure 2.5 do not show much varying. However when look carefully, there are some areas became slightly smoothly due to it been attacked by acid. Figure 2.6 clearly shown that the RH been attacked by acid. It was shown a more smoothly surface than Figure 2.5 in SEM micrograph. The SEM micrographs prove that acid can remove the organic matter on the surface of RH.

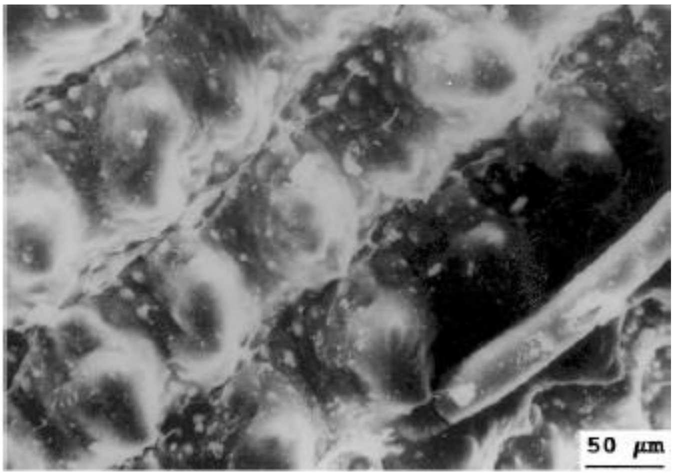

Figure 2.4 SEM Micrographs of Outer Epidermis of RRH (Krishnarao, Subrahmanyam and Jagadish Kumar, 2001)

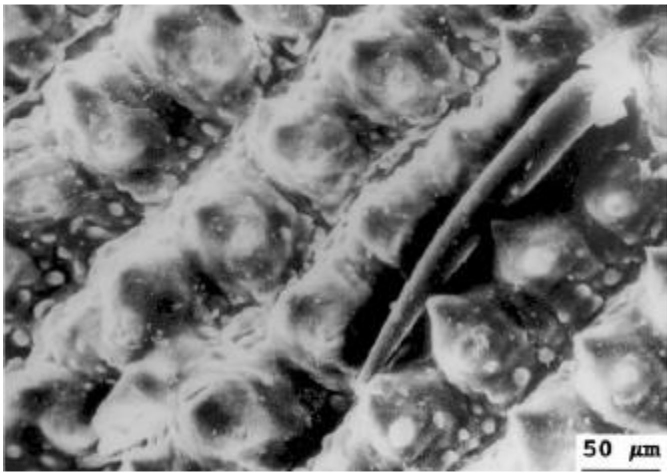

Figure 2.5 SEM Micrographs of Outer Epidermis of RH Treated by 3 N HCl(Krishnarao, Subrahmanyam and Jagadish Kumar, 2001)

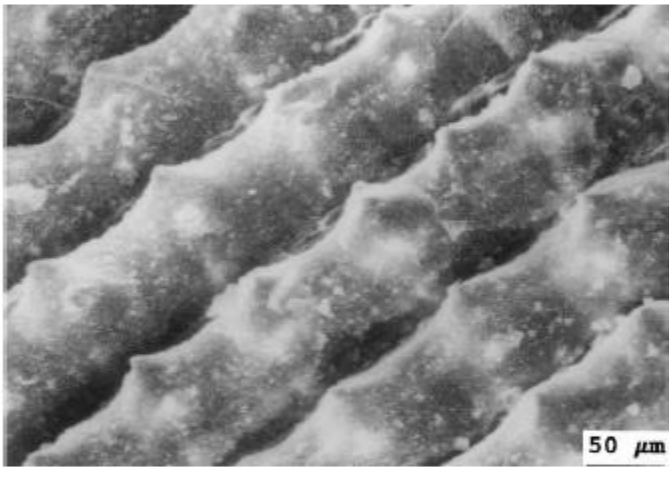

Figure 2.6 SEM Micrographs of Outer Epidermis of RH Treated by 5.6 N HCl(Krishnarao, Subrahmanyam and Jagadish Kumar, 2001)

2.5.2 Oxalic Acid as Leaching Agent

The RH was put into oxalic acid with temperature of 90 ^oC and 1 M concentration for 1 hour. After 1 hour, it was placed at other place to lower down it temperature and the TRH continue soaked in the oxalic acid for 20 hours before it sent to remove the oxalic acid completely. The TRH was put into oven with 110 ^oC temperature to let it dry for 1 day. After 1 day drying process, a thermal treatment been carried out by 700 ^oC with difference hours where 1 to 6 hours. TRH were used to compare to raw RH (RRH) with 3 difference hours.

The result shown that TRHA have the higher SiO_2 content than RRHA. Table 2.6 shows the oxalic acid TRHA burned for 3 hours contained the highest percentage of SiO_2 which 91.26 % among those six specimens.

Table 2.6 SiO_2 of RRHA and TRHA by Oxalic Acid which Burned at 700 ^oC(Olawale et al., 2012)

Thermal Treatment Hour	**1 hour**		**3 hours**		**6 hours**	
Types of RHA	RRHA	TRHA	RRHA	TRHA	RRHA	TRHA
SiO_2, %	86.78	89.87	89.16	91.26	88.74	89.92

2.5.3 Dilute Sulphuric Acid (H_2SO_4) as the Acid Leaching Agent

Using the high concentrated strong acid to running the treatment for RH is not an ideal way to remove metallic impurities in the RH. Hence an experiment using a dilute strong acid which has 5 % concentrated H_2SO_4 to leached the RH. The RH is leached in volume of 500 ml H_2SO_4 with 5 % concentration in 44 ^oC for 15 minutes. After that it was dried at 100^oC and burned at two different temperatures which are 600 ^oC and 1000 ^oC.

The result showed that dilute acid didn't change the morphology of the RH. The SEM shown the RRH and TRHA has high similarity appearance. Dilute H_2SO_4 only removed some of the metallic impurities on the surface. This study stated that the dilute acid was quite different from high concentration acid where high concentrated acid can caused the RH become fragment but dilute acid was not.

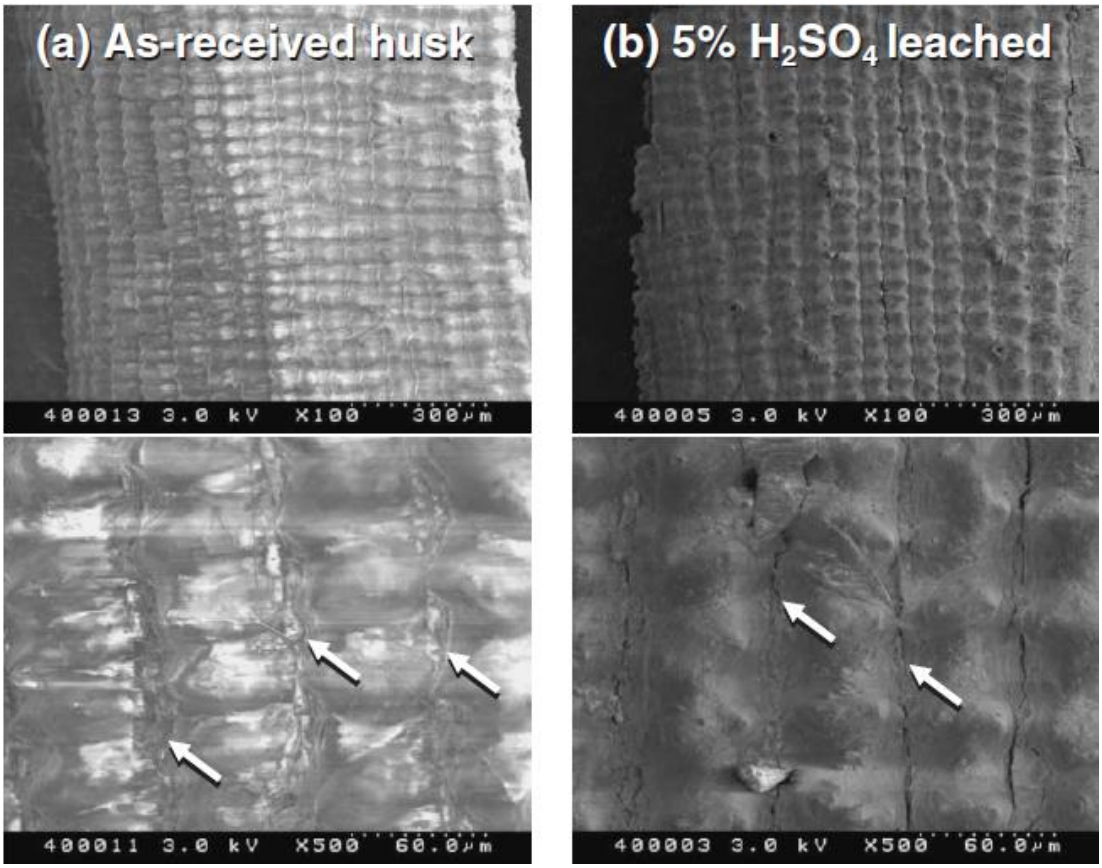

Figure 2.7 The SEM of RRH and TRHA by H_2SO_4(Umeda, Kondoh and Michiura, 2007)

This study concluded that the concentration of H_2SO_4 below 3 % is enough to break the impurities from the RH. The SiO_2 can be obtained more up to 99.3 wt.% when the temperature of thermal treatment is higher.

2.5.4 Citric Acid as the Acid Leaching Agent

The previous study shows that citric acid is useful to remove the metallic impurities in RH to increase the SiO_2 in RHA after combustion (Umeda, Kondoh and Michiura, 2007; Umeda and Kondoh, 2008). One study was carried out to determine the optimum concentration for citric acid and the temperature to leach the RH. This research was investigated the effect of citric acid concentration used for leach RH and temperature when leaching the RH.

The experiment was used citric acid with 1 % to 7 % concentrated citric acid to leach the RH with difference temperature started from 25 ^oC to 80 ^oC for 1 hour and the RH was stirred all the time. After the acid leaching process, the RH was washed with 20 ^oC distilled water for 15 minutes and dried at 100 ^oC for 1 hour. Lastly the leached RH sent to received thermal treatment at 800 ^oC for 30 minutes. Table 2.7 shows the RH undergo 1% of concentrated citric leaching for 1 hour at 50 ^oC produced 99.52 % of SiO_2 in TRHA meanwhile Table 2.8 shows that RH treated by 5 % concentrated citric acid at 80 ^oC can produced 99.77 % of SiO_2 in TRHA.

Table 2.7 SiO_2 in Citric Acid Leached RHA at 50 ^oC with Difference Acid Concentration (Umeda, Imai and Kondoh, 2009)

	Citric acid solution concentration, %				
	0	1	3	5	7
SiO_2 obtained, %	97.25	99.52	99.54	99.56	99.47

Table 2.8 SiO_2 in Citric Acid Leached RHA with 5% Concentration at Difference Temperature (Umeda, Imai and Kondoh, 2009)

	Citric acid solution temperature, ^oC				
	25.0	40.0	50.0	60.0	80.0
SiO_2 obtained, %	99.25	99.54	99.56	99.58	99.77

2.5.5 Comparison between Citric Acid and HCl Acid Treatment for RRH

A previous study was carried out to investigate the percentage to replace the citric acid to the strong acid, HCl in leaching the RH to get more SiO_2 in RHA. These studies have three groups of specimens which were RRH, citric acid treated RH (TRH1) which leached by 5 % concentrated citric acid with temperature of 80 ^oC for 15 minutes to 20 minutes and HCl treated RH (TRH2) which leached by 10 % concentrated HCl with temperature of 100 ^oC for 2 hours. After removed the acid and dried at 100 ^oC for 1 hour, the RHs were sent for thermal treatment for 5 hours at 750 ^oC. TRH1 produced more SiO_2 than TRH2 after thermal treatment hence citric acid could be used to replace HCl for leaching due to it is harmless compare to HCl.

Table 2.9 SiO_2 Content in RHs after Thermal Treatment of 750^oC Analysed by XRF (Sapei, Pramudita and Widjaja, 2014)

Wt.%	**SiO_2**
Without any leaching	91.5
Leached by citric acid	99
Leached by HCl	98.8

Besides that, one more study is carried out by three types of treatment for RH to get high percentage of SiO_2 where the first method was conduct the treatment for RRH with hot organic acid before the thermal treatment; second method was same with first method but replaced the hot organic acid by the boiling water while the third method was a post-treatment for RHA which RH had been burned at 250 ^oC and milled into powder.

This experiment separated into 5 processes: A, B, C, D and E where: Process A was the RRH directly received thermal treatment of 900 ^oC with a period of 6 hours. Process B was the RRH leached with 5 wt.% of HCl with 1 hour long period under 150 ^oC. The TRHA was then washed and undergoes 2 hours thermal treatment at 500 ^oC. Process C was replaced the HCl by citric acid and 700 ^oC thermal treatment while the other steps remained unchanged. Process D was consisted three different times of washing (1, 2 and 3 times, each time with 1 hour) to wash the TRHA with 150 ^oC hot water and followed by 2 hours thermal treatment at 700 ^oC. Process E was used the RH which finished the half an hour thermal treatment at 250 ^oC and been milled into powder before receive the treatment almost same as Process B to Process D with 700 ^oC thermal treatment.

The result on Table 2.10 shows that Process B and C have the higher percentage of SiO_2 which 99.5 % and 99.3 %. This experiment showed that acid leaching was more effective than hot water treatment and sent the RH to burned and milled it became powder before received treatment are not helping in increase the percentage of SiO_2 in RHA.

Table 2.10 The Percentage of Silica of the RHA (Souza, Magalhães and Persegil, 2002)

Process	SiO_2, %
A	94.0
B	99.5
C	99.3
D-i (treated with hot water for 1 hour)	97.6
D-ii (treated with hot water for 2 hour)	98.8
D-iii (treated with hot water for 3 hour)	99.1
E-i (treated with boiling HCl for 2 hour and burned for 6 hours)	98.9
E-ii (treated with hot water and burned for 2 hours)	96.6
E-iii (treated with tap water and burned for 6 hours)	96.0
E-iv (directly burned 6 hours after became black colour powder)	94.0

2.5.6 Comparison between Citric Acid and H_2SO_4 Acid Treatment for RRH

This study was to compare the effectiveness of citric acid and sulphuric acid in remove metallic impurities on RH. The reasons to used citric acid to do a comparison to H_2SO_4 are strong acids have economical problem for the equipment use to withstand themselves during acid leaching and the water treatment for strong acid removed from leaching treatment. Moreover it also brings the hazardous to human being and the surround environment (Umeda and Kondoh, 2008).

The test was carried out by soaked each 20 grams of RH in 500 ml of H_2SO_4 and citric acid with 5 % concentration for 15 minutes while the temperature was kept at 50 ^oC. A thermal treatment of 800 ^oC is introduced to TRH after removed the acid completely. The result showed that citric acid leaching more effective in removing metallic impurities on RH hence it can increase the quantity of SiO_2 in RHA.

Table 2.11 SiO_2 Obtained in RHA at 800^{o}C Thermal Treatment for RRH, Citric Acid Treated RH and H_2SO_4 Treated RH (Umeda and Kondoh, 2008)

RHA	**SiO_2, wt.%**
Without any acid leaching	94.58
Leached by citric acid	99.14
Leached by H_2SO_4	90.99

2.6 Concluding Remark

There were many researches regarding to the present of RHA in concrete and mortar to test their performances such as workability, compressive strength, frost resistance and etc. Those studies showed the temperature to prepare the RHA, RHA percentage in mixed concrete and etc are very important to the concrete's performances. It proven that the RHA can improve the performances of the concrete and mortar. Hence the future study can be conduct to find out the most appropriate mix proportional and temperature with the right pre-treatment of RH for the RHA to improve the performances of concrete and mortar.

However there is rare study shows which type of acid is the most effective leaching agent in remove the metallic impurities in RH. This is because different temperature, period of treatment and concentration of acid will give different results. Based on previous studies, citric acid pre-treatment showed slightly more effective than HCl that it can produce 99.77 % of SiO_2 in TRHA. Howeverthe research study on the effect of the room temperature citric acid with difference leaching period towards RH is rarely found.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

To achieve the aim of this study, the materials of TRHA will be determined and few tests are carrying out to analyse the mortar's properties.

3.2 Material

Since the aim of this research is to determine the optimum replacement ratio of TRHA in mortar hence the TRHA is the most important element and also other materials in composition of designed TRHA mortar. Those other materials included ordinary portland cement (OPC), fine aggregate and water.

3.2.1 Portland Cement

The first "Portland" cement was found on 1824 by Joseph Aspdin, and the real or true Portland cement produced 20 years later. The cement will have the chemical reaction when it contact to the water (Nawy, 2008). The cement act as an agent to bind all other materials in the concrete mixture, it will become harden after the react with water. The cement used for this research is Tasek Buaya cement. This cement quality was assured by MS EN 197-1: 2007 and it also certified by EN 196 . This cement must be place on a place that avoids any contact with water to prevent cement become hardened. Figure 3.1 indicates the ordinary portland cement that will be used to carried out the research.

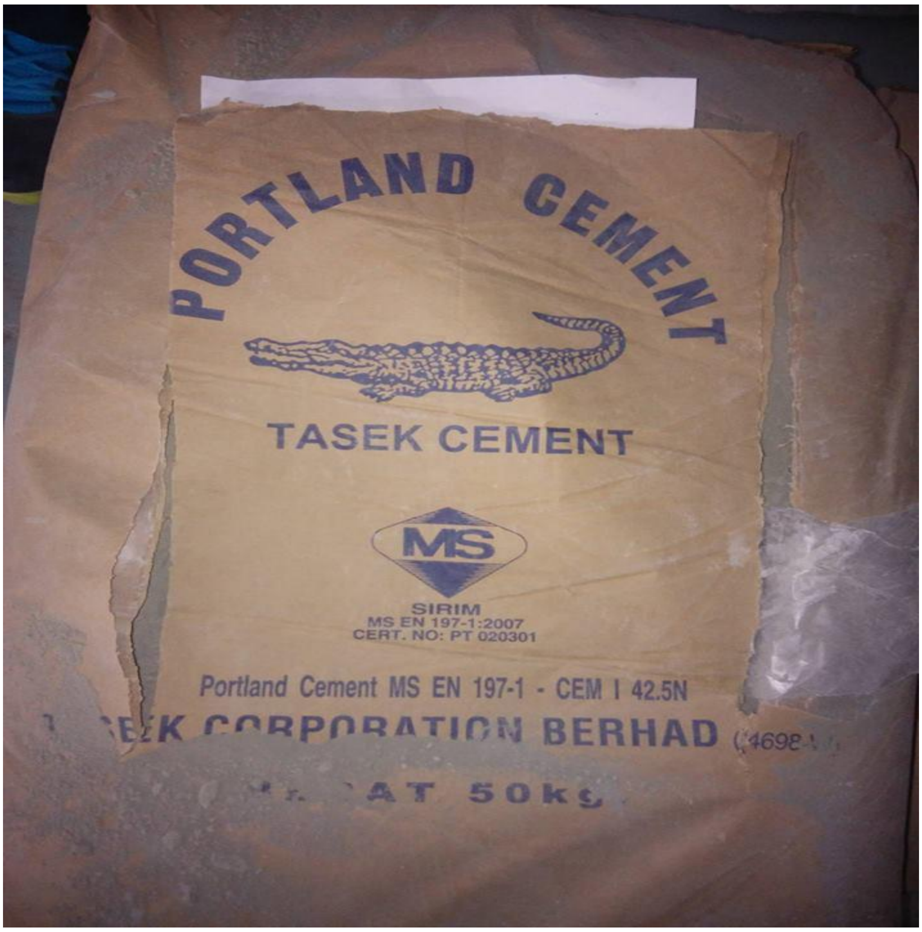

Figure 3.1 50 kg TasekBuayaOrdinary Portland Cement

3.2.2 Treated Rice Husk Ash

Rice husk is a convex shape and yellowish colour thing act as a protection agent to protect rice grain from the insects and natural weather. A paddy consists of rice (72 %), bran (5-8 %) and rice husk (20-22 %) (Yuzer et al., 2013). That means for every 100 kg of paddy there will has approximate 22 kg of rice husk. It is the by-product when the rice produced and it is important for this research. The RH used for this research is collected from the paddy milling plant at Perak. The steps to produce TRHA were showed in Table 3.1. Figure 3.2 showed the RH collected from paddy milling plant.

Figure 3.2 RH from Paddy Milling Plant Perak

The RHs are separated and some of them will send to receive the acid pre-treatment before running the thermal treatment by CARBOLITE Furnace. One of the specimens is doesn’t received any pre-treatment before changed to RHA. Figure 3.3 showed the furnace in Construction Management workshop.

Figure 3.3 CARBOLITE Furnace

Meanwhile another three specimens were been leached by 5 % concentrated citric acid by difference heat during acid leaching process and difference period. After the acid leaching process, they will send to remove the acid by washing with water. The thermal treatment will be conduct to these TRHs with temperature of 800 ^{o}C. The TRHAs are then grinding to become fine particles with 75 μm and it will be weighted before it use to carry out this research due to its weight reduced during the thermal treatment process. RH only left 20 %-22 % of its weight once became RHA (Ramezanianpour, Mahdi Khani and Hmadibeni, 2009).

Table 3.1 Steps to Produce Difference TRHAs

<table>
<tr><td rowspan="2">RRH</td><td colspan="3">5 % citric acid leaching</td><td rowspan="2">Wash three times to remove acid completely</td><td rowspan="2">Dry in furnace at 100 ^{o}C for 1 day</td><td rowspan="2">Thermal treatment at 800 ^{o}C for 3 hours</td></tr>
<tr><td>80 ^{o}C for 1 hour</td><td>Room temperature for 72 hours</td><td>Room temperature for 96 hours</td></tr>
</table>

3.2.3 Fine Aggregate

The fine aggregate was defined as the aggregate which can pass through the 4 mm sieve which stated in BS EN 12620. Typically the fine aggregate collect from land and marine. This fine aggregate is used more when there is a need of better surface area. Typically, the aggregate will be sieved to get the diameter less than 4 mm before mix with other materials to form concrete mixture. The fine aggregate used for this research is mining sand that smaller than 1.18 mm diameter and it is shows in Figure 3.4.

Figure 3.4 Mining Sand

3.2.4 Water

Water is the most important agent to have chemical reaction with cement to form a glue effect to bind all the materials in the concrete mixture such as aggregate, rebar etc. The water-cement ratio will affect the bonding strength between the cement molecules. Typically the quality of the concrete will increase when it's water: cement ratio is decrease. However if the water: cement ratio is too low which is not listed in the standard will give the negative result (Alawode and Idowu, 2011). Almost all the water that is drinkable without any smell or taste is suitable to form the concrete. However there is still a low portion of non-drinkable water that might still be able to use for concrete making. Hence the tap water which met standard of BS EN 1008 from Lembaga Air Perak (LAP) is suitable to make the concrete since it was treated and filtered for human daily uses. Besides that, the tap water also not acidic or alkaline water that might affect the strength of the normal concrete. Figure 3.5 showed the water source for this research.

Figure 3.5 Water Source

3.2.6 Superplasticizer

Superplasticizer used to increase the workability of the fresh concrete without affecting the w/c ratio. The workability of the fresh concrete affected by the mix proportion of concrete, w/c ratio, quantity of fine aggregate in the fresh concrete, the air content in fresh concrete and etc. However excess water, air content and fine aggregate will affect the strength of the concrete and sometimes segregation may occur when there is excess water in the fresh concrete. To prevent these kind of problem happen, the plasticiser or superplasticizer was added during or before mix the concrete.

Figure 3.6 Superplasticizer

Figure 3.6 showed the superplasticizer that will be used in this research. Typically the superplasticizer should add with the weight around 1 % of the binders' weight. This is because too much superplasticizer will cause the segregation in concrete and directly affect the strength of the concrete. According to Salas et al. (2009), it is necessary to add the plasticiser to the TRHA concrete to increase the workability of the TRHA mortar.

3.3 Mix Proportion of TRHAMortar

To find out the most optimum replacement ratio for TRHA in mortar, many ratios has been designed for running tests. The mix proportions of TRHA are show in Table 3.2. The TRHA 1 is the RH that treated by citric acid at 80 ^{o}C for 15 minutes meanwhile the TRHA 2 is RH treated by citric acid at room temperature for 72 hours and TRHA 3 is citric acid treated RH in room temperature for 96 hours. This research carry out by replace 5 %, 10 % and 15 % of cement by volume with TRHA1, TRHA 2 and TRHA 3.The weight of TRHA as partial replacement for cement is calculate by the using the percentage of cement replacement weight divide 3.15 multiple 2.3.

Table 3.2 Mix Proportions Table for TRHA Mortar

Mix code	**Ordinary Portland Cement, kg/m^3**	**Sand, kg/m^3**	**Water Ratio**		**TRHA 1, kg/m^3**	**TRHA 2, kg/m^3**	**TRHA 3, kg/m^3**	**Super plasticizer, %**
			W/C	**Sand weight, %**				
CTRL	733.33	1466.67	0.38	3.00	-	-	-	1.00
H 5	696.66	1466.67	0.38	3.00	26.77	-	-	1.00
H 10	659.99	1466.67	0.38	3.00	53.54	-	-	1.00
H 15	623.33	1466.67	0.38	3.00	80.32	-	-	1.00
TD 5	696.66	1466.67	0.38	3.00	-	26.77	-	1.00
TD 10	659.99	1466.67	0.38	3.00	-	53.54	-	1.00
TD 15	623.33	1466.67	0.38	3.00	-	80.32	-	1.00
FD 5	696.66	1466.67	0.38	3.00	-	-	26.77	1.00
FD 10	659.99	1466.67	0.38	3.00	-	-	53.54	1.00
FD15	623.33	1466.67	0.38	3.00	-	-	80.32	1.00

3.4 Mortar Mixing Process

Before mix the mortar, all the materials needed must be weight to make sure the mortar mixture are same as the mix proportion that been designed before. The mortar are mixing according to ASTM C 348. Due to this research is use different TRHA to replace some portion of cement in the mortar, hence the weighted TRHA is mix with the weighted cement with the ratio in mix proportion table before making the mortar mixture. For H 10,659.99 kg/m^3 of cement is mix with 53.54 kg/m^3 TRHA 1 before add in fine aggregate, water and superplasticizer. The well mixed OPC and TRHA put in a yellow coloured container while the sand put in another container. 3% of sand weight water is then pour into the sand and mix well before add in the OPC and TRHA. The superplasticizer is pour into the water and stir well. Next, insert all the OPC and TRHA into sand's container and mix them. After that, 50 % of water and plasticiser will be added to the dry mixture and mix again. Finally the remaining water is added to the mixture. The mortar mixture is always ensuring to mix evenly during the mixing process.

After complete the mixing process, the mortar mixture is cast in the mould and a rectangle wooden stick will be used to make the concrete mixture spread evenly in the mould by remove some of the air trapped in the mortar mixture during pour into the mould. The mortar mixture in the mould is then put in Construction Management workshop to avoid direct sunlight towards the mortar mixture and any vibration on it. According to BS EN 12390-2 (2009), the specimens should be place at the temperature of 20 ℃ ± 5 ℃ or 25 ℃ ± 5 ℃ for the higher temperature countries. Figure 3.7 illustrates the mortar casted in the mould.

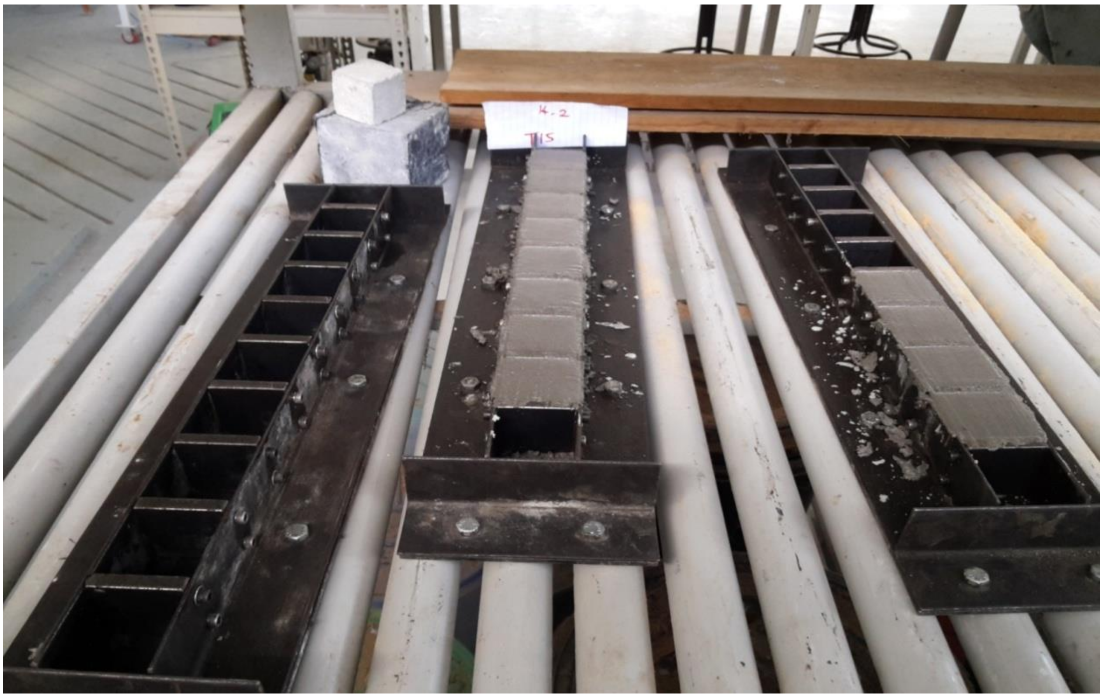

Figure 3.7 Mortar Casted in the Mould

3.5 Cast, Mould and Demould Mortar

The objective of this research is to find out the mechanical properties of the TRHA concrete hence few tests must be conduct for this research. Due to the test for this research will be carry out by laboratory test, hence the mortars are cast with two sizes; 50 mm x 50 mm x 50 mm and cylinder with 45 mm diameter and 40 mm height. The mortar is cast to the dimension of 50 mm x 50 mm x 50 mm for running compressive strength test while the cylinder is use for porosity and permeability test. Figure 3.8 illustrates the mould use to cast mortar cubes.

The mould will be make sure clear from any substances and coat it by used engine oil to let the mortar easier to remove from mould. The mortar mixture is pour into mould by two times. Each time mortar mixture poured into the mould, the rectangle wooden stick will be used by to remove the air that formed void in mortar mixture. The cement trowel used to remove the excess fresh concrete once it completely filled the mould. The mortar will be removed from the mould after 24 hours later and marked by marker to avoid any mistake.

Figure 3.8 Mould Use to Cast 50 mm x 50 mm x 50 mm Mortar

3.6 Curing

Curing actually is a process to provide suitable water content and satisfactory heat to concrete for a certain time (Mamlouk and Zaniewski, 2006). Hence, once the concretes demoulded from the mould, they will have the curing process according to the BS EN 12390-2 (2009) by tap water from LAP with the temperature of 25 ℃ to 29 ^{o}C until the age for testing strength. The ages for testing strength are 3 days, 7 day and 28 days. Figure 3.9 shows the place that the mortar undergo curing process.

Figure 3.9 Mortar Curing Process

3.7 Tests for the Fresh and HardenedMortar

In this research, there are few tests will be carried out for identified the performance, mechanical properties of the mortar. The tests that will be conducted are flow table test, compressive strength test,porosity test and gas permeability test. These tests are necessary for determine the workability of fresh mortar, compressive strength and the void in the hardened mortar. Those tests that will be carried out during the research was showed in Table 3.3.

Table 3.3 Tests' Details for Fresh and Hardened Mortar

Test	Testing age, days	No. of mortar
Flow Table	-	-
Compressive Strength	3	3
	7	3
	28	3
Porosity	28	3
Gas Permeability	28	3

3.7.1 Flow Table Test

When the fresh concrete has high workability, it is easy to handle and flowing into the formwork and around reinforcement. It can avoid the percentage of void forming in the hardened concrete. When the workability too low, the fresh concrete very hard to pour and separate evenly to the formwork. However when it is too high, there will occur segregation and bleeding which will affect the strength of the hardened concrete. In this study, the flow table test is the test that to identify the fresh mortar's workability.

The flow table test is carried out by follow the ASTM C 1437 which the mortar mixture is fill into the mould by two layers. Firstly, the mould must put in the centre of flow table disc before insert the mortar. Every layer filled with mortar mixture must be compact with at least 10 strokes of wooden stick. After the mould full with the mortar mixture, remove the mould by raising it carefully and switch on the start button for the flow table machine. Figure 3.10 shows the flow table machine that will be used to carry out the test.

Figure 3.10 Flow Table Test Equipment

The flow table machine will start to let the disc rises and drops for 25 times. The disc has 4 straight lines as guide line. Measure and record the diameters of the mortar by using the straight lines as guide line. After that remove the fresh mortar and wipe the disc by a wet cloth and the mould is send to wash by clean water to avoid the mortar from getting hardened.

3.7.2 Compressive Strength Test

The hardened concrete is send to the compressive strength test to determine the compressive strength of the concrete. It is important to those concrete that used as the structural member in a building such as footing, column, beam and etc. The compressive strength test is carrying out according the BS EN 12390-4:2000. Due to it is a destructive test for strength test, hence it only test on the concrete or mortar cube after conducted rebound hammer test. In this research, the mortar cube with size of 50 mm x 50 mm x 50 mm is send to the compressive machine and the machine will exert the force towards the mortar cube until the mortar cube fail to withstand the force. The result is record for the investigation of mortar strength. The Figure

3.11 demonstrates the compressive strength test machine which will be used for testing the mortar's strength.

Figure 3.11 Compressive Strength Test Machine

3.7.3 Porosity Test

Porosity is one of the factors that affect the concrete's durability. Porosity is measure the void inside the concrete or mortar. The amount of porosity increased, the shorter the durability for the concrete. The porosity test that will be carried out in this research follow RILEM CP 11.3, it required the sample size with 45mm diameter and 40mm height. These cylinders will put in the oven for 24 hours. After 24 hours of dehydration process, the cylinders are then send to weighing and marking one by one. When all the cylinders done marking and weighing, they will be moved into a glass desiccators and the opening will seal by silicone gel. Next step is switch on the generator for 15 minutes to let the glass desiccators in a vacuum state and 3 hours later switch on the generator again for 15 minutes and left it for 24 hours. Take out the samples, dry them by cloth and weight them in water and air one by one.

3.7.4 PermeabilityTest

According to Neville and Brooks (2010), the permeability test is used for checking the durability of the concrete which will affect the lifetime of the building by determine the rate of water or gas flow through the concrete. The permeability test will be carrying out according to Lynsdale Cabrera 1984. The mortar cylinder with 45 mm diameter and 40 mm height will be used to conduct the test at 28 days of testing age. This test is useful for mortar in the structures such as sewerage tanks and gas purifiers to avoid the poisonous gas escaped from those structure.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter discussed the results getting from the laboratory tests and analyse those results. The performances of mortars which replaced 5%, 10% and 15% OPC by different methods of treated rice husk ash were evaluated and compare their performances with the conventional mortar. Those performances are in term of mortar's workability, compressive strength, void inside the mortar and it's permeability ability against gas.

4.2 Rice Husk Ash Production

The RH was treated in heated citric acid for 1 hour, leached with citric acid in room temperature for 72 hours and leached with citric acid in room temperature for 96 hours. The acid pre-treatment can remove the metallic impurities in the raw rice husk (Kurama and Kurama, 2003; Ugheoke and Mamat, 2012; Salas et al., 2009). The weight of the RRH after pre-treatment was reduced around 15.8% - 15.97%. It is most probably that the citric acid removed the impurities in the RH.

Table 4.1 Weight of TRHAs

Code	**TRHA 1 80 ℃, 1 hour (Umeda, Imai and Kondoh, 2009)**	**TRHA 2 26 ℃, 72 hours**	**TRHA 3 26 ℃, 96 hours**
Weight before treatment, g	1000		
Weight after treatment, g	840.3	842.0	841.2
Wash 3 times remove acid and dry in oven for 24 hours			
Incineration with 800 ℃ for 3 hours			
Weight after incineration/ 1000g leached RHA	153.0	153.0	154.00

From this research, the most effective pre-treatment is leached by heated citric acid in term of weight which showed in Table 4.1 where the weight of TRHA 1 loss greater than TRHA 2 and TRHA 3. It produced up to 99% of SiO_2 (Umeda, Imai and Kondoh, 2009). Besides that, the result also shown that the longer the period of leaching, the higher the weight loss. It is most probably that the period of leaching affected the amount of impurities being removed.

4.3 Mortar Performances

To achieve this research's objectives, where determine the durability and mechanical properties of treated rice husk ash mortar with different types of treated rice husk ashes and it replacement volume to cement in mortar, few tests were carried out. Those tests included flow table test, compressive strength test, porosity test and gas permeability test.Figure 4.1 showed the sequence of those tests which were carried out to investigate the performances of the mortars.

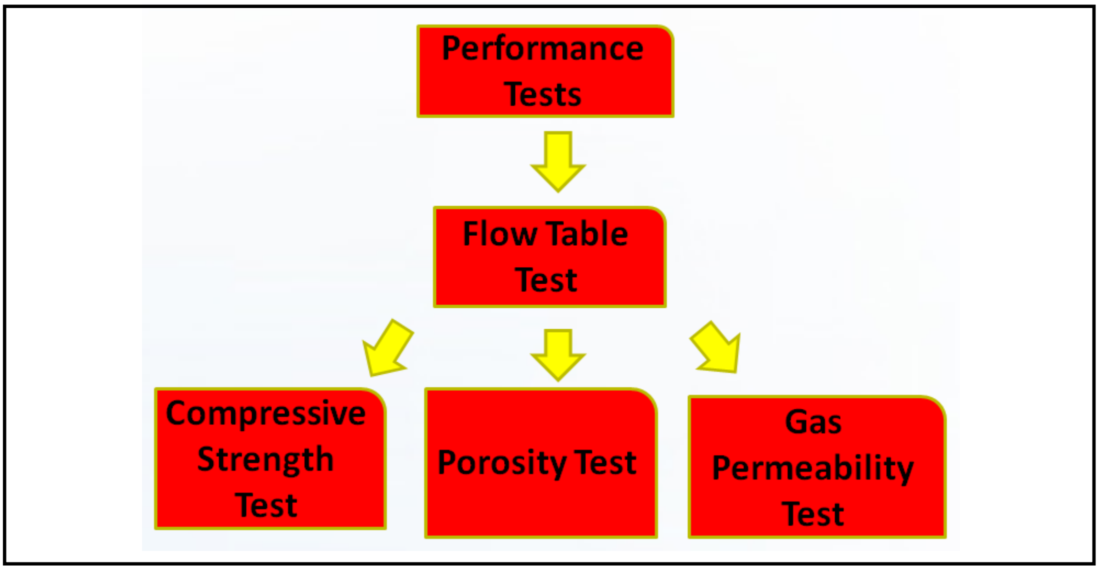

Figure 4.1 Mortar Performance Tests' Flowchart

4.3.1 Flow Table Test

The workability of the concrete is the ability of fresh concrete fill up the formwork that contained of rebar without exert any vibration on it. In this research, the workability of fresh mortar was determined by using flow table test. This flow table test is carried out according ASTM C 1437. The fresh properties of specimens were tested and the results are showed in Table 4.2. The results were obtained by using the equation as shown in Equation 4.1.

$$\text{Flow} = \frac{D_1 - d}{d} x\ 100 \tag{4.1}$$

Where :D_1 = diameter of flow

d = original diameter (100.0 mm)

Table 4.2 Flow Table Test Result

Mix Code	Flow diameter, mm				Average flow diameter, D_1(mm)	Flow
	One	Two	Three	Four		
CTRL	200.0	203.0	192.0	195.0	197.5	97.5
H 5	188.0	190.0	185.0	185.0	187.0	87.0
H 10	145.0	135.0	135.0	130.0	136.3	36.3
H 15	130.0	130.0	125.0	125.0	127.5	27.5
TD 5	175.0	177.0	180.0	185.0	179.3	79.3
TD 10	135.0	138.0	138.0	140.0	137.8	37.8
TD 15	130.0	130.0	134.0	130.0	131.0	31.0
FD 5	160.0	160.0	160.0	160.0	160.0	60.0
FD 10	135.0	135.0	135.0	135.0	135.0	35.0
FD 15	113.0	113.0	110.0	118.0	113.5	13.5

Figure 4.2 showed the flow of the conventional mortar and TRHA mortar with different portions of cement replacement. The graph showed the conventional mortar has the highest flow 97.5, among all other specimens in this research. While the fresh mortar with 15 % cement replacement by four days treated rice husk ash showed the lowest flow of 13.5. From the graph, it can be seen that the percentage of TRHA replace cement increases, the fresh properties of the mortar will decreases. The fresh properties of TRHA mortar lower than control mortar because the TRHA has the properties of water absorption due to the RHA has high specific surface area (Ganesan, K., K. Rajagopal,2008). It will absorb the water during the mortar mixing process and affect the water cement ratio. Hence the result getting from this experiment showed valid and reasonable.Besides that, as the workability decreased,

the porosity also decreased. This is because the chemical reaction of TRHA in forming C-S-H gel which required water and it water absorption properties (Kartini, Mahmud and Hamidah, 2010) hence the porosity and the workability decreased as the amount of TRHA increased.

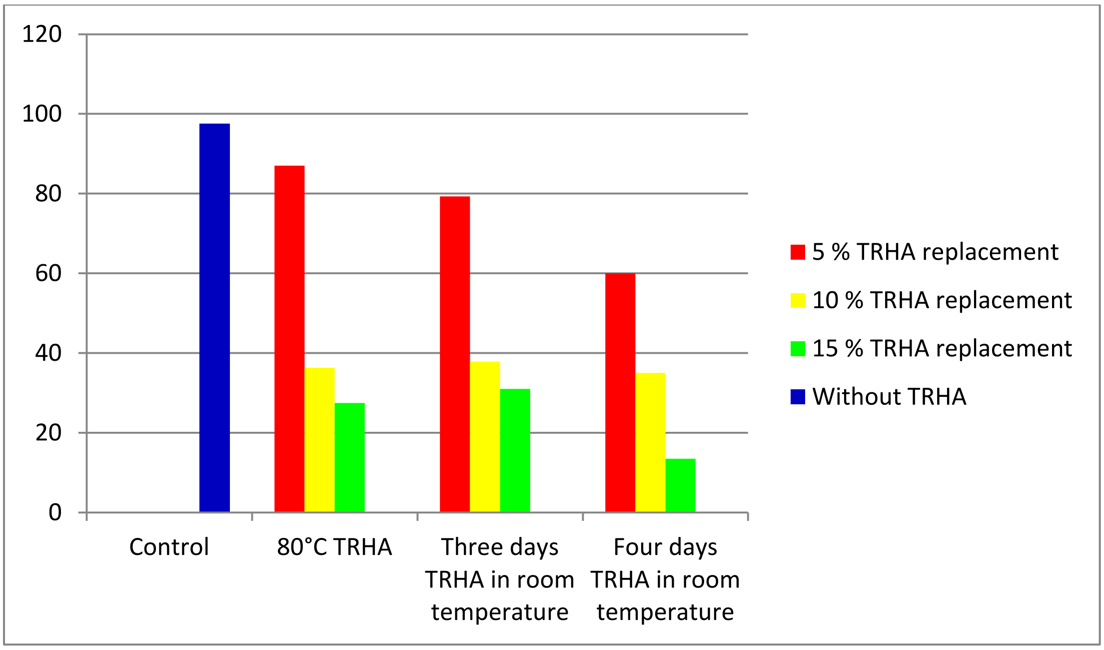

Figure 4.2 Flow Table Test Result Bar Chart

4.3.2 Compressive Strength Test

Compressive strength is the test that falls under mortar mechanical properties. The mortar specimens that required for this test are with size of 50 mm x 50 mm x 50 mm cube. These cubes were cured at the curing tank for 3 days, 7 days and 28 days. When the cubes reached their testing age, they were sent to receive the compressive strength test. Table 4.3 showed the 3 days, 7 days and 28 days compressive strength.

According to Khassaf, Jasim and Mahdi (2014), the strength of cement binders that contained of RHA will started gain higher at the later period. From Figure 4.3, it can be seen that most of the TRHAs mortars 28 days compressive strength are lower than control mortar. It is because when the amount of TRHA in mortar increase, the amount of cement will decrease and it will cause the capillary pore volume increase. The capillary pore will cause the calcium hydroxide accumulate on the interface. When this situation happened, the structures are less compact compared to conventional mortar hence the strength reduced (Khassaf, Jasim and Mahdi, 2014). According toNguyen (2011), when the amount of RHA in the mortar is higher, it will absorb the water and affect the cement hydration process at the beginning. As the time go, the mortar's strength will rise because the water absorbed by the RHA will be released to the cement and let it continue the hydration process. H 5 has the highest compressive strength at 28 days because it has higher water remaining in binder for cement hydration taking place compared with other. The result also showed the H 10 has the lowest compressive strength due to the TRHA volume increase and it directly affected the cement to gain strength through hydration process.

Table 4.3 Mortar Average Compressive Strength

Mix code	Average compressive strength, N/mm^2		
	3 days	7 days	28 days
CTRL	31.88	40.40	56.84
H 5	34.10	40.71	63.69
H 10	25.83	26.03	40.58
H 15	24.55	31.29	41.12
TD 5	28.09	36.14	47.98
TD 10	26.76	33.58	47.70
TD 15	27.55	39.65	45.09
FD 5	25.18	31.79	44.02
FD 10	26.54	33.31	40.77
FD 15	26.07	29.73	48.02

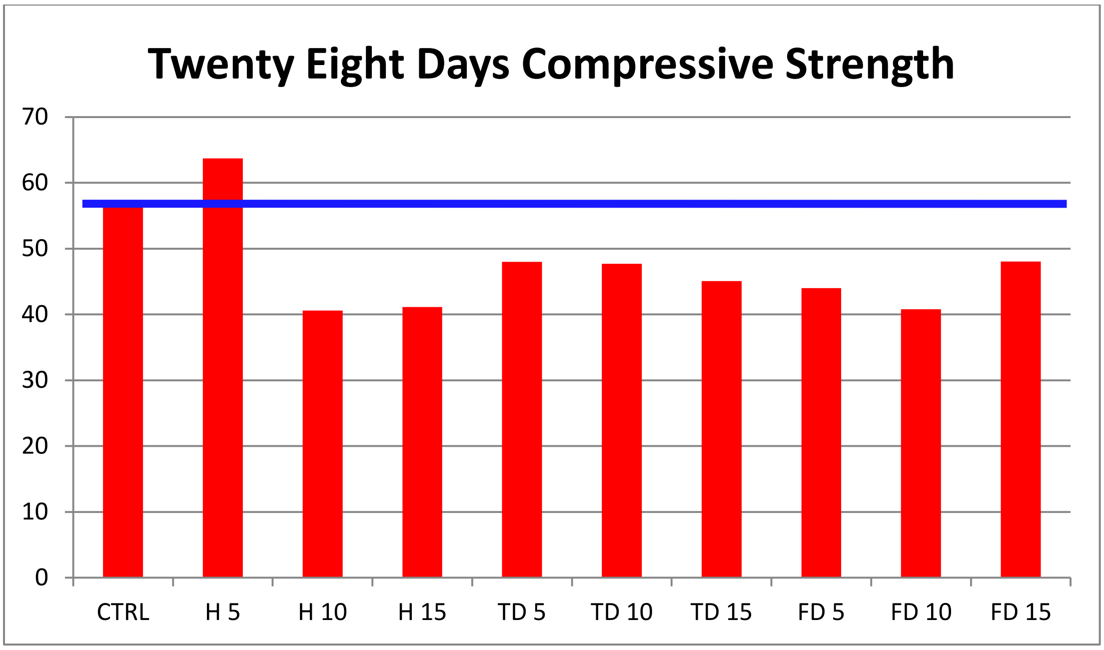

Figure 4.3 Twenty Eight Days Compressive Strength

C-S-H gel can bind the cement particles into a continuous form to reduce capillaries pores and increase strength (Kassim et al., 2004). In Figure 4.4, H 5 which used TRHA 2 which was treated with citric acid for 3 days showed a higher average compressive strength than TRHA 3 and TRHA 1 mortars with different replacement ratio. In general, the mortars with 5 % TRHA replacement have a higher compressive strength than 10 % TRHA replacement mortars. It is probably that the water in 5 % TRHA replacement mortars have better hydration than 10 % TRHA replacement mortars. It produced higher amount of C-H that react to silica from TRHA hence better strength gained. While the 10 % TRHA replacement mortar has lesser water for cement hydration hence the C-H became lesser than silica hence the strength gained was weaker than 5 % TRHA replacement mortar (Habeeb and Mahmud, 2010).

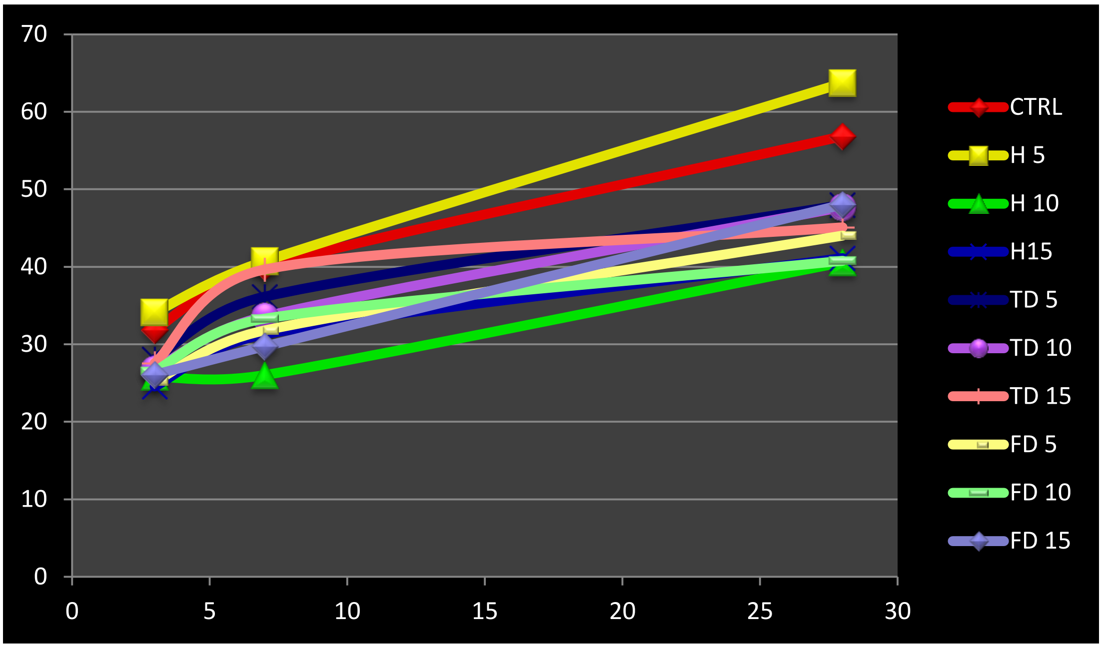

Figure 4.4 Mortar Average Compressive Strength

4.3.3 Gas Permeability Test

In this research, the durability of the TRHA mortars were determined by gas permeability test. The permeability is the ease of fluid or gasses pass through the specimen. Hence the higher permeability, the easier the fluids or gasses pass through it.The permeability is determined by the value of intrinsic permeability which will not affected by the size of the specimen. This test is using the time for the water in the flowmeter rising for every 10 cm to calculate the permeability level of the specimen. The faster the water rise means the permeability higher. The permeability is calculated by using the Equation 4.2.

$$K = 2P_2(1.76 \text{ x } 10^{-16} VL \,/\, \text{A} \,(P_1^{\,2} - P_2^{\,2})) \qquad (4.2)$$

Where K = intrinsic permeability, m^2

P_1= Absolute applied pressure bars (atmosphere pressure) usually 2 bars

P_2= Pressure at which the flow rate is measured (atmosphere pressure) usually 1 bar

A = Cross sectional areas of specimen, m^2

L = Length of specimen, m

V = Flow rate cm^3/s

The flow rate, $V = (D^2/4)\pi H/ T$

Where D = Flowmeter diameter, cm

H = Length read on flowmeter, cm

T = Average time, s

All the required data were showed in Table 4.4 which will be used to calculate the intrinsic permeability of the specimens. There were three specimens for each group of the TRHA mortar and control mortar. Meanwhile, the average intrinsic permeability was showed in Table 4.5.

Table 4.4 Intrinsic Permeability Result

Mix Code	Average time taken, *T* (s)			Flow rate, *V* (cm^3/s)			Intrinsic permeability, *K*(m^2)		
	1	2	3	1	2	3	1	2	3
CTRL	8	8	12.8	0.09	0.09	0.06	2.66E-16	2.66E-16	1.77E-16
H 5	23	16	30	0.03	0.04	0.02	8.86E-17	1.18E-16	5.90E-17
H 10	31	16.2	37	0.02	0.04	0.02	5.90E-17	1.18E-16	5.90E-17
H 15	30	16.5	26.6	0.02	0.04	0.03	5.90E-17	1.18E-16	8.86E-17
TD 5	42	30.49	24.59	0.02	0.02	0.03	5.90E-17	5.90E-17	8.86E-17
TD 10	61.82	23.52	54.8	0.01	0.03	0.01	2.95E-17	8.86E-17	2.95E-17
TD 15	57	51.78	32.2	0.01	0.01	0.02	2.95E-17	2.95E-17	5.90E-17
FD 5	26.2	46.21	17.35	0.03	0.02	0.04	8.86E-17	5.90E-17	1.18E-16
FD 10	51.34	44.63	49.78	0.01	0.02	0.01	2.95E-17	5.90E-17	2.95E-17
FD 15	23.83	54.85	56.14	0.03	0.01	0.01	8.86E-17	2.95E-17	2.95E-17

Table 4.5 Average Intrinsic Permeability Result

Mix Code	Intrinsic permeability, K(m^2)			Average Intrinsic permeability, K(m^2)
	1	2	3	
CTRL	2.66E-16	2.66E-16	1.77E-16	2.36E-16
H 5	8.86E-17	1.18E-16	5.90E-17	8.85E-17
H 10	5.90E-17	1.18E-16	5.90E-17	7.87E-17
H 15	5.90E-17	1.18E-16	8.86E-17	8.85E-17
TD 5	5.90E-17	5.90E-17	8.86E-17	6.89E-17
TD 10	2.95E-17	8.86E-17	2.95E-17	4.92E-17
TD 15	2.95E-17	2.95E-17	5.90E-17	3.93E-17
FD 5	8.86E-17	5.90E-17	1.18E-16	8.85E-17
FD 10	2.95E-17	5.90E-17	2.95E-17	3.93E-17
FD 15	8.86E-17	2.95E-17	2.95E-17	4.92E-17

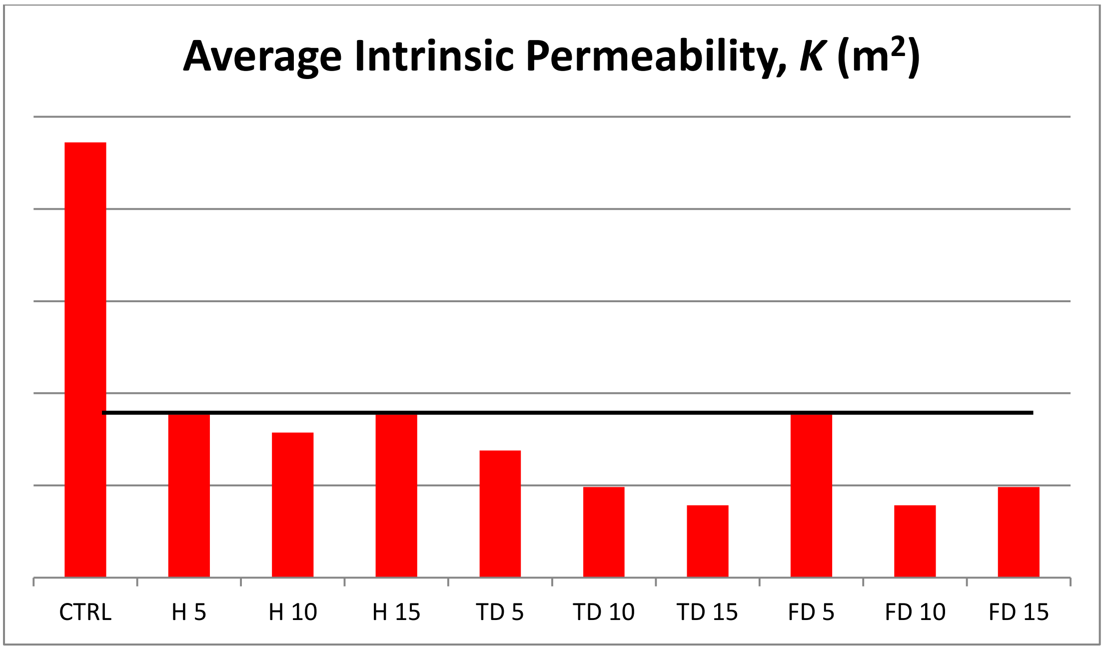

Figure 4.5 Average Intrinsic Permeability for TRHA Mortars and Conventional Mortar

Figure 4.5 showed the average intrinsic permeability for TRHA mortars and conventional mortar. It can be seen that conventional mortar has the highest permeability compared to other TRHA mortars, the mortar which contained TRHA as partial replacement of cement is 62.5% better than conventional mortar. It is because the TRHA will fill up the empty void in the mortar and effectively reduce the permeability (Kartini, Mahmud and Hamidah, 2010). Another research concluded that the RHA has tiny size of particles which believed can increase the particles packing density which helps to decrease the pores inside the specimen hence the permeability can be reduced (Saraswathy and Song, 2007).Hence when the amount of TRHA present in mortar increased, the permeability will be reduced. For the mortar H 15 and FD 15 which replaced with 15 % of TRHA have higher permeability than 10 % TRHA replacement are probably affected by the water for hydration. Mortar with 15 % TRHA replacement will remain less water for cement to run its hydration (Habeeb and Mahmud, 2010). The hydration process will produce C-H bond which will be used to react with S to form C-S-H gel to reduce the pore volume and permeability in the mortar (Dakroury et al., 2008; Kartini, Mahmud and Hamidah, 2010).

4.3.4 Porosity Test

Porosity test is to test the pore volume of the mortar in this research. It used to determine the durability of the mortar. According to Neville and Brooks (2010), the amount of pores in the specimen directly affect the strength of specimen, hence the higher the amount of pores in specimen, the weaker the strength. The samples used for this test were with 45 mm diameter and 40 mm height. Those specimens were at the 28 days testing age. The result obtained is calculated by the Equation 4.3.

$$P\ (\%) = \frac{W_1 - W_2}{W_1 - W_3}\ x\ 100 \tag{4.3}$$

Where :P= Porosity, %

W_1 = Weight of specimen in air after passed through the porosity test

W_2 = Weight of dry specimen

W_3 = Weight of specimen in water after passed through the porosity test

Table 4.6 showed the weight of the mortar before immersed into water and weight after immersed in water. The mortar's weight was increased after immersed in water. The result was then analysed by Equation 4.3 to find out the porosity of the TRHA mortar as shown in Table 4.7.

Table 4.6 Specimens Weight for Porosity Test

Mix Code	Weight in air, W_1(kg)			Weight in water, W_3(kg)			Weight for dry specimen, W_2(kg)		
	1	2	3	1	2	3	1	2	3
CTRL	0.145	0.148	0.153	0.079	0.084	0.087	0.131	0.135	0.139
H 5	0.151	0.149	0.147	0.084	0.083	0.082	0.138	0.137	0.134
H 10	0.145	0.148	0.152	0.080	0.083	0.086	0.134	0.134	0.140
H 15	0.144	0.144	0.141	0.078	0.077	0.077	0.132	0.132	0.130
TD 5	0.150	0.144	0.150	0.086	0.081	0.085	0.138	0.132	0.138
TD 10	0.148	0.149	0.147	0.083	0.084	0.083	0.137	0.139	0.136
TD 15	0.148	0.150	0.150	0.083	0.082	0.084	0.136	0.137	0.138
FD 5	0.149	0.146	0.145	0.085	0.085	0.082	0.136	0.134	0.133
FD 10	0.150	0.151	0.150	0.085	0.084	0.085	0.137	0.137	0.137
FD 15	0.145	0.148	0.148	0.080	0.081	0.082	0.132	0.134	0.135

Table 4.7 Porosity Result

Mix Code	Porosity, *P*(%)			Average porosity, *P*(%)
	1	2	3	
CTRL	21.21	20.31	21.21	20.91
H 5	19.40	18.18	20.00	19.19
H 10	16.92	21.54	18.18	18.88
H 15	18.18	17.91	17.19	17.76
TD 5	18.75	19.05	18.46	18.75
TD 10	16.92	15.38	17.19	16.50
TD 15	18.46	19.12	18.18	18.59
FD 5	20.31	19.67	19.05	19.68
FD 10	20.00	20.90	20.00	20.30
FD 15	20.00	20.90	19.70	20.20

From Figure 4.6, it can be seen that the mortar which contained TRHA have lower pore volume compare to conventional mortar which without THRA. According to Saraswathy and Song (2007), the volume of pores in the cement product can be reduced by increase the amount of RHA. RHA has smaller particles that can fill up the pore hence the pore volume been decreased. The mortars which replaced by TRHA 1 and TRHA 2 showed a better result than mortars which replaced by TRHA 3. It is probably the silica content in TRHA 1 and TRHA 2 are higher than TRHA 3. When the silica content in mortar decreased, the C-S-H gel which can reduce the pore size decreased because the calcium hydroxide bond cannot react with enough silica (Dakroury et al., 2008 ; Sugita et al., 1997).

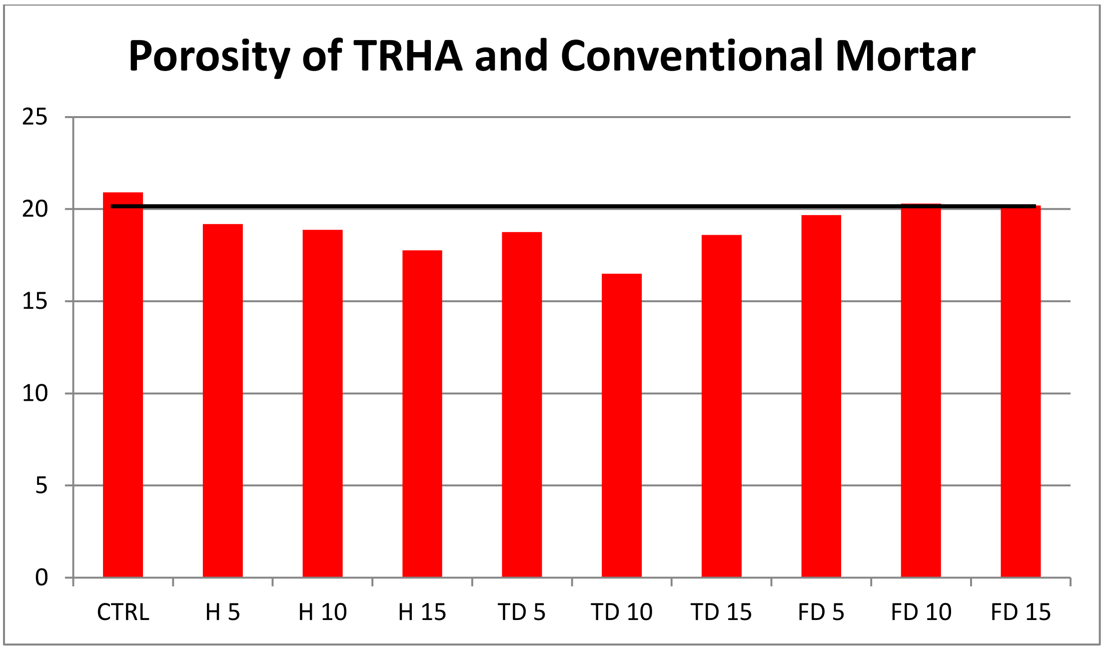

Figure 4.6: Porosity of TRHA and Conventional Mortar

Another researchers Kartini, Mahmud and Hamidah (2010) found that the pore in the RHA replacement concrete and mortar is higher when the amount of RHA replacement is lower. The reason is because the RHA water absorption characteristic. For 5 % RHA replacement mortar, the water amount remaining for hydration is higher than 10 % RHA replacement mortar. When the water is being evaporated, it will leave voids and contribute to the water absorption. Hence the higher the water remaining, the higher the amount of void in mortar after water evaporated.

4.4 Concluding Remark

From the results getting from the experiments, the workability showed that the higher the amount of TRHA in mortar, the lower the workability. In average, the mortars with TRHA 2 replacement are almost similar to TRHA 1 mortar while the TRHA 3 mortar gave a lowest workability among other groups mortar.

The compressive strength of mortars are affected by the amount of TRHA replacement and the type of TRHA. In overall, the TRHA mortars showed a lower compressive strength then control mortar but according to Khassaf, Jasim and Mahdi (2014), the higher the amount of THRA in mortar, the later the strength gained. Among three types of THRA mortars, the mortars groups which replaced by THRA 2 have the higher compressive strength in every testing ages.

This research showed the permeability and porosity of TRHA mortars are much lower than control mortar. This is because the TRHA filled up the voids inside the mortar (Kartini, Mahmud and Hamidah, 2010) and the C-S-H gel took place (Dakroury et al., 2008). TRHA 2 which treated with 3 days citric acid at normal room temperature exerted in the mortar given a better low permeability and porosity result.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

A research on the optimize replacement ratio of different types of treated rice husk ashes in mortar was conducted to investigate the mechanical properties and durability compared to conventional mortar. The results getting from this study were analysed and several conclusions can be make:

1) TRHA mortars have lower fresh properties than conventional mortar due to the TRHA has high specific surface area that will absorb the water during mixing process. When the amount of TRHA in mortar increases, the workability will decreases. Hence, an adjustment of w/c ratio and the amount of superplasticiser shall be made to improve the workability for high amount TRHA replacement mortar.

2) All the TRHA mortars showed a slow growth of compressive strength than conventional mortar due to the TRHA affected the water content in the mortar hence the cement has no enough water to process the hydration process to gain strength.

3) The permeability of the mortars which contained of TRHA are much lower than conventional mortar due to the TRHA were filled up the capillaries pores and formed C-S-H gel to reduce the void and permeability of mortar.

4) All the TRHA provided a better resistance against the pores in the mortar.

5) The TRHA mortar showed a good characteristic of gas penetration and low pores hence it is suitable to apply at the structure that contained of gas such as sewerage structure.

5.2 Recommendations

This study aim is mainly focused on the methods of preparing TRHA and the optimize replacement ratio in mortar. To achieve the aim of this research, the TRHAs mortars were undergo certain tests to determine the fresh properties and mechanical properties such as compressive strength, permeability and porosity test. There are some recommendations for future researchers to obtain a more accurate and useful information regarding to TRHA mortar:

1) A comprehensive study on the mechanical properties of TRHA mortar as the pozzolanic reaction took place in it.

2) Other durability test for TRHA mortar should be investigated as TRHA mortar has better porosity and permeability characteristics.

3) A study on acid treatment of rice husk with different molarity of citric acid as well as the period of treatment without extra heat.

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