Effects of Elevated Temperatures on Microwave Incinerated Rice Husk Ash blended Concrete

by

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CERTIFICATION OF APPROVAL

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Civil Engineering Programme
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BACHELOR OF ENGINEERING (Hons)
(CIVIL ENGINEERING)

Approved by,

____
(AP. Ir. Dr. Hj. Muhd Fadhil Nuruddin)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2008

CERTIFICATION OF ORIGINALITY

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

(CHANDRAKUMAR NADARAJAN)

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ABSTRACT

Concrete subjected to rapid heating at high temperatures causes spalling of concrete. Spalling occurs when the tensile stress due to the pore pressure exceeds the tensile strength of concrete. Agricultural waste such as rice husk ash (RHA) is disposed through open burning. This contributes to elevating air pollution problems and causes respiratory diseases. This research studies the possibility of using RHA as a partial replacement for cement in concrete subjected to elevated temperatures, hence simultaneously providing a solution for spalling and air pollution problems. The objective of this study is to evaluate MIRHA as a supplementary cementitious material with reference to strength of hardened concretes and identify the optimal level of replacement of cement to improve concretes resistance to elevated temperatures. Tests were conducted on normal and RHA blended concrete. This included tests on compressive strength, fire resistance, Ultrasonic-Pulse Velocity and Scanning Electro-optic Microscope. Results show that MIRHA at 800°C with 5% partial replacement of OPC is the optimum mix proportion that maximizes concretes resistance to elevated temperatures.

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ABBREVIATIONS

RHA	-	Rice Husk Ash
MIRHA	-	Microwave Incinerated Rice Husk Ash
OPC	-	Ordinary Portland Cement
SEM	-	Scanning Electron Microscopy
CO_2	-	Carbon dioxide
C-S-H	-	Calcium Silicate Hydrate
ITZ	-	Interfacial Transition Zone
SP	-	Superplasticizer

CHAPTER 1

INTRODUCTION

1.1 Background

Concrete provides good fire resistance, but when exposed to elevated temperatures for a long duration, spalling is prone to take place. Therefore, improvement of concrete durability and strength to increase its fire resistance are of great concern. The basic aspects of the production of high performance concrete are the use of lower water-cement ratio and supplementary cementing materials. Due to this growing environmental concern and the need to conserve energy and resources, considerable efforts have been made to utilize local natural waste and by-product materials in making concrete, e.g. Rice Husk Ash (RHA). Under controlled burning and with sufficient grinding, the rice husk ash can be used as a supplementary cementing material.

1.2 Problem Statement

Normal concrete subjected to rapid heating at high temperatures causes spalling. Spalling occurs when the tensile stress due to pore pressure exceeds the tensile strength of the concrete. The phenomenon is generally assumed to occur at high temperatures, yet it has also been observed in the early stages of a fire and at temperatures as low as 200°C.

The world's ecosystem is faced with the growing problem of global warming which is associated with the emission of CO_2 into the atmosphere. It is a well-known fact that for every tonne of Portland cement produced, approximately one tonne of

CO_2 is released. To reduce the CO_2 emissions related to cement production, the use of Portland cement needs to be reduced without compromising the performance of the concrete structures.

The introduction of a new breed of concrete is essential to increase the performance of concrete structures subjected to high temperature conditions. The current approach when introducing a new breed of concrete is to incorporate waste materials which are pozzolanic in nature.

One such agricultural waste that is gaining popularity is the rice husk ash (RHA). Rice husk ash is produced under controlled burning and with sufficient grinding. The burning procedure, method and maximum temperature affects the quality of RHA produced.

Therefore a standard burning procedure needs to be identified. Subsequently, the optimum incorporation of RHA into concrete needs to be established. Effects of elevated temperatures on concrete containing RHA can be tested by exposing the various samples at different temperatures.

1.3 Objective

The main objectives of the project are:

i. To identify a standard burning procedure and optimum temperature for rice husk.
ii. To determine the optimum percentage of rice husk ash as a partial replacement for cement in concrete subjected to fire.
iii. To establish the effects of fire on the structure, integrity and microstructure of MIRHA blended concrete.

1.4 Scope of Study

i. Various burning procedures of rice husk and MIRHA burnt at different temperatures.
ii. Concrete mix proportions for 0.45 water-cement ratio with 0, 5, 10, 15 and 20 percent replacement of rice husk ash.
iii. Testing the fire resistance of 13 concrete samples with various mix proportions under 800°C.
iv. Compressive strength of 13 concrete samples with various mix proportions at ages 3, 7 and 28 days.
v. Compressive strength of 13 burnt concrete samples.

CHAPTER 2

LITERATURE REVIEW

2.1 Concrete in Fire

Concrete provides good fire resistance. It does not burn like other materials in a building and it does not emit any toxic fumes, smoke or drip molten particles when exposed to fire.

This excellent fire performance is due to the concrete's constituent materials (i.e. cement and aggregates) which, when chemically combined, form a material that is essentially inert and has poor thermal conductivity. It is this slow rate of heat transfer that enables concrete to act as an effective fire shield not only between adjacent spaces but also to protect itself from fire damage.

2.1.1 Factors influencing fire resistance

Fire resistance of concrete is influenced by aggregate type, moisture content, density, permeability and thickness. Limestone, dolomite and limerock are called "carbonate" aggregates because they consist of calcium or magnesium carbonate or combinations of the two. During exposure to fire, these aggregates calcine - carbon dioxide is driven off and calcium (or magnesium) oxide remains. Since calcining requires heat, the reaction absorbs some of the fire's heat. The reaction begins at the fire-exposed surface and slowly progresses toward the opposite face. The result is that carbonate aggregates behave somewhat better than other normal-weight aggregates in a fire.

Moisture content has a complex influence on concrete's behavior in fire. Concrete that has not been allowed to dry may spall, particularly if the concrete is highly impermeable, such as concretes made with silica fume or latex, or if it has an extremely low water-cement ratio. Concretes that are more permeable will generally perform satisfactorily, particularly if they are partially dry.

Pertinent visual observations shows discoloration, damage and detachment of protective and structural materials, cracking, spalling, buckling, creation of gaps-openings, flame and gas penetration and other unusual behavior. Discolouration of the concrete suggested that the material around the reinforcement had reached about 700°C[1].

2.1.2 Correlation between Furnace Burning and Actual fire

The characteristics of fire resistance test furnaces show that the efficiency of a furnace, as measured in terms of the heat load it imposes on a test specimen, depends entirely on the size of the furnace and the nature of the furnace gas. A defective furnace with a material of very low thermal inertia, though helpful, is unlikely to bring its performance up to the required level. The theorem of uniformity heat load is recognized as a succinct descriptor of fires with respect to their destructive potential. Intensity of actual fires greatly depend on combustile material present in a specific fire. Actual fires also lack consistency as other variables, such as changes in ventilation influence the severity of a specific fire. Though furnace burning does not have constant supply of oxygen as opposed to actual fires, this does not affect the intensity of the fire in the furnace as the furnace itself produces the fire and is not influenced by its surroundings. As such, it forms the basis for correlating real-world fires with standard test fires.

2.1.3 Reaction of concrete to fire (temperature rise)

- **Chemical changes**

When subjected to heat, concrete responds not just in instantaneous physical changes, such as expansion, but by undergoing various chemical changes. This response is especially complex due to the non-uniformity of the material. Concrete contains both cement and aggregate elements, and these may react to heating in a variety of ways[1].

First of all, there are a number of physical and chemical changes which occur in the cement subjected to heat. Some of these are reversible upon cooling, but others are nonreversible and may significantly weaken the concrete structure after a fire. Most porous concretes contain a certain amount of liquid water in them. This will obviously vaporize if the temperature significantly exceeds the moisture plateau range of 100-140°C or so, normally causing a build-up of pressure within the concrete. If the temperature reaches about 400°C, the calcium hydroxide in the cement will begin to dehydrate, generating further water vapour and also bringing about a significant reduction in the physical strength of the material. Other changes may occur in the aggregate at higher temperatures, for example quartz-based aggregates increase in volume, due to a mineral transformation, at about 575°C and limestone aggregates will decompose at about 800°C. In isolation, the thermal response of the aggregate itself is more straightforward but the overall response of the concrete due to changes in the aggregate may be much greater. For example, differential expansion between the aggregate and the cement matrix may cause cracking and spalling[1].

These physical and chemical changes in concrete will have the effect of reducing the compressive strength of the material. Generally, concrete will maintain its compressive strength until a critical temperature is reached, above which point it will rapidly drop off. This generally occurs at around 600°C. This is only a little higher than critical temperatures for steel, but because of the much lower conductivity of concrete the heat tends not to penetrate very far into the depth of the material, meaning that the structure as a whole normally retains much of its strength.

- **Physical changes**

i. Spalling

One of the most poorly understood processes in the reaction of concrete to high temperatures or fire is that of 'explosive spalling'[3]. This is the process whereby chunks of concrete break off and are ejected from the surface of the concrete slab, often at fairly high velocities. The phenomenon is generally assumed to occur at high temperatures, yet it has also been observed in the early stages of a fire and at temperatures as low as 200°C. If severe, spalling can have a deleterious effect on the strength of reinforced concrete structures, due to enhanced heating of the steel reinforcement. Spalling may significantly reduce or even eliminate the layer of concrete cover on the reinforcement bars, thereby exposing the reinforcement to high temperatures, leading to a reduction of strength of the steel and hence a deterioration of the mechanical properties of the structure as a whole[1].

The mechanism leading to spalling is generally thought to involve large build-ups of pressure within the porous material which the structure of the concrete is not able to sufficiently dissipate, so fractures occur and chunks of the material are forced suddenly outward.

The main prerequisites for spalling are relatively well established, these being moisture content of at least 2% and most importantly steep temperature gradients within the material. Temperature gradients are dependent not only on gas-phase temperatures but also heating rates, so that it is not possible to define a threshold temperature per se. However, these values may be affected by the type of concrete, including the strength of the material and the presence of fibres, as described below.

There has been a large amount of recent research on the potential for inclusion of various types of fibres into concrete to mitigate the effects of spalling. Some studies have included polypropylene fibres into the concrete matrix. The theory is that when the concrete is subjected to heat, the polypropylene will melt, creating pathways within the concrete for the exhaust of water vapour and any other gaseous products, which will thereby reduce the build-up of pressure within the concrete. There has been some debate as to whether mono-filament or multi-filament fibres are better able to mitigate spalling. It has also been suggested that the melted polypropylene fibers can form a barrier to the transport of moisture further into the concrete, preventing pressure build-up at greater depth and forcing the moisture to escape instead. The same report suggests that the polypropylene fibers may provide a mechanism for cracking deeper within the concrete, which may mitigate spalling at the surface, but may have adverse structural consequences. Clearly, more work needs to be done in this area. Other studies have added steel fibers to concrete systems; the theory behind this is that the steel will increase the ductility of the concrete and make it more able to withstand the high internal pressures. Results are, so far, inconclusive[1].

Recently there has been increasing use of 'high strength concrete'. This material typically has considerably higher compressive strength than normal strength concrete, however it is also considerably less porous and moisture absorbent. While this generally reduces the water content of the cement, it is also harder for water vapour to escape during heating. Spalling has been suggested to be relatively more common in high strength concrete, due to the lower porosity of high strength concrete and hence the increased likelihood of high pressure developing within the concrete structure. However, some recent research has shown that this is not necessarily the case, with testing showing higher spalling resistance in high strength concrete than in normal strength[1].

ii. Cracking

The processes leading to cracking are believed to be essentially the same as those leading to spalling. Thermal expansion and dehydration of the concrete due to heating may lead to the formation of fissures in the concrete rather than, or in addition to, explosive spalling. These fissures may provide pathways for direct heating of the reinforcement bars, possibly bringing about more thermal stress and further cracking. Under certain circumstances the cracks may provide pathways for hot combustion products to spread through the barrier to the adjoining compartment, but this has not been the subject of significant research[1].

A case study was made on cracking in a concrete building subjected to fire, with particular emphasis on the depths to which cracking penetrates the concrete[4]. It was found that this relates to the temperature of the fire, and that generally the cracks extended quite deep within the concrete member. Major damage was confined to the surface near to the fire origin, but the nature of cracking and discoloration of the concrete suggested that the material around the reinforcement had reached about 700°C. Cracks which extended more than 3cm into the depth of the structure were attributed to a short heating/cooling cycle due to the fire being extinguished

2.2 Supplementary cementing materials (SCMs)

Mineral admixtures such as rice husk ash, silica fume, fly ash, and ground granulated blast-furnace slag improve the engineering properties and performance of concrete when they are used as mineral additives or as partial cement replacements [8,9]. Economic (lower cement requirement) and environmental considerations have also played a great role in the rapid increase in usage of mineral admixtures. Compared with Portland cement, cement with pozzolan helps to have concrete with less permeability and a denser calcium silicate hydrate (C–S–H) is obtained[7].

SCMs can be divided into two categories based on their type of reaction: hydraulic or pozzolanic. Hydraulic materials react directly with water to form cementitious compounds, while pozzolanic materials chemically react with calcium hydroxide (CH), a soluble reaction product, in the presence of moisture to form compounds possessing cementing properties[7].

Pozzolanic SCMs can be used either as an addition to the cement or as a replacement for a portion of the cement. Most often an SCM will be used to replace a portion of the cement content for economical or property-enhancement reasons. Current construction industry practices include the addition of rice husk ash, silica fume, fly ash, and ground granulated blast-furnace slag[7].

2.2.1 Rice husk ash

Rice husk is an abundantly available waste material in all rice producing countries. In certain regions, it is sometimes used as a fuel for parboiling paddy in the rice mills. The partially burnt rice husk in turn contributes to more environmental pollution. There have been efforts not only to overcome this but also to find value addition to these wastes using them as secondary source of materials[5].

Rice husk contains nearly 20% silica, which is present in hydrated amorphous form. On thermal treatment, the silica converts tocrystobalite, which is a crystalline form of silica. However, under controlled burning conditions, amorphous silica with high reactivity, ultra fine size and large surface area is produced. Due to the high pozzolanic activity, this rice husk silica also finds application in high strength concrete. Possibility of using this silica as filler in polymers is also studied. Introduction of more sophisticated instruments in the field of material characterization has enabled detailed structural studies of rice husk and its thermally treated products[5].

Rice husk ash (RHA) is a natural byproduct from the processing of paddy rice. The husks, which are approximately 50 percent cellulose, 30 percent lignin and 20 percent silica, are incinerated by controlled combustion leaving behind an ash that predominantly consists of amorphous silica. Rice husk ash is highly pozzolanic due to its extremely high surface area (50,000 to 100,000 m^2/kg). Research has shown that higher compressive strengths, decreased permeability, resistance to sulfate and acid attack, and resistance to chloride penetration can all be expected when a high-quality RHA is used in amounts of 5 percent to 15 percent by mass of cement.

Rice husk ash is one of the promising pozzolanic materials that can be blended with Portland cement for the production of durable concrete and at the same time it is a value added product. Addition of rice husk ash to Portland cement not only improves the early strength of concrete, but also forms a calcium silicate hydrate (CSH) gel around the cement particles which is highly dense and less porous. The rice husk ash rich in silica content is obtained by burning rice husk to remove the volatile organic carbon such as cellulose and lignin.

2.2.2 RHA in Malaysia

Rice husk is a waste product of the agriculture activity in most countries in Asia and in particular Malaysia. Rice husk has posed a major problem of disposal to the rice milling industry in Malaysia and elsewhere in the world. Currently, there are a few companies in Malaysia, mainly in Kedah and Penang, producing and exporting RHA. According to the statistics compiled by the Malaysian Ministry of Agriculture, there are 408,000 t of rice husk produced in Malaysia annually[6].

2.2.3 Comparison of burning methods

In the past, RHA was produced under uncontrolled combustion. The ensuing environmental concerns prompted the need to adopt controlled burning methods to extract amorphous silica from rice husk.

Naair, Jagadish and Fraaij have studied the properties of RHA produced from various types of field ovens. The various types of field ovens used were annular oven, brick oven and burning pit. The RHA obtained was then compared with those produced from laboratory muffle furnaces and waste ash collected from a rice factory. The results showed that the amount of SiO_2 obtained through controlled combustion was 5% - 6% higher compared to the uncontrolled combustion of fields ovens[10].

The time taken to complete the entire process also shows significant differences between the uncontrolled and controlled burning. The annular oven that used two arranged cylinders to burn the rice husk needed nine hours to completely burn its contents. The brick oven, which was a rectangular enclosure, was built from bricks having small numbers of openings in the body of the enclosure to allow smooth flow of air. This oven took three days to complete the burning process. The burning pit method took almost a week to complete burning and simultaneously cool the sample. Meanwhile, furnace burning, which is a controlled combustion method took 14 hours to complete the burning process[10]. These field oven methods stress the disadvantages of uncontrolled methods in terms of burning time efficiency.

Research on the pozzolanic activity of RHA produced through different burning methods have also been studied. The results show that RHA obtained through furnace burning has the highest value of pozzolanicity, followed by field oven samples, meanwhile waste ash from rice factories have the lowest. RHA produced in an annular oven has the highest pozzolanic activity[10].

2.3 Microwave incineration

To produce the best pozzolanas, the burning of the husk must be carefully controlled to ensure that the formation of carbon is kept to a minimum by supplying an adequate quantity of air. Proper burning is important in obtaining RHA with high reactive silica content. Modern incinerators such as microwave incinerators are designed to avoid environmental problems such as caused by open burning. Microwave incinerators are capable of producing RHA with high pozzolanic reactivity which can significantly enhance the properties of concrete.

2.3.1 Procedure

Microwaves are part of the electromagnetic spectrum and are located between 300MHz and 300GHz. Microwave heating is defined as the heating of a substance by electromagnetic energy operating in that frequency range. There is a fundamental difference in the nature of microwave heating when compared to conventional methods of heating material. Conventional heating relies on one or more of the heat transfer mechanisms of convection, conduction and radiation to transfer thermal energy into the material. In all three cases, the energy is deposited at the surface of the material and the resulting temperature gradient established in the material causes the transfer of heat into the core of the object. Thus, the temperature gradient is always into the material with the highest temperatures being at the surface[11].

In microwave heating, the microwave energy not only interacts with the surface material but also penetrates the surface and interacts with the core of the material as well. Energy is transferred from the electromagnetic field into thermal energy throughout the entire volume of the material that is penetrated by the radiation. Microwave heating does not rely on conduction from the surface to bring heat into the core region. Since the heating rate is not limited by conduction through the surface layer, the material can be heated quicker. Another important aspect of microwave heating is that it results in a temperature gradient in the reverse direction compared to conventional heating, meaning the highest temperature occurs at the centre of the object and heat is conducted to the outer layer of the material[11].

2.3.2 Advantages

Since the heating rate is not limited by the conduction through the surface layer, material can be heated quicker with through microwave heating. The shorter process time gives economical advantages to the whole production process. Problems such as cracking that may occur when drying material can be reduced because the heat is generated in all parts of the material that are receiving radiation. This effect reduces the internal stresses in the material and can help eliminate cracking that may occur when the internal stresses become too large[11].

The additional device called Flue Gas Filter is equipped to the microwave incinerator and provides significant positive effect to the environment. It distills all the ashes and dust that result from rice husk incineration, once again reducing the air pollution caused during the burning process[11].

2.4 Superplasticizers

The use of superplasticizers (high range water reducer) has become a quite common practice. Superplasticizers are linear polymers containing sulfonic acid groups attached to the polymer backbone at regular intervals. The sulfonic acid groups are responsible for neutralizing the surface charges on the cement particles and causing dispersion, thus releasing the water tied up in the cement particle agglomerations and thereafter reducing the viscosity of the paste and concrete[12] .

The main purpose of using superplasticizers is to produce flowing concrete with very high slump in the range of 7-9 inches (175-225 mm) to be used in heavily reinforced structures and in placements where adequate consolidation by vibration cannot be readily achieved. The other major application is the production of high-strength concrete at w/c ratio ranging from 0.3 to 0.4.

The family of superplasticizers which are based in polycarboxylic products are more recent (1980s). These materials are of higher reactivity, they do not contain the sulfonic group and they are totally ionized in alkaline environment. The superplasticizers of high reactivity, which in high dosages do not have the side-effect of delaying the curing of concrete, made the production of concrete with a big volume of fly ash or slag possible. As it is known the superplasticizers increase the workability of a mixture very much. However, this increase is not retained for more than 30–60 min. There are various ways (addition of admixtures during the placement or in doses) in which workability can be retained for longer time. The type of the admixture also seems to affect the loss of slump. Although the superplasticizers do not react by a chemical action on hydrated products, they affect the microstructure of cement gel and concrete. The porosity and the bleeding decrease significantly and, on a second level, the drying shrinkage and creep deformations. Thus, beyond the increase of strength, there is also an increase of the durability of concrete with the use of superplasticizers [12].

The ability of superplasticizers to increase the slump of concrete depends on such factors as the type, dosage, and time of addition of superplasticizer; w/c; and the nature or amount of cement. It has been found that for most types of cement, superplasticizer improves the workability of concrete.

One problem associated with using a high range water reducer in concrete is slump loss. The slump loss problem can be overcome by adding the admixture to the concrete just before the concrete is placed.

2.5 Pollution problems

Considerable efforts are being taken worldwide to utilize local natural waste and by-product materials in making concrete, such as silica fume (SF) or rice husk ash (RHA) as supplementary cementing materials to improve concrete properties (durability, strength, etc.).

Burning of agricultural wastes creates large amounts of soot particles. These soot particles can be blown over long distances and are mainly responsible for the haze that often covers the sky. These fires not only pollute the air but also destroy the rich habitat of the flora and fauna. In Malaysia, there have been reports of rice-millers who do not comply to the law which has resulted in the people living around the mills especially in Kedah, threatened by dust and rice husk.

2.6 Scanning Electron Microscopy

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. In an optical microscope, lenses are used to bend the light waves and the lenses are adjusted for focus. In the SEM, electromagnets are used to bend an electron beam which is used to produce the images on a screen. Beam of electron is produced in an electron gun by heating of a metallic filament. This electron beam will follow a vertical path through the column of the microscope and pass electromagnetic lenses, which focus and direct the beam towards the sample. When the electron beam hits the sample, other electrons, called as backscattered electron and secondary electron, are ejected from the sample. The detectors will collect these secondary and backsterred electrons and convert them to a signal that is sent to a display screen[13].

CHAPTER 3

METHODOLOGY

3.1 Material selection

Materials used in this research were selected according to the specifications that complemented the objectives of this research and complied to the requirements of appropriate standards used.

3.1.1 Microwave Incinerated Rice Husk Ash (MIRHA)

Rice husk used in this research was obtained from Bernas, a rice milling plant in Malaysia. Rice husk was burnt in an automatic microwave incinerator to produce amorphous MIRHA.

The combustion was based on the condition that the automatic microwave incinerator received its heat from microwave heating and heat of combustion. Straight combustion to a maximum of 550°C can cause over burning of rice husk. When the temperature reaches 550°C, not all of the volatile material in the rice husk is completely burnt and these un-burnt material will continue to generate fire that causes excess heat within the incinerator. This study required RHA to be burnt at 800°C, 700°C and 600°C, so to avoid over-burning, the incinerator was turned off once the temperature within the incinerator reached 500°C, 400°C and 350°C respectively during each burning process. Figure 3.1 shows the microwave incinerator used for this research. Figure 3.2 shows the microwave incinerated rice husk ash after a 24 hour cooling period.

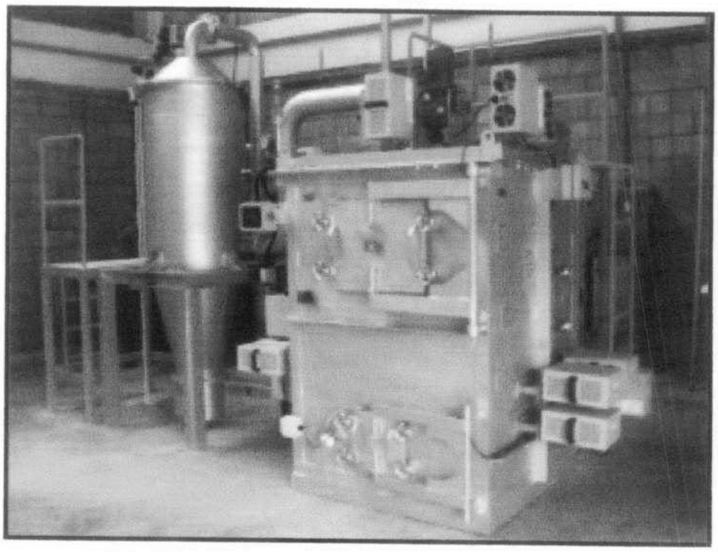

Figure 3.1

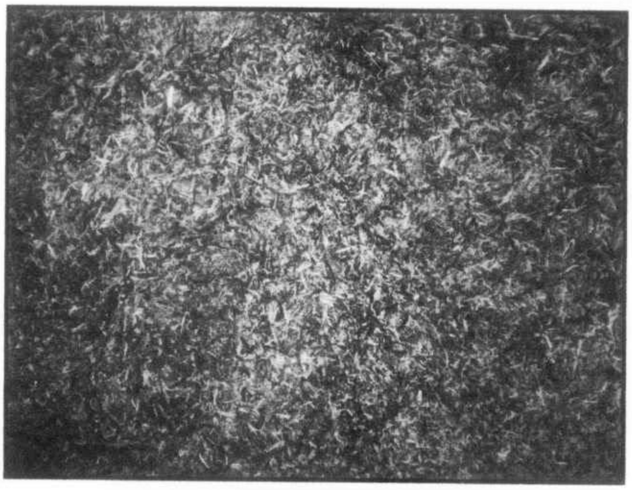

Figure 3.2

MIRHA was then ground in the ball mill to increase its fineness. Due to the large amount, MIRHA was separated into three batches. Each batch is ground for 999 cycles. A fineness similar to or greater than that of OPC is usually recommended for pozzolans. Figure 3.3 shows the Los Angeles grinder used for this research. Figure 3.4 shows the MIRHA obtained after the grinding process.

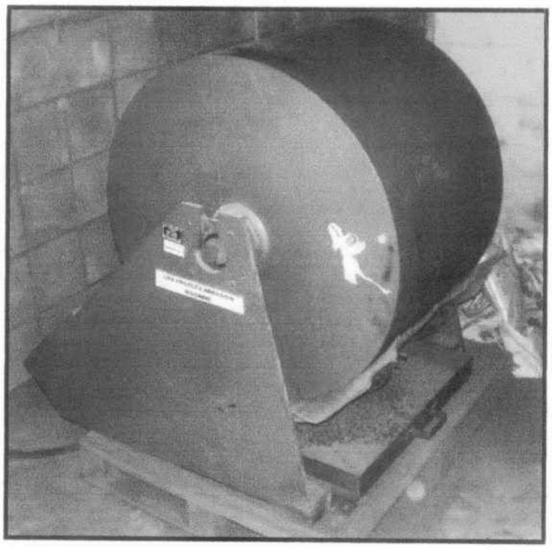

Figure 3.3

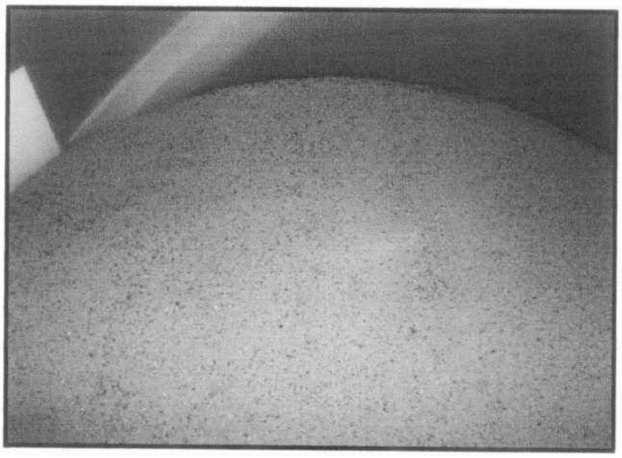

Figure 3.4

3.1.2 Water

The water used in the mix needs to be free from harmful chemicals, oil, chloride, silt or any harmful contaminant that could affect the performance of concrete. The water used during concrete mixing was tap water obtained from the laboratory.

3.1.3 Cement

Ordinary Portland Cement (OPC) Type 1 was used in this research, OPC Type 1 was selected based on its physical and chemical properties listed in BS EN 197-1 2000.

3.1.4 Superplasticizer (SP)

The superplasticizer used in this research was a high range water-reducing concrete admixture that complied to the requirements of BS EN 934-2:2001. This admixture, which is a polycarboxylate type, has a density of 1.11kg/l and pH value of 5.5.

3.1.5 Aggregate

The fine aggregate used was natural sand with a fineness modulus of 2.7 while the coarse aggregate used was crushed aggregate with a maximum size of 20 mm, both in accordance to BS 812-103.2 1989. Coarse aggregates were prepared as saturated surface dry aggregated by washing the aggregates. The wet aggregates were then left to dry but sheltered from direct sunlight.

3.2 Concrete mixing and sampling

The concrete mix proportion used in this research was 0.45w/c. MIRHA was used to partially replace the cement content in concrete by 5%, 10%, 15% and 20%. Concrete samples without the addition of MIRHA were used as control specimens. Superplasticizer was only used in concrete incorporating MIRHA, to increase its workability. The amount of superplasticizer used was designed for each mix proportion incorporating MIRHA to achieve a similar level of workability as control concrete, which does not contain the addition of superplasticizer in its mix proportion. Table 3.1 shows the mix proportions designed for this research.

Table 3.1: Designed mix proportion

w/c	Percentage of RHA (%)	Superplasticizer (%)	Cement (kg/m³)	Water (kg/m³)	Fine aggregate (kg/m³)	Coarse aggregate (kg/m³)
0.45	0	0	475.00	213.75	607.25	1127.75
	5	0.4	451.25	213.75	607.25	1127.75
	10	0.8	427.50	213.75	607.25	1127.75
	15	1.5	403.75	213.75	607.25	1127.75
	20	2.0	380.00	213.75	607.25	1127.75

The concrete mixed was mixed in accordance to BS 1881-125.1986. First, the fine and coarse aggregates were mixed for 1 minute. Half the water proportion was added to the mixed aggregates and mixed again for 1 minute. These wet aggregates were then left in the machine for 8 minutes. This is done to allow the aggregates to absorb the surrounding water. Cement was then added into the mixture and mixed for 1 minute. For the mix proportions containing MIRHA, both the cement and MIRHA will be added into to mixture during the same time. Finally, the remaining water is then added to the mixture and mixed for 1 minute. Before the fresh concrete is taken for a workability test (slump test) and cast into moulds, the mixture was hand-mixed to ensure the homogeneity of the concrete.

The moulds were greased with engine oil to enable easier removal of hardened concrete cubes. Fresh concrete was cast in 3 layers within the 150 mm x 150 mm iron mould and compacted using a poker vibrator. Next, the specimens in moulds were covered with plastic sheets to prevent contamination and left in the casting room for 24 hours. These specimens were then de-moulded and placed in a curing tank with a temperature range of 18°C - 22°C, until further testing.

3.3 Concrete testing

Testing of concrete specimens was conducted in two phases, fresh and hardened concrete. Fresh concrete was tested to analyze its workability characteristics using Slump test. Hardened concrete test were performed to analyze the structural uniformity, integrity and strength development of concrete using two test methods, destructive and non-destructive.

The non-destructive method involves the Ultrasonic Pulse Velocity (UPV) test to analyze the integrity of burnt concrete samples. The destructive method consists of compressive strength test and fire test. Table 3.2 shows the experimental details of concrete testing methods for this research.

3.3.1 Slump test

The slump test in this research was performed according to BS EN 12350-2:2000. The freshly mixed concrete is packed into a 305 mm high cone. The concrete sample was filled into the cone in 3 layers. Each layer was tamped 25 times with a standard 16 mm diameter steel rod, rounded at the end. Then the top of the cone is smoothed off level with the top rim of the cone, and the cone is then carefully lifted, so that the concrete is left unsupported. The slump is the distance that the centre of the cone top settles. The decrease in height of the centre of the slumped concrete was then measured. Figure 3.5 shows the apparatus used for conducting a slump test.

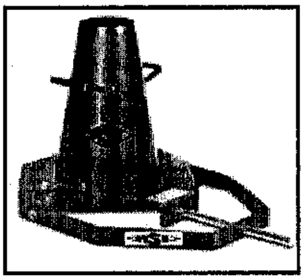

Figure 3.5

Table 3.2: Experimental details of concrete testing methods

Concrete type	Test type	Standard	Equipment	Testing age	Sample size	Number of test	Measurement
Fresh concrete	Slump test	BS EN 12350-2:2000	Slump cone	Fresh concrete	-	Each batch	mm
Hardened concrete (destructive test)	Compression strength (before fire test)	BS EN 12390-3:2002	Compression testing machine	3, 7 and 28 days	150 mm^3	3 cubes/mix/age	N/mm^2
	Compression strength (after fire test)	BS EN 12390-3:2002	Compression testing machine	56 days	150 mm^3	1 cube/mix/age	N/mm^2
	Fire test	ASTM E 119	furnace	28 days	150 mm^3	1 cube/mix/age	observation
Hardened concrete (Non-destructive test)	Ultrasonic Pulse Velocity (UPV)	BS EN 12504-4:2004	PUNDIT UPV tester	56 days	150 mm^3	1 cube/mix/age	m/s

3.3.2 Ultrasonic Pulse Velocity test

Ultrasonic Pulse Velocity test in this experiment utilized direct transmission method according to BS EN 12504-4:2004, where an electro acoustical transducer (transmitter) was placed on the opposite surface of longitudinal pulse receiver. The UPV test measures the time of travel of an ultrasonic pulse passing through the concrete. The time taken for the pulse to pass through the concrete is measured by electronic measuring circuits. In defect areas, the compressional wave velocity is slower than in sound areas and signal amplitude is often lower. For structural members containing large, severe voids, signal transmission may be completely lost. The principle use of this test is to check the homogeneity of the concrete and locate the presence and approximate extent of cracks and voids. The UPV test was conducted using Portable Ultrasonic Non-Destructive Digital Indicative Tester (PUNDIT). Measurement was taken for 1 cube per mix at 56 days.

3.3.3 Compressive Strength test

Compressive strength development of the concrete was measured according to BS EN 12390-3:2002 using a Digital Compressive Testing Machine. The test was performed on 3 concrete cubes per mix at ages 3, 7 and 28 days. During the test, concrete cubes were loaded with 6.8 KN/s of constant load without any sudden shock loads.

3.3.4 Fire resistance test

This Fire resistance test was performed according to ASTM E 119. Concrete cubes were placed into a pre-heated furnace at 800°C . The cube was kept in the furnace for 2 hours or until spalling occurred. The test was performed on 1 cube per mix at 28 days. Then, specimens were taken out and placed under room temperature for cooling down (natural cooling). The natural cooling method is to simulate the situation in which a concrete structure is exposed to fire and then cooled down naturally. Changes in colour and surface was observed. After the burning, the concrete cubes will be inspected to check if spalling has occurred, if yes, to what extent. Figure 3.6 shows the furnace used in this research.

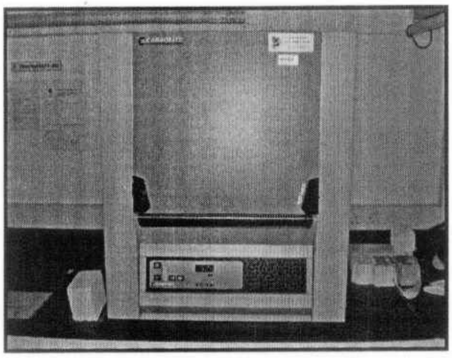

Figure 3.6

3.3.5 Microstructure analysis

Scanning Electron Microscopy (SEM) analysis was carried out to determine the inner microstructure condition of burnt concrete samples with MIRHA replacement. Analysis was conducted on concrete samples with high compressive strength. Concrete as a non-conductive material must be coated with gold atoms in a sputter coater. Coated concrete was then placed in the vacuum chamber inside the SEM. The SEM must be operated under specific pressure to facilitate the operation of filament and electrons within the SEM.

3.4 Hazard analysis

Microwave incineration does produce emissions of organic gases and dust but the emission levels do not violate the Department of Environment's list of regulations on air pollution control under the Environmental Quality Act (EQA) 1974.

CHAPTER 4

RESULTS

4.1 Slump test

The concrete mixture in this research was designed to have slump within 30 mm – 40 mm. High water reducing superplasticizer was added to the MIRHA concrete mix proportion to increase its workability. the slump for the natural concrete (0% MIRHA) measured 32 mm. The first mix of MIRHA blended concrete only measured 7 mm, prompting a change of w/c ratio from 0.40 to 0.45 and the addition of superplasticizer. The results show no significant pattern for the effects of MIRHA on the water absorbing capabilities of concrete. Table 4.1 shows the slump measurements for all mix proportions in this research.

Table 4.1: Slump measurements

Mix	Slump (mm)
600°C-5%	31
600°C -10%	35
600°C -15%	29
600°C -20%	34
700°C -5%	32
700°C -10%	32
700°C -15%	32
700°C -20%	29
800°C -5%	38
800°C -10%	33
800°C -15%	31
800°C -20%	41
control (0% MIRHA)	32

4.2 Ultrasonic Pulse Velocity test

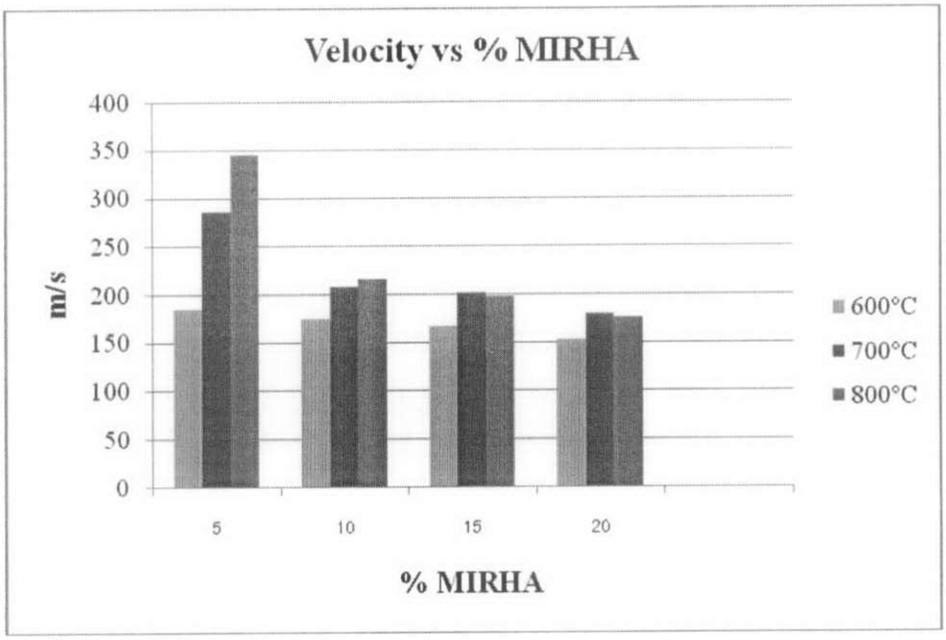

Figure 4.1

The results for the ultrasonic pulse velocity test performed on MIRHA blended concrete specimens are presented in Figure 1. It can be seen that the velocity of pulse continuously decreases with increase in the percentage MIRHA content. In a previous research, Andri Kusbiantoro found that for 0.45 w/c mix proportion, the average pulse velocity is 4400 m/s. The burnt concrete sample with the highest pulse velocity was MIRHA at 800°C-5% replacement with 345 m/s, hence, in comparison there is a 92% reduction in velocity between an unburnt sample and a burnt sample. This shows that exposure to a constant elevated temperature can adversely damage the structural integrity of concrete. The long duration of exposure during the fire test caused the further deterioration of the MIRHA blended cubes. This observation is true for all MIRHA blended cubes tested with 600°C, 700°C and 800°C MIRHA content. Thus, the integrity of MIRHA blended concrete tested under elevated temperatures is considerably increased by partial replacement of OPC with MIRHA. Concrete cubes with MIRHA at 800°C are the most consistent. The cube with 5% replacement of MIRHA at 800°C recorded the highest velocity at 345 m/s.

4.3 Compressive strength test before Fire resistance test

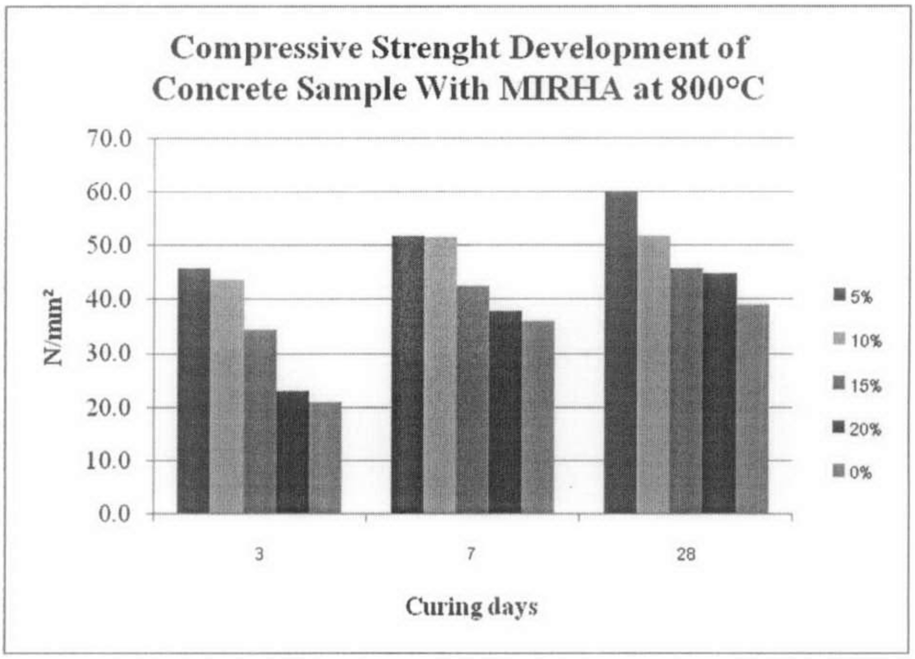

Figure 4.2

Table 4.2: Comparison of percentage increase in compressive strength of MIRHA at 800°C with control specimen

Percentage of increase in MIRHA at 800°C concrete compressive strength compared to control specimen (0% MIRHA)				
% MIRHA / Days curing	5%	10%	15%	20%
3	105	108	63	9.5
7	44	43	18	5
28	55	33	17	53

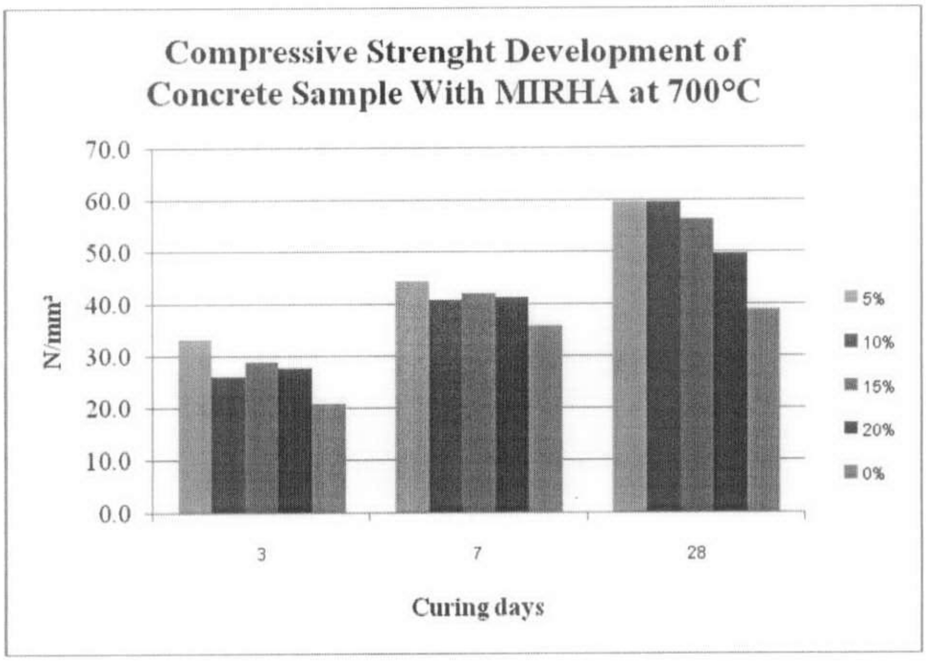

Figure 4.3

Table 4.3: Comparison of percentage increase in compressive strength of MIRHA at 700°C with control specimen

Percentage of increase in MIRHA at 700°C concrete compressive strength compared to control specimen (0% MIRHA)				
% MIRHA / Days curing	5%	10%	15%	20%
3	58	24	39	31
7	23	13	16	15
28	53	53	44	27

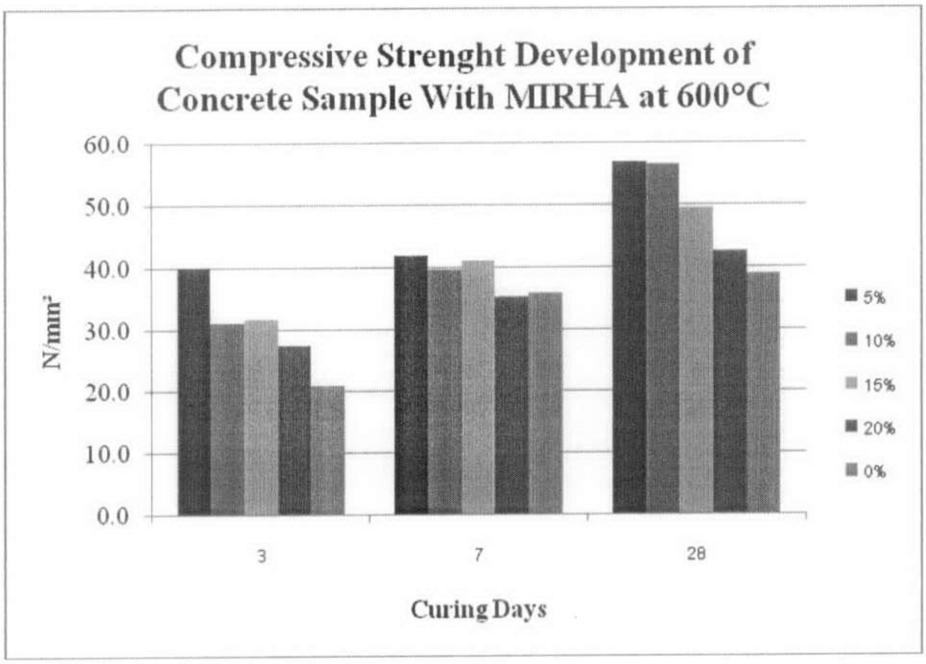

Figure 4.4

Table 4.4: Comparison of percentage increase in compressive strength of MIRHA at 600°C with control specimen

Percentage of increase in MIRHA at 600°C concrete compressive strength compared to control specimen (0% MIRHA)				
% MIRHA / Days curing	5%	10%	15%	20%
3	91	48	50	31
7	16	10	14	-1.9
28	46	45	26	8

4.4 Compressive strength test after Fire resistance test

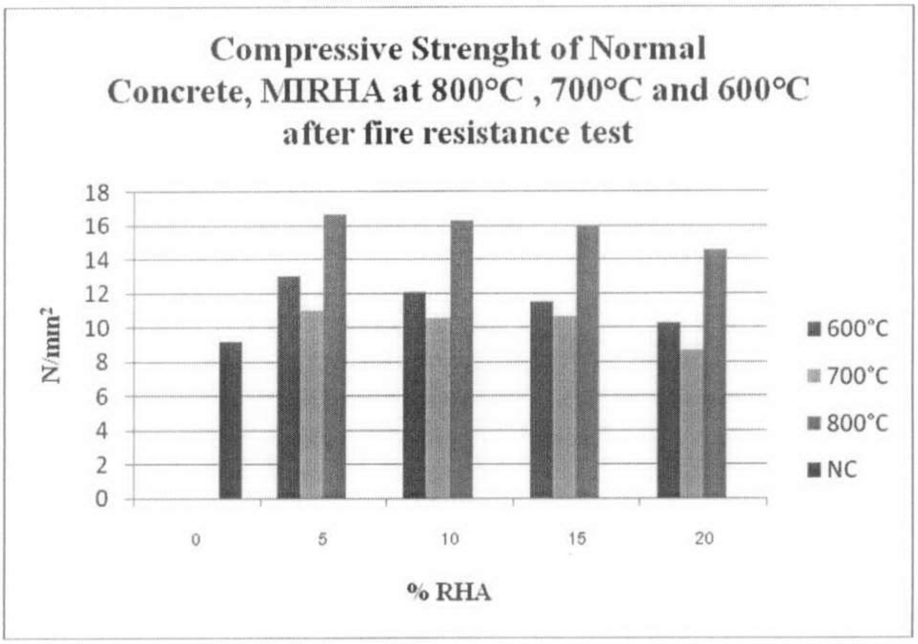

Figure 4.5

Table 4.5

Percentage of increase in MIRHA concrete compressive strength compared to control specimen (0% MIRHA)				
% MIRHA / MIRHA / Temperature (°C)	5%	10%	15%	20%
600	42	31	24	11
700	19	14	15	-5.7
800	81	77	74	58

The compressive strengths of MIRHA blended concrete specimens are shown in Figures 4.2, 4.3 and 4.4. Comparison of the data for 3, 7 and 28 days of curing time shows that the compressive strength increases with the number of curing days irrespective of the MIRHA content. Tables 4.2, 4.3 and 4.4 show the percentage increase of compressive strength of all MIRHA samples compared to that of the control specimen.

Figure 4.4 lists the compressive strength of MIRHA concrete samples at 600°C had minimal increase between 3 and 7 days of curing. After 28 days of curing the rapid development of C-S-H gel stimulated an increase in compressive strength. The compressive strength of 5% MIRHA at 600°C was 46% higher than of the control specimen.

Figure 4.3 lists the compressive strength of MIRHA at 700°C . MIRHA at 700°C produced a steady increase in compressive strength throughout the 3, 7 and 28 days of curing. After 28 days of curing, compressive strength of the 5% MIRHA at 700°C was 51% higher than of the control specimen.

The most consistent and highest increase in compressive strength can be seen in all 5% MIRHA blended concrete specimens. A similar pattern can be seen in the compressive strength comparison after the fire resistance test. Figure 4.2 lists the compressive strength of MIRHA at 800°C. The 5% MIRHA at 800°C concrete specimen produced the highest compressive strength post-fire resistance testing and after 28 days curing. This is because the MIRHA at 800°C had more complete burning compared to the MIRHA at 600 and700°C. A complete burning results in MIRHA with lesser carbon and more transition of hydrate amorphous to amorphous silica. Once the amorphous silica reacted with the calcium hydroxide in the presence of water during concrete mixing, the MIRHA at 800°C produced more calcium silicate hydrate gel, which bound the aggregate to the cement paste. After 28 days curing, the 5% MIRHA at 800°C concrete specimen recorded a compressive strength 55% higher than of the natural concrete.

Figure 4.5 shows the compressive strength of MIRHA concrete samples after the fire resistance test. These results depict the compressive strength results before the fire resistance test with the 5% MIRHA at 800°C as the MIRHA concrete specimen with the highest compressive strength. The only difference is that the MIRHA at

700°C shows lower compressive strength compared to before the fire resistance test. This is such, because the MIRHA at 700°C concrete samples are relatively younger compared to the MIRHA at 600°C and 800°C. The MIRHA at 700°C concrete samples were approximately 15 days younger at the time of fire testing.

4.5 Fire resistance test

Table 4.6: Fire resistance test results

Temperature MIRHA incinerated at (°C) and Percentage of cement replacement	Duration in furnace (min)	Observed changes
600-5%	120	Mild cracking
600-10%	120	Small red patches,cracking
600-15%	120	Small red patches, cracking
600-20%	120	Small red patches, cracking
700-5%	120	Small red patches, Mild cracking
700-10%	120	Small red patches,cracking
700-15%	120	Small red patches, cracking
700-20%	120	Small red patches, cracking
800-5%	120	Mild cracking
800-10%	120	Mild cracking
800-15%	120	Small red patches, cracking
800-20%	120	Small red patches, cracking
Normal concrete (0%)	40	spalling

The cracks begin to appear after 1 hour when the temperature was between 600°C – 700°C. The cracks get wider as exposure duration increases. Figures 4.6 and 4.7 show the difference between cracking and mild cracking respectively. Concrete cubes with only mild cracks developed cracks much later compared to the cubes with wider cracks. The small red patches appear on almost all concrete cubes. These small red patches indicate exposure to a temperature of 800°C and above. Figures 4.8, 4.9 and 4.10 show the small red patches on the surface of the burnt concrete specimens. The normal concrete cube failed after only 40 minutes. The top right corner of the cube cracked open in a small burst. Figures 4.10 and 4.11 show the effects of spalling after 40 minutes.

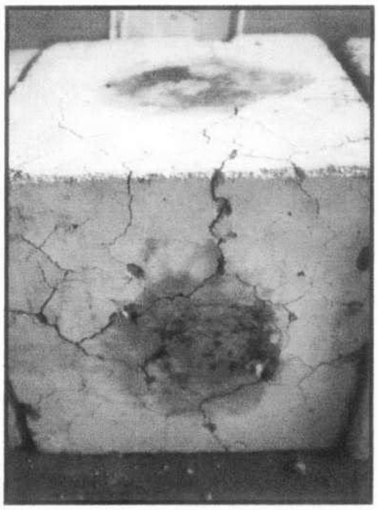

Figure 4.6

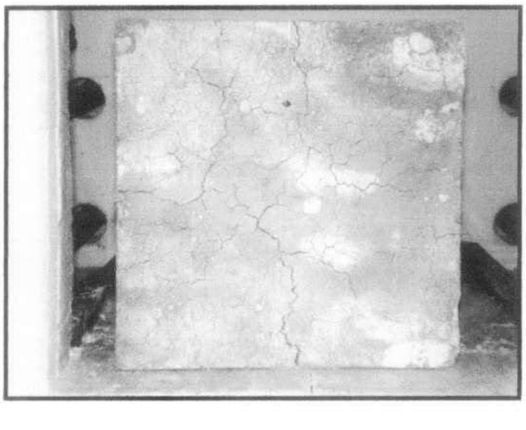

Figure 4.7

Figure 4.8

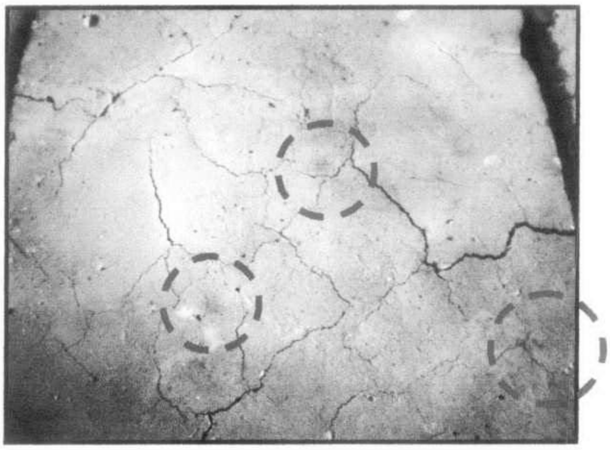

Figure 4.9

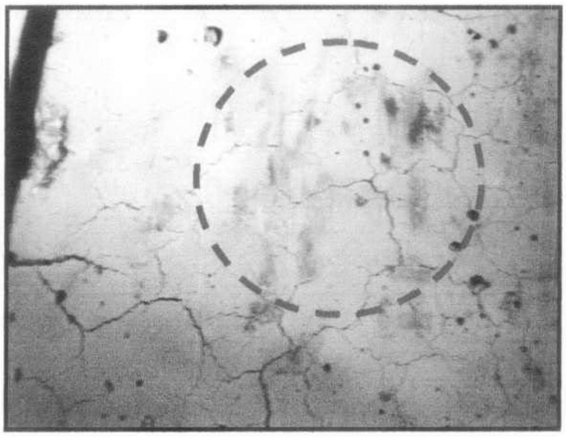

Figure 4.10

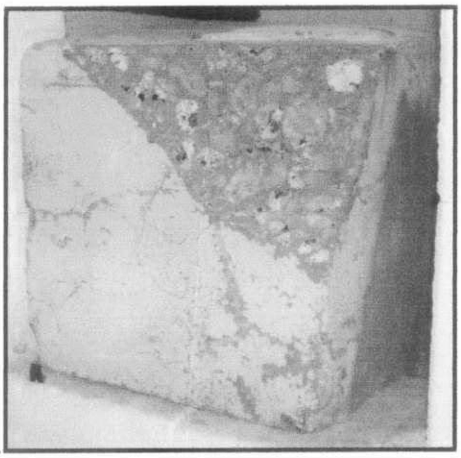

Figure 4.11

Figure 4.12

4.6 Scanning Electron Microscopy Analysis

Scanning Electron Microscopy (SEM) analysis was carried out to compare the inner conditions of burnt MIRHA concrete samples which produced the best results from the previous tests. Concrete samples for the SEM test were taken from:

- 5% MIRHA at 600°C
- 5% MIRHA at 700°C
- 5% MIRHA at 800°C

Figure 4.13 exhibits the SEM images of the burnt 5% MIRHA at 600°C concrete sample. The figure clearly shows the Interfacial Transition Zone (ITZ) between cement paste and aggregate. ITZ is a common phenomenon but could aid the development of a micro crack, hence the failure of the concrete structure. The function of MIRHA as a pozzolanic material is to hydrate in the presence of water to form Calcium Silicate Hydrate (C-S-H) and fill the gaps that exist in the ITZ. The C-S-H, which is in a gel form functions as a binder. The clear appearance of ITZ and the low concentration of cement paste explains the low compressive strength.

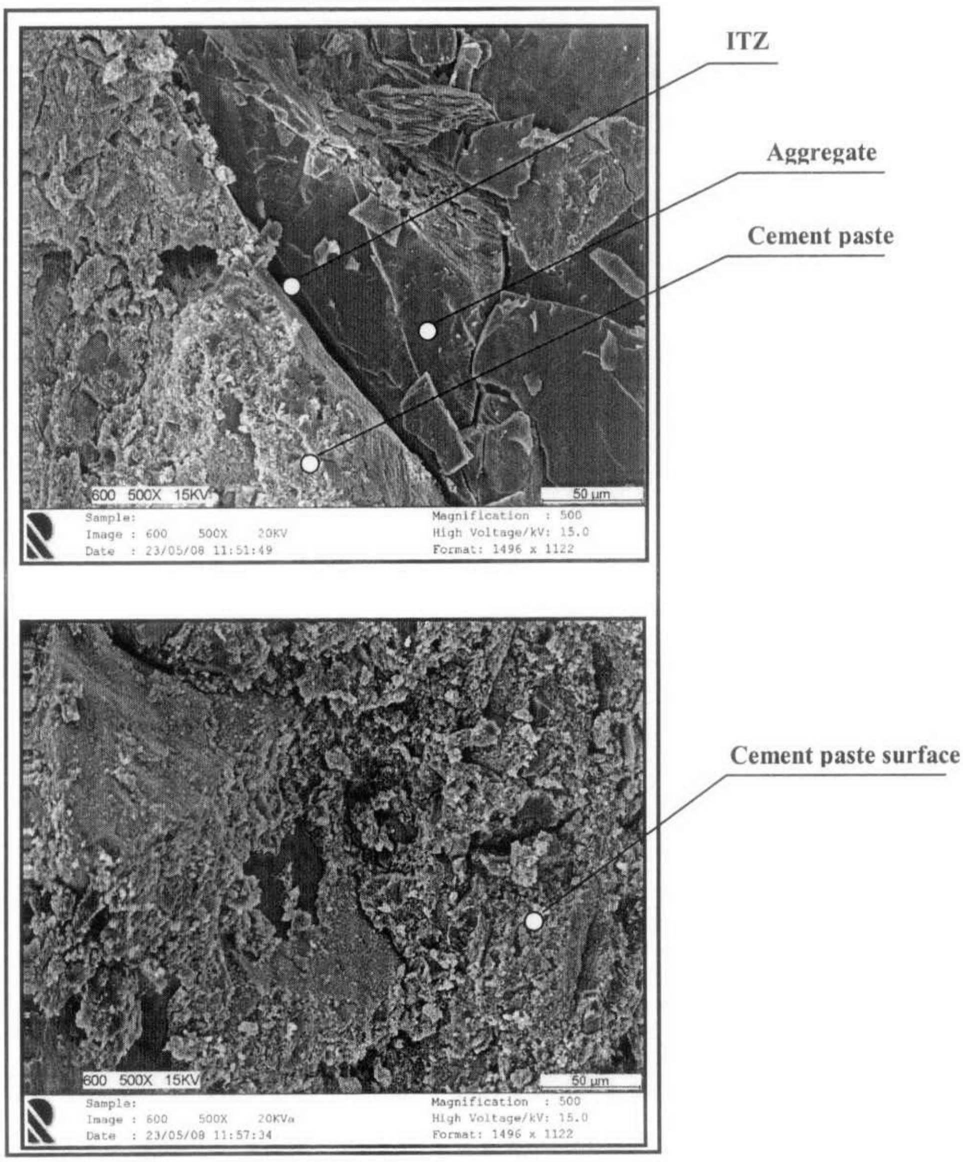

Figure 4.13

Figure 4.14 exhibits the SEM images of the burnt 5% MIRHA at 700°C concrete sample. The figure shows a micro crack path that passes through the ITZ between the aggregate and cement paste. This concrete sample also appears to be porous. These micro pores are the outcome of improper hydration. This shows that some spaces that were previously occupied with water, evaporated and was not filled by the C-S-H gel in time. This resulted in a low compressive strength.

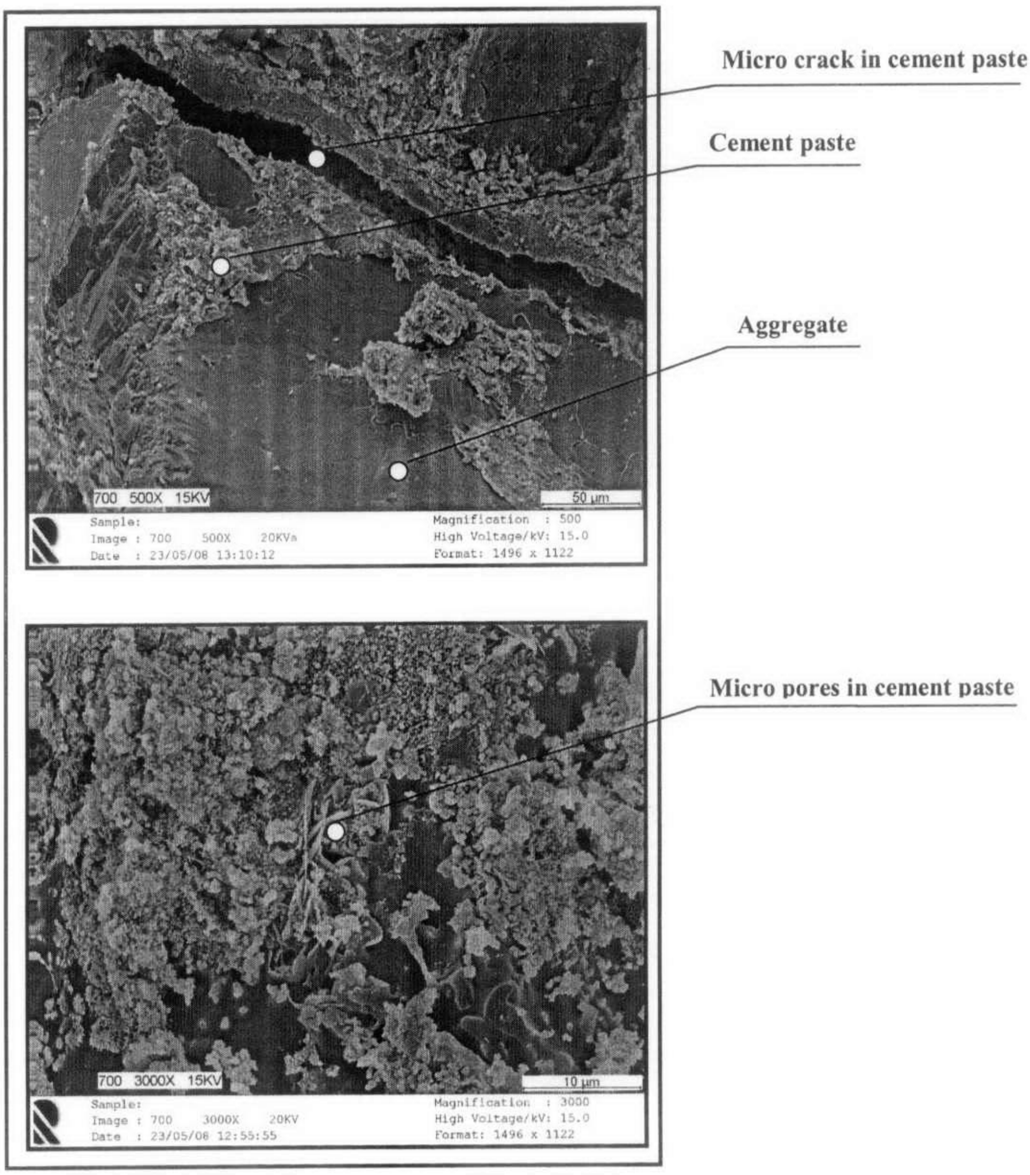

Figure 4.14

Figure 4.15 exhibits the SEM images of the burnt 5% MIRHA at 800°C concrete sample. The image shows an unfilled ITZ, yet it also shows the presence of C-SH gel. The formation of C-S-H gel was insufficient to fill the gap between the aggregate and cement paste. Compared to the previous samples, this concrete sample has almost intact cement paste even after the fire test. This could be because of the C-S-H gel, which bonded most of the cement paste together throughout the fire test.

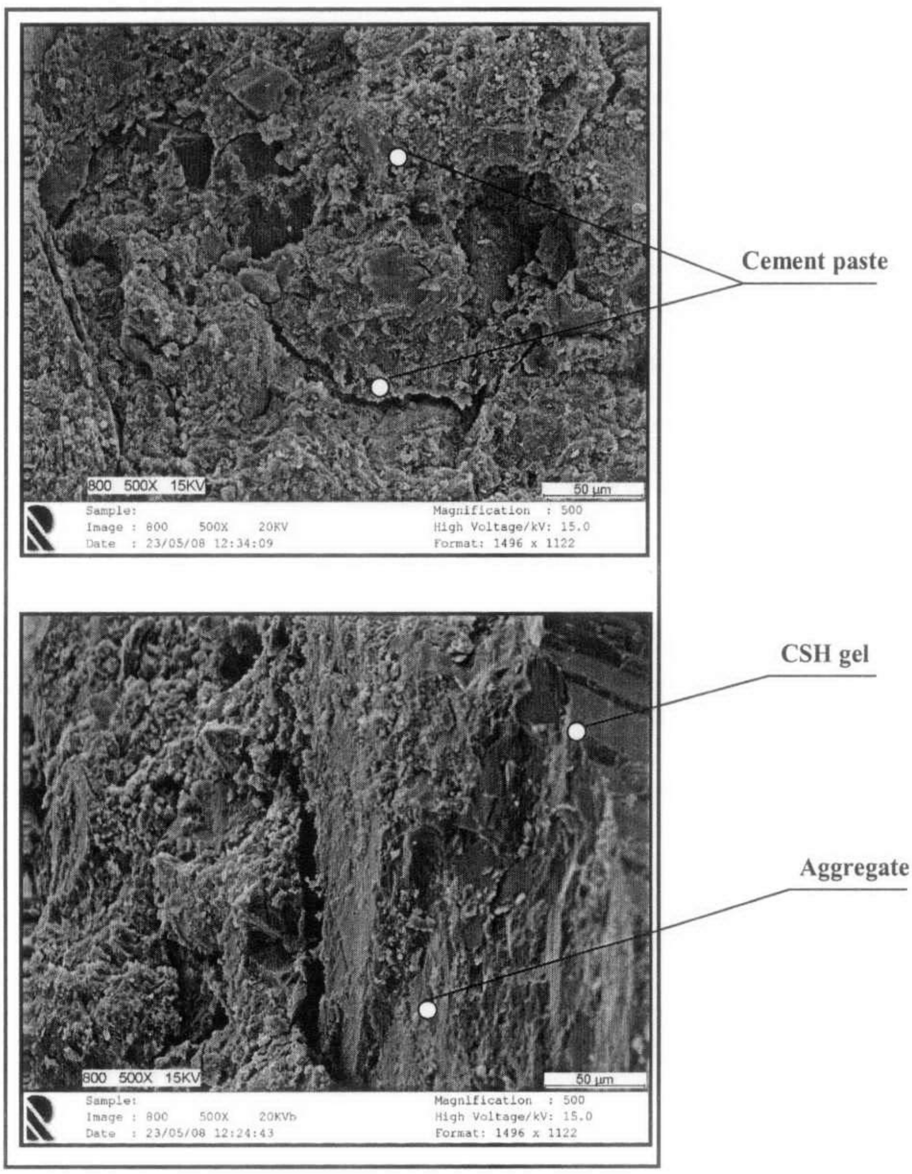

Figure 4.15

4.7 Water loss

Table 4.7: Weight loss after the fire resistance test

Temperature MIRHA incinerated at (°C) and Percentage of cement replacement	Weight loss after fire test (%)
600-5%	7.65
600-10%	7.80
600-15%	8.93
600-20%	8.08
700-5%	7.96
700-10%	8.35
700-15%	8.22
700-20%	7.97
800-5%	8.01
800-10%	8.03
800-15%	8.07
800-20%	8.25

The effect of elevated temperatures on the weight loss of the concrete specimens is insignificant. The average loss of weight post-fire testing is 8.11%. The weight loss in these MIRHA concrete samples during exposure to elevated temperatures is not attributed to just one factor. Various factors such as mechanical properties, pozzolanic composition, proper hydration of calcium silicate in MIRHA, loss of water during mixing of concrete, etc.

CHAPTER 5

CONCLUSION

The transport of ultrasonic pulse through MIRHA blended concrete depends on the pore structure of the concrete. The highly reactive pozzolana, such as rice husk ash is able to reduce the size of voids in hydrated cement pastes, thus, making them almost impermeable. This is the reason the pulse velocity for the 5% MIRHA at 800°C being higher than the other MIRHA concrete samples. The MIRHA at 800°C blended concrete specimens have the highest average values for the UPV test, the highest being the 5% MIRHA at 800°C blended concrete.

An inverse relationship is found to exist among the compression strength for all MIRHA blended concrete specimens tested with 600°C, 700°C and 800°C MIRHA content. As the partial replacement of OPC with MIRHA is decreased, there is an increase in compressive strength with the number of subsequent curing days. The most consistent increase in compressive strength can be seen in all 5% MIRHA blended concrete specimens.

The concrete specimen with MIRHA at 800°C with 5% partial replacement of OPC with MIRHA is found to be able to retain its properties better compared to all other specimens at elevated temperatures. This proves that MIRHA blended concrete does increase resistivity to fire compared to normal concrete. This may be because the optimum percentage of MIRHA in the concrete stimulated pozzolanic reaction with the calcium hydroxide existing in the cement to form calcium silicate hydrate. The 800°C temperature for rice husk incineration significantly reduced the presence of carbon. Microsilica particles could fill the voids in the cement paste because of their small size and spherical shape, leading to partial closure of these voids. This is the main reason for the improvement in concrete durability and strength and resistance to elevated temperatures.

5.2 Recommendations

MIRHA has too much value to be ignored. A research of this scale is not sufficient to explore its fullest potential. Before MIRHA is used in the construction industry, a comprehensive research on the feasibility of producing MIRHA should be carried out. Only once its production is justified economically, can any other study be pursued. RHA might be an agricultural waste, but the processes involved in transforming RHA to MIRHA, and in a large scale is very costly.

Should the production of MIRHA be feasible, then other factors such as reaction of MIRHA to other construction material such as steel, can be researched.

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[20] ASTM E 199. *Standard Methods of Fire Test of Building Construction and*

Appendix A

Classification of Mineral Admixtures. Adapted from Ramachandran

Classification	Chemical and Mineralogical Composition	Particle Characteristics
II. Highly active pozzolans		
a. Condensed silica fume	Consists essentially of pure silica in non crystalline form	Extremely fine powder consisting of solid spheres of 0.1 μm average diameter (about 20 m^2/g surface area by nitrogen adsorption).
b. Rice husk ash; (Mehta-Pitt process)	Consists essentially of pure silica in non crystalline form	Particles are generally less than 45 μm but they are highly cellular (about 60 $m^2.g$ surface area by nitrogen adsorption
III. Normal pozzolans		
a. Low calcium fly ash	Mostly silicate glass containing aluminium iron, and alkalies. The small quantity of crystalline matter present consists generally of quartz, mullite, silimanite, hematite, magnetite	Powder corresponding to 15-30% particles larger than 45 μm (usually 200-300 m^2/kg Blaine). Most particles are solid spheres of average 20 μm diameter. Cenospheres and plerospheres may be present.
b. Natural materials	Besides aluminosilicates glass, natural pozzolans contain quartz, feldspar, mica.	Particles are ground to mostly under 45 μm and have rough texture.
IV. Normal pozzolans		
Slowly-cooled blast furnace slag, bottom ash, boiler slag, field-burnt rice husk ash.	Consists essentially of crystalline silicate minerals and only a small amount of non crystalline matter.	The materials must be pulverised to very fine particle size in order to develop some pozzolanic activity. Ground particles are rough in texture.

APPENDIX B

Technical Specification for Microwave Incinerator Model Bentech Inc-21

Adapted from Bentech

ITEM			DESCRIPTION
A	**GENERAL DESCRIPTION**		
	i)	Manufacturer	Pollution Engineering Sdn Bhd
	ii)	Model	BENTECH INC-21
	iii)	Capacity	1 m^3 Chamber
	iv)	Type of Waste	Paddy Husk
	v)	Overall Dimension (m)	2.3 (H) x 4.0 (W) x 4.0 (L)
	vi)	Operating Temperature	800° C
	vii)	Emission & Ash Control System	Ceramic Filter
	viii)	Combustion Control	Temperature Controller
	ix)	Mode of Operation	PLC with Manual Overwrite
	x)	Mode of Loading	Manual
	xi)	Mode of Waste Ash Removal	Manual
B	**KILN CHAMBER**		
1	**Body Casing**		
	i)	Material and Thickness	SS 304 Plate Thickness 4.5 mm – 5.0 mm
	ii)	Support and Thickness	SS 304 Angle Iron 3 inch x 3 inch x t5.0 mm and above
2	**Charging Door**		
	i)	Dimension (mm) Big Door	580 x 455
	ii)	Dimension (mm) Small Door	315 x 315
C	**MICROWAVE INCINERATOR**		
	i)	Type	Air Cooled Magnetron
	ii)	Manufacturer/Model	Pollution Engineering Sdn Bhd/ MG-AIR 2450-1100
	iii)	Country of Origin	Malaysia
	iv)	Power Rating (l/hr)	1100 W

D	**THERMOCOUPLE**			
	i)	Length (mm)	300	450
	ii)	Type	In-Connel	
	iii)	Manufacturer/Brand	IPSH Sdn Bhd	
	iv)	Country of Origin	Malaysia	
	v)	Temperature Range	Up to 1600° C	
E	**SUPPLY AIR BLOWER**			
	i)	Type	TSB 50	
	ii)	Manufacturer/Brand	Fu-Tsu	
	iii)	Country of Origin	Taiwan	
	iv)	Motor rating	1.5 KW	
	v)	Air Capacity (m^3/min)	Not less than 1.87 m^3/min	
F	**CERAMIC FILTER**			
	i)	Type	CERAFIL XS-1000	
	ii)	Manufacturer/Brand	CERAFIL	
	iii)	Country of Origin	United Kingdom	
	iv)	Surface Area (m^2)	0.19	
G	**INDUCED DRAFT FAN**			
	i)	Type	HFD 3242 T	
	ii)	Manufacturer/Brand	Maxis Fan	
	iii)	Country of Origin	Malaysia	
	iv)	Motor Rating	4 HP	
	v)	Air Capacity (m^3/min)	Not less than 0.15 m^3/sec	
H	**AIR COMPRESSOR**			
	i)	Type	TS 05 120 H	
	ii)	Manufacturer/Brand	ELGI	
	iii)	Country of Origin	India	
	iv)	Motor Rating	5 HP	
	v)	Air Capacity (m^3/hr)	Not less than 24.6 m^3/hr at 10 kgf/cm^2	

I	**CONTROL PANEL**		
	i)	Enclosure	IP54
	ii)	Model of Operation	Programmable Logic Control (PLC)
	iii)	Type of PLC	Omron or equivalent
	iv)	Type of Cubicle	MERLIN GERLIN
	v)	Type of Contractor	TELEMECANIQUE
	vi)	Type of Starter	TELEMECANIQUE
	vii)	Touch Screen	GT21 4.7 Inch Panasonic
J	**WIRING WORKS**		
	i)	Type of Wiring	PVC
	ii)	Type of Conduit/Cable Tray	Galvanized Conduit