DISSERTATION

STUDY ON THE EFFECT OF RICE HUSK IN GEOPOLYMER COMPRESSIVE STRENGTH

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ACRONYMS

MIRHA = Microwave Incinerated Rice Husk Ash

RHA = Rice Husk Ash

NaOH = Sodium Hydroxide

OPC = Ordinary Portland Cement

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CHAPTER 1

ABSTRACT

Geopolymerization is a field where the main concern is the utilization of solid waste and by products. Industrial waste products such as fly ash, rice husk ash or silica fume are used as the source of alluminosilicate powder. Geopolymerization is found to be cost effective and environmentally friendly. Geopolymer involves the silicates and aluminates of by products to undergo process of geopolymerization. Rice is a primary source of food in the Asian region. A total of 600 million tons of rice paddy are produced every year. The husks causes disposal problems. Rice Husk ash is a super pozzolan and would act as a suitable material for geopolymer cement. The project attempts to propose MIRHA geopolymer cement as a substitute to OPC for well cementing purposes by studying whether it fulfills the compressive strength requirement for well cement. There are several problems identified in the current usage of OPC. It is found that the manufacturing of OPC, the current well cement, consumes a lot of energy and resources. Currently, cement production is responsible for 5% to 8% of yearly manmade CO_2 global emissions, or nearly 1.6 billion ton of CO_2 and therefore is the second largest CO_2 emitting industry behind power generation. Manufacturing of geopolymer cement releases up to 80% less CO_2 compared to OPC. The project aims to find out the factors affecting the MIRHA geopolymer's compressive strength and study them. This would further on lead to the possibility of substituting OPC with MIRHA geopolymer cement. By substituting OPC with MIRHA geopolymer cement, we could solve the RHA disposal issue and at the same time tackle the greenhouse gas emission from cement manufacturing that we are currently facing. The project manipulates several variables mainly the water/cement ratio, the concentration of NaOH solution, curing time, curing temperature, MIRHA grain size and ash/activator ratio in order to determine its effect on the MIRHA geopolymer compressive strength. The scope of study includes conducting research on MIRHA geopolymer cement. Devising the experiment procedures and methods of carrying them out is also researched. A few sets of experiments were conducted. In each experiment, a variable is manipulated and its effect on the geopolymer compressive strength is observed. From the experiments

conducted, we are able to figure out the optimum condition for MIRHA geopolymer cement that would result in a higher compressive strength. It is concluded that a water ration of 40% or lower should be used. It is found that a MIRHA to alkali activator ratio of 2:1 results in a better compressive strength. As for alkali activator, a 10M sodium hydroxide solution results in a higher compressive strength and can be concluded as the optimum molarity for geopolymer synthesis. A finer grain size results in a better compressive strength. A longer curing time results in an increased compressive strength. Finally, it is found that an optimum temperature of 60˚C should be used for curing rather than an elevated temperature. All of these results has been presented and discussed. The objectives of the final year project has been achieved.

CHAPTER 2

INTRODUCTION

2.1 Background of Study

The term "Geopolymer" was given by Davidovits. It describes a type of cementitious binder that is formed by alkali metal hydroxide activation of alluminosilicate powder. Geopolymerization is a field where the main concern is the utilization of solid waste and by products. Industrial waste products such as fly ash, rice husk ash or silica fume are used as the source of alluminosilicate powder. Geopolymerization is a process which involves naturally occurring silico-alluminates. Geopolymerization is cost effective and environmentally friendly. Geopolymer involves the silicates and aluminates of by products to undergo process of geopolymerization[2]. Any pozzolanic compound or source of silica and alumina that is readily dissolved in the alkaline solution acts as a source of geopolymer precursor species and this lends itself to geopolymerization[2]. The alkali component acts as an activator. This process is attractive since it utilizes waste materials generated from industries. Geopolymers utilize the polycondensation of silica and alumina precursors and a high alkali content to attain structural strength [2].

Rice is a primary source of food especially in the Asian region. Globally, it is found that 600 million tons of rice paddy are produced each year [3]. Rice husk are the by-products of the rice paddy milling industries. Each ton of dried paddy produces about 20% of husk [3]. Burning of these husk would produce rice husk ash (MIRHA). Assuming a husk ratio of 18%, we can assume that the total production of RHA annually could be up to 22 million tons [3]. Disposal of rice husk has always been a big problem. As open burning is illegal, most of the rice husk ends up in landfills. The properties of rice husk ash (RHA) which are unique and has high reactivity promotes its use as a substitute for cement. Rice husk ash (RHA) is known as a super pozzolan since it has a silica content in the range of 90% to 95%. If RHA is properly prepared, it is in an active form which behaves like a cement.

This project looks into the usage of MIRHA as a substitute for Portland cement and attempts to perform preliminary studies on the MIRHA geopolymer compressive strength.

The project attempts to propose MIRHA geopolymer cement as a substitute to OPC for well cementing purposes by studying whether it fulfills the compressive strength requirement for well cement.

Nowadays, green technology plays a vital role in industries. Industrialization has been a main contributor to undesirable pollutants to the environment. Corporations are moving forward in acquiring ways that would leave minimum negative imprint to the environment. There has been an increasing awareness in terms of solid waste generation as well as its adverse impact to mankind.

The threat of climate change is considered to be a major environmental challenge. Carbon dioxide is one of the major greenhouse gases that can be found in abundance in our atmosphere. It is found that major oil and gas companies along with cement manufacturing plants are one of the biggest emitters of greenhouse gases. This particular project will be looking into cement manufacturing specifically for the oil and gas sector. The cementing systems that are used for zonal isolation of wellbores often require complex designs that is necessary to achieve high performance for the extreme conditions downhole. The manufacturing of cement for the oil and gas sector must be technically and economically compliant. However, there is an increasing concern about the global warming. Due to this, sustainability and CO_2 footprints becomes a major concern for the cementing systems for wellbore exploration.

Currently, cement production is responsible for 5% to 8% of yearly manmade CO_2 global emissions, or nearly 1.6 billion ton of CO_2 and therefore is the second largest CO_2 emitting industry behind power generation [1]. Worldwide cement production was about 2.77 billion metric tons in 2007 and Portland Cement is the most common type used [1]. It is found that for the production of one ton of Portland cement, 2.8 ton of raw materials is needed. The process generates 6000 - 14000 m^3 of dust containing air stream which contains between 0.7 to 800 g/m^3 of dust and account for about one ton of greenhouse gas CO_2 [2]. This is alarming since the world's demand for cement is increasing over the years. Geopolymer cements are proven to leave smaller carbon footprint compared to this. By

engaging on the idea of using geopolymer cement to replace well cement, we are one step closer in cutting the amount of greenhouse gases produced due to cement manufacturing.

The project will look into the compressive strength properties of the cement and aim to find the best mixture. By moving into geopolymer cement, it is hoped that the detrimental effect on the environment could be reduced.

2.2 Problem Statement

Portland Cement is used as a primary substance in well cementing. Upon further inspection, it is found that geopolymers are a better replacement to Portland Cement. Portland Cement is a hydraulic cement, which means that water is an integral part of its chemical structure[4]. There are several disadvantages of Portland Cement when it comes to its usage:

- Portland cement absorbs water and expands and contracts significantly with temperature changes
- Poor performance in salty environments
- Damaged by fire, lower heat resistant compared to geopolymer cement
- Substantial CO_2 emission during manufacturing (8% of CO_2 emission worldwide)
- Increased usage of non-renewable raw material
- Manufacturing of Portland cement produces harmful substance
- Manufacturing of Portland cement uses substantial amount of energy
- Portland cement is reactive to acid, bases and salt
- Takes a longer time for curing compared to geopolymer cement
- Cost effectiveness
- Portland cement is more permeable compared to geopolymer cement, a condition not favoured for well cementing

As stated above, the main concern is due to the characteristic of Portland Cement which is water based. Moreover, it would be more beneficial if a slurry with a reduced CO_2 footprint is used compared to Portland Cement. Geopolymers are found to have better properties than Portland Cement. However, the compressive strength of the MIRHA geopolymer cement must be confirmed first, before it could be further analyzed as a potential replacement for Portland Cement.

2.3 Objective and Scope of Study

The objectives of this study are:

- To understand the concept of geopolymer
- To study the advantages of geopolymer compared to Portland Cement
- To investigate the effect of concentration of NaOH on the compressive strength of the MIRHA geopolymer cement
- To investigate the effect of curing time on the compressive strength of the MIRHA geopolymer cement
- To investigate the effect of curing temperature on the compressive strength of the MIRHA geopolymer cement
- TO investigate the effect of size of MIRHA on the compressive strength of the MIRHA geopolymer cement

The scope of study includes:

- Conducting research on the theory and definition of terms related to the study.
- Conducting research in developing a laboratory procedure for conducting lab testing and experiment
- Coming up with a work plan that will accommodate the research
- Conducting research to figure out the data obtained from the experiments

2.4 Relevancy of Project

The project will look into the usage of MIRHA geopolymer cement as a viable replacement for well cement. The oil and gas sector is an industry which harvests energy from the environment. The method used to extract this energy source leaves a huge impact to the environment if not monitored carefully. Energy extraction should be done in a way to minimize any ill effect on Mother Nature.

As shown in the problem statement, the current usage of Ordinary Portland Cement poses several problems. Its manufacturing poses the biggest detrimental effect on the environment. As of now, the cement industry is one of the largest producers of CO_2 [6]. Thus this project proposes geopolymer cement to be used. Geopolymer cement has been known to possess superior properties when compared to Ordinary Portland Cement. These properties include high compressive strength, excellent strength gain rate, fire resistance, maintenance of structural properties at elevated temperature, chemical stability in highly acidic environment, relatively low cost and multitude of environmental benefits [21].

The cement will be composed from industrial wastes and more importantly it releases less greenhouse gases during its making. The student believes that geopolymer cement could be the future of cementing as it has more benefits compared to weaknesses. Given more research on this particular area, disposal of industrial wastes could be reused and managed efficiently resulting in better cost saving.

2.5 Feasibility of Project within Scope and Time Frame

The project will look into the compressive strengths of the geopolymers cement. Based on the schedule composed for the experiments to be carried out, it is found that the project is feasible. The materials needed for the project could be found in the campus. The nly concern for the project was the methodology and the arising problems in case the experiments ended in failures.

The first 3 months will be focusing on the experimental part of the project. A Gantt chart has been planned to help carry out the project. The student believes that the project is entirely feasible and all the objectives stated could be accomplished.

CHAPTER 3

LITERATURE REVIEW

3.0 Literature Review

The study is focused on the compressive strength of the geopolymer cement and its ability to act as a well cement replacing OPC. The geopolymer mentioned is derived from rice husk. It is noted that the properties of MIRHA geopolymer cement are better than Portland Cement. This literature review is made based on the references from 30 papers.

3.1 MIRHA Geopolymer Cement

Rice husk is an agro-waste material which is produced in millions of tons every year. Approximately, 20kg of rice husk are obtained for 100kg of rice [10]. Rice husk comprises of organic substance and 20% of inorganic substance [10]. Waste managers have found it to be difficult to dispose this agro-waste which is found in abundance. Rice husk ash (RHA) is obtained by the combustion of rice husk and is found to be super pozzolanic[8]. A pozzolan is a siliceous and aluminous material in which, in itself, possesses little or no cementitious value but which will react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties [8]. RHA is very rich in silicon dioxide which makes it very reactive with lime due to its non-crystalline silica content and its specific surface [8]. RHA is rich in silica and has about 85% to 90% silica content [8].

Geopolymerization is a general term used to decribe all the chemical processes that are involved in reacting aluminoslicates with aqueous alkaline solution so produce a new class of inorganic binder called geopolymers[6]. Geopolymers are members of the inorganic polymers family. The polymerisation process involves a substantially fast

chemical reaction under alkaline condition on Si-Al minerals resulting in a three dimensional polymeric chain and ring structure consisting Si-O-Al-O bonds. The chemical reaction may comprise the following steps[30]

- Dissolution of of Si and Al atoms from the source material through the actions of hydroxide ions
- Transportation or orientation or condensation of precursor ions into monomers
- Setting or polycondensation/polymerisation of monomers into polymeric structures

A geopoloymer can take one of the three basic forms[30]

- Poly(sialate), which has [-Si-O-Al-O-] as the repeating unit
- Poly(sialate-siloxo), which has[-Si-O-Al-O-Si-O-] as the repeating unit
- Poly (sialate-disiloxo), which has [-Si-O-Al-O-Si-O-Si-O-] as the repeating unit.

RHA is a suitable material to be used in the production of geopolymer cement due to its high silica content. Fly ash is commonly used as a source for geopolymer, however, this study will look into the effects of using RHA as a source for geoploymer. Geopolymer cement is known for its versatility which enables the product to be engineered from a range of component ratios so that is able to deliver specific properties at a lowered cost. The geopolymer cement system offers [8] :

- Variable densities from 1200 to 1900 kg/m^3
- Thickening times from several minutes to several hours
- Superior early and late strength development
- Fast gel strength development
- Controlled fluid loss
- Enhanced flexibility and elasticity
- Zonal isolation through strong bonding to formation and casing
- Ease of operation and handling
- Compatibility with most common cement admixtures and additives

- Significantly reduced CO_2 and water footprints
- Reduced energy used for manufacturing
- Cost savings

Geopolymer is a technology that must be looked into in order to come up with a green solution. Geopolymer cement are environmentally friendly and need moderate energy to be produced compared to Portland Cement [2]. CO_2 emission is reduced for about 80% if compared to the amount released during the manufacturing of Portland Cement [2]. Ordinary Portland Cement attains its strength through hydration reaction, which leads to the formation of C-S-H gel under the presence of water. Portland cement requires water for curing over a period of 28 days. Whereas, geopolymer gains its strength through rapid exothermic polymerization reaction and therefore requires curing temperatures[21]. Geopolymers gain 70% of its final strength in the first 3-4 hours and immobilizes 90% of the toxic materials within the matrices [2].

3.2 MIRHA Geopolymer cement studies

3.2.1 Mixing Type

Rattanasak et al[28] stated that separate mixing gave better strength mortar compared to normal mixing. Vaidya et al[21] stated that ash and sodium hudroxide should be mixed first. Then sodium silicate can be added in. For the experiment, sodium hydroxide and sodium silicate will be mixed first. Then, the rice husk ash will be added. The method used will be separate mixing.

3.2.2 Alkali Activator

Petermann et all [15] stated that there are two types of alkali activator used for geopolymerization, mainly sodium hydroxide and potassium hydroxide . Since K^+ is more basic, it allows a higher rate of solubilized polymerization and dissolution leading to a dense polycondensation reaction that provides greater overall network formation and an increase in the compressive strength of the

matrix. However, a study by Arjunan et. al [16] revealed that sodium hydroxide in low concentration was found to be the most effective chemical activator for low calcium fly ash. Thus, sodium hydroxide is used for this experiment.

Petermann et all [15] stated that sodium silicate is commonly used with sodium hydroxide as the alkali activator for geopolymerization. Palomo et. al.[20] that the use of alkali silicates such as sodium silicate and potassium silicate increases the polymerization reaction rate and improves the mechanical performance of the outcome geopolymer. Skvara et. al[17] suggests that the ration of sodium-silicate to sodium hydroxide solution (by mass) be set to an approximate value of 2.5.

3.2.3 Alkali Activator Molarity

The concentration of alkali activator is determined through further research. Rattanasak et al[28] used a molarity of 5M, 10M, and 15M. He found that 10M gave the best compressive strength for fly ash geopolymer cement. Khale et al[2] stated that an increase from 5M to 10M would generally increase the compressive strength of any geopolymer cement. Petermann et al[15] stated that the pH of the activation solution strongly influences the final cement performance. It is stated that the strength formend from samples with an alkali pH 14 were five time stronger compared to the samples from an alkali of pH 12. Petermann et all[15] concluded that a range of pH 13-14 is the most suitable for geopolymer formation.

3.2.4 Curing Temperature and Curing Time

Petermann et. al.[15] stated that temperature in the range of 50˚C - 80˚C are widely accepted values used for successful geopolymer hydration. Vaidya et. Al [21] states that geopolymer gains its strength through rapid exothermic polymerization reaction and therefore requires curing temperatures (typical curing period for

geopolymer is 60˚C for 24 hours. Rattanasak et. al [28] conducted curing temperature of 65˚C for 48 hours. Vaidya et. Al [21] showed that geopolymer gains 60% of their compressive strength within the first week. The geopolymer curing time was set within the week to monitor the increase in strength.

3.4 Geopolymer cement as well cement

One of the main function of well cement is to support the casing string. The shear strength of the cement holds the casing string once the cement is set. The most support for the casing string comes from the cement shear strength. In order to compare the geopolymer cement with the actual well cement, a threshold of compressive strength is set. If the geopolymer cement is unable to achieve this particular compressive strength, then it is unable to perform its function as a well cement.

Generally cement has a shear strength of approximately 1/12 of it s compressive strength [27]. For example a 1MPa compressive strength has a shear strength of 12 psi.

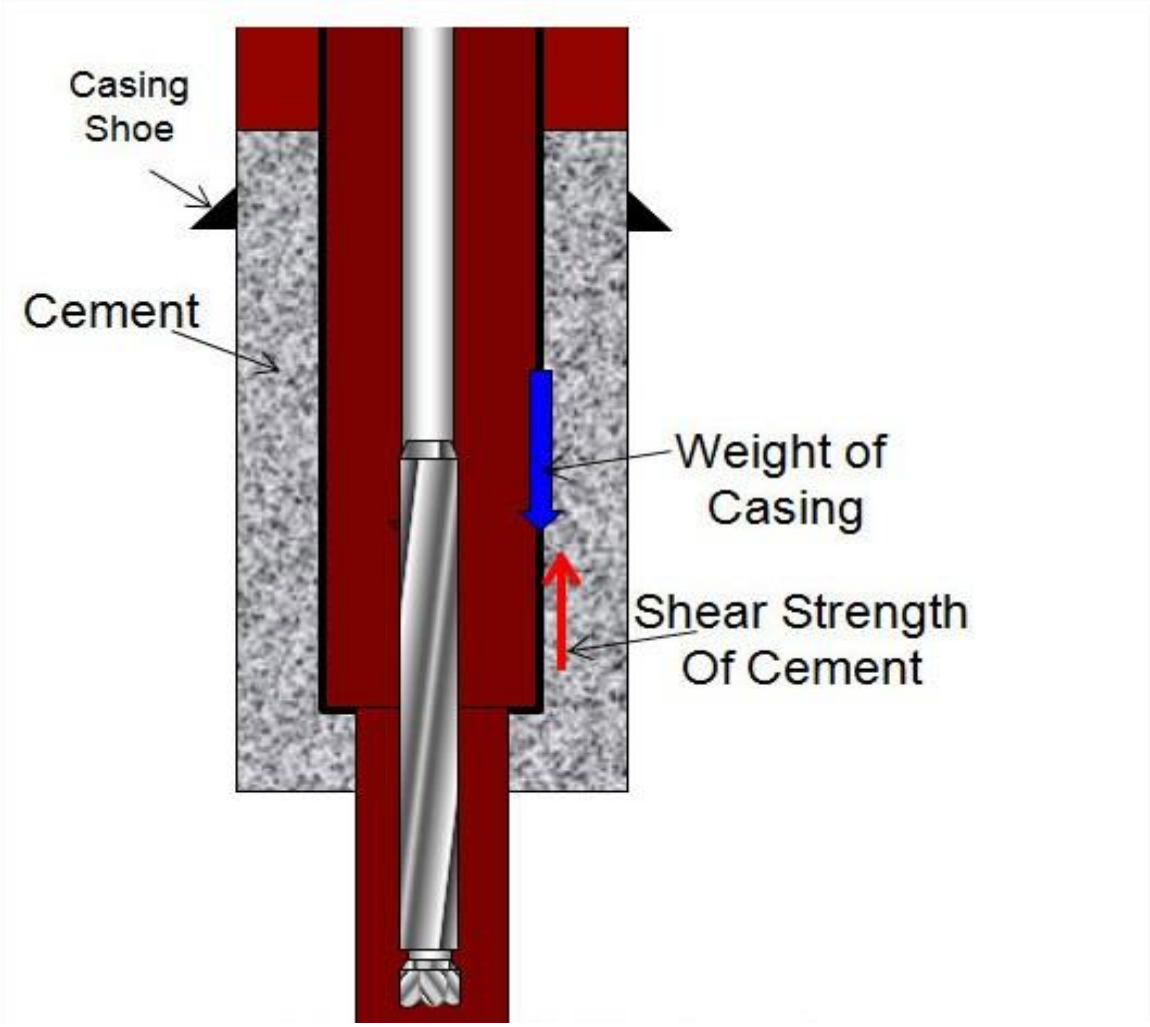

Figure 1: Cementing design and shear strength[27]

The following case study is taken based on a typical well completion design[27]. The information listed are as follows[27].

- Casing 9-5/8", 40 ppf (pound per foot), ID of casing = 8.835"
- Casing is set at 3,200'MD/3,000'TVD
- Top of cement at 600'MD/550'TVD
- Previous casing shoe (13-3/8") = 1000'MD/900TVD

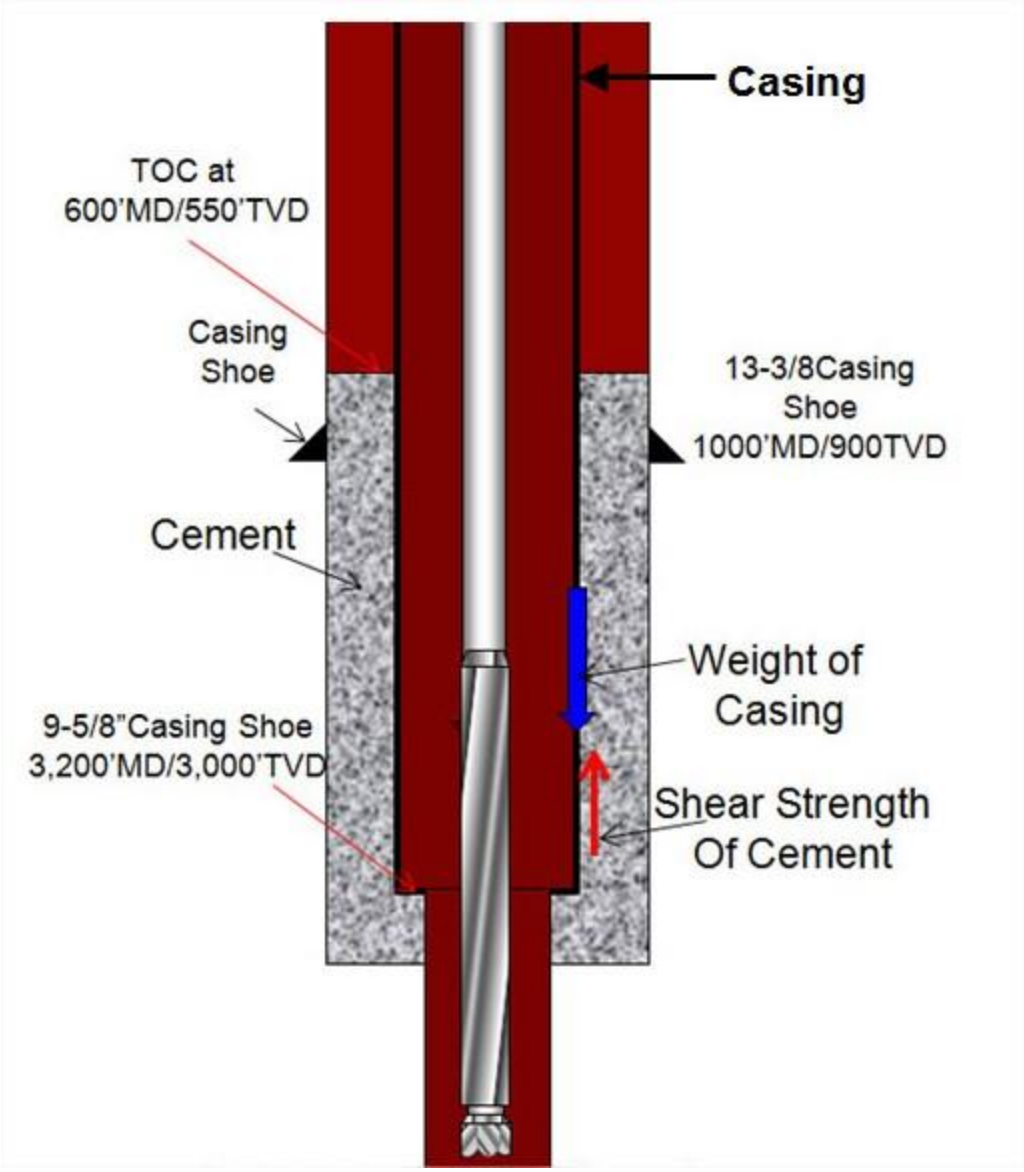

Figure 2 : Shear strength of well cement

The following calculation are employed to determine the minimum shear strength required to hold the casing in place. From the shear strength, we can calculate the compressive strength needed for a well cement [27]

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Surface area of casing = ∏ x 9.625" x 2600'

= <u>943,420 sq in.</u>

Casing weight = weight in ppf X length of casing
= 40 x 3200
=128000 lb

Shear strength = casing weight / area of casing covered by cement
= 128 000 / 943 420
= 0.136 psi

Compressive strength = 12 x 0.136
= 1.63 psi

This is a rough estimation without any consideration regarding complex load, thermal movement, etc [27]. The cement must be able to withstand the hydrostatic pressure and formation fluid pressure. The normal hydrostatic pressure is 0.433 psi/ft. Thus the pressure at the depths are as shown below

Table 2 : Pressure against depth(psi vs ft)

Pressure(psi)(MPa)	Depth(feet)
433(2.99)	1000
866(5.97)	2000
1299(8.96)	3000

Based on Table 3, we can conclude that a threshold of 3 MPa qualifies a geopolymer cement to perform its function as a well cement. At 3 MPa, the cement should be able to withstand a hydrostatic temperature up to 1000 ft and be able to hold the casing in place due to its high shear strength. This project aims to find the compressive strength of the MIRHA geopolymer cement. The purpose is to see whether MIRHA geopolymer can possess similar compressive strength as the normal well cement. However, other properties such as thickening time, hardening time and plastic deformation are not considered for this particular project.

3.5 Portland Cement

Portland Cement is a hydraulic product which is made by burning and grinding a mixture of calcereous and argillaceous materials such as limestone and clay. Portland Cement is widely known for its uses in the construction sector. However, it is found that Portland Cement is widely used in the oil and gas sector as well. Portland Cement is used in cementing operations which help to seal annulus between the wall of the wellbore and the casing, to provide zonal isolation, to protect the casing against aggressive wellbore fluids and to protect the casing against collapse by rock creeping in on the wellbore[4]. In Malaysia, class G cement is used widely in cementing the oil well[13]. Class G cement is an imported cement, its components are Tricalcium Silicate(C_3S), Dicalcium Silicate (C_2S) and Gypsum (CSH_2)[13]. This cement has low hydration rate and forms tight bond between the pebbles and the casing[13]. It can be used up to a depth of 2440m and temperature between 80 - 200˚F[13].

The main concern behind the usage of Portland Cement is its effect on the environment. It is found that Portland Cement manufacturing requires the usage of a huge amount of energy. Manufacturing of Portland Cement releases a large amount of CO_2. Cement manufacture is energy intensive [5]. Production of one ton of Portland cement typically requires 4200 MJ of thermal energy and 110 kWh of electric energy [5]. Modern cement plants might be more energy efficient, whereas older plants tends to use of higher amount of energy. The main source of CO_2 comes from the decarbonation of limestone which is the main raw material used to produce cement [5]. The remainder of the CO_2 comes from combusting the fuel required to drive the reactions necessary to make the clinker [5]. Cement manufacture requires high flame temperature (+1800˚C) in order to sinter and fuse the raw materials to the clinker compounds at 1450˚C [5]. Worldwide cement production is responsible for 8% of yearly man made global CO_2 emission, nearly 1.6 billion tonnes [1]. Worldwide cement production was about 2.77 billion metric tonnes in 2007 and Portland Cement is the most common type [1]. Portland Cement clinker manufacturing produces around 0;9 tons of CO_2 per ton of Portland Cement[1].

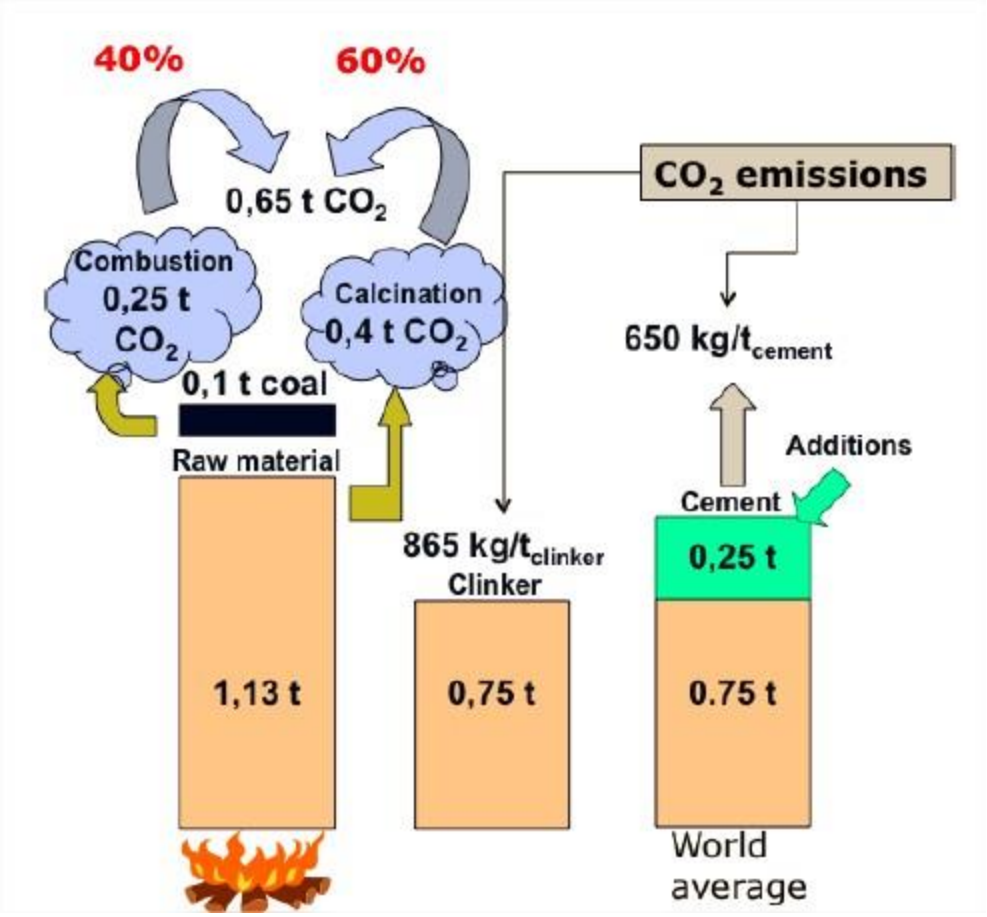

Figure 3 : Typical CO_2 emissions in the cement manufacturing process[5]

Thus, it can be observed that geopolymer cement is more nature friendly compared to OPC. An increased usage of geopolymer cement as a well cement would certainly cut down the detrimental effects to the environment.

CHAPTER 4

METHODOLOGY

4.1 Research Methodology

Figure 4: Flow of final year project

4.2 Project Activites

Table 3 : Project activities tabulation

Methodology	**Activities**
Project scope validation	□ Confirmation of project title with supervisor □ Problem statement identification □ Scope of study identification
Project introduction	□ Understanding the principle behind geopolymer □ Understanding the different types and factors that contribute to the compressive strength of the geopolymer
Identifying and selection experiment	□ Feasibilities study on each of the factor affecting geopolymer compressive strength □ Finalize on the factors to be tested
Experiment	□ Designed experiments to test the factors listed. □ Repeat experiment and find the best alternative in gaining the maximum compressive strength
Analysis of data	□ Analyze the data obtained □ Compare the data and come up with reasoning to explain the data
Conclusion and recommendation	□ Come up with a conclusion for the project and list down future recommendations.

4.3 Key Milestone

Table 4. Key Milestone for Project

Week	Objectives
FYP I	
5	Completion of preliminary research work
6	Submission of extended proposal
9	Completion of proposal defence
12	Confirmation on lab material and equipment for conducting experiment
13	Submission of Interim draft report
14	Submission of Interim report
FYP II	
5	Finalized the experiment procedure
6	Conducting experiment
7	Result analysis and discussion
8	Submission of progress report
9	Preparation for Pre-SEDEX
11	Pre-SEDEX
12	Submission of draft report
13	Submission of technical paper and dissertation
14	Oral presentation
15	Submission of project dissertation

4.3 Gantt Chart

Table 5 : Proposed Gantt chart for the project implementation for both FYP I and FYP II.

PROJECT ACTIVITIES	WEEKS													
	Final Year Project 1													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Project Scope Validation														
Project Introduction														
Submission of Extended Proposal														
Identify material and equipment														
Training on how to conduct experiment														
Proposal Defense														
Detailed Study														
Submission of Interim Draft Report														
Finalized Procedure														
Conducting Experiment														
Result analysis and discussion														
Submission of progress report														
Preparation for Pre-SEDEX														
Pre-SEDEX														
Submission of draft report														
Submission of technical paper and dissertation														
Oral presentation														
Submission of project dissertation														

Final Year Project 2													
1	2	3	4	5	6	7	8	9	10	11	12	13	14

As per shown in the proposed Gantt chart, the project is feasible given the time to complete it.

4.4 Project Activities/ Research Methodology

For this project, several lab testing will be conducted in the future. For now, the experiment procedures has been devised. Once this report has been approved, preparation of MIRHA geopolymer cement will take place. The lab testing or its compressive strength and its acid resistivity will follow. This project must be completed within a time period of 16 weeks. It is important that the experiments for this particular project to be of high efficiency. The objectives of this study is as stated in **1.3**.

- To understand the concept of geopolymer
- To study the advantages of geopolymer compared to Portland Cement
- To investigate the effect of concentration of NaOH on the mechanical properties of the MIRHA geopolymer cement
- To investigate the effect of curing time on the compressive strength of the MIRHA geopolymer cement
- To investigate the effect of curing temperature on the compressive strength of the MIRHA geopolymer cement
- TO investigate the effect of size of MIRHA on the compressive strength of the MIRHA geopolymer cement

The first objective is achieved since the extensive research was done in this particular field for the project. The next step to be taken is to design an efficient experimental procedure in order to achieve the remaining objectives. Due to the time restraint in this project, the experiment will aim find out the compressive strength of the MIRHA geopolymer cement only.

The MIRHA will be obtained from the combustion of rice husk. MIRHA is known to contain substantial amount of silica. If it is properly produced, the ash will contain 95% pure silica in an active form which behaves like a cement. It is found that concrete prepared from MIRHA which was burned at 800˚C had a higher compressive strength [25]. Thus, it is decided that the rice husk will be combusted at 800˚C for a period of 45 minutes. The MIRHA will then be grinded until its particles are in the range of size 300μm [25].

Alkali activator types, concentration of activators, ratios of solids to liquid, curing time and curing temperature are all relevant in the characterization of geopolymer cements and the mechanical properties attained. In this project we are unable to test all of the aforementioned variables due to the time constraint. It has been decide that the manipulated variable for the experiment will be the concentration of the alkali activator, ratio of ash/alkali activator, curing temperature as well as the curing time. These factors will be varied and the effect on the compressive strength of the MIRHA geopolymer will be recorded.

The experiment focuses on creating a MIRHA geopolymer cement. This means that OPC will not be added in. The cement created would be formed from 100% Microwave Incinerated Rice Husk Ash (MIRHA). MIRHA combined with the specific alkali activator would result in the formation of MIRHA geopolymer. There have been many experiments that have focused on using fly ash as the main material for the geopolymer cement. However as stated in the literature review, MIRHA is a super pozzolan and might possibly be more efficient than fly ash.

The most common activator are sodium hydroxide and potassium hydroxide [15] . Since K^+ is more basic, it allows a higher rate of solubilized polymerization and dissolution leading to a dense polycondensation reaction that provides greater overall network formation and an increase in the compressive strength of the matrix. However, a study by Arjunan et. al [16] revealed that sodium hydroxide in low concentration was found to be the most effective chemical activator for low calcium fly ash. Regardless of the type of alkali selected, the concentration of the alkali plays a bigger role. It is found that a range of 8M-12M NaOH gives out the best compressive strength[2] . For the experiment, two different concentration of NaOH will be used which are 10M and 15M. The pH level of the activating solution strongly influences the final cement performance [15]. It is reported that the strength measured from samples of pH 14 were five times greated than samples from pastes of pH 12; and it was concluded that a pH range of 13-14 was most suitable for the formation of geopolymers with a higher mechanical strength [2]. Since the properties of MIRHA is similar to fly ash, it is agreed that the alkali activator for the project is sodium hydroxide (NaOH). Sodium silicate, waterglass is commonly mixed with NaOH as a fortifying agent to enhance alkalinity and increase overall specimen strength. The most

common alkaline liquid used in geopolymerization is a combination of sodium hyroxide and sodium silicate [15]. A study conducted by Palomo et. Al[20] on using alkali silicates in addition to alkaline hydroxides to activate the source material concluded that the use of alkali silicates such as sodium silicate and potassium silicate increases the polymerization reaction rate and improves the mechanical performance of the outcome geopolymer[20].For this experiment, the alkali activator will be a combination of sodium hydroxide(NaOH) and sodium silicate(Na_2CO_3). Laboratory experience suggests that the ration of sodium-silicate to sodium hydroxide solution (by mass) be set to an approximate value of 2.5[17].

The ratio of pozzolanic material to a selected activator affects several critical properties of the geopolymer basis [15]. Overall strength is greatly affected by this variable. The alkaline liquid to ash ratio (by mass) is recommended to be maintained in the range of 0.30 to 0.45[18]. The ash to activator ratio appeared to be the most critical parameter regarding the general strength and fire resistance of the geopolymer paste [15]. MIRHA has a lesser density compared to fly ash. Due to this, MIRHA has a larger volume compared to the normal fly ash. This makes it a bit difficult for it to be mixed. Thus it is agreed that the ratios of ash to alkali activator will be varied. This would help determine which ratio is more suited for MIRHA geopolymer cement. The ratio used will be 1:1 and 2:1.

The biggest challenge in successful geopolymer cement production is obtaining the proper ambient temperature for curing. Geopolymer reaction takes place more easily with the addition of an external heat source to promote alkaline reactivity in pozzolanic material. It is found that the compressive strength of the geopolymer could go up to 50MPa for specimens cured at temperature ranging from 8C to 25C and relative humidity of 40%-60% [19]. Elevated temperature curing greatly affect the mechanical development of geopolymer binders. Temperature in the range of 50˚C - 80˚C are widely accepted values used for successful geopolymer hydration [15].Both curing temperature and curing time directly influence final compressive strength values of geopolymer specimens. Geopolymer gains its strength through rapid exothermic polymerization reaction and therefore requires curing temperatures (typical curing period for geopolymer is 60˚C for 24 hours [21].For the project, the MIRHA geopolymer cement will be cured at 60˚C for 24 hours followed by room temperature curing. In order to investigate the significance of the curing temperature, another batch of geopolymer cement will be cured at 100˚C for the

first 24 hours. This would allow us to map the effect tof curing temperature on the geopolymer cement.

For this particular project, the effect of concentration of alkali activator and curing time on the MIRHA geopolymer properties will be the focus. The alkali activator for this project is a combination of sodium hydroxide (NaOH) and sodium silicate,waterglass(Na_2CO_3). The concentration of the sodium hydroxide will vary to find its effect on the MIRHA geopolymer properties. The concentration that will be used in 10M and 15M. The ratio of silicate,waterglass(Na_2CO_3 to sodium hydroxide (NaOH) will be maintained at 2.5:1.

Another manipulating variable is the curing time. The test will be repeated with different curing time at room temperature. The curing time for room temperature is set to be 3 day, 5 days and 7 days. For the curing method, the project employs external exposure curing. In external exposure curing, the geopolymer cement will be placed in a transparent chamber outside the building. The heat radiation from sunlight will penetrate the chamber. The samples will be protected from rain water [24]. It is found that external exposure curing results in a higher compressive strength [24]. The MIRHA geopolymer cement will then be tested for its compressive strength and it shall be recorded.

4.5 Tools and Testing Procedures

For the experiment, there are a few tools that were needed. The tools needed are listed below;

Materials

1. Microwave Incinerated Rice Husk Ash
2. Sodium Hydroxide Solution (10M)
3. Sodium Silicate granules
4. Cement Mold

Tools

Table 6 : Tools used in the experiment and its description

Tools	Images	Function
Microwave Incinerator		- Used to incinerate the Riche Husk ash. - The rice husk ash is incinerated at 800˚C for a day.
LA Abrasion Machine		- Used to grind the incinerated rice husk ash - Grinding takes around 12 hours for 50kg MIRHA.
Cement Mould 50mm X 50mm X 50mm		- The mould holds the cement until it dries up. - Cement is retained in mould for a day.

Sieving Machine		- The grinded MIRHA is sieved to 300µm.
Constant Speed Mixer		- Used to mix the the rice husk ash with sodium hydroxide and sodium silicate to form geopolymer cement paste.
Compressive Strength Tester		- To test the compressive strength of the cement.

Preparation of MIRHA

1. The Rice Husk obtained is dried under the sunlight for 3-4 hours to reduce the moisture content.
2. The rice husk ash was incinerated in the UTP Microwave Incinerator (UTPMI) at 800˚C for 1 day.
3. The MIRHA was grinded using a Los Angeles abrasion machine until the particles are in a range of size 300µm.

MIRHA Geopolymer Cement Procedures

1. A suitable amount of MIRHA that would fill 3x50mm^{3}cement moulds was prepared.
2. A 0.5 parts of 10M NaOH solution was prepared with the MIRHA weight as reference.
3. A2.5 parts of sodium silicate to the mass of the NaOH solution was prepared.
4. The MIRHA was mixed with NaOH for 10 minutes at 120rpm.
5. The sodium silicate was added into the mixture and the mixture were mixed for another 1 minute at 120rpm.
6. The slurry is inserted into the cement moulds.
7. The cement were cured at 60˚C for 24 hours
8. The batches of cement were cured for a different range of time,3 day, 5 days and 7 days. External exposure curing was used.

Compressive Strength Testing Procedures

1. The cube was placed in compressive testing machine and load was applied uniformly.
2. The load at which the cube fails is noted.
3. The procedure is repeated with the remaining 2 cubes.
4. 3 specimens were tested and its average were taken as its final compressive strength.

Calculations

$$f_{ci} = \frac{F_i}{A_{ci}}$$

Where;

f_{ci} = the compressive strength, N/mm^2

F_i = the maximum load, N

A_{ci} = the cross-section area at which load is applied, mm^2

Figure 5 : Compressive Strength Tester[22]

Result and Data Tabulation

Compressive Strength Test

Table 7 : Example of compressive strength calculation

Cement Sample	**Cement Curing Time(d)**	$\mathbf{f_{ci}}$ (Compressive strength, MPa)	$\frac{\mathbf{Fcia + Fci}\boldsymbol{b} \mathbf{+ Fci}\boldsymbol{c}}{\mathbf{3}}$ (MPa)
A	3		
B	5		
C	7		

CHAPTER 5

RESULTS AND DISCUSSION

5.1 Experimentation Design

The major problem encountered with the MIRHA geopolymer cement is its tendency to clump and form hard cement and its inability to mix with the portion of alkali activator. Based on the review on geopolymer cement made using fly ash and other materials, it is found that MIRHA reacts faster and needs an additional amount of water for it to be able to mix together and form a cement. Several experiments were conducted to achieve the project's objectives. However, additional experiments were conducted to find the necessary parameters, such as water ratio in order to proceed. The list of experiment conducted are as follows;

- The first experiment focusses on investigating the best water ratio needed for the geopolymer cement.
- The second experiment focusses on the difference of cement compressive strength when the ratio of the ash to alkali activator is changed.
- The third experiment was done to find the effect of sodium hydroxide molarity on the strength of the cement
- The fourth experiment was conducted to effect of grain size on the cement strength
- The fifth experiment was conducted to find the effect of curing temperature on the cement's compressive strength

Experiment 1

As stated above, it is impossible to form MIRHA geopolymer cement with the alkali activator alone. A certain amount of water needs to be added in order for the cement to be able to form a slurry. The first experiment is focused on

- The ratio of water in the mixture with respect to the MIRHA mass.
- The ratio of alkali activator to the MIRHA mass

Table 8 : Samples tested for Experiment 1 with different water ratio

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M,(g)	Distilled Water, (g)
A	109	30	20	40
B	109	27	27	60
C	109	39	16	80

The purpose of the experiment is to look at the ratio of liquid content in the mixture that would give the highest compressive strength. The ratio of alkali activator was varied from 1:1 to 1:2.5. The total liquid content of the slurry also varies due to this. From here, we can find the best amount of water ratio to ash mass to be used in the experiment. The sodium hydroxide to sodium silicate ratio will be maintained at 1:2.5 as per said in the literature review.

Experiment 2

The second experiment is focused on finding the effect of the ratio of MIRHA:alkali activator on the strength of the cement. From experiment 1, it was found that a water ratio of 40% to MIRHA mass gave the best strength. Thus the water ratio is used for all of the experiments from here onwards. Two ratios were investigated in this experiment. The first ratio is 1:1. The second ratio is 2:1.

Table 9 : Sample ratio for experiment 2

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M/15M,(g)	Distilled Water, (g)
A	200	57.14	142.85	80
B	200	71.43	28.57	80

The two ratios above were tested with different molarity and curing days. The molarity of sodium hydroxide used were 10M and 15M. The compressive strength acquired would reveal the best ratio to be used.

Experiment 3

The third experiment focuses on finding the effect of Sodium Hydroxide concentration on the compressive strength of the geopolymer cement. The experiment is a continuation from experiment 2. The different ratios of cement are tested with different molarity of sodium hydroxide.

MIRHA : Alkali Activator = 1 : 1

Table 10 : 1:1 Sample ratio for experiment 3

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, (g)	Distilled Water, (g)
A	200	57.14	142.85	80

Table 11 : Manipulated variable for 1:1 ratio

Curing Time / Molarity	Compressive strength at 3 days(MPa)	Compressive strength at 5 days(MPa)	Compressive strength at 7 days(MPa)
10 M Sodium Hydroxide			
15 M Sodium Hydroxide			

MIRHA : Alkali Activator = 2 : 1

Table 12 : 2:1 Sample ratio for experiment 3

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, (g)	Distilled Water, (g)
A	200	71.43	28.57	80

Table 13 : Manipulated variable for 2:1 ratio

Curing Time / Molarity	Compressive strength at 3 days(MPa)	Compressive strength at 5 days(MPa)	Compressive strength at 7 days(MPa)
10 M Sodium Hydroxide			
15 M Sodium Hydroxide			

From this experiment, we will be able to tell the best NaOH molarity for MIRHA geopolymer cement.

Experiment 4

From experiment 3, we are able to conclude two things.

- The best ratio for MIRHA : alkali activator is 2:1
- The best molarity for MIRHA geopolymer cement is 10M

The next set of experiment is conducted with these two results in mind. The 4th experiment investigates the effect of grain size on the compressive strength of the MIRHA geopolymer cement. The sizes investigated are 300µm and 600µm. The ratios used are as follows. A curing temperature of 60˚C was used for a period of 1 day followed by external exposure curing.

Table 14 : Sample ratio for Experiment 4

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M (g)	Distilled Water, (g)
300µm	200	71.43	28.57	80
600µm	200	71.43	28.57	80

Experiment 5

For this part of the experiment, the curing temperature was investigated. For all of the cement before, a curing temperature of 60˚C was used for a period of 1 day. The experiment was repeated with different curing temperatures. The curing temperatures used are 60˚C and 100˚C. The cements were cured at these temperatures for 1 day.

Table 15 : Sample ratio for Experiment 5

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M (g)	Distilled Water, (g)
60˚C	200	71.43	28.57	80
100˚C	200	71.43	28.57	80

The ratio used were 2:1. The molarity of sodium hydroxide was 10M.

5.2 Experimentation Methods

The steps that were taken throughout the entire experiment is shown below. It is arranged chronologically in order to ease understanding.

Preparation

The first part of the entire experiment is to prepare the MIRHA for the experiment. The first part comprises of the following steps.

- Rice Husks were incinerated using the UTP Microwave Incinerator at 800˚C for 24 hours.
- The incinerated rice husk is grinded using the LA Abrasion Machine. The entire process takes 8 hours to complete in order to ensure thorough grinding.
- The grinded MIRHA is then sieved to 300 micron.

Cement Ratios

The second part of the process was coming up with the mixture for the geopolymer cement. The experiment was conducted with different ratios of ash to alkaline activator in order to determine the best ratio. The amount of water was also varied in order to determine the proper amount of additional water to be added. The experiment was done by manipulating the amount of alkaline activator and the amount of water with respect to the mass of MIRHA. The steps for the second part of the process are as follows

- Cement was prepared using the ration of MIRHA/alkali activator of 1:1 and 2:1.
- The amount of sodium hydroxide to sodium silicate is varied as well
- The amount of water to be added in is varied
- The ratio of materials used for the cement could be seen in the table below.

Ratios of material for experiment

Curing Method

3 cubes were made for each sample. They were cured in an oven at 60˚C for a period of 24 hours. The compressive strength of the cubes were tested after the given time. For the 5^{th} experiment, a batch of the cement were cured at at 100˚C for a period of 24 hours.

The cements were cured for 3 days, 5 days and 7 days. The cements were cured at room temperature after it has been taken out of the oven.

Compressive Strength Test

The cubes were tested for their compressive strength using the compressive strength tester. Three cubes were tested and the result is the average of the three cube's compressive strength.

5.3 Findings/Data Gathering

It is important that a medium of comparison was found in order for us to evaluate the strength of the cement compared to the other well cements that are used in the industry. The project looks at the idea of using geopolymer cement as a well cement. The threshold stated in the literature review at **2.2** is at **3MPa.** As shown, there is a minimum compressive strength that a cement must possess after a certain amount of curing time in order for it to qualify as a well cement. Thus the geopolymer cement must pass this particular compressive strength.

The data acquired are arranged as per the experiments done. The experiments are done stage by stage. The result from the first experiment will lead to the ratios used in the second experiment. The data from the experiments are as shown below.

Experiment 1

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M,(g)	Distilled Water, (g)
40%	109	30	20	40
60%	109	27	27	60
80%	109	39	16	80

Results

Table 16 : Results for Experiment 1

Sample	Trial 1,(MPa)	Trial 2,(MPa)	Trial 3,(MPa)	Compressive Strength,(MPa)
40%	0.67	0.72	0.73	0.7
60%	0.52	0.47	0.5	0.49
80%	0.32	0.35	0.28	0.32

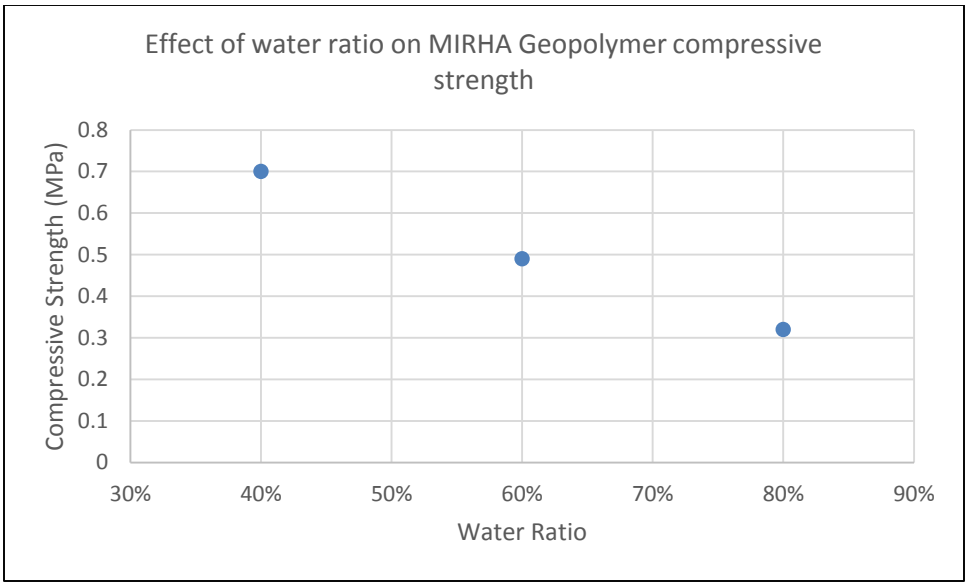

Figure 6: Effect of water ration on MIRHA geopolymer compressive strength

From the experiment, it is found that the amount of water directly affects the compressive strength of the geopolymer cement. From the first experiment it is concluded that the ratio of water should be maintained at 40% of the MIRHA mass. This enables it to achieve a better compressive strength.

Thus it can be concluded that the water ratio for MIRHA geopolymer cement should not exceed 40% of the MIRHA mass in order to achieve a high compressive strength.

*In the experiment, it was found that the cement cubes did not fill up the mold as it should have been. This could be attributed due to a low amount of ash used. For the next few experiments, a MIRHA mass of 200g will be used.

Experiment 2 & 3

In this experiment, different ratios of MIRHA to alkali activator was tested. The samples were subjected to different sodium hydroxide concentrations as well. The ratios are as shown in the table below.

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M/15M,(g)	Distilled Water, (g)
A	200	57.14	142.85	80
B	200	71.43	28.57	80

MIRHA : Alkali Activator = 1 : 1

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, (g)	Distilled Water, (g)
A	200	57.14	142.85	80

Table 17 : Results for 1:1 ratio

Curing Time / Molarity	Compressive strength at 3 days(MPa)	Compressive strength at 5 days(MPa)	Compressive strength at 7 days(MPa)
10 M Sodium Hydroxide	1.2	2.45	4.4
15 M Sodium Hydroxide	0.9	2.2	3.57

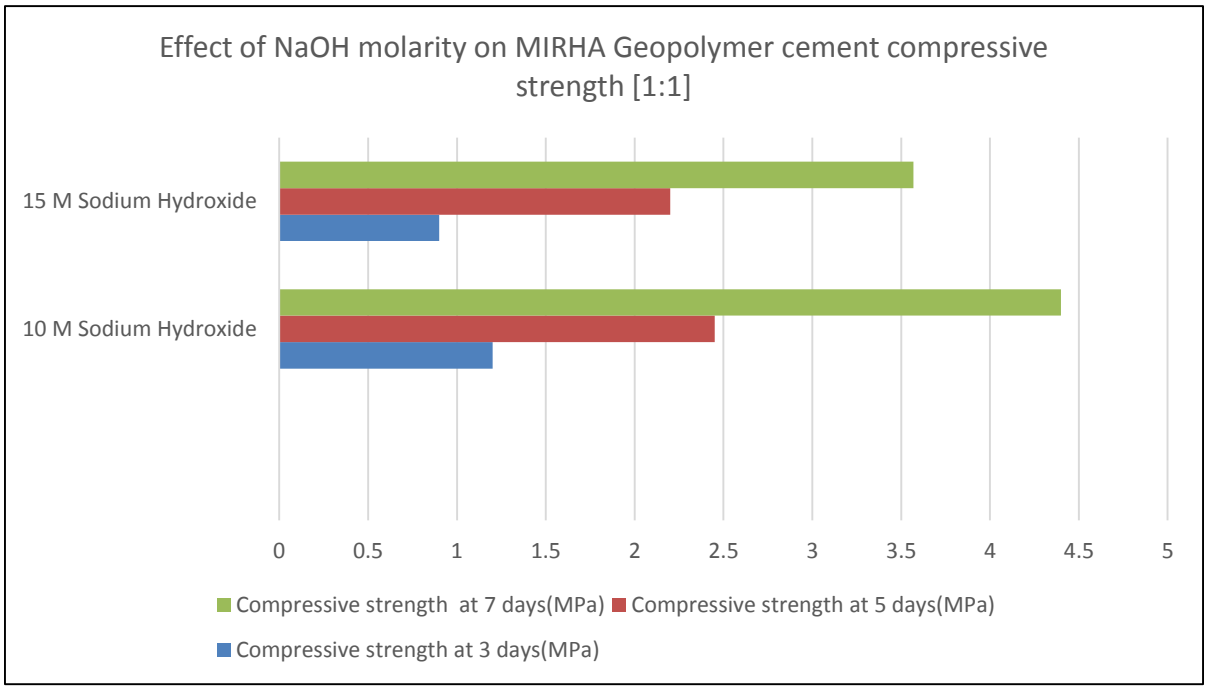

Figure 7 : Effect of NaOH molarity on MIRHA geopolymer compressive strength [1:1]

The overall compressive strength of the 15M NaOH cement is lower than the 10M NaOH cement. The highest compressive strength recorded is 4.4MPa @10M NaOH (7 days). The lowest compressive strength recorded is 0.9MPa@15M NaOH (1 day). The compressive strength for the 10M NaOH showed a 267% increase in compressive strength from day 1 to day 7. The compressive strength for the 15M NaOH showed a 297% increase in compressive strength from day 1 to day 7.

MIRHA : Alkali Activator = 2 : 1

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, (g)	Distilled Water, (g)
A	200	71.43	28.57	80

Table 18 : Results for 2:1 ratio

Curing Time / Molarity	Compressive strength at 3 days(MPa)	Compressive strength at 5 days(MPa)	Compressive strength at 7 days(MPa)
10 M Sodium Hydroxide	4.1	4.3	4.82
15 M Sodium Hydroxide	3.2	3.8	3.96

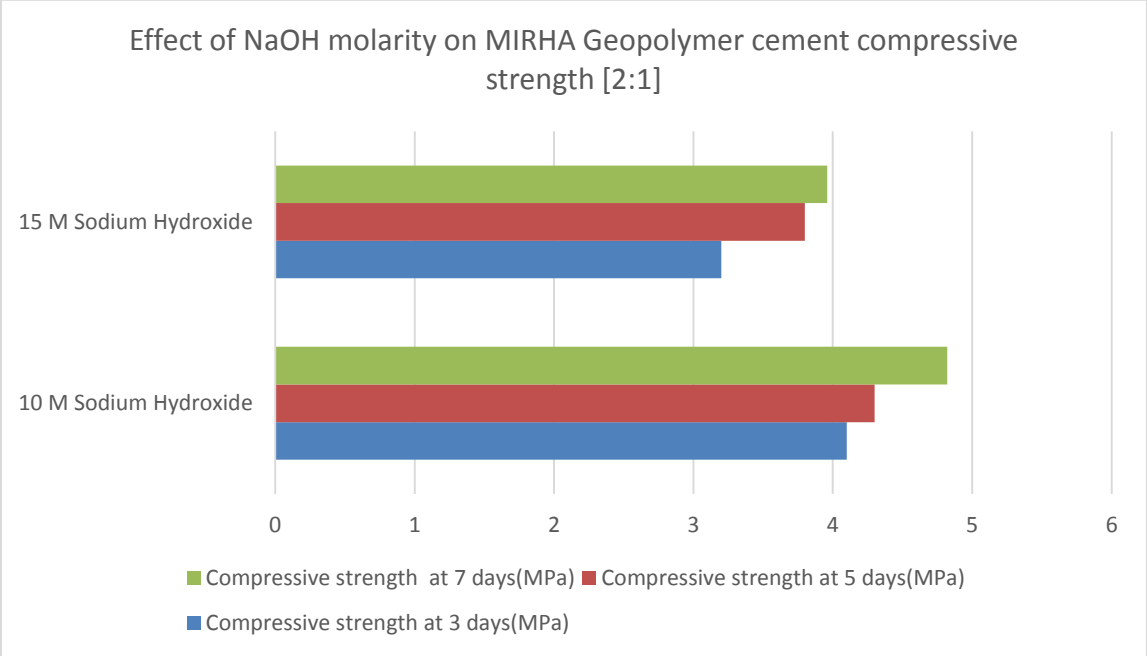

Figure 8 : Effect of NaOH molarity on MIRHA geopolymer compressive strength [2:1]

The overall compressive strength of the 15M NaOH cement is lower than the 10M NaOH cement. The highest compressive strength recorded is 4.82MPa @10M NaOH (7 days). The lowest compressive strength recorded is 3.2MPa@15M NaOH (1 day). The compressive strength for the 10M NaOH showed a 17% increase in compressive strength from day 1 to day 7. The compressive strength for the 15M NaOH showed a 24% increase in compressive strength from day 1 to day 7.

From the Figure 8, we can conclude that a 10M NaOH results in a higher compressive strength for the MIRHA geopolymer cement.

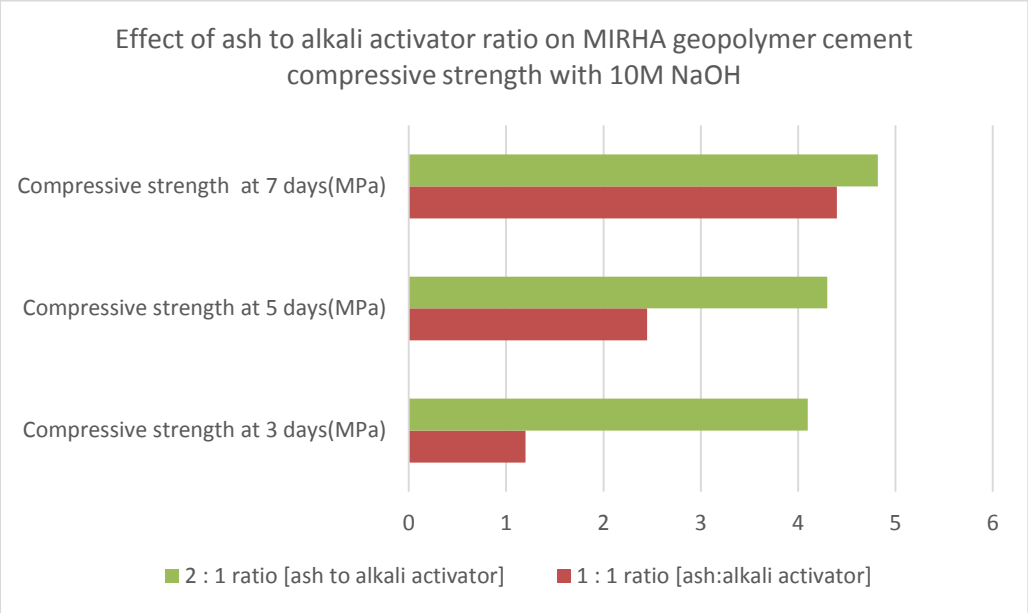

Figure 9 : Effect of ash to alkali activator ratio on MIRHA geopolymer cement compressive strength with 10M NaOH

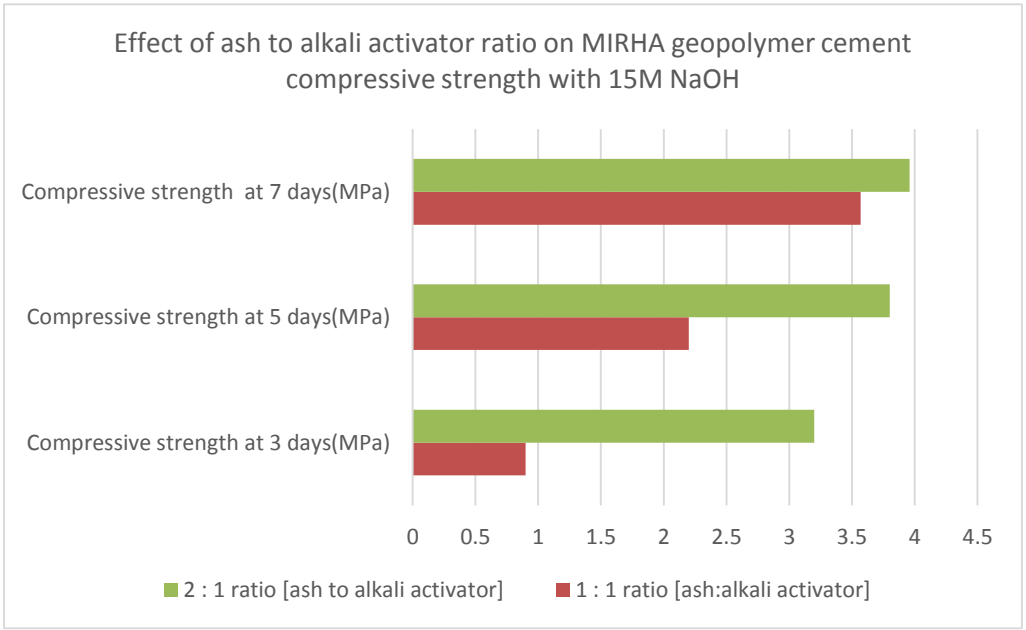

Figure 10 : Effect of ash to alkali activator ratio on MIRHA geopolymer cement compressive strength with 15M NaOH

The bar chart above proves that the ash to alkali activator ratio of 2:1 results in a higher compressive strength compared to the 1:1 ratio.

Thus, it can be concluded, that an ash to alkali activator ratio of 2:1 results in a better compressive strength.

Experiment 4

In this experiment, the grain size of the MIRHA was manipulated. The amount of water used is limited at 40% of ashes mass as recommended from the results of Experiment 1.The molarity of NaOH used is 10M since it has been determined from Experiment 2 that 10M NaOH gives out a higher compressive strength. The ash to alkali activator ratio used is 2:1 in concurrent to the result obtained from Experiment 3.

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M (g)	Distilled Water, (g)
300µm	200	71.43	28.57	80
600µm	200	71.43	28.57	80

Table 19 : Results for Experiment 4

Curing Time / Sample	Compressive strength at 3 days(MPa)	Compressive strength at 5 days(MPa)	Compressive strength at 7 days(MPa)
300µm	4.1	4.3	4.82
600µm	3.06	3.7	3.84

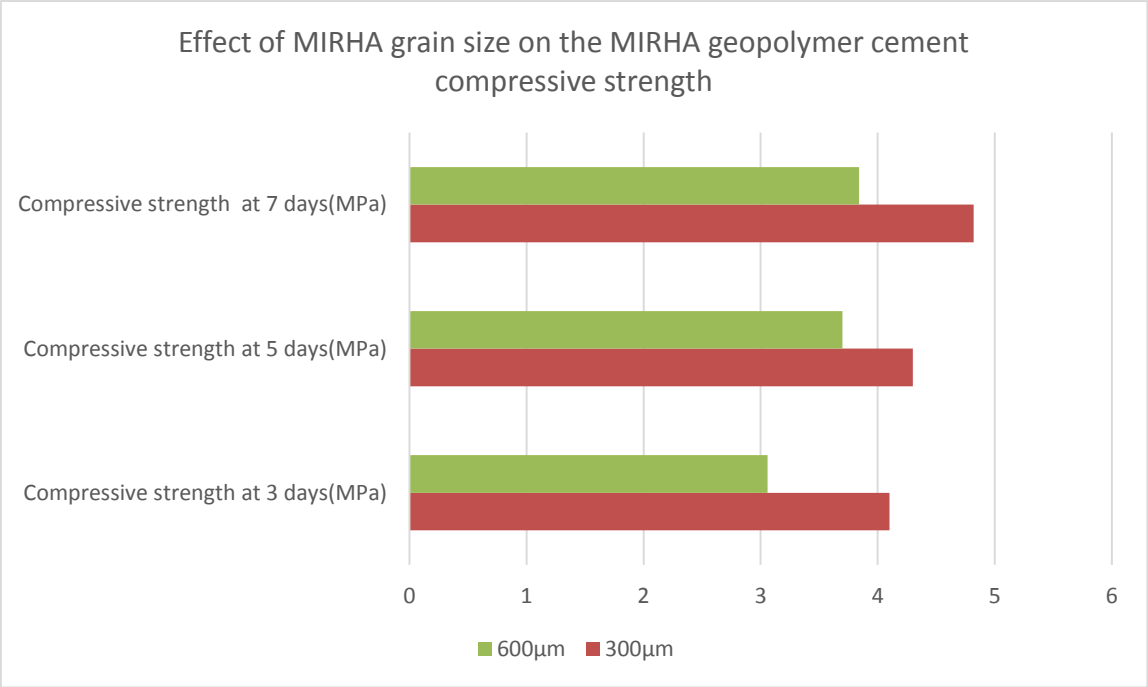

Figure 11 : Effect of MIRHA grain size on the MIRHA geopolymer cement compressive strength

The highest compressive strength recorded is from the 300µm at 7 day curing time. The overall compressive strength of the 600µm cement is lower than the 10M NaOH cement. The highest compressive strength recorded is 4.82MPa@300µm(7 days). The lowest compressive strength recorded is 3.06MPa@600µm (1 day). The compressive strength for the 300µm showed a 17% increase in compressive strength from day 1 to day 7. The compressive strength for the 15M NaOH showed a 25.5% increase in compressive strength from day 1 to day 7.

From Figure 11, it can be concluded that a 300µm grain size results in a higher compressive strength than the 600µm grain size cement.

Experiment 5

For this part of the experiment, the curing temperature was investigated. The experiment was repeated with different curing temperatures. The curing temperatures used are 60˚C and 100˚C. The cements were cured at these temperatures for 1 day. The amount of water used is limited at 40% of ashes mass as recommended from the results of Experiment 1.The molarity of NaOH used is 10M since it has been determined from Experiment 2 that 10M NaOH gives out a higher compressive strength. The ash to alkali activator ratio used is 2:1 in concurrent to the result obtained from Experiment 3. The grain size used for the experiment is set at 300µm as per shown in Experiment 4.

Sample	MIRHA (g)	Sodium Silicate, (g)	Sodium Hydroxide Solution, 10M (g)	Distilled Water, (g)
60˚C	200	71.43	28.57	80
100˚C	200	71.43	28.57	80

Table 20 : Results for Experiment 5

Curing Time / Sample	Compressive strength at 3 days(MPa)	Compressive strength at 5 days(MPa)	Compressive strength at 7 days(MPa)
60˚C	4.1	4.3	4.82
100˚C	1.1	1.5	2.1

Table 21 : Physical image of Experiment 5 results

Curing Temperature	Physical Appearance
60˚C curing temperature - The cubes are formed according to the mould	 

100˚C curing temperature - The cubes are deformed	

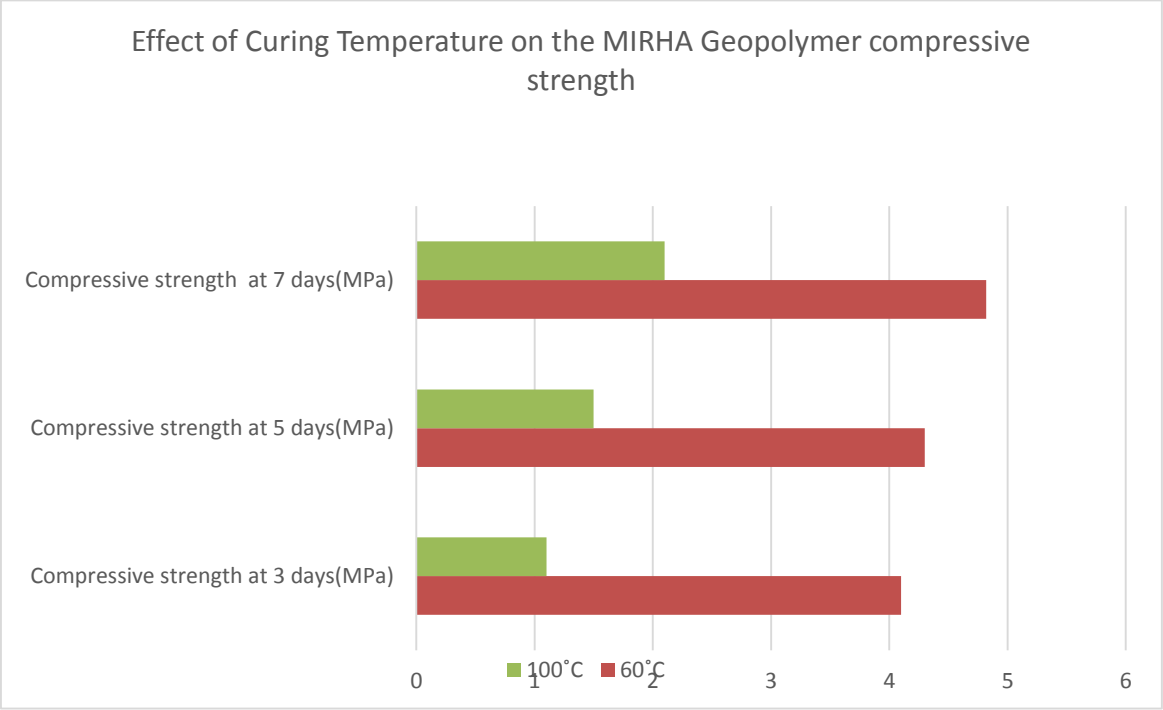

Figure 12 : Effect of curing temperature on the MIRHA geopolymer cement compressive strength

The overall compressive strength of the 100˚C cement is lower than the 60˚C cement. The highest compressive strength recorded is 4.82MPa@60˚C (7 days). The lowest compressive strength recorded is 1.1MPa@100˚C (1 day). The compressive strength for the 60˚C showed a 17% increase in compressive strength from day 1 to day 7. The compressive strength for the 15M NaOH showed a 91% increase in compressive strength from day 1 to day 7.

Thus, it can be concluded from Figure 12 that the curing temperature of 60˚C results in a better compressive strength than the 100˚C curing temperature.

5.3 Data Analysis/Discussion

The discussion part of the project will be divided into 5 parts. In this section, the student will analyze the findings and come up with a proper explanation on the reasons behind the results. The discussion will be tailor made to provide explanation for each of the experiment conducted.

Experiment 1

Experiment 1 is modelled to find the best water ratio to be used in order to obtain the highest compressive strength. The MIRHA needs a higher amount of water than usual due to its natural properties. It is found that a water ratio of 40% is optimum. As we go higher, there is a decrease in the compressive strength of the MIRHA geopolymer cement.

Once the water is mixed in the slurry, the hydration process will begin. It has been established that the water content in the cement at the time of hardening plays a large role in determining the ultimate strength of the cement. The water/cement ratio law states that as the water to cement ratio is reduced the strength is increase. As the water/cement ratio is increased, the distance between hydrated cement crystals is increased. This reduces the strength of the cement.

The primary factor in determining the strength of the cement is the density of the hydrated cement paste. The denser the cement paste, the higher the strength of the hardened cement. Thus it is important that a water to cement ratio is determined in order to produce the densest possible hydrated cement paste. From Experiment 1, we find that a 40% water to cement ratio is sufficient enough to result in a high cement strength. Using more than the described water amount results in reduced strength since the density of the hydrated cement paste is lower. An increased in water content pushes the hydrated cement crystals apart reducing the bonding contact area between them resulting in reduced strength.

For MIRHA, it is determined that the water/cement ratio should not exceed 40%. There is a possibility for reduced water/cement ratio if additives are included.

Experiment 2 & 3

In Experiment 2, the ratio of MIRHA to alkali activator and the effect of sodium hydroxide molarity on the compressive strength was tested. The result obtained showed that the MIRHA to alkali activator ratio of 2:1 is better than the 1:1 ratio. Other than that, it was also proven that 10M sodium hydroxide gives a better compressive strength compared to the 15M sodium hydroxide.

In terms of the ratio for MIRHA to alkali activator, we can conclude that it had to do with the density of the hydrated cement paste. The 1:1 ratio resulted in a less density due to a large amount of liquid present in the cement mixture. In the 2:1 ratio, there is a suitable amount of fly ash to alkali activator. The mixture was denser. As shown in the experiment, the ratio was applied to two different molarity of sodium hydroxide. It was found that in both cases, the 2:1 ratio was better compared to the 1:1 ratio. The 1:1 ratio had a higher liquid content in the overall cement slurry resulting in a reduced compressive strength.

Concentration of alkali solution affects the compressive strength of the geopolymer cement. MIRHA contains alumina and silica. Leaching of alumina and silica ions are generally high with sodium hydroxide solution. When alumino-silicate material comes into contact with alkali solution, leaching of both Si^{4+}, Al^{3+} and other ions start [28]. It is found that in 10M NaOH is higher compared to 15M NaOH[28]. In the 15M NaOH, the dissolution of the ions are reduced due to an increase in the coagulation of silica. The NaOH acts as a catalysis for the condensation reaction. Due to a higher dissolution of ions in the 10M NaOH, there is an increase in the formation of hydrated calcium silicates. Thus, 10M NaOH is appropriate for the synthesis of geopolymer compared to the 15M NaOH.

Experiment 4

In Experiment 4, it is found that 300µm MIRHA grain size resulted in a better compressive strength than the 600µm MIRHA grain size. A coarser cement will require more time to set [29]. Thus, this result in a lower degree of hydration [29]. The strength development of a coarser cement will also lag significantly behind that of finer cement. Thus it could be concluded that a finer cement will result in a higher compressive strength due to an increased degree of hydration.

Experiment 5

In Experiment 5, the curing temperature of the MIRHA geopolymer cement was tested. It is found that the curing temperature of 60˚C resulted in a better compressive strength. It can be concluded that the MIRHA geopolymer cement will not work well in high temperature environment. It could perform better if additives were added to improve its heat durability. Ambient temperature is needed for the geopolymer pozzolanic reaction. The reaction is generally accelerated with temperature increase. It is stated that for geopolymer cement, the ambient curing temperature should be between 30˚C to 90˚C [2]. It can be concluded that curing at elevated temperatures is effective (in the range of 30˚C to 90˚C) and has a more significant contribution to geopolymeric reactions. The temperature of 100˚C retarded the development of the compressive of the strength. The compressive strength decreased on curing at higher temperature, as prolonged curing at the elevated temperature broke the granular structure of the geoplymer mixture. It is theoretically accepted that a shorter exposure to higher temperature would lead to a better compressive strength. In the Experiment, the samples were cured for 24 hours at the given temperature. For this purpose, it could be concluded that a temperature of 60˚C is ambient for MIRHA geopolymer cement.

Curing Time

Based on the experiments conducted, it is found that the compressive strength increases with the curing time. Longer curing time improved the polymerization process that occurs in the geopolymer cement. This results in a higher compressive strength. Thus it can be concluded that a longer curing time at room temperature results in a stronger compressive strength.

CHAPTER 6

CONCLUSION

6.1 Relevancy to the objective

In conclusion, the ultimate objective of this project is to find out the efficiency of MIRHA geopolymer for well cementing. This project aims to prove the benefits of using MIRHA geopolymer. The environmental benefits will be derived and presented in this project. At the same time, the project aims to explore the economic advantages of using MIRHA geopolymer cement.

From the experiments conducted, we are able to figure out the optimum condition for MIRHA geopolymer cement that would result in a higher compressive strength. It is concluded that a water ration of 40% or lower should be used. It is found that a MIRHA to alkali activator ratio of 2:1 results in a better compressive strength. As for alkali activator, a 10M sodium hydroxide solution results in a higher compressive strength and can be concluded as the optimum molarity for geopolymer synthesis. A finer grain size results in a better compressive strength. A longer curing time results in an increased compressive strength. Finally, it is found that an optimum temperature of 60˚C should be used for curing rather than an elevated temperature. All of these results has been presented and discussed. The objectives of the final year project has been achieved.

Overall, there seems to be good potential in using MIRHA geopolymer cement as a well cement. However, we still need to tackle its slow hardening time. The compressive strength of the MIRHA geopolymer could be increased by mixing MIRHA with other waste substances such as fly ash. MIRHA geopolymer cement qualifies as well cement. With the addition of additives, it is believed that MIRHA geopolymer cement would possess better quality to perform as a well cement.

6.2 Suggested Future Work for Expansion and Continuation

There are some areas of the MIRHA geopolymer cement synthesis that could be researched further. This final year project has concluded its research on the effects of several factors on the MIRHA geopolymer strength. Based on the conclusion made, it is possible to synthesize stronger MIRHA geopolymer cement.

The next step would be to find out ways to increase the MIRHA geopolymer strength. As we know, well cement has to endure higher pressure in the wells. Another important factor that needs to be tackled would be the durability of the MIRHA geopolymer cement in an elevated temperature environment. The durability of the geopolymer could be increased by adding in additives. Other than that, it is proposed that a mixture of MIRHA and fly ash should tested since both of these wastes have similar properties.

MIRHA geopolymer properties are found to be acid resistive. This should be investigated further. The current OPC is unstable in acidic environment. The OPC is also unstable in CO_2 rich environment. The MIRHA geopolymer cement could act as a replacement for the OPC in these environments. This could prove useful, especially in carbon sequestration wells. Carbon sequestration wells have a higher saturation of carbonic acid and CO_2 concentration. Thus, the possibility of using MIRHA geopolymer cement for carbon sequestration well should be investigated.

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