# Analysis of Chemical and Mechanical Properties of Geopolymer Cement

By

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14355

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**CERTIFICATION OF APPROVAL**

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A project dissertation submitted to the

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BACHELOR OF ENGINEERING (HONS.)

(PETROLEUM ENGINEERING)

Approved by,

__________________________

(RAJA RAJESWARY SUPPIAH)

Supervisor

Date:

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2014

## 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 concept 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.

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(SITI FATEHAH ABDULLAH)

## ABSTRACT

Due to increasing of industrial waste product, as well as environmental concern on conventional cement due to $CO_2$ emission, a new green cement technology has been developed known as geopolymer cement. However, it is crucial that geopolymer cement can meet the specific requirement to ensure its efficiency in downhole condition. Industrial by products were utilized as raw material for geopolymer cement in this project. They are Fly Ash (FA) and Microwave Incinerated Rice Husk Ash (MIRHA). Numerous studies have been done on the application of fly ash in geopolymer cement and it has been proven that fly ash is a good raw material which can form geopolymer cement with high compressive strength. However, due to abundance of rice husk as waste materials that is not widely utilized, there are also several studies on the employment of MIRHA in geopolymer cement. Yet the contribution of MIRHA in compressive strength of geopolymer cement has not been extensively studied. Hence, this project studies the compressive strength of geopolymer cement composed of fly ash, and MIRHA as raw material and the effect of addition of different percentage of silica fume towards the strength development. Microstructure studies also were conducted to confirm the result of compressive strength of the sample by using X-Ray Diffraction (XRD), and Fourier Transform Infrared Spectroscope (FTIR) and Scanning Electron Microscope (SEM).

## ACKNOWLEDGEMENT

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I would also like to acknowledge with much appreciation to my fellow colleagues whom had given unconditional helps and cooperation in completion of my project.

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## ABBREVIATIONS AND NOMENCLATURES

| | |
|---|---|
| Al | Aluminum |
| API | American Petroleum Institute |
| ASTM | American Society of Testing Materials |
| $CO_2$ | Carbon Dioxide |
| CRM | Cement Replacement Materials |
| FA | Fly Ash |
| FTIR | Fourier Transform Infrared Spectroscopy |
| GHG | Green House Gases |
| IR | Infra-Red |
| ISO | International Organization for Standardization |
| KOH | Potassium Hydroxide |
| MIRHA | Microwave Incinerated Rice Husk Ash |
| $Na_2SiO_3$ | Sodium Silicate |
| NaOH | Sodium Hydroxide |
| OPC | Ordinary Portland Cement |
| PCC | Pulverized Coal Combustion |
| SF | Silica Fumes |
| Si | Silica |
| SPE | Society of Petroleum Engineers |
| UTP | University Technology PETRONAS |
| XRD | X-Ray Diffraction |

# CHAPTER 1

# INTRODUCTION

## 1.1 BACKGROUND STUDY

In oil or gas well drilling and completion, cementing is a major operation that contributes to stability and safety of the well. Due to uncertain downhole condition, cement slurry should be designed to meet the required downhole condition. One of the most important parameter in designing cement slurry is the compressive strength. Ordinary Portland Cement (OPC) is the conventional well cement that has been used widely in oil and gas industry. However, OPC contributes to significant gas house gaseous ($CO_2$) to the environment (McLellan, Williams, Lay, Van Riessen, & Corder, 2011), (Hewlett, 2003), (Hilsdorf & Kropp, 2004), (Vidivelli & Mageswari, 2010) . Hence, in order to reduce environmental effect from OPC, a green cement technology known as geopolymer cement is being developed.

Geopolymer cement provides comparable performance to OPC with an additional advantage of reduced gas house gaseous emission (Mahmoudkhani, Huynh, Sylvestre, & Schneider). Geopolymer is an alumino-silicate binder obtained through geopolymerisation process as shown in Figure 1. Solid alumino silicate is converted into a synthetic alkali aluminosilicate through the process shown in Figure 1. The properties of geopolymer cement depend on the raw material and the condition during preparation.

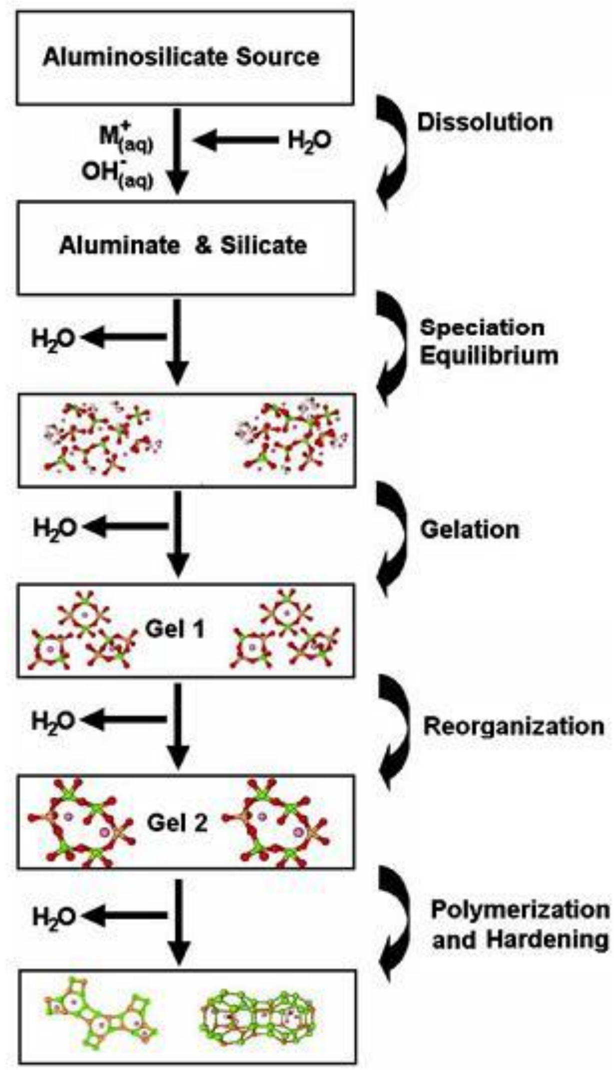



Figure 1: Conceptual model of geopolymerizataion (Duxson et al., 2007).

This project focuses on the study of geopolymer cement as a replacement for conventional cement in oil well which is known as Ordinary Portland Cement (OPC). In this project, Fly Ash (FA) and Microwave Incinerated Rice Husk Ash (MIRHA) were used as raw materials for geopolymer cement.

Fly ash is a popular cement replacement material of OPC due to the amorphous aluminosilica content in it which gives good compressive strength to geopolymer cement. The abundance of fly ash is also one of the factors of its inclusion as replacement material of OPC. According to (Kusbiantoro, Nuruddin, Shafiq, & Qazi, 2012), a total of 480million tons of fly ash was produced annually which makes waste management issue becomes severe. Hence, the utilization of fly ash in industry to form a new product is highly beneficial for the environment.

In the other hand, rice husk also having pozzolanic materials which is a good candidate as raw material for geopolymer cement. However, the utilization of rice husk in geopolymer cement has not been widely studied. The presence of rice husk also abundance in nature especially in Asian country which consume rice as its main food. About 130 million tons of rice husk were produced annually in the world and 446 thousand tons of the total are produced in Malaysia (Palacpac, 1978).

## 1.2 PROBLEM STATEMENT

The usage of cement is a vital element in oil well completion. According to (Nasvi, Ranjith, & Sanjayan, 2012), the conventional oil well cement, Ordinary Portland Cement (OPC) has been found to be unstable in $CO_2$ environment as it degrades, shrink and reduce in strength over time. It is reported by The Government of Canada that OPC is one of the major contributor of greenhouse gaseous such as Carbon Dioxide, $CO_2$ (Mahmoudkhani et al.). The emission of $CO_2$ which is ranging from 0.84 to 1.15 kg/kg of clinker (Mahmoudkhani et al.) is found to cause global warming and if it is uncontrolled, it is expected that the global temperature will increase significantly in next 50 to 100 years (Nasvi et al., 2012).

The development of geopolymer cement is an advantage to reduce environmental effect (Abdullah, Hussin, Bnhussain, Ismail, & Ibrahim, 2011) as it is believed to reduce net $CO_2$ emission up to 10% compared to Portland cement (Mahmoudkhani et al.). On the contrary to OPC, geopolymer cement surpasses OPC in many criteria. Geopolymer cement is claimed to have greater strength, superior acid resistant characteristics, and insignificant shrinkage as compare to OPC. Its pumpability is also superior to OPC (Nasvi et al., 2012).

According to (Suetsugu, Miyata, & Kogure), the discharge of coal fly ash from thermal electric power plants pattern increasing over year.

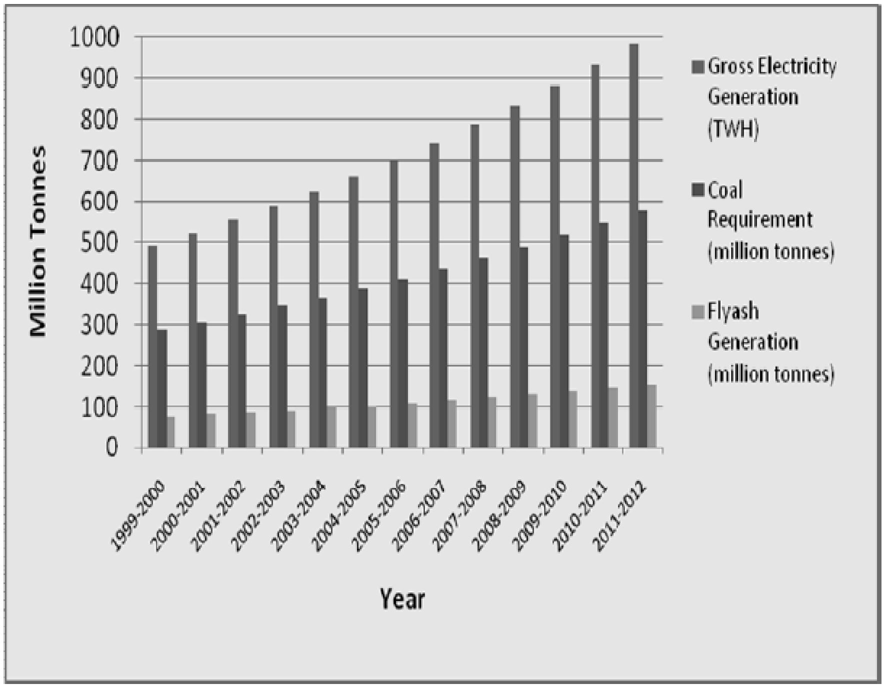



Figure 2: Fly ash generation chart retrieved by (Vidivelli & Mageswari, 2010)

Figure 2 shows the generation of fly ash from 1992 to 2012. The fly ash produced trend is directionally proportional to the gross electricity generation and coal requirement which is increasing over time (Vidivelli & Mageswari, 2010). Hence, due to increasing amount of fly ash, a new development of another product using fly ash would be a beneficiary effort to green environment. Other than fly ash, rice husk also can be developed into concrete binder. Rice husk production is estimated as much as one fifth of the total amount of world's annual gross rice production (Johan, Kutty, Isa, Muhamad, & Hashim, 2011). Both fly ash and rice husk can be developed into geopolymer cement in oil well operation.

Chemical component and microstructure analysis of raw materials is also one of the concerns in the formation of geopolymer cement bond. Thus, FTIR, XRD and SEM analysis are required to confirm the result of compressive strength obtained. The development of geopolymer technology in cement not only reduces $CO_2$ emission as compared to Portland Cement, it also develop an effective waste management problem by utilizing waste materials such as fly ash and MIRHA (Abdullah et al., 2011).

## 1.3 OBJECTIVE AND SCOPE OF STUDY

The objectives of this project are:

1) To utilize waste product and form a new product using it.
2) To compare the compressive strength of geopolymer cement with different size of MIRHA particles.
3) To study the effect of various proportions of Fly Ash and MIRHA towards compressive strength of geopolymer cement.
4) To study the effect of addition of different percentage of silica fumes to geopolymer cement.
5) To study microstructure of fly ash, MIRHA and silica fumes.

The scopes of study for this project are:

- Study the effect of different particle size of MIRHA, different proportion of Fly Ash and MIRHA, and addition of different percentage of silica fumes on compressive strength of geopolymer cement.
- Microstructure analysis by using FTIR and XRD and SEM.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 GEOPOLYMER CEMENT

Reaction between aluminosilicates and aqueous alkaline solution produces geopolymer which is one of synthetic binders (Mahmoudkhani et al.), (Bakharev, 2005). This reaction results in formation of $SiO_4$ and $AlO_4$ linked alternately by sharing the oxygen (Mahmoudkhani et al.). In other words, geopolymer also known as amorphous alumino silicate that is synthesized by polycondensation of geopolymeric antecedent and alkali polysilicates (Abdullah et al., 2011). Alkaline solution such as sodium hydroxide (NaOH) and sodium silicate ($Na_2SiO_3$) are commonly used as alkaline activators to produce geopolymer (Bakri et al., 2011), (Bakharev, 2005). The activation of Fly Ash (FA) requires heat since the activation energy is high to ensure the reaction will take place (Bakharev, 2005).

The heterogeneous reaction between solid aluminosilicates oxides and alkali metal silicates solutions at highly alkaline condition at trivial temperature produces amorphous to semi-crystalline polymeric structures. The structure consist of Si-O-Al and Si-O-Si bonds (Abdullah et al., 2011).

The presence of silica (Si) in fly ash and rice husk and alumina (Al) in clay such as kaolin makes them as good materials to be used to form geopolymer (Abdullah et al., 2011). The raw materials can be activated by using alkali silicate solutions precisely can be NaoH and $Na_2SiO_3$. The activation of silicate gives rise to promising mechanical properties of the geopolymer cement (Abdullah et al., 2011).

Geopolymer cement slurry consist of geopolymeric materials which exhibit greater mechanical and chemical resistance as well as superior cost saving and it requires lower water amount for slurry preparation (Mahmoudkhani et al.).

Based on a study by *Amir H. Mahmoudkhani, SPE, Diana N.T. Huynh, Chuck Sylvestre, and Jason Schneider, Sanjer Corporation*, it is summarized that geopolymer cement exhibits these advantages:

- It can be designed for various densities ranging from 1200 to 1900 $kg/m^3$.
- Having wide range of thickening time from several minutes to several hours.
- Having superior early and late strength development.
- Having fast gel strength development.
- Controlled fluid loss properties.
- Enhanced flexibility and elasticity.
- Applicable for well with zonal isolation through strong bonding to formation and casing.
- Having robust compatibility with most common cement admixtures and additives.
- Significantly reduce $CO_2$ and water footprints.
- Cost savings.

## 2.2 RAW MATERIALS

### *2.2.1 Fly Ash (FA)*

Fly ash is a waste material generated from pulverized coal combustion (PCC) at power stations to generate electricity (Seames, 2003). Fly ash particles primarily exist in spherical shape but there is also small portion of irregular shape particle presents such as quartz. The composition of fly ash is dependent on the inorganic part of the coal used for burning. However, generally fly ash composed of 40 to 60% of silica and 20 to 30% of alumina (Abdullah et al., 2011). Other constituents of fly ash are iron, alkalis, potassium and sodium (Abdullah et al., 2011).

Fly ash is categorized into class C and class F based on its chemical composition. The calcium, sodium and magnesium content in class C fly ash are relatively higher as compared to class F. However, class F fly ash contains higher silica and iron which forms from burning bituminous coal. The calcium, alumino silicate and crystalline presence in fly ash make it a good candidate as replacement material for Portland cement as it hydrates (Vidivelli & Mageswari, 2010) and forms cementitious material when it dissolved in water. A good quality of fly ash should contain low carbon and high in alumino silicate.

The existence of fly ash is abundant worldwide but the usage to date is very restricted (Abdullah et al., 2011). The usage of fly ash as cement material has been developed in Japan. About 70% of fly ash production in Japan has been utilized into fly ash cement (Suetsugu et al.).

Due to pozzolanic reaction in fly ash concrete, the utilization of fly ash as partial replacement in Portland cement beneficial in reducing cement shrinkage and gives higher strength which improves the cement durability (Vidivelli & Mageswari, 2010). However, at mixing ratio 30% and 40%, the compressive strength reduced compared to 100% conventional cement due to high ash content in which increases water to cement ratio (Vidivelli & Mageswari, 2010). The generally spherical shape of fly ash particles gives a good consolidation property which contributes to low permeability of the concrete. Fly ash also gives lower permeability of the concrete by reducing water to cement ratio (Abdullah et al., 2011).

### *2.2.2 Microwave Incinerated Rice Husk (MIRHA)*

Microwave Incinerated Rice Husk (MIRHA) is a waste product from paddy plantation. MIRHA production is estimated as much as one fifth of the total amount of world's annual gross rice production (Johan et al., 2011). MIRHA is also one of the raw materials from industrial by product which can be used as cement material. The high content of amorphous silica which is 95% in 20% of the rice husk ash makes it is one of a good Cement Replacement Material (CRM) for Portland cement (Bayuaji, 2014). Burnt fly ash may produce more than 80% pure silica which makes the ash properties act like cement (Nuruddin, Shafiq, & Kamal, 2008). The burning process of rice husk should be under controlled condition to ensure that the content of amorphous silica is optimum for application in oil well cement (Bayuaji, 2014). The content of $SiO_2$ increase as burning temperature increase, however, it is not recommended to burn rice husk ash for more than one hour at 800°C due to sintering effect which turns the particles to finer size (Nuruddin et.al).

## 2.3 CEMENT ADDITIVES

### *2.3.1 Sodium Hydroxide (NaOH) solution*

According to (Abdullah et al., 2011), combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate and potassium silicate are the most common activator used in geopolymerisation. Sodium hydroxide solution gives higher leaching rate of $Al^{3+}$ and $Si^{4+}$ ions compared with potassium hydroxide (Abdullah et al., 2011). NaOH solution must be prepared one hour before the procedure as the reaction is exothermic (Kusbiantoro et al., 2012).

### *2.3.2 Sodium Silicate ($Na_2SiO_3$) solution*

The presence of soluble silicate in alkaline activator either sodium silicate or potassium silicate gives higher reaction rate as compared with the utilization of sodium hydroxide only (Abdullah et al., 2011). Sodium silicate activator dissolve and bond fly ash together.

The ratio of sodium hydroxide to sodium silicate is 1:2.5 is considered as optimum as mentioned by Hardijo et al, which states that at the ratio of 1:2.5, the highest compressive strength was achieved for 28 days of testing (Abdullah et al., 2011).

## 2.4 CHEMICAL CHARACTERISTIC TEST

### *2.4.1 X-Ray Diffraction (XRD)*

X-Ray Diffraction (XRD) is used to identify crystalline phases according to the diffraction pattern (Kozak, 2011). The diffraction pattern shows by XRD the element present can be known as each pattern is indicated to a specified element or phase.

### *2.4.2 Fourier Transform Infrared Spectroscopy (FTIR)*

Fourier Transform Infrared Radiation (FTIR) is one of the common methods used in mineral characterization. FTIR works by passing infrared radiation through the sample. Due to the presence of different molecule in the sample, some infrared might be absorbed by the molecules in the sample and some might pass through the sample. Hence, the result of molecular absorption and molecular transmission creates a complete molecular fingerprint of the sample. In addition, FTIR also able to give information such as identification of unknown material, quality or consistency of the sample, and the amount of components present in a mixture.

FTIR analysis identifies the functional group of materials such as alcohol, carboxylic acid, alkanes, alkenes and other possible functional groups of cement compound. The curve of FTIR analysis shows the reading of IR spectrum gives the information of which functional group does the value belongs to. FTIR is conducted to confirm the result obtained. For example, in the testing for geopolymer formed by fly ash and kaolinite clay by (Bakharev, 2005) shows the utilization of FTIR when the observation does not show any presence of zeolitic products but FTIR analysis indicated their presence.

### *2.4.3 Scanning Electron Microscopy (SEM)*

Scanning Electron Microscopy Test is performed using Scanning Electron Microscope which uses electrons to produce high resolution to capture a three-dimensional image of the sample with wide ranges of magnifications. It also gives the information of shape, composition and surface of the sample (Ezumi & Todokoro, 1999). According to (Gao, Xu, Chen, Li, & Lu, 2013), SEM can also observe the morphology of sample. Morphology in other words means the study of structure, shape, color and pattern of the sample.

## 2.5 MECHANICAL PROPERTIES

### *2.5.1 Compressive Strength*

Cement compressive strength is a maximum amount of stress that cement can withstand under crush loading. It is simply a ratio of maximum load it can sustain to the total surface area of the cement cubes. Compressive strength of cement can be influenced by many factors. According to (Nazari, Bagheri, & Riahi, 2011), compressive strength of cement can be influenced by curing temperature as it affects setting and hardening rate of cement. Polymerization rate occurs faster at elevated temperature. However, (Swanepoel & Strydom, 2002) and (Chindaprasirt, Chareerat, & Sirivivatnanon, 2007) concludes that the optimum curing temperature for geopolymer cement is 60°C. In addition, (Nuruddin, Shafiq, & Kamal, 2009) claim that curing time also gives significant effects on compressive strength development of geopolymer cement. For fly ash based geopolymer cement, addition of MIRHA into the geopolymer cement contributes in compressive strength development of the cement. Up to 7% of MIRHA was added into fly ash based geopolymer cement and it shows the increase in compressive strength together with increasing proportion of MIRHA added (Nuruddin et al., 2009).

# CHAPTER 3

# METHODOLOGY

## 3.1 RESEARCH METHODOLOGY

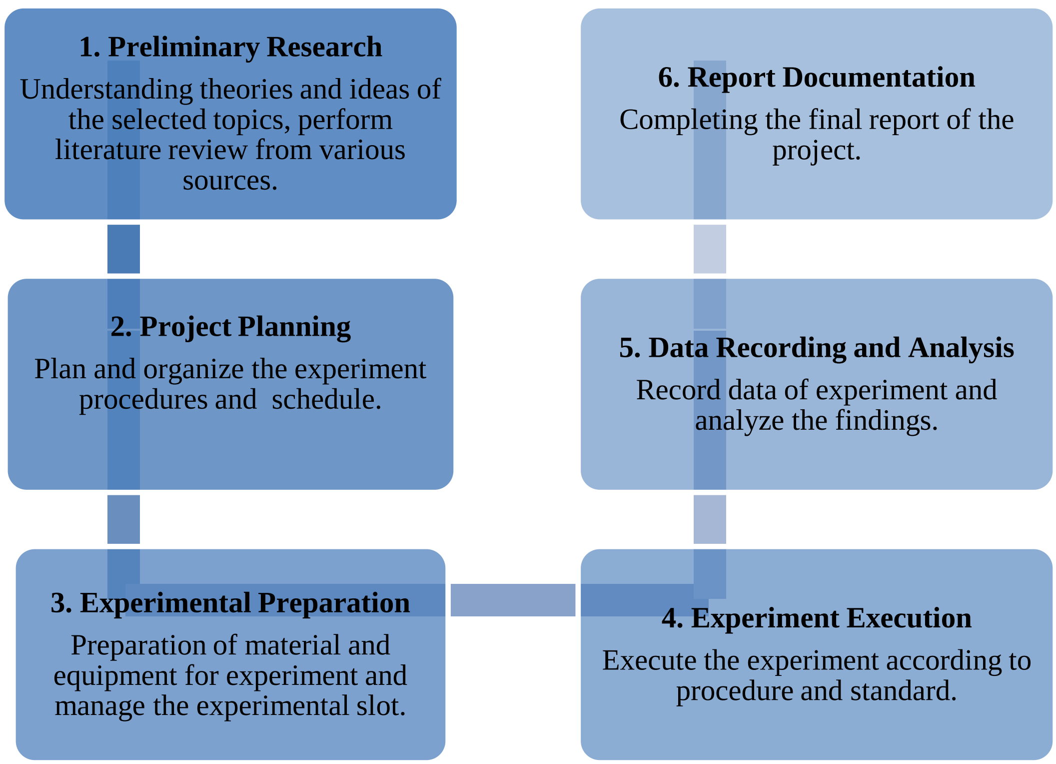



Figure 3: Project Flow

## 3.2 PROJECT ACTIVITIES

### *3.2.1 Materials and Equipment Preparation*

**Table 1: Materials and equipment used**

| Materials | Equipment |
|---|---|
| MIRHA | Sieve shaker |
| Fly Ash (ASTM Class F) | Electronic balance |
| Silica Fume | Constant speed mixer |
| Sodium Silicate | Magnetic stirrer |
| Sodium Hydroxide | 50x50x50mm cement mold |
| Distilled Water | Curing oven |
| | Compressive strength tester |

The materials that have been used in this project are shown in Table 1. Two sample of MIRHA was readily available in UTP laboratory. The first sample was burnt at 800°C with particle size distribution of 600µM as shown in Figure 8. Another MIRHA sample is finer in size with particle size distribution of 300µM which was burnt at 800°C. Fly Ash (ASTM Class F) and silica fumes were also readily available in UTP laboratory.

MIRHA with particle size distribution of 600µM

MIRHA with particle size distribution of 300µM

Fly Ash (ASTM Class F)

Silica Fume

**Figure 4: Materials for cement preparation**

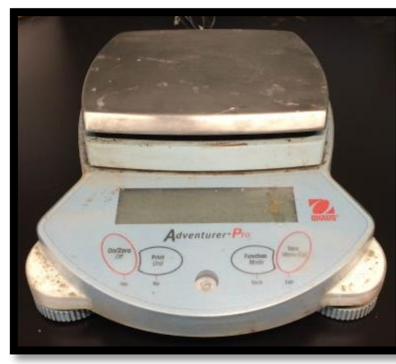
The equipment used during the experiment is shown in the Figure 5.


Sieve Shaker

Constant Speed Mixer


Compressive Strength Tester


Curing Oven




Mass Balance

50 x 50 x 50 mm Cement Molds

**Figure 5: Laboratory Equipment**

### *3.2.2 Laboratory Experiment*

The cement slurry was prepared as shown in the Figure 9. The samples were cured at 60°C as it is the optimum curing temperature for geopolymer cement (Swanepoel & Strydom, 2002), (Chindaprasirt et al., 2007). Sodium hydroxide of 12M was used in all the experiments. Water to cement ratio was maintained at 40% for all the experiments.

There are three experiments that are conducted with different objectives as follows:

**Experiment 1**:

To test the effect of MIRHA particle size to compressive strength of the sample.

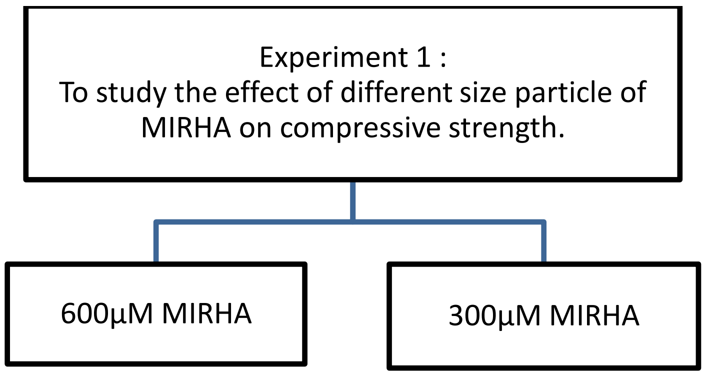



Figure 6: Structure of Experiment 1

Experiment 1 was conducted using the MIRHA and FA proportion as shown in Table 2. The cement samples were cured at 60°C for 24hours and the compressive strength was tested. Based on this experiment, the result of compressive strength obtained by using both size of MIRHA were compared and analyzed.

Table 2: Cement formulation for Experiment 1

| Sample | Fly Ash (g) | MIRHA (g) | NaOH solution (g) | $Na_2SiO_3$ solution (g) | Distilled Water (g) |
|---|---|---|---|---|---|
| A (60% FA;40% MIRHA) | 138 | 92 | 33 | 82 | 92 |
| B (70% FA;30% MIRHA) | 161 | 69 | 33 | 82 | 92 |
| C (80% FA;20% MIRHA) | 184 | 46 | 33 | 82 | 92 |

**Experiment 2**:

To test the optimum ratio of MIRHA and Fly Ash which produce highest compressive strength of geopolymer cement.

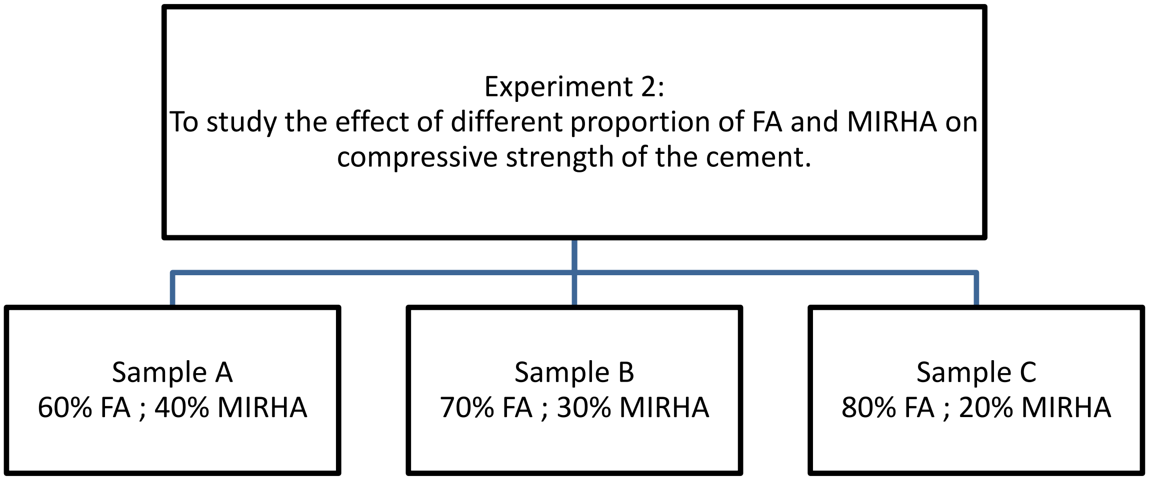



Figure 7: Structure of Experiment 2

Experiment 2 was conducted by using MIRHA which gives the highest strength based on Experiment 1. The proportion of MIRHA and Fly Ash was varied according to Table 3. The cement samples were cured at 60°C for 24hours and the compressive strength was tested for each cement cubes and compared.

Table 3: Cement formulation for Experiment 2

| Sample | Fly Ash (g) | MIRHA (g) | NaOH solution (g) | $Na_2SiO_3$ solution (g) | Distilled Water (g) |
|---|---|---|---|---|---|
| A (60% FA;40% MIRHA) | 138 | 92 | 33 | 82 | 92 |
| B (70% FA;30% MIRHA) | 161 | 69 | 33 | 82 | 92 |
| C (80% FA;20% MIRHA) | 184 | 46 | 33 | 82 | 92 |

**Experiment 3**:

To test the effect of adding different proportion of silica fumes (5%, 10% and 15%) on the compressive strength of geopolymer cement.

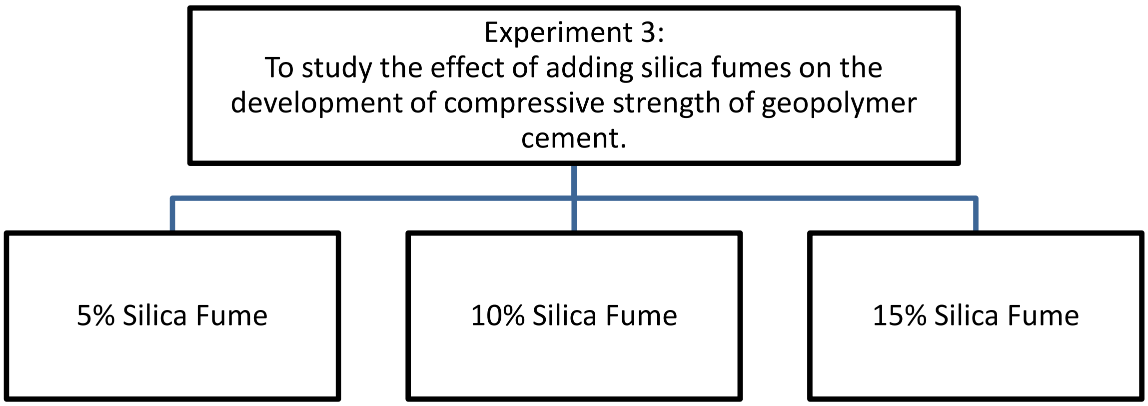


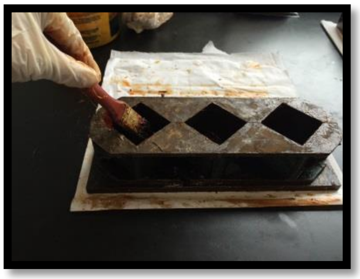

Figure 8: Structure of Experiment 3

Experiment 3 was conducted by varying the percentage of Silica Fumes as shown in Table 4 to observe the effect of adding silica fumes on the compressive strength of the geopolymer cement. The proportion of Fly Ash and MIRHA was chosen based on Experiment 2 which gives the highest compressive strength which is 40% MIRHA and 60% Fly Ash. The cement samples were cured at 60°C for 24hours and at atmospheric temperature for 7days before the compressive strength were tested.

Table 4: Cement formulation for Experiment 3

| Sample | Fly Ash (g) | MIRHA (g) | Silica Fume (g) | NaOH solution (g) | $Na_2SiO_3$ solution (g) | Distilled Water |
|---|---|---|---|---|---|---|
| 0% Silica Fume | 138 | 92 | 0 | 33.00 | 82.00 | 92.0 |
| 5% Silica Fume | 138 | 92 | 11.5 | 34.50 | 86.25 | 96.6 |
| 10% Silica Fume | 138 | 92 | 23.0 | 36.14 | 90.36 | 101.2 |
| 15% Silica Fume | 138 | 92 | 34.5 | 37.79 | 94.46 | 105.8 |

I. Preparation of Cement Sample



1) Grease the mold

2) Weigh the materials

3) Mix the materials in the mixer until turns into slurry state

4) Pour the slurry into the mold

5) Cure the cement slurry in the oven at 60°C for 24 hours

**Figure 9: Procedure for cement slurry preparation**

II. Compressive Strength Test

1) Measure the area of cement cube to be tested. The area is according to the size of the mould used.
2) Place cement cube in compressive strength tester.
3) Apply load increasingly.
4) Record the load where the cement cubes crushed.
5) Calculate the compressive strength using the equation below:

$$\sigma = \frac{F}{A}$$

Equation 1: Compressive Strength

Where:

$\sigma = compressive\ strength, \frac{N}{mm^2}$
$F = maximum\ load\ which\ the\ specimen\ can\ withstand, N$
$A = cross\ sectional\ area\ in\ contact\ with\ the\ load, mm^2$

III. XRD, FTIR and SEM Test

For these tests, the samples were sent to a lab for testing.

## 3.3 GANTT CHART

| | Final Year Project I | | | | | | | | | | | | | | Final Year Project II | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Week | | | | | | | | | | | | | | Week | | | | | | | | | | | | | |
| **Activities** | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 23 | 24 | 25 | 26 | 27 | 28 |
| Project Topic Selection | ■ | ■ | ■ | | | | | | | | | | | | | | | | | | | | | | | | | |
| Research Study and Project Planning | | | | ■ | ■ | ■ | | | | | | | | | | | | | | | | | | | | | | |
| Preparation of Extended Proposal | | | | | | ■ | ■ | | | | | | | | | | | | | | | | | | | | | |
| Submission of Extended Proposal | | | | | | | | ▲ | | | | | | | | | | | | | | | | | | | | |
| Proposal Defense | | | | | | | | | ■ | | | | | | | | | | | | | | | | | | | |
| Project Work Continues | | | | | | | | | ■ | ■ | ■ | ■ | | | | | | | | | | | | | | | | |
| Submission of Interim Draft | | | | | | | | | | | | | ▲ | | | | | | | | | | | | | | | |
| Submission of Interim Report | | | | | | | | | | | | | | ▲ | | | | | | | | | | | | | | |
| Experimental material and equipment preparation | | | | | | | | | | | | | ■ | ■ | ■ | ■ | | | | | | | | | | | | |
| Experiment execution | | | | | | | | | | | | | | | | | ■ | ■ | ■ | ■ | ■ | ■ | | | | | | |
| Submission of Progress Report | | | | | | | | | | | | | | | | | | | | | ▲ | | | | | | | |
| Data collection and analysis | | | | | | | | | | | | | | | | | | | | | | ■ | ■ | | | | | |
| Pre-Sedex | | | | | | | | | | | | | | | | | | | | | | | ▲ | | | | | |
| Submission of Final Draft & Technical Paper | | | | | | | | | | | | | | | | | | | | | | | | | | ▲ | | |
| SEDEX | | | | | | | | | | | | | | | | | | | | | | | | | | ▲ | | |
| Final presentation / VIVA | | | | | | | | | | | | | | | | | | | | | | | | | | | | ▲ |
| Documentation | | | | | | | | | | | | | | | | | | | | | | | | | | | | ■ |

Figure 10: Gantt Chart

## 3.4 KEY MILESTONE

Table 5: Project's Key Milestone

| | WEEK | TASK | REMARKS |
|---|---|---|---|
| FYP 1 | 1-3 | Project topic selection | Done |
| | 4-6 | Research study and project planning | Done |
| | 8 | Submission of extended proposal | Done |
| | 9 | Proposal defense | Done |
| | 14 | Submission of interim report | Done |
| FYP 2 | 15 | Lab and Equipment booking | Done |
| | 16 | Lab and Equipment handling demonstration by lab technician | Done |
| | 17 | Equipment and material preparation | Done |
| | 18 | FTIR test of Class F Fly Ash was executed | Done |
| | 19 | Experiment on water to cement ratio | Done |
| | 20 | First batch of cement using 300µM MIRHA was cured for 24hours and compressive strength was tested. | Done |
| | 21 | Second batch of cement using µM MIRHA was cured for 24 hours and compressive strength was tested. | Done |
| | 23 | Third batch of cement mixing and curing for 7 days. (Same proportion of MIRHA and FA but different proportion of Silica Fumes) | Done |
| | 24 | Compressive strength test on third batch cement sample. | Done |
| | 25 - 27 | Data gathering and interpretation. | Done |
| | 28 | Final Presentation & Documentation | Done |

# CHAPTER 4

# RESULT AND DISCUSSION

## 4.1 COMPRESSIVE STRENGTH TEST

***Experiment 1: Determining the effect of different MIRHA particle size on compressive strength of geopolymer cement.***

Table 6: Mixture Proportion of Geopolymer Cement for Experiment 1

| Sample | Fly Ash (g) | MIRHA (g) | NaOH solution (g) | $Na_2SiO_3$ solution (g) | Distilled Water (g) |
|---|---|---|---|---|---|
| A (60% FA;40% MIRHA) | 138 | 92 | 33 | 82 | 92 |
| B (70% FA;30% MIRHA) | 161 | 69 | 33 | 82 | 92 |
| C (80% FA;20% MIRHA) | 184 | 46 | 33 | 82 | 92 |

Table 7: Compressive strength of geopolymer cement sample with different size of MIRHA

| | Compressive Strength (MPa) | |
|---|---|---|
| **Sample** | **600µM MIRHA** | **300µM MIRHA** |
| A (60% FA;40% MIRHA) | 0.80 | 4.21 |
| B (70% FA;30% MIRHA) | 1.60 | 3.37 |
| C (80% FA;20% MIRHA) | 1.00 | 2.99 |

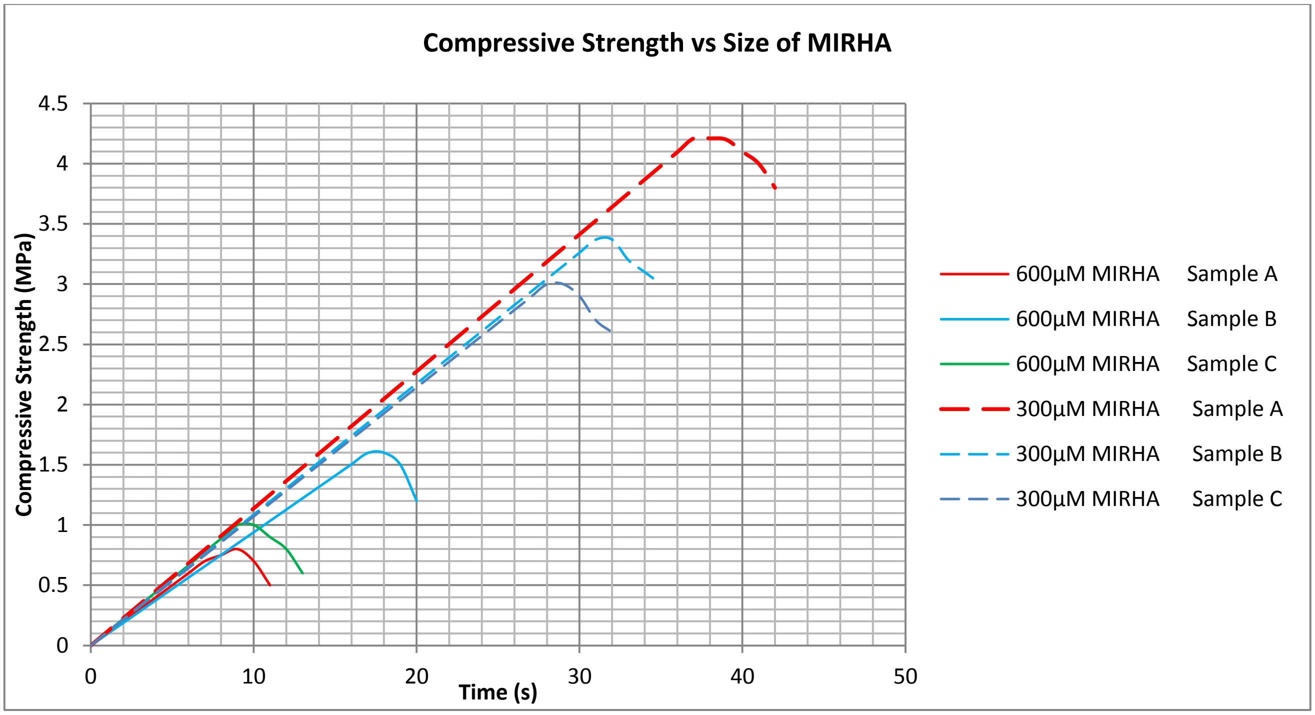



**Figure 11: Compressive strength of cement sample using different particle size distribution of MIRHA.**

Figure 15 shows the comparison of compressive strength of cement cube by using two different sample of MIRHA. It was observed that 300µM MIRHA gives higher compressive strength as compared with 600µM MIRHA. Coarser MIRHA will require longer time to set. This results in lower degree of hydration and lagging in cement strength development. This concludes that the size of MIRHA does give significant effect on compressive strength of geopolymer cement. The finer MIRHA size contributes to higher compressive strength as compared to coarser particle size of MIRHA due to higher degree of hydration.

Figure 12: Cement sample by using 600µM MIRHA

Figure 13: Cement cube by using 300µM MIRHA

Figure 16 and 17 shows cement cube sample by using 600µM MIRHA and 300µM MIRHA respectively. It can be observed that the cement cube in Figure 16 which is using 300µM MIRHA having dark layer on top which is a result of suspended MIRHA particles during curing. Meanwhile, cement cubes in Figure 17 which use 300µM MIRHA has less suspended MIRHA.

***Experiment 2: Determining the optimum proportion of Fly Ash and MIRHA that gives the highest compressive strength of geopolymer cement.***

This experiment was a continuation of Experiement 1. Based on the result of Experiment 1, 300µM MIRHA gives better compressive strength as compare to 600µM MIRHA. Hence, the MIRHA used in Experiment 2 is 300µM while fly ash was used from Class F Fly Ash.

Table 8: Mixture Proportion of Geopolymer Cement for Experiment 2

| Sample | Fly Ash (g) | MIRHA (g) | NaOH solution (g) | $Na_2SiO_3$ solution (g) | Distilled Water (g) |
|---|---|---|---|---|---|
| A (60% FA;40% MIRHA) | 138 | 92 | 33 | 82 | 92 |
| B (70% FA;30% MIRHA) | 161 | 69 | 33 | 82 | 92 |
| C (80% FA;20% MIRHA) | 184 | 46 | 33 | 82 | 92 |

Table 9: Compressive strength of geopolymer cement sample with different proportion of FA and MIRHA

| Sample | Compressive Strength (MPa) |
|---|---|
| A (60% FA;40% MIRHA) | 4.21 |
| B (70% FA;30% MIRHA) | 3.37 |
| C (80% FA;20% MIRHA) | 2.99 |

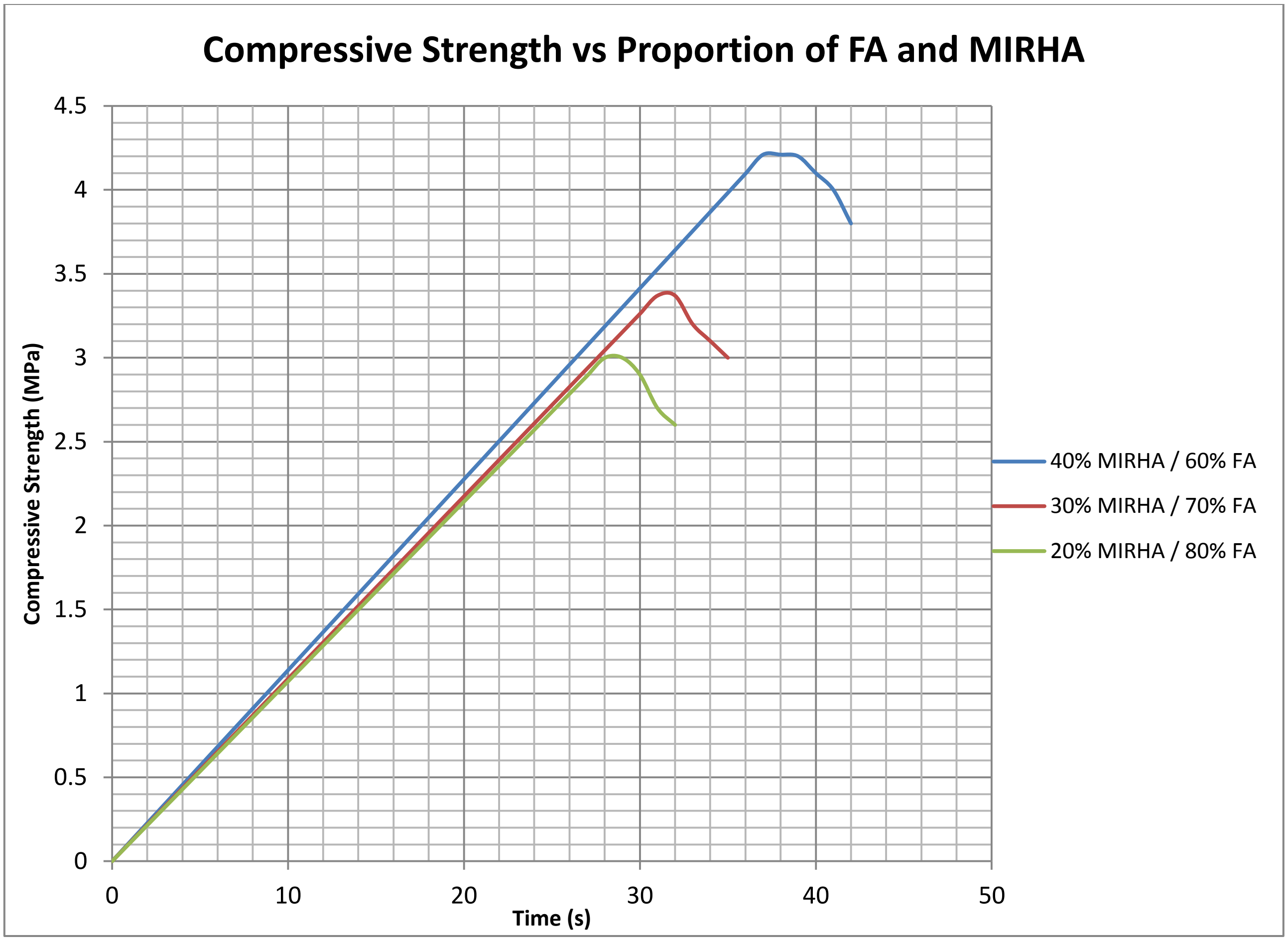



**Figure 14: Compressive strength of cement sample at different proportion of MIRHA and Fly Ash**

Based on the result, the higher the MIRHA to FA ratio gives better compressive strength to the geopolymer cement. The experiment was done until the ratio of 40% MIRHA and 60% FA. Further increasing MIRHA proportion will require additional water to cement ratio since MIRHA has high absorbance properties which will absorb the water quickly and cause the cement paste not being mixed entirely.

The mixture of MIRHA with FA as raw materials will alter $SiO_2$-$Al_2O_3$ ratio in the mixture and improves interfacial transition zone. This will directly results in higher compressive strength of the geopolymer cement (Kusbiantoro et al., 2012). Activation of Si and Al materials in the MIRHA and FA was completed by adding alkaline activator which is NaOH and $Na_2SiO_3$ solution.

***Experiment 3: Determining the effect of adding silica fumes in geopolymer cement.***

Experiment 3 is a continuation of Experiment 2. In Experiment 2, the proportion of 40% MIRHA and 60% FA gives the highest compressive strength to geopolymer cement. Thus, this experiment aims to study whether the addition of silica fumes will improves the compressive strength of the geopolymer cement. The proportion of MIRHA to FA used in this experiment is 40% and 60% respectively with silica fumes percentage of 0%, 5%, 10% and 15% to the total weight of MIRHA and FA.

**Table 10: Mixture Proportion of 40% MIRHA and 60% FA Geopolymer Cement for Experiment 3**

| Sample | Fly Ash (g) | MIRHA (g) | Silica Fume (g) | NaOH solution (g) | $Na_2SiO_3$ solution (g) | Distilled Water |
|---|---|---|---|---|---|---|
| 0% Silica Fume | 138 | 92 | 0 | 33.00 | 82.00 | 92.0 |
| 5% Silica Fume | 138 | 92 | 11.5 | 34.50 | 86.25 | 96.6 |
| 10% Silica Fume | 138 | 92 | 23.0 | 36.14 | 90.36 | 101.2 |
| 15% Silica Fume | 138 | 92 | 34.5 | 37.79 | 94.46 | 105.8 |

**Table 11:Compressive strength of 40% MIRHA and 60% FA geopolymer cement sample with different proportion of Silica Fumes**

| Sample | Compressive Strength (MPa) |
|---|---|
| 0% Silica Fume | 4.21 |
| 5% Silica Fume | 5.50 |
| 10% Silica Fume | 5.53 |
| 15% Silica Fume | 5.67 |

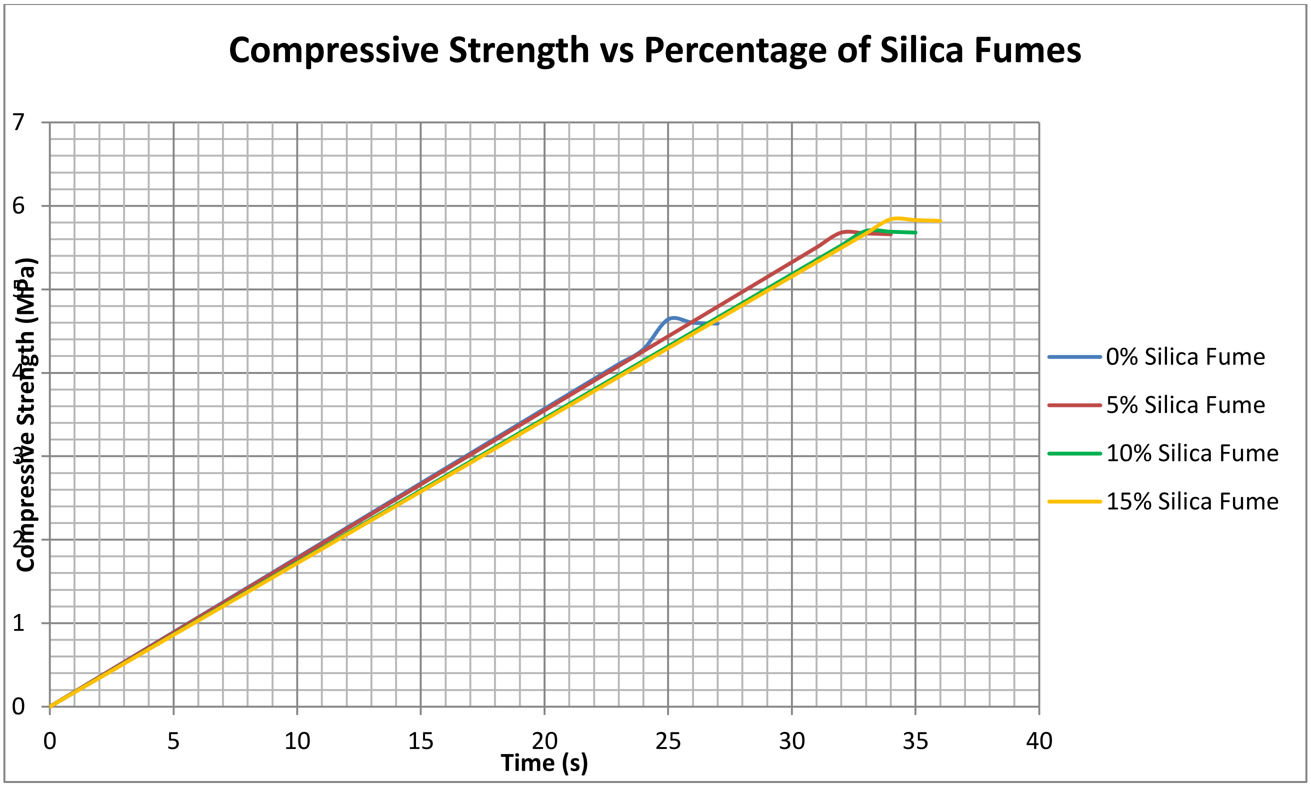



Figure 15: Compressive strength of 40% MIRHA and 60% FA with different proportion of Silica Fumes

Based on the result, inclusion of silica fumes to FA and MIRHA gepolymer cement increase the compressive strength of the cement. The increment from 0% silica fumes to 5% silica fume added is significant which is from 4.21MPa to 5.50MPa but the increment of silica fumes from 5%, 10% up to 15% in the geopolymer cement result in small increment of compressive strength which is from 5.50 MPa, 5.53MPa to 5.67MPa respectively. To confirm the experiment, another batch of cement slurry with similar proportion was performed and the result shows similar pattern.

In this experiment, silica fumes acts a binding agent which improves the bonding of the particles in the cement. Due to the crystalline nature of MIRHA particles, it results in high pore spaces in the gepolymer cement microstructure. The higher the pore spaces in the structure gives reduced compressive strength. Hence, the addition of silica fumes reduced the pore and void spaces which improves the compressive strength of the cement. However, there is a controversial issue in addition of silica fumes in cement and concrete. According to (Cong, Gong, Darwin, & McCabe, 1992), there are two opinion regarding the inclusion of silica fumes in concrete and cement. Some opinions support the claim that addition of silica fumes in cement paste but there is evidence against this claim. The contradict evidence claims that the addition of silica fumes does not increase the compressive strength of cement paste but it increase the compressive strength of concrete by strengthening the bond between cement paste and aggregate.

## 4.2 MICROSTRUCTURE ANALYSIS

### *4.2.1 Fourier Transform Infrared Spectroscopy (FTIR) Analysis*

FTIR analysis gives information of functional group of a material. In this experiment, FTIR analysis was conducted on fly ash, MIRHA and Silica Fumes samples.

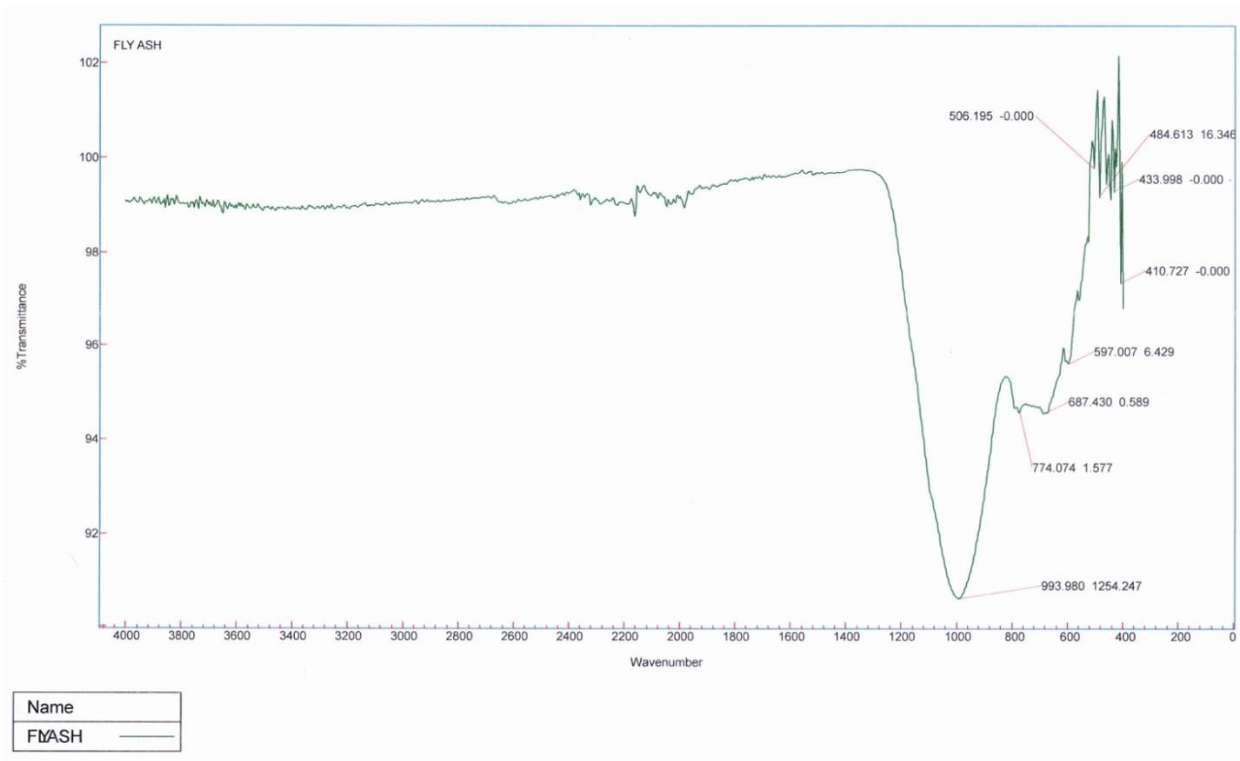



Figure 16: FTIR analysis of Class F Fly Ash

Figure 16 shows FTIR analysis of Fly Ash (Class F). The IR spectrum shows main absorption bands at 410.727, 433.998, 484.613, 506.195, 597.007, 687.430, 774.074 and 993.980 $cm^{-1}$. The broad component at 993.980 $cm^{-1}$ is due to Si-O-Si and Al-O-Si asymmetric vibration (Mohd Mustafa Al Bakri et al., 2012). The rest of the peak shows existence of fingerprint region. Fingerprint region is a complex area which shows many bands, frequently overlapping each other. Due to the complexity of the region, the interpretation was omitted.

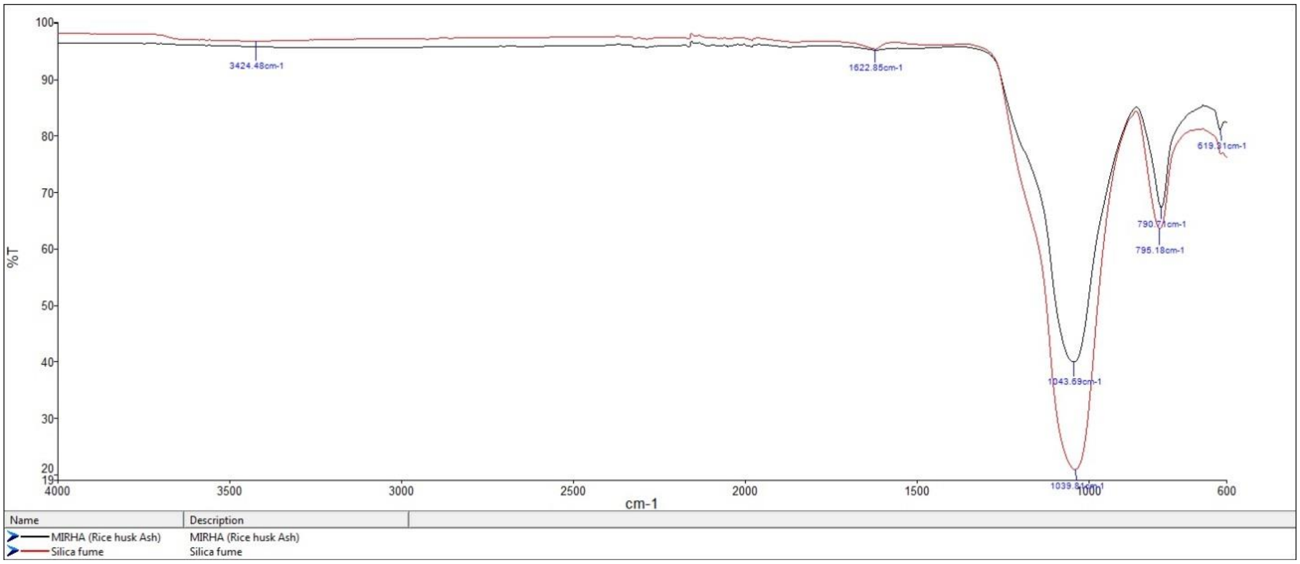



Figure 17: FTIR analysis of MIRHA and Silica Fumes

Figure 17 shows FTIR analysis of MIRHA and Silica Fumes. The trend of variation for both sample are almost similar. For MIRHA, the main absorption occurs at 1043.69, 790.71 and 619.31 $cm^{-1}$ while Silica Fumes shows main absorption at 1039.81, 795.18, and 619.31 $cm^{-1}$. From 3700 until 1300 $cm^{-1}$, weak O-H (alcohol) stretching band and asymmetric C=C=C stretching band was detected. The presence of double bond in MIRHA and Silica Fumes shows the reason of increasing compressive strength with increasing MIRHA proportion to Fly Ash and the additional of Silica Fumes to geopolymer cement. The peak at 1043.69 and 1039.81 $cm^{-1}$ distinguish stretching frequency of Si-O-Si. The peak at 790.71 and 795.18 $cm^{-1}$ shows the symmetric stretching of Si-O-Si. The band at 619.31 $cm^{-1}$ shows asymmetrical vibration of Si-O bond (Moenke, 1974). The weak peak at 3424.48 and 1622.85$cm^{-1}$ shows presence of O-H-O bond which is water bond with hydrogen bridges (Kocak, 2010).

*4.2.2 X-Ray Diffraction (XRD)*

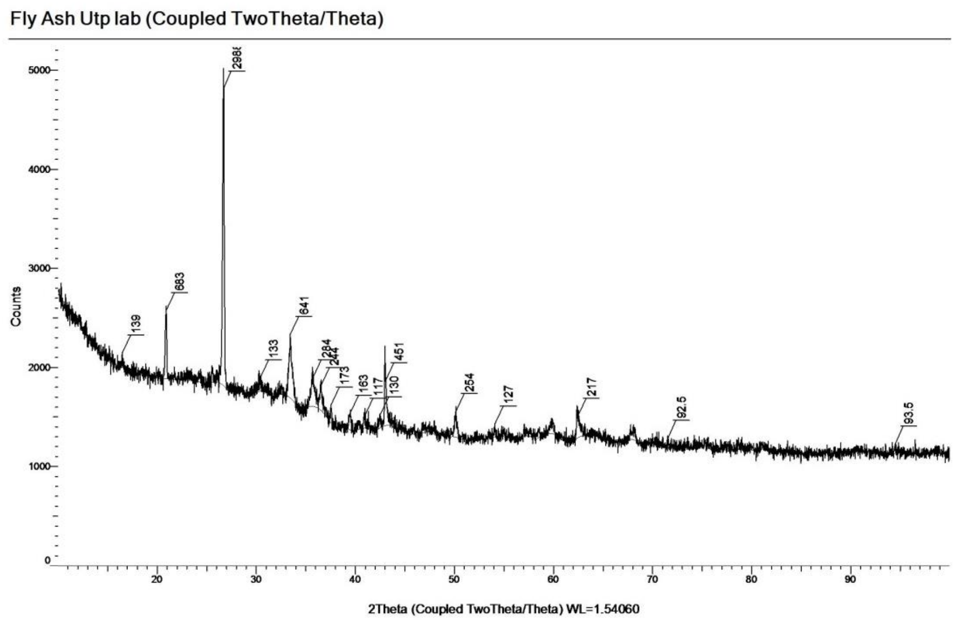



Figure 18: XRD analysis of Class F Fly Ash

Figure 18 shows XRD analysis of Class F Fly Ash. It is observed that the highest peak shows the presence of quartz. Meanwhile the other peaks are not so obvious. This is due to the generally spherical shape of fly ash particles which gives a good consolidation property which contributes to low permeability of the cement (Abdullah et al., 2011). To confirm this statement, SEM analysis was conducted on this fly ash sample and the result is shown in the next section.

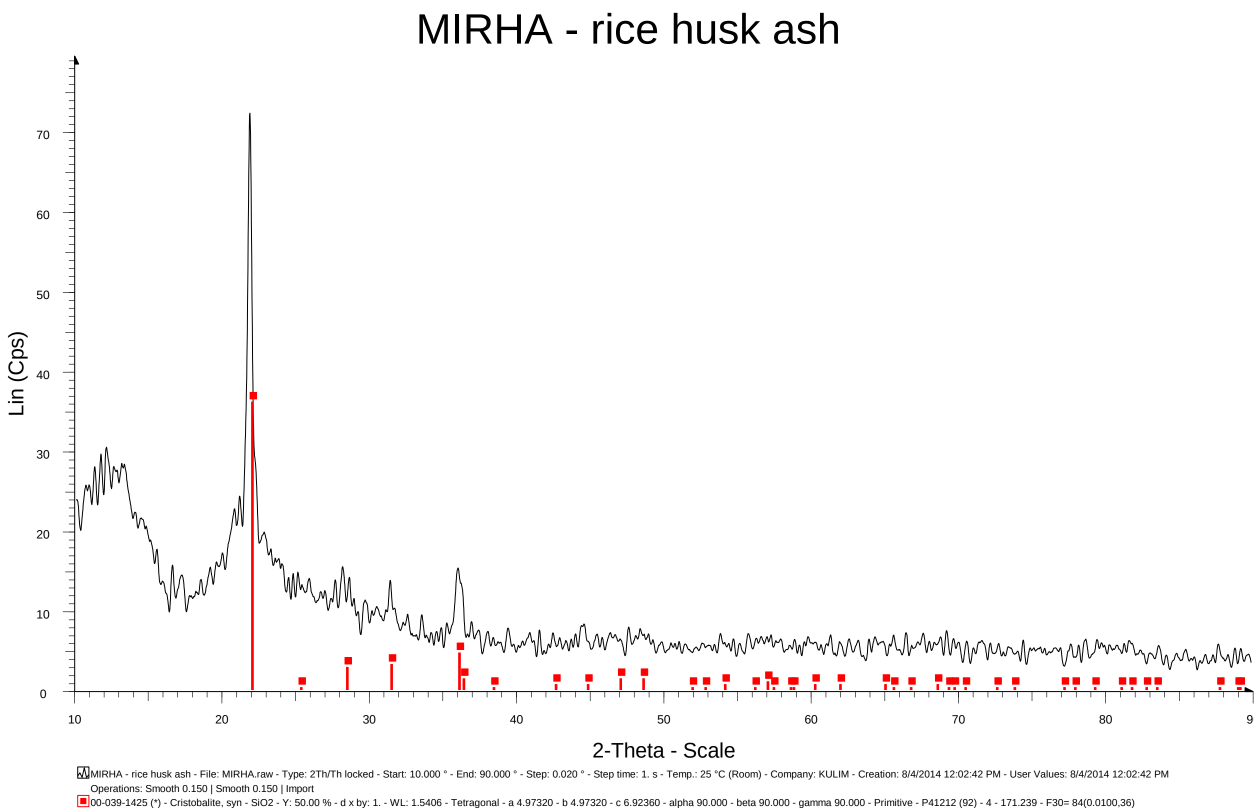



**Figure 19: XRD analysis of MIRHA**

Figure 19 shows XRD analysis of MIRHA. The result shows that $SiO_2$ compound exist in the form of cristobalite crystal. Cristobalite is a tetragonal crystalline which forms due to burning of rice husk. Burning the rice husk at 800°C, the ash convert into cristobalite (Nuruddin et al., 2009).

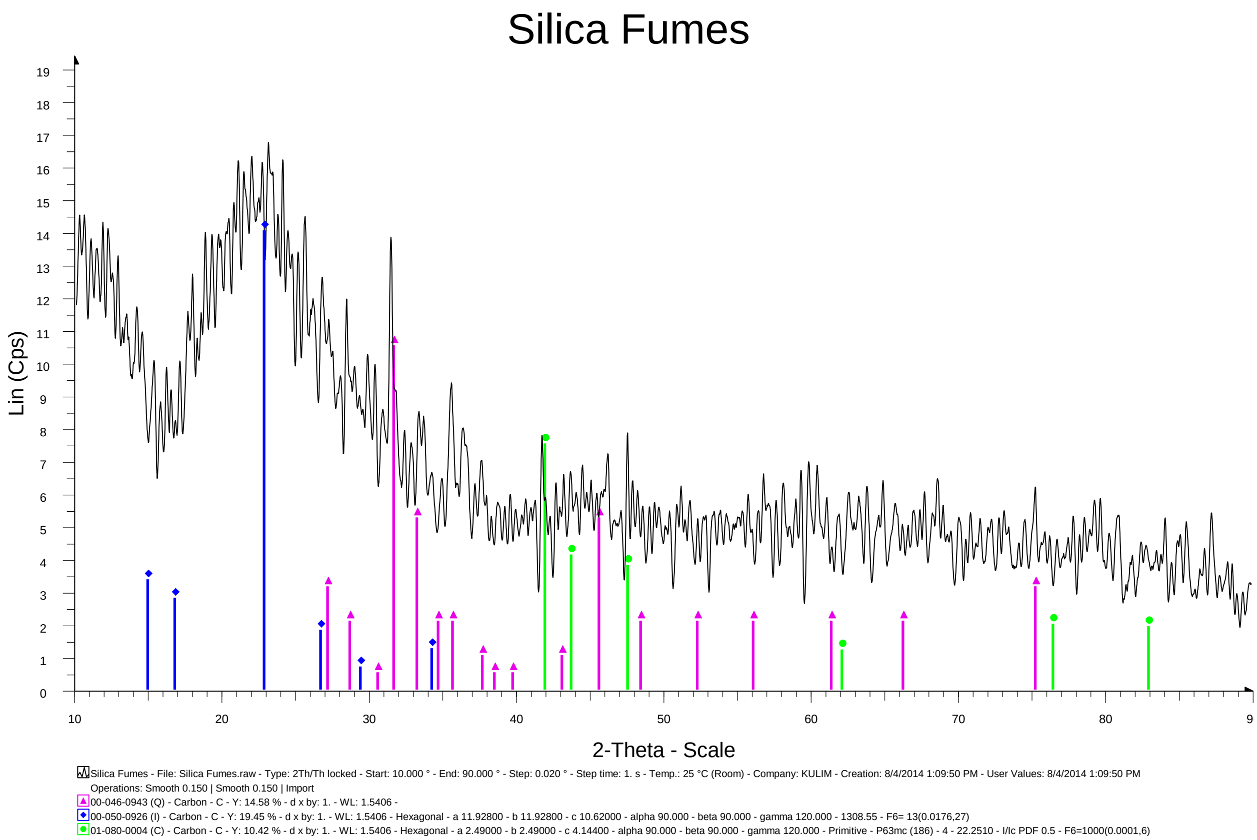



**Figure 20: XRD analysis of silica fumes**

Figure 20 shows the diffraction pattern for silica fumes. It can be notified that silica fumes are mainly built with amorphous quartz and cristobalite which are in the forms of SiO2. Cristobalite is in crystal forms whereas amorphous quartz in non-crystalline allotropic form. Amorphous silica is more flexible and easy to work on with while cristobalite requires high amount of activation energy to form.

### *4.2.3 Scanning Electron Microscope (SEM)*

Additional test which is, SEM analysis was conducted to study the morphology of Class F Fly Ash.

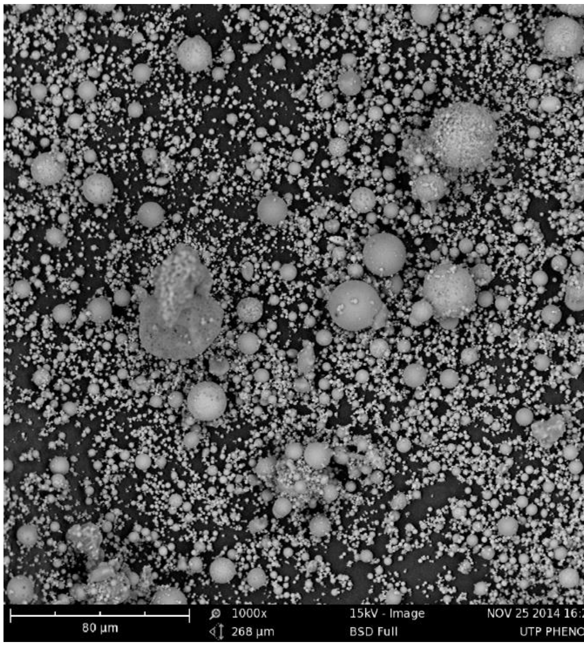



Figure 21: SEM analysis of Class F Fly Ash (1000x magnification)

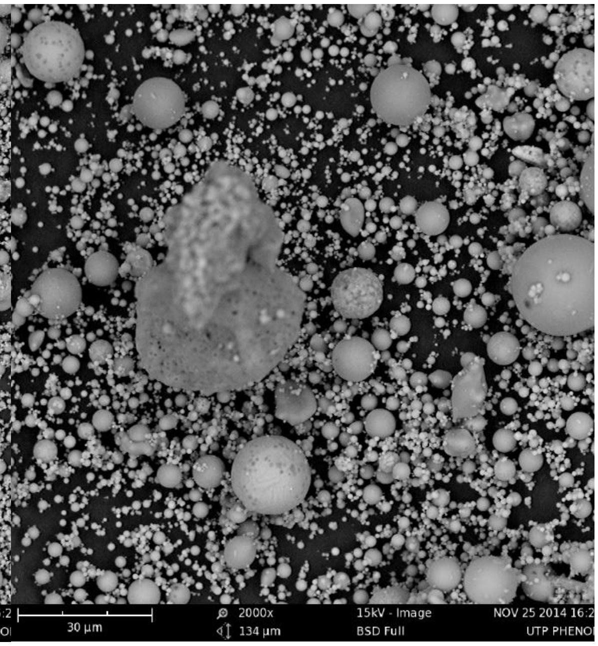



Figure 22: SEM analysis of Class F Fly Ash (2000x magnification)

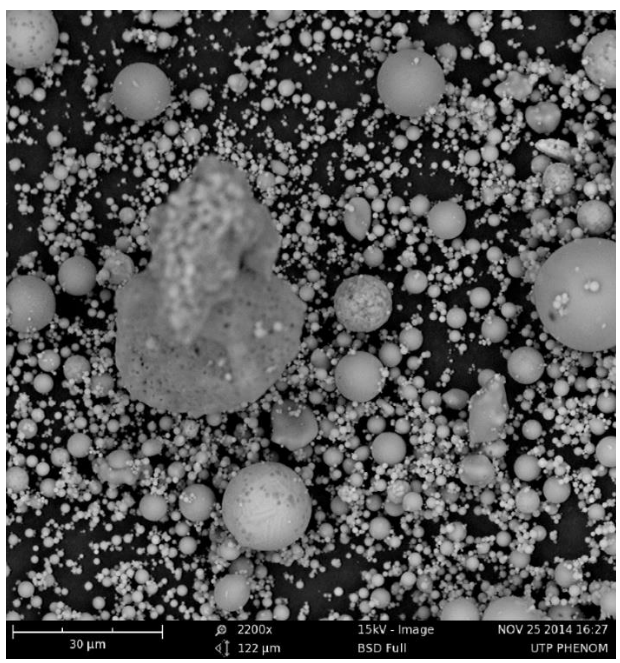



Figure 23: SEM analysis of Class F Fly Ash (2200x magnification)

Scanning Electron Microscope (SEM) shows the morphology of fly ash. Based on the result, the fly ash samples are composed of small, spherical materials with high regularity. According to (Ismail, Hussin, & Idris, 2007), fly ash sample consist of cenospheres particles with diameter ranging from 2µm to 14µm.

# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

## 5.1 RELEVANCY TO THE OBJECTIVE

The usage of fly ash and MIRHA as raw materials in geopolymer cement is definitely advantageous in reducing environmental pollution as it has been widely studied previously. Based on the result obtained, it can be concluded that:

1) Size of MIRHA particles gives significant effect on compressive strength of geopolymer cement. In Experiment 1, 300µM MIRHA particles give better compressive strength as compared with 600µM MIRHA particles.

2) Addition of MIRHA together with fly ash in geopolymer cement contributes in formation of geopolymer matrix of the cement, thus improves the compressive strength. In Experiment 2, 40% MIRHA and 60% FA proportion gives the highest compressive strength as compared with 20% MIRHA, 80% FA and 30% MIRHA, 70% FA, provided MIRHA particle size distribution is 300µM. In Experiment 1, when using 600µM MIRHA, the highest compressive strength obtained is at 30% MIRHA, 70% FA and it degrades as the proportion of MIRHA is increased further. This concludes that the proportion of MIRHA included also depending on the MIRHA particle size distribution.

3) Addition of silica fumes in geopolymer cement improves the compressive strength of the cement. As compared with the sample of 0% silica fumes, the inclusion of 5% silica fumes gives significant increase in compressive strength. However, further increasing silica fumes to 10% and 15% do not gives significant increase in compressive strength. This is supported by the idea that

silica fumes contributes significantly in compressive strength of concrete by increase the bonding between cement and aggregates but the effect is non-significant in cement.

4) Based on FTIR analysis, the presence of C=C=C double bond gives the reason of improved compressive strength with addition of MIRHA and silica fumes. XRD analysis shows MIRHA is rich in crystalline while silica fumes contain crystalline and non-crystalline materials. SEM images of Class F fly ash show that the shapes of fly ash particles are regular and spherical. The particle size distribution of fly ash also mostly fines. This is an indication that fly ash is a good material for cement as it is packed densely , indirectly reduces porosity of the cement and forms high strength cement.

In conclusion, the combination of MIRHA and FA as raw materials in geopolymer cement can be utilized in the industry as the compressive strength obtained in this project is up to 5.67MPa which exceeds the compressive strength of cement for most well applications which is 500psi or 3.5MPa. Any further improvement in compressive strength is believed can be done by applying the recommendations given in the next section.

## 5.2 SUGGESTED FUTURE WORK FOR EXPANSION AND CONTINUATION

In order to improve compressive strength of geopolymer cement, these modifications are recommended:

1) Extending curing time to be longer to enable cement to hydrates completely and compressive strength development to be higher.
2) Utilize the usage of nano-silica. The usage of nano silica can reduce the porosity and permeability of cement hence, improving compressive strength,
3) Study of physical properties of the geopolymer cement such as thickening time and fluid loss properties should be done to compare the result with API standard of class G oil well cement.
4) Curing the cement at reservoir temperature instead of atmospheric condition will give more accurate and improved result of strength development.
5) SEM analysis should be done on cement sample to identify the morphology of the cement and supporting the result of compressive strength obtained.
6) Curing the cement using HPHT curing chamber will determine the suitability of MIRHA and fly ash geopolymer cement in HPHT wells.

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# APPENDIX

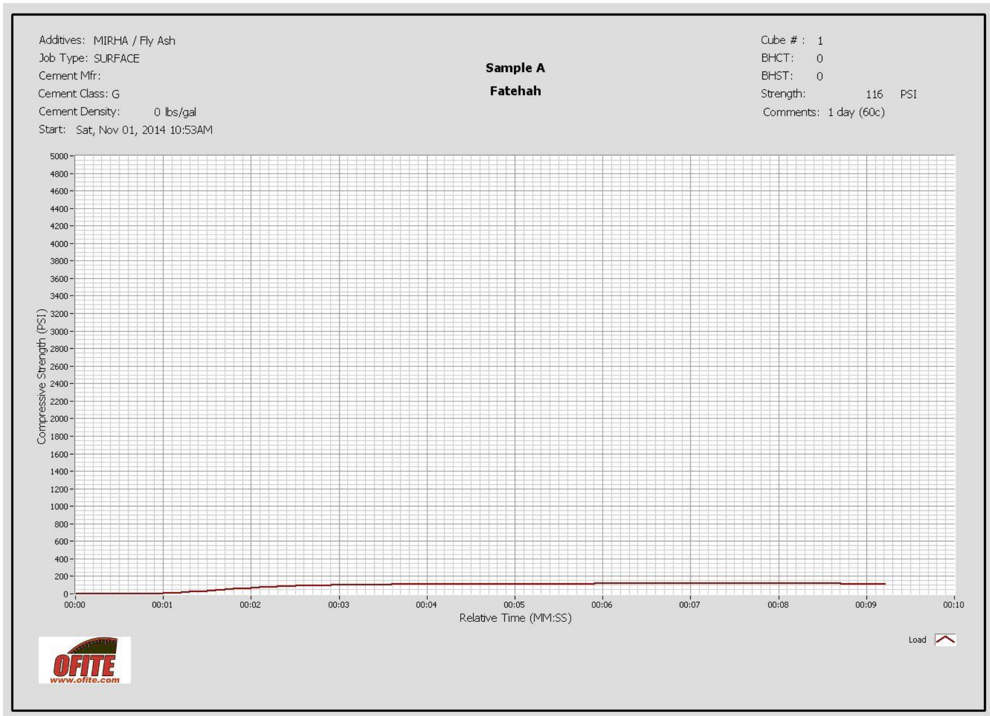


Figure 24: Compressive Strength Test for 600µM MIRHA Sample A ( 60%FA ; 40% MIRHA )

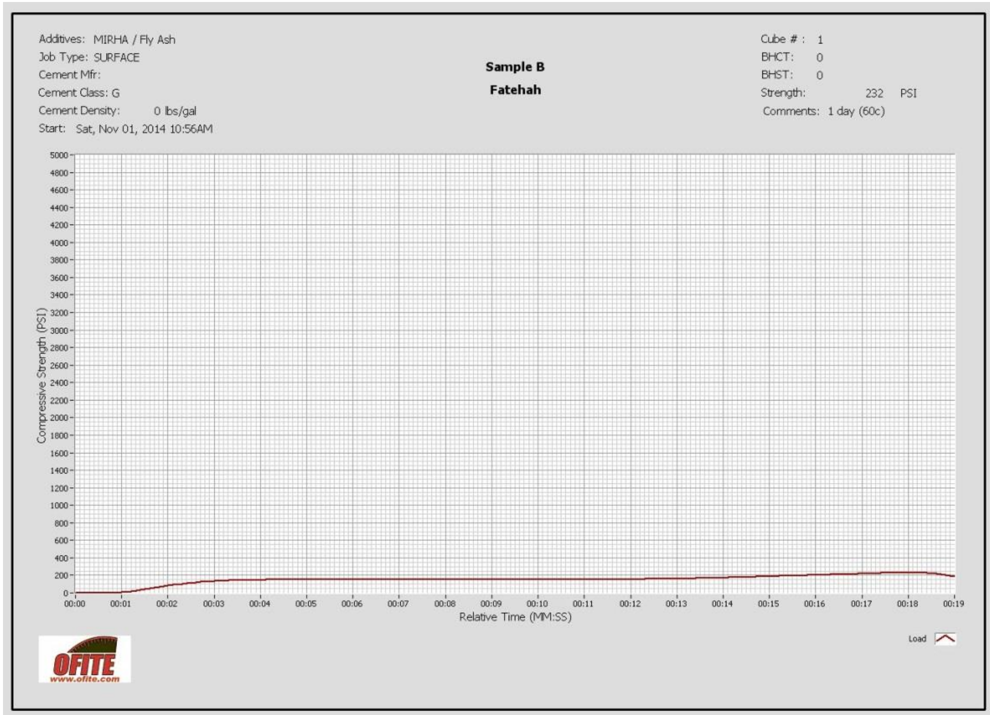


Figure 25: Compressive Strength Test for 600µM MIRHA Sample B (70% FA; 30% MIRHA)

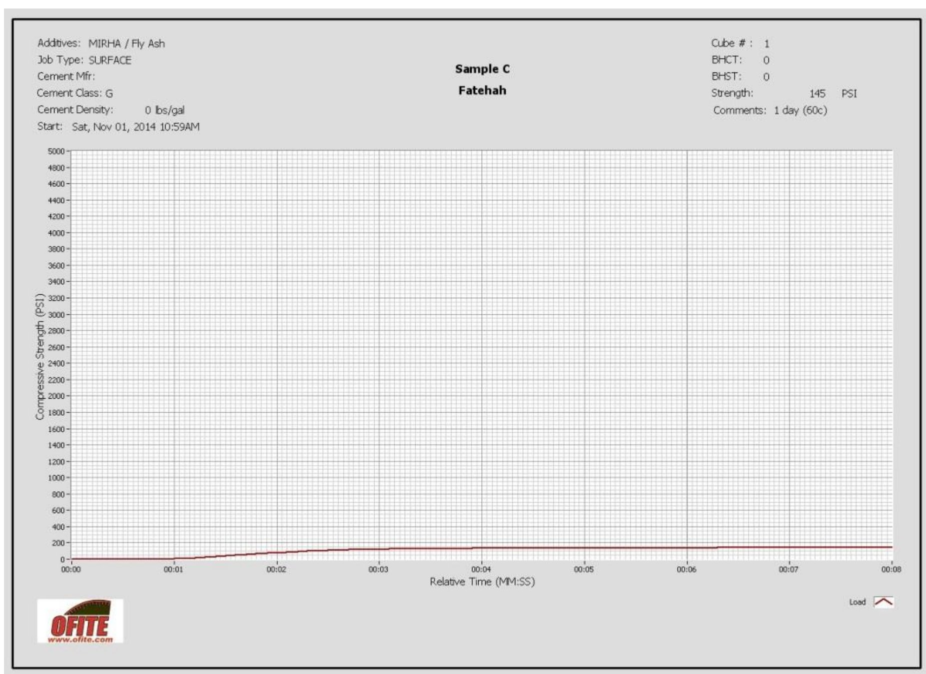


Figure 26: Compressive Strength Test for 600µM MIRHA Sample C (80%FA; 20% MIRHA)

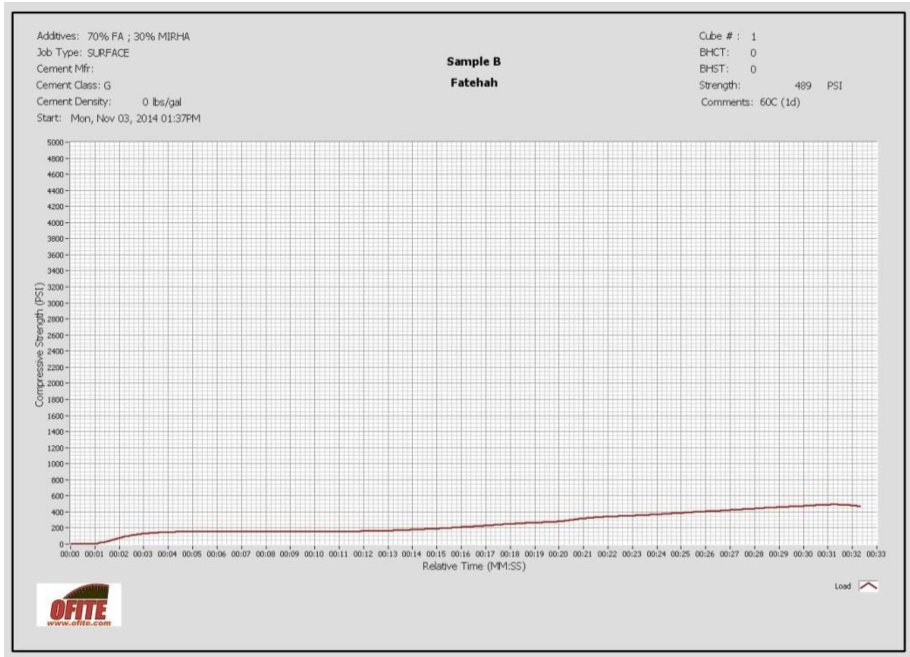


Figure 27: Compressive Strength Test for 300µM MIRHA Sample B (70%FA; 30% MIRHA)

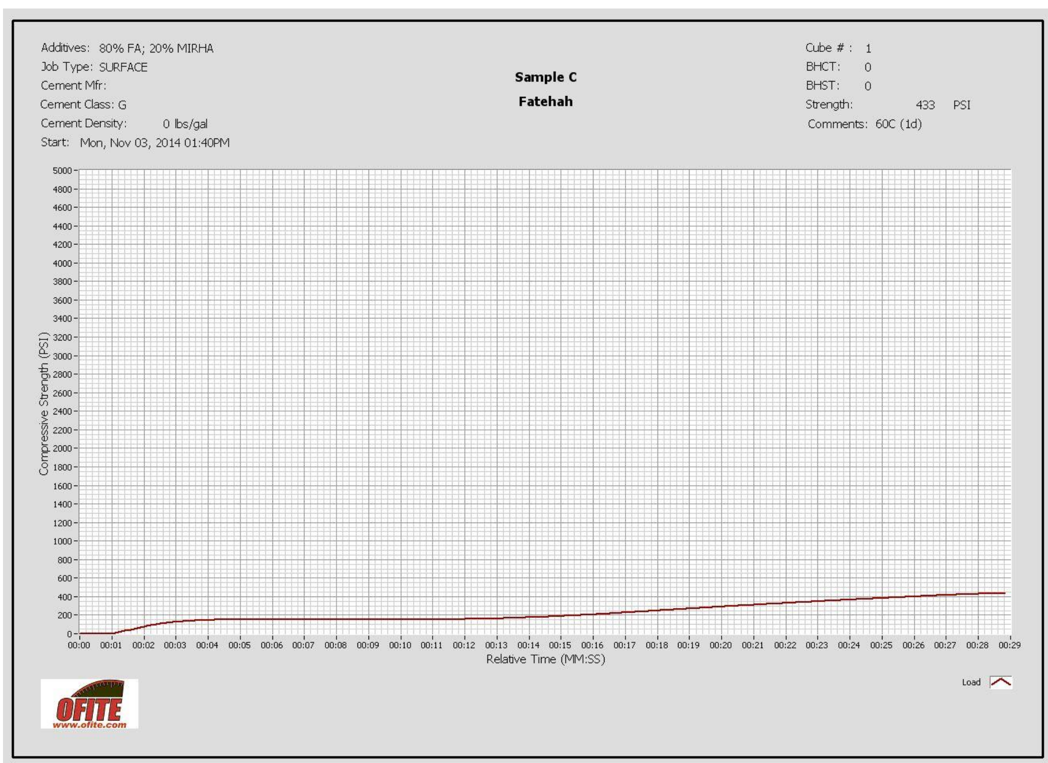


Figure 28: Compressive Strength Test for 300µM MIRHA Sample C (80%FA; 20% MIRHA)