Study on the Effect of Micro-Incinerated Rice Husk Ash (MIRHA) and Fly Ash (FA) in Geopolymer Cement

By

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12795

Dissertation submitted in partial fulfillment of the requirements for the
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CERTIFICATION OF APPROVAL

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Petroleum Engineering Programme
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In partial fulfillment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(PETROLEUM ENGINEERING)

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
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CERTIFICATION OF ORIGINALITY

This is to certify that I, Norsharmila binti Abdul Wahab (I/C No: 901204-01-5270), am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

The objective of this research is to study Fly Ash (FA) and Micro Incinerated Rice Husk Ash (MIRHA) as a cement binder replacement for Ordinary Portland cement (OPC). Both Fly Ash and MIRHA are categorized as pozzolonic materials in which when combined with calcium hydroxide, will exhibits cementitious properties. This supplementary cementitious material is proven to be effective to meet most of the requirement of durable concrete as well as cement. In the modern oil and gas industry, the utilization of both these materials as cement blend is gaining the attention of many. When compared to OPC, its application is generally cheaper, reduce the environmental effects especially on carbon dioxide (CO_2) emission and improve the ordinary cement blend. Both materials are easily obtained from waste or by-products generated through industrial and agricultural activities. MIRHA was mixed with FA by the ratio of 1:1 and 3:7 without any addition of OPC, fine aggregate or coarse aggregates. The effect of curing time for 3, 7 and 14 days, water to binder ratio (w/b), water ratio and different mixture composition were studied through the observation of the final compressive strength result of the samples. The project is solely based on experimental analysis. The laboratory works will be carried out in Universiti Teknologi PETRONAS (UTP) Petroleum Engineering and Civil Engineering laboratories. The experiments start from the incineration process to retrieve MIRHA and Rice Husk Ash (RHA), sieving, mixing, blending of the raw material and finally compressive strength test. The results indicate that the compressive strength development was the highest for batch A3 at 5 MPa by the 14^{th} day, with 30wt.% MIRHA to 70wt.% FA, 10% water and w/b ratio of 0.95 in which the ratio of MIRHA and water was the lowest. Though the targeted compressive strength was no achieved, it was identified that the reduced amount of MIRHA and water appear to be the main contributor to the increasing compressive strength of geopolymer binder.

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABBREVIATION AND NOMENCLATURES

FA	: Fly Ash
FTIR	: Fourier Transform Infrared Spectroscopy
MIRHA	: Micro Incinerated Rice Husk Ash
MPa	: Mega Pascal
NaOH	: Sodium Hydroxide
Na_2SiO_3	: Sodium Silicate
OPC	: Ordinary Portland cement
SiO_2	: Silicon Oxide
W/b	: Water to Binder Ratio
Wt.%	: Weight Percent by Mass

CHAPTER 1

INTRODUCTION

1.1 Project Background

Low cement quality and improper cementing job can jeopardize safety and causing huge amount of economic losses. Oil spills such as the recent Gulf of Mexico deep water horizon oil spills are some of the causes of oil loss from the global reserve, beside economic losses, oil spills cause environmental disasters particularly in marine habitats because of toxic substances. It is reported that the main cause of the tragedy was faulty cement work allowing the wall supporting the steel casing to come apart (Weiss & Donn, 2010). The industries have been spending billions of dollars to invent more technologically advanced materials to enhance cementing job and to minimize financial losses. Nonetheless, the fact remains that it is virtually impossible to solve every new problem that may arise.

The most common type of cement used in the industries is ordinary Portland cement (OPC) (Davidovits, 1999). It was produced commonly from limestone and either clay or shale. Differs from concrete and mortar, cement acts as a binder to hold materials together. It was the main component to produce mortar and concrete mixtures along with the combination of sand, aggregates and water. According to several standards, OPC is produced by grinding 90% of Portland cement clinker with limited amount of calcium sulfate and 5% minor constituents. It should consist of at least two-thirds calcium silicates by mass, CaO-SiO_2, aluminum and other compounds. The fraction of CaO to SiO_2 shall not be less than 2 while magnesium oxide content, MgO, shall not excedd 5% by mass (Bakri *et al*., 2012). Over the last century, OPC was one of the lowest cost materials widely used throughout the world. With rapid development and rises of new building everyday, the demand for OPC continues to increase. This situation leads to high concerned on the depleting natural resources to produce that huge amount of OPC as well as the increasing price.

In oil and gas industries, cementing is part of the process of preparing a well for further drilling, production or abandonment. It was done by pumping cement slurry into the well to displace the drilling fluids and left to harden in its designated location. Cementing may be used for a number of different reasons for protecting and sealing the wellbore. Most commonly, it was used to permanently shut off water breakthrough into the well. During the completion process of prospective production well, cementing is conducted to seal the annulus after the casing string has been run into wellbore. In directional drilling, cementing is used to plug an existing well to enable another directional drilling operation from that point. Additionally, cementing is used to plug a well for abandonment. Cementing is performed when the cement slurry is deployed into the well via pumps, displacing the drilling fluid in the well, and eventually replacing them with cement. The cement then flows through the casing to the bottom of the wellbore. From there it fills in the space between the casing and the formation and hardens. As a result, it seals and preventing materials from entering the well, as well as positions the casing in place ("How Does Cementing Work", 2013).

Nowadays, in line with the nations's effort towards ecofriendly environment, large number of researches are directed towards the utilization of waste material and natural resources. For cementing, the development and use of blended cements termed as "inorganic polymer" or "geopolymer" is growing rapidly (Duzson *et al*., 2006). Pozzolans, from the industrial and agricultural by-products such as Fly Ash (FA) and Micro Incinerated Rice Husk Ash (MIRHA) specifically; Palm Oil Frond Ash (POFA) and Silica Fumes generally, are receiving more attention since their usage provide comparable performance to traditional cementitious binder in range of application, but with added advantage of significantly reduced greenhouse emission. Their abundant existence caused it to be generally cheaper (Duzson *et al*., 2006). Pozzolon is a material which when combined with calcium hydroxide, exhibits cementitious properties. In addition, their uses also proven to be applicable since they exhibit excellent durability characteristic to act as cement. Geopolymers can exhibit a wide variety of properties and characteristics depending on the raw material selection and processing conditions, including high compressive strength, low shrinkage, fast or low setting, acid resistance, fire resistance and low thermal conductivity (Akyuz & Permezei, 2002). Numerous researches have also shown that the use of Fly Ash and Micro Incinerated Rice Husk Ash enhance the properties of cement and concrete. Blended cements, though not altogether a new concept, are in the forefront of durable building materials.

1.2 Problem Statement

The millennium era has resulted in rapid rises of building and bigger construction industries. This situation leads to the increase in demand for OPC all over the world. As a result, the sources of raw material (limestone) to produce OPC decreases proportionally while the cost of the material increasing every day. At the same time, it is also claimed that the cement industry contribute to fast polluting environment through the emission of Carbon Dioxide, CO_2 gas. The cement industry is responsible for about 7% of total CO_2 emission, since one ton of Portland cement emits approximately one ton of CO_2 into the atmosphere (Mc Caffrey, 2002; Davidovits, 1994; Bhikshma *et al*., 2012). As such, alternative materials have to be introduced to replace OPC as cement and binder. The problem statements are listed below:

1. Natural sources of raw material to produce OPC which is limestone are decreasing although the demand increases.
2. OPC usage is responsible for 7% of total CO_2 emission in the world. Approximately, 1 ton of OPC produced will emits one ton of CO_2.
3. Increasing cost of OPC due to rapid development, high demand and limited limestone sources left.
4. Increasing demand on higher durability cement and binder.

Eventually, the project will give a significant impact towards the utilization of improved alternative raw materials in terms of environmental friendly, cost effective and durability as cement. The behavior of the material chosen will be analyzed for optimum usage. Hopefully, the knowledge gained could ultimately allow the optimization of blended oil well cements, leading both to ecological and economic benefits.

1.3 Scope of Study and Objectives

Industrial by-products are good options for the problems. With regard to various types of by-products listed earlier, the study will only focuses on MIRHA and Fly Ash. Nowadays, Fly Ash and MIRHA application as the components in cement and thus replacing the normal concrete blend has captured the attentions of many researches. The use of Fly Ash and Micro Incinerated Rice Husk Ash will definitely help in reducing significant amount of cost, improve the properties of the cement blend and at the same time respond to environmental responsibility. The process design and synthesis of Fly-Ash-MIRHA-based geopolymers will continue to undergo intense research and development until the application in the industry has been optimized.

The main objective of the whole project is to study the effect of Fly Ash and MIRHA blend as cement binder. These two materials are abundantly found around Malaysia at cheaper cost or even for free. Not to forget, the use of these by-products will help to reduce the need for dumping job thus lessens the environmental pollution. Hence, it is expected that the blend between MIRHA and FA will result in improvement in durability of the cement paste. The study will include the effect of curing time chosen as 3, 7 and 14 days for the development of compressive strength. Additionally, water to binder ratio (w/b), water ratio and different composition of raw material’s effects are also studied. 0.40 and 0.95 w/b ratio were used with 60% and 10% water inclusion while source material is varied from 50% FA to 50% MIRHA and 70% FA to 30% MIRHA. The compressive strength results will be an indicator on the influence of these manipulative variables. From this, improved understanding on geopolymer properties can be developed. Finally, based from the result, the study will conclude the feasibility and limitations of using MIRHA and FA in geopolymer cement.

The specific objectives of this study are:

1. To replace the existing use of OPC as main ingredient in cement blend, being environmental friendly and save cost.
2. To investigate the effect of curing time, raw material composition, water ratio and alkaline solution ratio to the compressive strength of geopolymer.
3. To develop an improved understanding of the geopolymer properties of Fly Ash and Micro Incinerated Rice Husk Ash as cement binder through compressive strength development.
4. To determine the feasibility of using Fly Ash and MIRHA as cement binder.

It is practicable to conduct the study within the time frame as geopolymer cement has the characteristic of quick compressive strength development. Hence, experimental job can be done within the allocated time frame. Furthermore, FA and MIRHA can be easily obtained from UTP's laboratories. Study can be done in the Mud and Cementing Laboratory located in UTP as it well equipped with the equipment needed.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Geopolymer Cement

Geopolymers' theory was first developed by Davidovits (Ali Nazari, 2011). Geopolymer is a term used to describe inorganic polymers based on aluminosilicate, which can be produced by reacting pozzolonic compounds or aluminosilicate source materials with highly alkaline solutions (Kong *et al*., 2007). The aluminosilicate source can be a natural mineral or by-product materials, for instance kaolinite, clay, Fly Ash, silica fume, rice husk, or slag (Al Bakri Abdullah *et al.*, 2012). Geopolymer is an inorganic polymer that can be formed at room temperature by using industrial waste or by-products as source materials to form a solid binder that looks like and perform similar function to OPC (Zeobond, 2012). Normally, concrete is made of the basic ingredients of hydraulic cement, namely Portland cement, mineral aggregates, and water (Mehta & Monteiro, 2006) with typical densities of 2000-2500 kg/m^3 for concrete and 1800-2200 kg/m^3 for mortar (CEMEX UK Operations Ltd, 2009). However recently, the potential for replacing the ordinary Portland cement with geopolymer has been explored extensively. It was used in variety of applications not only in construction industry but also as binder to hold the casing in place during drilling operation. Numerous research publications related to geopolymers have been released, with some reporting on chemical composition aspects or reaction processes while others present results related to mechanical properties and durability.

The main process dissimilarity between OPC and geopolymer cement is that OPC depend on a high-energy manufacturing process that imparts high potential energy to the material through calcination. This means the activated material will respond readily with a low energy material for example water. On the other hand, geopolymer binder uses very low energy materials such as Fly Ashes, slags or other industrial wastes and a small amount of high chemical energy materials (alkali hydroxides) to initiate reaction only at the surface of particles to acts as glue (Zeobond, 2012). The process of geopolymerization starts with hydrolysis on the solid surface through exchange of H^+ with movalent cations (K^+ or Na^+) from the bulk solution (Davidovits, 1999).

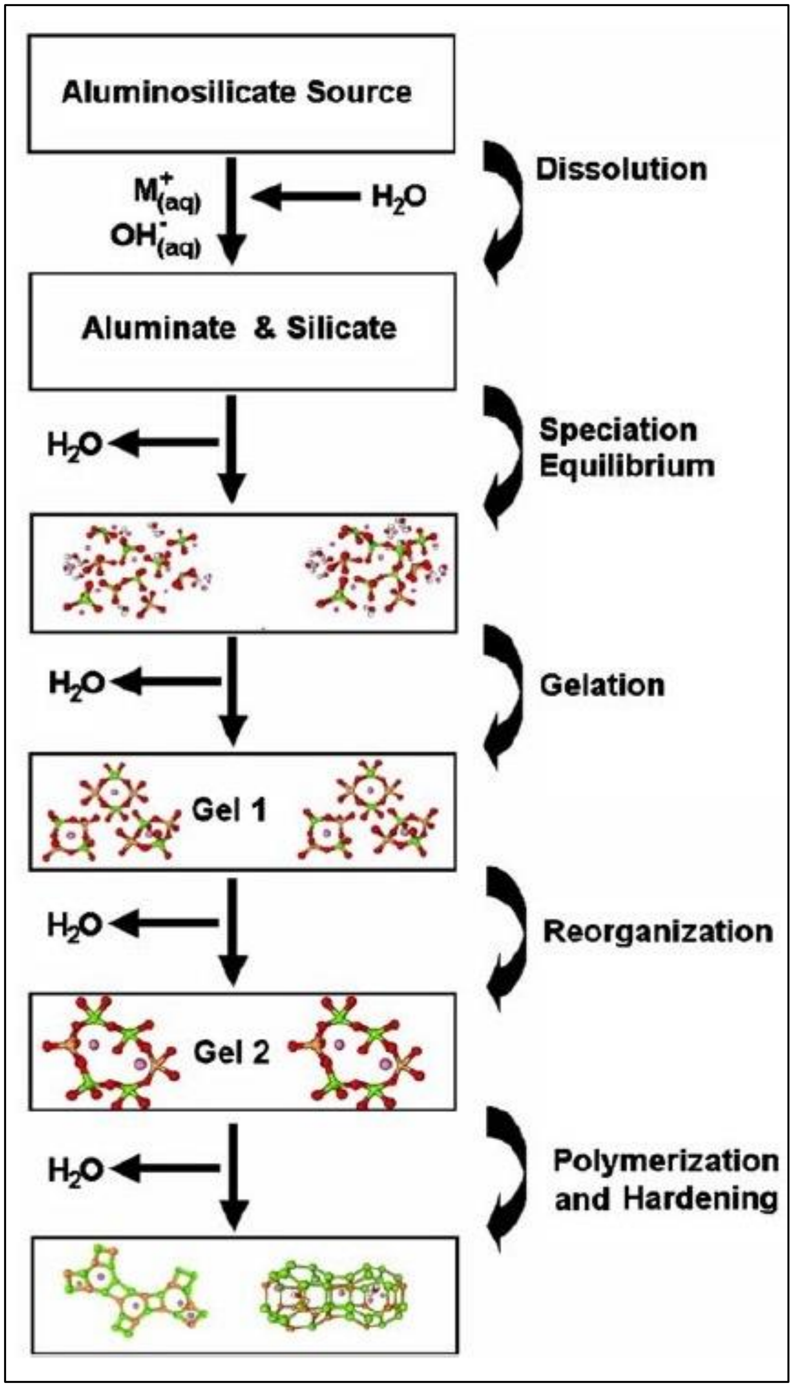

Figure 1: Conceptual Model for Geopolymerization

The subsequent step is believed to be the continuous dissolution of aluminosilicate recursors by the breaking of Si-O-Si or Si-O-Al bonds from the solution particles to form the reactive precursors $Si(OH)_4$ and $Al(OH)_4^-$ in solution (Xu & Van deventer, 2000). The dissolution stage occurs concomitantly with precipitation on the solid surface, which is known as the rearrangement of silicates and aluminates (Lee & van Deventer, 2002). Next, polymerization occurs through condensation of Si and Al, dismissing water and leaving unreacted extra alkali in the liquid phase (Davidovits, 1999; Sindhunata *et al.,* 2006). **Figure 1** presents a highly simplified reaction mechanism for geopolymerization process. The reaction mechanism shown outlines the key processes occurring in the transformation or curing of solid aluminosilicate source into final state as geopolymer (Duzson *et al*., 2006).

The study concerned on the geopolymers properties specifically compressive strength development of two source materials; Fly Ash and MIRHA. Compressive strength is the most basic test for a geopolymer to succeed before proceeding to the next stage of work. Other main properties of geopolymers are low permeability, resistance to acid attack, good resistance to freeze-thaw cycles, tendency to drastically decrease the mobility of most heavy ions contained within the geopolymeric structure (Jaarsveld *et al*., 1997), low shrinkage, fast of low setting, fire resistance and low thermal conductivity (Duzson *et al*., 2006). Despite this wide variety of commonly outlined attributes, these properties are not necessarily inherent to all geopolymeric formulations. Inorganic polymers should not be considered a universal panacea for all material selection problems, but rather a solution that may be tailored by correct mixing and processing design to optimize properties and/or reduce cost for a given application. The term 'geopolymer' is also commonly referred to as 'low-temperature aluminosilicate glass' (Rahier *et al*., 1996), 'alkali-activated cement' (Palomo & Fuente, 2003), 'geocement' (Krivenko, 1994), 'alkali-bonded ceramic' (Mallicoat *et al*., 2005), 'inorganic polymer concrete' (Sofi et al., 2006), and 'hydroceramic' (Bao *et al*., 2005). Despite this variety of nomenclature, these terms all describe materials synthesized utilizing the same chemistry. Supplementary cementitious materials observed from MIRHA and FA were prove to be effective to meet most of the requirement of durable concrete (Abdul Aziz *et al*., 2010).

This is also supported by the improved result in terms of strength at the low replacement level and at the later age from the use of ternary blend of OPC, RHA and FA (P. Chindaprasirt, 2008).

2.2 Alkaline Activators and Workability of Fresh Geopolymer

Alkaline liquid could be used to react with the source material to produce binders. These chemicals help in completing the activation of the source materials (Kusbiantoro *et al.*, 2012). The alkaline activation of material can be defined as a chemical development that provides a quick change of some specific structures, partly or totally amorphous, into compact cemented frameworks (Bakri *et al.*, 2012). The most common alkaline solutions used in producing Fly-Ash-MIRHA-based geopolymer are sodium hydroxide, NaOH and sodium silicate, Na_2SiO_3 (Bakri *et al*., 2010). The use of the sodium silicate solution of approximately 2 and sodium hydroxide solution with 97-98% purity is also recommended. The concentrations of the sodium hydroxide solution that can be used range from 8 to 16 M. however, it was found that 12 M NaOH solution gives the highest compressive strength result of all (Al Bakri Abdullah et al., 2012; Ali Nazari, 2011). It is also indicated that the use of a Na_2SiO_3/NaOH ratio of 2.5 resulted in highest compressive strength (Al Bakri Abdullah *et al.*, 2012).

Normally, water is added to increase the workability of the concrete. However, it will increase the porosity due the evaporation of water during curing process at elevated temperature (Sathia *et al*., 2008). Chindaprasirt (2008) discovered that the use of superplasticiser has an adverse effect on the strength og geopolymer. As such, extra water was chosen as it gives greater strength than the addition of superplasticiser. Kusbiantaro (2012) come out with the optimum composition for FA-MIRHA-based geopolymer concrete by using 10% of water by mass of raw material and 0.4 water to binder ratio.

2.3 Raw Materials

2.3.1 Fly Ash (FA)

Fly Ash is one of the waste residues generated during coal combustion at coal power station, and is composed of spherical micrometer-sized particles that would rise with the flue gases if not collected by dust collection system. (Guo & J. Reardon, 2012). The total amount of FA produced in the world has now reached 480 million tons annually, while the total OPC production in the world is reaching 3.3 billion tons in 2010 (Kusbiantoro *et al*., 2012). Based from the gap of these number, FA will contribute to an effective way to replace OPC production. Note that the composition of Fly Ash produced by different powerplants differs greatly, although it always contains a large amount of amorphous and crystalline silicates, aluminosilicates, and calcium oxide. This difference is related to the source of the parent coal, the combustion conditions in the furnace, and even the way of collecting Fly Ash from the flue gases (Qi & Hlavacek, 2005). Toxic elements include arsenic, beryllium, boron, cadmium, chromium, cobalt, lead, manganese, mercury, molybdenum, selenium, strontium, thalium, and vanadium (About Civil, 2013). FA is used as a source material to produce geopolymer because of its suiTable chemical compositions, favorable size, shape and consists mostly of glass, hollow and spherical particles (Kumar *et al.*, 2005). There are two typical categories of FA which are class C and class F as detailed in **Table 1**.

FA is the most common pozzolan and is being used worldwide in concrete works. There are known for their porperties, which are better than those of normal concrete due to their lower creep (Wallah, 2010), lower shrinkage (Hardjito *et al.* , 2004), better fire and acid resistance (Guo *et al.*, 2010), and resistance to sulfate attack (Harditjo et al., 2005; Brungs, 2005). The replacement of cement with Fly Ash up 10% to 30% by mass could help to reduce global cement consumption, which result in the reduction of CO_2 emissions associated with cement manufacturing (Gartner, 2004). The addition of Fly Ash to OPC, particularly class F Fly Ash with low calcium content, is found to lessen the porosity and fluid permeability in the cement paste (Dhir & Byars, 1993).

Table 1: Comparison between Class C and Class F of Fly Ash

	Class C	**Class F**
Source	Produced from the burning of younger lignite or subbituminous coal	Produced from the burning of older anthracide or bituminous coal
Lime content	More than 15%	Less than 15%
Requirement in order to produce cementitious properties	• Have some self-cementing properties. • In the existence of water, it will harden and develop strength over time.	• Require the presence of water along with cementing agent for example OPC, quicklime, etc • Require activator
Common application	• Prestressed application • In situation where higher early strength are important • Soil stabilization	• Ideal cementitious material in mass concrete and high strength mixtures • Answer to a wide range of summer concreting problem • In condition where concrete may be exposed to sulfate ion in soil and ground water.
Adapted from Headwater Resources (2005)		

Futhermore, the cost of the geopolymer materials that are derived from Fly Ash is generally lower than OPC by a factor of about 10% to 30% (Duzson *et al.*, 2006). This experiment will be conducted using Class C Fly Ash as it is more abundantly found nowadays than Class F Fly Ash. MIRHA was brought together in the mixture to replace certain percentage of Fly Ash content as source material. The purpose is to adjust the SiO_2-Al_2O_3 ratio in the source material hence improvement expected on the interfacial transition zone and strength properties of geopolymer cement (Kusbiantoro *et al.*, 2012).

2.3.2 Micro Incinerated Rice Husk Ash (MIRHA)

Rice Husk is one of the major agricultural by-products and is available in many parts of the world (P. Chindaprasirt, 2008). The total world production of Rice Husk has reached 130 million tons annually, with 446 thousands tons of them are produced from Malaysia (IRRI and FAOSTAT database, 2008). Currently, the disposal method used are by burning and dumping which create environmental pollution through the emission of greenhouse gasses. Hence, cement replacement would be a ‘green’ alternative to take care of this matter. In order to produce MIRHA, a specific set of temperature and duration of burning has to be maintained. It is a very fine material with average size ranges from 5 to 10µm. Rice husk contains high silica content generally more than 80-85% in the form of non-crystalline or amorphous silica. Therefore, it is considered as pozzolanic material and can be used as supplementary cementitous materials (Mehta, 1979). The used of these fine amorphous silica in the production of special cement and concrete mixes result in enhanced performance, high strength, low permeability concrete and higher resistance against cracking (Abdul Aziz *et al.*, 2010). Nevertheless, the research on producing Rice Husk Ash that can be used in concrete is not new. In 1973, Mehta investigate the effect of pyroprocessing on the pozzolonic reactivity of RHA. Since then, many attempts have been made to produce and use pozzolonic RHA in several countries around the world. The rice husk replacement of cement was found effective in improving resistance of concrete to sodium chloride attack (Abu Bakar *et al.*, 2012). The MIRHA contained concrete showed better compressive strength performance in sodium chloride solution comparing with the normal control concrete specimens. This clearly indicates a positive added high value from the use of both materials (Akyuz & Permezei, 2002). **Figure 2** shows rice husk before incineration process while **Figure 3** show ungrinded MIRHA obtained after incineration process.

Figure 2: Rice Husk before Incineration

Figure 3: MIRHA before Grind

2.4 Compressive Strength Development

Curing temperature has a significant effect on the compressive strength development because it affects specimens setting and hardening (Ali Nazari, 2011). When the curing temperature increases, polymerization becomes more rapid, and the concrete can gain 70% of its strength within 3 to 4 hours of curing. The compressive strength of dried cured geopolymer concrete is 15% higher than that of stem cured geopolymer (Bakri et al., 2012). Both curing time and temperature influence the result for compressive strength. Al Bakri *et al.* (2012) found that the optimum curing temperature of 60℃ gives the highest compressive strength. Studied conducted by Kusbiantaro *et al.* (2012) reported that higher strength development retained by MIRHA based geopolymer concrete in ambient curing. The enhancement on the compressive strength of MIRHA based geopolymer concrete was up to 22.34% higher than non-MIRHA based specimens placed in ambient curing. **Table 2** shows the compressive strength's range of standard API cements which served as benchmark for this study.

Table 2: Physical Requirement for API Cements

Well cement class:				A	B	C	G	H
Mix water, wt% of well cement:				46	46	56	44	38
Fineness tests (alternative methods):								
Turbidimeter (specified surface, minimum, m_2/kg):				150	160	220	—	—
Air permeability (specified surface, minimum, m_2/kg):				280	280	400	—	—
Free-fluid content, maximum, mL:				—	—	—	3.5	3.5
Compressive-strength test, 8-hour curing time	Schedule number, Table 7	Curing temp., °F (°C)	Curing pressure, psi (kPa)	Minimum Compressive Strength, psi (MPa)				
	—	100 (38)	Atmos.	250 (1.7)	200 (1.4)	300 (2.1)	300 (2.1)	300 (2.1)
	—	140 (60)	Atmos.	—	—	—	1,500 (10.3)	1,500 (10.3)
Compressive-strength test, 24-hour curing time	Schedule number, Table 7	Final curing temp., °F (°C)	Final curing pressure, psi (kPa)	Minimum Compressive Strength, psi (MPa)				
	—	100 (38)	Atmos.	1,800 (12.4)	1,500 (10.3)	2,000 (18.8)	—	—

Other than that, the strength of concrete us also influenced by the composition of cement, aggregates, water, and various admixtures. The ratio of water is the principal factor for defining concrete strength as shown in **Figure 4** (Alilou & Teshnehlab, 2012). When the water-cement ratio decreases, the compressive strength increases. However, as mention earlier, a certain minimum amount of water is required for the proper chemical action in the hardening of the concrete; extra water may provide workability but diminishes its strength (Alilou & Teshnehlab, 2012).

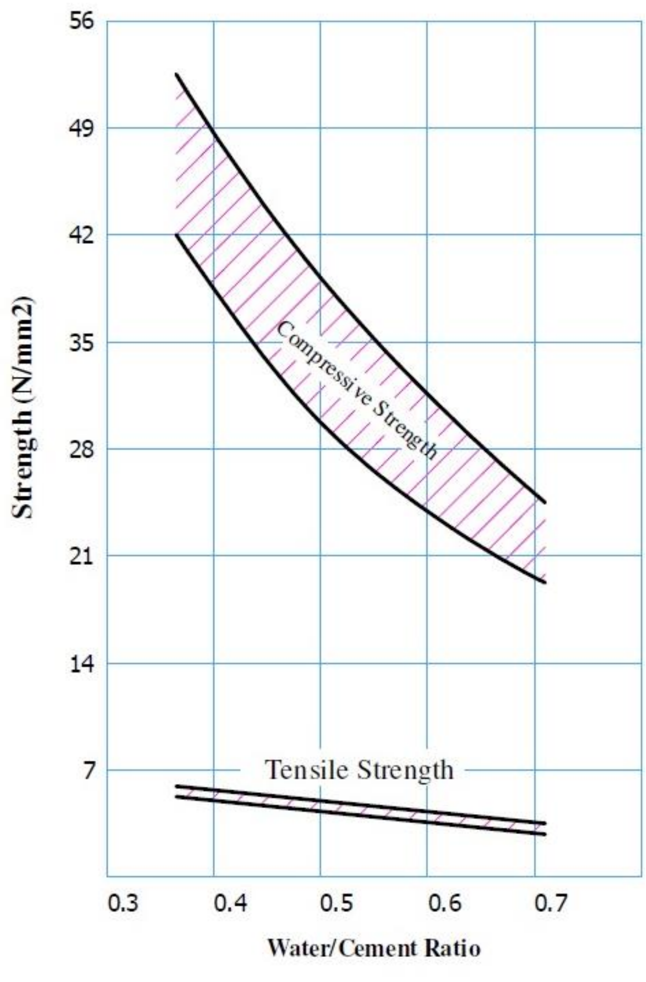

Figure 4: Effect of Water/Cement Ratio in Concrete Strength

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

With respect to the project's objectives listed earlier, several steps are taken in order to achieve it. The details of each activity will be elaborated in the next section of the report.

a. Use **only** Fly Ash and MIRHA to mix the cement paste without the addition of OPC, aggregates or fine sand. It is generally more environmentally friendly and cost saving.
b. Test the samples at different condition (curing time, different source material composition, alkaline solution ratio (w/b ratio) & water ratio using laboratory compressive strength machine for better understanding of the geopolymer properties and behavior.
c. Analyzed the effect of each variable parameter to the development of compressive strength of the cement.

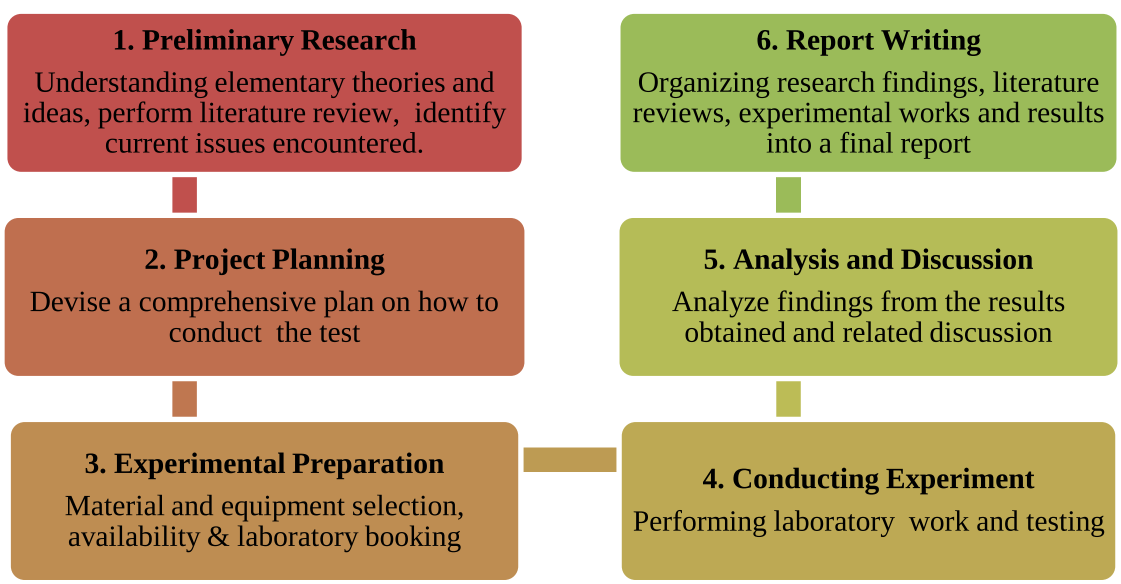

Figure 5: Research Methodology

3.2 Project Activities

3.2.1 Materials and Equipment Preparation

Only two raw materials will be involved in these experiments which are MIRHA and Fly Ash. Rice Husk is readily found in UTP laboratory. Microwave Incinerated Rice Husk Ash (MIRHA) was obtained through incineration process conducted using UTP-Microwave Incinerator at 600℃ incineration temperature for 1 day as in **Figure 6**. Then, it was grinded using 12 balls mill grinder machine for 2000 cycles to obtain finer ashes (refer to **Figure 7**). Meanwhile, class C Fly Ash in this research was obtained from Charcoal Factory operating in Taiping, Perak. Both of these materials are sieve up to 300µm to eliminate larger size particles. Equipment utilized during the experiment is shown in **Figure 8**.

Figure 6: Incineration Process

Figure 7: Grinding Process Using Ball Mill Grinder

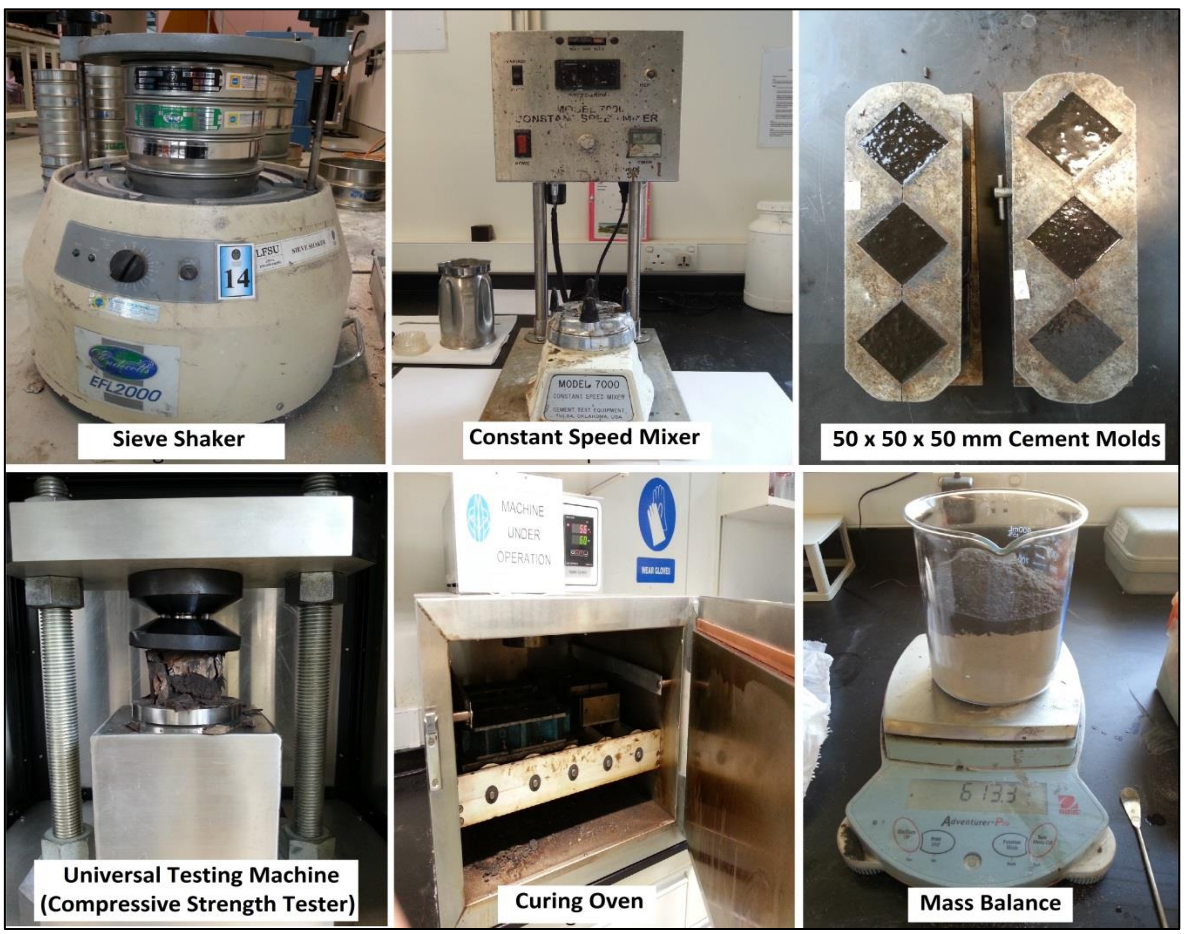

Figure 8: Laboratory Equipment

3.2.2 Laboratory Experiment

The experiment procedure was conducted as in **Figure 9**. The curing of the samples will took place in the curing oven for 1 day at 60℃ and continued to be cured at room temperature. The A1 and A2 batches of samples were mixed using 50wt.% of Fly Ash and 50wt.% of MIRHA while A3 batch was using 70wt.% Fly Ash and 30wt.% MIRHA. The details and comparison in the composition of each batch are shown in **Table 3** to **5**. Mix A1 are using 0.4 w/b ratio and 60% water while mix A2 and A3 of 0.95 w/b ratio and 10% water.

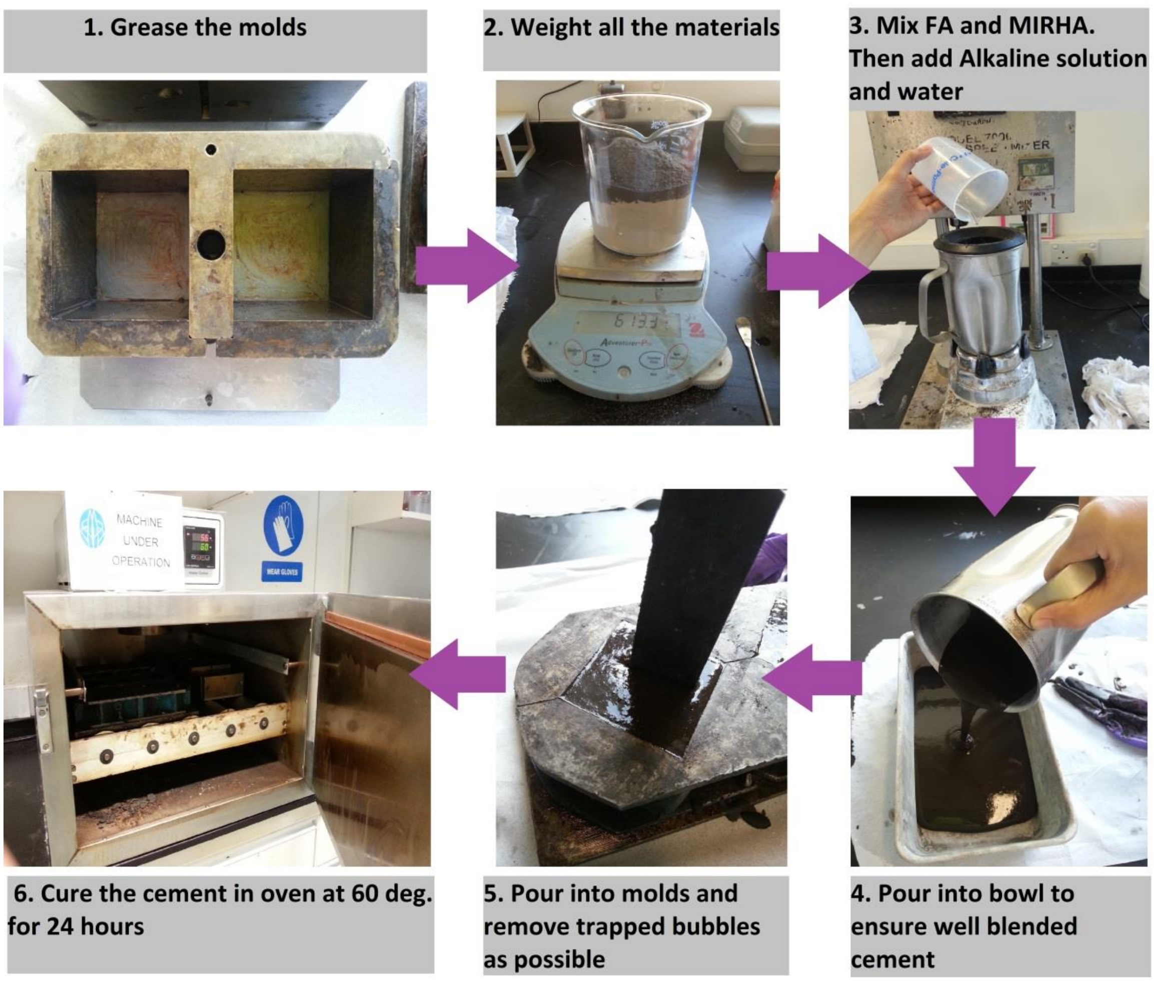

Figure 9: Experiment Procedures

Table 3: Mixture Proportion for Batch A1

Mix code	Proportion in kg/m3				
	Fly Ash	MIRHA	NaOH solution	Na_2SiO_3 solution	Extra Water
A1	175	175	41	103	210

Table 4: Mixture Proportion for Batch A2

Mix code	Proportion in kg/m3				
	Fly Ash	MIRHA	NaOH solution	Na_2SiO_3 solution	Extra Water
A2	175	175	96	237.5	35

Table 5: Mixture Proportion for Batch A3

Mix code	Proportion in kg/m3				
	Fly Ash	MIRHA	NaOH solution	Na_2SiO_3 solution	Extra Water
A3	245	105	96	237.5	35

3.2.3 Sample Testing

3.2.3.1 Curing Time and Curing Condition

Curing plays an important role on strength development and durability of the concrete. It is defined as a procedure for ensuring the hydration of the cement in newly-placed concrete (Curing Concrete, 2013). In this project, the sample will be tested for **3, 7** and maximum of **14 days** only. All these specimens will be initially placed in curing oven for 24 hours at 60℃ after mixing. The samples will then be cured in room temperature (25℃ to 30℃). The specimens will be constantly protected from direct sunlight and rainfall until the testing day for compressive strength analysis.

3.2.3.2 Compressive Strength

The compressive strength is a measure of the concrete's ability to resist loads which tend to crush it. The compression test shows the compressive strength of hardened concrete.

In this experiment, the cube specimen of size 50x50x50 mm will be used. The compression test shows the best possible strength concrete can reach in perfect conditions. The strength is measured in Megapascals (MPa). This test will be conducted using Universal Testing Machine at a rate of 4000lbf/min for 3 samples each with curing time of 3 days, 7 days and 14 days as shown in **Figure 10**. The output generated were graphs of compressive strength vs. time attached in **Appendix 1**.

Figure 10: Compressive Strength Test

3.2.3.3 Chemical Activator

Sodium hydroxide of 12M was use based as it was the optimum molarity proven to produce optimum compressive strength (Ali Nazari, 2011). The use of high molarities NaOH solution (such as 12M) could accelerate dissolution and hydrolysis but obstruct polycondensation (Z. Zuhua). Thus, 12M NaOH can be considered as the suitable solution for preparing geopolymer cement. NaOH solution was prepared by diluting 480g NaOH pellets with distilled water filling up to one liter of the volumetric flask. To prevent excess heat, NaOH solution was prepared one day before conducting the experiment. Sodium silicate solution is readily obtained in solution form with 98-99% concentration. In this experiment, w/b ratio refers to the ratio of both alkaline solutions to the raw material.

3.3 Gantt Chart and Key Milestones

The experiment has been conducted successfully within the time period allocated. Table 6 shows project Gantt chart while Table 7 shows the project milestones. Result and data analysis is discussed in **Chapter 4** of this report.

Table 6: Project's Gantt chart

Activities	Final Year Project (FYP I and FYP II) (Jan – Aug 2013)																											
	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
Selection of project topic																												
Data/theory gathering and literature review																												
Extended proposal preparation																												
Proposal defense																												
Sample preparation																												
Laboratory and equipment requisition																												
Submission of interim draft report																												
Interim report preparation																												
Laboratory experiment and analysis																												
Data collection and interpretation																												
Submission progress report																												
Comparison analysis against normal OPC to FA+RHA																												
Reports Preparation and Presentations																												

Table 7: Project's Key Milestones

Week	Project Milestones
3	Topic and title selection
7	Submission of extended proposal
9	Proposal defense presentation
14	Submission of interim report
15	Start of laboratory works
22	Submission of progress report
23	End of laboratory works
25	Submission of final draft report and technical paper
27	Poster presentation
28	Viva and submission of dissertation

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Results and Data Gathering

The basic indicator to the performance of this alternative raw material is the development of compressive strength for hardened cement. It serves as the fundamentals description on the quality of geopolymerization products (Kusbiantoro *et al*., 2012). **Figure 11-13** and **Table 8-10** show the compressive strength test result of hardened geopolymer cement according to the specified proportion stated earlier while **Appendix 1** provides each compressive strength test result of the samples. The maximum compressive strength obtained by the end of 14th days was 1.62MPa, 3.97MPa and 4.87Mpa for batch A1, A2 and A3 respectively although the targeted minimum compressive strength by 14 days is 20MPa. It can be observed that as the curing time increase, the compressive strength is also increased. However, the slope for the strength development is steeper (higher) from 3 to 7 days when compared with 7 to 14 days curing time for all three batches.

Figure 14 shows the compressive strength development comparison for batch A1, A2 and A3. Batch A1 with 50wt.% inclusion of MIRHA and 60wt.% water gave lowest compressive strength reading. Lowering the water content in the cement mixture has significantly improved the geopolymer cement compressive strength. This is shown by batch A2, using 50wt.% MIRHA and 10wt.% water has compressive strength up to 64% higher than batch A1 using 60wt.% of water. Accordingly, batch A3 has compressive strength increment up to 71%. The average density of samples recorded by on the 14th days were 1176 kg/m^3, 1508 kg/m^3 and 1652 kg/m^3 for batch A1, A2 and A3. As a binder, the density are relevant since it is lower than the typical density for mortar and concrete (refer Chapter 2 for density values). Similarly, the higher the density of binder, the higher the compressive strength observed.

Table 8: Compressive Strength Result for Batch A1

	Compressive strength, MPa **Batch A1** **(50FA:50MIRHA, w/b: 0.4, 60% water)**		
	3 days	7 days	14 days
Sample 1	0.91	1.37	1.69
Sample 2	0.98	1.40	1.46
Sample 3	0.95	1.38	1.71
Average	**0.95**	**1.39**	**1.62**

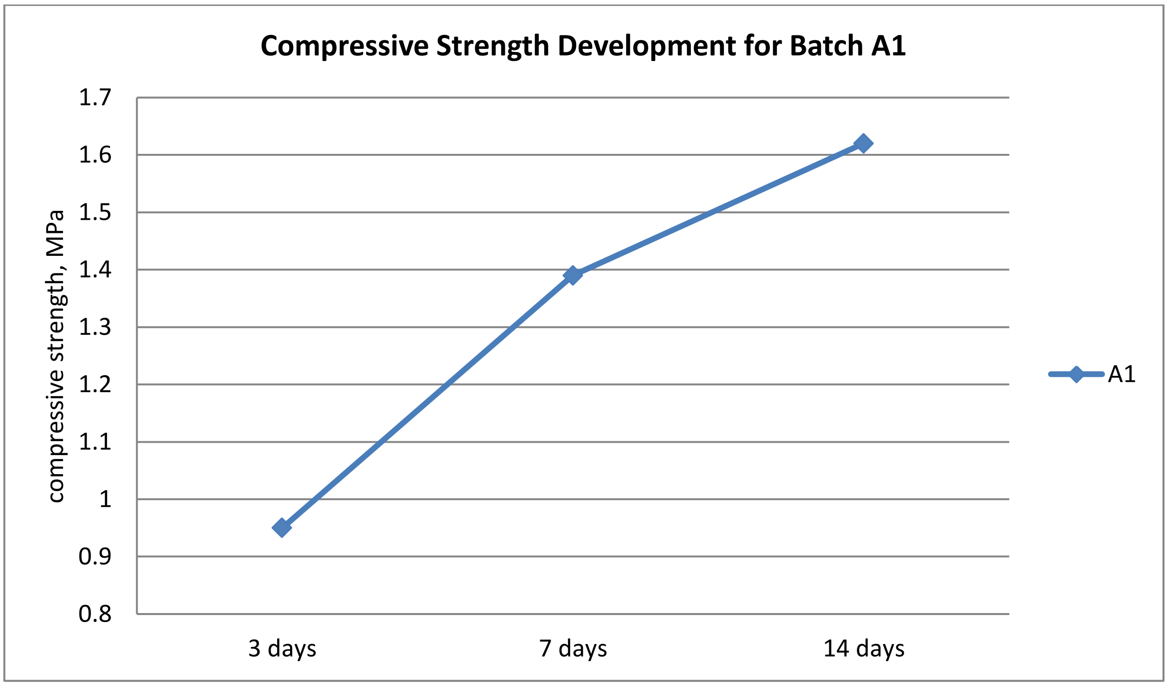

Figure 11: Compressive Strength Development for Batch A1

Table 9: Compressive Strength Result for Batch A2

	Compressive strength, MPa **Batch A2** **(50FA:50MIRHA, w/b: 0.95, 10% water)**		
	3 days	7 days	14 days
Sample 1	2.58	3.76	4.31
Sample 2	2.75	3.80	3.98
Sample 3	2.68	3.57	3.61
Average	**2.67**	**3.71**	**3.97**

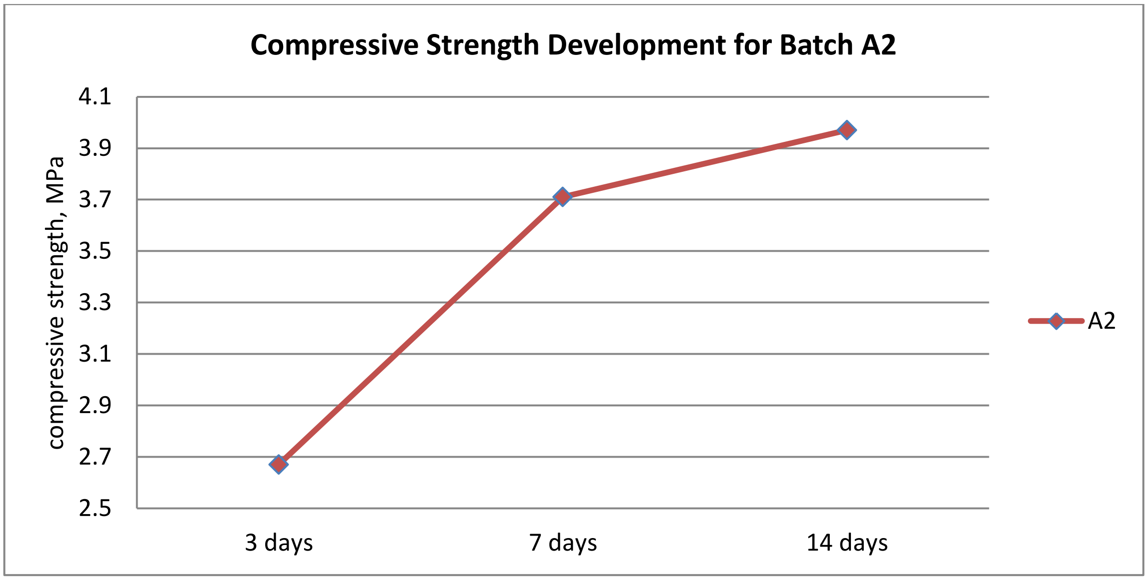

Figure 12: Compressive Strength Development for Batch A2

Table 10: Compressive Strength Result for Batch A3

	Compressive strength, MPa **Batch A3** **(70FA:30MIRHA, w/b: 0.95, 10% water)**		
	3 days	7 days	14 days
Sample 1	3.06	3.25	4.97
Sample 2	2.67	4.40	4.54
Sample 3	4.07	4.05	5.09
Average	**3.26**	**3.90**	**4.87**

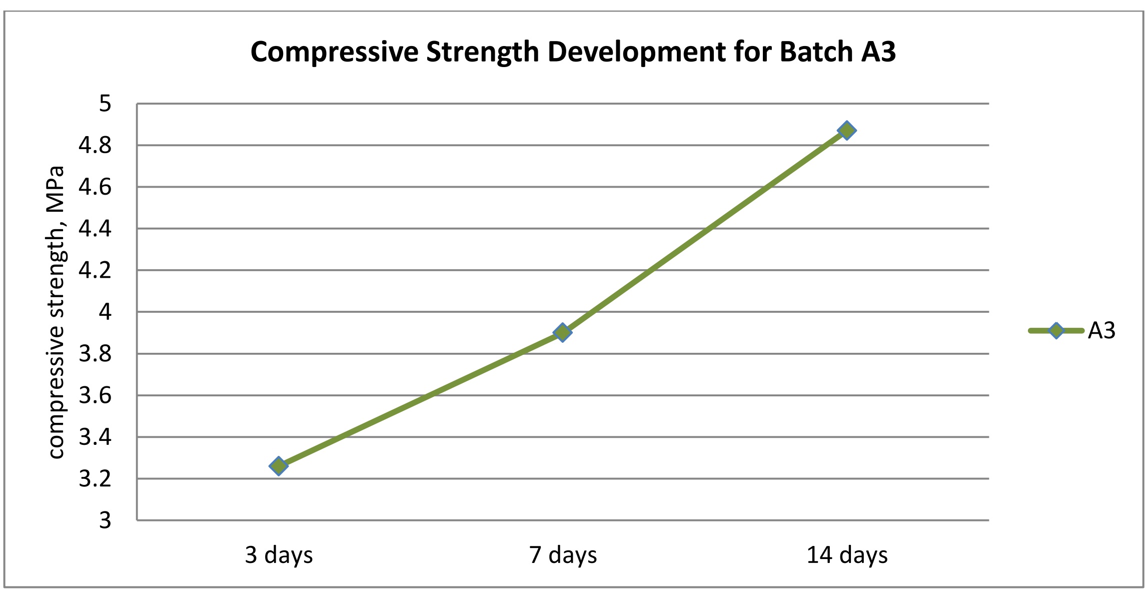

Figure 13: Compressive Strength Development for Batch A3

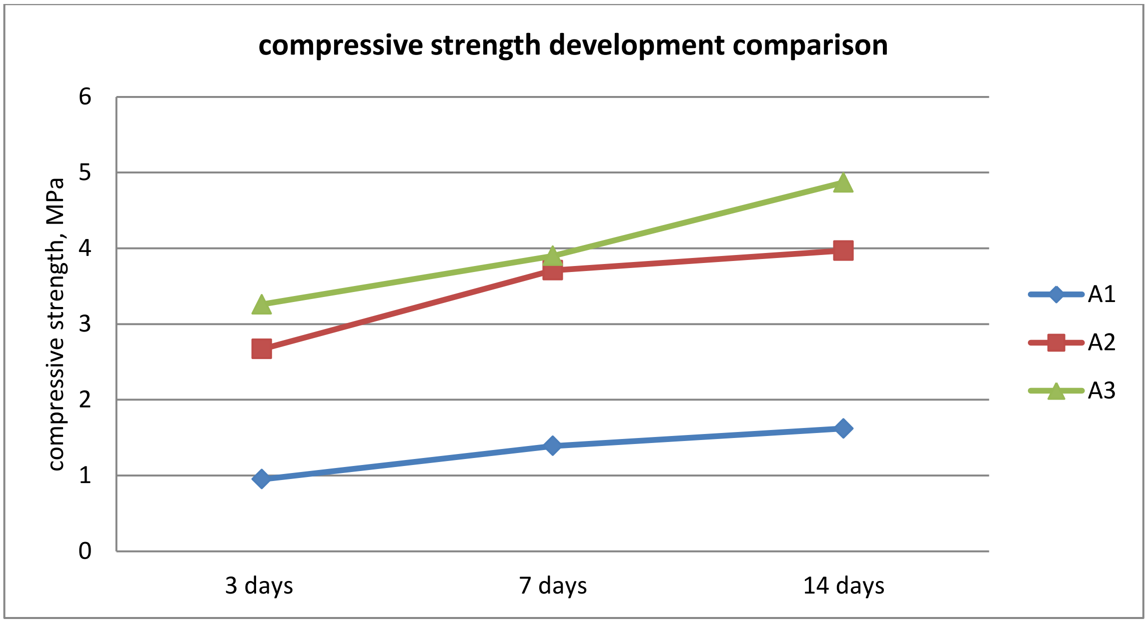

Figure 14: Compressive Strength Development Comparison

Figure 15 shows the influence of water inclusion to the compressive strength. Both batches A1 and A2 are using 50% FA and 50% MIRHA. Batch A1 with 60% water content appear to have lower strength compared to batch A2 with 10% water inclusion. The increase in strength is up to 1.5 times when the water was reduced to 10%. In contrast, the increase in w/b ratio leads to increasing compressive strength.

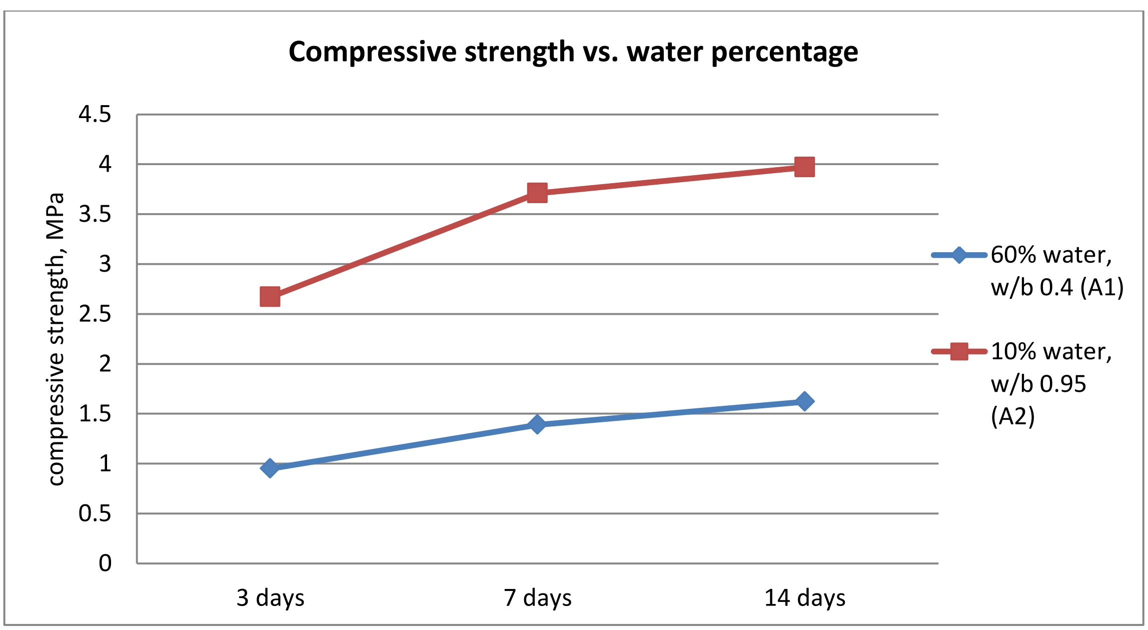

Figure 15: Compressive Strength vs. Percentage of Water

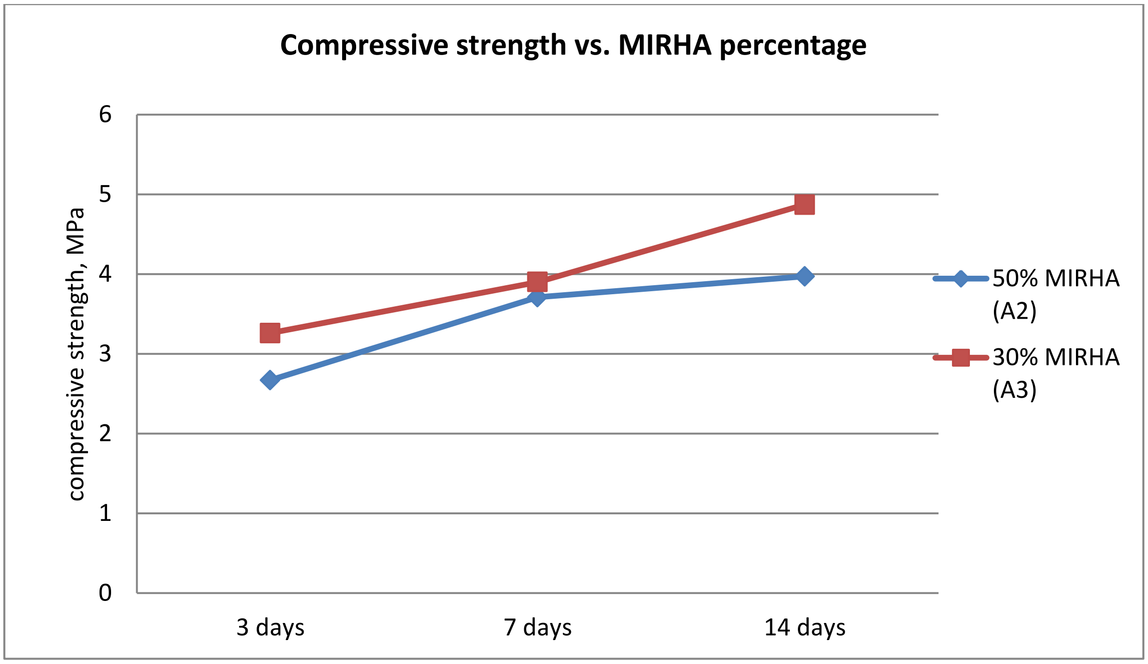

Figure 16: Compressive Strength vs. Percentage of MIRHA

From **Figure 16**, it can be observed that MIRHA ratio does affect the compressive strength. Lowering the MIRHA content would result in increasing cement strength.

4.2 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR analysis works by identifying the functional group of materials. In this analysis, raw FA and MIRHA were examined. **Figure 17** shows the IR bonds of the Fly Ash. The IR spectrum shows main absorption bands at 609.73, 712.49, 875.83, 1443.77, 1798.69, 2513.25, and 3440.84 cm^{-1}. **Table 11** summarizes the functional groups based from IR bonds obtained for both FA and MIRHA.

In addition, **Figure 18** shows the FTIR result for class F Fly Ash (Al Bakri *et al*., 2012). Its respective fuctional groups are Alkenes at 1428 cm^{-1} and Alcohols at 1004 cm^{-1}. Comparing these two results of FA clarify the large different between the chemical constituents of FA used in this experiment with the typical Class F FA. Despite that, the presence of aromatic group in the FA for this study explain the reasons for increasing compressive strength results when the compostion of FA increased. Aromatic compound contained conjugated double bonds allowing added stability for the material.

Table 11: Characteristic of IR band for Fly Ash and MIRHA

Functional Group	Fly Ash (cm^{-1})	MIRHA (cm^{-1})
Alcohol	3440.84	1090.99
Carboxylic Acids	2513.25 1798.69	-
Alkanes	1434.77	2922.61
Aromatics	875.83	-
Alkyl Halides	712.49	-
Alkynes	609.73	621.67
Alkenes	-	1631.45
Amides	-	3453.08

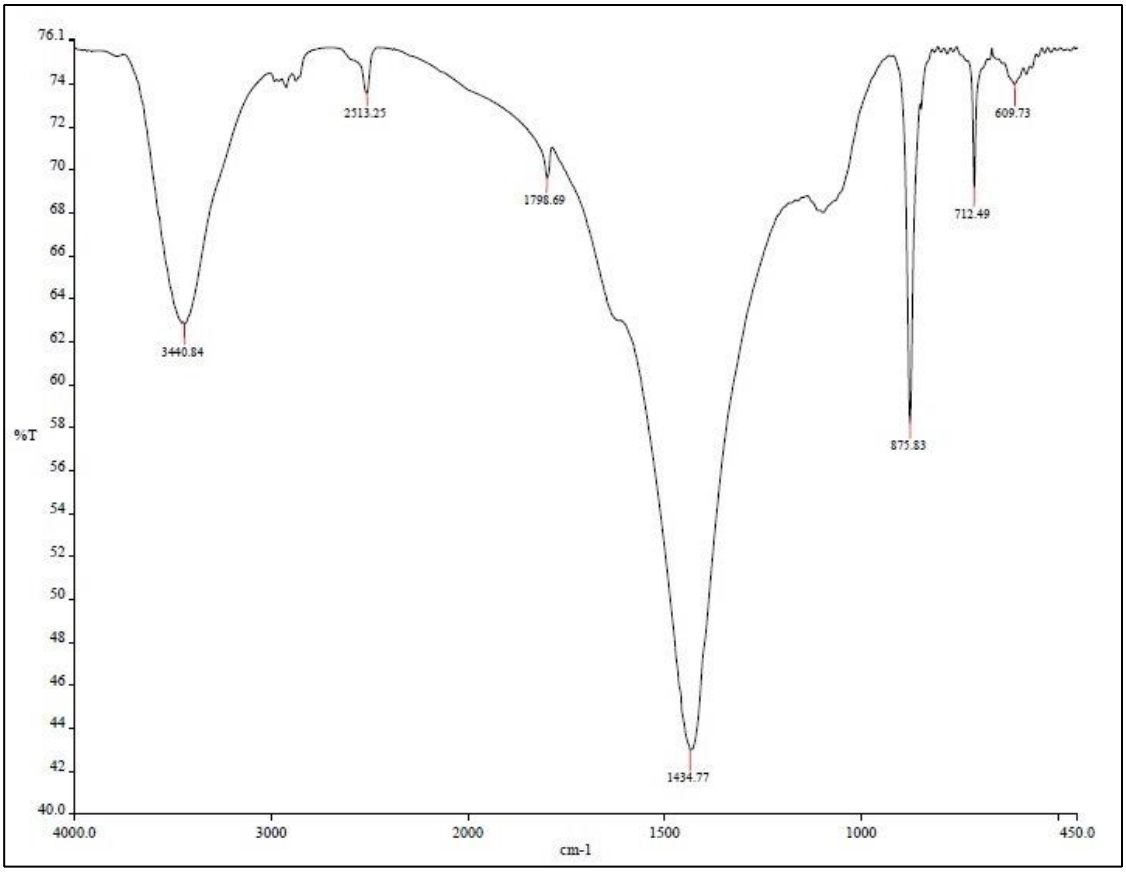

Figure 17: FTIR Analysis of Fly Ash

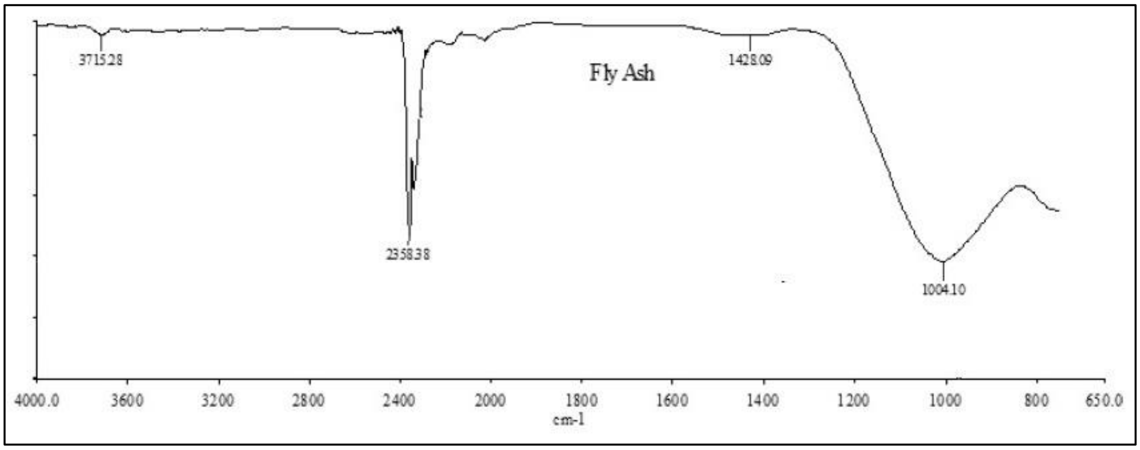

Figure 18: FTIR Analysis of Class F Fly Ash

Adapted from Al Bakri et al. (2012)

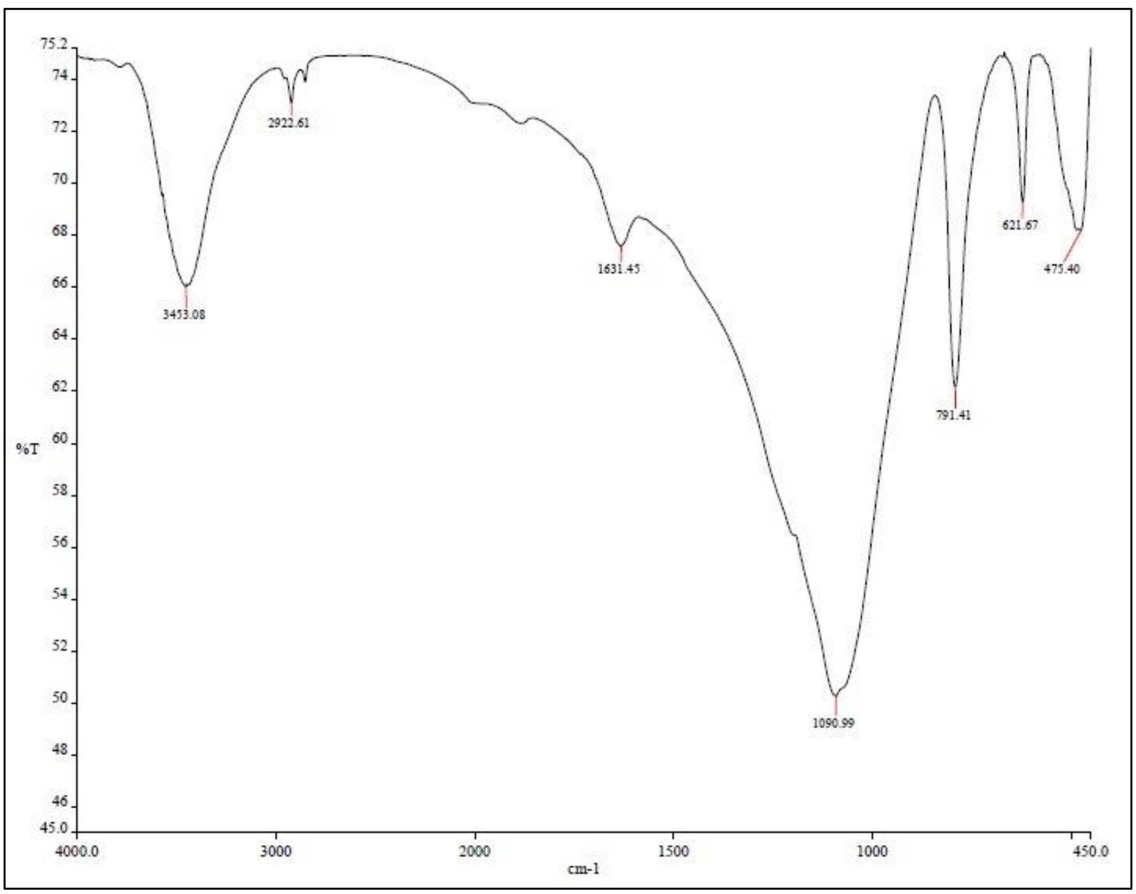

Figure 19: FTIR Analysis of MIRHA

On the other hand, **Figure 19** shows the IR bonds of MIRHA. It has main absorption at 475.40, 621.67, 791.41, 1090.99, 1631.45, 2922.61, and 3453.08 cm^{-1}. MIRHA samples were taken from the same sources as Kusbiantoro *et al*. (2012) hence similar functional group would be expected.

4.3 Discussion

Many researchers claimed that curing temperature and time influenced the development of geopolymers compressive strength. Their relationship is described with the theory that increased in curing time and temperature will result in increased of compressive strength with regard to certain optimum point. This is believed due to unfinished development of the specimens' compressive strength. As expected, increasing curing time of the cement will result in increased compressive strength of the cement (Ali Nazari, 2011) as in **Figure 11-14**. The strength development was higher during 3 to 7 days. After that, gentler slope will be observed. This indicated that the cements are nearer to the optimum curing period. However, the study was not able to identify the optimum curing time for each sample due to limited time. There are possibilities that the compressive strength will continue to increase for a long time. Nonetheless, it failed to prove that geopolymer cement is having added advantage of quick compressive strength build up.

Additionally, it is shown that the composition of MIRHA, percentage of water and, w/b ratio in the composition of the materials gave significant effect on the geopolymer strength development. The inclusion of MIRHA is limited up to certain stage only. Any further increase in MIRHA content will yield low strength cement. It appears that MIRHA particles possess slightly different silicate structure, hence when SiO_2/Al_2O_3 ratio is altered to a higher ratio; the species of silicate that has large cyclic structure hinders the polycondensation process (Swaddle, 2001). It is well understood that in polycondensation, the monomeric $Si(OH)_4$ and larger linear silicate anions only react with uncomplexed tetrahedral aluminate $AL(OH)_4^-$. The presence of large silicate cyclic structure inhibits the kinetics and reduces the production rate of geopolymer gel (Kusbiantoro *et al*., 2012). Study done by Ali Nazari *et al*. (2011) included up to only 40% MIRHA into the concrete mixture composition. Referring to **Figure 20**, the result shows that 40% MIRHA has the lowest compressive strength result compared to 30% and 20% MIRHA inclusion.

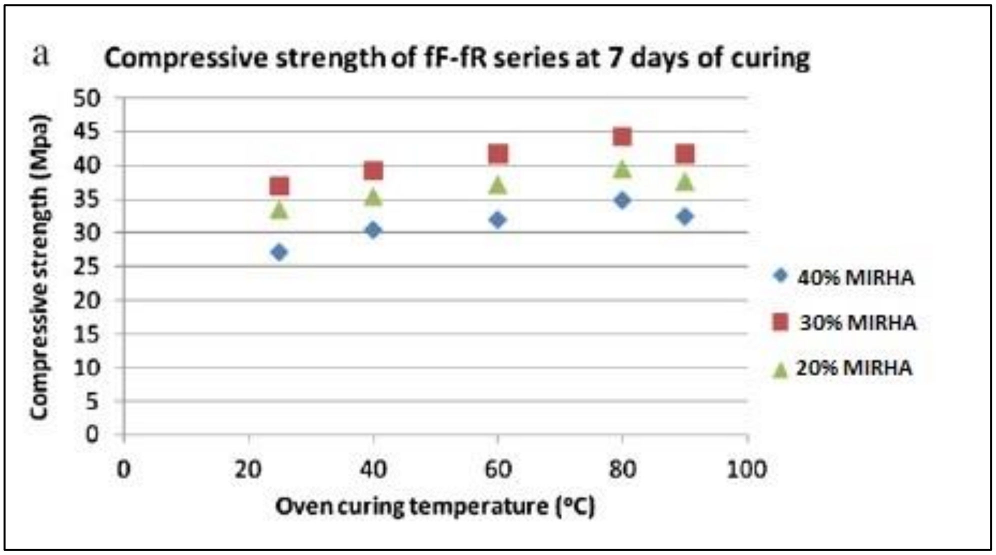

Figure 20: Compressive Strength of Fly Ash-MIRHA Based Concrete

Adapted from Ali Nazari et al. (2012)

Besides that, excessive addition of water also leads to lower compressive strength. The situation can be observed from **Figure 15**. This situation occurs because excessive water inclusion into the samples especially for ambient curing was also hindering the polycondensation process of the cement (Toreanu, 1991) thus increasing the porosity (Duzson *et al*., 2006) and pore sizes (Abalaka & Okoli, 2013). Although water is a necessity to provide workability to cement, unnecessary amount can cause reverse effect to the compressive strength of the cement. Unfortunately, the absorption characteristic of MIRHA induces more solution to be added on the mixture either through the addition of water or alkaline solution. At first, the composition was based on the optimum Fly Ash-MIRHA-based concrete proportion obtained by Kusbiantoro *et al*. (2012). Optimum w/b ratio of 0.4 and 10wt.% of water serves as basis for batch A1. However, 10wt.% water consumption was not able to dissove the mixture. The addition of fine and coarse aggregates into concrete cause the surface area to be lower than cement binder (where no fine or coarse aggregates are present). Therefore, less amount of solution would be needed to bind the concrete materials compared to cement. Alternatively, the demand for water can be compensated by using superplasticizers (Bui et al., 2004).

To proceed with the experiment, the percentage of water was raised to 60wt.% to increase the workability of the cement (batch A1). Batch A2 was vice versa. It increase the amount of alkaline solution by fixing the water content of 10wt.%. It is proved that the inclusion of high water content resulted a low strength cement (Toreanu, 1991). This is also supported by the compressive strength result for batch A1 to batch A2 in **Figure 15**. Hence, the next batch A3 was prepared by only manipulating alkaline solution ratio (w/b ratio) and fixing the water content up to only 10wt.% where improvement on compressive strength were seen.

The maximum compressive strength obtained from this study is 5MPa which is way lower than the expected result would be (approximately 20MPa for the first 24 hours as in **Table 2**). Among the possible causes is low quality Fly Ash. This is supported by the FTIR analysis where there are large different between the chemical behavior of FA used in this study and normal class F FA. It must be noted that different samples of Fly Ash may give different reactivity due to their varying chemical compositions. The FA obtained might be generated from younger coal where it can hardly perform without any addition of additives. Other than that, the burning temperature of MIRHA was believed to be at 600℃ (planned to be at 800℃) only. This can be identified by the darker colour of MIRHA produced. It indicates that lower Silicon Oxide (SiO_2) content thus lower performance. Research done by Kamal *et al*. (2008) shows that burning MIRHA at 800℃ produce concrete with higher compressive strength compared to 700℃ and 600℃ burning temperature. Nevertheless, it is not suggested ti burn rice husk above 800℃ longer than one hour to prevent sintering effect. In addition, the particle sizes also plays an important role. The study uses 300µm for both FA and MIRHA which considered larger when compared to others. The strength will increase as fineness of MIRHA increase (Bui *et al*., 2004).

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

There is no doubt that the use of MIRHA and Fly Ash reduce the environmental pollution that comes from the OPC-based cement industry. Instead of just industrial by-products, both of these materials are not only cost saving but also are becoming more popular as substitution in concrete cement. In all mixtures, batch A3 showed the highest compressive strength result by using only 30wt.% MIRHA, 10wt.% water and 0.95 w/b ratios. Although the expected result was not as desirable, but the knowledge gained through the study will absolutely helped in improving the understanding on MIRHA and Fly Ash' behavior as cement binder. From the study, it can be concluded that:

1. Increasing water content in cement mixture will result in lower compressive strength development although it helps to increase workability.
2. Addition of MIRHA in the mixture will help in improving compressive strength but up to only certain point.
3. Increasing FA content help to gain higher compressive strength.
4. Increasing w/b ratio does increase the compressive strength of the cement.
5. As curing time increases, the compressive strength is also increased.

The current knowledge shows that the influence of NaOH molarity, FA-MIRHA/alkaline activator ratio, source material composition, and curing temperature are essential for achieving the optimum strength of geopolymer. Further study on this topic should be done by:

1. Lowering the content of MIRHA or increasing the FA composition in the mixture.
2. Incineration of MIRHA should be done at 800℃ for optimum performance.
3. Class F Fly Ash would be a better substitution to be used instead of class C.

4. Identification of suitable FA chemical constituents should be done to ensure smooth study.
5. Extending the curing time would also a good option since there is still high compressive strength development observed during the 14^{th} days of curing.
6. Water ratio should be maintained at 10% for workability purpose.
7. Increase the fineness of both MIRHA and FA

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APPENDICES

Appendix 1: Compressive Strength Test Result for Each Sample

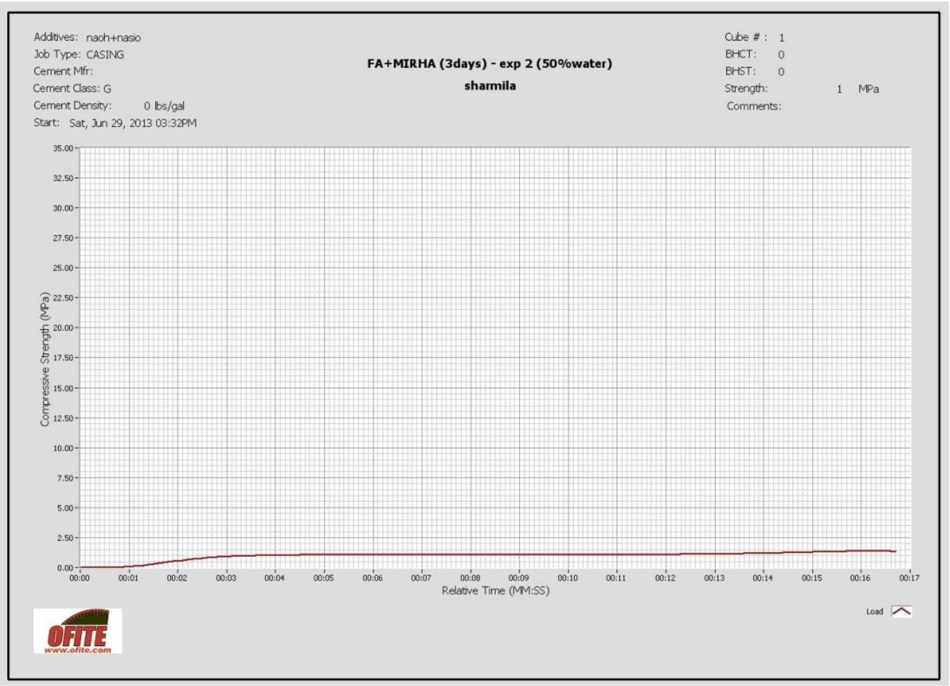

Figure 21: Compressive Strength Test Result for Batch A1, 3 days (sample 1)

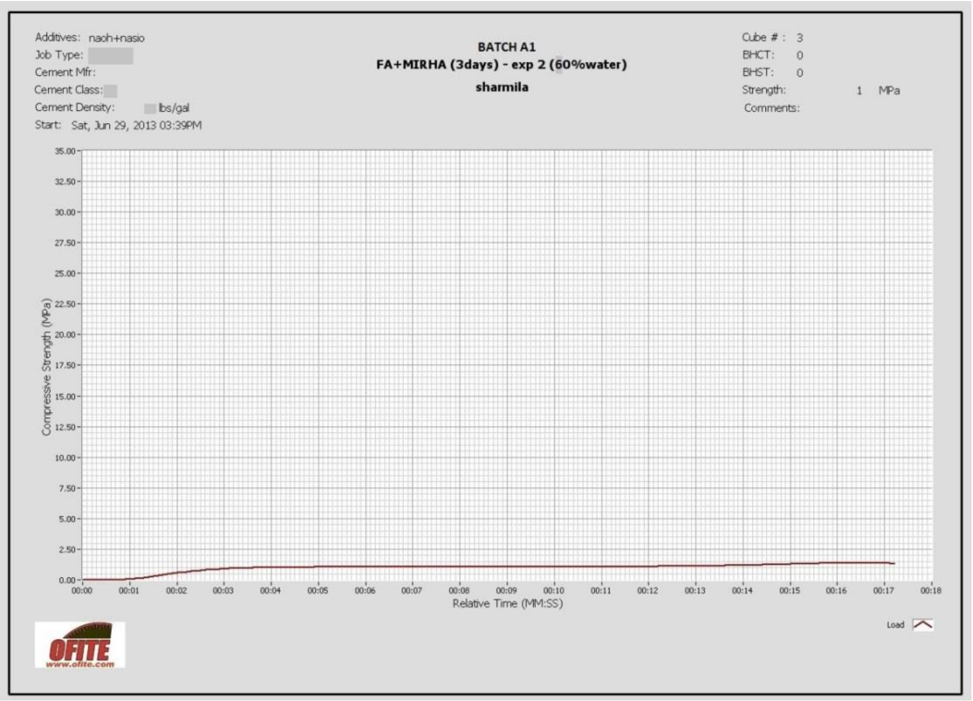

Figure 22: Compressive Strength Test Result for Batch A1, 3 days (sample 2)

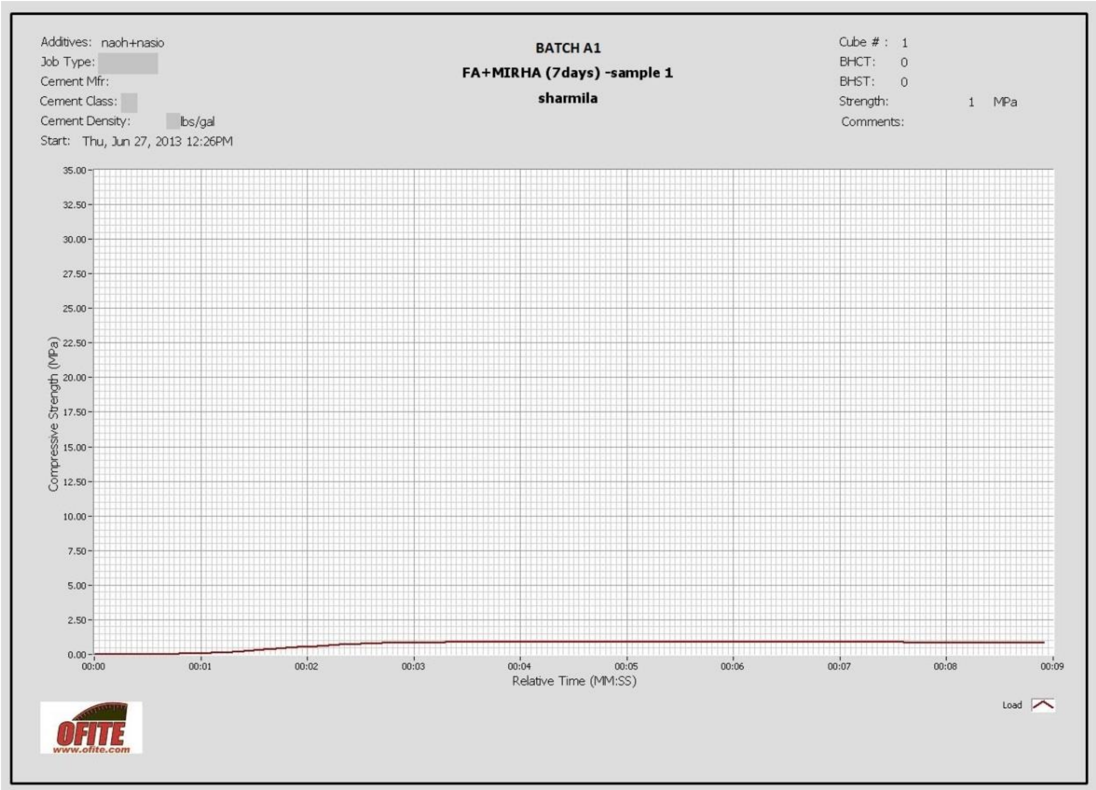

Figure 23: Compressive Strength Test Result for Batch A1, 7 days (sample 1)

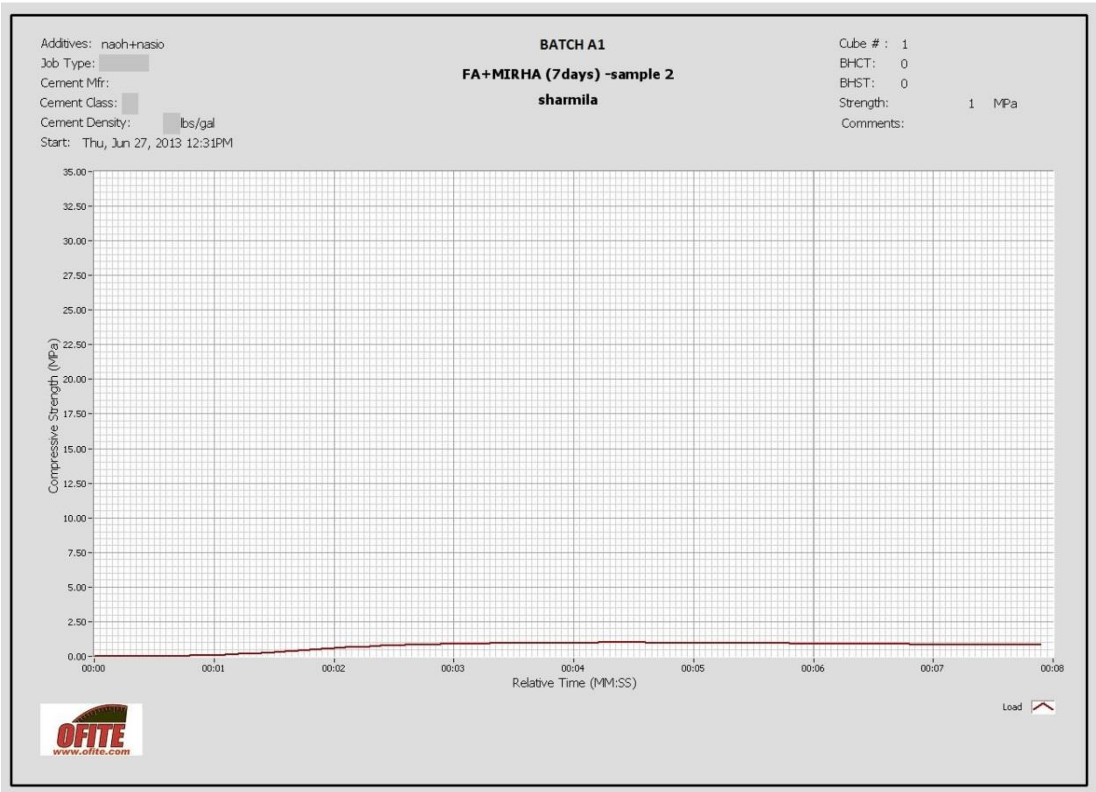

Figure 24: Compressive Strength Test Result for Batch A1, 7 days (sample 2)

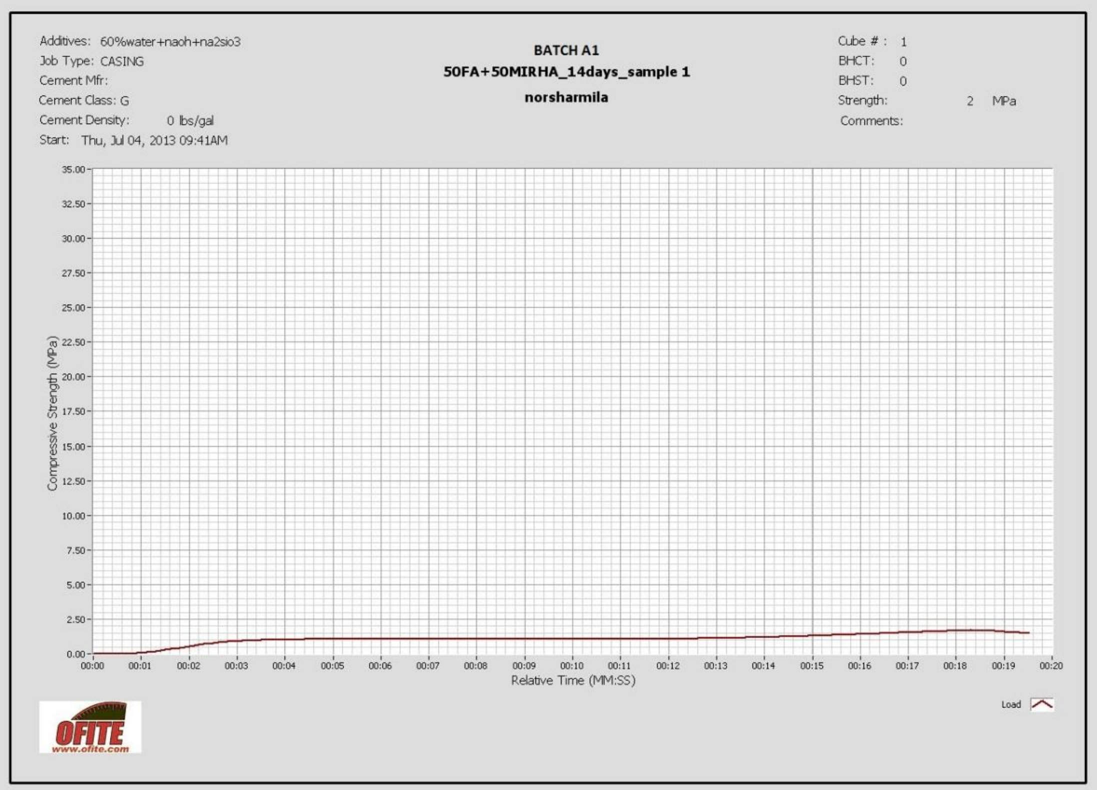

Figure 25: Compressive Strength Test Result for Batch A1, 14 days (sample 1)

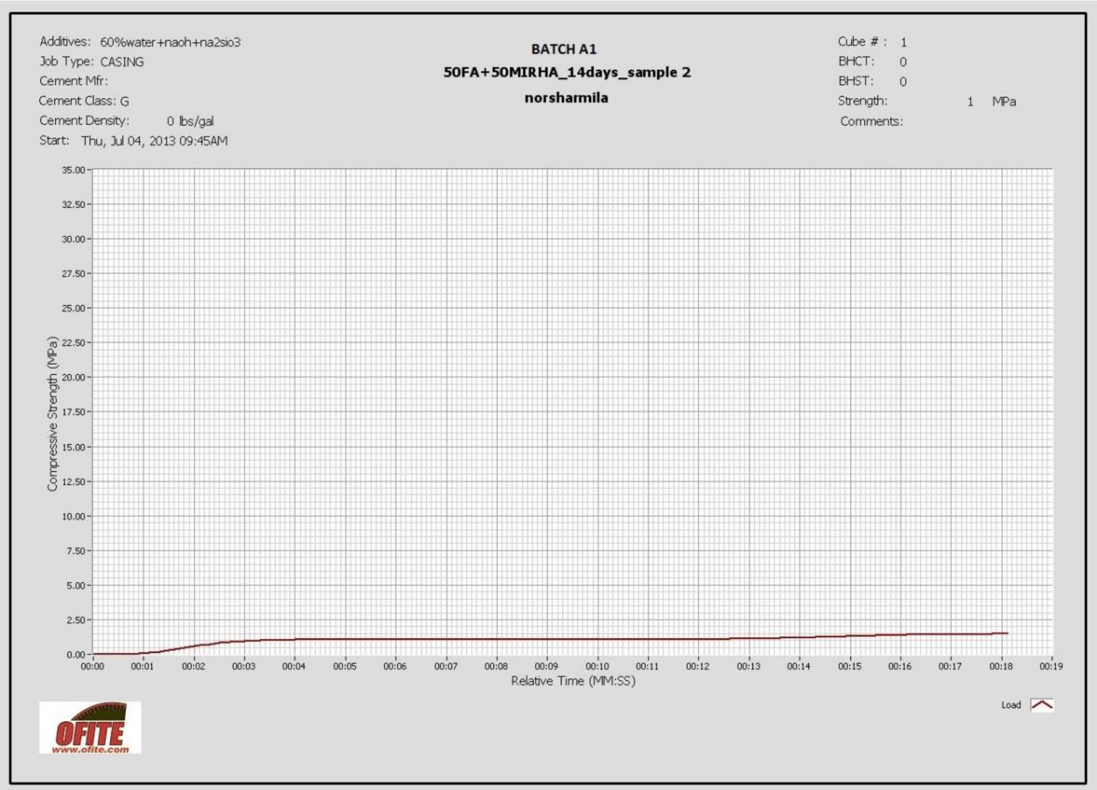

Figure 26: Compressive Strength Test Result for Batch A1, 14 days (sample 2)

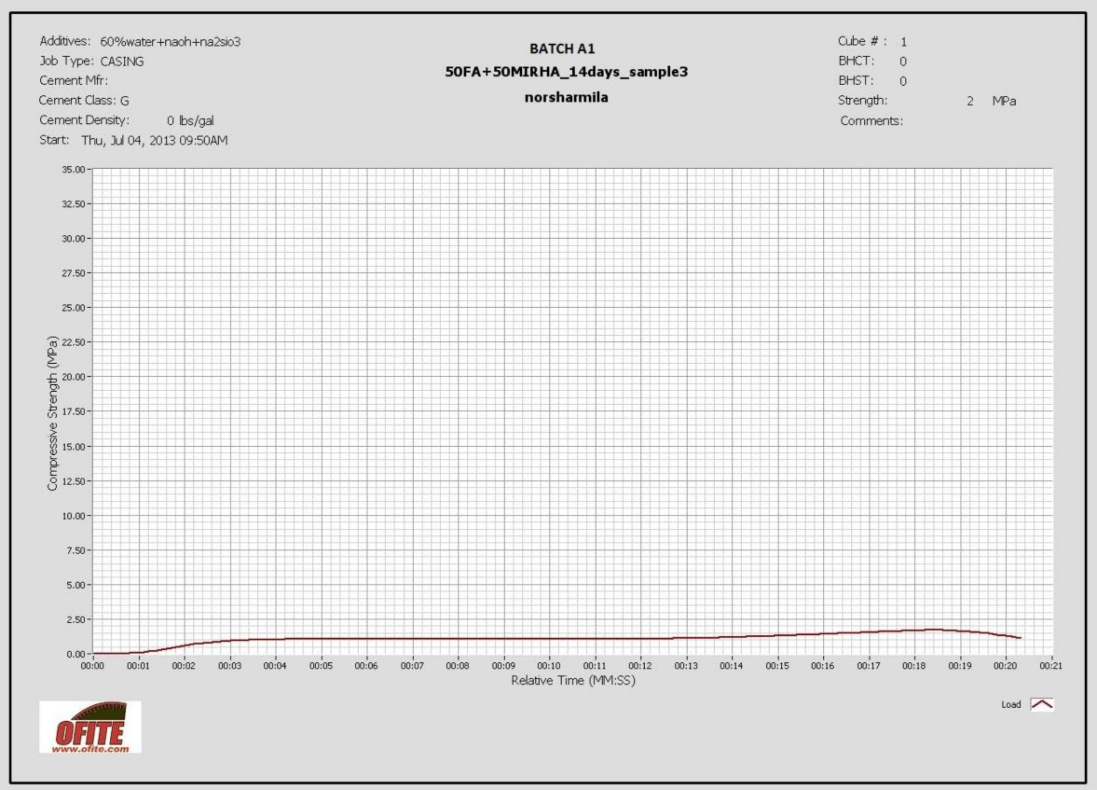

Figure 27: Compressive Strength Test Result for Batch A1, 14 days (sample 3)

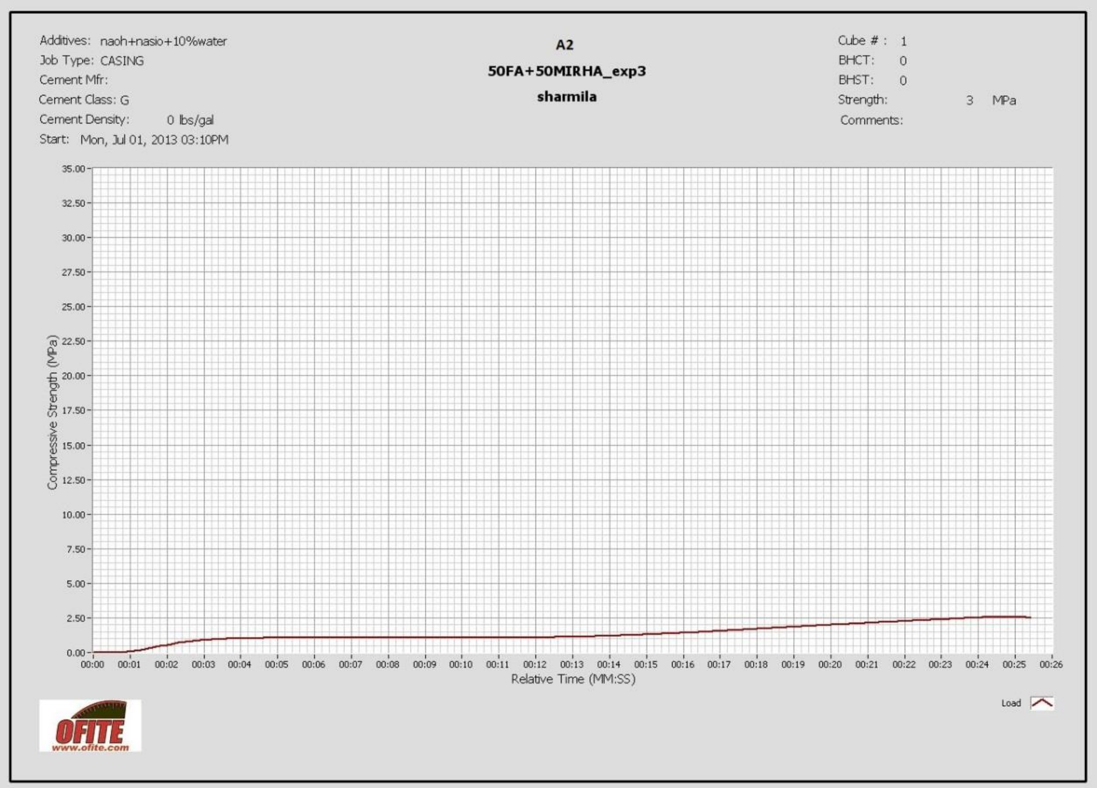

Figure 28: Compressive Strength Test Result for Batch A2, 3 days (sample 1)

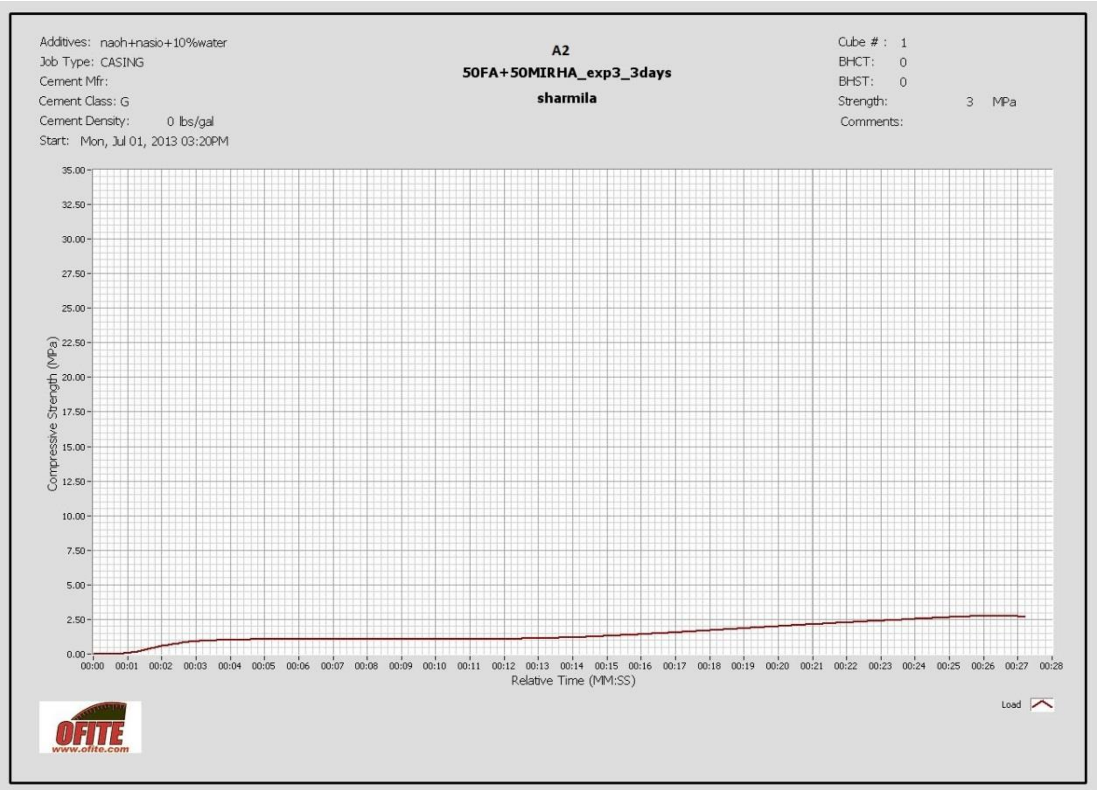

Figure 29: Compressive Strength Test Result for Batch A2, 3 days (sample 2)

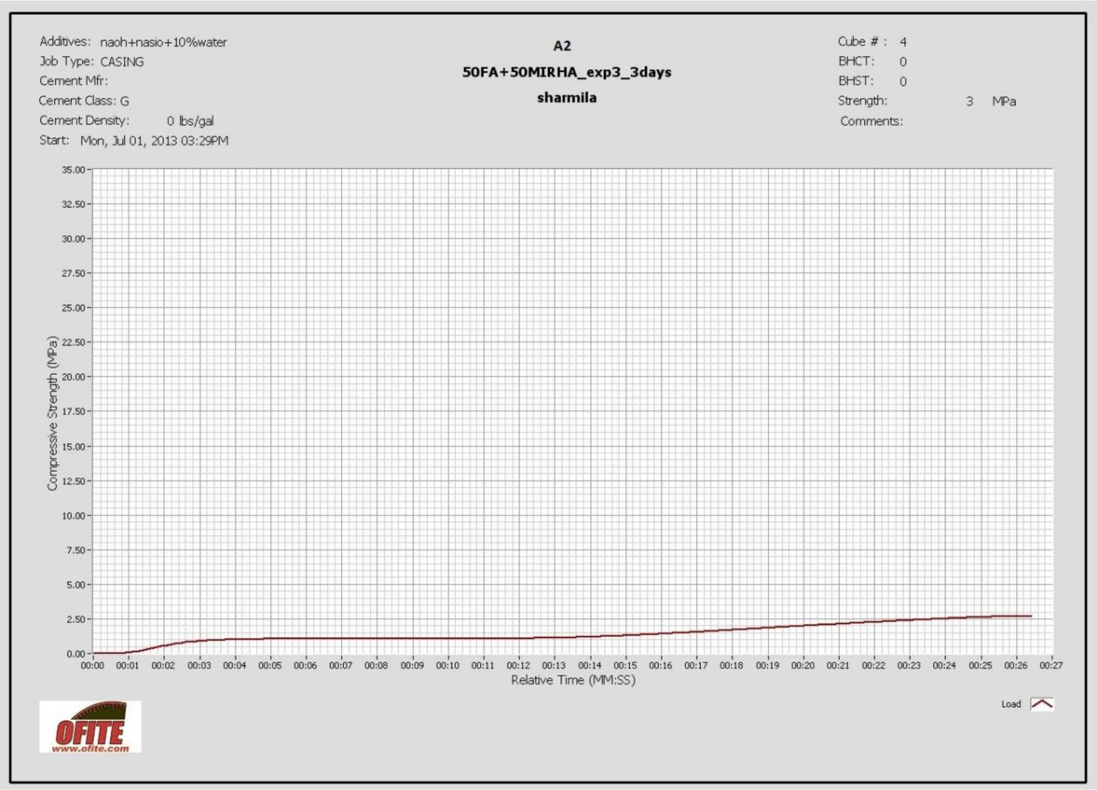

Figure 30: Compressive Strength Test Result for Batch A2, 3 days (sample 3)

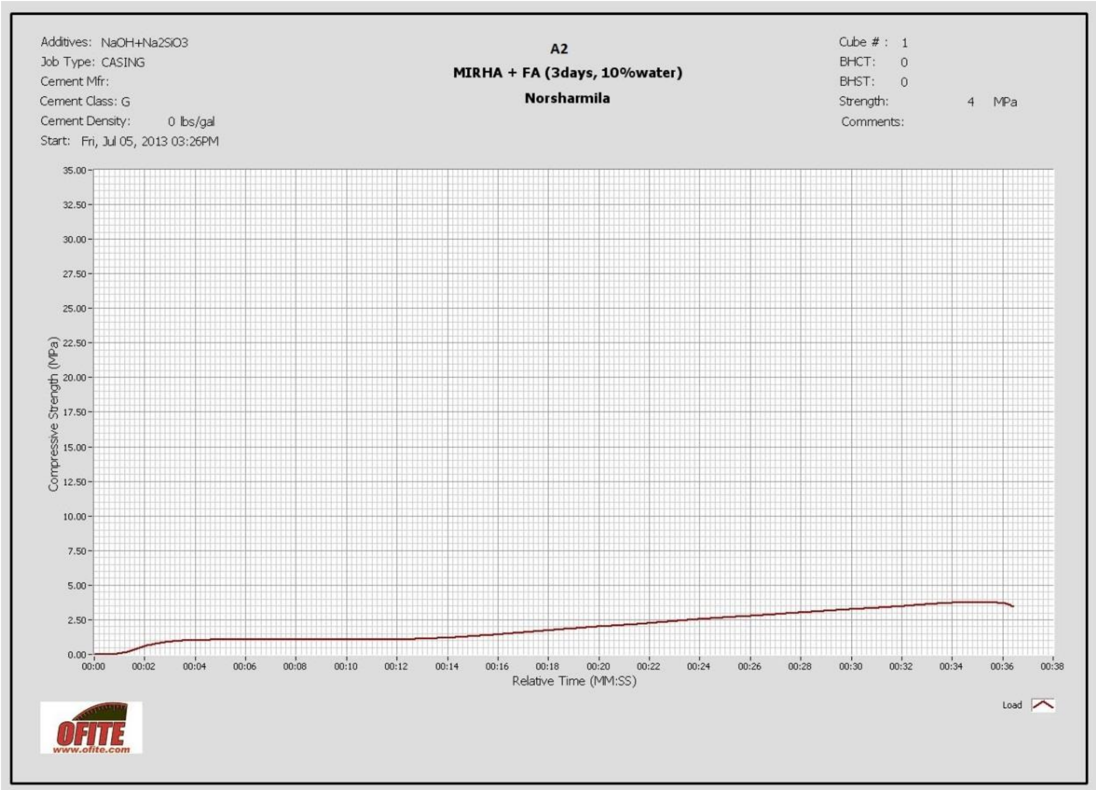

Figure 31: Compressive Strength Test Result for Batch A2, 7 days (sample 1)

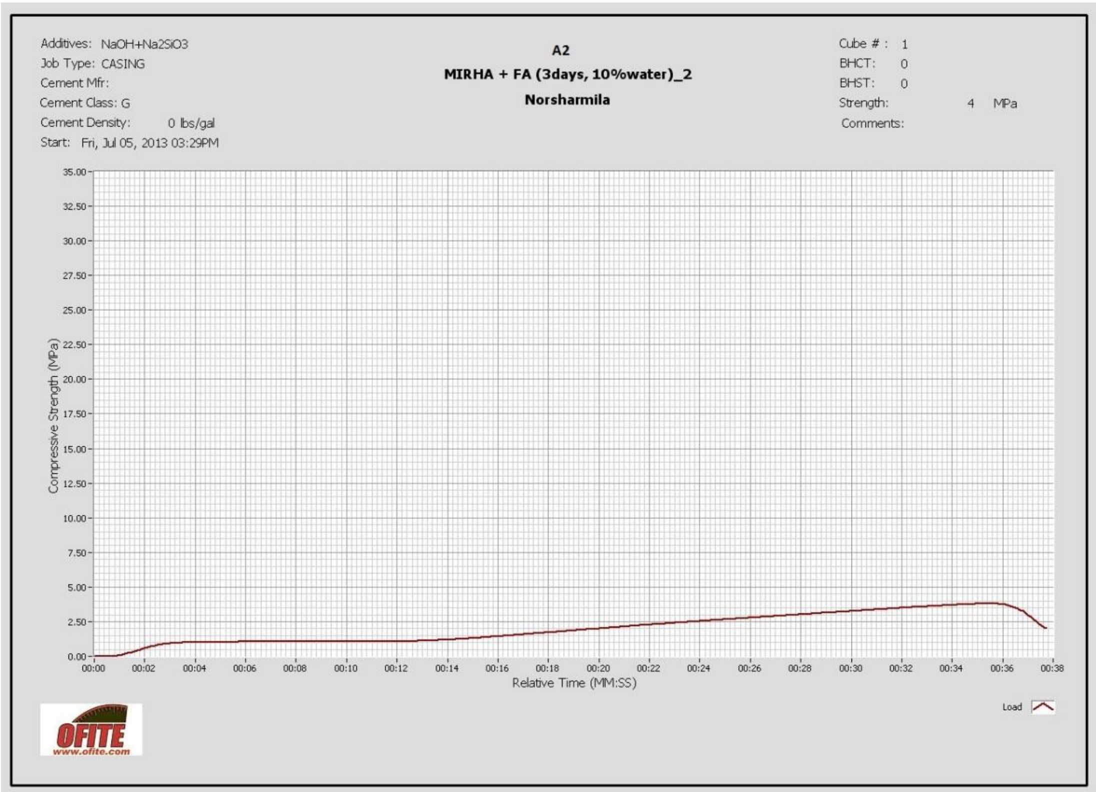

Figure 32: Compressive Strength Test Result for Batch A2, 7 days (sample 2)

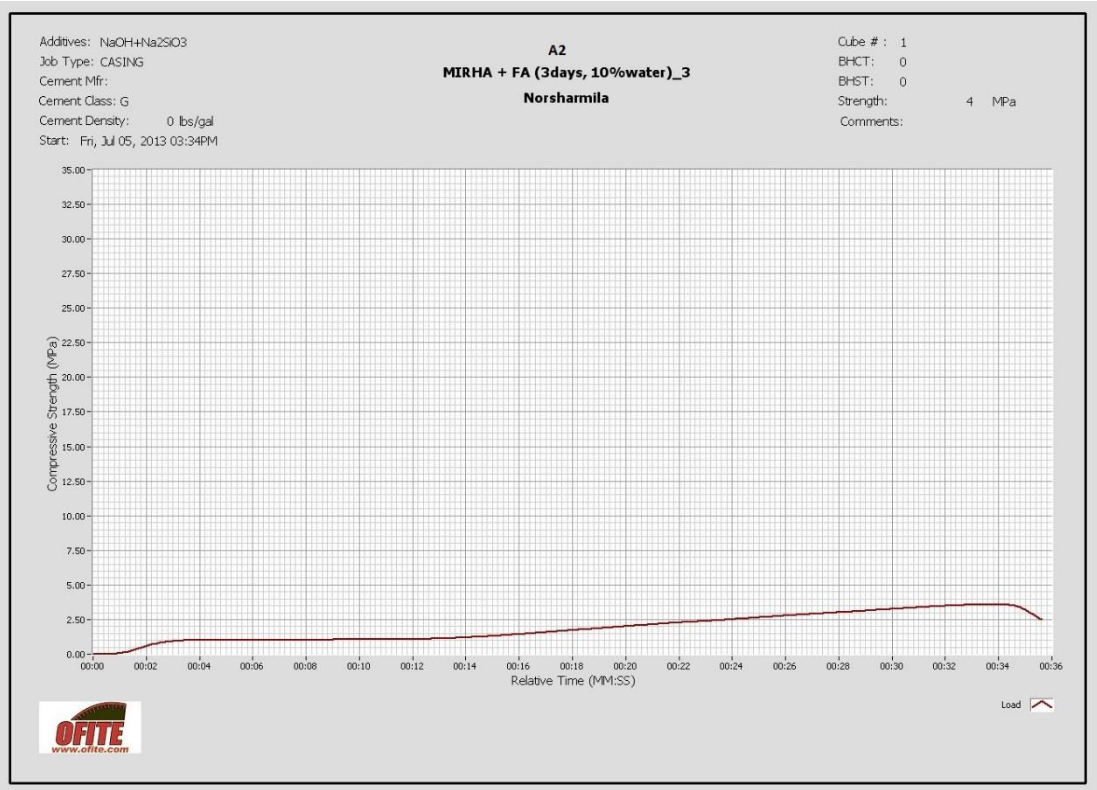

Figure 33: Compressive Strength Test Result for Batch A2, 7 days (sample 3)

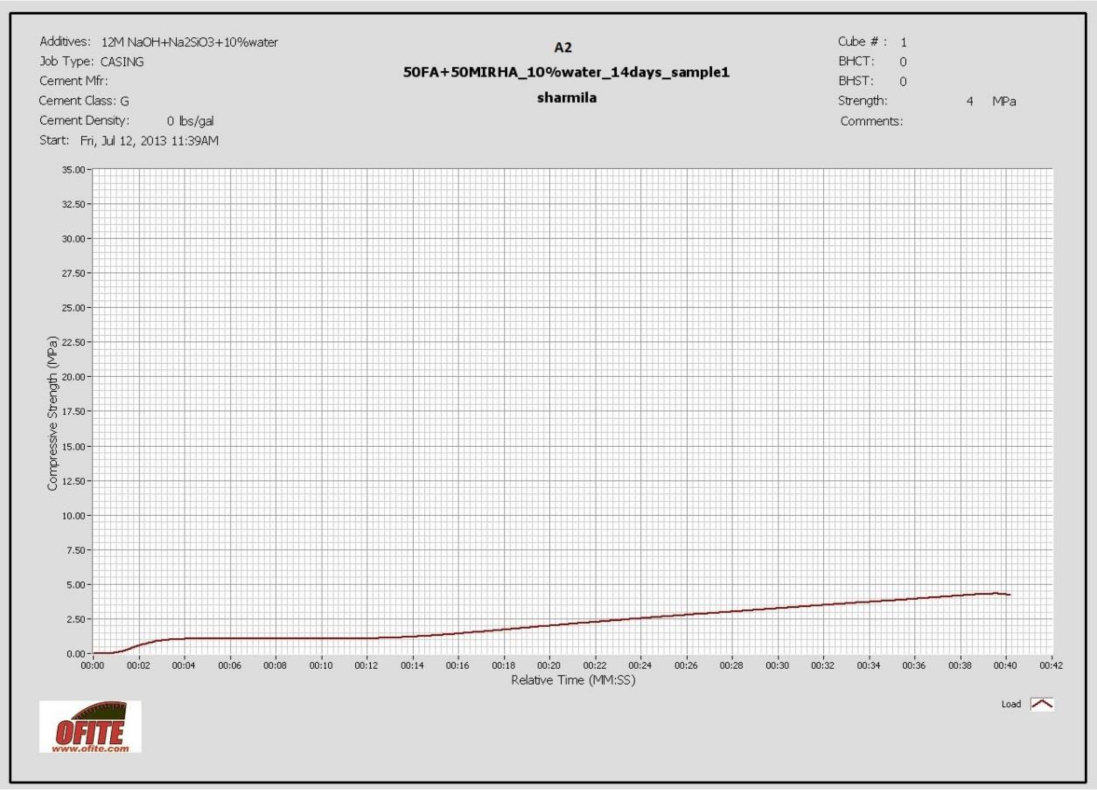

Figure 34: Compressive Strength Test Result for Batch A2, 14 days (sample 1)

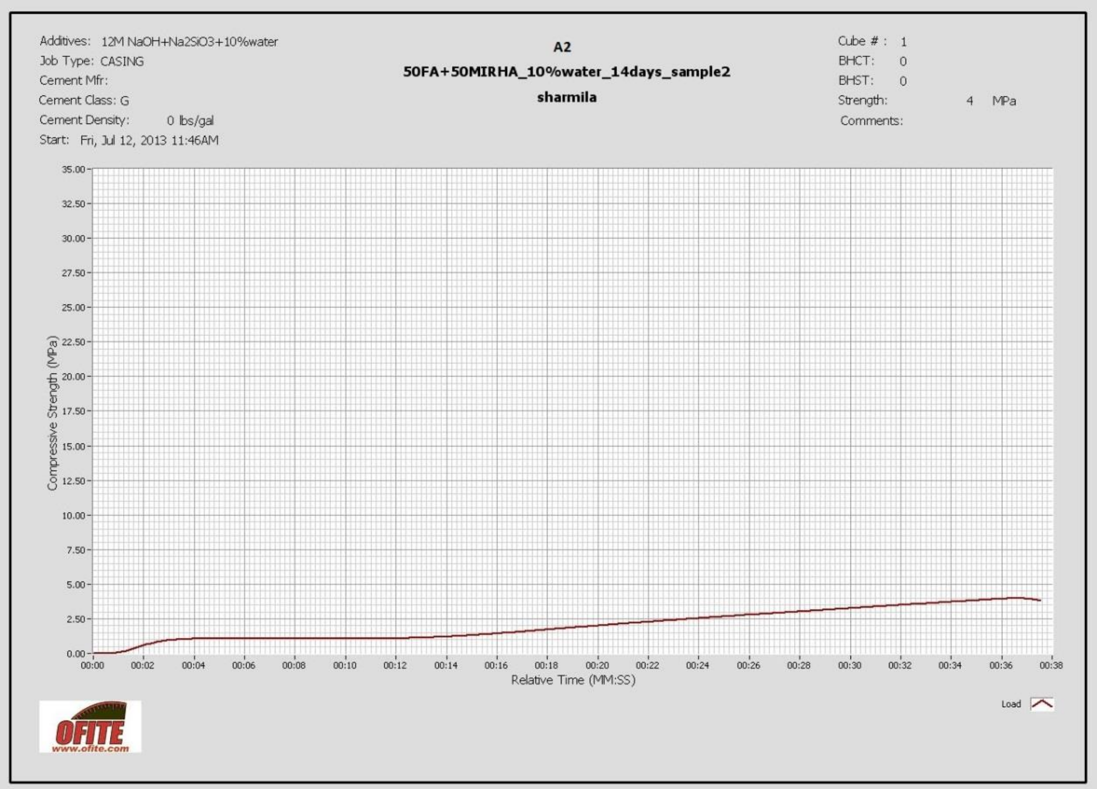

Figure 35: Compressive Strength Test Result for Batch A2, 14 days (sample 2)

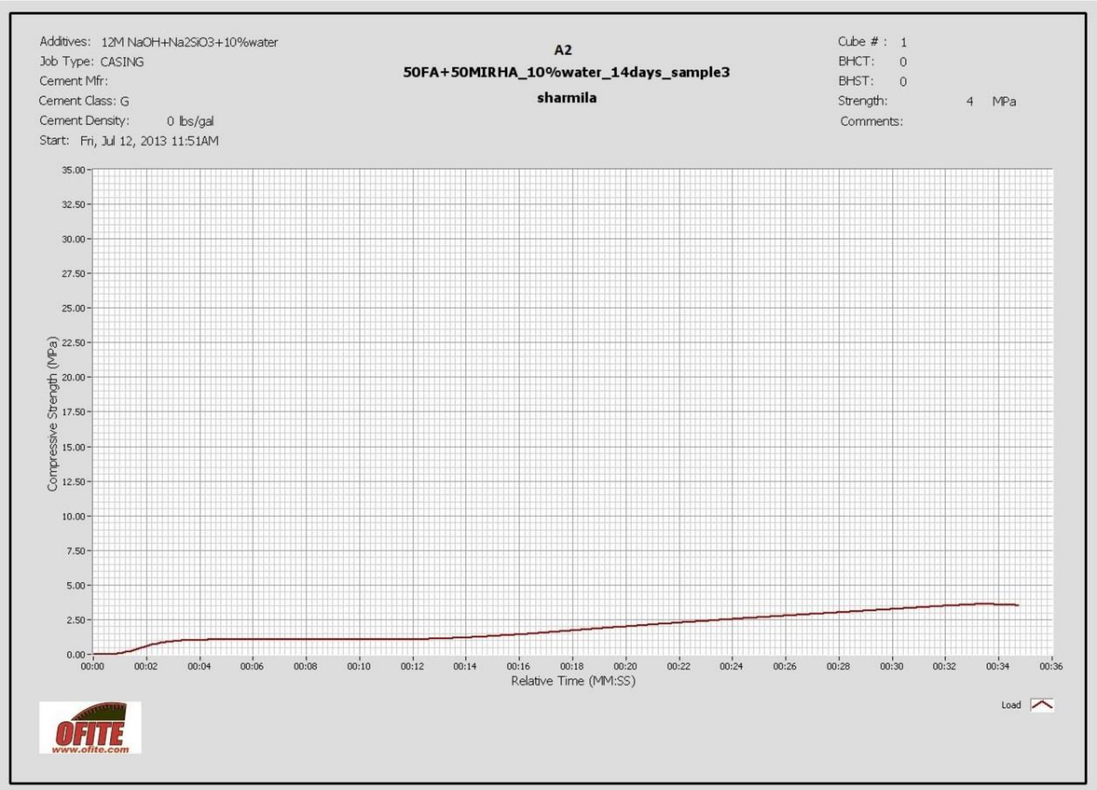

Figure 36: Compressive Strength Test Result for Batch A2, 14 days (sample 3)

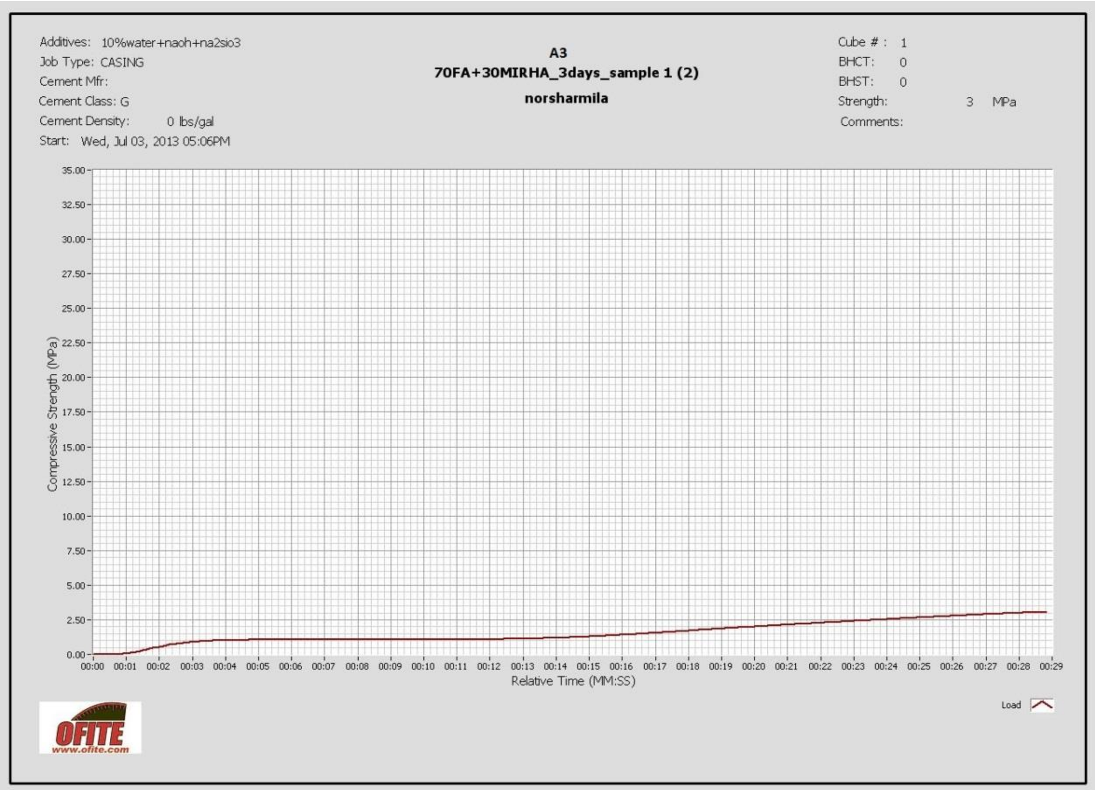

Figure 37: Compressive Strength Test Result for Batch A3, 3 days (sample 1)

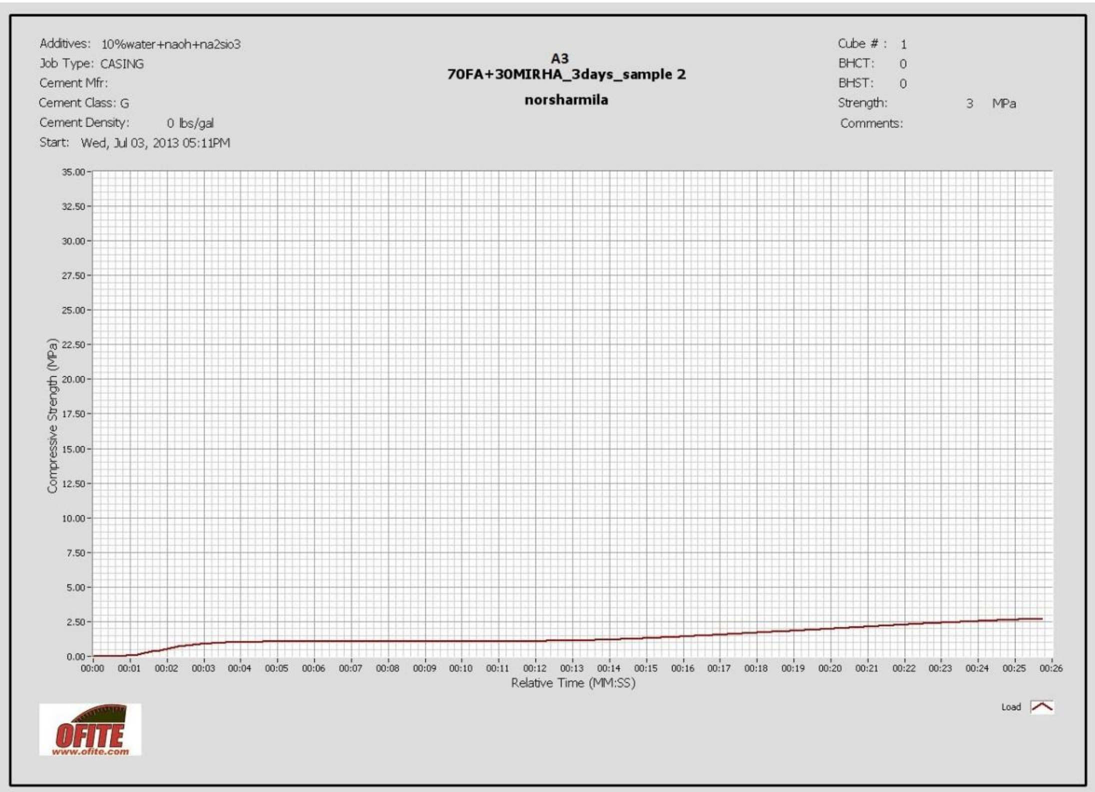

Figure 38: Compressive Strength Test Result for Batch A3, 3 days (sample 2)

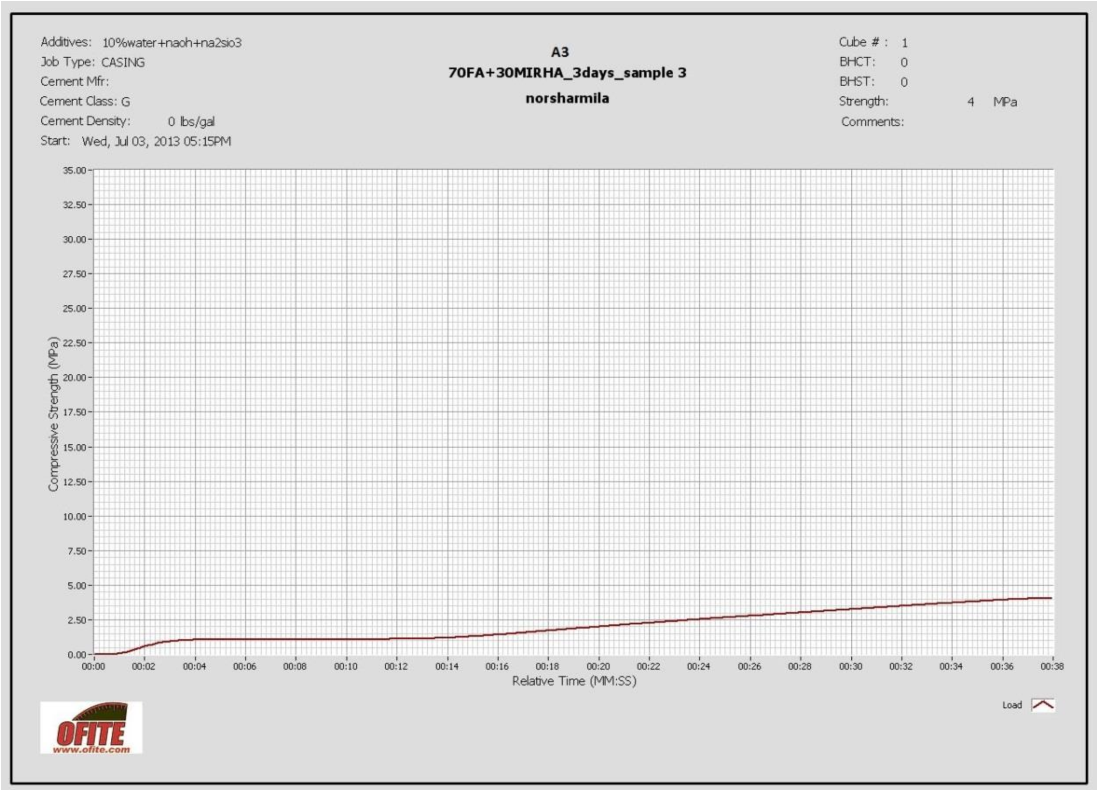

Figure 39: Compressive Strength Test Result for Batch A3, 3 days (sample 3)

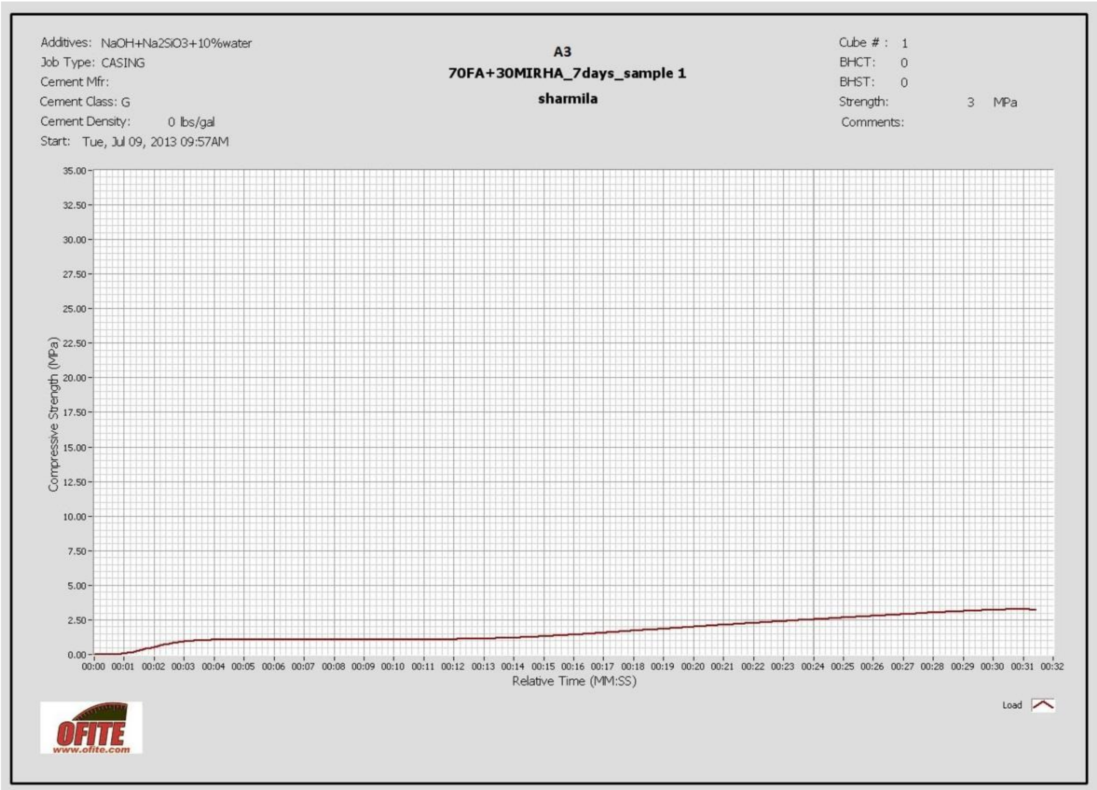

Figure 40: Compressive Strength Test Result for Batch A3, 7 days (sample 1)

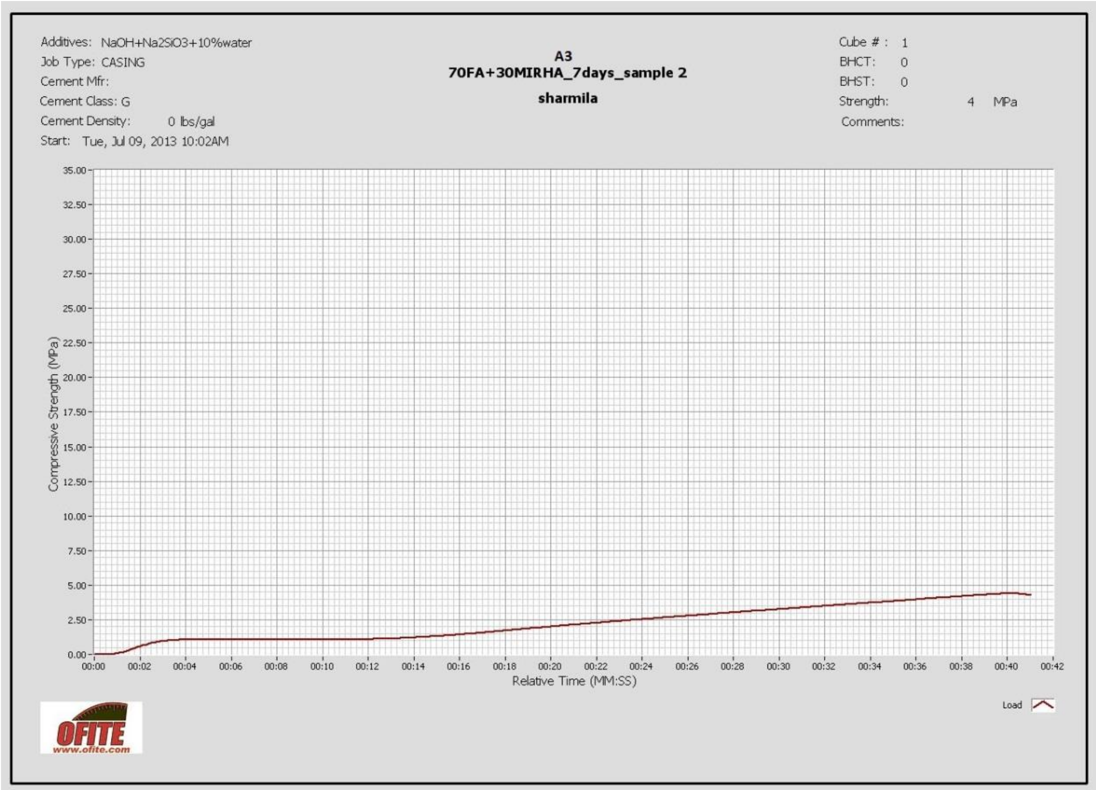

Figure 41: Compressive Strength Test Result for Batch A3, 7 days (sample 2)

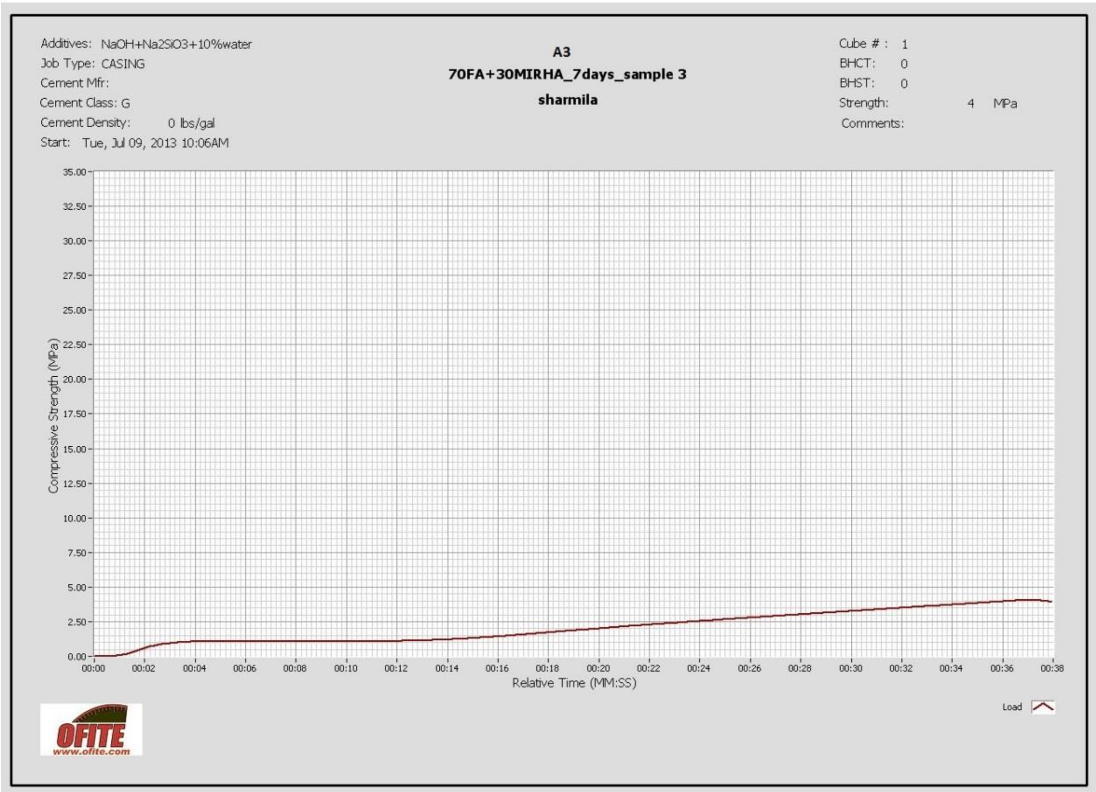

Figure 42: Compressive Strength Test Result for Batch A3, 7 days (sample 3)

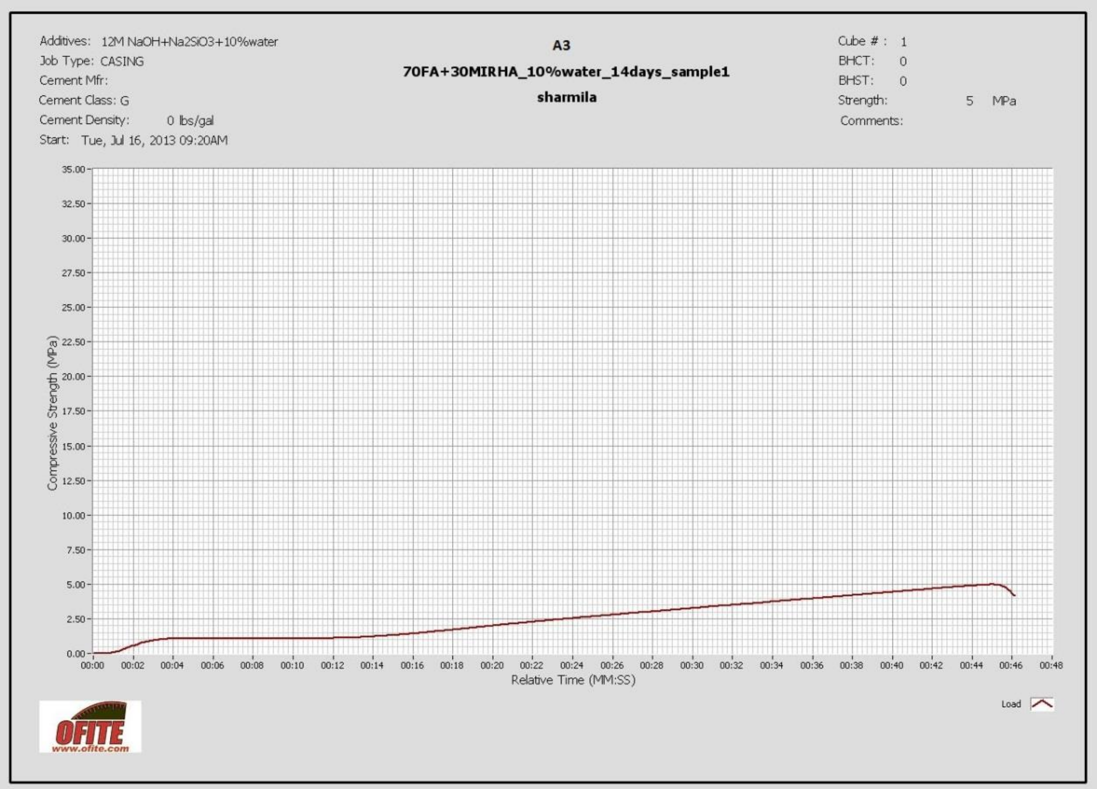

Figure 43: Compressive Strength Test Result for Batch A3, 14 days (sample 1)

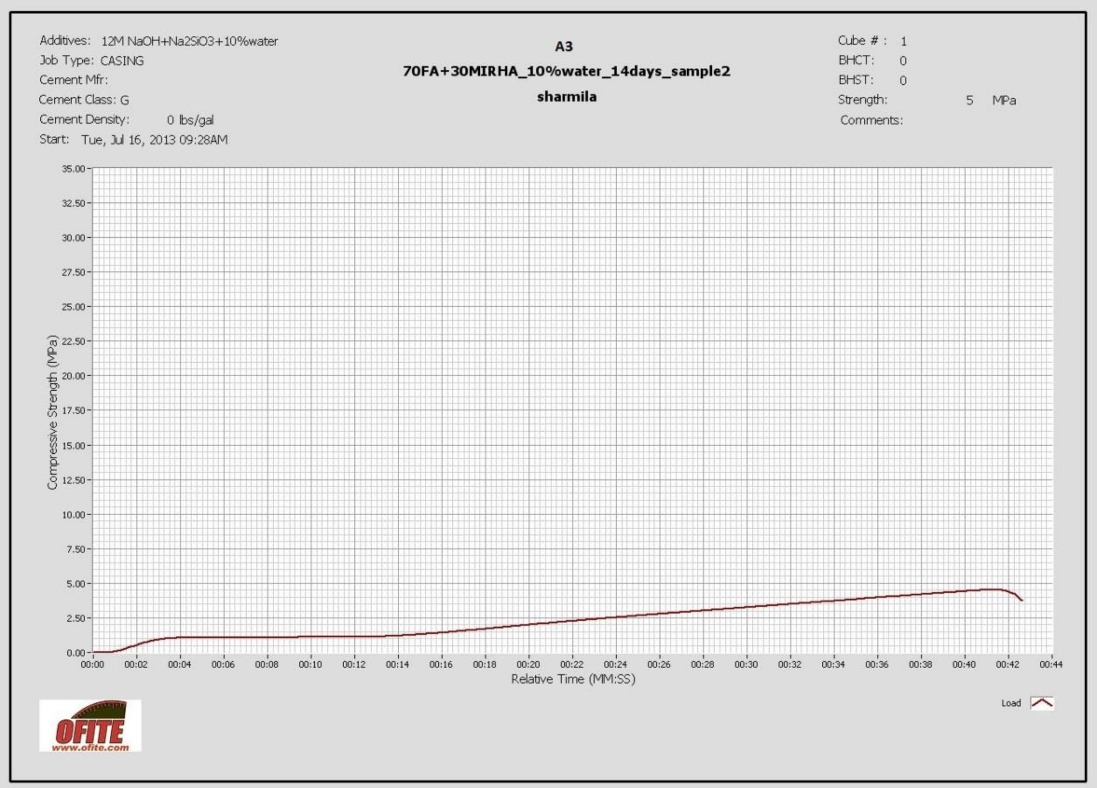

Figure 44: Compressive Strength Test Result for Batch A3, 14 days (sample 2)

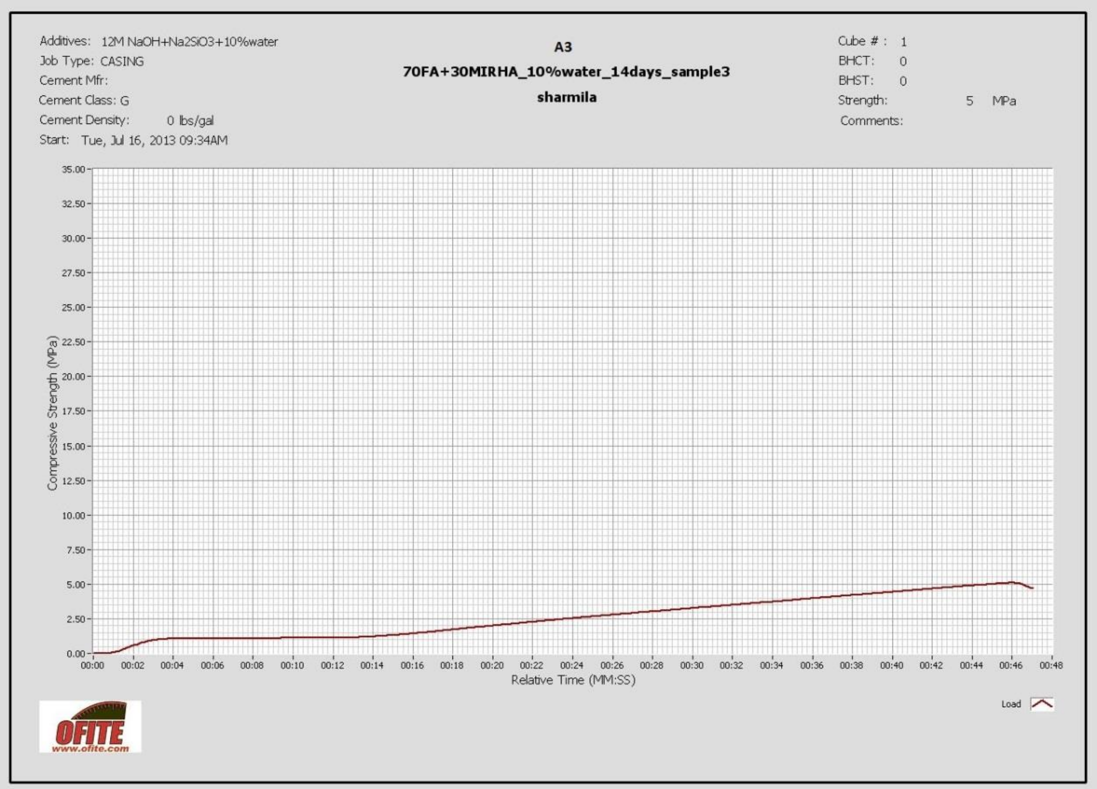

Figure 45: Compressive Strength Test Result for Batch A3, 14 days (sample 3)