Study on the Effects of Palm Oil Frond Ash (POFA) in Geopolymer Cement Properties

By

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Dissertation submit in partial fulfillment of

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Supervised By

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CERTIFICATION OF APPROVAL

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A project Dissertation submitted to the
Petroleum Engineering Programme
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Approved by,

(Raja Rajeswary A/P Suppiah)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible to the works 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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SAR-IYAH HAJIBERAHENG

ABSTRACT

Geopolymerization is the current booming field of research for utilizing solid waste and by-products. It does give a cost-effective solution to many problems where hazardous residue has to be treated and stored at critical environmental conditions. From geopolymerization, a new technology which is geopolymer cement also known as green cement has been introduced. Geopolymer cement is also said to reduced greenhouse gas footprints when compared to conventional cement slurries used in oil and gas well cementing operations.

In this project, the writer has been assigned to carry out the study on geopolymer characteristic properties of Palm Oil Frond Ash (POFA) and its effects as geopolymer cement materials. The main objectives of this project are to study the properties of palm oil frond ash based geopolymer, to find the compressive strength of the geopolymer cement by using palm oil frond ash and will be decide whether palm oil frond ash can be an alternative to replace the ordinary Portland cement (OPC) that we are currently using or not.

The experiments will be done by mixing the POFA with other materials such as alkaline activators to produce cement paste. The cement paste is then poured into specific mould size and left for curing at different curing time and curing temperature. The results of compressive strength test are recorded and analyzed.

In conclusion, the POFA geopolymer cement gives higher compressive strength in 12M of sodium hydroxide solution and with increment of alkaline activators to POFA mass ratio, the compressive strenght will increase but 100 percent of POFA geopolymer cement might not be suitable to be used as cement replacement material.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Industrialization leads to the generation and release of undesirable pollutants into the environment. In order to keep pace with the rapid industrialization there is a necessity to select such process which would cause minimum pollution in environment. In recent years, there is an increasing awareness on the quantity and diversity of hazardous solid waste generation and its impact on human health.

Geopolymer cement has been identified as one of the methods in reducing the emission of CO2. However, it is important that geopolymer cement can meet the specific requirement in order to be the substitute of current conventional cement system. The use of geopolymer in cement system is a new technology that yet needs proper study to yield better advantages of it.

In Malaysia the oil palm frond (OPF) was produced 26.2 million tonnes per year from palm oil industries. Oil palm is one of the most valuable plants and contributes to high amount of agriculture waste in Malaysia. Malaysia is the one of the country that exports the palm oil in the world besides Indonesia and Ghana. Abundant of waste from the oil palm replanting such as oil palm fronds, oil palm trunk and empty fruit bunch will be produced each year. Recycling is needed to produced something that can be used and reduce the pollution of environment.

1.2 PROBLEM STATEMENT

1.2.1 Problem Identification

Even though the Portland cement are widely use in the world to construct the concrete but the production of Portland cement still release approximately 7% of CO_2 into the environment that can be making a major greenhouse effect and the global warming of the Earth (Bondar et al, 2011). Increasing economic factor that industry should look forward to recycling and reuse of waste material as a better option to landfill and expel.

So that, the introduce of plentiful materials which are fly ash (Matthew kambic & Joshua Hammaker, 2012) and palm oil frond ash which has been suggested as alternative material that can be potential use & replace the ordinary Portland cement (OPC).

1.2.2 Significant of the Project

The Significant of this study will be to:

- Improve the literature on effect of palm oil frond ash in geopolymer properties.
- Improve public understanding about the geopolymer properties.
- Enhance and develop the geopolymer properties in related industries.

1.3 OBJECTIVES

1.3.1 To study the properties of palm oil frond ash based geopolymer.

1.3.2 To find the compressive strength of the geopolymer cement by using palm oil frond ash.

1.3.3 To decide whether palm oil frond ash can be an alternative to replace the ordinary Portland cement (OPC) that we are currently using or not.

1.4 SCOPE OF STUDY

This project will involve in the understanding of palm oil frond ash based geopolymer, its properties and application. At the end of study; the experimental result will be evaluated in order to identify and determine that palm oil frond ash as the alternative can be able to replace the ordinary Portland cement (OPC). To research on the theory and definition of terms related to the study as well as to conduct the experiment that can provide the results determining the conclusion that whether or not palm oil frond ash can be utilized as base material to making geopolymer concrete. The samples will be tested their compressive strength by compressive testing machine.

The concrete property studies included the compressive and indirect tensile strengths, the elastic constants, the stress-strain relationship in compression and the workability of fresh concrete.

1.5 THE RELEVANCY OF THE PROJECT

Nowadays people have more concern about green technology, environmental and human health hazards of CO_2 emission. Based on previous study the reason of having an environmental friendly, geopolymer cement is to reduce the impact on the environment. Portland cement is widely used in many industries such as industrial mill and concrete manufacture. Geopolymer with properties such as abundant raw resource little CO_2 emission, less energy consumption, low production cost, high early strength, fast setting. These properties make geopolymer find great applications in many fields of industry such as civil engineering, automotive and aerospace industries, non-ferrous foundries and metallurgy, plastics industries, waste management, art and decoration, and retrofit of buildings.

1.6 FEASIBILITY OF THE PROJECT

Feasibility of this project highly possible to be completed within the scope and time frame, some of the reasons are as following:

- Availability of UTP laboratory in block 15 including facilities, equipment and apparatus for students to use at all time.
- The project involves a process flow diagram that was developed by other researcher and its unit operations were known. Therefore, the experiment is possible to generate and complete in a given time period.
- Some reference has taken from previous research of UTP student.

CHAPTER 2

LITERATURE REVIEW

2.1 GEOPOLYMER

Geopolymers is a new form of binder used in cements and concrete compounds, are produced by the interaction of aluminosilicate material with alkaline solutions (Davidovits, 1991) known as geopolymerization. Slag and fly ash become popular sources materials for geopolymer instead of metakaolin because they have high silica and alumina contents and are plentiful available in landfill sites (Olivia & Nikraz, 2012).

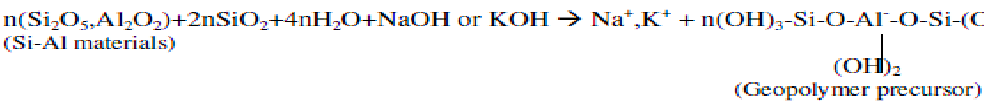
The polymerisation process involves a substantially fast chemical reaction under alkaline condition on Si-Al minerals that results in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds, as follows (Davidovits 1999):

$$Mn\ [-(SiO_2)z\text{-}AlO_2]n.\ wH_2O \qquad (2\text{-}1)$$

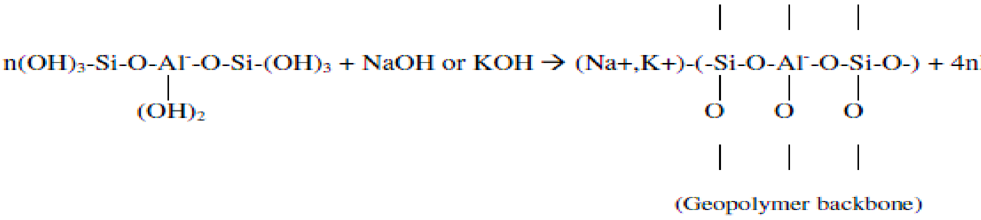
Where:

M = the alkaline element or cation such as potassium, sodium or calcium; the symbol – indicates the presence of a bond, n is the degree of polycondensation or polymerisation; z is1,2,3, or higher, up to 32.

The schematic formation of geopolymer material can be shown as described by Equations (2-2) and (2-3) (van Jaarsveld et al. 1997; Davidovits 1999):



(2-2)



(2-3)

The chemical reaction may comprise the following steps (Davidovits 1999; Xu and van Deventer 2000):

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

Davidovits (1999) proposed the possible applications of the geopolymer depending on the molar ratio of Si to Al, as given in **Table 1**.

Table1: Application of Geopolymeric Materials Based on the Silica to Alumina Atomic Ratio

Si : Al ratio	Applications
1	Bricks Ceramics Fire protection
2	Low CO2 cements and concrete Radioactive and toxic waste encapsulation
3	Fire protection fiber glass composite Foundry equipment Heat resistant composites, 200 0C to 1000 0C Tooling for aeronautics titanium process
>3	Sealants for industry 200 ^{0}C to 600 ^{0}C Tooling for aeronautics SPE aluminium
20-35	Fire resistant and heat resistant fiber composite

2.1.1 Geopolymer Cement

Davidovits (1988) stated that geopolymer cement is a type of three-dimensional CaO-free aluminosilicate binder and can be synthesized by mixing calcined kaolin and strongly alkaline solutions such as NaOH or KOH. With the development of reaction, water is gradually split out and the SiO4 and AlO4 tetrahedral units are linked alternatively to yield three types of geopolymer products: poly-sialate [–SiO4–AlO4–] (PS type), poly-sialate-siloxo [–SiO4–AlO4–SiO4–] (PSS type) or poly-sialatedisiloxo [–SiO4–AlO4–SiO4–SiO4–] (PSDS type) by sharing all oxygen atoms between two tetrahedral units (Davidovits, 1989).

Compared to Portland cement the good properties of geopolymer which are less energy consumption, less CO2 emission, high early strength, less shrinkage, low permeability, good fire and acid resistance and excellent durability (Davidovits, 1988, 1989; Davidovits et al., 1990; Duxson et al., 2007; Hongling et al., 2005; Nowak, 2008; Sofi et al., 2007; Van Jaarsveld and Van Deventer, 1999; Bakharev, 2005; Lyon et al., 1997) They agreed with the good properties above from that reason geopolymers have wide range of applications in the field of industry such as civil engineering, bridge, pavement, hydraulic, underground and militia engineering (Davidovits, 1994a).

2.1.2 Advantages and Applications of Geopolymer

As stated in Li et al, (2004) to compare the geopolymer characteristics with Portland cement characteristics as following:

1. Abundant raw materials resources: any pozzolanic compound or source of silicates or almino-silcates that is readily dissolved in alkaline solution will suffice as a source of the production of geopolymer.
2. Energy saving and environment protection: geopolymers don not require large energy consumption. Thermal processing of natural alumino-silicates at relative low temperature (600° to 800°) provides suitable geopolymeric raw materials, resulting in 3/5 less energy assumption than portland cement. In addition, a little CO2 is emitted.
3. Simple preparation technique: Geopolymer can be synthesized simply by mixing alumino-silicate reactive materials and strongly alkaline solutions,then curing at room temperature. In a short period, a reasonable strength wil be gained. It is very similar to the preparation of portland cement concrete.
4. Good volume stability: geopolymers have 4/5 lower shrinkage than Portland cement.
5. Reasonable strength gain in a short time: geopolymer can obtain 70% of the final compressive strength in the first 4 hours of setting.
6. Ultra-excellent durability: geopolymer concrete or mortar can withdraw thousands of years weathering attack without too much function loss.
7. High fire resistance and low thermal conductivity: geopolymer can withdraw 1000° to 1200° without losing functions. The heat conductivity of geopolymer varies form 0.24w/m·k to 0.3w/m·k, compared well with lightweight refractory bricks (0.3 w/m·k to 0.438 w/m·k).

2.2 THE GEOPOLYMER PROPERTIES FOR OTHER MATERIAL

2.2.1 Fly Ash

Fly ash is one of the most abundant materials on the Earth that comes from the combustion of coal in coal-fired power plants. It is important to form of geopolymer concrete due to its role in the geopolymerization process (Matthew kambic & Joshua Hammaker, 2012).There are two classes of fly ash which are Class F and Class C. Class F fly ash is made from the burning of either anthracite or bituminous coal and it has little to no self-cementing properties and contains very little calcium oxide or lime. In concrete it must be combined with some type of cementing agent such as Portland cement. This is not a very economic process if it is going to be made into ordinary concrete. In the other hand class C fly is produced through the combustion of lignite or subbituminous coal. The chemical composition depends on the mineral composition of the coal crowd (the inorganic part of the coal). Usually silica changes from 40 to 60% and alumina from 20 to 30% (Abdullah et al, 2011).

(Terzano et al, 2005) reported that the significant role in the development of the mechanical strength after material activation, the finesses of the fly ash was considered. They reported that, when the particle fraction sized higher than 45 *l*m. Is removed mechanical strength increased remarkably reaching 70 MPa in one day.

2.2.2 Alkali Activators

The most common used in alkaline activators are a mixture of sodium silicate and potassium hydroxide (NaOH, KOH) (Pacheco-Torgal et al, 2008) in producing fly ash-based geopolymer. This material was mixed with sodium hydroxide to produce the alkaline solution and the molarity (M) of alkaline solution is 7 to 10 M. The alkaline solution was prepared one day before it is mixed with fly ash.

Chindaprasirt et al. (2007) found that the optimum sodium silicate to sodium hydroxide ratio was in range of 0.67 to 1.00 give the higher strength geopolymer, meanwhile the concentration of NaOH between 10 and 20 M give small effect on the strength.

2.2.3 Microwave-Incinerated Rice Husk Ash (MIRHA)

Rice husk ash (RHA) produced from the burning process of paddy husk is a pozzolanic material that contains around 85 % -90 % amorphous silica (Ou et al, 2007). The husk itself comes from rice milling. During milling of paddy is received about 78 % weight of rice, broken rice and bran. The remaining 22 % of the weight of paddy is received as husk. This husk contains about 75 % organic volatile matter and during the firing process this husk is converted into ash is 25 % of the weight (Kamal et al, 2008). Rice husk ash has some properties that can be effect the geopolymer. The compressive strength can be develop through polymerization process of alkaline solution and fly ash blended with microwave incinerated rice husk ash (MIRHA) as Nuruddin et al. (2011) found that mixture of 95%fly ash and 5% MIRHA generate compressive strength 36% and 24 % higher than non-blended mixture by curing condition during maturing period.

2.2.4 Palm Oil Fuel Ash

Palm oil fuel ash (POFA) is one of agro-waste ash from which palm oil remains such as palm fiber and shells. The temperatures that use to burn to produce steam for electricity generation in biomass thermal power plants are 800–1000 ^{0}C. Nowadays the utilization of POFA has not been investigated as a pozzolanic material to partially replace Portland cement. (Tangchirapat et al, 2009).

Tangchirapat et al. (2009) has confirmed that increasing the proportion of POFA in high-strength concrete and reduce the water permeability of concrete. It will lead to reductions in cement usage and the cost of high-strength concrete and and good for the environment by reducing the volume of waste disposed of in landfills.

2.3 ORDINARY PORTLAND CEMENT (OPC)

Ordinary Portland cement (OPC) is the most common type of cement in general use around the world. Portland cement is a hydraulic material, in order to form exothermic bond, the cement requires adding of water and is not soluble in water. Initially designed as a cement which would set slowly, allowing enough time for it to be properly placed, and a water resistant cement which could be used in construction applications where water would come in contact with the cement. In 1824 the OPC was first patented by an English man, Joseph Aspdin, but the mix which became truly successful, and which is still used nowadays, was designed by his son, William Aspdin in around 1843. (Raw Polymers Ltd)

2.4 CEMENT PROPERTIES

2.4.1 Compressive Strength

The strength of concrete varies considerably depending on a number of variables. The source of materials is high requirement to produce a geopolymer with a high compressive strength (Xu and Van Deventer JSJ, 2002). Palomo et al. (1999b) stated that the significant factors affecting the compressive strength are the type of alkaline activator, the curing temperature and curing time. The result from some experiment stated by Kamhangrittirong et al. which is the compressive strength of geopolymers depends on a number of factors including fly ash content, the ratio of the Si/Al in gel phase, alkali concentration and its increased with the decrease of sodium silicate to sodium hydroxide ratio and increase of fly ash content in the synthesis of geopolymers.

2.4.2 Curing Temperature

The important factor if we need to setting of the concrete: the curing temperature always required (Brooks JJ, 2002). Brooks reported that for type I cement and fly ash concrete setting time was decreased by a factor of six when pozzolanic reactions were accelerated by temperature increase from 6 ^{0}C to 80 ^{0}C. The compressive strength of geopolymer concrete is increase when increasing of curing temperature in range of 30 ^{0}C to 90 ^{0}C (Hardjito et al, 2004). As shown in the figure below.

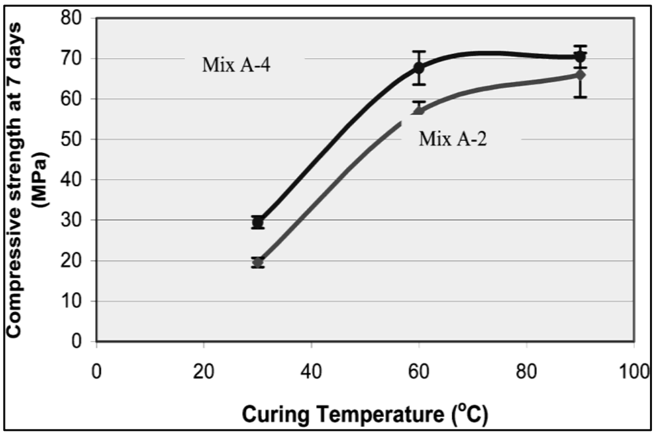


Figure 1: Effect of curing temperature on compressive strength.

Kirschner (2004) demonstrated that processing at ambient temperature was unfeasible due to a delayed beginning of setting. He also reported that curing at 75^{0}C for 4 h completed a major part of geopolymerization process and resulted in satisfactory properties of the material.

2.4.3 Curing Time

Prolonged curing time improve the polymerization process resulting in higher compressive strength. However, increase in strength for curing periods beyond 48 h. was not very significant (Hardjito et al, 2004). Puertas et al, (200) observed that the compressive strength for one day was higher when the curing was carried out at 65 ^{0}C and at rest of the age paste cured at 25 ^{0}C developed higher compressive strength than those treated at 65 ^{0}C. Compressive strength decreased on curing at higher temperature for longer period of time as prolonged curing at elevated temperature breaks the granular structure of geopolymer mixture.

2.4.4 pH

pH is considered as the most significant factor that used to control the compressive strength (Khale & Chaudhary 2007). Roy et al agreed with Khale & Chaudhary 2007 that the activating solution of pH will increase when the decreasing of cement setting time. The viscous will occur at lower pH values of geopolymeric mix and performs like cement while at higher pH: the mix attained a more fluid gel composition which was less viscous and is more workable (Phair JW & Van Deventer JSJ, 2001). Khale & Chaudhary (2007) found that pH range 13–14 is most suitable for the formation of the geopolymers with better mechanical strength.

2.4.5 Age of Concrete

Based on curing Palomo, (2003) said that about 70 % of the geopolymer strength gain in first 3-4 hr. Strength of concrete does not vary with the age of concrete when cured for 24 h, which is in contrast with the performance of ordinary Portland cement which undergo hydration process and gains strength overtime (Hardjito et al, 2004).

2.4.6 Silicate and Hydroxide Ratio

The ratio of sodium silicate to sodium hydroxide plays an important role in the compressive strength. The matrix activated with potassium silicate (KOH) obtained the greatest compressive strength while sodium silicate (NaOH) activated matrix were generally weaker.

2.4.7 Silicate and Aluminum Ratio

A high soluble silicate dosage is necessary for synthesizing alumino-silicate gel that provides good interparticle bonding and physical strength of geopolymer. Higher sodium silicate concentration was found to be beneficial to the geopolymerization of the Matakaolin / sand mixture.

2.4.8 Workability

Workability is one of the physical parameters of concrete which affects the strength and durability. Factor affecting workability are water content in the concrete mix, amount of cement & its properties, aggregate grading, temperature of the concrete mix. Chindaprasirt et al. (2006) stated that the workable flow of geopolymer motar was in the range of 110 to 135 depending on mass sodium silicate to NaOK and concentration of NaOH.

2.5 CLASSIFICATION OF CEMENT

The cement type are characterize according to the API classification as published in API Standards 10 entitled, ―Specification for Oil-Well Cement and Cement Additives. Table 2 below shows the API classification of cement

Table 2: API classification of cement (Source: (Smith, 1987)

API Classification	Mixing Water (gal/sack)	Slurry Weight (lbm/gal)	Well Depth (ft.)	Static Temperature (°F)
A (Portland)	5.2	15.6	0 to 6,000	80 to 170
B (Portland)	5.2	15.6	0 to 6,000	80 to 170
C (high early)	6.3	14.8	0 to 6,000	80 to 170
D (retarded)	4.3	16.4	6,000 to 12,000	170 to 260
E (retarded)	4.3	16.4	6,000 to 14,000	170 to 290
F (retarded)	4.3	16.2	10,000 to 16,000	230 to 320
G (basic)**	5.0	15.8	0 to 8,000	80 to 200
H (basic)**	4.3	16.4	0 to 8,000	80 to 200

Can be accelerated or retarded for most well conditions

There are a number of cementitious materials used very successfully for cementing wells besides the specific API classification cements. These materials include (1) pozzolanic-Portland cements, (2) pozzolan-lime cements, (3) resin or plastic cements, (4) gypsum cements, (5) diesel oil cements, (6) expanding cements, (7) refractory cements, (8) latex cement and (9) cement for permafrost environment. (Smith, 1987)

Other than that, there are a few physical properties of API cements, as stated in the Table 3 below:

Table 3: Physical properties of API cements (PetroWiki, 2012)

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)	—	—
Pressure/ temperature thickening-time test	Specifi-cation test schedule number, Table 10		Maximum consistency, 15 to 30 min stirring period, B_c	Minimum Thickening Time, min				
	4		30	90	90	90	—	—
	5		30	—	—	—	90	90
	5		30	—	—	—	120 max.	120 max.

B_c = Bearden units of consistency, obtained on a pressurized consistometer, as defined in Sec. 9 of API *Spec. 10A* and calibrated as per the same section.[8]

2.6 PALM OIL FROND ASH STUDIES

Oil palm grows well in wet humid parts of tropical Asia (mainly South-east Asia) and Central and South America. A tremendous amount of fibrous biomass from both the palms and the fruit processing are generated by the industry. Currently, Malaysia is still the leader in oil palm/palm oil production which produced approximately 18.77 million mt (on a dry matter basis) of oil palm fronds in 1994.

2.6.1 Palm Oil Frond

Palm Oil Frond is one of the most abundant by product of oil palm plantation in Malaysia and also available daily throughout the year when palm are pruned during the harvesting of fresh fruit bunches for the production of oil. Palm Oil Frond consist of leaflets and petioles is by product of the oil palm industry in Malaysia and their abundance has resulted in major interest potential use for animal feed.

2.6.2 Availability of Oil Palm Frond

The average economic life-span of the oil palm is 25 years. A marked increase in the cultivation of oil palm began in 1960, so that the year 1990 onwards will see a peak in replanting as shown in the table below. This will be a good opportunity to harness the ligno-cellulosic biomass or by-products of the oil palm including the fronds. Currently oil-palm fronds are left rotting between the rows of palm trees mainly for soil conservation, erosion control and ultimately the long-term benefit of nutrient recycling. The large quantity of fronds produced by a plantation each year make these very promising source of roughage feed for ruminants.

Table 4: Estimated availability of oil-palm trunks and frond (mt, dry matter basis) in Malaysia (*Source: Mohamad H, 1986*)

Year	Trunks	Fronds		Total
		Replanting	Pruning	
1990	1.32	0.25	16.92	18.49
1992	2.39	0.64	17.64	21.67
1994	4.60	0.88	17.89	22.37
1996	4.36	0.83	19.09	24.28
1998	7.48	1.42	18.18	27.08
2000	7.02	1.34	17.85	26.21

2.6.3 Mechanical Properties of Palm Oil Frond (Fiber)

Mechanical properties such as tensile strength and modulus related to the composition and internal structure of the fibers. It reported that generally the tensile strength and young’s modulus of plant fiber increases with increasing cellulose content of the fibers (Aji et al. 2009).

Some researcher reported that mechanical properties of palm oil frond fiber are hard and tough and also found to be potential reinforcement in polymer composites.

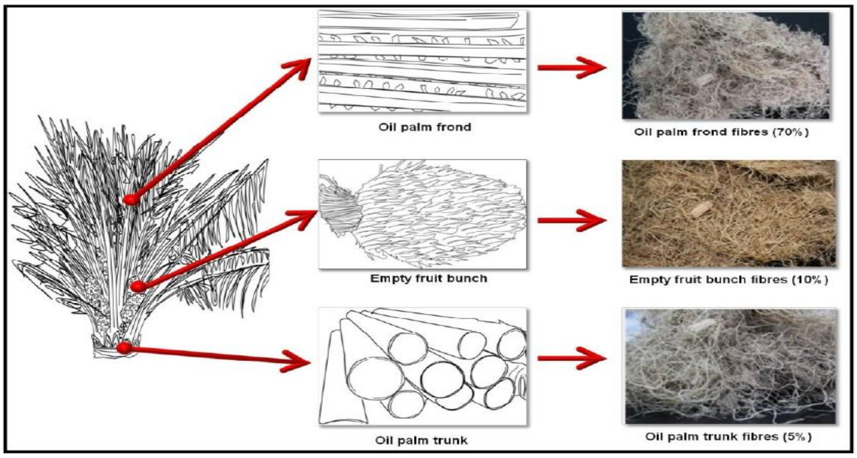


Figure 2: Oil Palm Biomass Fibers from Oil Palm Tree

2.6.4 Application of Oil Palm Frond Ash

For the application will be classified into 2 categories:

First the palm oil frond it play major role in ruminant feed source or food for animal, its use in biomass to generate energy. Second once it is transform to the ash by combustion process it can be used in the cement and concrete industry.

Figure 3: Oil Palm Frond (OPF)

Figure 4: Oil Palm Frond Ash (OPFA)

2.6.5 Why Oil-Palm Frond Technology was Well Sustained as a Viable Enterprise by Product

The oil-palm frond technology was well received because it met five attributes of innovations favorable for adoption. There do not seem to be any negative attributes with regards to oil-palm frond technology except perhaps the cost of the chopping machine. In some cases, this high cost has been overcome by reverse engineering and local fabrication of the machines. One common factor recognized among the producers was the cost-saving effect of using oil palm fronds in their production (especially in terms of feeding and labor costs). This is very significant in the context of the Malaysian animal industry as in production elsewhere.

CHAPTER 3

METHODOLOGY

3.1 PROJECT PLANING

3.1.1 Research Methodology.

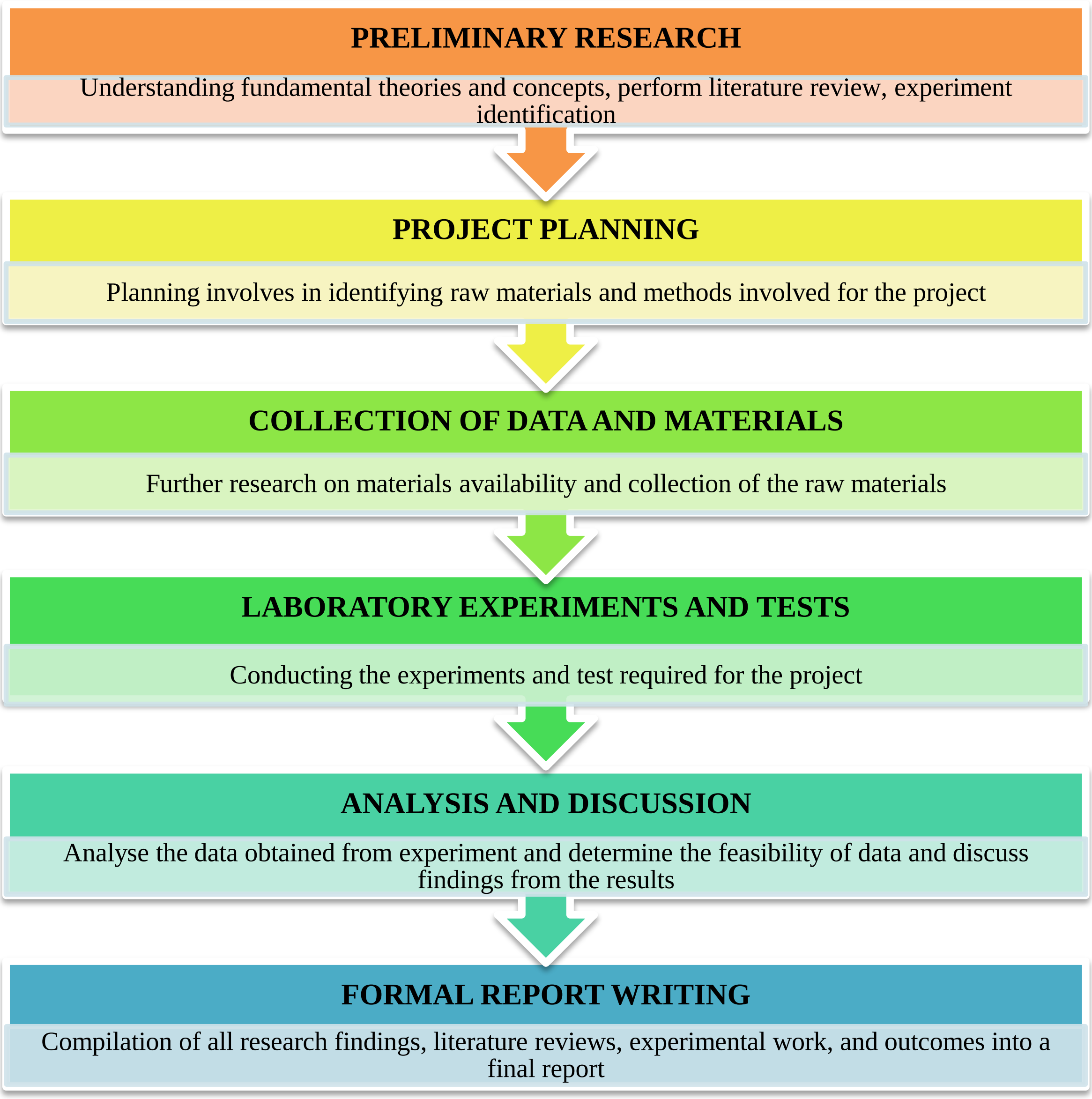


Figure 5: Flow of Final Year Project II Research Methodology

3.2 PROJECT ACTIVITIES

All project activities will be done accordingly to the mentioned sequence flow in the research methodology earlier. The detailed project activities will be further explained below:

Table 5: Project Activities

Methodology	Activities
Project scope validation	▪ Confirmation of project title with coordinator and supervisor ▪ Problem statement identification ▪ Scope of study identification
Project introduction	▪ Understanding the principle behind geopolymer ▪ Understanding the types and factors that contribute to the compressive strength of the geopolymer
Identifying and selection experiment and tools	▪ Feasibility study on each of the factor affecting geopolymer compressive strength ▪ Finalize on the factors to be testes ▪ Select an appropriate machine or tool and learn how to operate the tool
Experiment	▪ Designed experiments to test the factors listed. ▪ Repeat experiment and find the best alternative in gaining the maximum compressive strength
Analysis of data	▪ Analyze the data obtained ▪ Compare the data and come up with reasoning to explain the data
Conclusion and Recommendation	▪ Come up with a conclusion for the project and list down future recommendations.

3.3 GANTT CHART

Table 6: Project Timeline for FYP I

No	Activity in FYP I / Week	1	2	3	4	5	6	7	7	8	9	10	11	12	13	14	15	16
1	Selection of project topic		■	■					SEM BREAK							STUDY WEEK	EXAMINATION	
2	Preliminary research work (Literature review)			■	■	■	■											
3	Submission of proposal defense report							★										
4	Proposal defense (Oral Presentation)											★						
5	Project work continues									■	■	■	■					
6	Submission of interim report												★					

★ **Suggest Milestone** ■ **Process**

Table 7: Project Timeline for FYP II

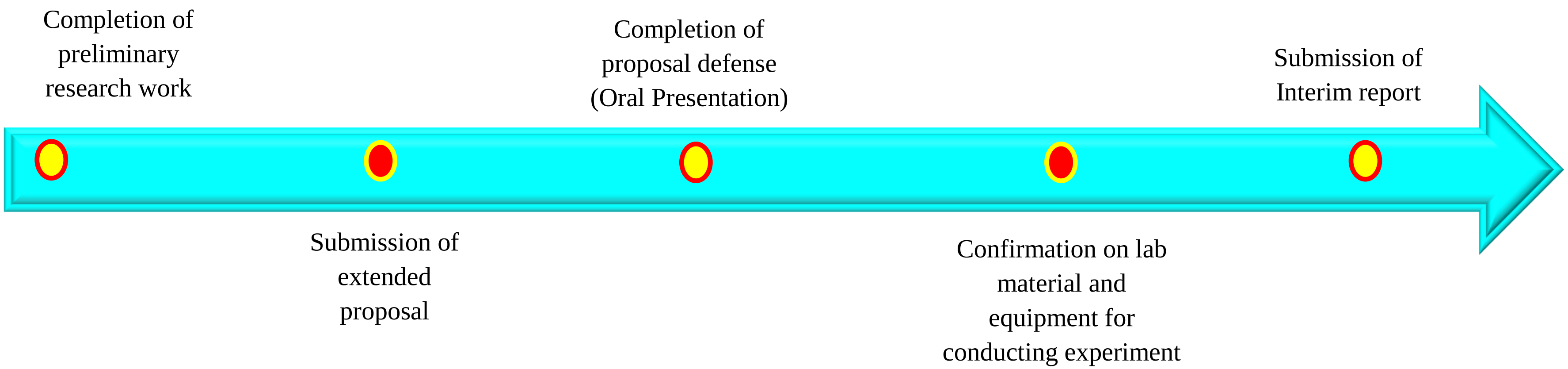
No	Activity in FYP II / Week	1	2	3	4	5	6	7	7	8	9	10	11	12	13	14	15	16
1	Prepare Lab equipment and material for experiment								SEM BREAK							STUDY WEEK	EXAMINATION	
2	Rheology study of palm oil frond ash and geopolymer cement																	
3	Submission of Progress report																	
4	Analysis of obtained result																	
5	Pre-EDX and poster presentation																	
6	Final Report and Technical Paper Submission																	
7	EDX																	
8	Final Oral Presentation																	

 **Suggest Milestone**

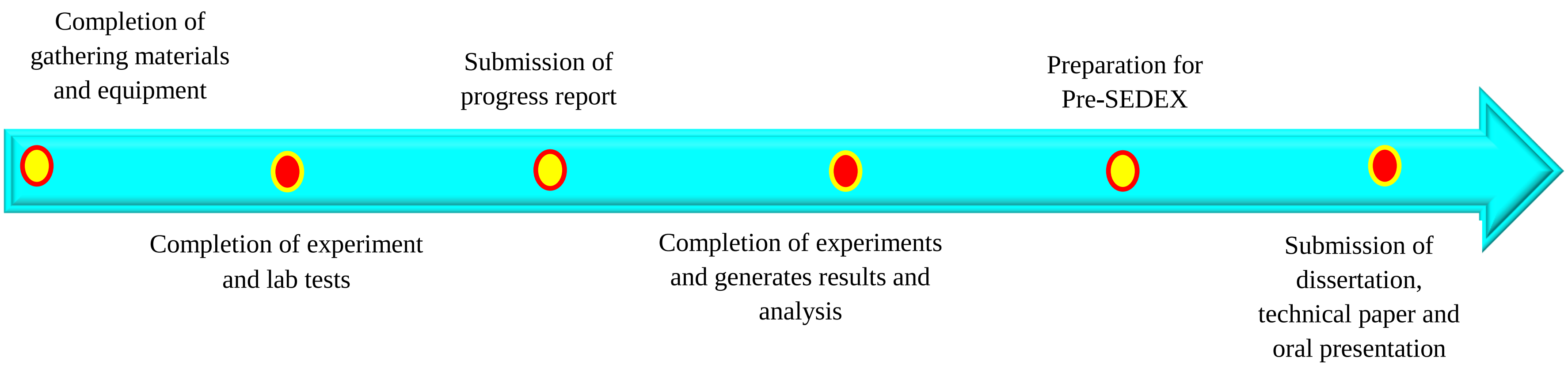
Process

3.4 KEY MILESTONE FOR THE PROJECT

FYP I



FYP II



3.5 MATERIALS, EQUIPMENT AND TESTING PROCEDURES

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

Material Required

1. Palm oil frond ash (POFA)
2. Fly ash (FA)
3. Alkaline activator (NaOH 10M, 12M, 15M)
4. Sodium Silicate Solution (Na_2SiO_3)
5. Water

Equipment Required

Table 8: Tools Used in the Experiment and its Description

Equipment	Image	Function
Cement Mould 50mm X 50mm X 50mm		✓ The mould holds the cement until it dries up. ✓ Cement is retained in mould for a day.
Grinding Machine		✓ Used to grind the incinerated palm oil ash

Weighing Machine		✓ A measuring instrument for weighing; shows amount of mass
Constant Speed Mixer		✓ Used to mix the Palm Oil ash with sodium hydroxide and sodium silicate to form geopolymer cement paste.
Compressive Strength Tester		✓ To test the compressive strength of the cement.
Sieving Machine		✓ To sieved the grinded Palm Oil ash.

3.5.1 POFA Geopolymer Cement Procedures

1. Prepare a suitable amount of POFA by drying and grinding into finer particles.
2. Weight the POFA to desired weight.
3. Prepare 1 part of 10M of sodium hydroxide with POFA weight as reference.
4. Prepare 2.5 parts of sodium silicate solution to the mass of the sodium hydroxide solution
5. Mix the sodium silicate solution together with sodium hydroxide solution in the mixer cup.
6. Mix the POFA with alkaline solution for 10 minutes at 125 rpm.
7. Add in 10 % of water (based on ash powder weight) to give workability of the cement slurry.
8. Fill up the cement slurry into 50 mm X 50 mm cement mould.
9. Mould should be tightly covered with aluminium foil. Cure the cement at 60 ^{0}C for 24 hours.
10. Cure the batches of cement at ambient temperature (room temperature) for 3 day, 7 days, 14 days and 21 days.
11. Repeat the experiment with 12M and 15M of sodium hydroxide solution.

3.5.2 Compressive Strength Testing Procedures

1. Measure the dimensions of the surface in which the load is to be applied. Since it is standardised mould of 50 mm X 50 mm, the cross-section area is constant at 2500 mm^2.
2. Place the cube in compressive testing machine and apply load uniformly.
3. Note the load at which the cube fails.
4. Calculate the compressive strength of the cube.
5. Repeat the same procedure with the remaining 1 cube.
6. 2 specimens should be tested and its average should be taken as its final compressive strength.
7. The calculation of compressive strength is done by using the following formula:

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

Where;

σ_i = the compressive strength, N/mm^2

F_i = the maximum load, N

A_i = the cross-section area at which load is applied, mm^2

Figure 6: Compressive Strength Test

3.5.3 Scanning Electron Microscope (SEM) Test

For this project, POFA and FA samples were sent to the lab for the SEM test.

CHAPTER 4

RESULT AND DISCUSSION

4.1 EXPERIMENTATION DESIGN

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

- The first experiment focusses on investigating the best water ratio needed for the geopolymer cement.
- The second experiment focusses on the different mix composition of geopolymer cement (Mix with fly ash).
- The third experiment was conducted to find the effect of curing temperature on the cement's compressive strength.
- The fourth experiment focusses on the difference of cement compressive strength when the ratio of the ash to alkali activator is changed.
- The fifth experiment was conducted to effect of grain size on the cement strength.

4.1.1 Experiment 1

It is impossible to form POFA geopolymer cement with the alkali activator alone. A certain amount of water needs to be added in order for the cement to be able to form slurry. The first experiment is focused on

- The ratio of water in the mixture with respect to the POFA mass.

Table 9: Sample tested for experiment 2 with different water ratio

Sample	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide (g)	Distilled Water (g)
A	120	35.7	14.3	10 %
B	120	35.7	14.3	20 %
C	120	35.7	14.3	30 %

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

4.1.2 Experiment 2

The experiment is focused on using different mix composition of geopolymer cement (25:75 POFA: FA). The curing time is kept constant at 7 days, whereas the sodium hydroxide concentration is varies using 10M, 12M and 15M.

4.1.3 Experiment 3

For this part of experiment, the curing temperature was investigated. For all of the cement before, a curing temperature of 60 ^{0}C was used for a period of 1 day. The experiment was repeated with different curing temperatures. The curing temperatures used are 60 ^{0}C and 100 ^{0}C. The cement was cured at these temperatures for 1 day and the curing time is 3 days, 7 days, 14 days and 21 days.

Table 10: Sample ratio for Experiment 3

Sample	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide Solution, 10M (g)	Distilled Water (g)
60 ^{0}C	200	71.4	28.6	20
100 ^{0}C	200	71.4	28.6	20

4.1.4 Experiment 4

For this experiment is focused on finding the effect of the ratio of POFA: alkali activator on the strength of the cement. From experiment 1, it was found that a water ratio of 10% to POFA mass gave the best strength. Thus the water ratio is used for all of the experiments from here onwards. Two ratios were investigated in this experiment. The first is 1:1. The second ratio is 2:1.

Table 11: Sample ratio for experiment4

Sample	Ratio	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide Solution 10M, 12M, 15M (g)	Distilled Water (g)
A	2:1	200	71.4	28.6	20
B	1:1	200	142.8	57.2	20

The two ratios above were tested with different molarity and curing days. The molarities of sodium hydroxide used were 10M, 12M and 15M. The compressive strength acquired would reveal the best ratio to be used.

4.1.5 Experiment 5

The 5th experiment investigates the effect of grain size on the compressive strength of the POFA geopolymer cement. The sizes investigated are 300 µm and 600 µm. The ratio used is as follow. A curing temperature of 60 ^{0}C was for a period of 1 day follow by external exposure curing.

Table 12: Sample ratio for experiment 5

Sample	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide Solution 10M, 12M, 15M (g)	Distilled Water (g)
300 µm	200	71.4	28.6	20
600 µm.	200	71.4	28.6	20

4.2 FINDING/ DATA GATHERING

The data acquired are arranged as per the experiments done. The experiments are done stage by stage.

4.2.1 Compressive Strength Testing Results

Table 13: Compressive Strength Test result for 100 percent POFA geopolymer cement

NaOH Concentration	Compressive strength (Mpa)			
	3 days	**7 days**	**14 days**	**21 days**
10 M	0.68 0.72	0.76 0.74	0.88 0.68	0.80 0.84
Average	**0.70**	**0.75**	**0.78**	**0.82**
12 M	1.00 0.88	0.96 1.00	1.04 1.16	1.24 1.28
Average	**0.94**	**0.98**	**1.10**	**1.26**
15 M	0.68 0.75	0.89 0.73	0.91 0.93	0.95 0.97
Average	**0.72**	**0.81**	**0.92**	**0.96**

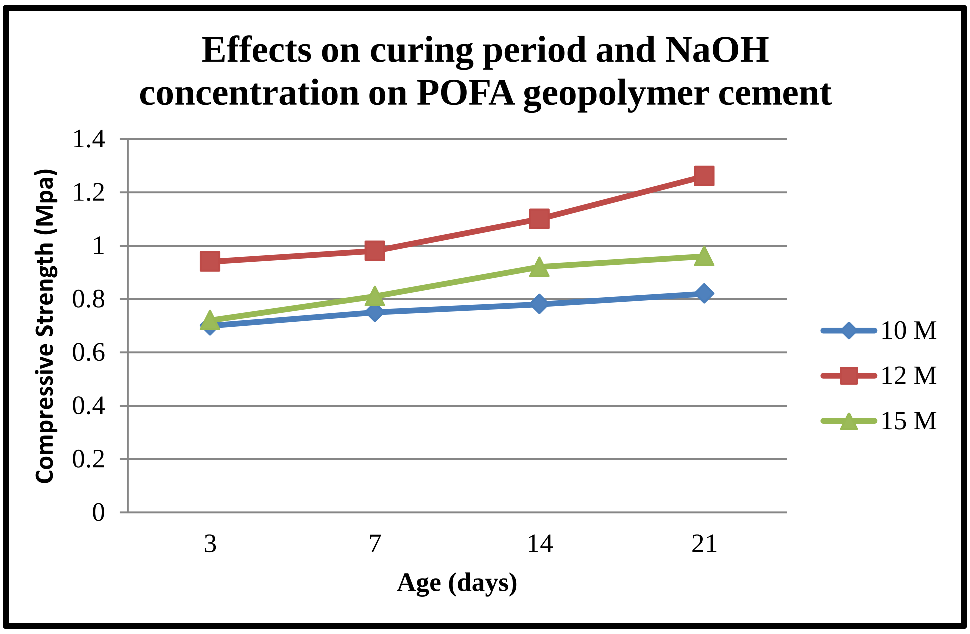


Figure 7: Effects of curing time and NaOH concentration on POFA geopolymer cement

Figure 7 above shows the effects between curing time and sodium hydroxide concentration on POFA geopolymer cements compressive strength.

For 3 day of curing and 10M of sodium hydroxide solution showed the compressive strength of 0.70 Mpa. Whereas, for the 7 days of curing time, the compressive strength obtained is 0.75 Mpa, for the 14 days of curing time, the compressive strength obtained is 0.78 Mpa. The compressive strength result increases to 0.82 Mpa for 21 days of curing time.

For 12 M of sodium hydroxide solution concentration, the compressive strength obtained for 3 day curing time is 0.94 Mpa. The compressive strength obtained for 7 days curing time is 0.98 Mpa, for 14 days of curing time, the compressive strength obtained is 1.10 Mpa, while the compressive strength recorded for 21 days curing time is 1.26 Mpa.

The compressive strength results for 15M of sodium hydroxide solution concentration, for 3 days, 7 days, 14 days and 21 days curing time are 0.72 Mpa, 0.81Mpa, 0.92 Mpa and 0.96 Mpa respectively.

From Figure 7 it indicated that the compressive strength is increasing from 3 days, 7 days, 14 days and 21 days curing time. For all sodium hydroxide solution concentrations, the compressive strength also increased from 10M to 12M of sodium hydroxide. However, the compressive strength decreases when using 15M of sodium hydroxide solution.

The result from the first experiment will lead to the ratios used in the second experiment. The data from the experiments are as shown below.

4.2.2 Experiment 1

Sample	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide (g)	Distilled Water (g)
A	120	35.7	14.3	10 %
B	120	35.7	14.3	20 %
C	120	35.7	14.3	30 %

Table 14: Results for Experiment 1

Sample	Trial 1,(MPa)	Trial 2,(MPa)	Compressive Strength,(MPa)
10%	1.60	1.52	**1.56**
20%	1.35	1.42	**1.38**
30%	1.28	1.20	**1.24**

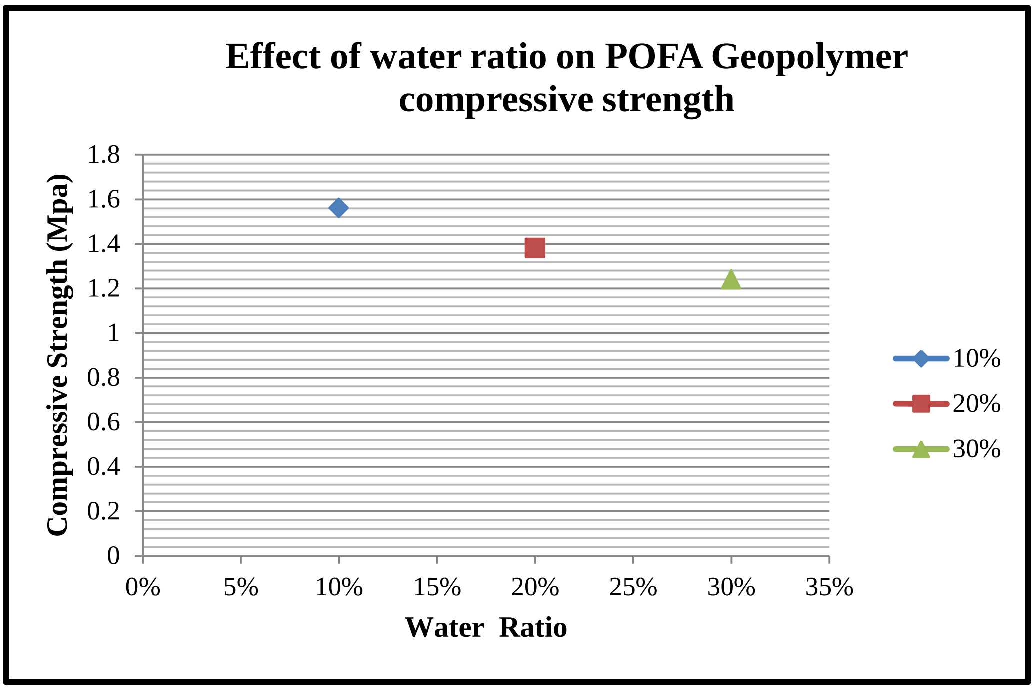


Figure 8: Effect of water ratio on POFA geopolymer compressive strength

This experiment it is found that the compressive strength of the geopolymer cement was affected by amount of water. From the first experiment it is concluded that the ratio of water should be maintained at 10% of the POFA mass. This enables it to achieve a better compressive strength.

Thus it can be concluded that the amount of water ratio for POFA geopolymer cement should not exceed 10% in order to achieve a high compressive strength.

*** From this experiment, it was found that the cement cubes did not fill up the mold as it should have been and sometime if we put more water it could be liquid, therefore it is hard to form cement. This could be attributed by add more amount of ash used.

4.2.3 Experiment 2

The experiment is repeated using different mix composition of geopolymer cement. The curing time is kept constant at 7 days, whereas the sodium hydroxide concentration is varies using 10M, 12M and 15M. The result is presented in the Table 12 and Figure 9 below.

Table 15: Compressive strength test for different composition of geopolymer cement

NaOH Concentration	**Compressive strength (Mpa)**	
	Geo Cement A 100 : 0 (POFA: FA)	**Geo Cement B 25 : 75 (POFA: FA)**
10 M	0.74	27.73
	0.76	31.28
Average	**0.75**	**29.50**
12 M	0.96	34.09
	1.00	26.60
Average	**0.98**	**30.34**
15 M	0.89	37.25
	0.73	32.93
Average	**0.81**	**35.09**

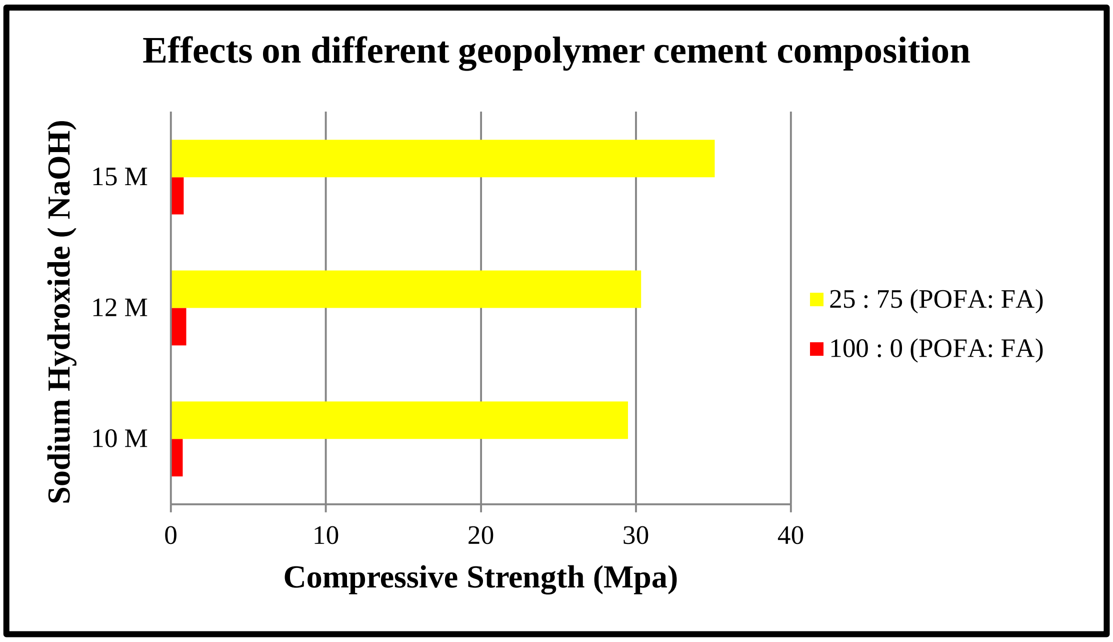


Figure 9: Effects on different geopolymer cement composition on POFA geopolymer cement

Figure 9 show that the effects on different geopolymer cement composition and sodium hydroxide on its compressive strength. The new cement composition is using 25:75 ratios of POFA and Fly ash.

The compressive strength of Geo Cement A which is using 100% POFA and10M, 12M, 15M of sodium hydroxide is recorded at 0.75 Mpa, 0.98 Mpa and 0.81 Mpa respectively.

The Geo Cement B which using 25% of POFA added with 75% of Fly Ash has higher compressive strength of 29.50 Mpa when using 10M of sodium hydroxide solution. The compressive strength increases to 30.34 Mpa when increasing the molarity concentration of sodium hydroxide to 12M. The compressive strength recorded for Geo Cement B using 15M of sodium hydroxide solution is 35.09 Mpa.

From the above chart shown is Figure 9 the compressive strength is increases when fly ash is added into the cement mix composition. The compressive strength also increases as the concentration of sodium hydroxide solution is increases from 10M to 12M for both geopolymer cement compositions. It shows that the addition of fly ash helps in improving the cement properties of POFA geopolymer cement.

4.2.4 Experiment 3

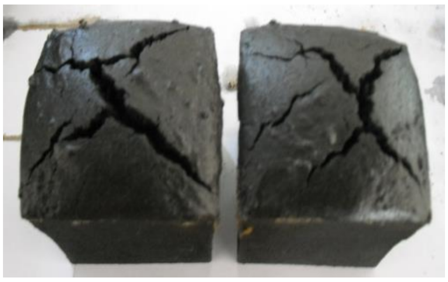
For this part of the experiment, the curing temperature was investigated. The experiment was repeated with different curing temperatures. The curing temperatures used are 60˚C and 100˚C. The cements were cured at these temperatures for 1 day. The amount of water used is limited at 10 % of ashes mass as recommended from the results of Experiment 1.The molarity of NaOH used is 12M since it has been determined from previous experiment that 12M NaOH gives out a higher compressive strength.

Sample	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide Solution, 10M (g)	Distilled Water (g)
60 ^{0}C	200	71.4	28.6	20
100 ^{0}C	200	71.4	28.6	20

Table 16: Result for Experiment 3

Curing Time / Sample	Compressive Strength at 3 days (MPa)	Compressive Strength at 7 days (MPa)	Compressive Strength at 14 days (MPa)	Compressive Strength at 21 days (MPa)
60 ^{0}C	1.00	0.96	1.04	1.24
	0.88	1.00	1.16	1.28
Average	**0.94**	**0.98**	**1.10**	**1.26**
100 ^{0}C	2.88	2.96	1.76	1.49
	5.00	3.00	1.48	1.52
Average	**3.94**	**2.98**	**1.62**	**1.50**

Table 17: Physical image of experiment 3 results

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

From Table 17 above shows that the sample of 60 ^{0}C curing temperature; the cubes are formed according to the mould and does not have any fracture occur while the sample of 100 ^{0}C curing temperature the cubes are deformed and we can see the top of sample it is began to break since it was in the oven.

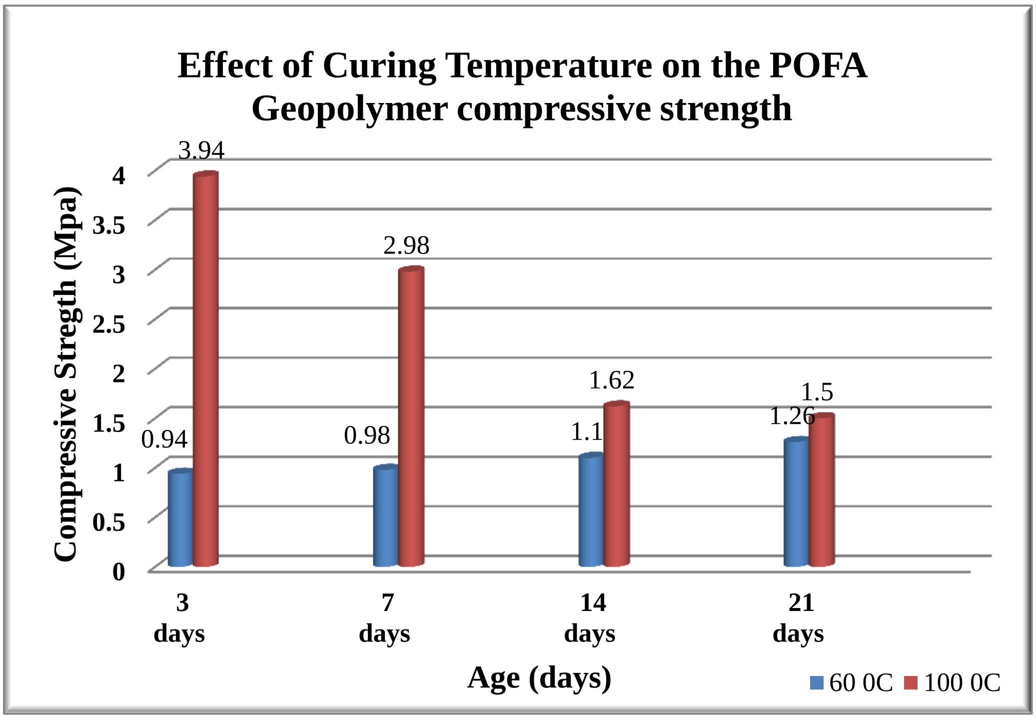


Figure 10: Effect of curing temperature on the POFA geopolymer cement compressive strength

From figure above we can see the overall compressive strength of the 100˚C cement. For 3 days of curing the compressive strength is 3.94 Mpa follow by 7 days of curing the compressive strength is 2.98 Mpa. The compressive strength of 14 days curing time show is 1.62 and for 21 days the compressive strength is 1.50 Mpa.

The compressive strength results for 60˚C curing temperature of cement; for 3 days, 7 days, 14 days and 21 days curing time are 0.94 Mpa, 0.98Mpa, 1.10 Mpa and 1.26 Mpa respectively.

Based on this chart indicated that the highest compressive strength recorded is 3.94 MPa@100˚C (3 days) and the lowest compressive strength recorded is 0.94 MPa@60˚C (3day). Although the 100 ˚C of curing temperature for cement is highest compressive strength but we cannot conclude that the result is better than 60˚C curing temperature due to many factors and can discuss in the discussion part.

4.2.5 Experiment 4

Sample	Ratio	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide Solution 12M, (g)	Distilled Water (g)
A	2:1	200	71.4	28.6	20
B	1:1	200	142.8	57.2	20

Table 18: Result for Experiment 4 (1:1 and 2:1)

Molarity \ Curing Time		Compressive strength (MPa)			
		3 days	**7 days**	**14 days**	**21 days**
10 M	**A**	0.68 0.72	0.76 0.74	0.88 0.68	0.80 0.84
	Average	**0.70**	**0.75**	**0.78**	**0.82**
	B	2.72 2.72	2.25 2.72	2.72 2.36	1.96 1.80
	Average	**2.72**	**2.49**	**2.54**	**1.88**
12 M	**A**	1.00 0.88	0.96 1.00	1.04 1.16	1.24 1.28
	Average	**0.94**	**0.98**	**1.10**	**1.26**
	B	1.64 1.76	1.44 1.16	1.04 1.36	0.92 0.84
	Average	**1.70**	**1.30**	**1.20**	**0.88**
15 M	**A**	0.68 0.75	0.89 0.73	0.91 0.93	0.95 0.97
	Average	**0.72**	**0.81**	**0.92**	**0.96**
	B	1.16 1.08	0.68 0.76	0.76 0.64	0.72 0.68
	Average	**1.12**	**0.72**	**0.70**	**0.70**

Table 19: Physical image of experiment 4: (Ratio of POFA: Alkaline solution 1:1)

Molarity	Physical Appearance
10 M	
12 M	
15 M	

Table above shows that the 10 M of sodium hydroxide after 14 days it is began to crack and for sample of 12 M of sodium hydroxide it is being fracture since 14 days. The most cracked in this experiment is sample for 15 M of sodium hydroxide since after 3 days curing time.

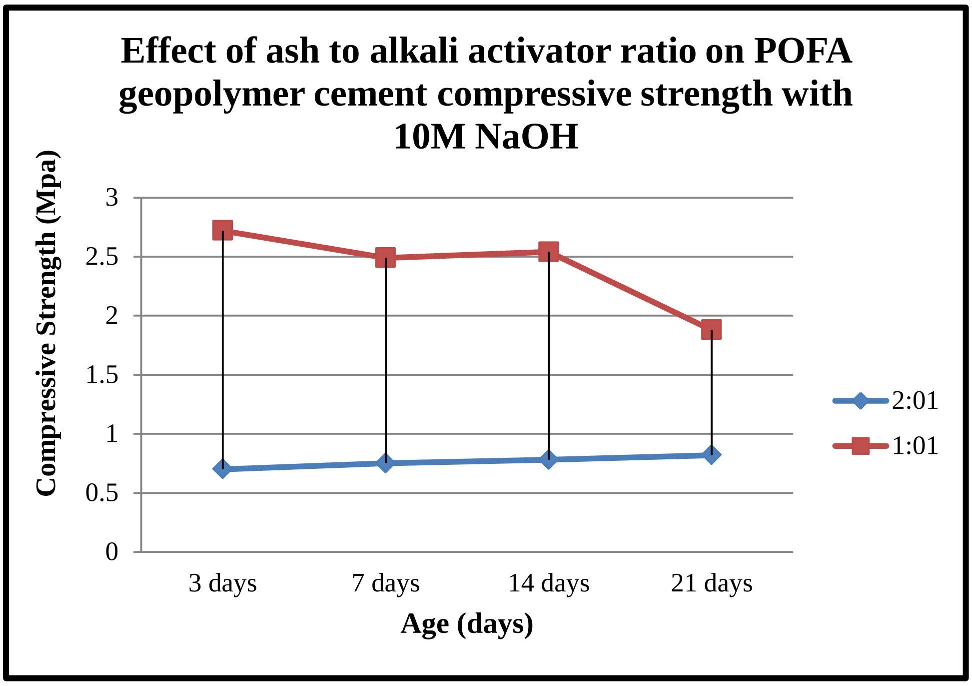


Figure 11: Effect of ash to alkali activator ratio on POFA geopolymer cement compressive strength with 10M NaOH

Figure 11 above shows the effects of ash alkaline activator ratio on POFA geopolymer cement compressive strength with 10M NaOH.

The ratio of POFA: Alkaline activator (2:1) for 3 day of curing and 10M of sodium hydroxide solution showed the compressive strength of 0.70 Mpa. Whereas, for the 7 days of curing time, the compressive strength obtained is 0.75 Mpa, for the 14 days of curing time, the compressive strength obtained is 0.78 Mpa. The compressive strength result increases to 0.82 Mpa for 21 days of curing time.

The compressive strength of POFA: Alkaline activator (2:1) for 3, 7, 14 and 21 days of curing time is recorded at 2.72 Mpa, 2.49 Mpa, 2.54 Mpa and 1.88 Mpa respectively.

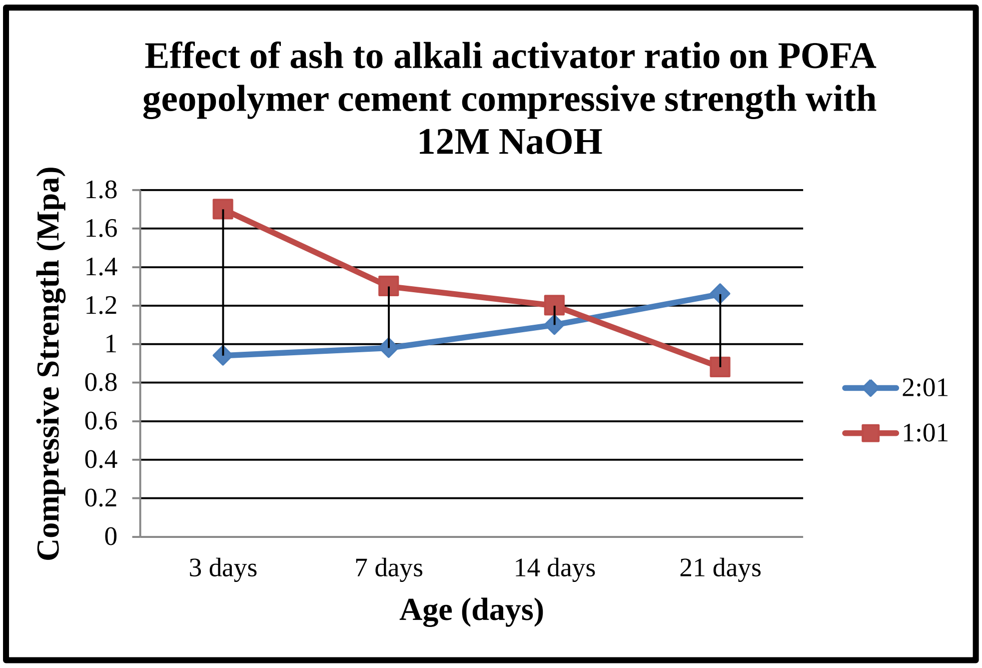


Figure 12: Effect of ash to alkali activator ratio on POFA geopolymer cement compressive strength with 12M NaOH

The ratio of POFA: Alkaline activator (2:1) for 12 M of sodium hydroxide solution concentration, the compressive strength obtained for 3 day curing time is 0.94 Mpa. The compressive strength obtained for 7 days curing time is 0.98 Mpa, for 14 days of curing time, the compressive strength obtained is 1.10 Mpa, while the compressive strength recorded for 21 days curing time is 1.26 Mpa.

The compressive strength recorded for 1:1 ratio of POFA to alkaline activator indicate that it will be decrease from 1.70 Mpa to 0.88 Mpa for 3 days, 7days, 14 days and 21 days of curing time respectively.

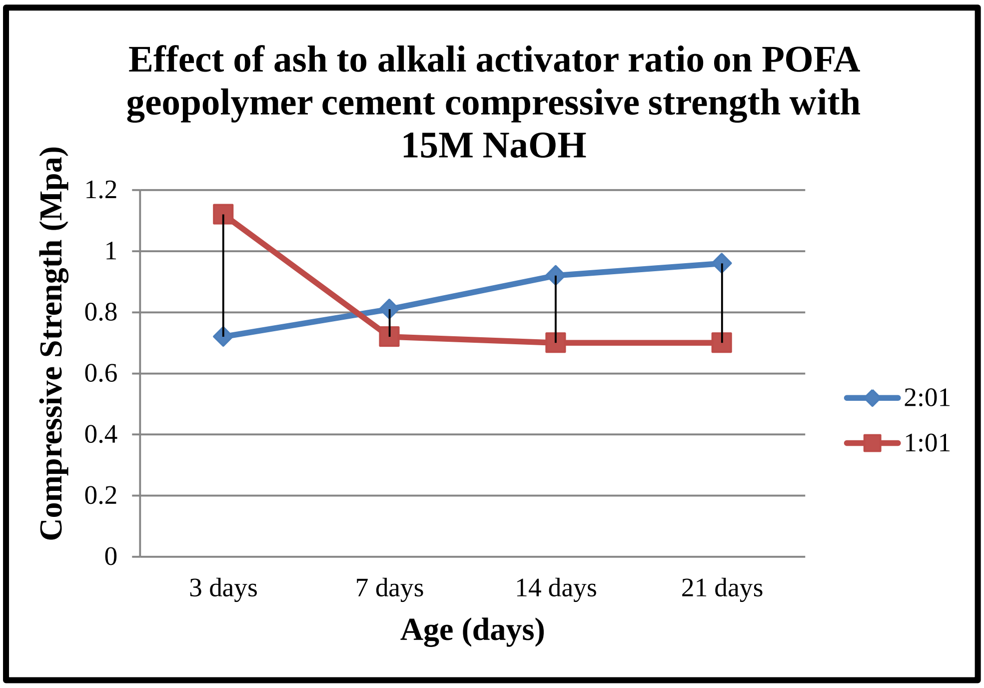


Figure 13: Effect of ash to alkali activator ratio on POFA geopolymer cement compressive strength with 15M NaOH

For 15M of sodium hydroxide solution concentration of POFA to alkaline activator (2:1), the compressive strength results for 3 days, 7 days, 14 days and 21 days curing time are 0.72 Mpa, 0.81 Mpa, 0.92 Mpa and 0.96 Mpa respectively.

The ratio of POFA: Alkaline activator (1:1) for 3 day of curing and 15M of sodium hydroxide solution showed the compressive strength of 1.12 Mpa. Whereas, for the 7 days of curing time, the compressive strength decreased to 0.72 Mpa, and will be decreased until 0.70 Mpa for the 14 days and 21 days of curing time.

4.2.6 Experiment 5

In this experiment, the grain size of the POFA was handled. The amount of water used is limited at 8 % of ashes mass as recommended from the results of Experiment 1.The molarity of NaOH used is 12 M since it has been determined from previous experiment that 12M NaOH gives out a higher compressive strength.

Sample	POFA (g)	Sodium Silicate (g)	Sodium Hydroxide Solution 12M (g)	Distilled Water (g)
300 µm	200	71.4	28.6	20
600 µm.	200	71.4	28.6	20

Table 20: Result for experiment 5

Curing Time / Sample	Compressive Strength at 3 days(MPa)	Compressive Strength at 7 days(MPa)	Compressive Strength at 14 days(MPa)
300 µm	0.84 0.84	0.92 0.88	1.05 0.98
Average	**0.84**	**0.90**	**1.02**
600 µm.	1.24 1.12	1.25 1.28	1.36 1.29
Average	**1.18**	**1.27**	**1.33**

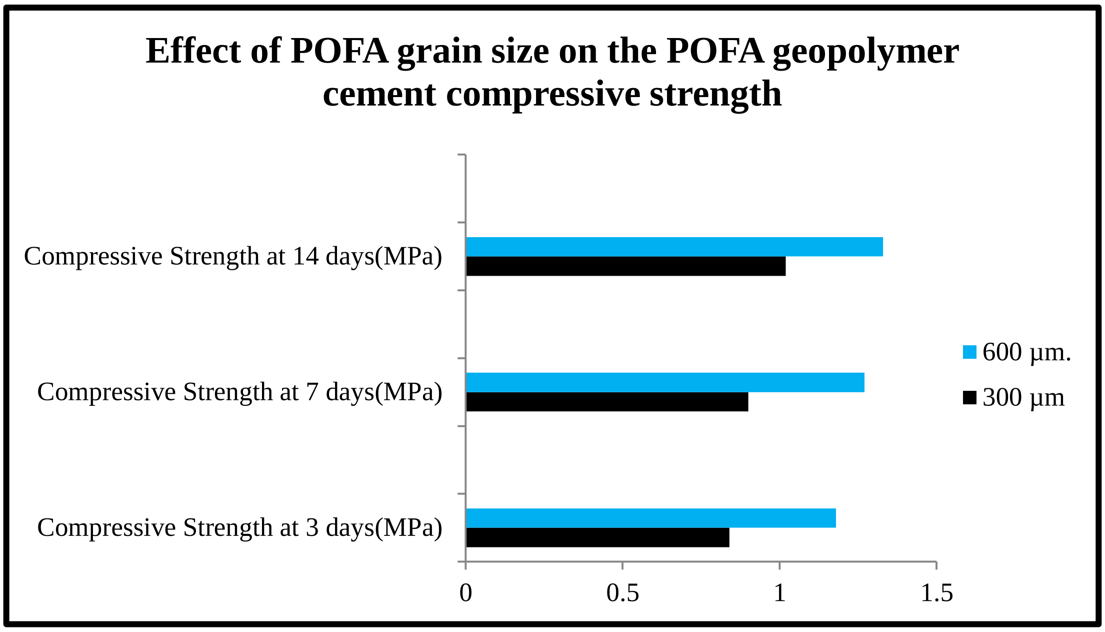


Figure 14: Effect of POFA grain size on the POFA geopolymer cement compressive strength

From figure above indicated that the highest compressive strength is 1.33 Mpa from 600µm at 14 days curing time. The compressive strength from 600µm recorded is for 3 days curing time the compressive strength is 1.18 Mpa, for 7 days curing time the compressive strength is 1.27 Mpa and 1.33 Mpa was the highest compressive strength from 14 days curing time.

The compressive strength results for 300µm of cement; for 3 days, 7 days and 14 days curing time are 0.84 Mpa, 0.90Mpa and 1.02 Mpa respectively. We can see the lowest compressive strength from this size which is 0.84 Mpa for 3 days curing time.

From Figure 14, it can be concluded that a 600µm grain size results in a higher compressive strength than the 300µm grain size cement.

4.2.7 Scanning Electron Microscope (SEM) Test Result

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens including external morphology (texture), chemical composition, crystalline structure and grain size of sample.

For this project, POFA and FA samples were sent to the lab for the SEM test. Figure 15 to 18 below show the result of SEM test 20µm and 40µm for both POFA and FA.

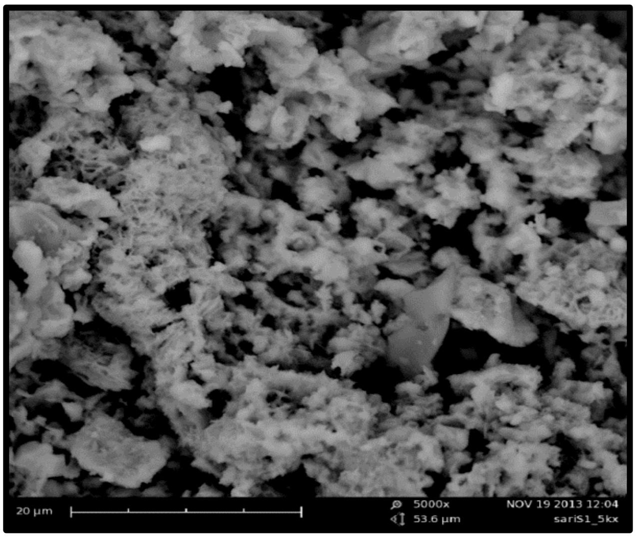


Figure 15: SEM of palm oil frond ash (20µm)

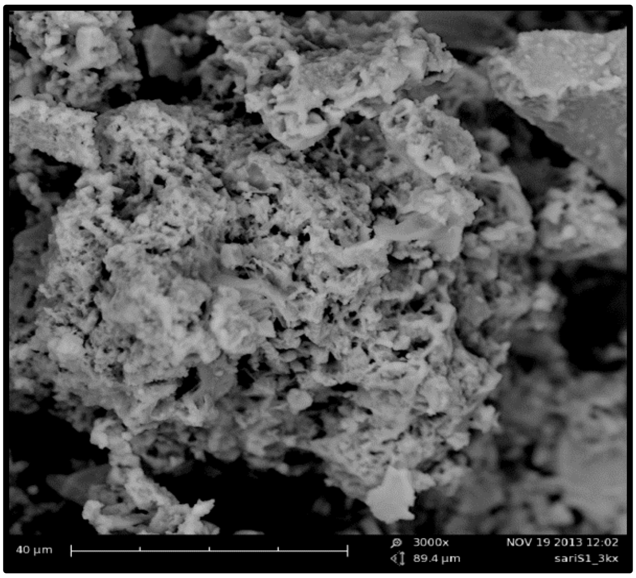


Figure 16: SEM of palm oil frond ash (40µm)

From SEM test result for POFA we can found that medium size particle of sample with crush shape structure and POFA have found spongy and porous structure of varied shape. The sample contains more porosity which may lead the sample to become brittle and weak affecting the overall mechanical properties of the sample. The main components were found to have angular and irregular shapes. The surface quality is more roughness than fly ash.

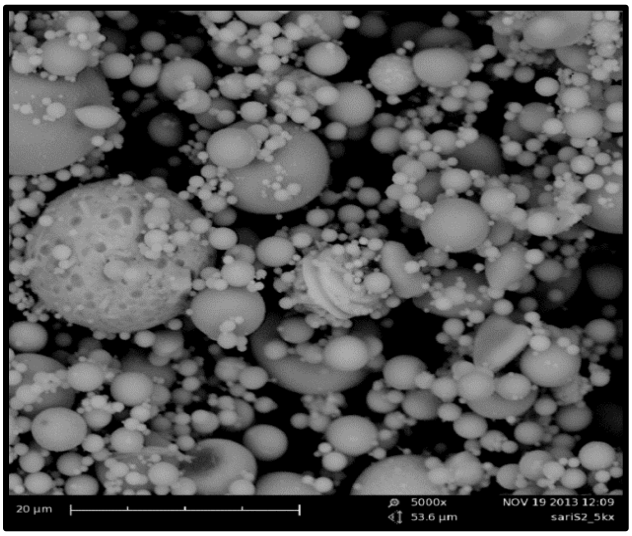


Figure 17: SEM of fly ash (20µm)

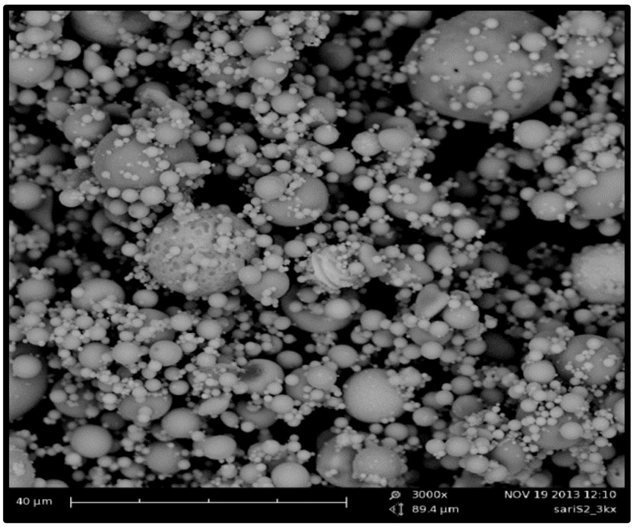


Figure 18: SEM of fly ash (40µm)

From the figures above shows that the particles shape of fly ash is spherical shape which is very important in regards of water requirement and very suitable for pozzolanic purposes. The size is also very fine; diameter of majority particles lies between 100 µm to even less than 1 µm.

The calcium hydroxides formed by the hydration of cement paste can react with fly ash more effectively due to specific surface of such higher degree.

4.3 DISCUSSION

The discussion part of the project will be divided into 6 parts. In this section, the student will analyze the findings and come up with a proper explanation on the reasons behind the results. The discussion will be made to provide explanation for each of the experiment conducted.

4.3.1 Various Molarity of Sodium Hydroxide Solution

The objective of this project is to determine the compressive strength of POFA geopolymer cement and compare the results with the API specifications for oil well cement. To identify the compressive strength; the method that has been used is producing the POFA geopolymer cement cubes and test its compressive strength.

From the results tabulated in Table 13, the compressive strength increases when increasing the molarity of sodium hydroxide solution from 10M to 12M. However, after the 12M of sodium hydroxide solution, decreases in compressive strength can be observed. This result is in similar with the results of Alonso and Palomo (2011) study, which also found that a 12M sodium hydroxide solution produced better results than the higher sodium hydroxide solution concentration. (Al Bakri et al., 2011)

On the other hand, the compressive strength is increasing proportionally with increasing in concentration of sodium hydroxide; the research was done by done by Hardjito et al. (2008). This might due to different source materials used. Hardjito used low calcium fly ash (ASTM Class F) whereas Alonso and Palomo (2001) used a high purity metakaolin. The result may vary due to these differences. (Hardjito et al., 2008)

4.3.2 Experiment 1

Experiment 1 is to find the best water ratio to be used in order to get more workability and the highest compressive strength of cement. The POFA needs a lower amount of water than usual due to its natural properties. It is the fiber so that can absorb the water easily. It is found that a water ratio of 10% is optimum. As we put more water, the compressive strength of the POFA geopolymer cements will be decreased.

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

The density of the hydrated cement paste is the primary factor to determine the strength of cement. The higher denser of cement paste, the higher the strength of the hardened cement. Thus it is important that a water to cement ratio is determined in order to produce the densest possible hydrated cement paste. From Experiment 1, we find that a 10% water to cement ratio is sufficient enough to result in to make the high cement strength. If using more than the suitable amount of water results in reduced strength since the density of the hydrated cement paste is lower. Increased in water content pushes the hydrated cement crystals apart reducing the bonding contact area between them resulting in reduced strength.

For POFA; the water / cement ratio should use 10 % of water and should not exceed that amount.

4.3.3 Experiment 2

From experiment 2 we can obtained that the compressive strength is very low strength of POFA geopolymer cement. This is however expected might due to different materials composition used from other study.

However, by addition of Fly Ash into the geopolymer cement composition, the compressive strength is higher. This is shown in the third test of this experiment whereas the compressive strength of Geo Cement B using 25:75 ratio of POFA:Fly ash is increased from 29.50 Mpa to 35.09 Mpa at 15M sodium hydroxide solution concentration and 7 days curing time. The result obtained is corresponding with study done M. Azreen et al., (2011) which found out that the compressive strength of POFA geopolymer cement with addition of FA is improved at the blended ash ratio of 70:30 ratios. (M. Azreen et al., 2011)

Aside from that, when referring to the SEM test results, we can observed that the FA has spherical shape that suitable for pozzolanic process because it is regard of water requirement and the size more fine than POFA. Therefore we may conclude that the presence of shape in FA does gives impact to the geopolymer cement by improving the compressive strength.

From this experiment; we can concluded that the geopolymer cement using 100% POFA show very low compressive strength which may be attributed to very slow chemical reaction due to less alumina in POFA.

4.3.4 Experiment 3

In Experiment 3, the curing temperature of the POFA geopolymer cement was tested. It is found that the curing temperature of 100˚C resulted in a better compressive strength than 60˚C.

Although the 100˚C curing temperature resulted in a better compressive strength than 60˚C but from this experiment we can observe that the sample of 100 ^{0}C curing temperature the cubes are deformed and at the top of sample it is began to break since it was in the oven and the result will be decreased from 3.94 Mpa to 1.50 Mpa while increasing the curing time which are 3, 7 14 and 21 days.

For curing temperature of 60˚C from this experiment can show that even though the result is lower if compared to 100 ^{0}C but from Figure 10 above indicated that the compressive strength will increased when increasing the curing time; for this case it is stated that for geopolymer cement, the ambient curing temperature should be between 30˚C to 90˚C (Hardjito et al, 2004) and the cube are formed according to the mould which is does not have any fracture occur during cure time. Ambient temperature is needed for the geopolymer pozzolanic reaction. The reaction is generally accelerated with temperature increase. It can be concluded that curing at elevated temperatures is effective (in the range of 30˚C to 90˚C) and has a more significant contribution to geopolymeric reactions.

The temperature of 100˚C retarded the development of the compressive of the strength. The compressive strength increased on curing at higher temperature, as prolonged curing at the elevated temperature broke the granular structure of the geoplymer mixture. It is theoretically accepted that a shorter exposure to higher temperature would lead to a better compressive strength. In the Experiment, the samples were cured for 24 hours at the given temperature. For this purpose, it could be concluded that a temperature of 60˚C is ambient for POFA geopolymer cement.

4.3.5 Experiment 4

From the experiment 4 we can see the effect of ash to alkaline activator ratio POFA geopolymer cement compressive strength. For 10M, 12M and 15M of sodium hydroxide the compressive strength of ash to alkaline activator (2:1) will be increased when increasing the curing time from 3 days, 7days, 14 days and 21 days. While for 1:1 ratio of ash to alkaline activator, the compressive strength will be decreased at 7 days and increased again at 14 days and for 21 days of curing time also decreased. The result not consistent due to the cracking of sample.

Refer to table 19 above for 10M sample after 14 days, it begins to break. For 12M of sodium hydroxide compressive strength start to drop because of the cracking of sample after 7 days and for 15M sample it begins to crack after 3 days resulting steadily decreasing. I observe the fracture of the sample occurred when take it out of oven and during ambient temperature curing time. For this reason, compressive strength of sample is very low because during the test of compressive strength, the apply load touch the fail part of sample then it will stop detecting the reading. From this experiment clearly indicate that as increasing the alkaline activator, it will affect to increase the adhesion of molecule of the sample and moreover decreased in the compressive strength thus it is inadvisable for the material to add alkaline activator with POFA.

4.3.6 Experiment 5

In Experiment 5, it is found that 600μm POFA grain size resulted in a better compressive strength than the 300μm POFA grain size. POFA is coarse fiber material that easy to compacted and absorbed water so that it is easy to form the cement. Finer material will require more time to set. Thus, this result in a higher degree of hydration. From this experiment; the strength development of finer material will also lag significantly behind that of coarser material. Sometime it is also depending on the type and sources of material. Based on this experiment we can conclude that coarser cement will result in a higher compressive strength due to an increased degree of hydration.

4.3.7 Curing Time

Based on the experiments conducted, it is found that the compressive strength increases with the curing time. The increase in compressive strength can be observed at all tested sodium hydroxide solution concentration. Longer curing time improved the polymerization process that occurs in the geopolymer cement. The results are reliable with study and review done by Khale and Chaudhary (2007). Thus it can be concluded that a longer curing time at room temperature results in a stronger compressive strength.

4.3.8 Source of Raw Materials

The source of raw materials is accepted as one of the contributing factor that slightly affects the compressive strength. Galau and Ismail was study that the compressive strength of geopolymer cement containing POFA from different palm oil mill gives different range of results. It shows that POFA from different mill have different characteristic based on their mill operation. (Galau and Ismail, 2004)

CHAPTER 5

CONCLUSIONS & RECOMMENDATIONS

5.1 CONCLUSION

The project entitled Study on Effect of Palm Oil Frond Ash in Geopolymer Properties has able to achieve the three objectives which are to study the properties of palm oil frond ash based geopolymer, to find the compressive strength of the geopolymer cement by using palm oil frond ash and to decide whether palm oil frond ash can be an alternative to replace the ordinary Portland cement (OPC) that we are currently using or not.

From the experiments conducted, we are able to figure out the optimum condition for POFA geopolymer cement that would result in a higher compressive strength. The compressive strength of POFA geopolymer cement is expressively influenced by the concentration of the NaOH solution. As the NaOH concentration increases, the compressive strength also increases. This might be due to the acceleration in the geopolymerization process with the increase of the NaOH concentration or molarity in certain curing time.

The curing time is importance factor that impact to the compressive strength. It is observed that, the longer the curing time, the higher the compressive strength. The longer curing time help in enhancing the polymerization process to occur.

By replacing OPC with POFA, it does help in reducing the by-product waste from palm oil factory. However, the additions of additives and other type of blended ashes may help in increasing the compressive strength of the geopolymer cement.

In conclusion, 100 percent of POFA geopolymer cement might not be suitable to be used as cement replacement material. The objective to study the effects of the POFA as the raw materials for cementing and its cement properties is achieved.

5.2 RECOMMENDATIONS

The current OPC is unstable in acidic environment. The OPC is also unstable in CO2 rich environment. The further research on POFA geopolymer cement should be continued because this technology will help in solves problems such as excessive agriculture by products and landfilling.

The next step would be to find out ways to increase the POFA geopolymer cement. As we know, well cement has to endure higher pressure in the wells and other study can be improved by adding other agricultural industrial wastes as new source of geopolymer cement. The addition of other Agro- Industrial Wastes may improve the properties of the geopolymer cement.

In addition, aside from testing the compressive strength of geopolymer cement, the thickening time and fluid loss should also be tested. The results can be compared with the physical requirement of API standard for oil cements to determine the suitability of the new geopolymer cement to be used as oil well cement.

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