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Geopolymer Paste

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THESIS

THE EFFECT OF CALCIUM HYDROXIDE ON THE STRENGTH OF
FLY ASH GEOPOLYMER PASTE



A Thesis Submitted in Partial Fulfillment of
the Requirements for the Degree of
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This study aimed to find the effect of calcium hydroxide on fly ash geopolymer. Calcium hydroxide was added to increase the compressive strength of fly ash geopolymer paste. Three weight percentages of fly ash were used, 60, 65 and 70 for class C and 50, 55 and 60 for class F respectively. The amounts of calcium hydroxide added to fly ash were 2, 4 and 7% and 10, 17 and 22.5% for classes C and F by weight respectively. Alkaline activation was by 12 M sodium hydroxide. Curing of the the geopolymer was in room temperature at 24 °C for 24 hours, 3 and 7 days under dry environment or 70 °C for 24 hours. The specimens were then tested for compressive strength and microstructure.

The result indicated improvement in microstructure of geopolymer paste. Adding calcium hydroxide enhanced the compressive strength compared with control sample. The increase in compressive strength was in the range 3.7-38.5 %. The addition of 7 % calcium hydroxide to 70 percent of solid under room temperature curing for fly ash class C appeared to improve the strength of fly ash class C and class F respectively. This study indicated the potential for improvement of mechanical properties of the fly ash based geopolymer with the addition of calcium hydroxide.

Paste with class C fly ash cured at room temperature, when calcium hydroxide was added, was more compressed, had less pores and less unreacted fly ash. When cured at 70 °C, the surface of geopolymer paste was smooth and nearly uniform, because of a near completion of geopolymerization reaction.

Student's signature

Thesis Advisor's signature

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LIST OF ABBREVIATIONS

Al	=	Aluminum
ASTM	=	American Standard Testing Method
Ca	=	Calcium
ksc	=	Kilogram per square centimeter
Kg.	=	Kilogram
M	=	Molarity
MIP.	=	Mercury Intrusion Porosimetry
min	=	Minute
mm	=	Millimeter
NaOH	=	Sodium Hydroxide
Na ₂ SiO ₃	=	Sodium Silicate
SiO ₂	=	Silica
Std	=	Standard
XRD	=	X-ray Diffractometer
XRF	=	X-Ray Fluorescence
°C	=	Celsius degree temperature
w/b	=	Water to binder ratio

THE EFFECT OF CALCIUM HYDROXIDE ON THE STRENGTH OF FLY ASH GEOPOLYMER PASTE

INTRODUCTION

The process of generating electricity by burning pulverized coal produces two kinds of ashes. The first one is called bottom ash, found at the bottom of the furnace. The other one is fly ash, which has relatively small size and light weight, and so can be found floating inside the furnace. To prevent it from polluting the air, fly ash is captured by electrostatic precipitator. Fly ash is used to mix with Portland cement to create an environmental-friendly concrete without sacrificing its strength.

Fly ash can also be used in making a new kind of construction material, without having to use Portland cement. Since the production of Portland cement gives off some 800 kg of carbon dioxide for every ton of Portland cement, and since the use of Portland cement is over 3,000 million tons each year, this new material is a major step forward to the reduction of the greenhouse gas. The new material, called Geopolymer (Davidovits, 1976). There are essentially two parts, solid and liquid. The solid part is an alumina/silica bearing material such as fly ash or metakaolin, in powder form. The liquid part is a mixture of alkaline of sodium or potassium and the solution of sodium silicate. The mixture of the solid and liquid parts result in a paste that set and harden within one to three hours. Researchers have shown that addition of calcium hydroxide into the mix yielded the paste with better strength.

This study will concentrate on geopolymers prepared using class C fly ash (high calcium) and class F fly ash (low calcium), which have a different compositions and properties.

OBJECTIVES

The main objective of this research is to investigate the effect of proportion of calcium hydroxide on the fly ash based geopolymer. The specific objectives of this study are:

1. To determine the effects of fly ash percentages on, flow, setting time and compressive strength
2. To determine the effect of various concentration of sodium hydroxide, on the dissolution of silica, calcium and alumina ion from fly ash.
3. To determine the microstructure of geopolymers.
4. To determine the relationship between compressive strength and microstructure.

Scope of Research

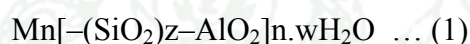
This research study will be performed within the scope listed.

1. Two classes of fly ash were used in this research, namely, class C and class F.
2. The percentages of calcium hydroxide added in class C fly ash were 2, 4 and 7% and class F fly ash were 10, 17 and 22.5%
3. The Alkaline used in this research was 12M sodium hydroxide.
4. There were two sets of specimens, the first set was cured at 24 °C for 24 hours the second set was cured at 70 °C for 24 hours, 3 days and 7 days.
5. Setting time and flow tests were conducted on all mixes.
6. Mercury Intrusion Porosimeter (MIP) was used in this research to determine the sizes of pores in specimens.
7. Microscopic photos were taken with Scanning Electron Microscope (SEM).
8. Phase compositions were identified by X-ray diffraction (XRD).

LITERATURE REVIEW

1. Introduction to geopolymers

Geopolymer is a cementitious material that consists mainly of pozzolan. When the pozzolan is blended with a solution mixture of silicate and solid alkali, the reactions result in a material that can set and harden, shows strength improvement, and has the cementitious properties like those of Portland cement paste. The pozzolanic materials that are broadly utilized as a part of this method are fly ash and metakaolin. The chemical composition occurring in geopolymer are thought to be as follows:



where

M is an alkaline element

– represents the bond between oxide groups

z is the number of SiO₂ molecule in the group.

It can take the value of 1, 2, or 3.

n is the number of the particular molecule in a chain

w is the number of water molecule

Geopolymers are based on fundamental poly (sialates). Sialates is an abbreviation of alkaline silicon-oxo-aluminate. The alkaline is from sodium-potassium-lithium-calcium and the term poly (sialates) covers all geopolymer containing at least one (Na,K,Li-Ca) (Si-o-Si),(Na,K,Li,Ca)-sialate unit (Davudovits, 1976) Sialate molecular structures involve at least four elementary unit classified according to the Si: Al atomic ratio shown in the Figure 1

1.1 sialates, poly(sialates) Si:Al = 1,(-Si-O-Al-O-) are chain and ring polymers that are the result of the polycondensation of the monomer, ortho-sialate (OH)₃-Si-O-Al-(OH)₃

1.2 sialates – siloxo, poly (sialates – siloxo) Si:Al = 2, (-Si-O-Al-O-Si-O-) may be considered as the condensation result of orthosialate with ortho-silicic acid Si(OH)₄. There are three isomorphs, a linear (-Si-O-Al-O-Si-O-), mono-siloxo-sialate and 3 cycles.

1.3 sialates – disiloxo, poly (sialates – disiloxo) Si:Al = 3 (-Si-O-Al-O-Si-O-Si-O-) may be considered as the condensation result of orthosialate with two ortho-silicic Si(OH)₄. The sialate unit may be at the beginning, in the middle or at the end of the sequence. There are six isomorphs: 2 linear, 2B ranched and 2 cycles.

1.4 sialate link , poly (sialates-multisiloxo) Si:Al > 3 It designates the bridge Si-O-Al between two poly(sialate), poly (silanol) or poly (sialate) chains.

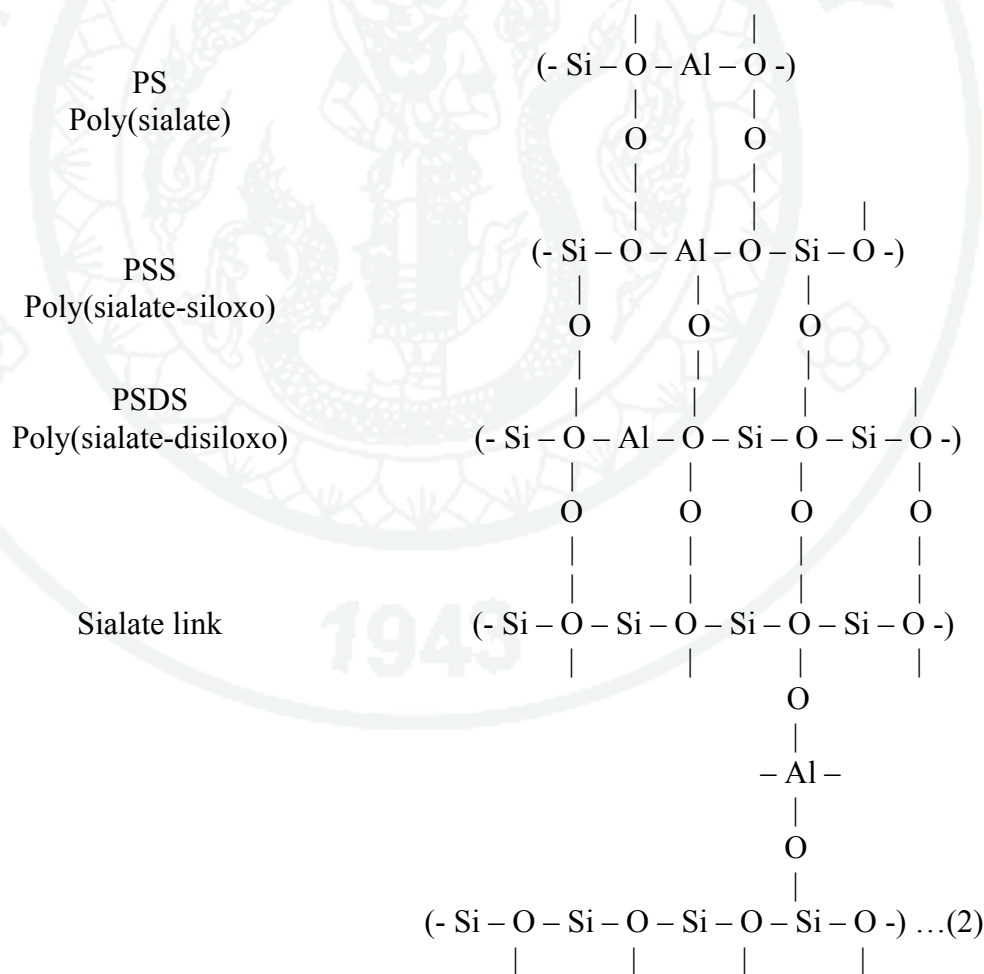


Figure 1 Geopolymeric molecular networks

2. Fly ash

Fly ash is the ash from the combustion of coal. It can be classified into 3 types based on its conditions:

1. Bottom ash is the ash with large particles. Generally, its size is approximately 0.5-10 mm and found at the bottom of a kiln. Bottom ash is transferred from a kiln using a belt and constitutes about 18% of overall ash.

2. Slag ash is the ash that melts and aggregates becoming a slag on the wall of a kiln, generally found in tiny amount.

3. Fly ash is the ash with fine particles having size between 0.001-1.00 mm flying along with hot gas in a chimney and caught by an electrostatic precipitator.

Since fly ash obtained after coal has been used is abundant and has the potential in the construction industry, there are studies that focus on its applications. Coal from each location has different composition and properties, which subsequently has an effect on the physical and chemical properties of fly ash.

2.1 Chemical Properties

The major oxides of fly ash include SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , K_2O , SO_3 . These chemical compositions vary quantitatively according to the source of coal. This is a problem in using fly ash because of the difficulty in quality control since fly ash is a material produced from the combustion of coal. Coals from America, Europe, and Japan are mostly bituminous. They are fine quality coal and produce high heat. Lignite, found at Mae Mo, Lampang of Thailand, on the other hand, produced low heat. Therefore, the chemical compositions of fly ash obtained are different. Fly ash, in general, has total SiO_2 , Al_2O_3 , and Fe_2O_3 approximately 66-92%. For fly ash from Mae Mo in Thailand from 1990-1997, the amount of these compositions was between

72-80% and the amount of CaO was 12% (Electricity Generating Authority of Thailand, 1999).

2.2 Physical Properties

The physical property crucial to the properties of concrete is the particle size of fly ash, which depends on the type of coal and how it's combusted and the efficiency of the electrostatic precipitator. In general, the particle size of fly ash is 0.001-1 mm and the fly ash found at Mae Mo Lampang has particle size between 0.03-0.42 mm.

2.3 Type of fly ash

Fly ash can be classified into three classes according to the ASTM standard (ASTM C619-91). The classifications are class C, F and N. Two classes of fly ash were used in this research for class C and class F. The classification factor of each class is shown in the Table 1.

Table 1 Classification of fly ash

Chemical requirements for fly ash classification	Class of fly ash	
	Class F	Class C
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	70	50
Sulfur trioxide (SO_3), max, %	5	5
Moisture content, max, %	3	3
Loss on ignition, max, %	6	6

3. Alkali hydroxide solutions and silicate compound

The most popular alkali hydroxides used for catalyzing the synthesis of geopolymer are sodium hydroxide and potassium hydroxide since they have good corrosive property.

3.1 Sodium hydroxide

Sodium hydroxide is an activator and mostly used in geopolymer synthesis because its price is not very high and it is the most popular alkali hydroxide. Sodium hydroxide is used for the synthesis of geopolymer when both fly ash and metakaolin are used as the starting material. It is widely used because it is low cost and the viscosity is not too high. A significant property of sodium hydroxide is its high corrosive property compared to other alkali because geopolymer is needed in very high quantity and sodium hydroxide can be used in a wide range of temperature and high humidity condition.

Using sodium hydroxide as an activator of the starting materials to produce geopolymers is known to form zeolitic structure. In an investigation in some systems, there was no obvious negative impact including basic factor or the combination of factors that cause the formation of zeolite and the reduced strength.

3.2 Silicate compounds

Silicate compounds widely used is sodium silicate solution. Sodium silicate solution that has high Na_2O to SiO_2 ratio will give high compressive strength. However, this problem is resolved by adding water to make it works better.

4. Consideration for curing of geopolymers

Accelerated curing of Portland cement has received wide attention. There is a method to predict the strength of 28 day old concrete from only 1 day of incubation by using basic principle as a tool for quality control for the construction industry because strength is very important. But in terms of microstructure it's usually not given very much interest in academic consideration. The objective of curing by accelerating temperature is important and there is a difference between curing and aging test of geopolymer (Resheidat and Ghanma ,1997)

Tests to shorten curing time is important to reduce testing steps in order to accept the period of each age of geopolymer in a short period of time in many conditions and variables. To achieve the objectives, the followings should be focused. (Provis and van Deventer, 2009)

1. In the first period of curing, normal curing should be performed completely before entering the stage of curing acceleration to develop microstructure as normal as possible.
2. Curing by temperature acceleration should be compared with normal curing in order to compare the significance of curing and experimental results.
3. Curing should be maintained in a closed system to prevent moisture loss, exposure to carbon dioxide, and leaching in the pores, etc. in order to prevent the impact from the outside.

A study has found that for the development of compressive strength of metakaolin-based geopolymer mortar cured at ambient temperature the development of compressive strength will increase quickly in the initial stage of curing, approximately 3 days, with compressive strength nearly 70 MPa. Compressive strength begins to be stable after 28 days of curing. When comparing curing at 28 and 360 days, it can confirmed that the compressive strength develops rapidly in the initial stage of curing. (Lloyd, 2008)

4.1 Curing of geopolymers synthesized from metakaolin

4.1.1 Curing at ambient temperature

For the compressive strength development of metakaolin geopolymer mortar cured at 23 C, the initial stage has quick development with nearly 70 MPa in 3 days. The rate decreases when cured to 28 days. (Palomo *et al*, 1999)

4.1.2 Curing at 95 °C

A study of the result of curing until the desired age by accelerating the temperature at 23°C found that the compressive strength of metakaolin-based geopolymer mortar cured at 23 °C for 28 days is decreasing compared to curing at 95 °C. For samples with similar compositions cured at 40°C for 7 days, the compressive strength that increases for sample cured for 1 day at 23°C. The curing for 1 and 3 days at 95 °C shown decrease of compressive strength of approximately 30 MPa after 25 days of curing, a decrease of about 60%. This is a comparison of strength regression occurred in calcium aluminum cement with high water/cement ratio and may lead to a failure of the concrete production with this type of binder (Dexson, 2006)

5. Effect of calcium

Role of calcium in a mixture of geopolymer was still not clear, but it was noted that geopolymer gel and calcium silicate hydrate formation occurred during the reaction.

For geopolymer, the concentration of alkali hydroxide played an important role in determining the pattern and calcium silicate hydrate form in geopolymer with low alkaline hydroxide and the product of the reaction will favor the formation of calcium silicate hydrate while higher concentration of alkaline hydroxide will favor the formation of geopolymer gel. This difference is the fact that shows that high hydroxide concentration is an obstacle for Ca^{2+} for the dissolution silicate and aluminum in the form of geopolymer gel and on the other hand when the concentration of OH^- is low, the dissolution efficiency of Ca^{2+} will be higher and more calcium silicate hydrate will be formed.

The addition of calcium has been noted that it has an influence on the hardening process which improved the mechanical properties in terms of compressive strength of fly ash-based geopolymer. The addition of calcium helps improve the compressive strength, a mechanical property, from curing at ambient temperature. At the same

time, it also decreases mechanical property of curing at elevated temperature because there is calcium compound that hinders the development of three-dimensional network structure.

Dombrowski, *et al.* (2006) investigated the influence of calcium content on the structure and structure and thermal performance of fly ash-based geopolymers. The aluminosilicate hard coal ash was mixed with 8 M sodium hydroxide solution. 0, 4, 8, 14 and 20% calcium hydroxide solutions were added. The samples were cured at 40°C and 100% relative humidity for 3 days, and were cured at normal temperature for 28 and 111 days. Some samples were treated at 600, 800 and 1,000 °C. The resistance of binder of Portland cement and geopolymer were compared. The test results showed that addition of 8% calcium hydroxide solution resulted in higher compressive and flexural strengths.

Temujin (2009) studied the effect of the addition of calcium oxide and calcium hydroxide into fly ash based geopolymer and curing the mixtures at 20 °C and at 70 °C. The result showed that the geopolymer specimens under 20°C curing yielded higher compressive strength than those that went through 70 °C curing. The study also showed that addition of calcium hydroxide gave higher strength than addition of calcium oxide.

Khater (2012) studied the effect of calcium on geopolymerization of aluminosilicate waste. Demolished wall/waste concrete Ratio of 60:40 % by weight use as partial replacement of metakaolin. Sodium hydroxide and sodium silicate of 3:3 % ratio by weight were used. Curing was at 23°C in tap water and 40 °C and drying treatment at 80°C to 24 hour. The study found 80C cured specimens with 10 % calcium hydroxide showed an enhancement of mechanical and micro structural properties of aluminosilicate waste.

MATERIALS AND METHODS

Material

1. Two classes of fly ash were used in this study. Class C fly ash was obtained from Mae Moh district, Lampang province, to the north of Thailand. Class F fly ash was obtained from Indonesia. The oxide compositions have been obtained by X-Ray Fluorescence (XRF) analysis and are as shown in Table 2.

Table 2 Oxide composition of the two samples of fly ash used in the study

Chemical composition of Fly ash	Fly ash Class C (%)	Fly ash Class F (%)
SiO ₂	35.3	62.9
Al ₂ O ₃	20.6	25.18
Fe ₂ O ₃	14.06	3.25
CaO	18.17	0.85
MgO	2.03	0.64
Na ₂ O	1.09	0.35
K ₂ O	2.49	1.28
LOI	0.34	3.4

2. Sodium silicate solution, grade B (Na₂O =14.7%, SiO₂ = 29.4% and water = 55.9% by mass.).

3. Sodium Hydroxide (NaOH) in flake from (98% purity).

4. Calcium hydroxide

5. Water.

6. Volume metric flask 1,000 ml
7. Beaker 1,000 ml
8. Cylinder flask 1,000 ml
9. Compressive strength testing machine
10. Plastic cylindrical molds (40x80mm).
11. Vicat test set
12. Flow test set
13. Hot air oven
14. Scanning Electron Microscope SEM
15. X-Ray Fluorescence (XRF)
16. X-Ray Diffractometer (XRD)
17. Mercury Intrusion Porosimetry (MIP)
18. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)

Methods

1. Mixing of Geopolymer

1.1 Fly ash class C – based geopolymer

Determine to compressive strength by using a mixture of 60% w/w class C fly ash and 2, 4, and 7% w/w calcium hydroxide and adding 12 M sodium hydroxide and sodium silicate solution ratio 1:1. The mixture was cured at room temperature for 1, 3, and 7 days and at 70 °C for 1 day. The specimens were then tested for compressive strength. The productive was repeated using 60, 65 and 70 % w/w of the ash. Details of the mixes are shown in Table.3

1.2 Fly ash class F – based –geopolymer

To determine the compressive strength by using a mixture of 50% w/w class F fly ash and 10, 17 and 22.5 w/w calcium hydroxide and adding 12 M sodium hydroxide and sodium silicate solution. The mixture was cured at room temperature for 1, 3 and 7 days and at 70 °C for 1 day. The specimens were then tested for compressive strength. The productive was repeated using 50, 55 and 60 % w/w of the ash. Details of the mixes are shown in Table.3

Table 3 Mix proportions for geopolymer

Mix NO.	Designation	Solid % by weight	Fly ash % by weight	Ca(OH) ₂ % by weight	Solution (% by weight)	
					NaOH	Na ₂ SiO ₃
1	C60-0	60	60	0	20	20
2	C60-2	60	58	2	20	20
3	C60-4	60	56	4	20	20
4	C60-7	60	53	7	20	20
5	C65-0	65	65	0	17.5	17.5
6	C65-2	65	63	2	17.5	17.5
7	C65-4	65	61	4	17.5	17.5
8	C65-7	65	58	7	17.5	17.5
9	C70-0	70	70	0	15	15
10	C70-2	70	68	2	15	15
11	C70-4	70	66	4	15	15
12	C70-7	70	63	7	15	15
13	F50-0	50	50	0	25	25
14	F50-10	50	40	10	25	25
15	F50-17	50	33	17	25	25
16	F50-22.5	50	27.5	22.5	25	25
17	F55-0	55	55	0	22.5	22.5
18	F55-10	55	45	10	22.5	22.5
19	F55-17	55	38	17	22.5	22.5
20	F55-22.5	55	32.5	22.5	22.5	22.5
21	F60-0	60	60	0	20	20
22	F60-10	60	50	10	20	20
23	F60-17	60	43	17	20	20
24	F60-22.5	60	37.5	22.5	20	20

1.3 Preparation the fly ash , sodium hydroxide solution 12 molar concentration, sodium silicate solution and calcium hydroxide. Mixing procedures was as follows.

1.3.1 Place the sodium hydroxide solution and fly ash into the mix machine and mixed for 5 minutes.

1.3.2 Pour in the sodium silicate solution and mix for 5 minutes.

1.3.3 Fill the geopolymer in cylindrical mold, cure the geopolymer in hot air oven 70 °C for 24 hours and room temperature 24 hours, 3days and 7days

2. Compressive strength

Compressive strength is one of the most important properties of concrete, and in this case, of geopolymer. This is because the development of geopolymer as a construction material is aimed at being used as compression member material, similar to concrete. Also, as several engineering properties such as shrinkage, durability, tensile strength, flexural strength and compressive strength of a material are normally closely related, the compressive strength is widely used as an index indicating the overall quality of the material. The compressive strength of the geopolymer is tested in accordance with a standard method for testing the compressive strength of cement mortar ASTM C109. The method specifies the use of 40mm x 80 mm cylinder molds. In this study geopolymer paste is first placed in the molds and then left to set and harden for 24 hours. It was then demolded and left to cure in the specified room temperature for 1 day, 3 days and 7 days and elevate temperature at 70 °C for 1 day. The specimens were removed from curing room and tested under compression until it reached failure. The compressive strength was equal to the force that breaks the specimen divided by the cross section area of the specimen.

3. Dissolution of ion

To determine leaching test of silica calcium and alumina fly ash by using 9 and 12 M sodium hydroxide to determine leaching value after 3, 10, 20 and 30 minute

by choosing the maximum compressive strength of mixtures of both class C and class F fly ash for testing. Place sodium hydroxide and fly ash in the mixer and mixed for 3, 10, 20 and 30 minute .Varying the each of mix designation and mixing procedures as shown in the Table 4.

3. 1Fill fly ash into the mixer and fill 9 M of sodium hydroxide solution mixed for 3, 10, 20 and 30 minutes and fill the same by adding calcium.

3.2 Preparation mix was pour in vacuum filter set by using a 0.50 μm membrane filter.

3.3 Turn on the vacuum pump shown in to increase efficiency of sample filtrated through a 0.50 μm membrane filter as shown in the Figure 2 and 3

3.4 Filtrated solution was analyzed to determine quantities of dissolution ion from fly ash by using Inductively Coupled Plasma Optical Emission Spectroscopy shown in the Figure 4.



Figure 2 Filtration set

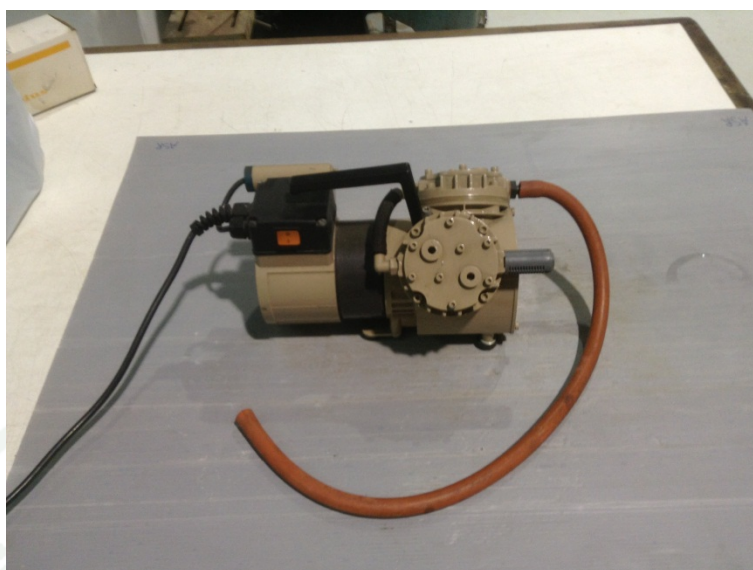


Figure 3 Vacuum pump

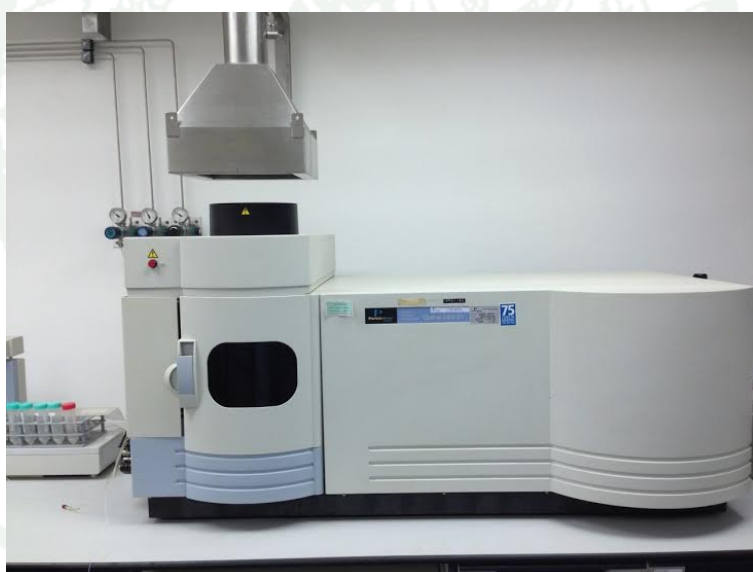


Figure 4 Inductively Coupled Plasma Optical Emission Spectroscopy

Table 4 Mix proportions for dissolution of fly ash with 9 and 12 M hydroxide

Mix	Designation	% by weight			
		Solid	Fly ash	Ca(OH) ₂	9 M NaOH
1	C60-0	60	60	0	20
4	C60-7	60	53	7	20
22	F60-10	60	50	10	20
23	F60-17	60	43	17	20

Mix	Designation	% by weight			
		Solid	Fly ash	Ca(OH) ₂	12 M NaOH
1	C60-0	60	60	0	20
4	C60-7	60	53	7	20
21	F60-0	60	60	0	20
23	F60-17	60	43	17	20

4. Microstructure

4.1 Scanning Electron Microscope, SEM

Accelerated electrons in an SEM carry significant amounts of kinetic energy, and this energy is dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample. These signals include secondary electrons, backscattered electrons (BSE), diffracted backscattered electrons (EBSD that are used to determine crystal structures and orientations of minerals), photons (characteristic X-rays that are used for elemental analysis and continuum X-rays), visible light (cathodoluminescence-CL), and heat. Secondary electrons and backscattered electrons are commonly used for imaging samples: secondary electrons are most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples (i.e. for rapid phase discrimination). X-ray generation is produced by inelastic collisions of the incident electrons with electrons in discrete orbitals (shells) of atoms in the sample. As the excited electrons

return to lower energy states, they yield X-rays that are of a fixed wavelength (that is related to the difference in energy levels of electrons in different shells for a given element). Thus, characteristic X-rays are produced for each element in a mineral that is "excited" by the electron beam. SEM analysis shown in the Figure.5 is considered to be "nondestructive"; that is, x-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyze the same materials repeatedly. The Scanning electron microscope is as shown in the Figure 5

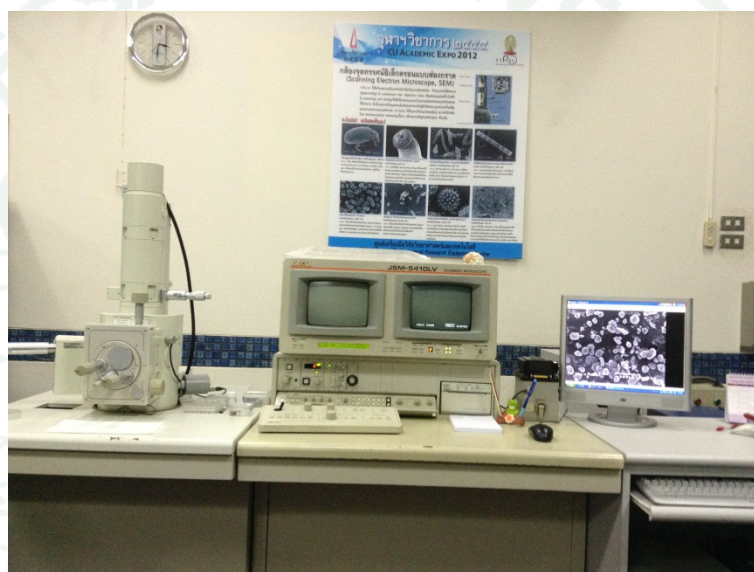


Figure 5 Scanning electron microscope

4.2 X-Ray Diffraction (XRD)

X-ray diffraction relies on the dual wave/particle nature of X-rays to obtain information about the structure of crystalline materials. A primary use of the technique is the identification and characterization of compounds based on their diffraction pattern. The dominant effect that occurs when an incident beam of monochromatic X-rays interacts with a target material is scattering of those X-rays from atoms within the target material. In materials with regular structure (i.e. crystalline), the scattered X-rays undergo constructive and destructive interference. This is the process of diffraction. The diffraction of X-rays by crystals is described by Bragg's Law, $n\lambda = 2d \sin \theta$. The directions of possible diffractions depend on the size and shape of the unit

cell of the material. The intensities of the diffracted waves depend on the kind and arrangement of atoms in the crystal structure. However, most materials are not single crystals, but are composed of many tiny crystallites in all possible orientations called a polycrystalline aggregate or powder. When a powder with randomly oriented crystallites is placed in an X-ray beam, the beam will see all possible interatomic planes. If the experimental angle is systematically changed, all possible diffraction peaks from the powder will be detected. The X-Ray Diffractometer is as shown in the Figure 6.



Figure 6 X-Ray Diffractometer

4.3 Mercury Intrusion Porosimetry (MIP)

Mercury Intrusion Porosimeter as shown in the Figure.7 uses mercury intrusion or intrusion of any other non-wetting liquid to determine pore volume. The Mercury Intrusion Porosimeter fills the penetrometer and sample chamber with mercury under vacuum and takes a volume reading. The sample, however, is not initially intruded with mercury or other non-wetting test liquid because of the high surface tension. Gradually, increasing amounts of pressure are applied on the non-

wetting liquid. For each incremental increase in pressure, the change in intrusion volume is equal to the volume of the pores whose diameters fall within an interval that corresponds to the particular pressure interval.



Figure 7 Pore size distribution MIP tests

4.4 Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

Inductively coupled plasma atomic emission spectroscopy (ICP-AES), also referred to as inductively coupled plasma optical emission spectrometry (ICP-OES), is an analytical technique used for the detection of trace metals. It is a type of emission spectroscopy that uses the inductively coupled plasma to produce excited atoms and ions that emit electromagnetic radiation at wavelengths characteristic of a particular element. The intensity of this emission is indicative of the concentration of the element within the sample.

RESULTS AND DISCUSSION

1. Properties of fly ash

Chemical composition analysis in the form of metal oxide of fly ash using X-ray fluorescence spectrometer showed that the main component of fly ash obtained from Mae Moh, Lampang had 35.3% silica oxide, 20.6% alumina oxide, 14.6% ferric oxide, and 18.17% calcium oxide, while fly ash obtained from Indonesia had 62.9% silica oxide, 25.18% alumina oxide, 3.25% ferric oxide, and 0.85% calcium oxide. The classification of fly ash according to ASTM 618 found that fly ash from Mae Moh Lampang used in the test was in class C because it had more than 50% of total silica, alumina, and ferric oxides and more than 10% of calcium oxide and fly ash from Indonesia was in class F since the total silica, alumina, and ferric oxides is over 70% shown in Table 5

Table 5 The composition of fly ash by X-Ray fluorescence spectrometer

Fly ash	Composition (%)								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	MnO	LOI
Class C	35.3	20.6	14.06	18.17	2.03	35.3	1.09	0.12	0.34
Class F	62.9	25.18	3.25	0.85	0.64	62.9	0.35	0.03	3.4

2. Flow

Flow test were performed on 60, 65 and 70 % class C fly ash with 2, 4 and 7% calcium hydroxide and on 50, 55 and 60 % class F fly ash with 10, 17 and 22.5% calcium hydroxide.

The result showed that class C fly ash mixes yielded the flow between 107.9 to 152.5 percent while the value for class F mixes were 127.8 to 152.5 percent as shown in Table.6

Table 6 Flow of geopolymer paste

Mixed	Designation	% Flow
1	C 60-0	152.5
2	C 60-2	152.5
3	C 60-4	152.5
4	C 60-7	107.9
5	C 65-0	152.5
6	C 65-2	152.5
7	C 65-4	152.5
8	C 65-7	152.5
9	C 70-0	122.8
10	C 70-2	152.5
11	C 70-4	152.5
12	C 70-7	152.5
13	F50-0	152.5
14	F50-10	152.5
15	F50-17	152.5
16	F50-22.5	152.5
17	F55-0	152.5
18	F55-10	152.5
19	F55-17	152.5
20	F55-22.5	152.5
21	F60-0	127.7
22	F60-10	127.7
23	F60-17	152.5
24	F60-22.5	152.5

3. Setting time

The initial and final setting time of geopolymer paste decreased as the fly ash percentage increased. Geopolymer paste obtained from class C fly ash had initial

setting time decreased from 165 to 92 minute, 52.5 to 12 minute, and 25 to 1.5 minute, respectively, and the final setting time reduced from 340 to 210 minute, 180 to 90 minute, and 60 to 30 minute, respectively, as shown in Figure 8.

Geopolymer paste obtained from class F fly ash did not set at room temperature or at 70 °C. Setting time was observed only after adding 10, 17 and 22.5% calcium hydroxide. Fly ash was found to decrease setting time. The initial setting time decreased from 30 to 11.5 min, 50 to 6 min, and 120 to 36 min, respectively, and the final setting time reduced from 150 to 105 min, 180 to 90 min, and 225 to 50 min, respectively, as shown in Figure 9.

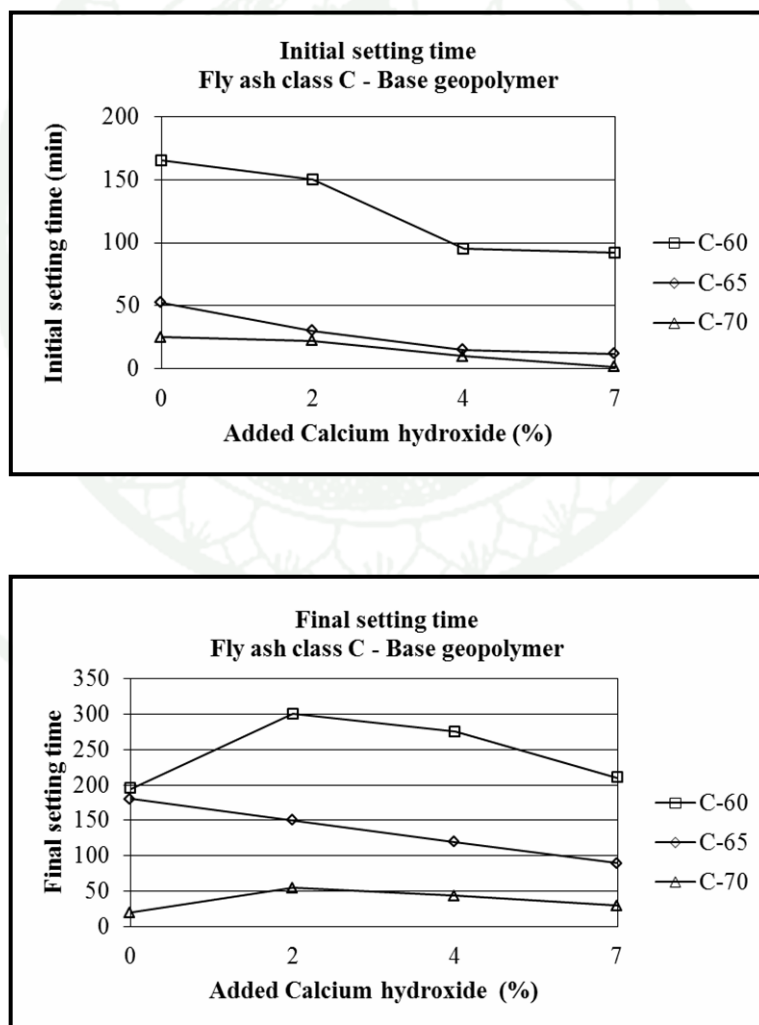


Figure 8 Initial and final setting time Fly ash class C - Based geopolymers

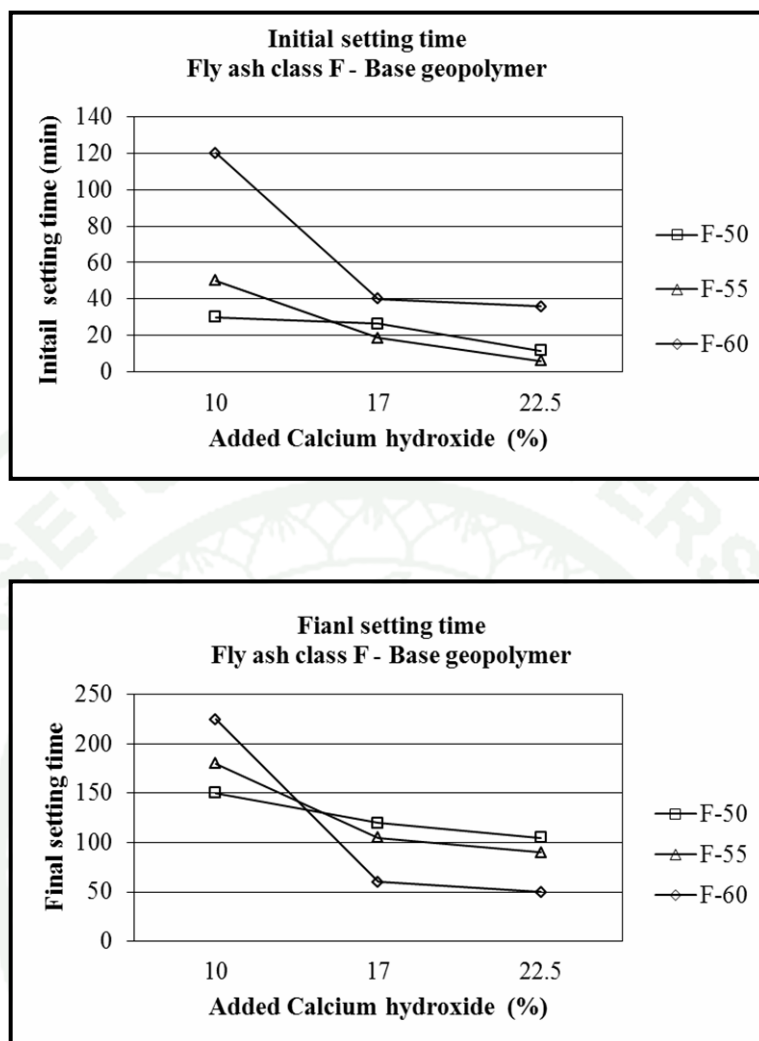


Figure 9 Initial and final setting time Fly ash class F - Based geopolymers

4. Compressive strength

For samples cured at room temperature for 7 days, addition of calcium hydroxide for 7 % w/w in class C fly ash to ratio of solid at 60, 65 and 70 % w/w had maximum compressive strength of 189.77, 187.61 and 179.54 ksc, respectively as shown in the Figure10, which were consistent with the study by Temujin (2003) research by adding 1.3, 2.6 and 3.9% of calcium hydroxide to fly ash using 14 M sodium hydroxide as a catalyst and curing at room temperature and at 70 °C.

Addition of suitable amount of calcium hydroxide resulted in better strength. As for curing at 70 °C for 1 day, addition of calcium hydroxide for 7% w/w in class C fly ash to ratio of solid at 60, 65, and 70 % w/w had maximum compressive strength of 155.05, 201.81 and 198.81 ksc respectively. is as shown in the Figure.10 Compare curing at room temperature and 70 °C, curing at 24 °C of 60% solid ratio the compressive strength was higher than curing 70 C, but for 65% and 70% solid ratio curing at 70 C had higher compressive strength as shown in the Figure 10

Curing at 70 °C, addition of calcium hydroxide at 2% w/w in class C fly ash to the solid ratio of 60 and 65% w/w and addition of calcium hydroxide at 7% w/w in class C fly ash to the solid ratio of 70% w/w resulted in maximum compressive strength of 161.01, 201.81 and 198.56 ksc and had higher compressive strength compared to the control without addition of calcium hydroxide, which was consistent with the study by Weiping and Brown (1997). Addition of higher amount of calcium hydroxide and curing at higher temperature had positive effect on compressive strength and for class F fly ash addition of 17% w/w calcium hydroxide in fly ash with solid ratio of 50, 55 and 60% w/w resulted in maximum compressive strength of 111.73, 184.12 and 189.71 ksc, respectively is as shown in the Figure 11

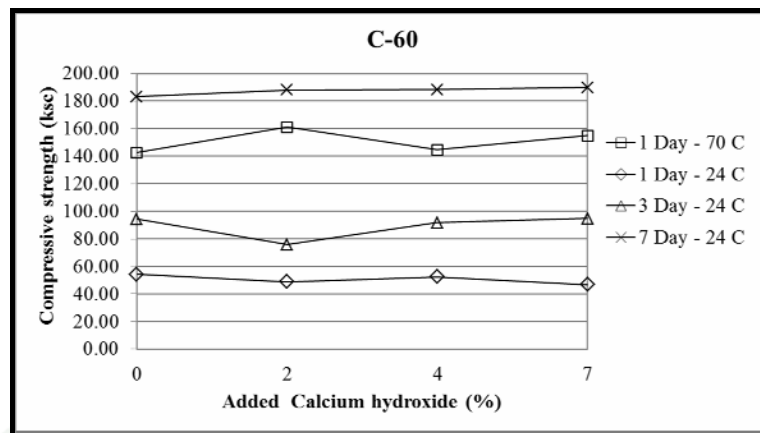
The trend of the impact of temperature on compressive strength of geopolymers paste as shown in the graph indicated that compressive strength at curing temperature of 70 °C for 1 day is similar to that of curing at 24 °C for 7 days of class C fly ash with 7% calcium hydroxide addition and class F fly ash with 17% calcium hydroxide addition because the increase in curing temperature helped accelerate hydration which will cause geopolymers paste to have high compressive strength in its initial lifetime. Curing at high temperature is a crucial factor for compressive strength of geopolymers curing by using temperature which will take some time, approximately 24-48 hours (P. Chindaprasirt, 2006).

For control geopolymers paste produced from class F fly ash without the addition of calcium hydroxide, it would not set for both curing at room temperature and at 70 °C because the main component of fly ash had low calcium oxide value

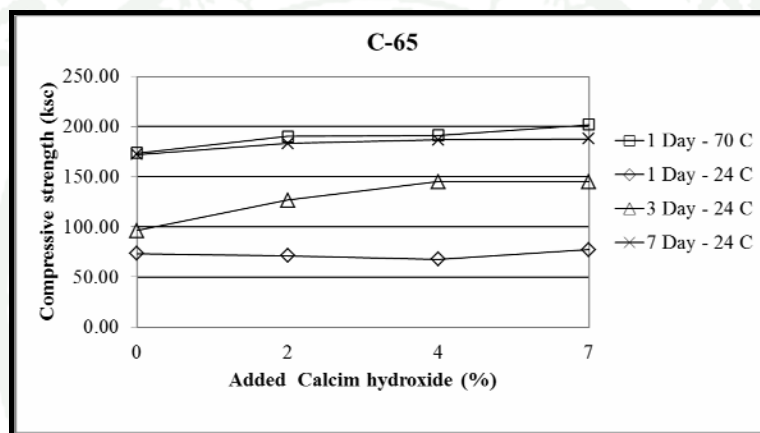
causing it to set slowly. The addition of calcium hydroxide in class F fly ash at suitable ratio had effect on the setting and could withstand compressive strength as a result of the curing time. According to the graph that show the trend of curing time, compressive strength increased quite fast as the curing time increased and the results of compressive strength test were not much different for class C fly ash with 60 and 65 percentage of fly ash and class F fly ash with 50 percentage of fly ash.

Effect of the addition of calcium hydroxide in class C fly ash, the development of compressive strength of fly ash that has been added with calcium hydroxide for 7 days caused hydration and pozzolanic reaction as evidenced by the compressive strength mixed with calcium hydroxide with solid ratio of 60 and 65 percentage that had compressive strength more than the control. For the control of class F fly ash without the addition of calcium hydroxide, it did not set so the compressive strength could not be tested. Considering the solid to liquid ratio for Class C fly ash, when cured at room temperature for 7 day higher solid to liquid ratio resulted in higher compressive strength than lower solid to liquid ratio, while when cured at 70 °C lower solid to liquid ratio resulted in higher compressive strength than higher solid to liquid ratio. For Class F fly ash, when cured at room temperature for 7 days and at 70 °C higher solid to liquid ratio had lower compressive strength than lower solid to liquid ratio.

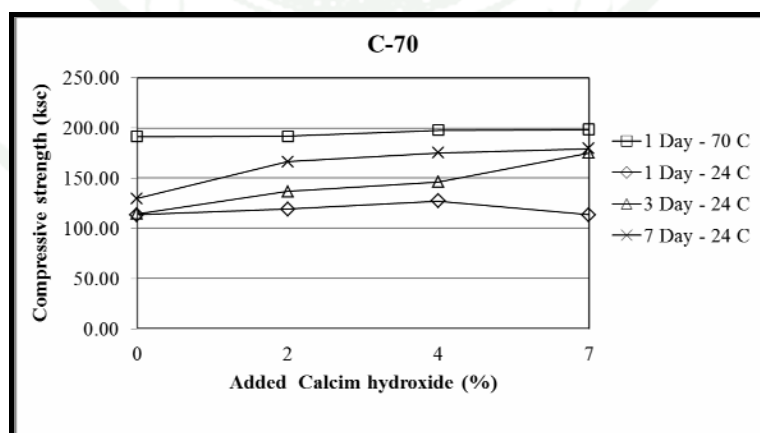
Effect of the amount of fly ash replaced by calcium hydroxide, the compressive strength of fly ash mixed with calcium hydroxide had the maximum compressive strength for class C fly ash with 2-4 percentage of calcium hydroxide and 15-18 percentage for class F fly ash. In other words, the amount of calcium hydroxide in percent by weight added to the fly ash was sufficient for complete pozzolanic reaction as shown in the Figure15. The addition of too much calcium hydroxide would result in a leftover and there was excess calcium hydroxide in the geopolymer causing incomplete agglomeration and lower compressive strength.



(a) Fly ash based geopolymer 60 % fly ash

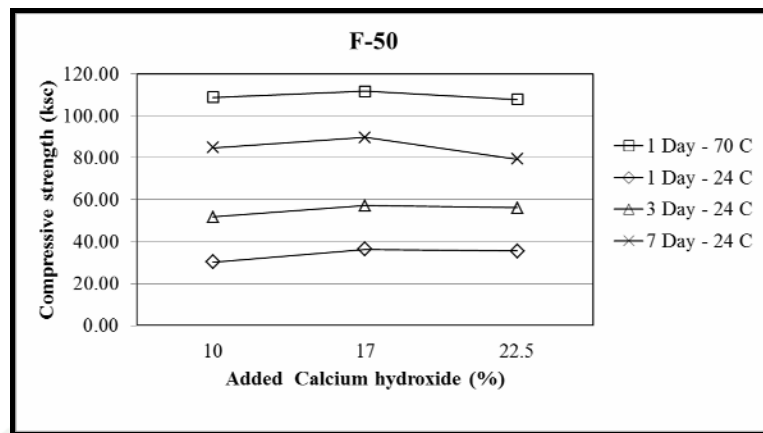


(b) Fly ash based geopolymer 65 % fly ash

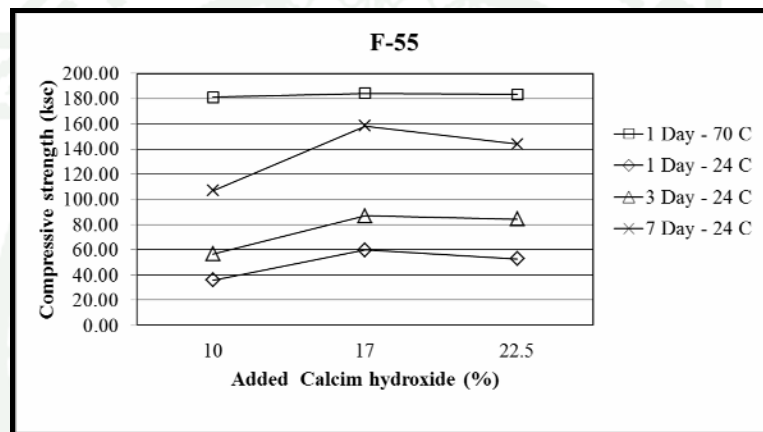


(c) Fly ash based geopolymer 70 % fly ash

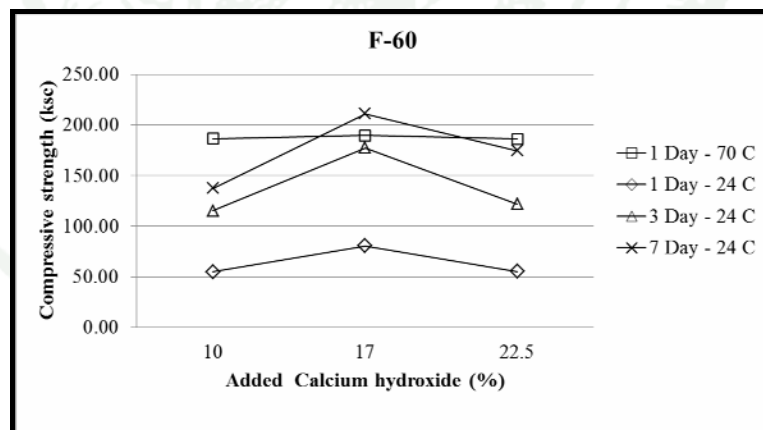
Figure 10 The compressive strength development of geopolymer (Class C)



(a) Fly ash based geopolymer 50 % fly ash



(b) Fly ash based geopolymer 55 % fly ash



(c) Fly ash based geopolymer 60 % fly ash

Figure 11 The compressive strength development of geopolymer Class F

5. Dissolution

The study of silica, alumina, and calcium leaching of class C and F fly ash was performed using 9 and 12 M Sodium hydroxide solution for 3, 10, 20 and 30 minute to determine the amount of silica, alumina, and calcium leached at different time and to find suitable time and amount for producing geopolymer materials from fly ash. This research determined the amount of silica, alumina, and calcium using inductively coupled plasma optical emission spectrometry (ICP-OES), which is an accepted technique for determination of concentration of elements in solution.

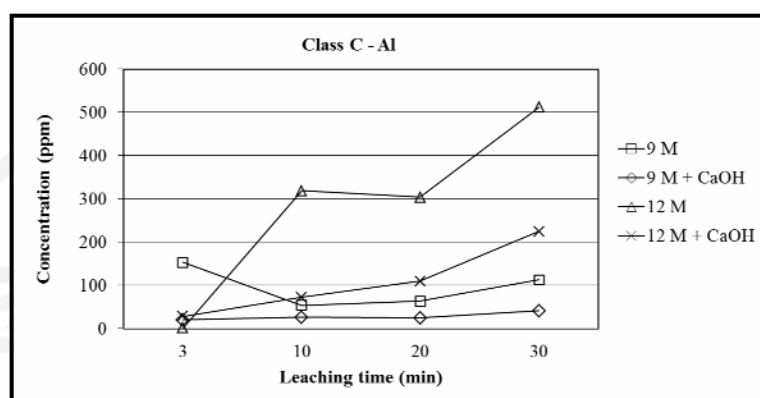
The experiment to determine the amount of alumina calcium and silica leached from fly ash by 9 and 12 M sodium hydroxide solution found that for class C and F fly ash 12M sodium hydroxide solution could cause alumina leaching more than these produced by 9 molar sodium hydroxide solution. Except for calcium leaching of class F.

The addition of calcium hydroxide did not have any effect on leaching. The addition of calcium hydroxide caused higher calcium leaching rate when compared to samples that calcium hydroxide were not added. However, it cannot be determined whether the addition of calcium hydroxide caused higher calcium leaching in fly ash for both class C and F fly ash.

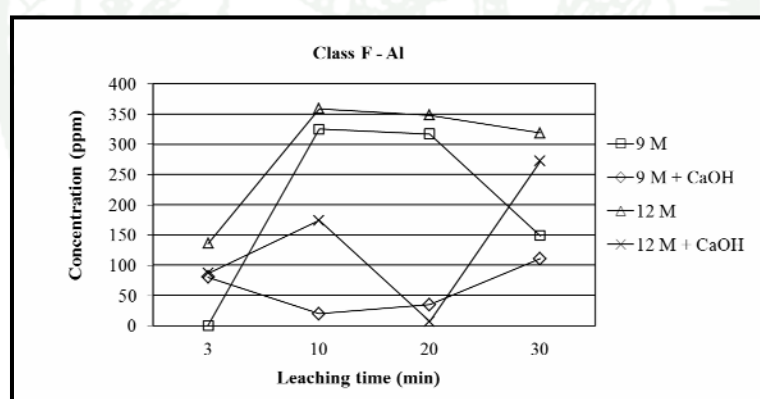
The optimum duration for alumina, calcium, and silica leaching in class C and F fly ash was relatively spread and diverse. When considering the results from graph, it can be concluded that there were two periods which were at 3 minute that compound leaching was quite high and gradually decreased and at 20-30 min, indicating continuous reaction as shown in Figure 12, 13, and 14.

Silica leaching by 9 and 12 molar Sodium hydroxide solution for both class C and F, it was found that 12 molar sodium hydroxide solution could cause more silica leaching. Compare leaching by sodium hydroxide solution at the same concentration between fly ash with and without the addition of calcium hydroxide, the sample

without calcium hydroxide had higher silica leaching because sample with calcium hydroxide, calcium hydroxide would replace fly ash reducing the amount of fly ash and it showed that addition of calcium hydroxide did not have any effect on calcium leaching, while the suitable time was 3 minute.

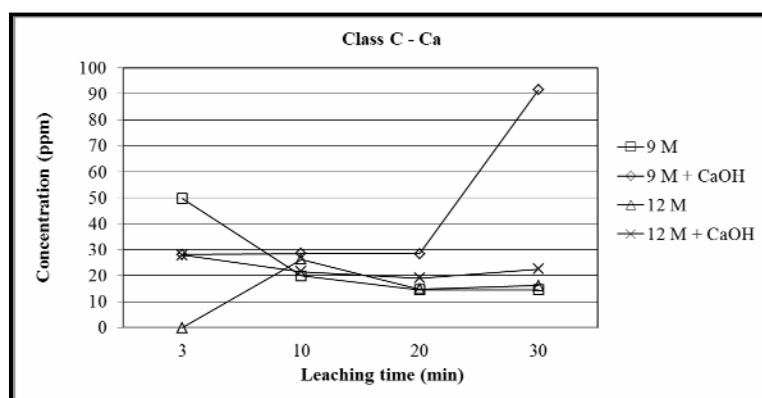


(a)

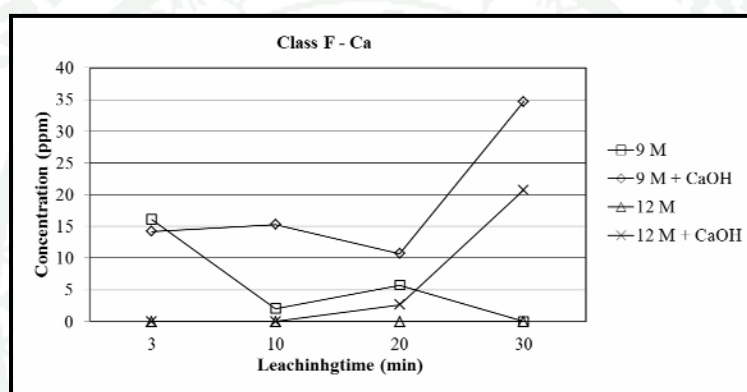


(b)

Figure 12 The alumina ion dissolution from fly ash

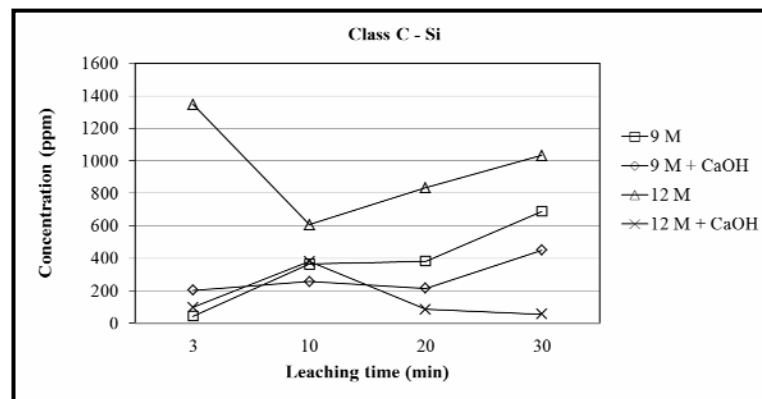


(a)

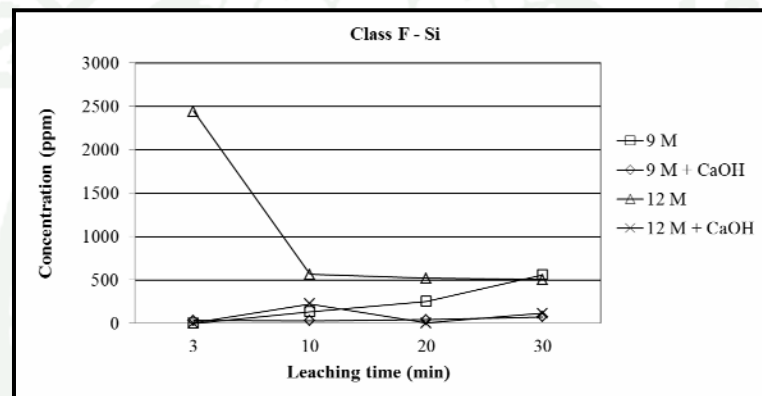


(b)

Figure 13 The calcium ion dissolution from fly ash



(a)



(b)

Figure 14 The silica ion dissolution from fly ash

6. Microstructure

6.1 Scanning electron microscopy (SEM)

Scanning electron microscopy images showed that for geopolymer paste from class C fly ash cured at room temperature, when calcium hydroxide was added the paste was more compressed and had less pores and the amount of fly ash remained from the reaction was less, as shown in Figure 15a and 15b. For curing at 70 °C, the surface of geopolymer paste was smooth and nearly uniform, which was because of a near completion of geopolymerization reaction. In addition, it was found that all

unreacted fly ash, because of insufficient solution or mixing, was combined into geopolymer. In addition, it was found that acceleration of the reaction by curing at 70 °C would cause silica and alumina leaching from the surface of fly ash in basic condition causing bonds of H₂O to break and bind with silicon and aluminium ion becoming and producing gel of aluminosilicate on the surface of fly ash increasing binding efficiency as shown in Figure 16a and 16 b. As a result, geopolymer paste from fly ash is denser and less porous and improves mechanical properties of the fly ash based geopolymers. This was consistent with the total porosity. Curing at room temperature and at 70 °C and the addition of calcium hydroxide caused lower total porosity.

Paste obtained from class C and F fly ash as shown in Figure 17a and 17 b shows residues of unreacted fly ash in the matrix of geopolymer. But when calcium hydroxide was added the reaction was better and the structure of geopolymer became denser for both curing at room temperature and at 70 °C, illustrating that it was due to higher amount of calcium hydroxide. The addition of calcium hydroxide helped producing calcium silicate hydrate and calcium aluminosilicate hydrate improving the compressive strength of geopolymer fly ash.

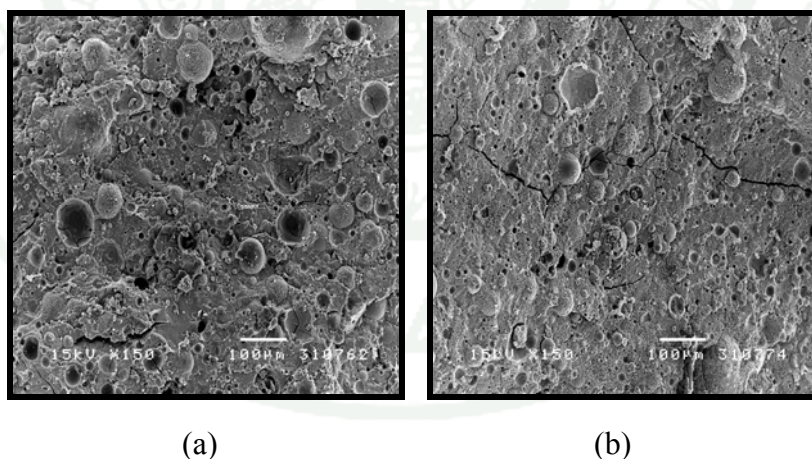


Figure 15 SEM micrograph of class C fly ash based geopolymer curing 24 °C no added calcium hydroxide (a) and added calcium hydroxide (b)

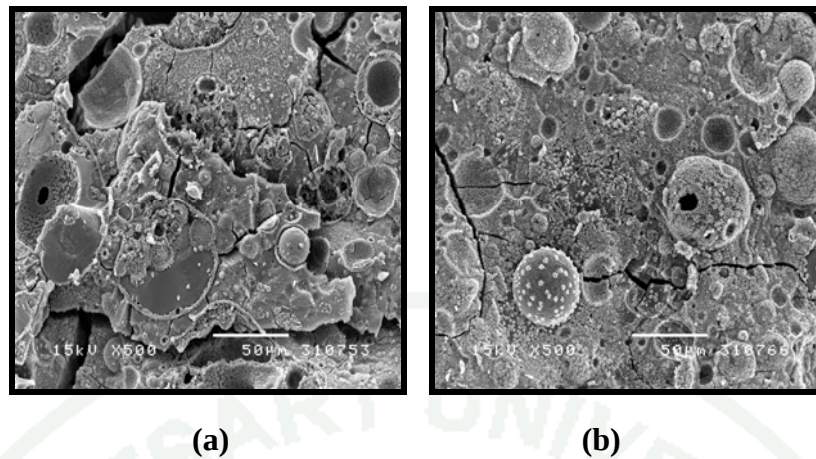


Figure 16 SEM micrograph of class C fly ash based geopolymer curing 70 °C no added calcium hydroxide (a) and added calcium hydroxide (b)

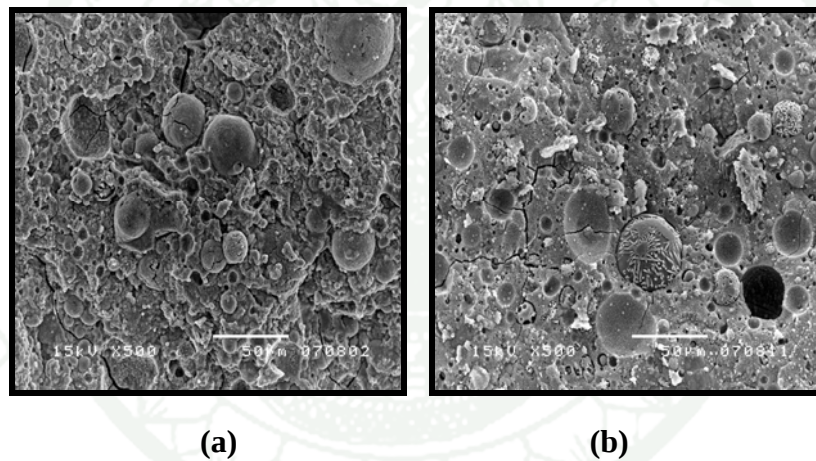


Figure 17 SEM micrograph of class F fly ash based geopolymer added calcium hydroxide 17 % curing 70 °C (a) and curing 24 °C (b)

6.2 Pore size distribution

In this study some mixtures of fly ash-based geopolymer were tested for porosity. For class C fly ash, the study also determined the effect of calcium hydroxide after curing at room temperature for 7 days and at 70 °C for 1 day. For class F fly ash, because calcium hydroxide was not added, the samples did not set for both curing at room temperature for 7 days and curing at 70 °C for 1 day. Therefore, a

comparison was made between fly ash cured at room temperature for 7 days and curing at 70 °C for 1 day with solid to liquid ratio of 1.00 and 0.67 as shown in Table.7

For class C fly ash cured at room temperature, the addition of calcium hydroxide caused an increase in average pore size but the total porosity decreased. For curing at 70 °C for 1 day, average pore size decreased when calcium hydroxide was added while total porosity decreased as well. When compare with curing at 70 °C of Temujin, the results were consistent in which when the addition of calcium hydroxide would cause a decrease in total porosity. Considering compressive strength, the addition of calcium hydroxide caused higher compressive strength for curing at both 24 and 70 °C and when the relationship between compressive strength and total porosity was considered, the total porosity increased as the compressive strength increased. On the other hand, when the compressive strength decreased, total porosity decreased, which was consistent with the study by Temujin. As has been mentioned, for the compressive strength it was found that curing at room temperature showed lower total porosity percentage while curing at room temperature caused a decrease in total porosity percentage, which was contradicting.

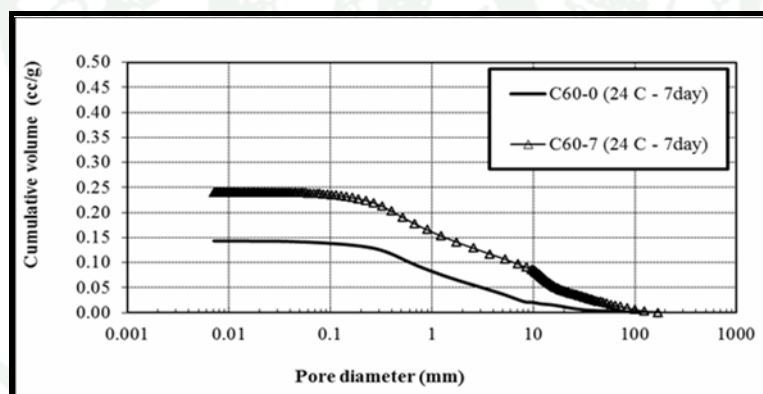
The cumulative volume for curing at 24 °C was 0.1434 cc/g and when calcium hydroxide was added the cumulative volume increased to 0.2390 cc/g. For curing at 70 °C, the cumulative porosity decreased when calcium hydroxide was added. For instance, the sample without the addition of calcium hydroxide had cumulative volume of 0.1593 cc/g but when calcium hydroxide was added cumulative volume decreased to 0.0566 cc/g as shown in the figure 18 and 19.

For class F fly ash, being low calcium fly ash, sample without the addition of calcium hydroxide, which was the control, did not set for both curing at 24 and 70 °C. There fore, this research compared the ratio of solid to liquid at 1.00 and 0.67 and found that for curing at 24 and 70 °C when the solid to liquid ratio decreased the average pore diameter increased while the total porosity decreased for curing at room temperature but increased for curing at 70 °C. When the relationship between

compressive strength and total porosity was considered, at curing temperature of 24 °C the compressive strength increased as the total porosity decreased, which was consistent with the experiments by Temujin, but at 70 °C curing temperature, the compressive strength decreased as the total porosity decreased shown in the Table.7

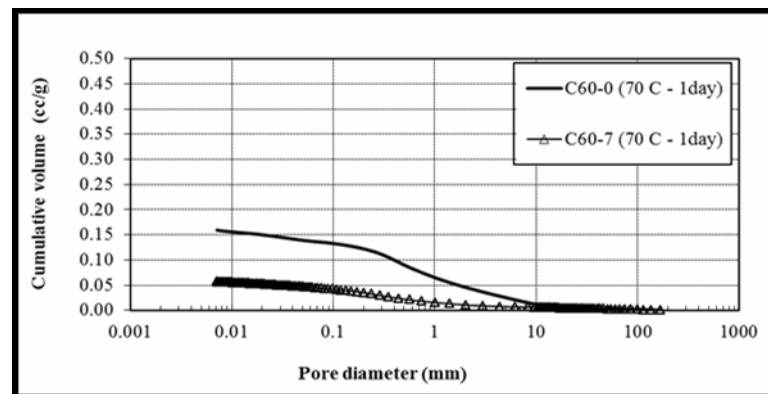
Table 7 Total porosity and pore size distribution of geopolymer paste

Mixed	Designation	Curing 24 °C			Curing 70 °C		
		Total	Cumulative	Average	Total	Cumulative	Average
		Porosity	Volume	Pore	Porosity	Volume	Pore
		(%)	(cc/g)	Diameter	(%)	(cc/g)	Diameter
1	C 60-0	23.83	0.143	1.33	21.97	0.159	0.61
4	C 60-7	21.73	0.239	12.13	10.6	0.056	0.2
15	F50-17	12.1	0.214	21.93	14.62	0.212	0.2
23	F60-17	8.28	0.051	145.8	22.08	0.107	0.76

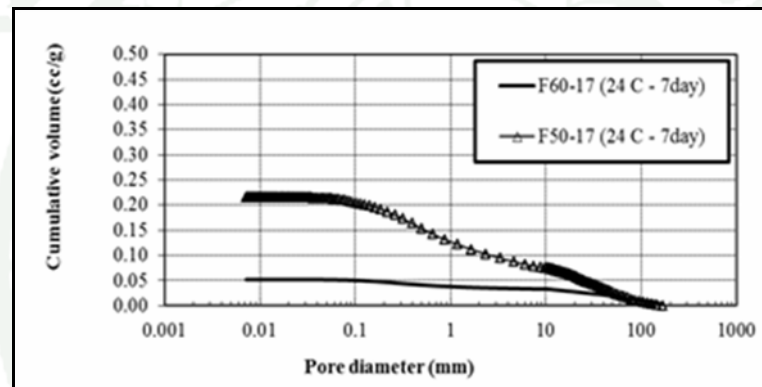


(a) Geopolymer MIP test results added fly ash class C 0 and 7 % Ca(OH)_2 and curing 24 °C

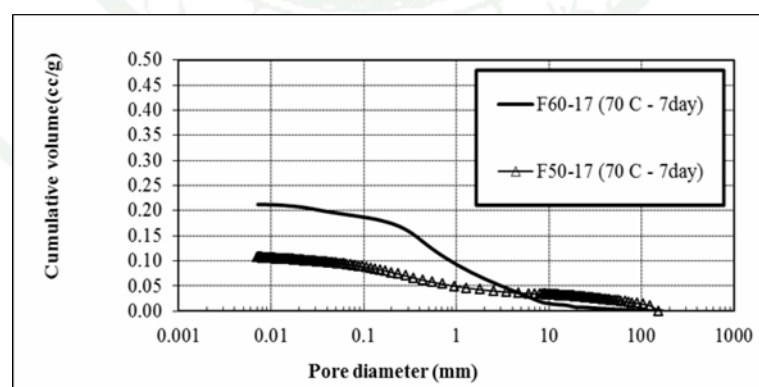
Figure 18 Cumulative volume of intrusion vs pore diameter in the MIP tests for the fly ash based geopolymer



(b) Geopolymer MIP test results added fly ash class C 0 and 7 % Ca(OH)_2 and curing 70 °C

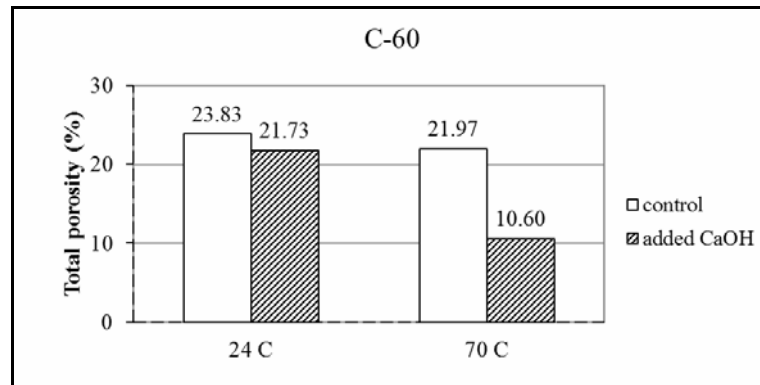


(c) Geopolymer MIP test results added fly ash class F 17 % Ca(OH)_2 and curing 24 °C

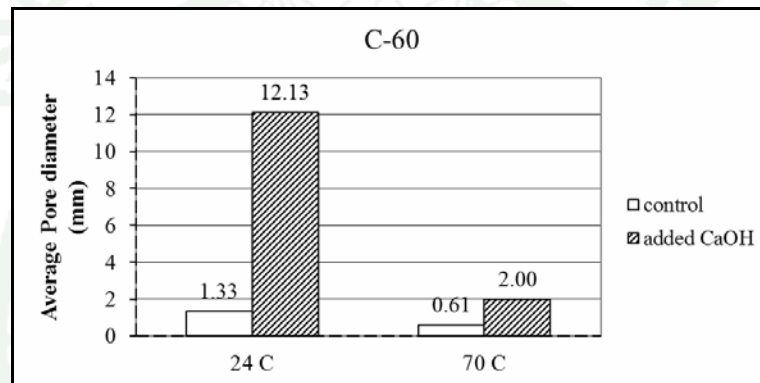


(d) Geopolymer MIP test results added fly ash class F 17 % Ca(OH)_2 and curing 70 °C

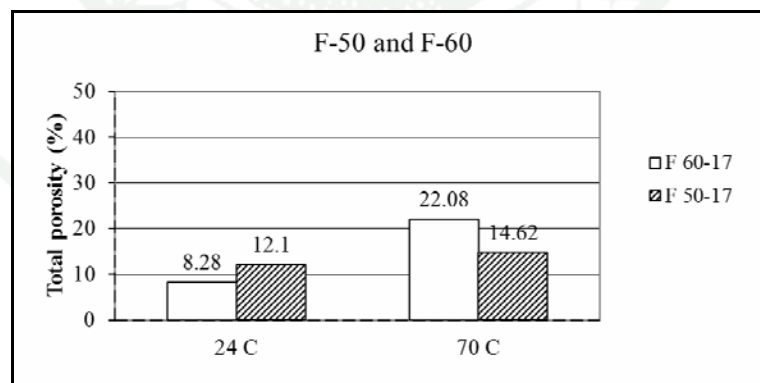
Figure 18 (Continued)



(a) The development of total porosity (%) fly ash based geopolymer class C

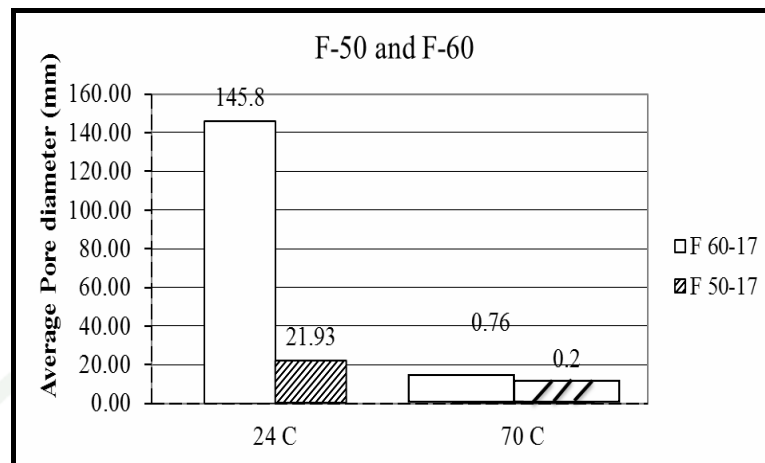


(b) The development of average pore diameter (um) fly ash based geopolymer class C



(c) The development of total porosity (%) fly ash based geopolymer class F

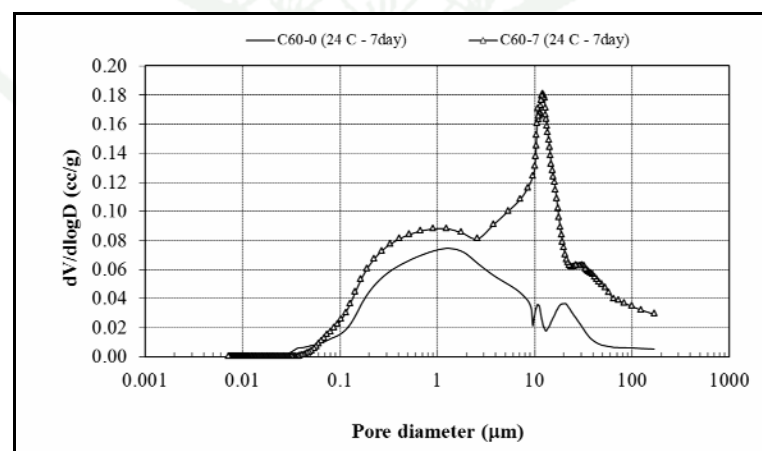
Figure 19 Total porosity and average pore diameter in the MIP tests for the fly ash based geopolymer class C and F



(d) The development of average pore diameter (um) fly ash based geopolymer class F

Figure 19 (Continued)

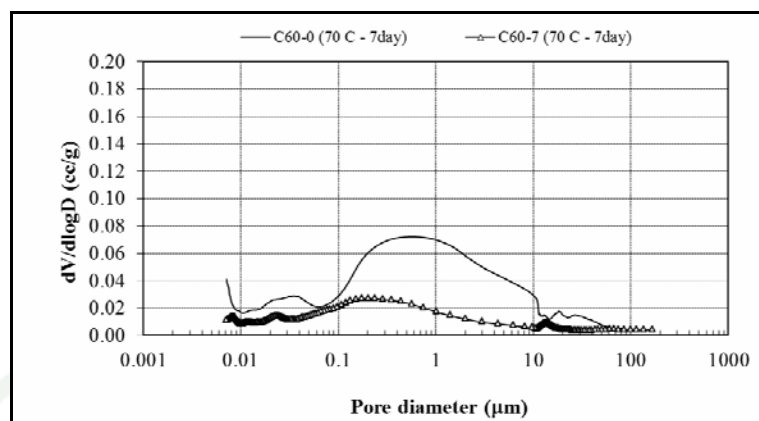
Pore volume and pore size of paste. When comparing Class C fly ash sample that was cured at 70 °C, the addition of calcium hydroxide caused lower amount of pores and when comparing to curing at 24 °C it was found that the graph shifted to the left indicating that the pores were significantly smaller as shown in Figure.20 (a) and (b) consistent with the results of the compressive strength test in which the addition of calcium hydroxide resulted in higher compressive strength and consistent with the results of geopolymer structure which was denser.



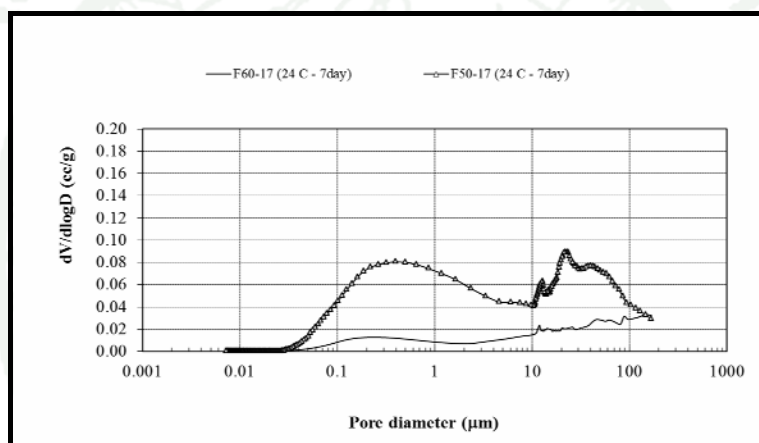
(a)

Figure 20 Pore size distribution in the MIP tests for the fly ash based geopolymer

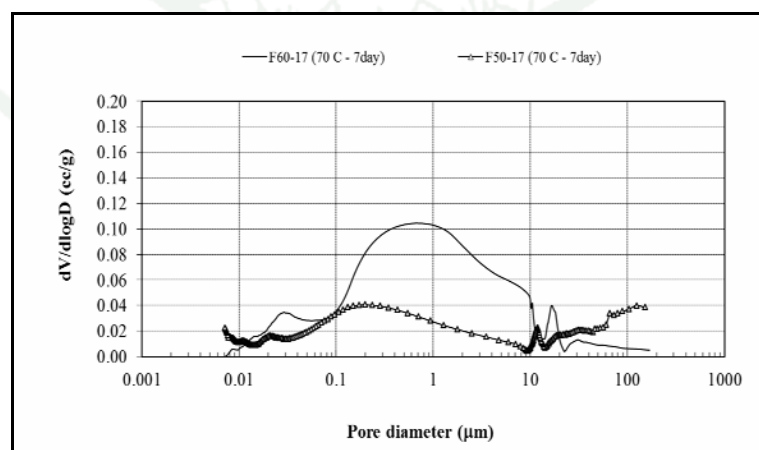
class C and F



(b)



(c)

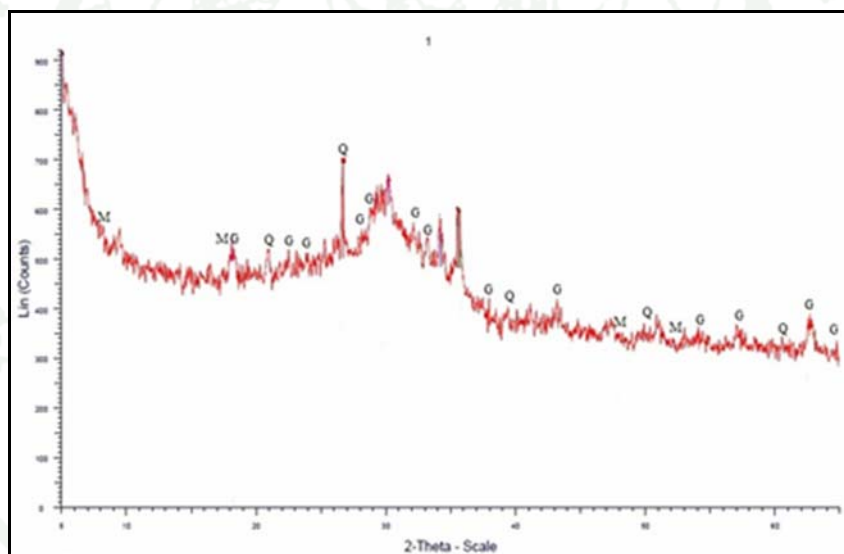


(d)

Figure 20 (Continued)

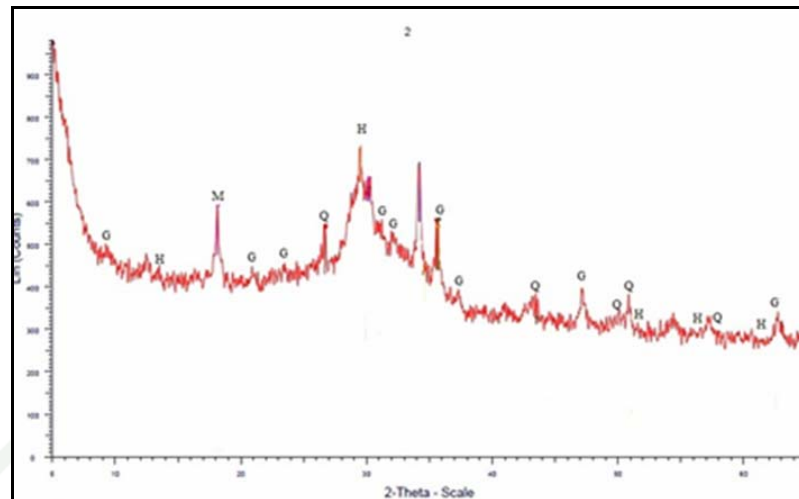
6.3 X –ray Diffraction Spectroscopy (XRD)

The study of calcium hydroxide addition in class C and F fly ash of mixtures of geopolymer was done by choosing the mixtures with the highest compressive strength to test by adding sodium silicate solution and 12 molar sodium hydroxide solution with a reaction time of 5 minute. The obtained crystal of geopolymer mixed with calcium hydroxide studied by X –ray Diffraction Spectroscopy showed that class C fly ash without calcium hydroxide had quartz, iron silicate, iron silicon oxide, minamiite, margarite, and hedenbergite. When compared with samples that calcium hydroxide was added, the samples with calcium hydroxide had an increase of hedenbergite shown in the Figure.21 a and b.



(a)

Figure 21 X-ray diffractograms of fly ash class C –base geopolymer at 7 days and 24 °C : Q = Quartz, M = Minamillte, G = Margarite, H = Hedenbergite

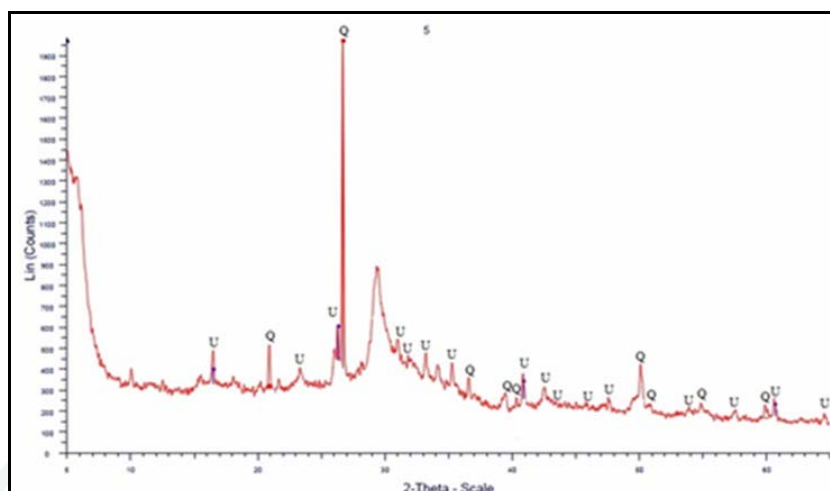


(b)

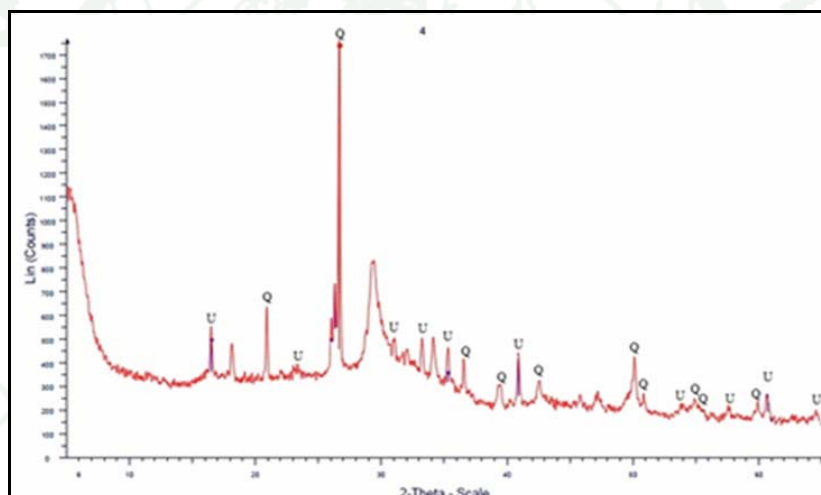
Figure 21 (Continued)

For class F fly ash, because the samples were not added with calcium hydroxide, the samples did not set. The crystal study of geopolymer paste obtained only from fly ash mixed with calcium hydroxide samples. For 50 and 60 percent by weight, the addition of all ratio of calcium hydroxide at 17% by weight showed quartz and mullite compounds as the two major components as shown in Figure 15. Mineralogy analysis found that for percentage by weight, the higher the amount of solid or the lower solid to liquid ratio caused higher crystallinity as illustrated by taller graph and at 2 theta of 25-30 degree is the range that shows amorphous geopolymer Figure.22 a and b.

The addition of calcium hydroxide caused geopolymer to become more amorphous and produced calcium silicate hydrate (CSH) improving the compressive strength which was consistent with compressive strength test in which the addition of calcium hydroxide resulted in higher compressive strength.



(a)



(b)

Figure 22 X-ray diffractograms of fly ash class F –base geopolymer at 7 days and 24 Celsius degree: Q = Quartz, U = Mullite

CONCLUSION

1. Class F fly ash produced paste with higher flow than the one with class C fly ash.
2. Calcium hydroxide helped decreased the paste setting time for both classes of fly ash. At the same fly ash percentage of 60, class C yielded paste with faster setting time.
3. Paste with class F fly ash had long setting time. Addition of calcium helped decrease the setting time and increased strength development.
4. Calcium hydroxide helped increased strength development of geopolymer. Accelerated curing yielded compressive strength comparable to 7 days of curing for paste from class C fly ash. The accelerated strength was even higher than the 7 days strength with class F fly ash was used.
5. Highest strength was achieved when 7% calcium hydroxide was added to class C fly ash paste. The optimum percentage was 17 for class F fly ash.
6. Addition of calcium hydroxide did not significantly affect leachate of silica and alumina from the paste.
7. The silica and alumina and calcium ions leached out were found to be maximum at 10 minutes leaching time. After that the concentrations decreased.
8. SEM study showed calcium hydroxide yielded paste with denser structure, in line with the increase in strength and reduction in porosity.
9. Addition of calcium hydroxide decreased the total porosity of the paste. This was also true for the accelerated curing, when compared with normal curing.

10. XRD study indicated increase in the degree of crystallinity in class C fly ash paste. For paste with class F fly ash, the reverse was found, ie, addition of calcium hydroxide increased the amorphicity of the paste and increased the paste strength.

11. The results of flow percentage compared between class C and class F fly ash showed similar values. Comparison of setting time between class C and class F fly ash at the same ratio of solid mixture showed that class F fly ash had better setting time than class C fly ash. Class C fly ash based geopolymer cured at 24°C had better compressive strength than class F fly ash based geopolymer. However, when cured at 70 °C with the same ratio of solid mixture class F fly ash based geopolymer had better compressive strength, but when the ratio of solid mixture is higher, class C fly ash possessed better compressive strength. Microstructure was considered the addition of sodium hydroxide caused the paste to be denser and more homogeneous, and had lower total porosity clearly corresponding to higher compressive strength.

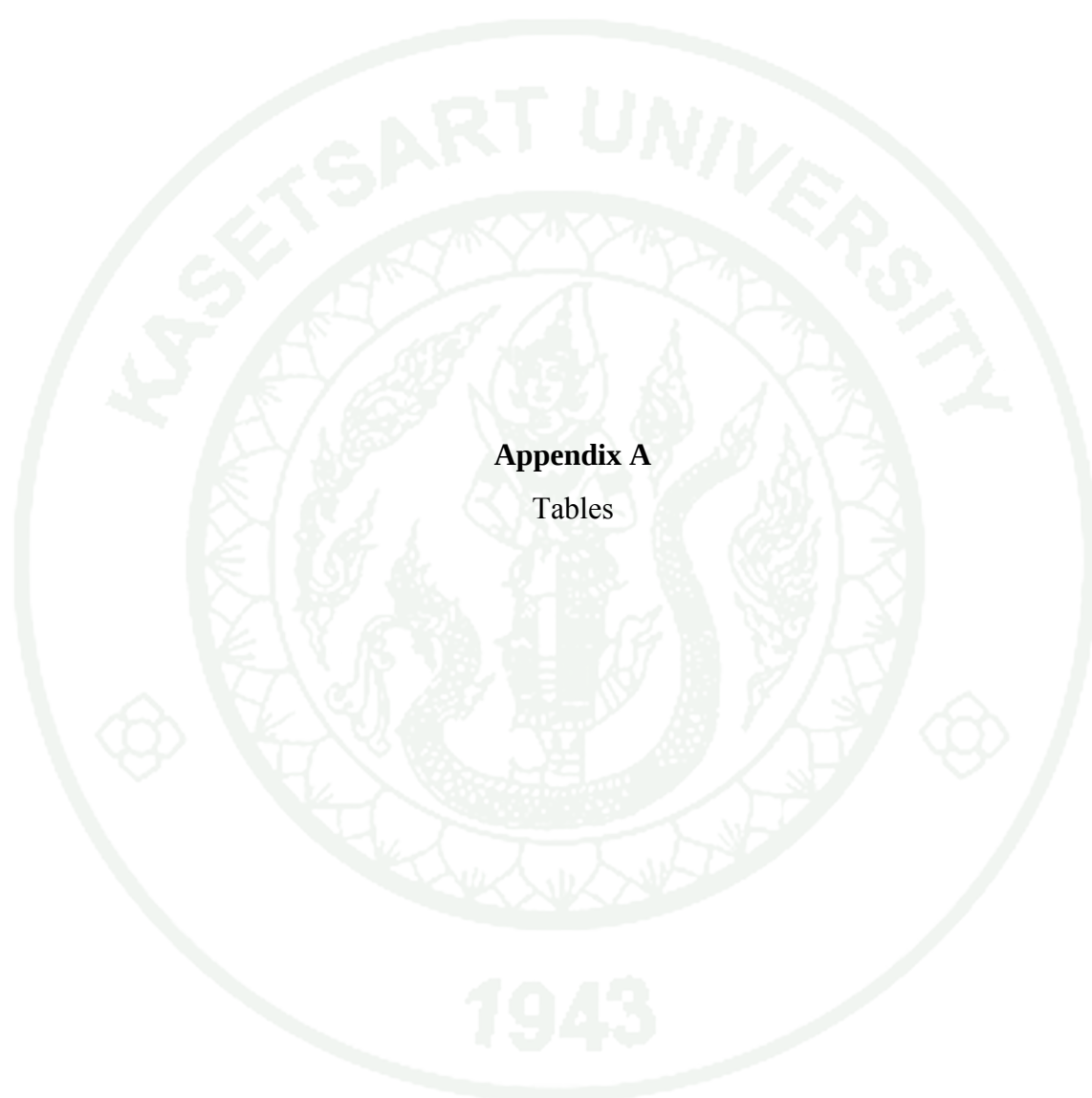
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APPENDICES



Appendix A
Tables

Appendix Table A1 Mix design of fly ash – based geopolymer

Mixed	Designation	Solid (% by weight)		Solution (% by weight)		Solution :FA Ratio
		Fly ash	Ca(OH) ₂	NaOH	Na ₂ SiO ₃	
1	C 60-0	60	0	20	20	0.67
2	C 60-2	58	2	20	20	0.67
3	C 60-4	56	4	20	20	0.67
4	C 60-7	53	7	20	20	0.67
5	C 65-0	65	0	17.5	17.5	0.54
6	C 65-2	63	2	17.5	17.5	0.54
7	C 65-4	61	4	17.5	17.5	0.54
8	C 65-7	58	7	17.5	17.5	0.54
9	C 70-0	70	0	15	15	0.43
10	C 70-2	68	2	15	15	0.43
11	C 70-4	66	4	15	15	0.43
12	C 70-7	63	7	15	15	0.43
13	F50-0	50	0	25	25	1.00
14	F50-10	40	10	25	25	1.00
15	F50-17	33	17	25	25	1.00
16	F50-22.5	27.5	22.5	25	25	1.00
17	F55-0	50	0	22.5	22.5	0.82
18	F55-10	40	10	22.5	22.5	0.82
19	F55-17	33	17	22.5	22.5	0.82
20	F55-22.5	27.5	22.5	22.5	22.5	0.82
21	F60-0	50	0	20	20	0.67
22	F60-10	40	10	20	20	0.67
23	F60-17	33	17	20	20	0.67
24	F60-22.5	27.5	22.5	20	20	0.67

Appendix Table A2 Result of Flow test

Mixed	Designation	D2	D1	% Flow
1	C 60-0	25.5	10.1	152.5
2	C 60-2	25.5	10.1	152.5
3	C 60-4	25.5	10.1	152.5
4	C 60-7	21.0	10.1	107.9
5	C 65-0	25.5	10.1	152.5
6	C 65-2	25.5	10.1	152.5
7	C 65-4	25.5	10.1	152.5
8	C 65-7	25.5	10.1	152.5
9	C 70-0	22.5	10.1	122.8
10	C 70-2	25.5	10.1	152.5
11	C 70-4	25.5	10.1	152.5
12	C 70-7	25.5	10.1	152.5
13	F50-0	25.5	10.1	152.5
14	F50-10	25.5	10.1	152.5
15	F50-17	25.5	10.1	152.5
16	F50-22.5	25.5	10.1	152.5
17	F55-0	25.5	10.1	152.5
18	F55-10	25.5	10.1	152.5
19	F55-17	25.5	10.1	152.5
20	F55-22.5	25.5	10.1	152.5
21	F60-0	23.0	10.1	127.7
22	F60-10	23.0	10.1	127.7
23	F60-17	25.5	10.1	152.5
24	F60-22.5	25.5	10.1	152.5

Appendix Table A3 Result of setting time

Mixed	Designation	Setting time (min)	
		Initial	Final
1	C 60-0	165	340
2	C 60-2	150	300
3	C 60-4	95	275
4	C 60-7	92	210
5	C 65-0	52.5	180
6	C 65-2	30	150
7	C 65-4	15	120
8	C 65-7	12	90
9	C 70-0	25	60
10	C 70-2	22	55
11	C 70-4	10	44
12	C 70-7	1.5	30
13	F50-0	Unsetting	Unsetting
14	F50-10	30	150
15	F50-17	26.5	120
16	F50-22.5	11.5	105
17	F55-0	Unsetting	Unsetting
18	F55-10	50	180
19	F55-17	18.5	105
20	F55-22.5	6	90
21	F60-0	Unsetting	Unsetting
22	F60-10	120	225
23	F60-17	40	60
24	F60-22.5	36	50

Appendix Table A4 Result of dissolution of Alumina ion

Mixed	Designation	NaOH (Conc)	Dissolution Alumina ion (ppm)			
			3 (min)	10 (min)	20 (min)	30 (min)
1	C 60-0	9M	153	54	63.7	113
		12M	1.7	319	304	512
4	C 60-7	9M	20.2	25.9	24.6	40.8
		12M	28.3	72.2	110	225
21	F60-0	9M	0.474	325	317	149
		12M	136	359	348	319
23	F60-17	9M	80.5	19.8	34.8	111
		12M	86.9	174	6.84	272

Appendix Table A5 Result of dissolution of Calcium ion

Mixed	Designation	NaOH (Conc)	Dissolution Calcium ion (ppm)			
			3 (min)	10 (min)	20 (min)	30 (min)
1	C 60-0	9M	49.7	19.9	14.5	14.6
		12M	<0.005	26.3	14.8	16.2
4	C 60-7	9M	28.1	28.5	28.4	91.6
		12M	27.9	21.5	19	22.5
21	F60-0	9M	16.1	2.02	5.74	<0.005
		12M	<0.005	<0.005	<0.005	<0.005
23	F60-17	9M	14.2	15.3	10.7	34.7
		12M	<0.005	<0.005	2.62	20.7

Appendix Table A6 Result of dissolution of Silica ion

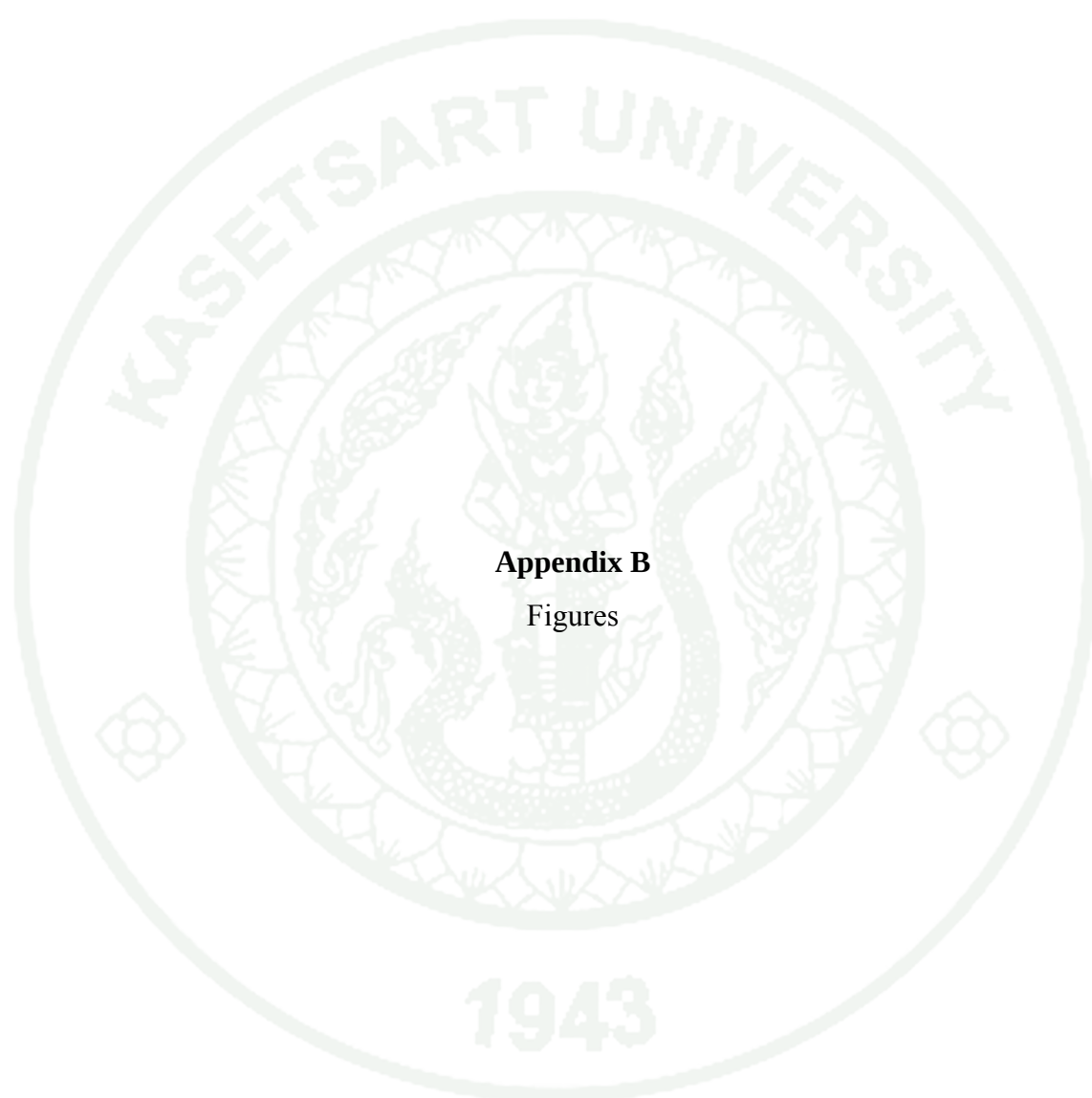
Mixed	Designation	NaOH (Conc)	Dissolution Silica ion (ppm)			
			3 (min)	10 (min)	20 (min)	30 (min)
1	C 60-0	9M	42.6	364	383	689
		12M	1347	607	834	1033
4	C 60-7	9M	202	257	214	449
		12M	97.1	381	84.9	54.8
21	F60-0	9M	0.736	135	252	560
		12M	2443	563	520	503
23	F60-17	9M	34.2	29.6	40.4	72.7
		12M	3.766	222	3.49	115

Appendix Table A7 Result of compressive strength testing

Mixed	Designation	Compressive strength (ksc)			
		1 day - 70 °C	1 day - 24 °C	3 day - 24°C	7 day - 24°C
1	C 60-0	142.60	54.15	94.40	183.21
2	C 60-2	161.01	48.74	75.81	188.09
3	C 60-4	144.77	52.35	91.70	188.33
4	C 60-7	155.05	46.69	94.83	189.77
5	C 65-0	173.65	73.47	96.39	172.02
6	C 65-2	190.07	71.30	126.71	183.03
7	C 65-4	191.16	67.69	144.95	186.64
8	C 65-7	201.81	77.08	145.01	187.61
9	C 70-0	191.52	113.36	114.56	129.60
10	C 70-2	191.70	119.13	136.70	166.55
11	C 70-4	197.65	127.26	146.21	175.09
12	C 70-7	198.56	113.48	175.33	179.54
13	F50-0	0.00	0.00	0.00	0.00
14	F50-10	108.84	30.32	51.74	84.84
15	F50-17	111.73	36.34	57.22	89.65
16	F50-22.5	107.76	35.50	56.14	79.42
17	F55-0	0.00	0.00	0.00	0.00
18	F55-10	181.05	35.98	56.80	106.98
19	F55-17	184.12	59.93	86.88	158.24
20	F55-22.5	183.21	52.83	84.48	143.68
21	F60-0	0.00	0.00	0.00	0.00
22	F60-10	186.46	54.75	115.40	137.30
23	F60-17	189.71	80.26	177.50	211.19
24	F60-22.5	186.10	55.23	121.90	174.73

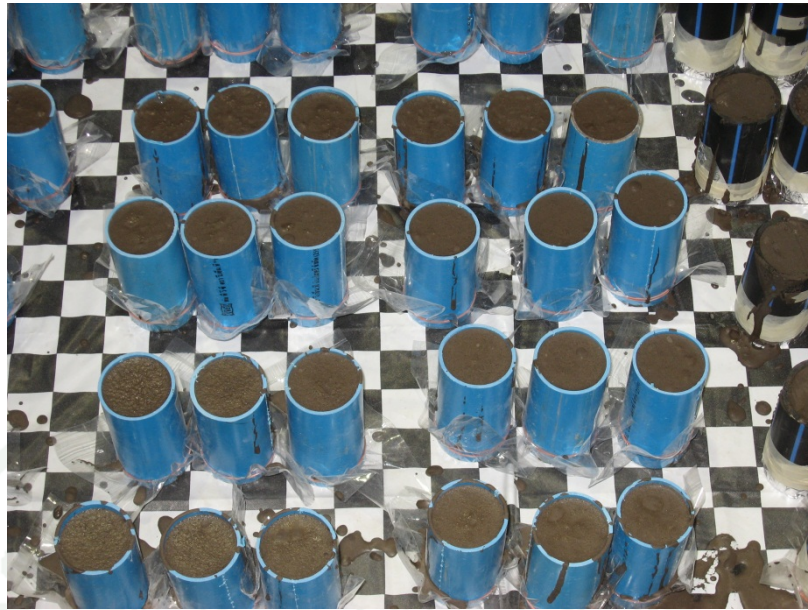
Appendix Table A8 Total porosity and pore size distribution of geopolymer paste

Mixed	Designation	Curing 24 °C			Curing 70 °C		
		Total	Cumulative	Average	Total	Cumulative	Average
		Porosity	Volume	Pore	Porosity	Volume	Pore
				Diameter			Diameter
		(%)	(cc/g)	r (µm)	(%)	(cc/g)	r (µm)
1	C 60-0	23.83	0.1434	1.33	21.97	0.1592	0.61
4	C 60-7	21.73	0.239	12.13	10.6	0.0566	0.2
15	F50-17	12.1	0.2144	21.93	14.62	0.2123	0.2
23	F60-17	8.28	0.0519	145.8	22.08	0.1073	0.76



Appendix B

Figures



Appendix Figure B1 Specimens of fly ash- based geopolymer



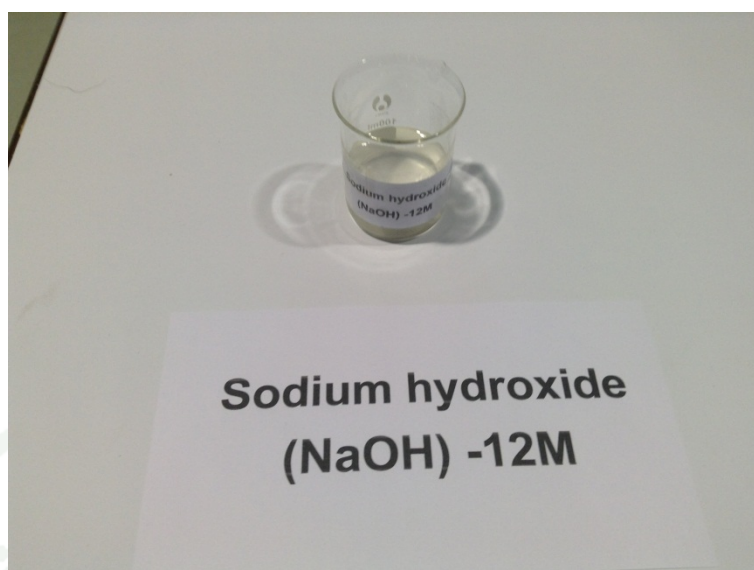
Appendix Figure B2 Compressive strength testing machine



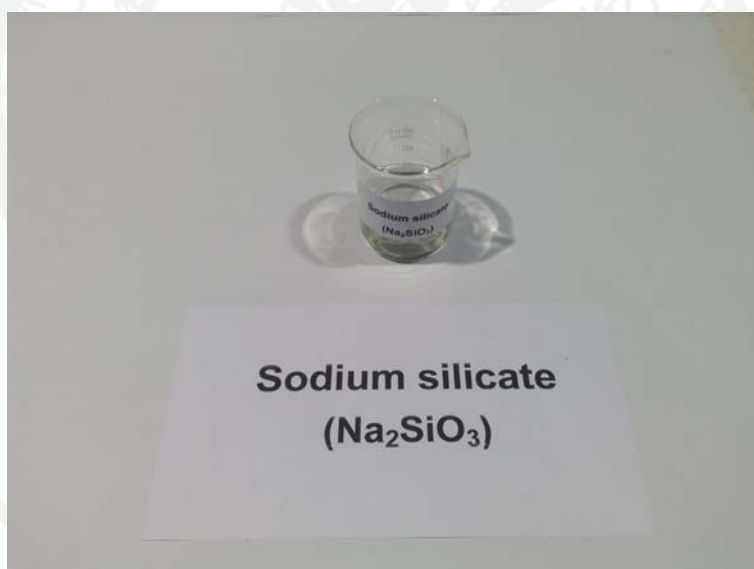
Appendix Figure B3 Vicat test set



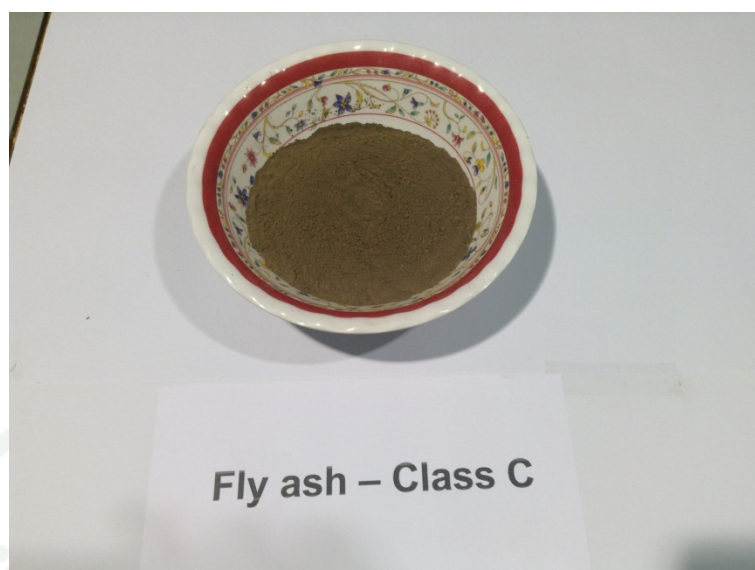
Appendix Figure B4 Flow test set



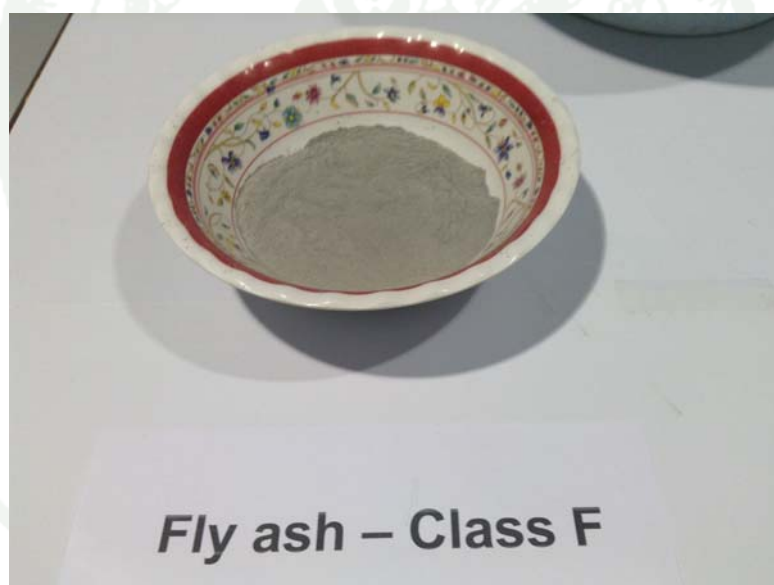
Appendix Figure B5 Sodium hydroxide solution



Appendix Figure B6 Sodium silicate



Appendix Figure B7 Fly ash class C



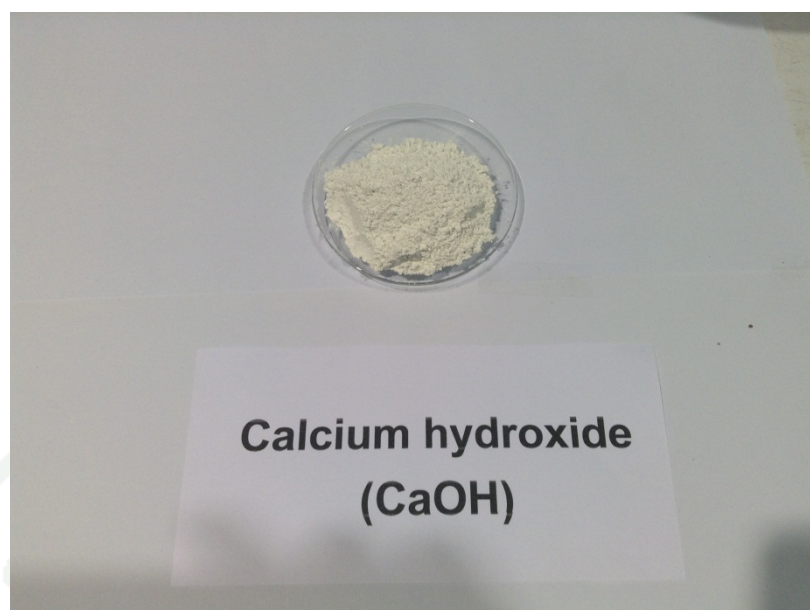
Appendix Figure B8 Fly ash class F



Appendix Figure B9 Specimen of fly ash class C- based geopolymer.



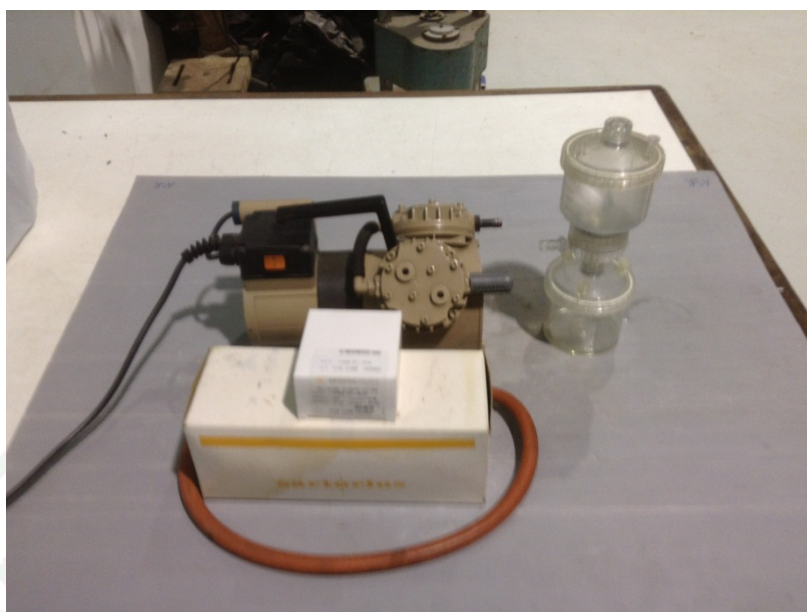
Appendix Figure B10 Specimen of fly ash class F- based geopolymer.



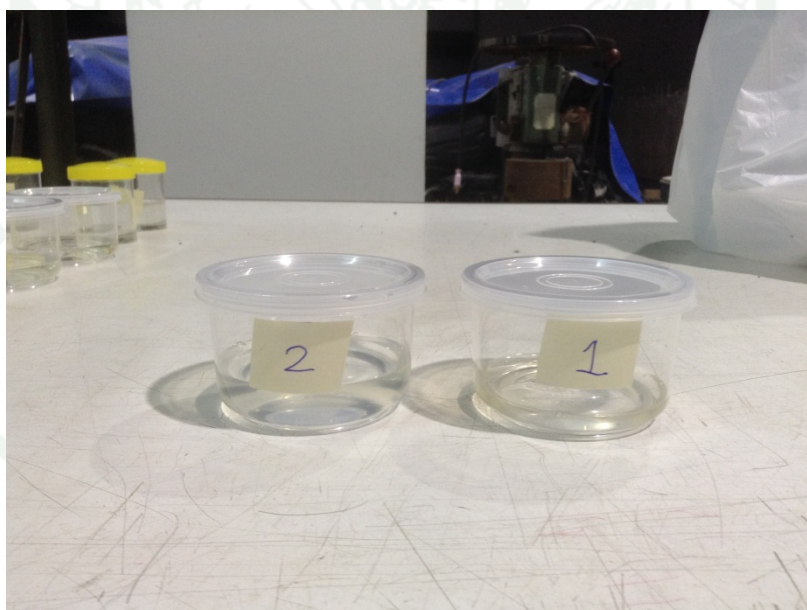
Appendix Figure B11 Calcium hydroxide (CaOH)



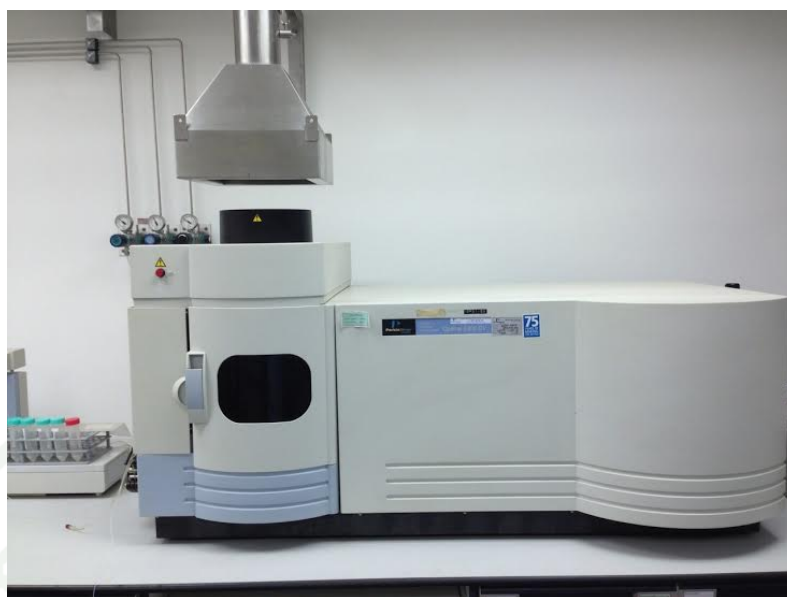
Appendix Figure B12 Hot air oven.



Appendix Figure B13 Dissolution test set



Appendix Figure B14 Result of dissolution liquid.



Appendix Figure B15 Inductively Coupled Plasma Optical Emission Spectroscopy



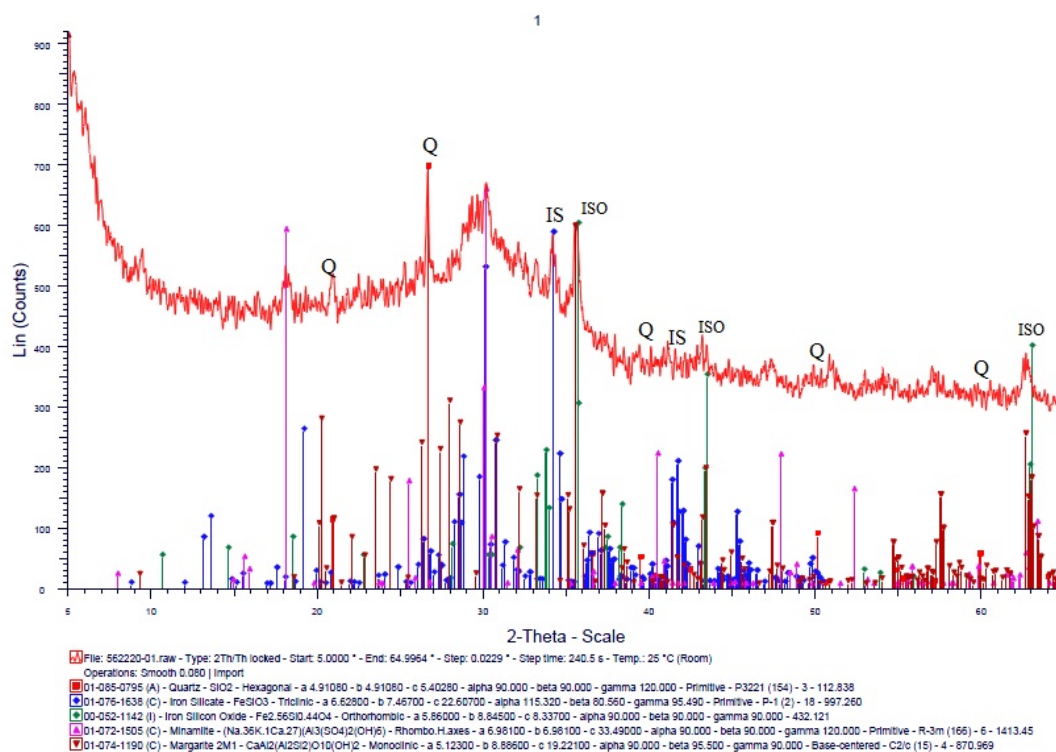
Appendix Figure B16 Scanning electron microscope



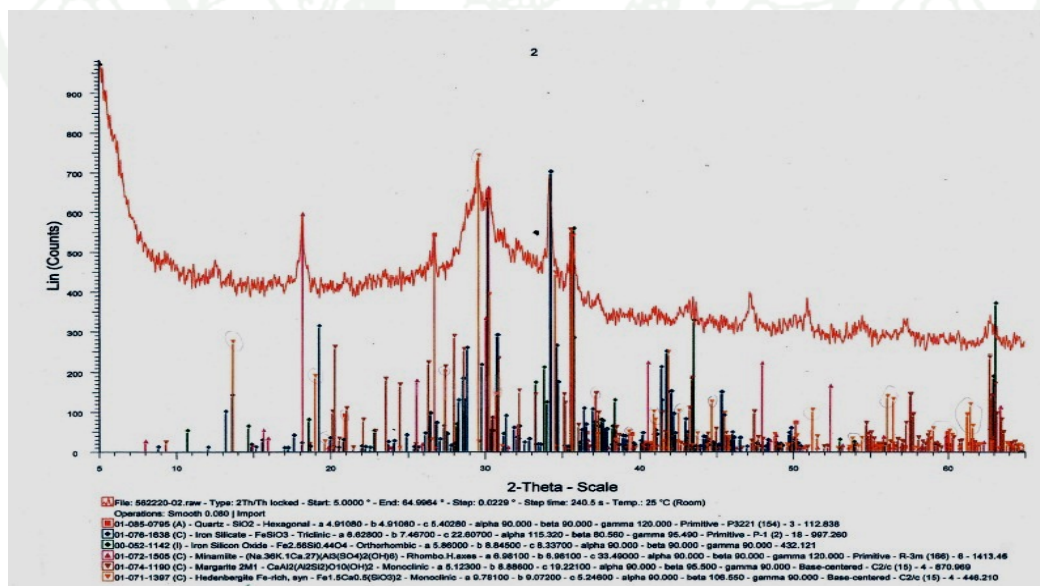
Appendix Figure B17 X-Ray Diffractometer



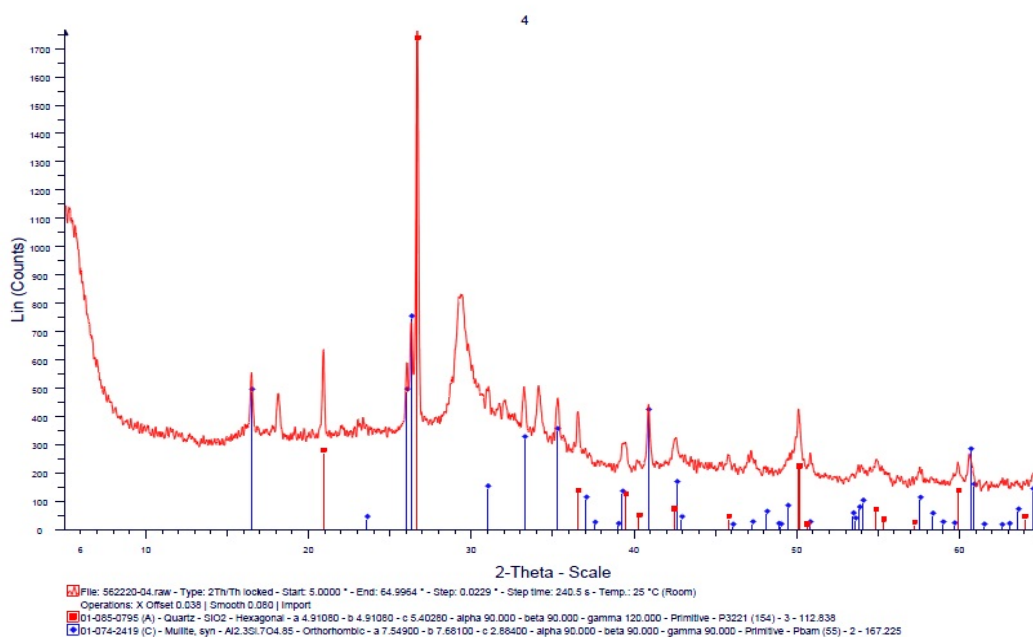
Appendix Figure B18 Pore size distribution MIP tests



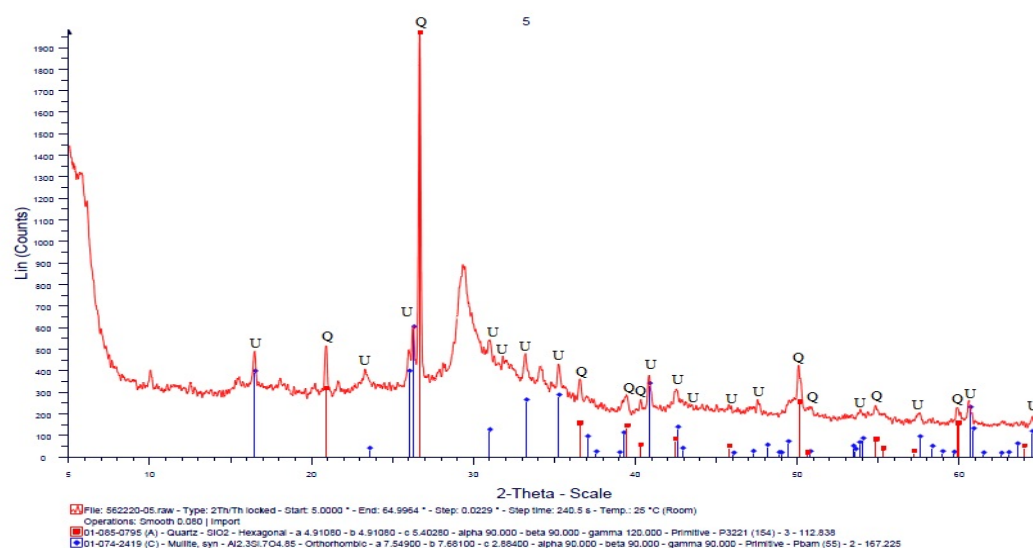
Appendix Figure B19 X – ray diffractograms of class C fly based geopolymer with solid 60% (fly ash 60%, Calcium hydroxide 0 %)



Appendix Figure B20 X – ray diffractograms of class C fly based geopolymer with solid 60% (fly ash 53%, Calcium hydroxide 7%)



Appendix Figure B21 X – ray diffractograms of class F fly based geopolymer with solid 60% (fly ash 43%, Calcium hydroxide 17%)



Appendix Figure B22 X – ray diffractograms of class F fly based geopolymer with solid 50 % (fly ash 33.0, Calcium hydroxide 17 %)

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