Development of Fly Ash Based Geopolymer As Anti Erosion Coating

by

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Dissertation submitted in partial fulfillment of
the requirement for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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MALAYSIA

CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Approved by,

....

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
May 2012

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own 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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NOOR HAFIZAH BINTI RAMLI@YUSOF

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In the name of God, Most Gracious and Most Merciful

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Thank you and best regards,

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ABSTRACT

Rapid development of marine resources increasingly demands the huge range of concrete coastal structures and offshore structures. However, the concrete exposed in rigorous marine environment is readily damaged by the erosive ocean-atmosphere and seawater. Besides, erosion also happens because of acidic environment especially from sewage and wastewater chemical treatment system. Several types of coatings such as acrylic, polyurethane, epoxy and others now have been tried as surface protection materials to inhibit the intrusion of corrosive and erosive ions. However, organic coating usually covers the concrete surface by physical absorption that makes it caduceus. The durability of thin coatings is also doubtful. The use of inorganic polymer coatings to substitute organic coatings seems to be an alternative way of improving the durability of marine concrete structures. The alternative material which is geopolymer has been introduced to the concrete that is more environmental friendly and saves cost. It is also known as inorganic polymer or alkali activated binder has gained worldwide interest and its high anticorrosion makes it a novel coating material. This development to the composite has been investigated widely over the past 50 years. By using the fly ash that contains high aluminosilicate and calcium to produce the geopolymer, the project will investigate and determine the best formula for geopolymer as anti erosion coating. Moreover, fly ash as has advantages over metakaolin in terms of lower cost as it is a waste from coal and easy to produce geopolymer. The best formula will be use to produce geopolymer and coat the concretes. The concretes that coat with fly ash based geopolymer will go through erosion evaluation to prove that the coating is effective anti erosion coating under acidic environment and rigorous marine environment.

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ABBREVIATIONS AND NOMENCLATURES

DE	Diatomaceous Earth
FYP 1	Final Year Project 1
H_2SO_4	Acid Sulfuric
KOH	Potassium
NaOH	Sodium Hydroxide
MA	Mechanical Activation
MgO	Mangan Oxide
PP	Polypropylene
RHBA	Rice husk-bark ash
SEM	Scanning Electronic Microscopy
XRD	X-Ray Diffraction
FESEM	Field Emission Scanning Electron Microscope

CHAPTER 1: INTRODUCTION

1.1 Background of Study

The project will study about the development of fly ash based geopolymer as anti erosion coating through some researches and experiments on the geopolymer. The purpose of research is to find the right formula to create the geopolymer based on fly ash as effective anti erosion coating to the concrete. The geopolymer coatings have to fulfill the requirement: a) acceptable setting time or solidifying time, b) low permeability of water, c) high anti-erosion due to sewage, wastewater and ocean-atmosphere, d) high bond strength to existing marine concrete, e) high bond strength to the concrete and f) high compressive strength. (1) The experiment will be conducted by using different concentration of sodium hydroxide, different ratio of Si/Al and different ratio of solid/liquid in order to find the best formula to produce geopolymer and to characterize the geopolymer samples. The geopolymers will be cured at 26°C and 60°C. All the samples will be characterized by using compressive strength test and field emission scanning electron microscope (FESEM). After the result is obtained, the project will continue by making the geopolymer as a coating. And the last stage is testing the concretes that coating with the geopolymer in acidic environment for anti-erosion evaluation.

1.2 Problem Statement

Rapid development of marine resources increasingly demands a huge range of concrete coastal structures and offshore structures. However, the concrete exposed in rigorous marine environment is readily damaged by the erosive ocean-atmosphere and seawater. Erosion also happens because of acidic environment especially from sewage and wastewater chemical treatment system. They can harm the concretes that are used as a connector to send the sewage and wastewater to the sea or river. Once the damage occurs, it cost a lot of money to repair. The durability of marine concrete structures, particularly to improve the anti-erosion property, has become the focus of the civil engineering, chemical engineering and material science. Several types of coatings such as acrylic, polyurethane, epoxy and others now have been tried as surface protection materials to inhibit the intrusion of corrosive and erosive ions. However, organic coating usually covers the concrete surface by physical absorption that makes it caduceus. Furthermore, the durability of thin coatings is also doubtful, particularly given the aging due to the exposure to the sun and the destructibility of waves although it has been reported that their service life was near 10 years. The use of inorganic polymer coatings to substitute organic coatings seems to be an alternative way of improving the durability of marine concrete structures. (1) One of the inorganic polymer is geopolymer that has potential to become novel material for coating as it possess high anti corrosion.

1.2.1 Erosion

Erosion is the displacement of solids (soil, mud, rock and other particles) by the agents of wind, water, ice or living organisms or by down-slope movement in response to gravity. (2) The building that is located near an ocean usually is prone to erosion because of the wind and the sea itself that contain high concentration of salt.

There are many types of erosion:

- **Gravity erosion** which is the down-slope movement of rocks and sediments caused mainly by the force of gravity.
- **Water erosion** which is the detachment and airborne movement of small soil particles caused by the impact of raindrops falling on the soil.
- **Shoreline erosion** occurs primarily through the action of currents and waves but sea level changes can also play a role.
- **Ice erosion** is caused by the movement of ice, typically in the form of glaciers.

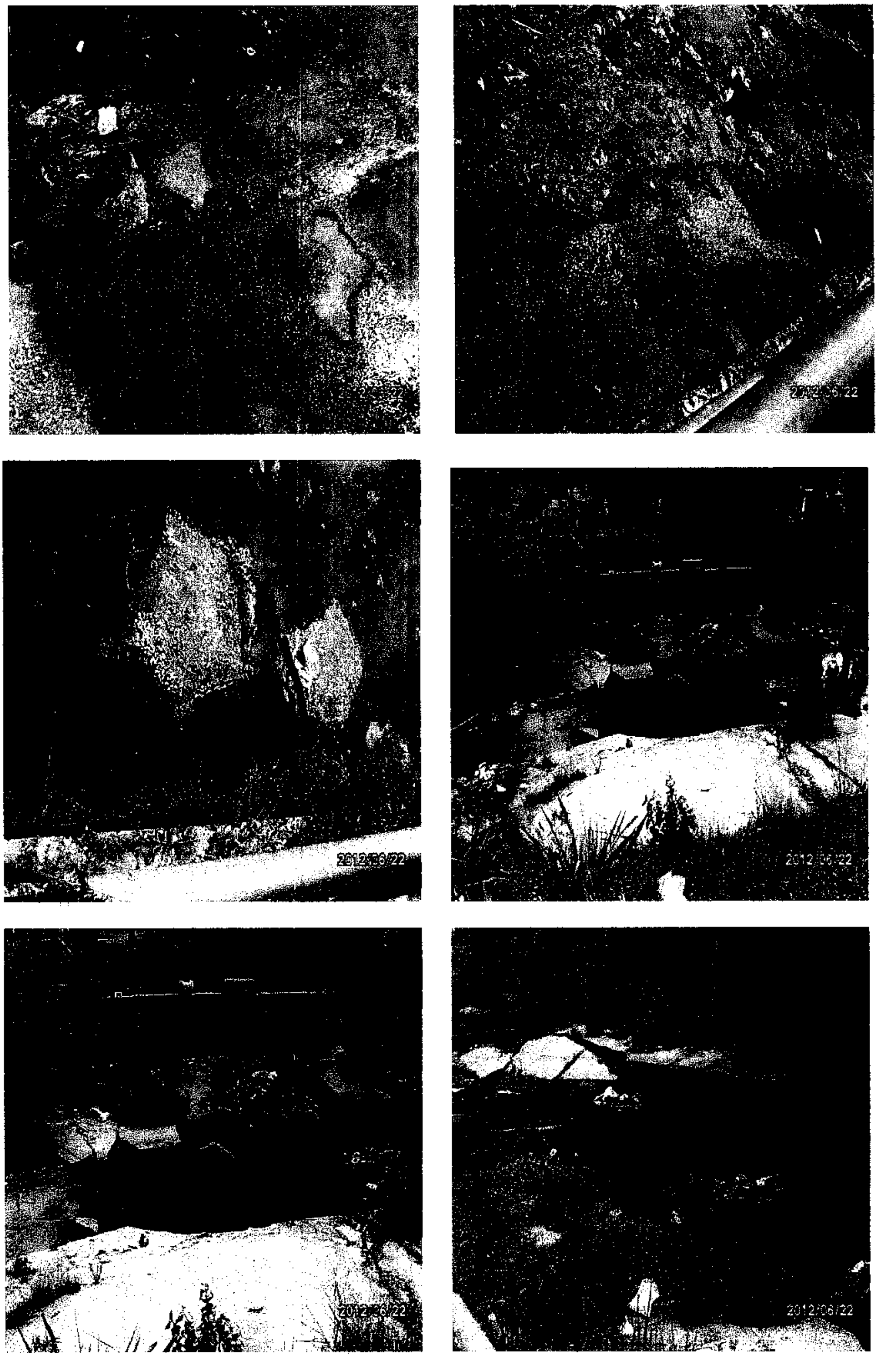


Figure 1.1: Effects of erosion at Medan Gopeng, Ipoh, Perak

1.3 Objectives and Scope of Study

1.3.1 Objective

The aim of the project is to find the right formula to produce geopolymer coatings that meet all the requirements as anti-erosion coatings. This project will investigate further the geopolymerisation of fly ash in different molarity of sodium hydroxide, different ratio of Si/Al and different of solid/liquid ratio in order to find the good strength of geopolymer for anti-erosion geopolymer coatings and the best setting time. Besides, at the end of the project, all the samples will be test in erosion test to check either the formula is good to produce geopolymer as anti erosion coating or otherwise.

1.3.2 Scope of Study

The project will investigate and determine the best formula to produce fly ash based geopolymer as anti erosion coating by running three experiments which are different concentration of sodium hydroxide, different Si/Al ratio and different solid/liquid ratio. The best formula will be used as a coating to the concretes. The concretes will be cured at 26°C and 60°C for a 24 hours and 7 days before conduct the erosion test to study the erosion profile effectively. The geopolymer samples will be test using compressive strength test and FESEM to characterize the samples.

CHAPTER 2: LITERATURE REVIEW

2.1 Geopolymer Background

Geopolymer is synthesized by **mixing aluminosilicate-reactive material with strong alkali solutions such as sodium hydroxide (NaOH), Potassium (KOH), sodium silicate or potassium silicate.** The mixture can be cured at room temperature or temperature cured. Under a strong alkali solution, aluminosilicate-reactive materials dissolve and form free SiO_4 and AlO_4 tetrahedral units. Water is gradually removed and the alkali clusters are linked to yield polymeric precursors through the sharing of all oxygen atoms between two tetrahedral units, thereby forming amorphous geopolymers. **Three common types of geopolymer are the polysilicate Al-O-Si chain, polysialate siloxo Al-O-Si-Si chain and polysialate disiloxo Al-O-Si-Si-Si chain.** (3)

Geopolymer also has **impressive acid and fire resistances and ability to immobilize toxic and radioactive materials. It is also a very promising material for protective coating of different surfaces including metal due to their superior mechanical, chemical and thermal resistance properties.** (4)

Geopolymer is also known as **inorganic polymer or alkali activated binder, has gained worldwide interest and its high anticorrosion makes it a novel coating material.** Geopolymers have **low permeability and excellent anticorrosion property. They could also efficiently bond with cement paste and mortar that probably results from the coexistence of C-S-H gels on cement and geopolymer surface.** The large shrinkage problem could be solved by appropriate addition of PP fiber and MgO as expansion agent as well as careful curing at an early age. (1)

2.2 Fly Ash Background

Fly ash is the most common source material for making geopolymers. Normally, **good high strength geopolymers can be made from class F fly ash.** However, it has been shown that **high calcium fly ash from lignite can also be used to produce geopolymer mortar with compressive strength up to 65.0 MPa.** The microstructure of fly ash geopolymer consists of aluminosilicate gel, unreacted fly ash and other crystalline phases. The negatively charged and tetrahedrally coordinated Al atoms inside the network are charge-balanced by alkali metal cations from the alkali solution. (3)

Fly ash is a by-product of coal-fired electric power stations. Rice husk–bark ash (RHBA) is a solid waste generated by biomass power plants using rice husk and eucalyptus bark as fuel. The major chemical constituent of RHBA is SiO2 (about 75%). Therefore, blending FA and RHBA can adjust the ratio of Si/Al as required. (5)

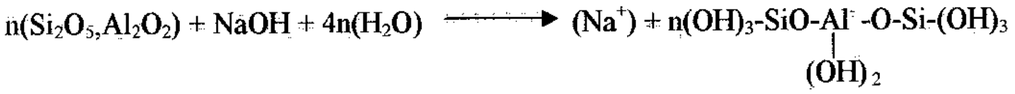
Fly ash has an advantage over metkaolin and slag. Fly ash geopolymers are being used in structural applications such as large concrete columns and railway sleepers and have demonstrated excellent mechanical properties and durability. (6)

2.3 Geopolymer formula

Mechanism I:

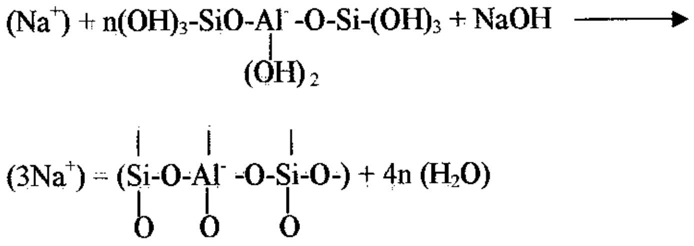
Fly Ash + Sodium Hydroxide ——→ Geopolymer precursor

(Si-Al source) (Alkaline liquid)



$$n(Si_2O_5,Al_2O_2) + NaOH + 4n(H_2O) \longrightarrow (Na^+) + n(OH)_3\text{-SiO-}\underset{(OH)_2}{\underset{|}{Al^-}}\text{-O-Si-}(OH)_3$$

Geopolymer precursor + Alkaline ions ——→ Geopolymer backbone



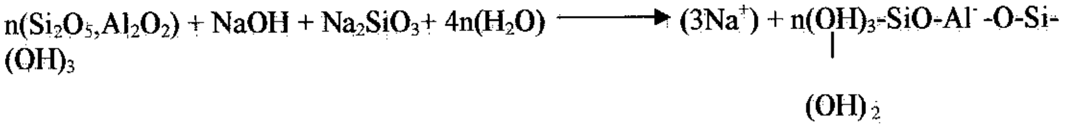
$$(Na^+) + n(OH)_3\text{-SiO-}\underset{(OH)_2}{\underset{|}{Al^-}}\text{-O-Si-}(OH)_3 + NaOH \longrightarrow$$

$$(3Na^+) = (\overset{|}{\underset{\underset{O}{|}}{Si}}\text{-O-}\overset{|}{\underset{\underset{O}{|}}{Al^-}}\text{-O-}\overset{|}{\underset{\underset{O}{|}}{Si}}\text{-O-}) + 4n\ (H_2O)$$

Mechanism II:

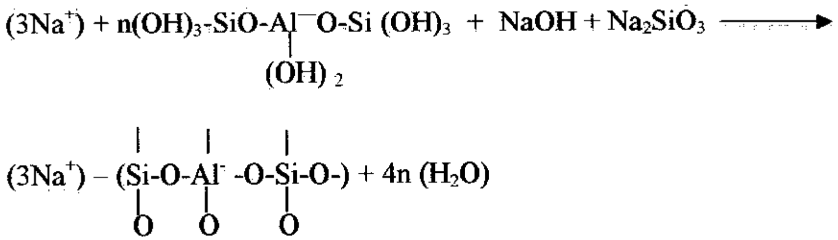
Fly Ash + Sodium Hydroxide + Sodium Silicate ——→ Geopolymer precursor

(Si-Al source) (Alkaline liquid)



$$n(Si_2O_5,Al_2O_2) + NaOH + Na_2SiO_3 + 4n(H_2O) \longrightarrow (3Na^+) + n(OH)_3\text{-SiO-}\underset{(OH)_2}{\underset{|}{Al^-}}\text{-O-Si-}(OH)_3$$

Geopolymer precursor + Alkaline ions ——→ Geopolymer backbone



$$(3Na^+) + n(OH)_3\text{-SiO-}\underset{(OH)_2}{\underset{|}{Al^-}}\text{-O-Si}\ (OH)_3 + NaOH + Na_2SiO_3 \longrightarrow$$

$$(3Na^+) - (\overset{|}{\underset{\underset{O}{|}}{Si}}\text{-O-}\overset{|}{\underset{\underset{O}{|}}{Al^-}}\text{-O-}\overset{|}{\underset{\underset{O}{|}}{Si}}\text{-O-}) + 4n\ (H_2O)$$

2.3.1 The mechanism of geopolymer gel formation investigated through seeded nucleation (7)

In particular, the geopolymer system is highly constrained; kinetically accessible amorphous structures are more likely to form than their highly crystalline counterparts. This is particularly true in high-silica systems, where the ability of the gel components is much lower and so the opportunities to rearrange into a more favourable crystalline structure are fewer. Recent work has shown that attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy is capable of providing key information regarding both the initial setting and later transformations taking place in geopolymer systems. In particular, an in situ ATR-FTIR study of the early period of geopolymer formation enabled analysis of changes in the rate-determining step as the alkalinity of the activator was increased above a critical value.

To synthesise the fly ash-based geopolymer control sample, 20.8 g of a 6M NaOH solution was mixed with 60 g of fly ash (Gladstone Power Station, Queensland, Australia, oxide composition and detailed characterisation given in Ref. and stirred mechanically for no more than 2 min. Additional samples were prepared with the same composition, but with either 0.01 or 0.1 g of Al_2O_3 nanoparticles (NanoScale Materials, USA, mean particle size 200 nm and BET surface area 275m2/g) dispersed in the activating solution immediately before mixing with the fly ash, to act as potential nucleation sites. In situ ATR-FTIR spectroscopy was performed on all samples for reaction periods of up to 3 days as described previously. Ex situ ATR-FTIR and X-ray diffraction (XRD) analysis were also performed after 100 days at 30 °C, again following procedures developed in previous work. Briefly, FTIR spectra of the samples were collected using a variance FTS 7000 FT-IR spectrometer, with a Specac MKII Golden Gate single reflectance diamond ATR attachment (45° angle of incidence) with KRS-5 optics and heater top plate maintained at 30 .C. Absorbance spectra were collected from 4000 to 400 cm^{-1} at a resolution of 2 cm^{-1}, with 64 scans per spectrum. In the ex situ experiments, geopolymer samples were removed from sealed containers and a freshly fractured surface immediately mounted onto the ATR crystal and clamped to obtain good contact. This procedure was designed to minimise atmospheric exposure, which can cause nanostructural changes through carbonation and evaporation of water from pore solutions or partially reacted samples. The ATR-FTIR spectra of all geopolymers were analyzed for the position of the Si.O.T (T: Si or Al) asymmetric stretching band (henceforth referred to as the gmain band h) using spectral subtraction of water. This uncovered the main band, particularly in the poorly reacted samples which display only a weak absorbance overshadowed by the contribution of the solvent.

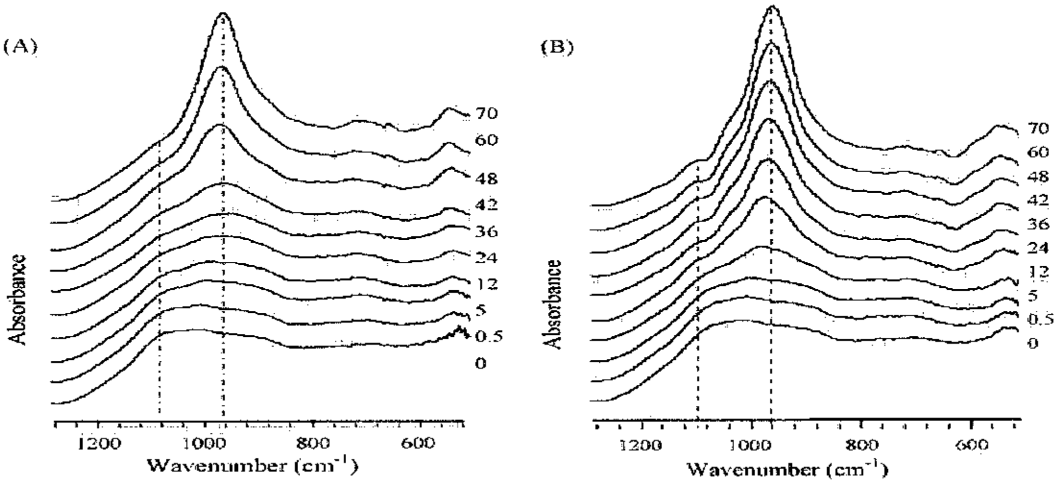


Figure 2.1: In situ ATR-FTIR spectra showing geopolymer development. (A) Control sample without nanoparticle addition and (B) with 0.01 mass % Al_2O_3 nanoparticles added.

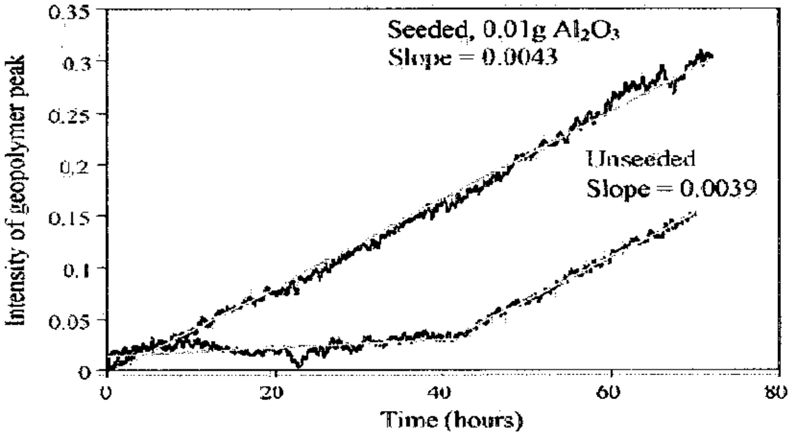
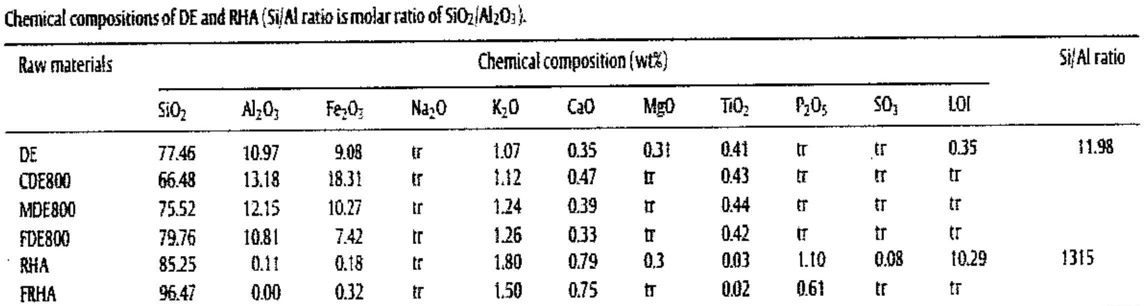


Figure 2.3: Changes in intensity at 960 cm^{-1} with time for seeded and unseeded geopolymers.

High surface area Al_2O_3 nanoparticles were added in very small quantities («1%) to seed growth processes. It was found that the 42 h reaction lag occurring in regular sodium hydroxide activation of fly ash does not occur when the synthesis mixture was seeded. Furthermore, there was a phase separation in the gel in the system seeded with nanoparticles, where a new gel very high in silica was formed.

2.3.2 Lightweight geopolymer made of highly porous siliceous materials with various Na_2O/Al_2O_3 and SiO_2/Al_2O_3 ratios (8)

Nowadays, lightweight construction materials are used to reduce the weight of building structures and improve thermal insulation efficiency of buildings. Suitable lightweight source materials containing silica and alumina such as diatomaceous earth (DE) and rice husk ash (RHA) are available in large quantity in Thailand. DE contains both silica and alumina and RHA contains mainly silica. They both have very high specific surface but particle morphologies are very different. DE is a sedimentary rock consisting principally of highly porous skeletons of diatom, in other word, a loosely packed mineral. The porous silica structure gives DE useful characteristics such as unique particulate structure, low bulk density, high absorptive capacity and high surface area.



Chemical compositions of DE and RHA (Si/Al ratio is molar ratio of SiO_2/Al_2O_3).

Raw materials	Chemical composition (wt%)											Si/Al ratio
	SiO_2	Al_2O_3	Fe_2O_3	Na_2O	K_2O	CaO	MgO	TiO_2	P_2O_5	SO_3	LOI	
DE	77.46	10.97	9.08	tr	1.07	0.35	0.31	0.41	tr	tr	0.35	11.98
CDE800	66.48	13.18	18.31	tr	1.12	0.47	tr	0.43	tr	tr	tr	
MDE800	75.52	12.15	10.27	tr	1.24	0.39	tr	0.44	tr	tr	tr	
FDE800	79.76	10.81	7.42	tr	1.26	0.33	tr	0.42	tr	tr	tr	
RHA	85.25	0.11	0.18	tr	1.80	0.79	0.3	0.03	1.10	0.08	10.29	1315
FRHA	96.47	0.00	0.32	tr	1.50	0.75	tr	0.02	0.61	tr	tr	

Tr=trace (0–0.049%).

Table 2.1: chemical composition of DE and RHA

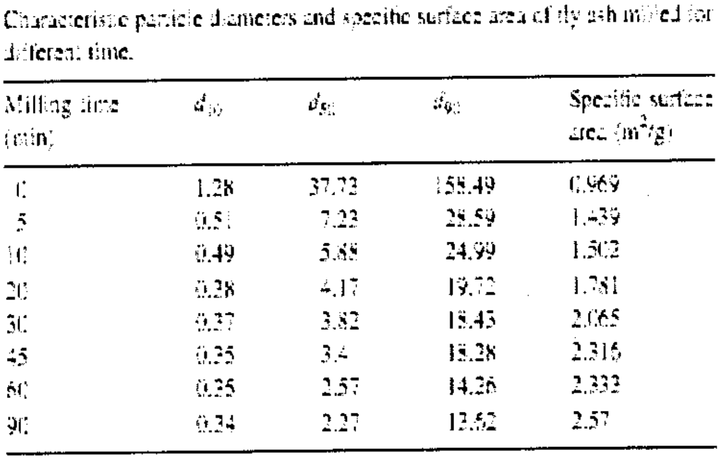
The mixing procedure started with mixing of NaOH (or KOH) solution, sodium silicate (Na_2SiO_3) and DE for 5 min. Water was then added and mixed for a further 5 min. The mixtures were cast in 50 mm×50 mm×50 mm acrylic cube mould. The specimens were then wrapped with plastic film to prevent moisture lost during curing. After delayed time of 1 h, the specimens were cured in an electric oven. The specimens were cooled down in the oven and then demoulded and kept at 23 ◦C room under ambient conditions until the scheduled testing. The compressive strength tests were performed at 7 days in accordance with ASTM C109, density measurements were also conducted at 7 days.

Based on the investigation, diatomaceous earth (DE) appears to be a good candidate material for producing lightweight geopolymeric material. It was further confirmed that calcined and sieved DE produced fine reactive particles suitable for use as a source material. The optimum calcination temperature of DE was 800 °C and the finer DE was more reactive due to the increase in the surface area. This allowed greater contact of DE particle surface with alkali solution and hence a faster leaching of silica and alumina. The XRD patterns of the calcined DE indicated the transformation of chemical structures of montmorillonite and kaolinite. With regards to the types of alkali, geopolymer pastes activated with 10 M NaOH possessed higher compressive strength than that with 10 M KOH. Curing temperature and duration also affected the properties of the geopolymers. The optimum curing temperature and time were 75 °C and 5 days. The starting Na_2O/Al_2O_3 ratios of mixtures also affected the properties of the geopolymer pastes. **At starting SiO_2/Al_2O_3 ratio of 13.0, the increase in starting Na_2O/Al_2O_3 ratios from 1.0 to 3.0 increased the compressive strength from 11 to 60 kg/cm2 but the samples with Na_2O/Al_2O_3 ratios of 2.0 and 3.0 were not stable as indicated in water immersion test. However, the bulk density values also increased from 0.93 to 1.5 g/cm3. RHA was incorporated in mixtures maintain the low density of geopolymer paste samples. High SiO_2/Al_2O_3 ratios used resulted in the lightweight geopolymer materials.**

2.3.3 Mechanical activation of fly ash: Effect on reaction, structure and properties of resulting geopolymer (9)

The merit of using mechanical activation (MA) for improving bulk and surface reactivity is well accepted. MA offers the possibility to alter the reactivity of solids through physicochemical changes in bulk and surface without altering overall chemistry of the material. Some very interesting findings on the MA of blast furnace slag and fly ash has been recently reported by us. Complete hydration of slag can be achieved for mechanically activated slag and without any chemical addition. Mechanical induced reactivity of fly has been exploited to tailor properties of geopolymer, and geopolymer having compressive strength of up to 120 MPa can be produced through judicious application of MA along with other processing parameters. It was earlier reported by us that the selection of milling device for MA influenced the reactivity of fly ash, i.e. fly ash of same fineness behaves differently depending upon milling device. Geopolymer prepared from fly ash that was mechanically activated in a vibratory mill showed superior mechanical properties as compared to their counterparts prepared from fly ash of similar particle size but obtained using other high energy mills, e.g. attrition mill. Recently, suitability of vibratory mill for MA of fly ash for application in geopolymer concrete has also been reported by other researchers.

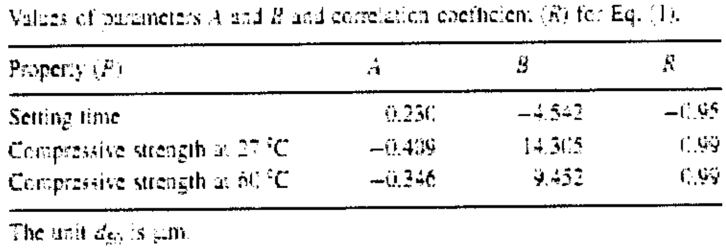
2 kg batch size was used for milling. The size of stainless steel media balls was 12.5 mm. Material to media ratio of 1:35 was maintained during milling. The fly ash was milled for 5, 10, 20, 30, 45, 60 and 90 min, and the milled samples are referred to as FA5, FA10...FA90 in subsequent description. Raw fly ash (FA0) was used as a reference. . The sample preparation involved the following steps: (a) preparation of alkaline activator solution, (b) mixing of alkaline activator with the powder sample, and (c) loading of the mix in calorimeter. Analytical grade sodium hydroxide in flaky form (98% purity) was used to prepare alkaline activator solution. Alkaline activator of 6 M concentration was prepared in distilled water at least 24 h before use.7 g solid sample and 3.5 ml of activator solution were used throughout the study. The physical properties of geopolymer were measured as follows: a) setting time at 27°C, b) compressive strength of samples after curing for 28 days and c) compressive strength of samples at 27°C for 24 hr and geopolymerisation at 60°C for 4 hr.



Characteristic particle diameters and specific surface area of fly ash milled for different time.

Milling time (min)	d_{10}	d_{50}	d_{90}	Specific surface area (m^2/g)
0	1.28	37.73	158.49	0.969
5	0.51	7.23	28.59	1.439
10	0.49	5.88	24.99	1.502
20	0.28	4.17	19.72	1.781
30	0.27	3.82	18.43	2.065
45	0.25	3.4	15.28	2.316
60	0.25	2.57	14.26	2.332
90	0.34	2.27	12.62	2.57

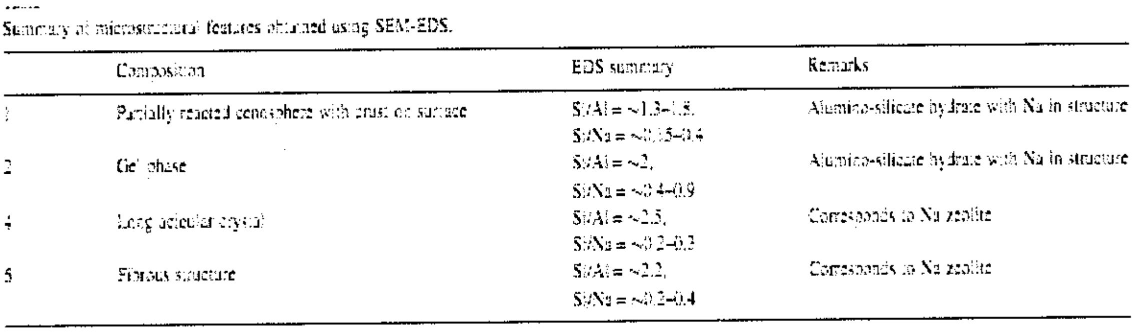
Table 2.2: characteristic particle diameters and specific surface area of fly ash



Values of parameters A and B and correlation coefficient (R) for Eq. (1).

Property (P)	A	B	R
Setting time	0.230	−4.542	−0.95
Compressive strength at 27 °C	−0.439	14.305	0.99
Compressive strength at 60 °C	−0.346	9.452	0.99

The unit d_{50} is μm.



Summary of microstructural features obtained using SEM-EDS.

	Composition	EDS summary	Remarks
1	Partially reacted cenosphere with crust on surface	Si/Al = ~1.3–1.8, Si/Na = ~0.15–0.4	Alumino-silicate hydrate with Na in structure
2	Gel phase	Si/Al = ~2, Si/Na = ~0.4–0.9	Alumino-silicate hydrate with Na in structure
4	Long acicular crystal	Si/Al = ~2.5, Si/Na = ~0.2–0.3	Corresponds to Na zeolite
5	Fibrous structure	Si/Al = ~2.2, Si/Na = ~0.2–0.4	Corresponds to Na zeolite

Table 2.3: Value of parameter A and B and correlation coefficient and summary of microstructural features

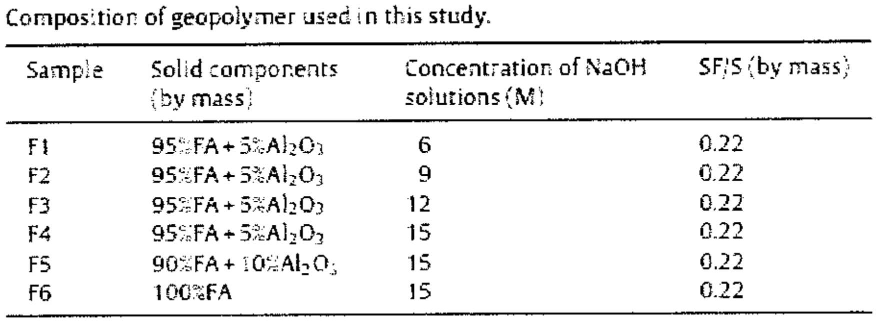
In conclusion, **mechanical activation in eccentric vibratory mill increases the reactivity of fly ash. The reactivity of fly ash varies with median particle size and increases vary rapidly when the particle size is reduced to less than 5–7 mm.** As a result, geopolymerisation at ambient temperature is possible. Besides, the effect of mechanical activation on structural reorganisation is evident from FTIR spectrum corresponding to Si–O stretching. The splitting of peak is associated with formation of less polymerised structure at 997 cm^{-1} and non-dissolved high polymerised structures at 1091 cm-1. A high degree of correlation between the properties of geopolymer (setting time and compressive strength) an inverse of median size of fly ash is observed. Combined effect of particle size (increase in surface area) and change in reactivity due to mechanical activation altered the geopolymerisation reaction. The improvement in physical properties is related to the intrinsic structure developed due to enhanced geopolymerisation.

2.4 Coating

Coatings are used both to protect and to decorate metal. A high quality, well applied coating can protect a substrate for up to 25 years before new application is required. **Coatings will protect the composite or the building or the concrete from cracking easily and increase the strength of the structures.**

2.5 The influence of α-Al_2O_3 addition on microstructure, mechanical and formaldehyde adsorption properties of fly ash-based geopolymer products.

It has been proven that **lower Si/Al ratio results in larger surface area of geopolymer and is beneficial for adsorption capacity.** Al_2O_3 mainly has two crystalline modifications: α-Al_2O_3 and γ-Al_2O_3. α-Al_2O_3 is highly crystalline and the most thermodynamically stable form of alumina, and therefore only slowly soluble in highly concentrated alkaline solution **while γ-Al_2O_3 is less crystalline and is easily soluble in strong alkaline solution.** It is well understood that aluminum component of fly ash tends to dissolve more easily than the silicon component at early stage of geopolymerization so that the addition of γ-Al_2O_3 leads to very high Al concentration at this stage. However, the addition of α-Al_2O_3 can be expected to averagely adjust the Si/Al ratio at the whole stage. (10)



Composition of geopolymer used in this study.

Sample	Solid components (by mass)	Concentration of NaOH solutions (M)	SF/S (by mass)
F1	95%FA + 5%Al_2O_3	6	0.22
F2	95%FA + 5%Al_2O_3	9	0.22
F3	95%FA + 5%Al_2O_3	12	0.22
F4	95%FA + 5%Al_2O_3	15	0.22
F5	90%FA + 10%Al_2O_3	15	0.22
F6	100%FA	15	0.22

Table 2.4: Composition of geopolymer

It is commonly believed that compressive strength of geopolymer increases with increasing Si/Al ratio under the same synthesis conditions and -Al_2O_3 addition is unlikely to increase this ratio.

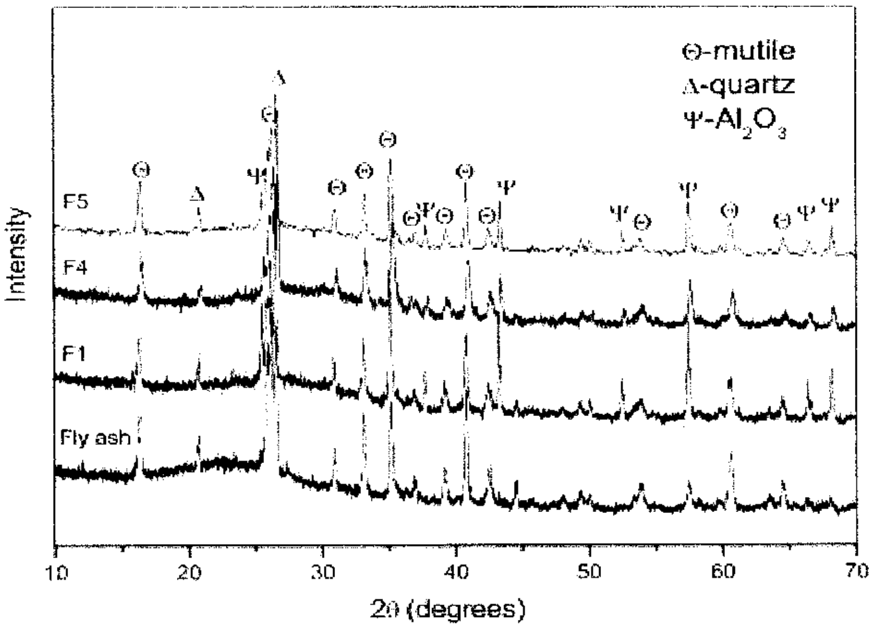


Figure 2.4: XRD patterns of geopolymer products

From XRD analysis, as to Al_2O_3 detectable in geopolymer samples, its peak intensity in sample F4 (5 wt% α-Al_2O_3, 15 M NaOH) is lower than that in sample F1 (5 wt% α-Al_2O_3, 6 M NaOH) and F5 (10 wt% α-Al_2O_3, 15 M NaOH). This suggests that α-Al2O3 can partially dissolve in alkaline activator solution as mentioned in introduction and the solubility increases with the increase of alkalinity. Besides, from FTIR analysis, it can be summarized that appropriate amount of -Al2O3 addition (such as 5 wt%) can reduce the Si/Al ratio in activator solution so promotes the geopolymerization and allows a greater level of structure reorganization of geoploymer gel, which can be explained by the higher ability of the gel components in high-alumina system. This behavior takes positive influence on the microstructure and mechanical properties of geopolymer products.

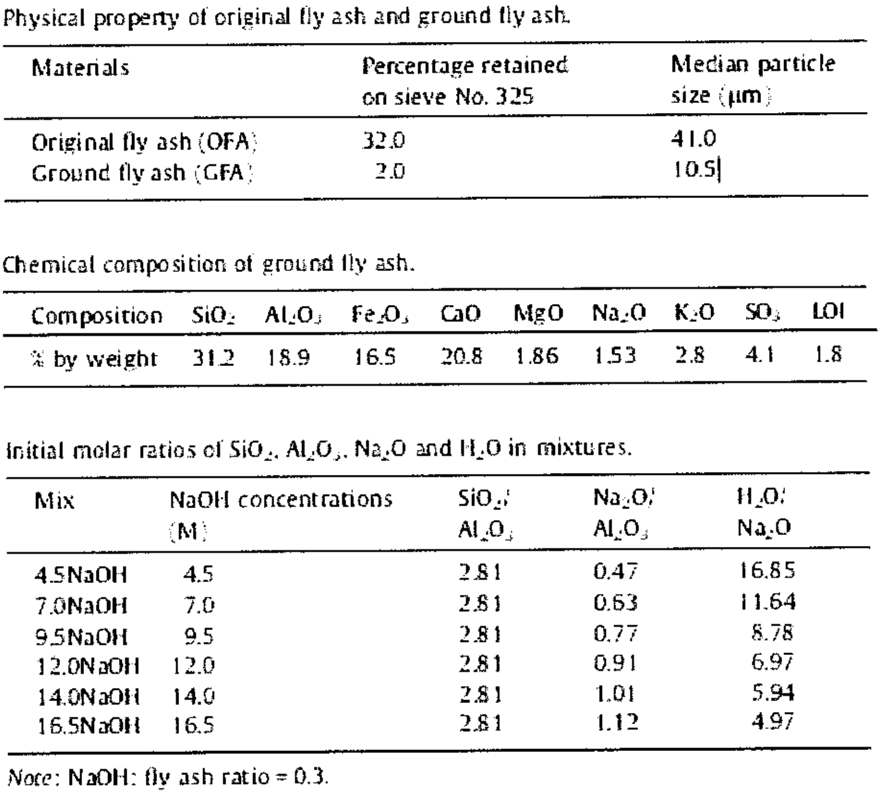
Addition of α- Al_2O_3 can reduce the Si/Al ratio of geopolymer and takes influence on the microstructure and mechanical properties of the fly ash-based geopolymer products. α- Al_2O_3 addition with appropriate amount (such as 5 wt%) increases the geoplymerization extent. This results in higher compressive strength and surface area. A better level of structural ordering of amorphous geopolymer gel for sample with 5 wt% α- Al_2O_3 addition can also be observed through FTIR analysis. In contrast, **excessive addition (such as 10**

wt%) gives rise to the dramatic decrease of compressive strength and surface area. The tests of formaldehyde adsorption capacity indicate that fly ash-based geopolymer products exhibit much better property of purifying indoor formaldehyde vapor than fly ash itself. (10)

2.6 Concentration of sodium hydroxide (NaOH)

Alkali activating solution is important for the dissolving of Si and Al atoms to form geopolymer precursors and finally aluminosilicate material. The most commonly used alkaline activators are NaOH and KOH. In the synthesis of geopolymers, NaOH was found to significantly affect both the compressive strength and structure of geopolymers. The NaOH concentration in the aqueous phase of the geopolymeric system acts on the dissolution process, as well as on the bonding of solid particles in the final structure. When fly ash comes into contact with NaOH, leaching of Si, Al and others minor ions begins. The amount of leaching is dependent on NaOH concentration and leaching time. The mixing of fly ash with 10 M NaOH for 10 min is appropriate for synthesis of geopolymers.

According to Kiatsuda Somna, from his journal, NaOH concentrations of 4.5, 7.0, 9.5, 12.0, 14.0 and 16.5 M were used to investigate the effects of NaOH concentration on geopolymer pastes. Fly Ash was continuously mixed with NaOH solution for 5 min. The paste samples were cast in plastic molds with a diameter of 30 mm and height of 60 mm and kept in a controlled chamber at 25–28 °C until the testing age. Besides, compressive strengths of specimens at 7, 14, 28, 42 and 60 days were determined following the procedure described in ASTM D1633 and Energy dispersive spectroscopy (EDS) was used to study the chemical composition of the geopolymers. Scanning electron microscope (SEM) was used to study the fracture surface of the geopolymer. An X-ray diffractrometer was used to provide fundamental information on geopolymer structure. Infrared spectra were used to record molecular absorption and transmission to create a molecular fingerprint of the samples. The tests were performed at the age of 60 days.



Physical property of original fly ash and ground fly ash.

Materials	Percentage retained on sieve No. 325	Median particle size (μm)
Original fly ash (OFA)	32.0	41.0
Ground fly ash (GFA)	2.0	10.5

Chemical composition of ground fly ash.

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	LOI
% by weight	31.2	18.9	16.5	20.8	1.86	1.53	2.8	4.1	1.8

Initial molar ratios of SiO_2, Al_2O_3, Na_2O and H_2O in mixtures.

Mix	NaOH concentrations (M)	SiO_2/ Al_2O_3	Na_2O/ Al_2O_3	H_2O/ Na_2O
4.5NaOH	4.5	2.81	0.47	16.85
7.0NaOH	7.0	2.81	0.63	11.64
9.5NaOH	9.5	2.81	0.77	8.78
12.0NaOH	12.0	2.81	0.91	6.97
14.0NaOH	14.0	2.81	1.01	5.94
16.5NaOH	16.5	2.81	1.12	4.97

Note: NaOH: fly ash ratio = 0.3.

Table 2.5: Physical property of original fly ash and ground fly ash, chemical composition and initial molar ratios of SiO_2, Al_2O_3, Na_2O and H_2O in mixtures.

From the journal, it showed that **the fine fly ash is also more effective in decreasing capillary pores than as-received coarser fly ash.** In addition, **the strength development of GFA pastes at the early ages of 7–14 days was high, and strength gain after 14 days was small except for the 4.5 NaOH sample which showed a high rate of strength development up to 28 days. An increase in the NaOH concentration from 4.5 to 9.5 M clearly increased the strengths of the pastes. Increasing NaOH concentration from 9.5 to 14.0 M NaOH also increased the strength of paste but to a lesser extent. A maximum strength of 25.5 MPa at 60 days was obtained with the 14.0 NaOH mix.** Additionally, the use of a low alkali solution resulted in a weak chemical reaction. The compressive strength increased with an increase in NaOH concentration mainly through the leaching of silica and alumina with high concentrations of NaOH. **An increase in NaOH concentration from 4.5 to 14.0 M increased the compressive strength of pastes. The relatively high compressive strengths of 23.0–25.5 MPa were obtained with the NaOH concentrations of 9.5–14.0 M. However, when the concentration of NaOH**

was 16.5 M, the compressive strength started to decline. When OH^{-1} concentration was high enough, dissolution of fly ash was accelerated, but polycondensation was hindered. An increase in alkali concentration enhanced strength development of the geopolymers, but excess hydroxide ion concentration caused aluminosilicate gel precipitation at the very early stages, resulting in lower strength geopolymers. From EDS analysis, The Si/Al ratio decreased as concentration of NaOH increased. This result indicated that leaching of Si and Al was dependent on NaOH concentration. At low NaOH, leaching of Si was higher than of Al, and subsequent reactions resulted in a geopolymer paste with an average Si/Al ratio of 1.54. At higher NaOH concentrations, rates of Al leaching were improved, resulting in geopolymer pastes with lower Si/Al ratios. From SEM analysis, the formation of C–S–H contributed partly to the strength development and resulted in aluminosilicate phase with a low Si/Al ratio. A decrease in Si/Al ratio could lead to the aluminosilicate compound of lower strength, accompanied by microstructure with increased crystalline phase.

Therefore, **for relatively high strength, NaOH-activated ground fly ash geopolymer pastes that are cured at ambient temperature (25–28°C), NaOH concentrations of 9.5–14.0 M are recommended. This condition is appropriate for the ground fine fly ash with median particle size of 10.5μm.** Moreover, XRD, SEM, EDS and FTIR studies showed that sodium hydroxide-activated ground fly ash geopolymerization occurred at room temperature. Therefore, ground fine fly ash can be used as a source material for making geopolymers cured at ambient temperature. (3)

2.7 Acid Rain

Acid rain is a rain or any other form of precipitation that is unusually acidic, meaning that it possesses elevated levels of hydrogen ions (low pH). It can have harmful effects on plants, aquatic animals, and infrastructure. Acid rain is caused by emissions of carbon dioxide, sulfur dioxide and nitrogen oxides which react with the water molecules in the atmosphere to produce acids. Governments have made efforts since the 1970s to reduce the release of sulfur dioxide into the atmosphere with positive results. Nitrogen oxides can also be produced naturally by lightning strikes and sulfur dioxide is produced by volcanic eruptions. **The chemicals in acid rain can cause paint to peel, corrosion of steel structures such as bridges, and erosion of stone statues.**(11)

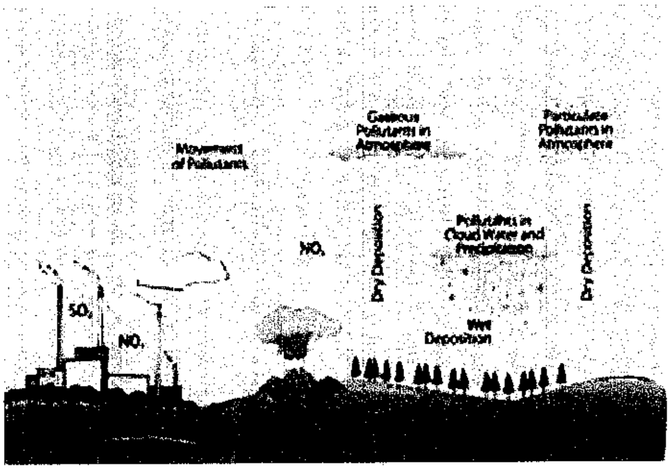


Figure 2.5: Acid rain mechanism

2.8 Acid Sulfuric Environment

Degradation of concrete members exposed to aggressive sulfuric acid environments is a key durability issue that affects the life cycle performance and maintenance costs of vital civil infrastructure. Sulfuric acid in groundwater, chemical waste or generated from the oxidation of sulfur bearing compounds (e.g. pyrite) in backfill can attack substructure concrete members. Moreover, concrete structures in industrial zones are susceptible to deterioration due to acid rain of which sulfuric acid is a chief component. Considerable damage can occur to sewage systems by biogenic sulfuric acid corrosion. Parker attributed this to a chemical–microbial interaction in sewage systems. **The effect of sulfuric acid**

on concrete is more detrimental than that of sulfate attack; in addition to attack by sulfate ions, there is a dissolution effect caused by hydrogen ions.

Corrosion of concrete due to sulfuric acid can generally be characterized by the following reactions (12):

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4.2H_2O \qquad (1)$$

$$CaSiO_2.2H_2O + H_2SO_4 \rightarrow CaSO_4 + Si(OH)_4 + H_2O \qquad (2)$$

$$\begin{aligned}&3CaO.Al_2O_3.12H_2O + 3(CaSO_4.2H_2O)\\&\quad + 14H_2O \rightarrow 3CaO.Al_2O_3.3CaSO_4.32H_2O. \qquad (3)\end{aligned}$$

To evaluate the resistance of concrete to sulfuric acid, three testing approaches have been adopted in the literature: chemical, micro-biological and in-situ tests. In chemical tests, mortar or concrete specimens are immersed in 1 to 5% sulfuric acid solutions for a specified time period with or without pH control. Micro-biological tests are peculiar to simulating biogenic sulfuric acid corrosion of concrete by accelerating growth of bacteria, which produce sulfuric acid, on concrete samples placed in environmentally controlled chambers. Exposure of concrete to specific sulfuric acid environments can also be investigated using in-situ tests. So many articles discuss about how to test concretes on acid sulfuric environment. As example, in a series of chemical tests with different sulfuric acid concentrations of 1–3%, Fattuhi and Hughes (13) showed that sulfate resistant portland cement (SRPC) did not offer marked improvement compared to that of ordinary portland cement (OPC) in reducing the mass loss of mortar or concrete specimens. Also, they indicated that for high (greater than 1%) sulfuric acid concentrations, minimizing the cementitious materials content in concrete can effectively reduce the rate of acid attack expressed by mass loss. Because acid specifically attacks cementitious constituents, concretes with a low w/cm and a high cementitious volume fraction are more vulnerable to greater mass loss. The effect of reducing the w/cm on improving the resistance of concrete to sulfuric acid attack was only significant at lower acid concentrations (1%). For instance, Durning and Hicks (14) and Mehta (15) reported that the incorporation of silica fume increased the resistance of concrete to 1% sulfuric acid attack due to reduced calcium hydroxide content and lower permeability. Conversely, Monteny et al. (16) reported a negative effect of silica fume incorporation in concrete specimens exposed to 0.5% sulfuric acid. They stated that a refined pore structure with higher capillary suction would cause deeper penetration of acidic solutions into concrete and increase the exposed surface area in contact with acid. These studies applied wire brushing to specimens. At higher concentrations of sulfuric acid solutions, Durning and Hicks (14) and Roy et al. (17) (no wire brushing) showed that silica fume did not improve the resistance of concrete and mortar to 5% sulfuric acid solutions. On the contrary, in a 5% sulfuric acid solution, Yamoto et al. (18) showed that time intervals to reach 25% mass loss were almost doubled for 30% silica fume concrete specimens relative to control specimens without silica fume. No information was given on the method of loose materials removal in Yamoto et al. (18) Monteny et al. (16) reported that the highest resistance to a 0.5% sulfuric acid solution was achieved by a binary binder mixture comprising more than 60% ground granulated blast furnace slag. Conversely, Chang et al. (19) recently reported that binary binder concrete mixtures prepared with 60% slag and ternary binder mixtures with 56% slag and 7% silica fume had inferior performance compared to that of a 100% OPC mixture when immersed in a 1% sulfuric acid solution with a pH of 1.27.

For this project, I choose **3% sulfuric acid solution to be use to represent the worst reported condition in the wastewater system.** (20) At first, the normal concretes will be test by merge them in acid sulfuric solution in one month with different temperature to prove that concrete is easily erode and to form erosion profile and compare it with erosion profile of concretes with geopolymer coating. The mass loss will be determined the erosion profile. Thus, the initial weight and the current weight of concretes will measured in every week.

$$\%\ of\ mass\ loss = \frac{initial\,mass - current\,mass}{initial\,mass} x100\% \quad (12)$$

2.9 Concrete

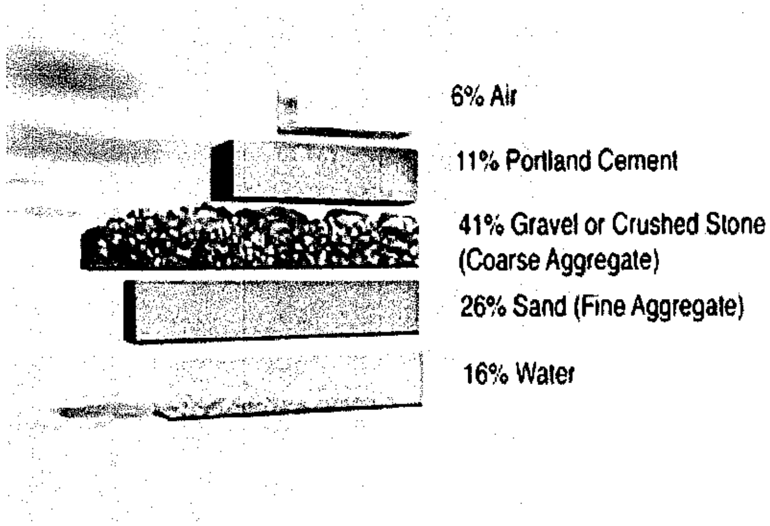
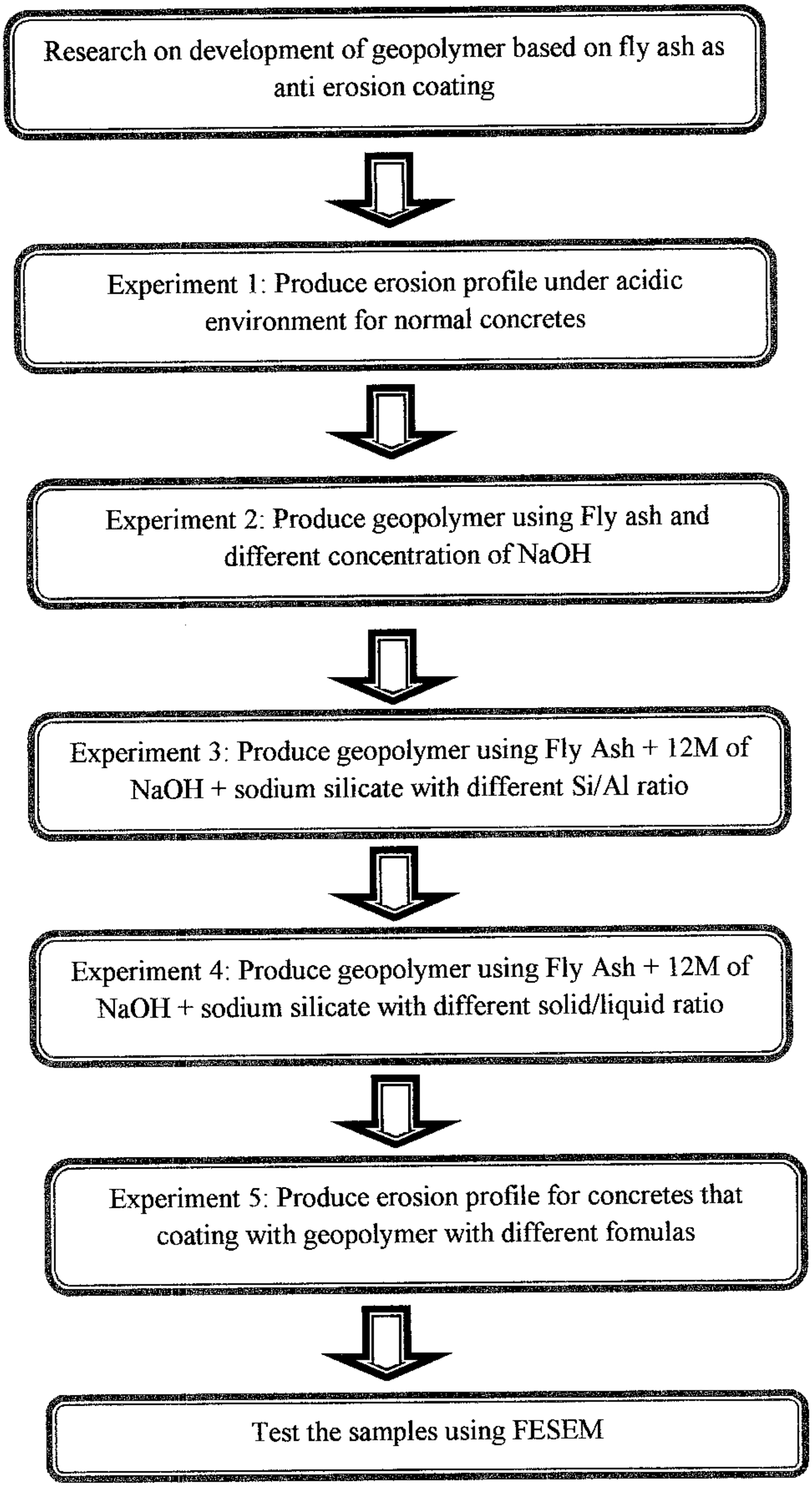


Figure 2.6: Concrete content (21)

There are many defects in concrete. One of the defects is crazing that caused by minor shrinkage in rapid drying conditions. Spalling is the slab edges and joints or break leaving an elongated cavity. It caused by heavy loads or impact with hard objects. As concrete expands and contracts the weak edges may crack and break. Besides, blistering can caused when the fresh concrete surface is sealed by trowelling while trapping air. This may particularly occur in thick slabs or on hot, windy days when the surface is prone to drying out. (22).These statements show that the concretes have many disadvantages and the strength is low.

CHAPTER 3: METHODOLOGY

3.1 Research Methodology



Research on development of geopolymer based on fly ash as anti erosion coating

Experiment 1: Produce erosion profile under acidic environment for normal concretes

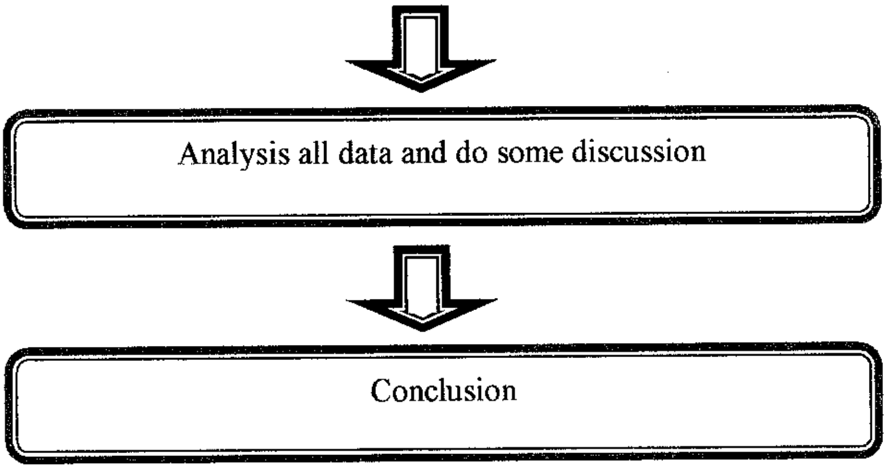
Experiment 2: Produce geopolymer using Fly ash and different concentration of NaOH

Experiment 3: Produce geopolymer using Fly Ash + 12M of NaOH + sodium silicate with different Si/Al ratio

Experiment 4: Produce geopolymer using Fly Ash + 12M of NaOH + sodium silicate with different solid/liquid ratio

Experiment 5: Produce erosion profile for concretes that coating with geopolymer with different fomulas

Test the samples using FESEM



3.2 FYPII Gantt chart and Milestones

Table 3.1: Timelines for FYP 2

No	Detail	May		June				July				August				September	
		1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Experiment 2	▨	▨	▨	▨												
2	Experiment 3					▨	▨	▨		▨							
3	Experiment 4			▨	▨	▨	▨										
4	Experiment 5				▨	▨	▨	▨									
5	Submission of progress report									●							
6	Test samples using FESEM									▨	▨	▨					
7	Pre-EDX												●				
8	Submission of draft report													●			
9	Submission of dissertation (soft bound)														●		
10	Submission of technical paper														●		
11	Oral presentation															●	
12	Submission of project dissertation (hard bound)																●

▨ Process ● Suggested Milestone

3.3 Experimental Sections

3.3.1 Raw Materials

These are the chemicals that will be use in this project:

i) Fly ash
ii) Sodium Hydroxide
iii) Sodium Silicate
iv) Acid Sulfuric
v) Concretes

3.3.2 Tools

These are the equipment or tools that will be use in this project:

i) FESEM/XRD
ii) Beakers
iii) Magnetic Bar
iv) Measuring Cylinders
v) Brush
vi) Moulds
vii) Water bath
viii) Mixer

3.3.3 Experiment 1: Produce Erosion Profile under Acidic Environment

1. Measured the initial weight of the concretes and labeled 1 and 2 to the concretes.
2. Prepared 3% acid sulfuric in 5L solution (see Appendix II).
3. Put 300 ml of acid sulfuric in two beakers.
4. Immersed the concrete 1 and 2 in the beakers.
5. Concrete 1 has been put at room temperature, 26°C.
6. Concrete 2 has been put at 35°C in the water bath.
7. After 1 week, brushed the concretes to remove the weakly adhering particles.
8. Dried the concretes in the oven at 100°C for 3 hours.
9. Measured the current weight of both concretes.
10. Repeated the step 3 to 9 for 1 month.
11. Produced erosion profile.

3.3.4 Experiment 2: Produce Geopolymer using Different Concentration of NaOH

1. 500ml of 6M, 10M and 12M of NaOH have been prepared (see Appendix II).
2. 1.155 kg of FA + 0.270 kg of 6M NaOH have been mixed in the mixer for 1.50 minutes.
3. Find the setting time.
4. Pour the sample into moulds. Prepared 6 samples.
5. 3 samples have been cured at 26°C and 3 samples have been cured at 60°C in the oven for 28 days.
6. After 7 days, compressive strength test has been conducted to 1 sample that cured at 26°C and 1 sample that cured at 60°C in the oven.
7. Repeat step 7 after 14 days and 28 days by using different samples.
8. 50g has been taken from each sample that used for compressive strength test after 28 days to characterize the samples using FESEM.
9. Repeated step 2 to 8 by change the concentration of NaOH to 10M & 12M
10. At the same time, produced normal concrete to compare the samples of geopolymer by mix 0.031kg of Portland cement + 0.013 kg of fly ash +

0.1155 kg of sand and 0.1444kg of coarse aggregate by using ratio of water/concrete is 0.5

11. Repeat step 4 to 8 for normal concretes
12. Compare the result of geopolymer samples with the normal concretes.

3.3.5 Experiment 3: Produce Geopolymer by using Different Si and Al Ratio by Adding Sodium Silicate

1. Prepared 1.155 kg of FA + 0.135 kg of 14M NaOH + 0.385 kg of Na_2SiO_3 for Si/Al ratio is 2.5:1 (see Appendix II).
2. Mixed all the chemicals using mixer for 1.50 minutes
3. Determined the setting time
4. Pour the sample into moulds. Prepared 6 samples.
5. Cured 3 samples at 26°C and cured 3 samples at 60°C in the oven for 28 days.
6. After 7 days, conducted compressive strength test to 1 sample that cured at 26°C and 1 sample that cured at 60°C in the oven.
7. Repeat step 6 after 14 days and 28 days by using different samples.
8. Taken 50g of each sample that used for compressive strength test after 28 days to characterize the samples using FESEM.
9. Repeated the step 1 to 8 by using 3.5:1 of Si/Al ratio.
10. Produced normal concrete to compare the samples of geopolymer by mix 0.031kg of Portland cement + 0.013 kg of fly ash + 0.1155 kg of sand and 0.1444kg of coarse aggregate by using ratio of water/concrete is 0.5
11. Repeated step 4 to step 8 for normal concretes.
12. Compared the result of geopolymer samples with the normal concretes.

3.3.6 Experiment 4: Produce Geopolymer by using Different Solid and Liquid Ratio by Adding Sodium Silicate

1. Prepared 1.155 kg of FA + 0.135 kg of 14M NaOH + 0.385 kg of Na_2SiO_3 for Solid/Liquid ratio is 2.5:1 (see Appendix II).
2. Mixed all the chemicals using mixer for 1.50 minutes
3. Determined the setting time
4. Pour the sample into moulds. Prepared 6 samples.
5. Cured 3 samples at 26°C and cured 3 samples at 60°C in the oven for 28 days.
6. After 7 days, conducted compressive strength test to 1 sample that cured at 26°C and 1 sample that cured at 60°C in the oven.
7. Repeat step 6 after 14 days and 28 days by using different samples.
8. Taken 50g of each sample that used for compressive strength test after 28 days to characterize the samples using FESEM.
9. Repeated the step 1 to 8 by using 3.5:1 of Si/Al ratio.
10. Produced normal concrete to compare the samples of geopolymer by mix 0.031kg of Portland cement + 0.013 kg of fly ash + 0.1155 kg of sand and 0.1444kg of coarse aggregate by using ratio of water/concrete is 0.5
11. Repeated step 4 to step 8 for normal concretes.
12. Compared the result of geopolymer samples with the normal concretes.

3.3.7 Experiment 5: Erosion Prevention Using Fly Ash Based Geopolymer Coating

1. Produced geopolymer by using fly ash and 12M of sodium hydroxide solution.
2. Dip the concretes into the geopolymer solution and repaired the coat using paste tool to make every side has same layer.

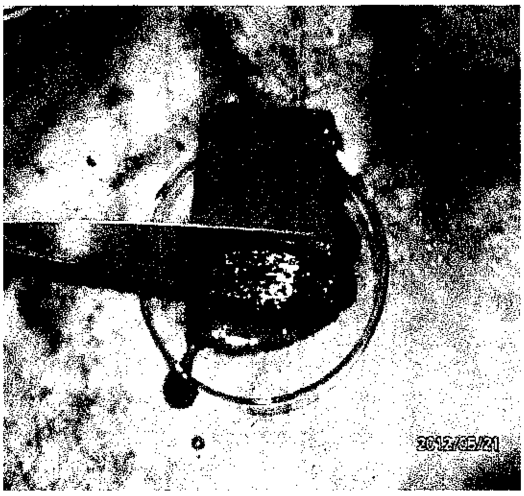


Figure 3.1: How to coat the concrete

3. Cured two concretes at 60°C and two concretes at 26°C for 24 hours.
4. Repeat step 3 by cured the concretes for 7 days
5. For the concretes that cured for 24 days, measured the initial weight of the concretes and label all the concretes with liquid paper the next day.
6. Prepared 3% acid sulfuric in 5L solution (see Appendix II).
7. Put 200 ml of acid sulfuric in 8 microwaves Tupperware.
8. Immersed the concretes in the Tupperware:
    - 1 concrete that cured at 60°C immersed in H_2SO_4 at 35°C in the water bath
    - 1 concrete that cured at 60°C immersed in H_2SO_4 at 26°C
    - 1 concrete that cured at 26°C immersed in H_2SO_4 at 35°C in the water bath
    - 1 concrete that cured at 26°C immersed in H_2SO_4 at 26°C
9. Repeat the step 5 to 8 for concrete that cured for 7 days. Conducted the experiment after 7 days.
10. After 1 week, brushed the concretes to remove the weakly adhering particles.
11. Dried the concretes in the oven at 100°C for 3 hours.
12. Measured the current weight of the concretes.

13. Repeated the step 10 to 12 for 1 month.
14. Produced erosion profile.
15. Repeat the step 1 to 14 by using fly ash, 12M of sodium hydroxide solution and sodium silicate solution with solid/liquid ratio are 2.5:1 and 3.5:1

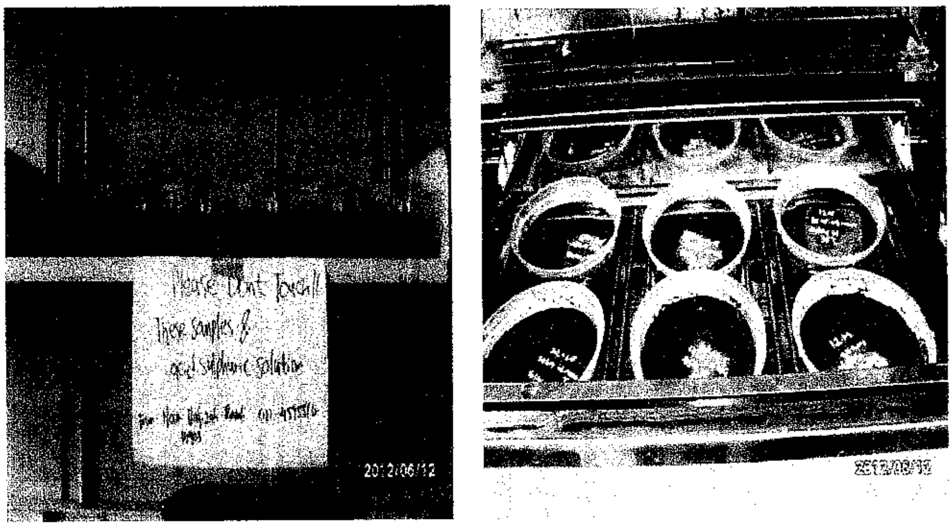


Figure 3.2: The concretes immersed in H_2SO_4 at 26°C and at 60°C

3.3.8 Characterized the Geopolymer samples using Field Emission Electron Microscope (FESEM)

The particle sizes of geopolymer samples before and after merged in acid sulfuric were analyzed by FESEM. This experiment conducted by laboratory technician. The samples will be scanned and X-ray using magnificent of 300X, 1.00 kX and 3.00 kX. The samples will be measured under EDX to determine the element of the samples.

Table 3.2: Geopolymer samples that have been characterized by FESEM

Samples	**Molarity of NaOH**	**Si:Al**	**Solid:Liquid**	**Cure Temperature**
FA+12M of NaOH	12M	1.81:1	4.3:1	60°C
FA+ 6M of NaOH	6M	1.81:1	4.3:1	26°C
3.5:1 of S/L	8.4M	2.2:1	3.5:1	60°C
2.5:1 of Si/Al	6.8M	3.5:1	1.1:1	26°C
FA+12M of NaOH and immersed in H_2SO_4 for 30 days	12M	1.81:1	4.3:1	60°C
FA+12M of NaOH and immersed in H_2SO_4 for 30 days	12M	1.81:1	4.3:1	26°C
3.5:1 of S/L and immersed in H_2SO_4 for 30 days	8.4 M	2.2:1	3.5:1	60°C
2.5:1 of S/L and immersed in H_2SO_4 for 30 days	8.4M	2.2:1	2.5:1	26°C

CHAPTER 4: RESULTS AND DISCUSSIONS

4.1 Effect of Acid on Normal Concrete

Normal concretes has been immersed in 3% of acid sulfuric that represent the worst reported condition in wastewater system to produce erosion profile for normal concretes. After 30 days immersed the concretes in acid sulfuric solution, the mass loss of the concretes increased rapidly week by week. The effect of acid on normal concrete can see in Appendix I.

Table 4.1: Mass loss of concretes in percentage

Temperature/ Week	**1**	**2**	**3**	**4**
26	0	6.236	9.423	14
35	0	7.23	12.553	18.23

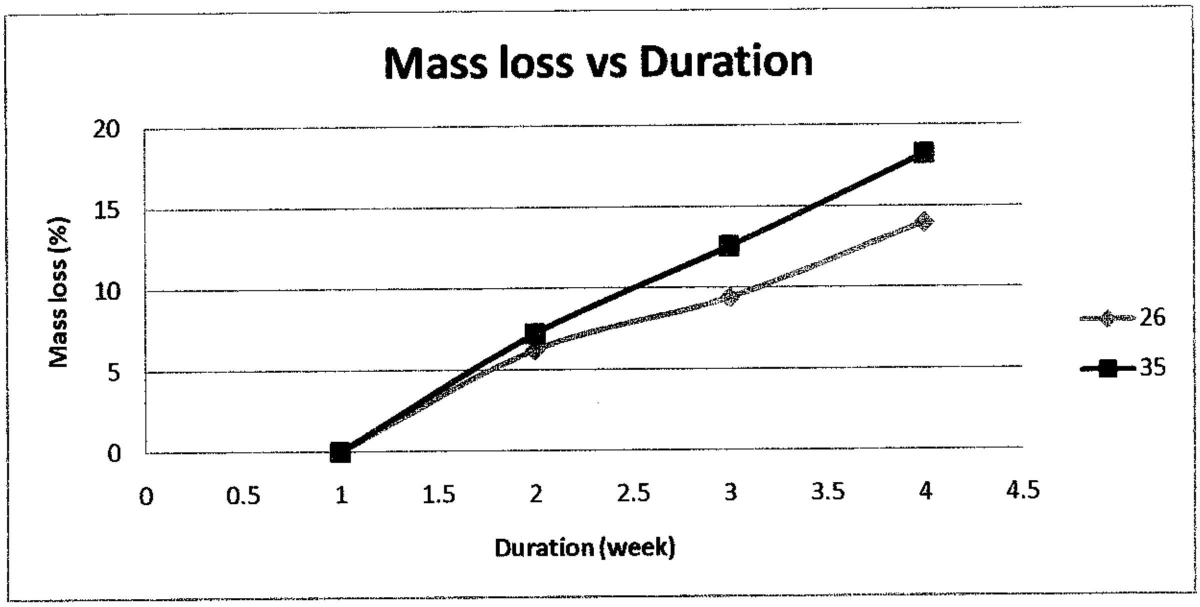


Figure 4.1: Graph of mass loss versus duration

Table 4.2: Compressive strength test result for normal concretes

Type of concrete	**Stress (Mpa)**
normal concrete	29.94
concrete that immersed in H2SO4 at 26°C for 1 month	15.22
concrete that immersed in H2SO4 at 35°C for 1 month	5.484

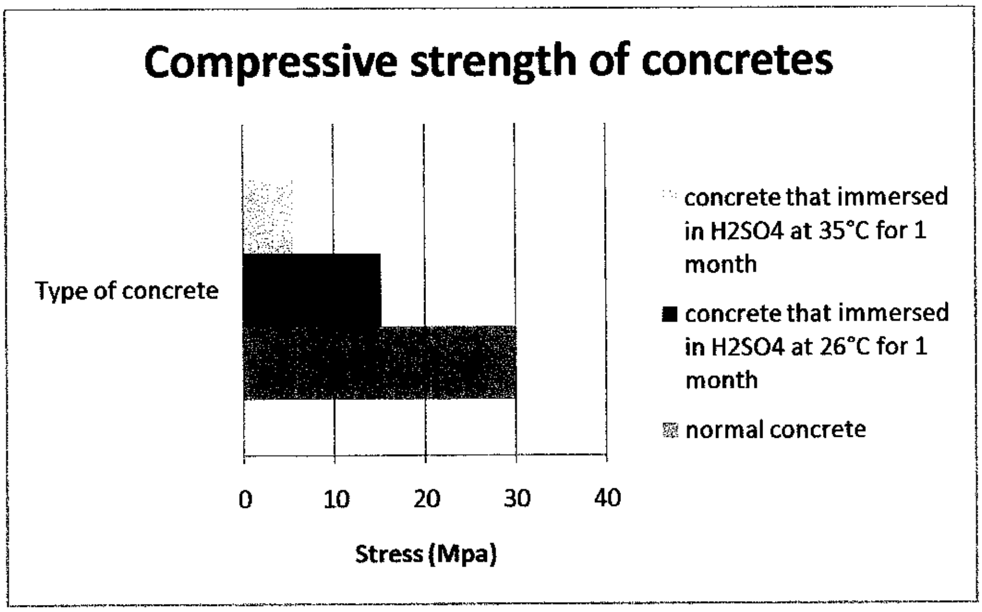


Figure 4.2: Bar chart of compressive strength of different condition of concretes

From the observation, the weakly adhering particles become more and more in every week when immersed them in 300ml of acid sulfuric solution. From the Figure 4.1 also showed that the concrete that immersed in acid solution at 35°C has higher mass loss compared to the concrete that immersed in acid solution at 26°C. This is because higher temperature can make the concretes more easily react to the acid sulfuric.

From the Figure 4.2, we can see that the concrete that immersed in acid sulfuric at 35°C is less strength that the concrete that immersed in acid sulfuric at 26°C and normal concrete. Thus, the temperature also affects the strength of the concretes.

In summary, it has been proven that concrete can easily erode and decrease the strength due to acidic environment. In summary, high temperature can make concrete erode and decrease strength faster than lower temperature.

4.2 Finding the Best Formula to Produce Fly Ash based Geopolymer as Anti Erosion Coating

The requirements to become as an effective anti erosion coating are:

- Good setting time
- High compressive strength
- High bond strength to the concrete
- High anti erosion

4.2.1 Effect of Concentration of Sodium Hydroxide to the Geopolymer Samples

In order to find the best formula to produce fly ash based geopolymer as anti erosion coating, the concentration of sodium hydroxide (NaOH) need to be analyze to find the best concentration of NaOH to mix with fly ash. The 6, 10 and 12 molarity of NaOH has been used in this study.

Table 4.3: Formula of mix the FA with different molarity of NaOH (refer to Appendix II)

Geopolymer samples	Si:Al	Molarity of NaOH	Solid:Liquid	Mass of NaOH
FA+6M of NaOH	1.81:1	6M	4.3:1	270g
FA+10M of NaOH	1.81:1	10M	4.3:1	270g
FA+12M of NaOH	1.81:1	12M	4.3:1	270g

Table 4.4: Setting time of different concentration of NaOH

Samples	**Setting time**
FA+ 6M of NaOH	300 minutes
FA + 10M of NaOH	150 minutes
FA + 12M of NaOH	50.50 minutes
Normal Concrete	180 minutes

Table 4.5: Compressive strength test for different concentration of NaOH

	6M of NaOH cure at 60°C	**6M of NaOH cure at 26°C**	**10M of NaOH cure at 60°C**	**10M of NaOH cure at 26°C**	**12M of NaOH cure at 60°C**	**12M of NaOH cure at 26°C**	**Normal concrete cure at 60°C**	**Normal concrete cure at 26°C**
Day	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)
7	24.56	11.77	43.37	8.991	53.53	15.23	33.45	28.23
14	16.89	14.18	51.2	19.98	56.82	24.5	32.65	29.72
28	34.75	27.65	55.6	33.19	74.61	37.5	35.4	30.11

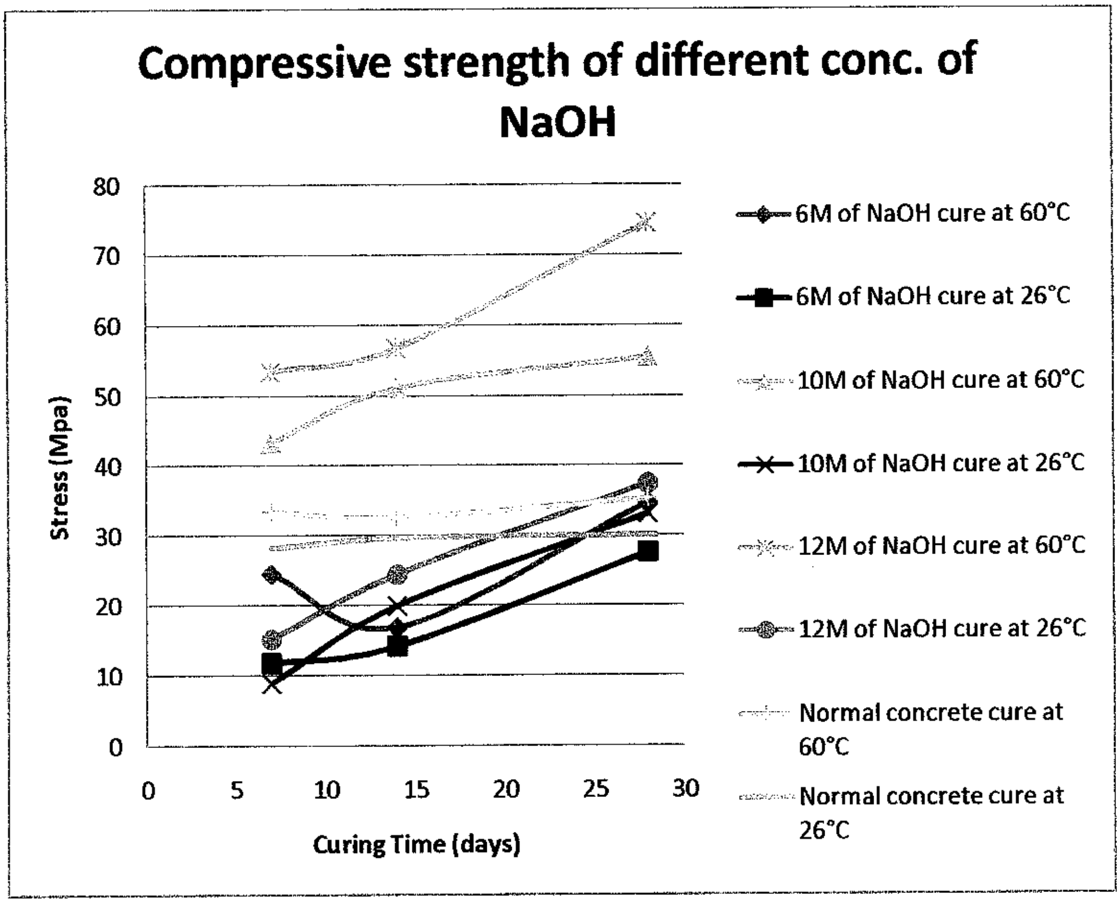


Figure 4.3: Graph of compressive strength test for different concentration of NaOH

From the Table 4.4, the samples that produced from FA+12M of NaOH has good setting time than FA mix with 10M and 6M of NaOH which is 50.50 minutes. Besides, it also better than normal concrete's setting time as the setting time of normal concrete is 180 minutes. Thus, higher concentration of NaOH will make the sample harden quickly and give better setting time compared to lower concentration of NaOH.

From the Table 4.5 and Figure 4.3, the samples that produce from FA+12M of NaOH has highest strength than FA that mix with 10M and 6M of NaOH which is the highest stress that can reach is 74.61 MPa. Besides, the samples that cure at 60°C have higher strength than samples that cure at 26°C, as example, the stress of the FA+10M of NaOH sample that cure at 60°C for 14 days is 51.20 MPa which is higher than the stress of the FA+10M of NaOH sample that cure at 26°C for 14 days which is only 19.98 MPa. Moreover, the samples that cure for 14 days have more strength than the samples that cure only 7 days, for example, the stress of the

FA+6M of NaOH sample that cure at 26°C for 14 days has 14.18MPa which is slightly higher than the stress of the FA+6M of NaOH sample that 26°C for 7 days which is 11.77MPa. On the other hand, from the observation, the strength of the normal concrete is almost constant although increase the curing time compare to the geopolymer samples which are keep increasing as the curing time increase. Thus, the higher concentration of NaOH can give more strength to the samples and when the samples cure at high temperature, the bonding of each molecules also become more strong time to time.

The best formula of geopolymer is FA+12M of NaOH that cure at 60°C for more than 7 days. This is because it has the best setting time and high strength. The strength of the samples will increase when increase the curing time. It also proved that the strength of geopolymer samples can be higher than normal concrete. In conclusion, the best concentration of the NaOH is 12M of NaOH.

4.2.2 Effect of Different of Silicate and Alumina ratio to the Geopolymer Samples

The Si/Al ratio also has been measured in order to find the best formula for anti erosion coating. The Si/Al ratios are changed by adding the sodium silicate with different amount. However, the sodium silicate that has been used in this experiment has 55.52% of water content. This fact make the concentration of NaOH decreased and the geopolymer need more time to harden because of molecules of water make the reaction become long.

Table 4.6: Formula of different Si/Al ratio (refer to Appendix II)

Geopolymer Samples	**Mass of Na_2SiO_3**	**Mass of NaOH**	**Si:Al**	**Molarity of NaOH**	**Solid:Liquid**
2.5:1 of Si:Al	385g	135g	2.5:1	9.8M	2.2:1
3.5:1 of Si:Al	943g	135g	3.5:1	6.8M	1.1:1

Table 4.7: Setting time of different Si/Al ratio and normal concrete

Sample	**Setting time**
Si:Al = 2.5:1	52 minutes
Si:Al = 3.5:1	665 minutes
Normal concrete	180 minutes

Table 4.8: Result of compressive strength of different Si/Al ratio

	Si/Al = 2.5/1 cure at 60°C	**Si/Al = 2.5/1 cure at 26°C**	**Si/Al = 3.5/1 cure at 60°C**	**Si/Al = 3.5/1 cure at 26°C**	**Normal concrete cure at 60°C**	**Normal concrete at 26°C**
Days	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)
7	55.13	26.23	Crack	16.16	33.45	28.23
14	58.23	45.92	Crack	24.08	32.65	29.72
28	61.76	48.5	Crack	28.9	35.4	30.11

The samples for 3.5:1 of Si/Al ratio that cured in 60°C are failed to conduct the compressive strength because all the samples crack in the middle of curing time.

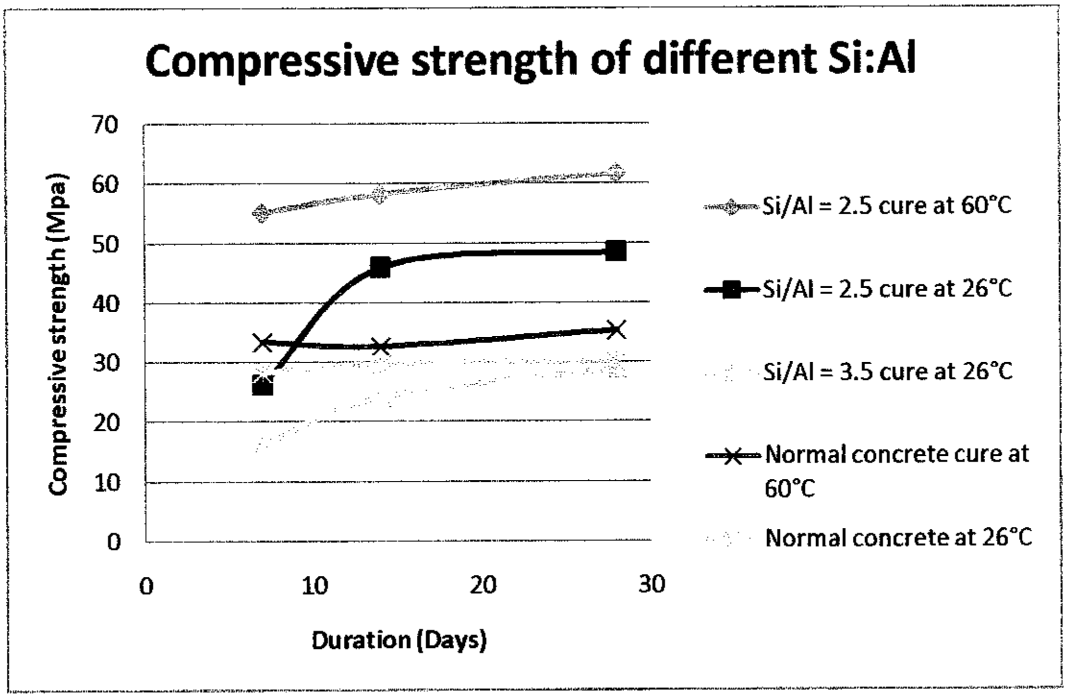


Figure 4.4: Graph of compressive strength of different Si/Al ratio

From the Table 4.7, the increasing of Si/Al ratio make the setting time is increased. As example, the setting time of 3.5:1 of Si/Al ratio is 665 minutes compared to previous experiment. The setting time is increasing because the water content in sodium silicate make the molarity of NaOH decreased and makes the samples hard to harden. However, the setting time of 2.5:1 of Si/Al still can categorized as a good setting time which is 52 minutes as this study used high concentration of NaOH that can help the samples to harden quickly.

From the Table 4.8 and Figure 4.4, 2.5:1 of Si/Al ratio has the highest strength; for example the stress for the sample that cure at 60°C for 14 days is 67.2 MPa compared to the stress for 3.5:1 of Si/Al ratio that cure at 26°C for 14 days is 24.08 MPa. For normal concrete, the strengths of the concretes are almost constant although the curing time increased. Therefore, the formula to produce fly ash based geopolymer using 3.5:1 of Si/Al ratio is not suitable to use to produce geopolymer as anti erosion coating because it has lowest strength.

Therefore, the less Si/Al ratio is more suitable to produce geopolymer based fly ash if adding the sodium silicate solution. This is because the water content which is 55.52% in sodium silicate affects the strength of the samples and increase the setting time as it decreases the molarity of sodium hydroxide. The best way to improve is change sodium silicate with silica powder or use the sodium silicate solution that has less water content.

4.2.3 Effect of Different Solid and Liquid ratio to the Geopolymer Samples

The objective of this study is to determine the best solid/liquid ratio. This experiment also used sodium silicate to produce the geopolymer samples.

Table 4.9: Formula of different Solid/Liquid ratio

Geopolymer samples	**Mass of Na_2SiO_3**	**Mass of NaOH**	**Si:Al**	**Molarity of NaOH**	**Solid:Liquid**
2.5:1 of S:L	218g	244g	2.2:1	8.4M	2.5:1
3.5:1 of S:L	218g	112g	2.2:1	8.4M	3.5:1

Table 4.10: Setting time for different Solid/Liquid ratio

Solid/Liquid Ratio	**Setting time (min)**
2.5: 1	57
3.5: 1	35
Normal concrete	180

Table 4.11: Result of compressive strength of different solid/liquid ratio

	S/L = 2.5/1 cure at 60°C	**S/L = 2.5/1 cure at 26°C**	**S/L = 3.5/1 cure at 60°C**	**S/L = 3.5/1 cure at 26°C**	**Normal concrete cure at 60°C**	**Normal concrete at 26°C**
Days	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)	Stress (Mpa)
7	49.07	35.83	81.02	35.92	33.45	28.23
14	62.02	41.1	82.13	41.19	32.65	29.72
28	84.5	83.29	84.66	57.26	35.4	30.11

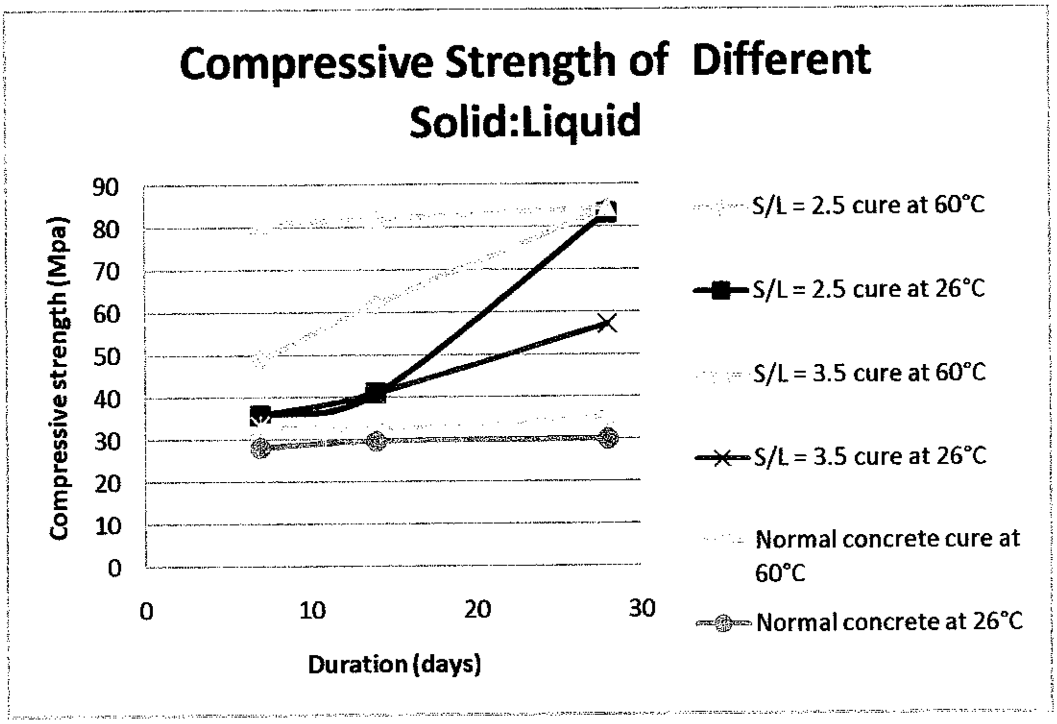


Figure 4.5: Graph of compressive strength of different solid/liquid ratio

From the Table 4.10, the setting time will decrease when the solid/liquid ratio is increased. The setting time for 3.5:1 of solid/liquid ratio is 35 minutes is lower than setting time for 2.5:1 of solid/liquid ratio which is 57 minutes. Decreasing the amount of liquid make the setting time decreased because the use of sodium silicate will be less. So, it will make the setting time become faster if the solid/liquid ratio is increased.

From the Table 4.11 and Figure 4.5, 3.5:1 of solid/liquid ratio has the highest strength which is cured at 60°C for 28 days, the stress is 84.66 MPa. From the observation, the strength of samples for geopolymer will increase as the curing time increase. On the other side, for normal concrete, the strength will remain the same although increased the curing time.

In summary, increasing the solid/liquid ratio will make the strength of the sample to increase and decrease the setting time. However, it was found that the solid/liquid ratio more than 3.5:1 the geopolymer based fly ash is not suitable to be used as anti erosion coating because the coating is easily crack and the strength reduces. Thus, the best solid/liquid ratio is 3.5:1 and it also has been proved that the strength of geopolymer can be higher than normal concrete. Although 2.5:1 of solid/liquid ratio has the lower strength than

3.5:1, it still can be used to produce geopolymer coating because the setting time still reasonable to coat the concrete.

From overall observation, the best concentration of sodium hydroxide is 12M, the best Si/Al ratio is in the range 1.81:1 - 2.5:1 and the best solid/liquid ratio is in the range 3.5:1 – 4.3:1 as they have the good setting time and high compressive strength.

4.3 Erosion Prevention using Fly Ash based Geopolymer Coating

After finding the best formula to produce the fly ash based geopolymer coating, all the concretes that already coated with geopolymer will be immersed in acid sulfuric solution like experiment 1. The erosion profile for normal concrete will be used as reference to determine to what extent geopolymer coating can reduce the erosion effect to the concrete. There are 3 formulas has been choose which are FA+12M of NaOH, 3.5:1 of Solid/Liquid ratio and 2.5:1 of Solid/Liquid ratio to conduct the erosion test to the geopolymer coatings.

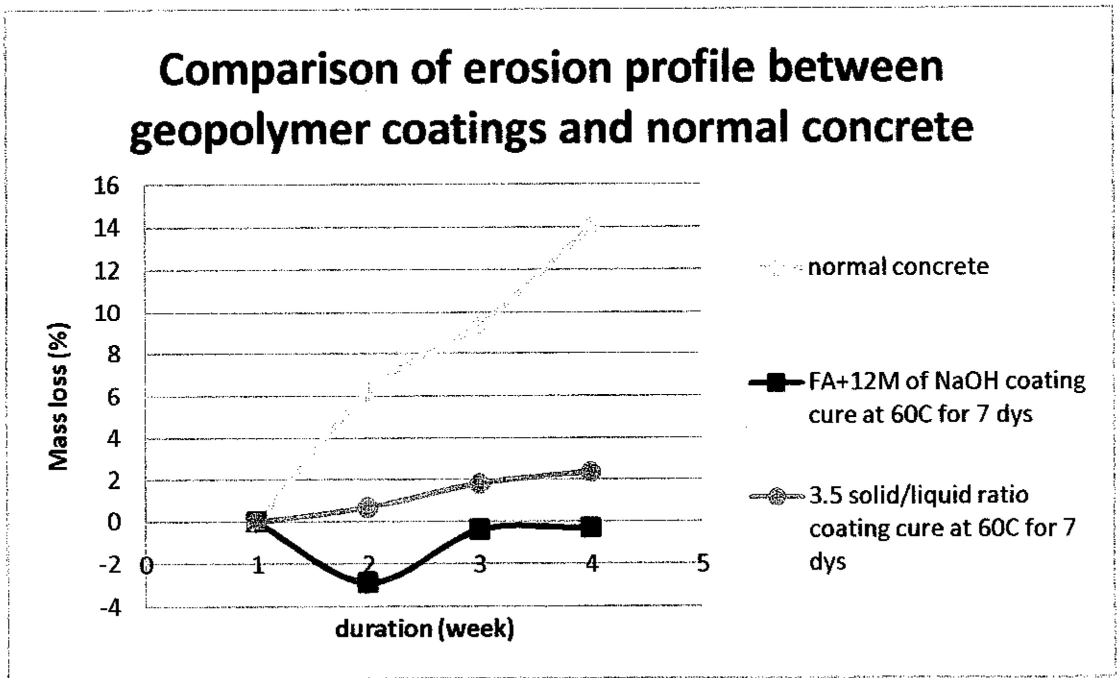


Figure 4.6: Graph of erosion profile between geopolymer coatings and normal concrete

In summary, **the geopolymer based fly ash as anti erosion coatings are succesfully reduce the erosion effect to the concretes.** The best formula to produce geopolymer based fly ash as effective anti erosion coating are **FA+ 12M of NaOH that cured at 60°C for 7 days** and **3.5 of solid/liquid ratio that cured at 60°C for 7 days.** However, the thickness of the coating should have been improves as it also effect the erosion profile.

4.4 Characterizations of the Geopolymer Samples

4.4.1 Field Emission Scanning Electron Microscope (FESEM)

FESEM analysis was carried out to analyze the morphology and determine the elements in the samples. The geopolymer samples before and after immersing in acid sulfuric solution for 30 days were analyzed and determined the morphology of each samples. The structures of the samples observed by magnificent of 300X, 1.00 KX and 3.00 KX and the determination of elements in the samples are using EDX with the length of 50μm.

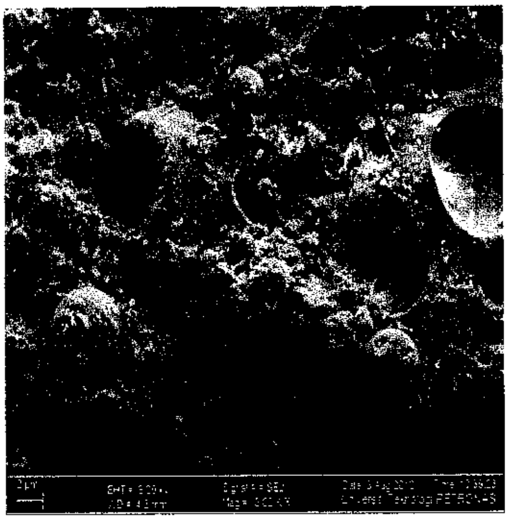


Figure 4.7: Morphology of FA+12M of NaOH with 4.3:1 of S/L, 1.81/1 of Si/Al and 50.50 mins of setting time that cured at 60°C.

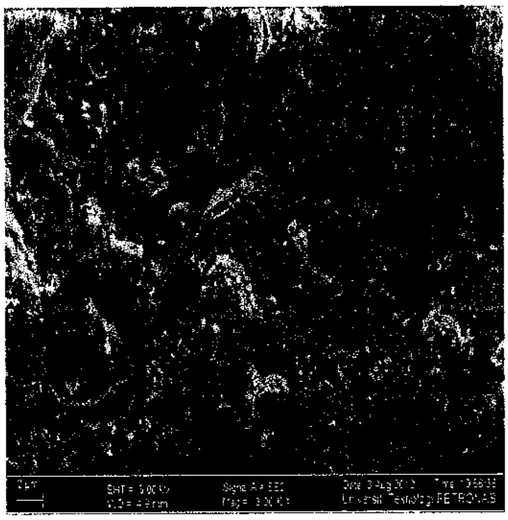


Figure 4.8: Morphology of FA+6M of NaOH with 4.3:1 of S/L, 1.81/1 of Si/Al and 300 mins of setting time that cured at 26°C.

From Figure 4.7, the morphology of the samples show spherical cenosphere of various sizes which can be related to the structure of fly ash. The figure also show that the structure very clearly as compared to Figure 4.8 which proved that the sample of FA+12M of sodium hydroxide has higher strength than the sample of FA+6M of NaOH. Figure 4.8 also show that the structures of the samples cracked after curing. This is indicates that 6M of NaOH is not suitable to use to produce geopolymer as it has less strength and very easy to crack. It is clearly showed that sample with low concentration of NaOH (Figure 4.8) contained more unreacted component as compared to Figure 4.7.

Figure 4.9: Morphology of FA+12M of NaOH+Na_2SiO_3 with 3.5:1 of S/L ratio, 2.2:1 of Si/Al and 35 mins of setting time that cured at 60°C.

Figure 4.10: Morphology of FA+12M of NaOH+Na_2SiO_3 with 1.1:1 of S/L ratio, 3.5:1 of Si/Al and 665 mins of setting time that cured at 26°C.

Figure 4.9 shows that sample of 3.5:1 of solid/liquid ratio has very good strength as the structural of the samples are very clear till the spherical structure of fly ash can be seem like 3D. Different with the Figure 4.10, it showed the sample has been cracked very badly which mean showed that 3.5:1 of Si/Al ratio is not suitable formula to produce fly ash based geopolymer.

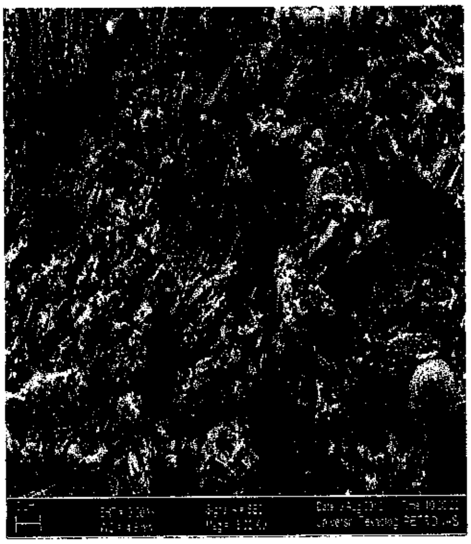


Figure 4.11: Morphology of FA+12M of NaOH that cured at 60°C and immersed in H_2SO_4 at 26°C for 30 days

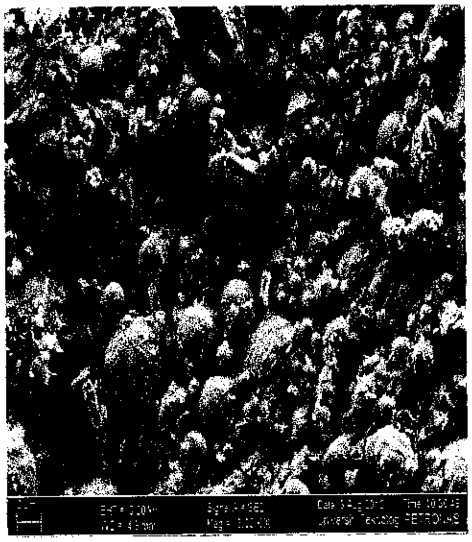


Figure 4.12: Morphology of FA+12M of NaOH that cured at 26°C and immersed in H_2SO_4 at 26°C for 30 days

Figure 4.13: Morphology of 3.5:1 of solid/liquid ratio that cure at 60°C and immersed in H_2SO_4 at 26°C for 30 days.

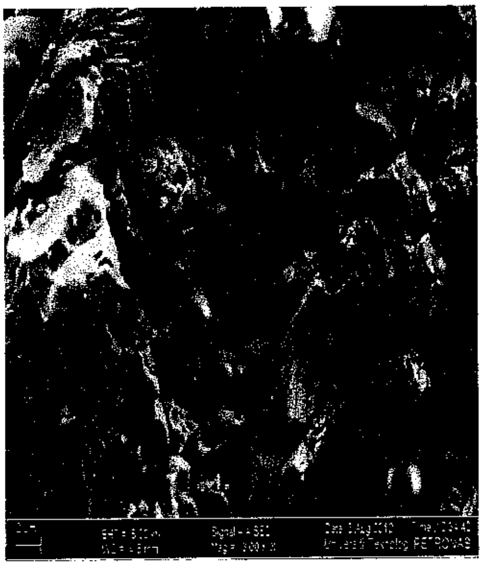


Figure 4.14: Morphology of 2.5:1 of solid/liquid ratio that cure at 26°C and immersed in H_2SO_4 at 26°C for 30 days.

Figure 4.11, 4.12, 4.13 and Figure 4.14 show the structures of the geopolymer samples after immersing them in acid sulfuric solution for 30 days. The morphologies of the samples show that the structural still can be seem clearly and still have a good strength between each molecule although acid sulfuric make the structure break and become compact structure and radiating acicular structure. The high strength of the geopolymer makes the sulfur molecules hard to break the geopolymer molecules. Thus, we can conclude that fly ash based geopolymer can reduce the erosion effect to the concrete as the strength between molecules in the geopolymer have a very good strength to defend them from harm because of acidic environment.

Table 4.12: Elements in geopolymer samples

	O (%)	**Na (%)**	**Mg (%)**	**Al (%)**	**Si (%)**	**K (%)**	**Ca (%)**	**Fe (%)**
12M	52.48	4.22	1.08	23.46	19.21	1.27	3.49	3.92
6M	50.88	5.3	2.22	9.24	19.24	1.49	6.34	6.54
S/L 3.5	48.29	6.45	1.55	8.39	19.39	1.71	5.75	8.12
Si/Al 3.5	51.81	9.84	1.8	7.49	21.22	1.47	5.06	6.34

The codes of the geopolymer samples:

- 12M = FA+12M of NaOH that cured at 60°C for 28 days sample
- 6M = FA+12M of NaOH that cure at 26°C for 7 days sample
- S/L 3.5 = 3.5:1 of solid/liquid ratio that cured at 60°C for 28 days sample
- Si/Al 3.5 = 3.5 :1 of Si/Al ratio that cured at 26°C for 7 days sample

The elements in each chemical:

- ➢ Fly ash elements: Fe, Si, Al, K, Mg and Ca
- ➢ Sodium hydroxide elements: Na and O
- ➢ Sodium Silicate: Na, Si and O

Table 4.13: Elements in geopolymer samples that have been merged in acid sulfuric solution

	O (%)	**Na (%)**	**Mg (%)**	**Al (%)**	**Si (%)**	**K (%)**	**Ca (%)**	**Fe (%)**	**S (%)**
12MA	54.36	0	0.63	3.67	15.88	0.6	11.65	4.2	9.02
12MB	54.43	0	1.02	7.93	20.73	1.23	4.54	7.06	3.05
S/L 3.5	56.36	0	0.59	0	16.87	0.74	6.55	2.22	6.55
S/L 2.5	54.32	1.73	2.05	7.86	20.85	0.97	8.36	6.22	0

The codes of the geopolymer samples:

- 12MA = FA+12M of NAOH that cured at 60°C for 7 days and merged in H_2SO_4 at 26°C sample
- 12MB = FA+12M of NAOH that cured at 26°C for 24 hours and merged in H_2SO_4 at 26°C sample
- S/L 3.5 = 3.5:1 of solid/liquid ratio that cured at 60°C for 7 days and merged in H_2SO_4 at 26°C sample
- S/L 2.5 = 2.5:1 of solid/liquid ratio that cured at 26°C for 24 hours and merged in H_2SO_4 at 26°C sample

From the Table 4.13, the element of Sulfur has been detected in all the samples because all of the samples were merged in acid sulfuric solution for 1 month. Sulfur elements are from the acid sulfuric solution. From the Table 4.13 also can see that the elements of sodium, Na cannot be detected from the samples and cannot be explained at the moment.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This project has been proved that the geopolymer has potential to improve the global issue which is erosion effect to the concretes especially in the marine environment, sewage and wastewater chemical treatment system. The formula of FA+12M of sodium hydroxide and 3.5:1 of solid/liquid ratio to produce fly ash based geopolymer already proved themselves that they are the best formula to produce fly ash based geopolymer for effective anti erosion coating during the erosion evaluation under acidic environment as they successfully reduces the effect of the erosion to the concretes. Besides, the characterization of the geopolymer samples using compressive strength test and FESEM also confidently show that FA+12M of NaOH and 3.5:1 of solid/liquid ratio that cured at 60°C for 28 days has a very high compressive strength and high bond strength between each component. In conclusion, after finishing the study of the development of fly ash based geopolymer as anti erosion coating, FA+12M of NaOH and 3.5:1 of solid/liquid ratio that cured at 60°C for 28 days has the best setting time, high compressive strength, high strength bond to the concretes and high anti erosion that make them the best formula to produce geopolymer as effective anti erosion coating.

5.2 Recommendation

There are some elements and parts that need to be improve. One of the main recommendations is the thickness of the coatings. In order to make the concretes can handle erosion problem effectively, the thickness of the geopolymer coatings to the concretes need to be increase. Besides, the compressive strength test sometimes gave the results with error, so to reduce the error; we should use 3 samples for one result so that we can take the average of the result. The setting time also need to conduct professionally by using the integrated machine so that we can determine the gel time and the viscosity profiles of the samples. One of the suitable machines for this study is rheometer.

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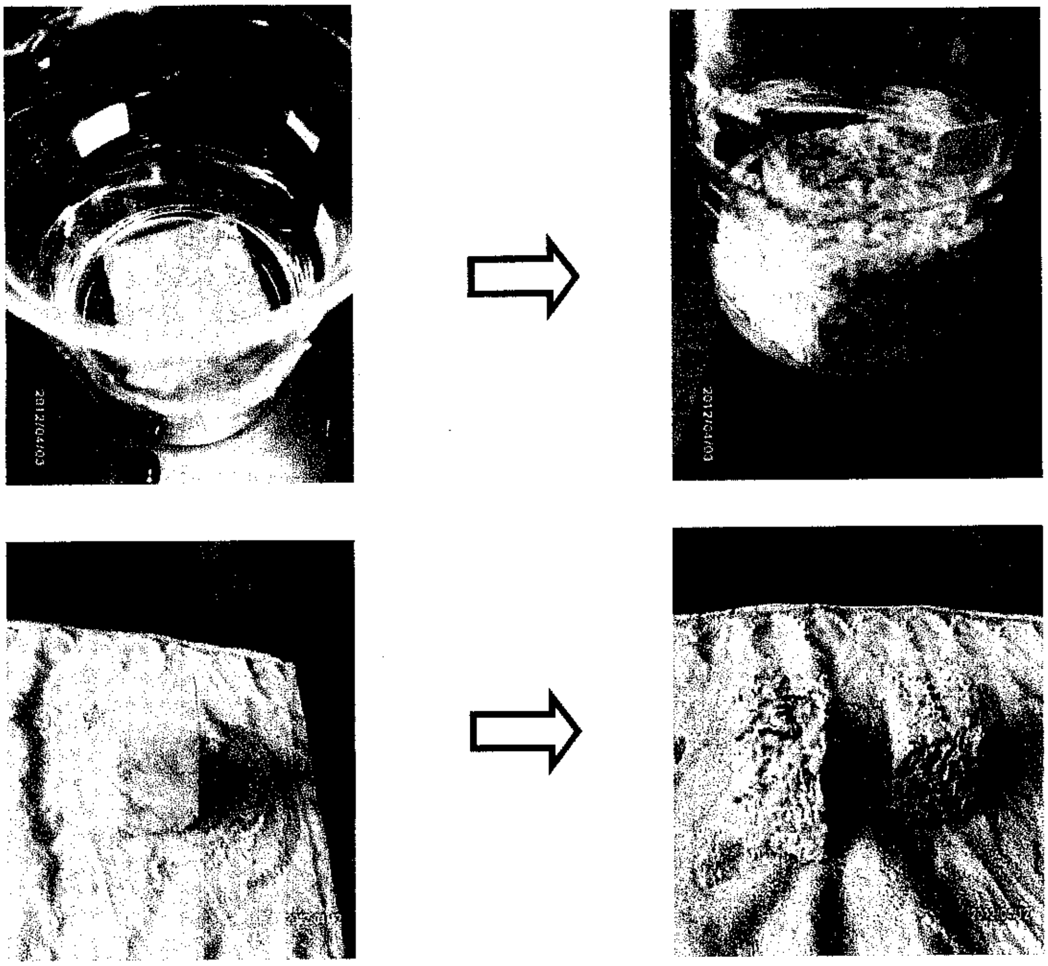
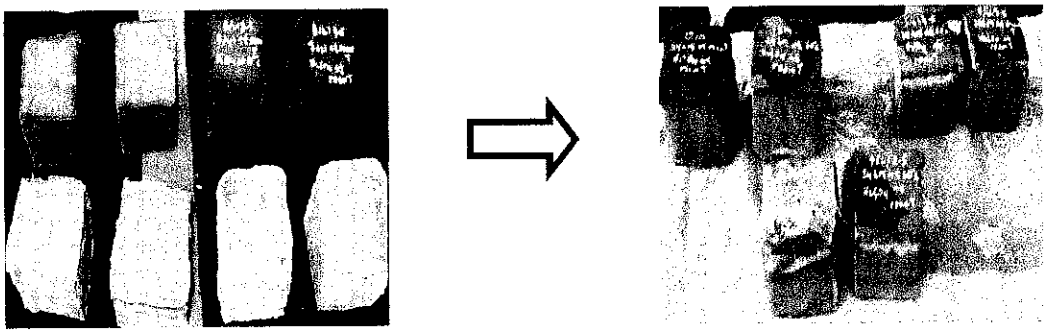
APPENDIX I: Effect of Acid Environment to the Concretes

i) Erosion test to the normal concrete



Concretes condition before and after test at acidic environment

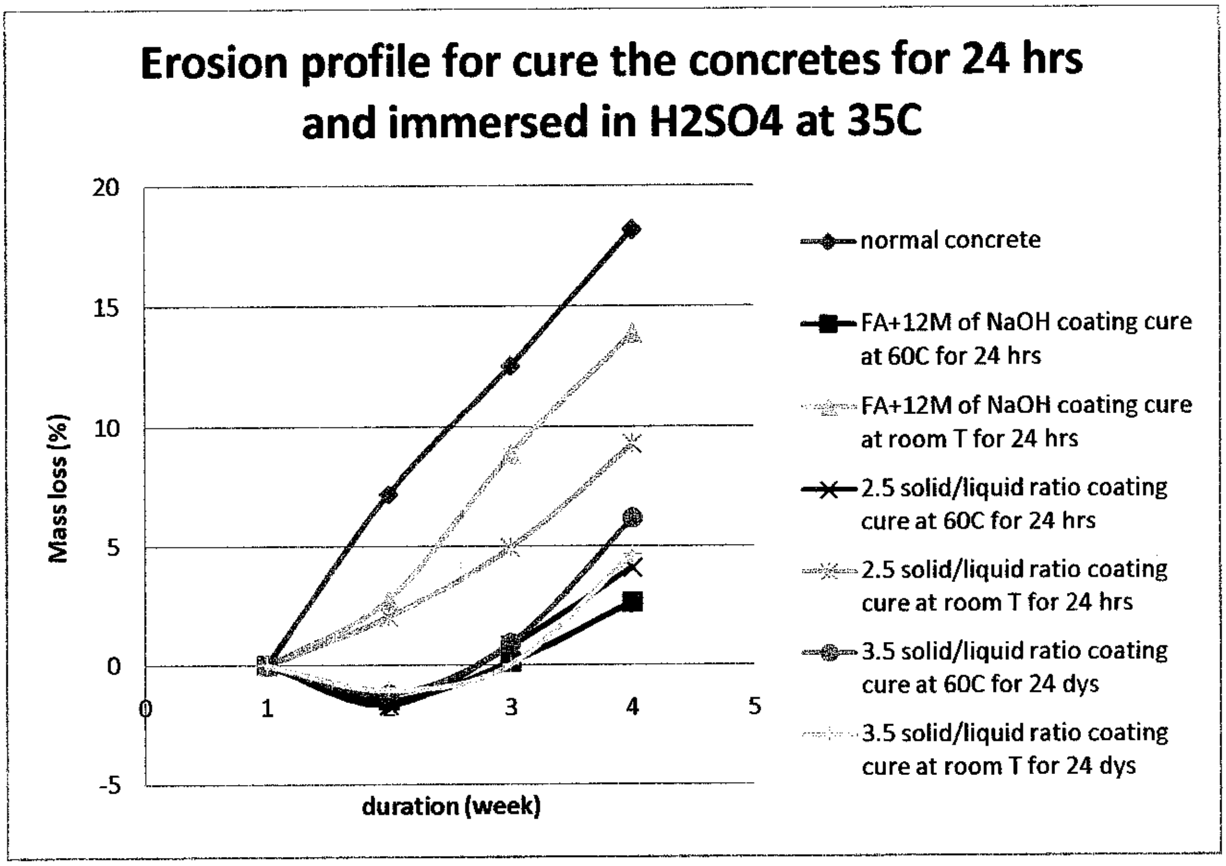
ii) Erosion test to the concretes that coating with fly ash based geopolymer



Concretes the coating with geopolymer condition before and after test in acidic environment

Table of mass loss of concretes that cure for 24 hrs and immersed in H_2SO_4 at 35°C

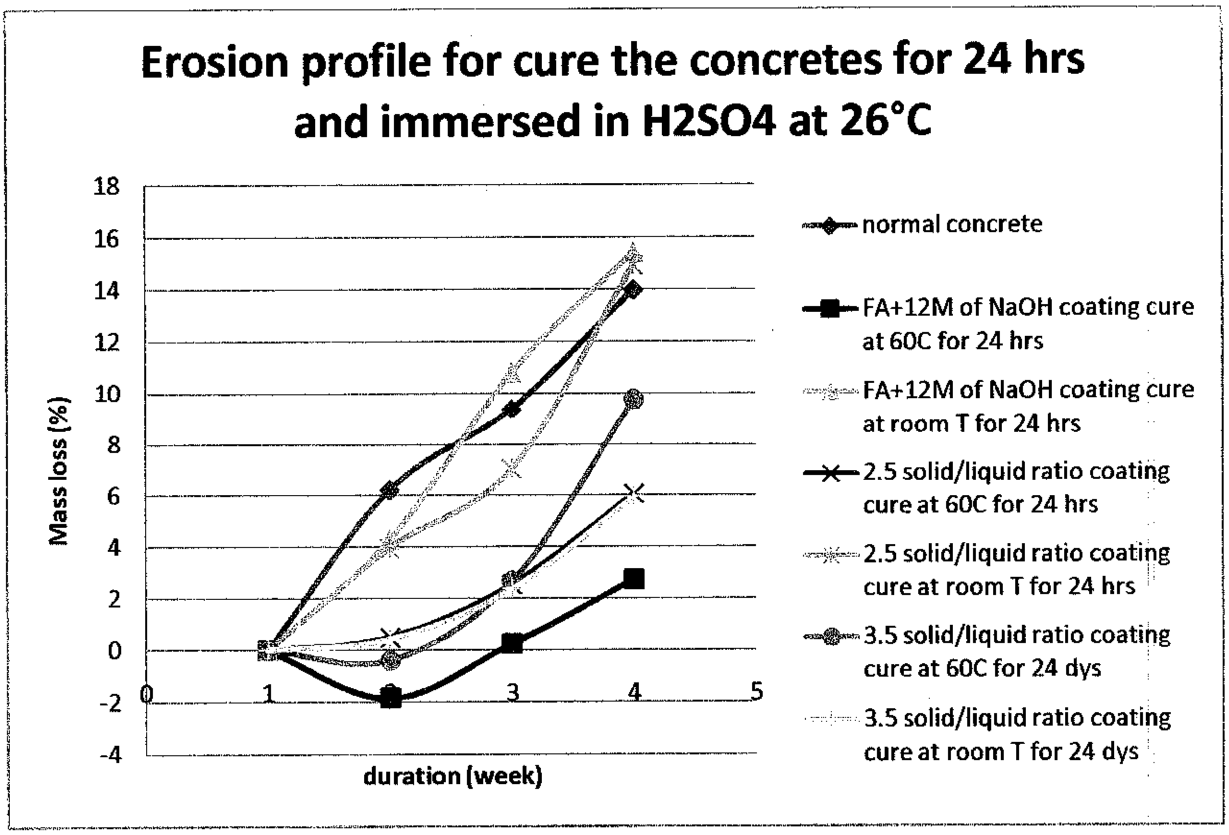
Type of concrete / Week	1	2	3	4
normal concrete	0	7.23	12.553	18.23
FA+12M of NaOH coating cure at 60C for 24 hrs	0	-1.46	0.13	2.65
FA+12M of NaOH coating cure at room T for 24 hrs	0	2.79	8.89	13.9
2.5 solid/liquid ratio coating cure at 60C for 24 hrs	0	-1.67	0.83	4.12
2.5 solid/liquid ratio coating cure at room T for 24 hrs	0	2.05	4.96	9.3
3.5 solid/liquid ratio coating cure at 60C for 24 dys	0	-1.22	0.92	6.18
3.5 solid/liquid ratio coating cure at room T for 24 dys	0	-1.06	0.03	4.7



Graph of erosion profile for cure the concretes for 24 hrs and immersed in H_2SO_4 at 35°C

Table of mass loss of concretes that cure for 24 hrs and immersed in H_2SO_4 at 26°C

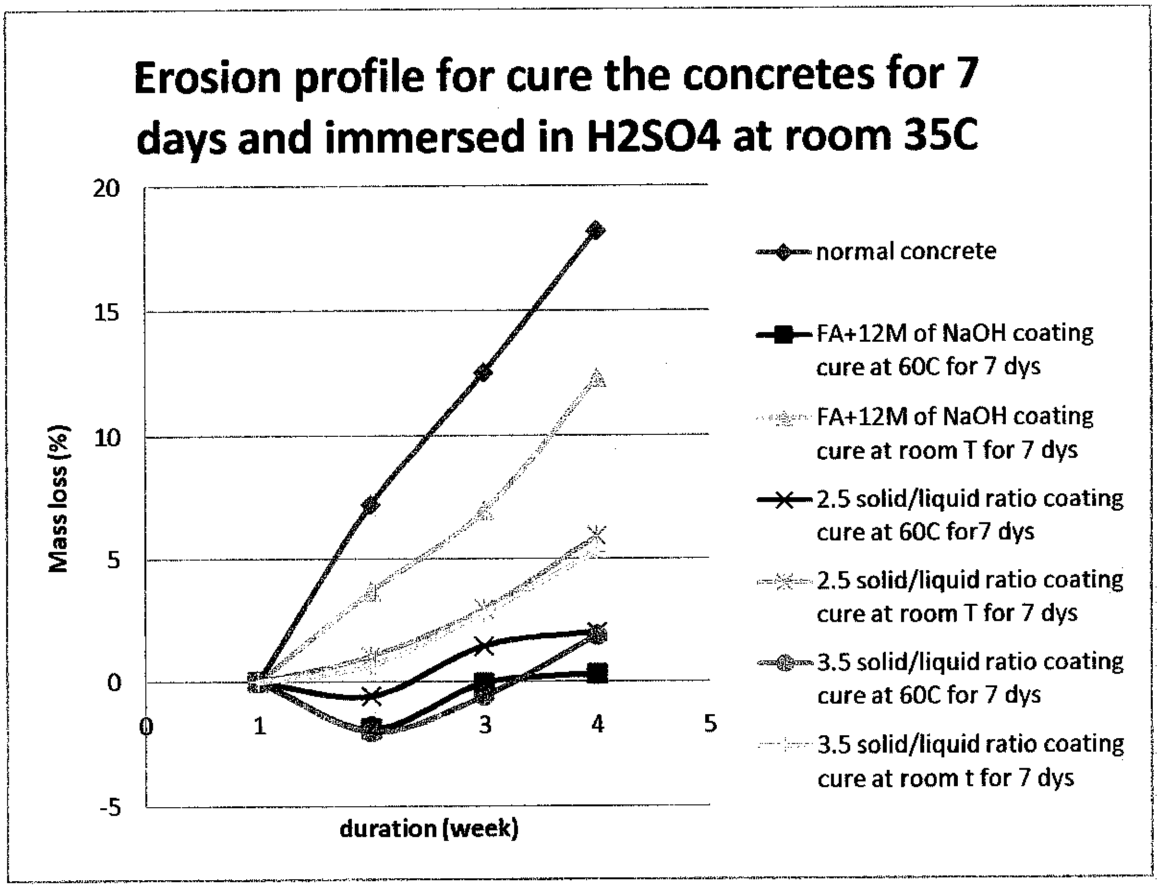
Type of concrete	1	2	3	4
normal concrete	0	6.24	9.42	14
FA+12M of NaOH coating cure at 60C for 24 hrs	0	-1.84	0.25	2.74
FA+12M of NaOH coating cure at room T for 24 hrs	0	4.3	10.81	15.44
2.5 solid/liquid ratio coating cure at 60C for 24 hrs	0	0.51	2.56	6.09
2.5 solid/liquid ratio coating cure at room T for 24 hrs	0	3.98	7.06	14.96
3.5 solid/liquid ratio coating cure at 60C for 24 hrs	0	-0.38	2.69	9.78
3.5 solid/liquid ratio coating cure at room T for 24 hrs	0	0.43	2.38	5.97



Graph of erosion profile for cure the concretes for 24 hrs and immersed in H_2SO_4 at 26°C.

Table of mass loss of concretes that cure for 7 days and immersed in H_2SO_4 at 35°C

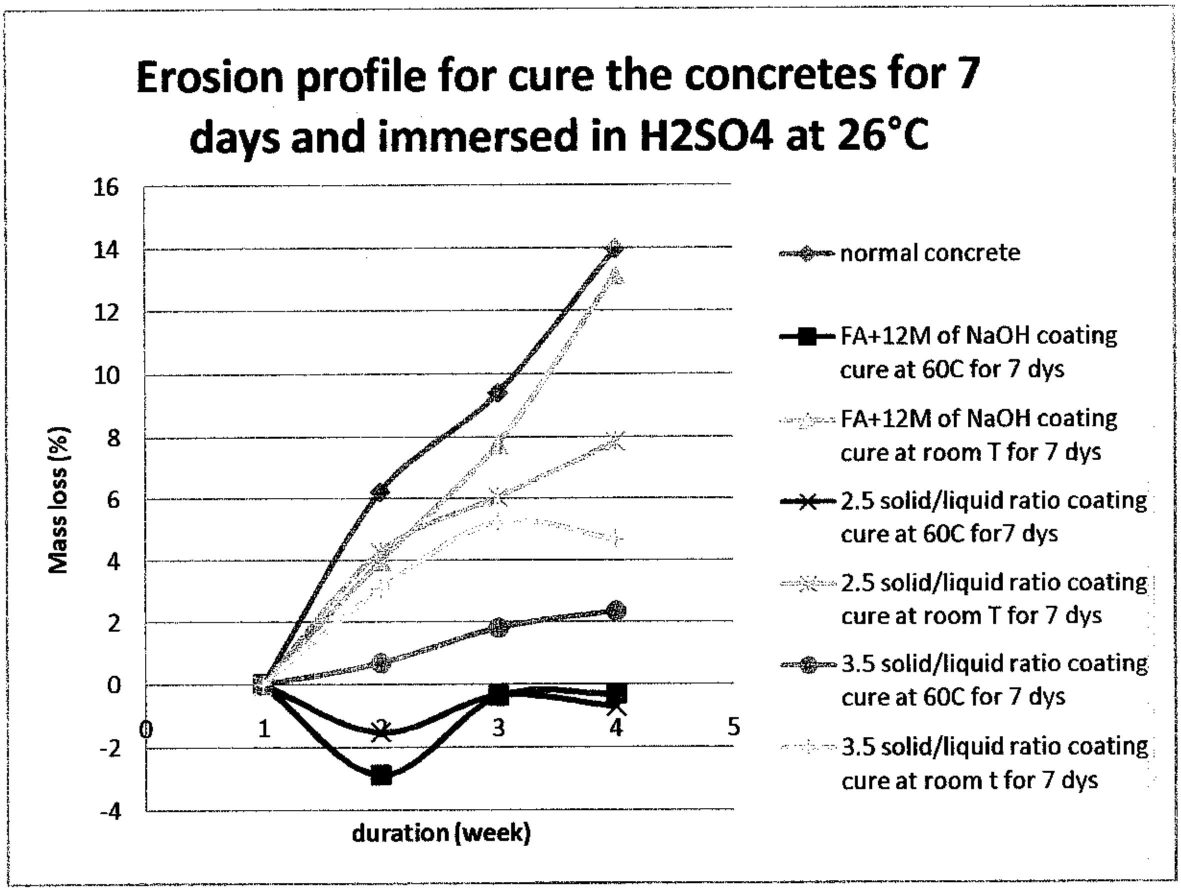
Type of concrete	1	2	3	4
normal concrete	0	7.23	12.55	18.23
FA+12M of NaOH coating cure at 60C for 7 days	0	-1.89	-0.06	0.3
FA+12M of NaOH coating cure at room T for 7 days	0	3.7	6.96	12.33
2.5 solid/liquid ratio coating cure at 60C for7 days	0	-0.57	1.46	2.03
2.5 solid/liquid ratio coating cure at room T for 7 days	0	1.06	2.99	5.93
3.5 solid/liquid ratio coating cure at 60C for 7 days	0	-2	-0.58	1.9
3.5 solid/liquid ratio coating cure at room t for 7 days	0	0.68	2.8	5.28



Graph of erosion profile for cure the concretes for 7 days and immersed in H_2SO_4 at 35°C

Table of mass loss of concretes that cure for 7 days and immersed in H_2SO_4 at 26°C

Type of concrete	1	2	3	4
normal concrete	0	6.236	9.42	14
FA+12M of NaOH coating cure at 60C for 7 days	0	-2.86	-0.38	-0.31
FA+12M of NaOH coating cure at room T for 7 days	0	4	7.8	13.17
2.5 solid/liquid ratio coating cure at 60C for7 days	0	-1.51	-0.35	-0.71
2.5 solid/liquid ratio coating cure at room T for 7 days	0	4.3	6.06	7.87
3.5 solid/liquid ratio coating cure at 60C for 7 days	0	0.69	1.82	2.37
3.5 solid/liquid ratio coating cure at room t for 7 days	0	3.09	5.26	4.69



Graph of erosion profile for cure the concretes for 7 days and immersed in H_2SO_4 at 26°C

APPENDIX II: Sample of Calculation of All Experiments

i) Calculation to Dilute 3% acid sulfuric in 5L solution.

3% of H_2SO_4 + 97% of H_2O in 5L solution

$$H_2SO_4 = \frac{3\ x\ 5000ml}{100\%} = 150ml$$

$$H_2O = \frac{97\ x\ 5000ml}{100\%} = 4850ml$$

ii) Calculation of Mass Loss

$$\%\ of\ mass\ loss = \frac{initial\ mass - current\ mass}{initial\ mass} x100\%$$

iii) Calculation to Dilute of 6M, 10M and 12M of NaOH in 500ml of H_2O.

$$Molarity = \frac{moles\ of\ solute}{liters\ of\ solution}$$

For 1L of 6M,

$$\frac{(weight\ of\ NaOH\ pellet), y}{MW\ NaOH} x\ 1L = 6M$$

$$y = 240\ g\ of\ NaOH\ pellet$$

For 0.5 L of 6M,

$$y = 240\ g\ of\ NaOH\ pellet/2 = 120g$$

iv) Calculation to Find Si/Al ratio in pure Fly Ash

$SiO_2 = \frac{43.34\%+42.69\%+43.73\%}{3} = 43\%$ $Al_2O_3 = \frac{20.77\%+20.81\%+20.18\%}{3} = 21\%$

Assume:

100g of FA = 43g of SiO_2 + 21g of Al_2O_3

1mol SiO_2 = 28.0855g + 32g = 60g

Thus, in 60g of SiO_2 has 28.0855g of Si

$$60g\ of\ SiO_2\ \equiv 28.0855g\ of\ Si$$

$$43g\ of\ SiO_2\ \equiv \frac{43\ x\ 28.0855}{60} = 20.13g\ of\ Si$$

1mol of Al_2O_3 = (26.98 x 2)g + 48g = 101.96g

Thus, in 101.96g of Al_2O_3 has 54g of Al

$$101.96g\ of\ Al_2O_3\ \equiv 54\ g\ of\ Al$$

$$21g\ of\ Al_2O_3\ \equiv \frac{21\ x\ 54}{101.96} = 11.12g\ of\ Al$$

Thus, 100g of FA = 20.13g of Si + 11.12g of Al

$$100g\ of\ FA\ \equiv 20.13g\ of\ Si$$

$$1155g\ of\ FA\ \equiv \frac{20.13\ x\ 1155}{100} = 232.50g\ of\ Si$$

$$100g\ of\ FA\ \equiv 11.12g\ of\ Al$$

$$1155g\ of\ FA\ \equiv \frac{11.12\ x\ 1155}{100} = 128.44g\ of\ Al$$

$$\frac{Si}{Al} = \frac{232.50}{128.44} = 1.81$$

Si/Al = 1.81:1

v) Calculation of mass of Sodium Silicate

1mol of Na_2SiO_3 = (23 x 2)g + 28.0855g + 48g = 122.09g

Thus, 122.09g of Na_2SiO_3 has 28.0855g of Si

$$100g\ of\ FA\ \equiv 20.13g\ of\ Si$$

$$1155g\ of\ FA\ \equiv \frac{20.13\ x\ 1155}{100} = 232.5g\ of\ Si$$

$$100g\ of\ FA\ \equiv 11.12g\ of\ Al$$

$$1155g\ of\ FA \equiv \frac{11.12\ x\ 1155}{100} = 128.44g\ of\ Al$$

1155g of FA + 122.09 of Na_2SiO_3 = (232.5g + 28.0855g) of Si and 128.44g of Al

Si/Al ratio = 2.03:1

$$\frac{232.5 + x}{128.44} = 2.5$$

x= 88.6g of Si

$$28.0855g\ of\ Si \equiv 122.09g\ of\ \mathrm{Na_2SiO_3}$$

$$88.6g\ of\ Si \equiv \frac{122.09\ x\ 88.6}{28.0855} = 385.15g\ of\ \mathrm{Na_2SiO_3}$$

vi) Calculation to Find the Molarity of Sodium Hydroxide and Solid/Liquid ratio after Adding Sodium Silicate

Si/Al ratio = 2.5:1

In the sodium silicate, it has:

- 14.73% of Na_2O
- 29.75% of SiO_2
- 55.52% of H_2O

$$0.5552\ x\ 0.385kg\ of\ \mathrm{Na_2SiO_3} = 0.214\mathrm{kg\ of\ H_2O}$$

$$\rho = 1000\ kg/m^3$$

$$0.214\ kg\ x\ \frac{m^3}{1000\ kg} x \frac{1000L}{m^3} = 0.214\ L = 214\ \mathrm{ml}$$

H_2O in 14M of NaOH + H2O in $0.385kg\ of$ Na_2SiO_3:

$$500ml + 214ml = 714\ ml$$

$$M_1V_1 = M_2V_2$$

$$14\ x\ 500 = 714\ M_2$$

$$\boldsymbol{M_2 = 9.8M\ of\ NaOH}$$

Calculate the solid/liquid ratio:

Si/Al ratio = 2.5:1

Solid = 1.155 kg of FA

Liquid = 0.135 kg of NaOH + 0.385 kg of Na_2SiO_3 = 0.520 kg

$$\frac{Solid}{liquid} = \frac{1.155}{0.520} = 2.22$$

Solid/Liquid ratio=2.22:1

vii) Calculation of mass of sodium hydroxide and sodium silicate for different solid/liquid ratio

Make the Si/Al ratio constant first. For experiment 3, the Si/Al ratio 2.2:1 is used.

For Solid/Liquid ratio = 2.5:1

1155g of FA + 122.09 of Na_2SiO_3 = (232.5g + 28.0855g) of Si and 128.44g of Al

Si/Al ratio = 2.03:1

$$\frac{232.5 + x}{128.44} = 2.2$$

x= 50.068g of Si

$$28.0855g\ of\ Si\ \equiv 122.09g\ of\ \mathrm{Na_2SiO_3}$$

$$88.6g\ of\ Si\ \equiv \frac{122.09\ x\ 50.068}{28.0855} = 217.65g\ of\ \mathrm{Na_2SiO_3}$$

$$\frac{Solid}{Liquid} = \frac{1.155\ kg}{y} = 2.5$$

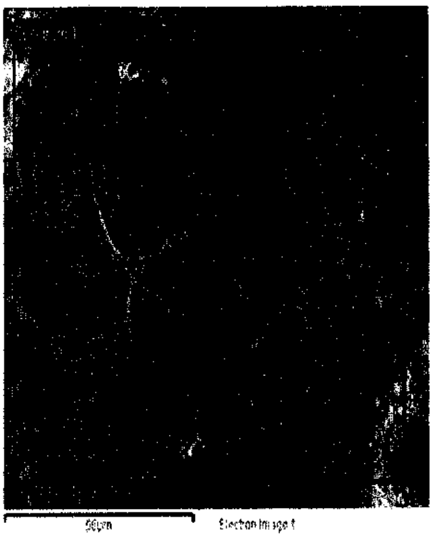
y = 0.462 kg

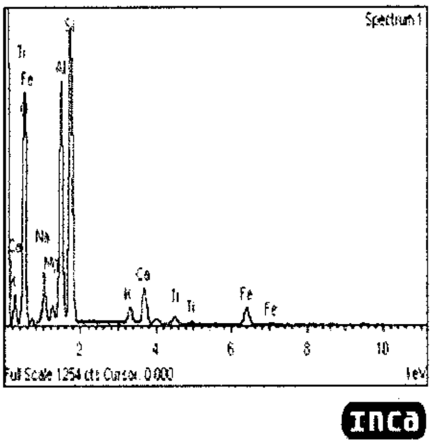
$$mass\ of\ NaOH = 0.462\ kg - 0.218\ kg = 0.244\ kg$$

Thus, for Solid/Liquid ratio is 2.5:1, the amount of FA is 1.155 kg, the amount of NaOH is 0.244 kg and the amount of Na_2SiO_3 is 0.218 kg.

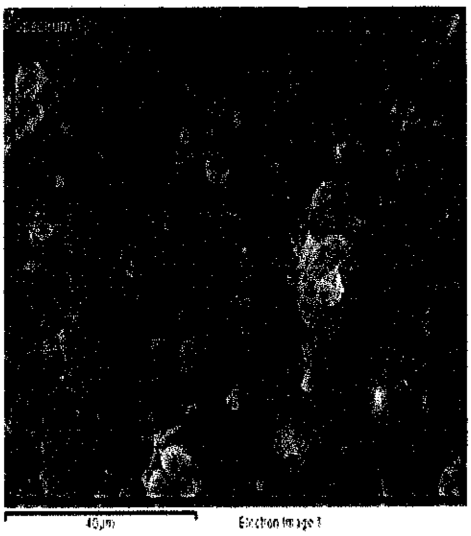
APPENDIX III: Characterization of Geopolymer Samples

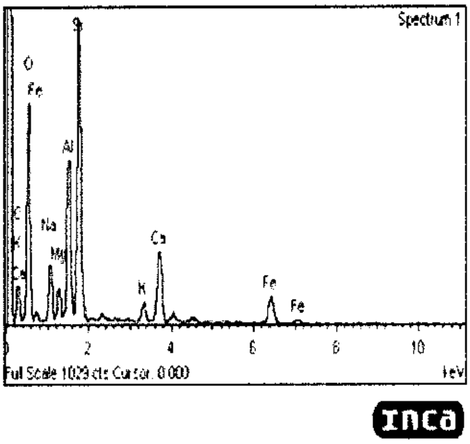
i) FESEM



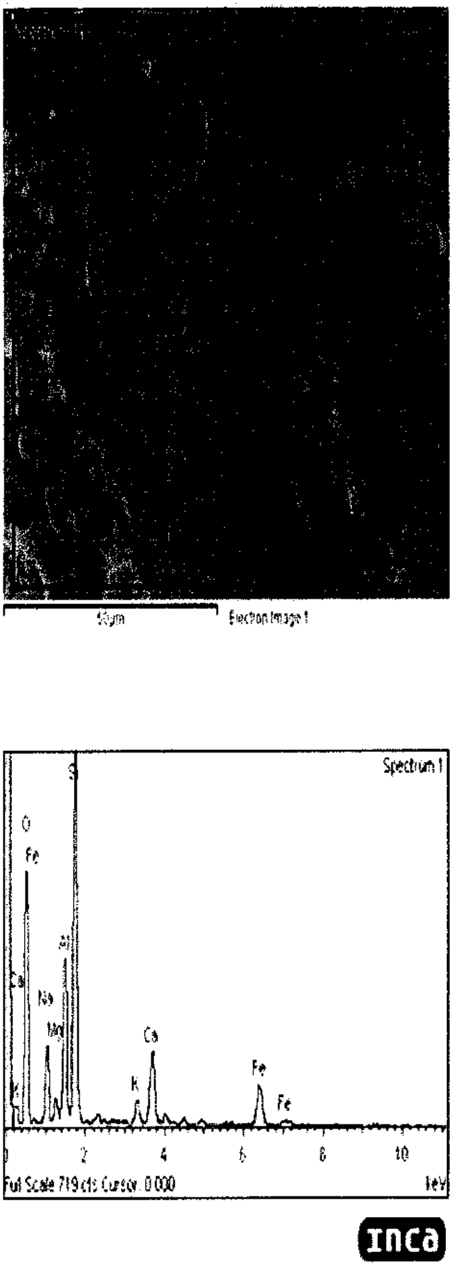


FA+12M of NaOH that cured at 60°C sample

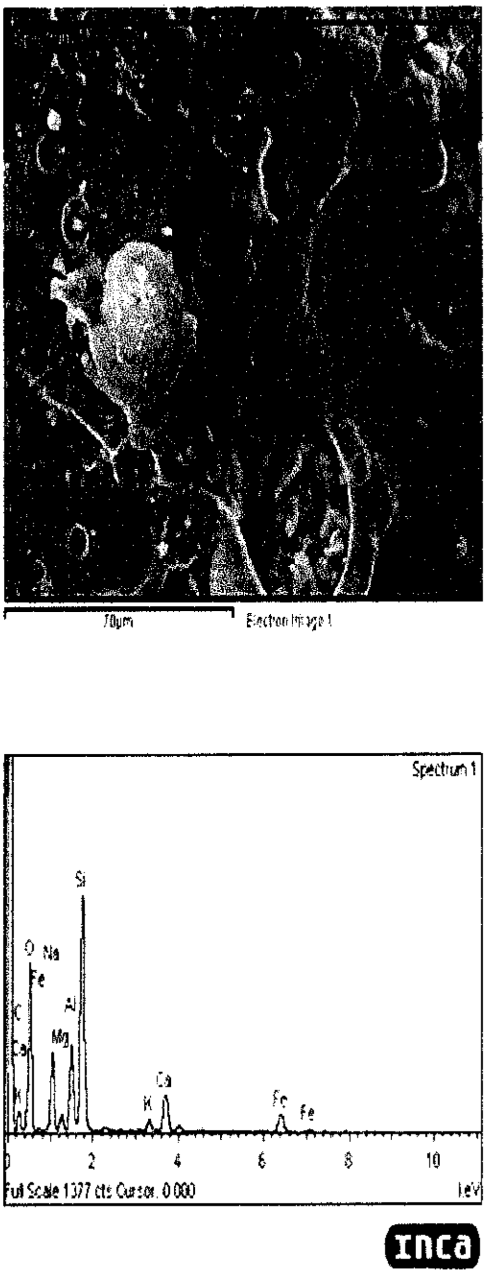




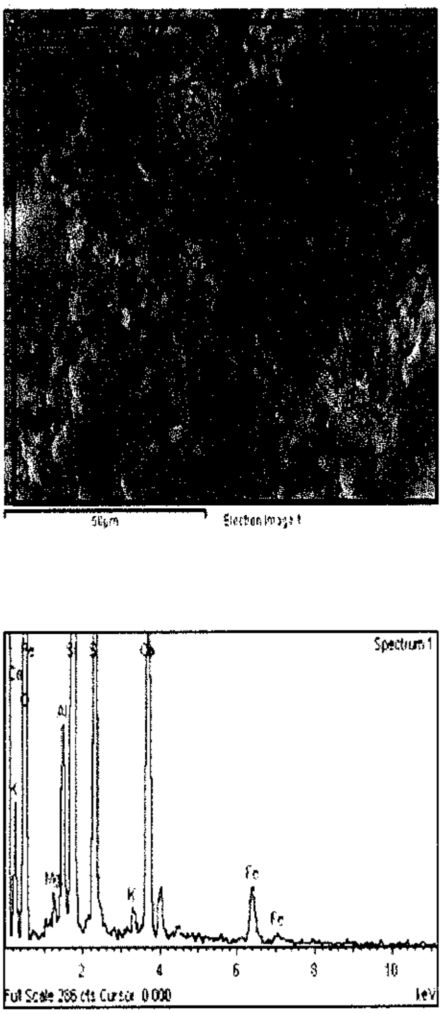
FA+6M of NaOH that cured at 26°C sample



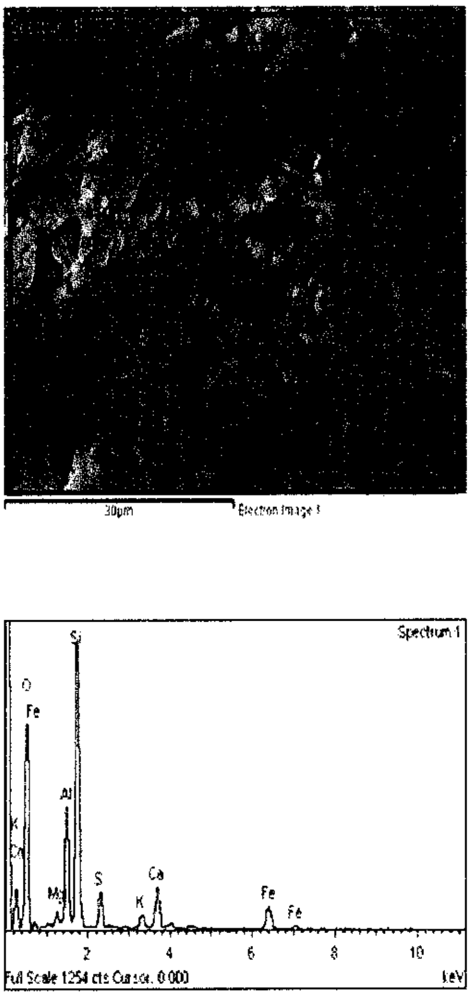
3.5:1 of solid/liquid ratio that cured at 60°C sample



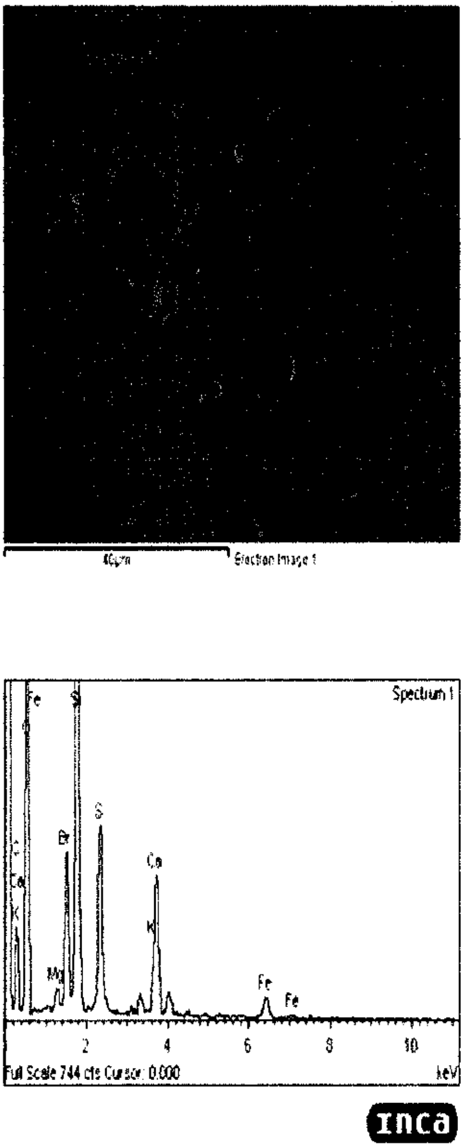
3.5:1 of Si/Al ratio that cured at 26°C sample



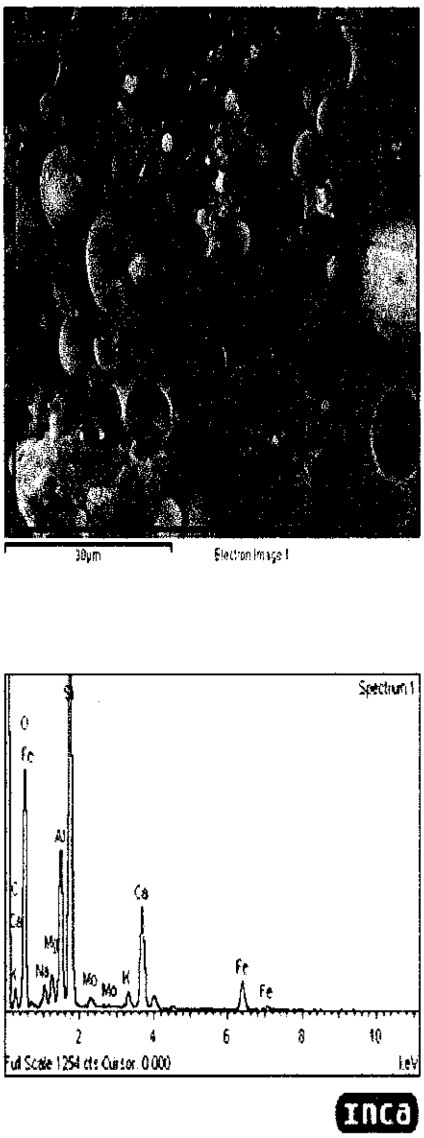
FA+12M of NaOH that cured at 60°C and immersed in H_2SO_4 at 26°C sample



FA+12M of NaOH that cured at 26°C and immersed in H_2SO_4 at 26°C sample

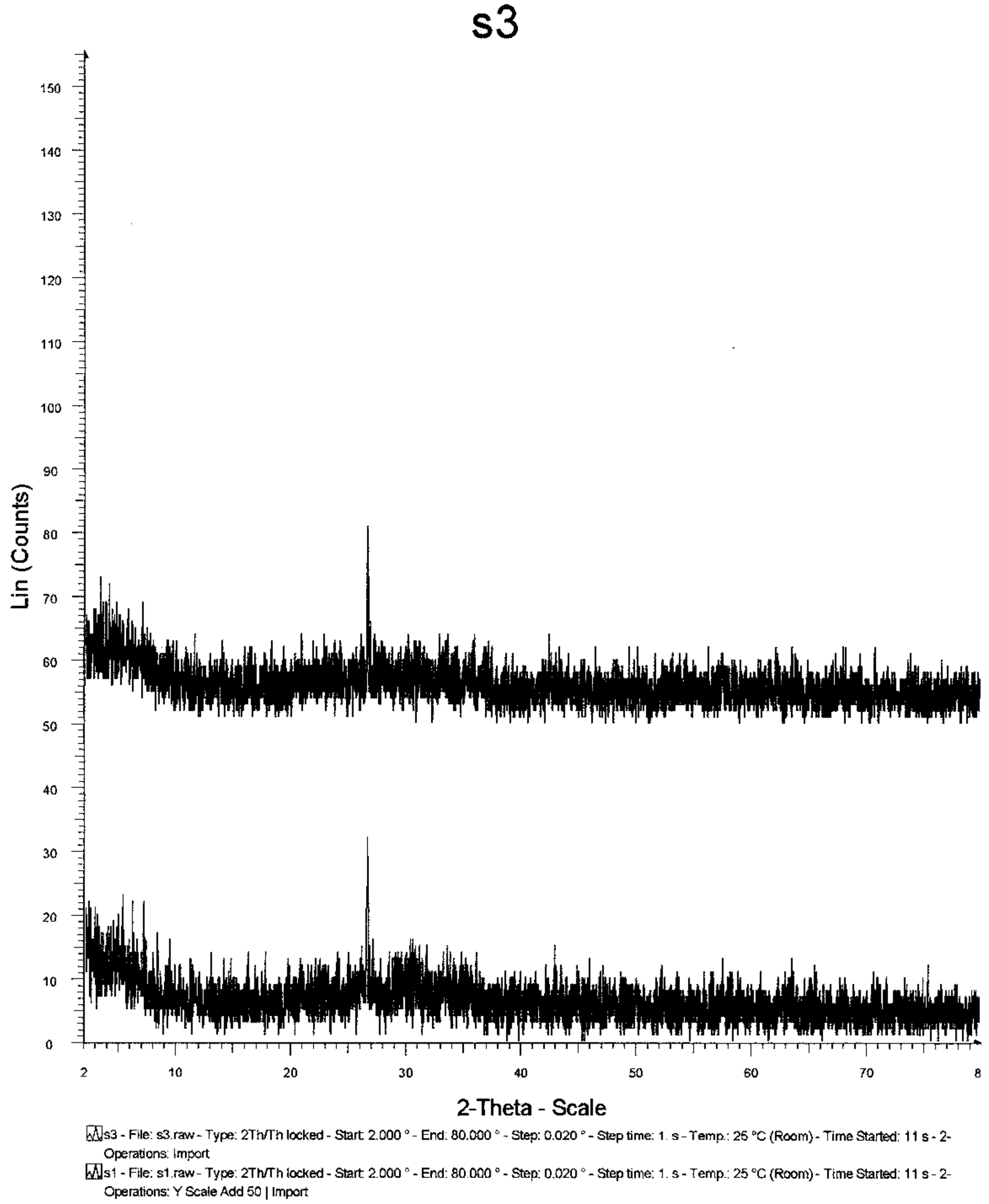


3.5:1 of solid/liquid ratio that cured at 60°C and immersed in H_2SO_4 at 26°C sample



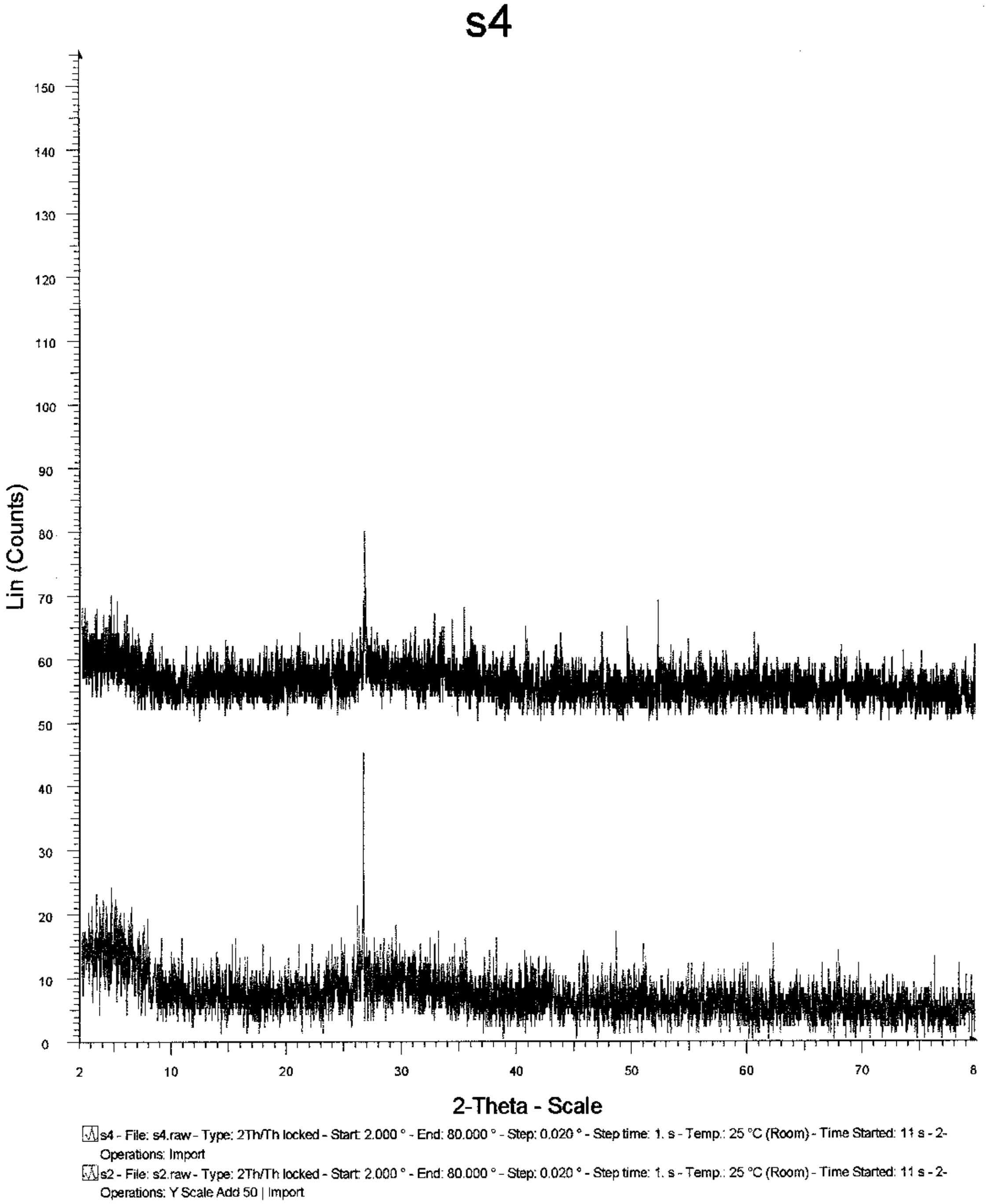
2.5:1 of solid/liquid ratio that cured at 26°C and immersed in H_2SO_4 at 26°C sample

ii) XRD



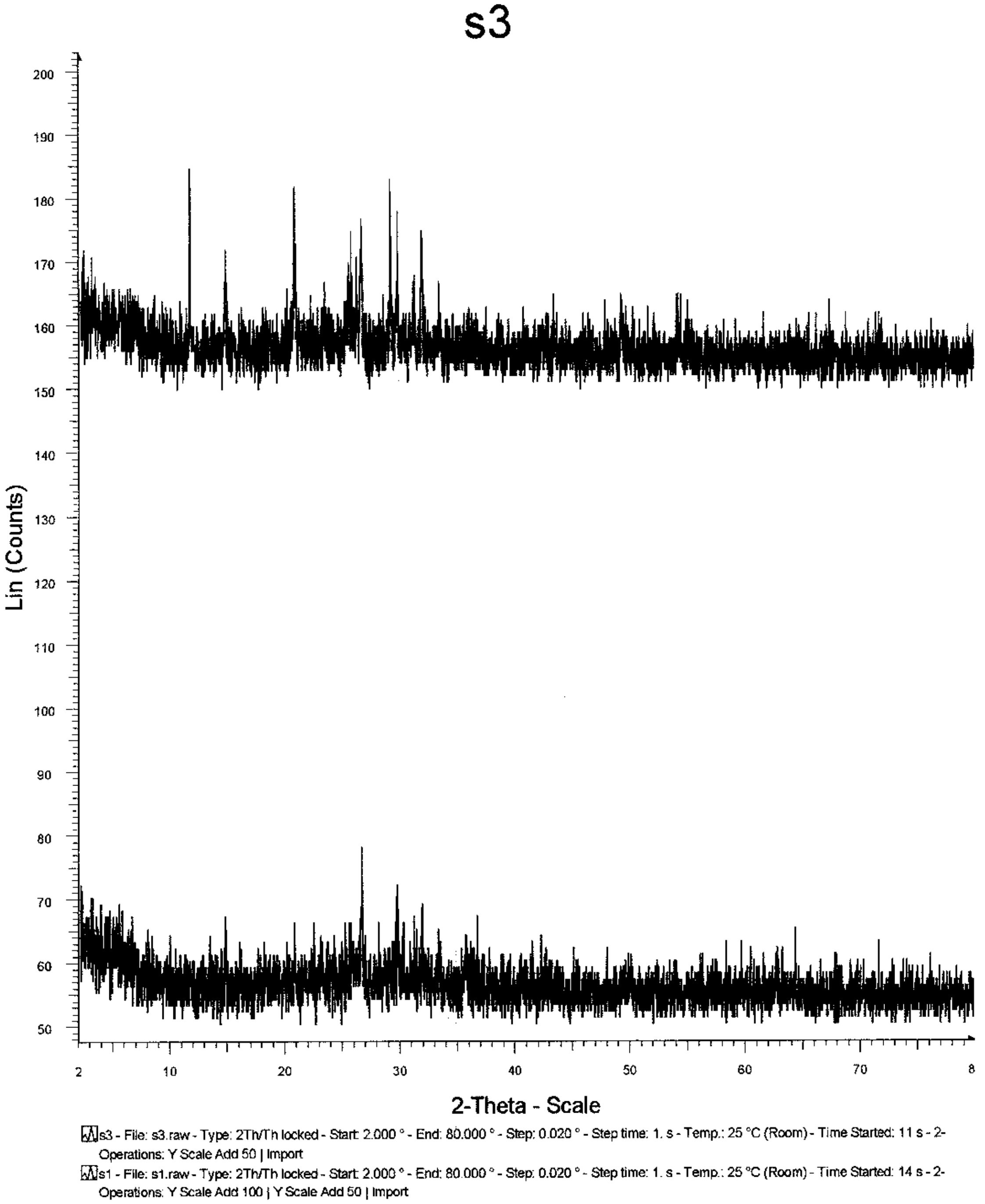
s1: FA+12M of NaOH that cured at 60°C sample

s3: FA+6M of NaOH that cured at 26°C sample



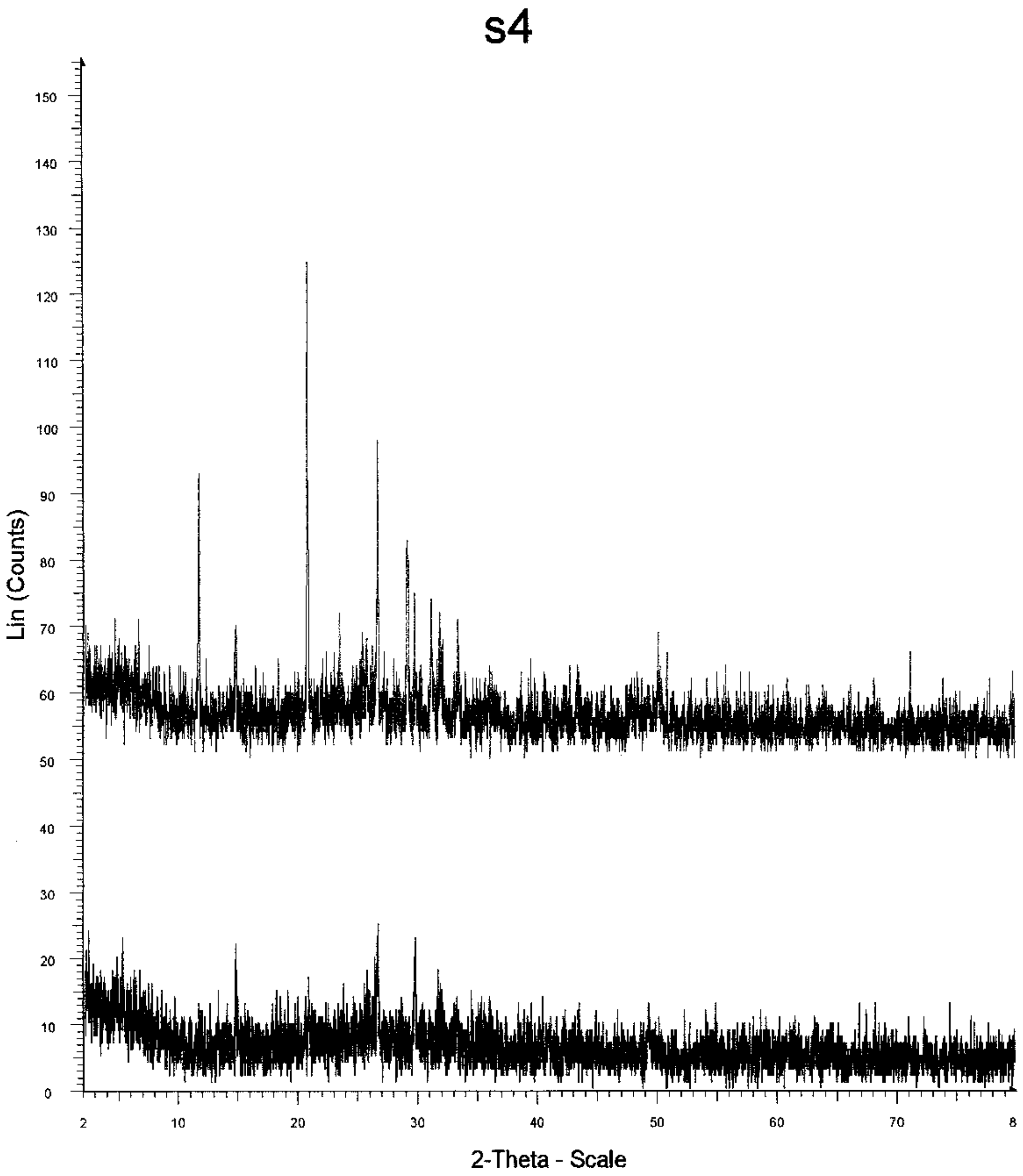
s2: 3.5:1 of solid/liquid ratio that cured at 60°C sample

s4: 3.5:1 of Si/Al ratio that cured at 26°C sample



s1: FA+12M of NaOH that cured at 60°C and immersed in H_2SO_4 at 26°C sample

s3: FA+12M of NaOH that cured at 26°C and immersed in H_2SO_4 at 26°C sample



s4 - File: s4.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.020 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Operations: Import

s2 - File: s2.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.020 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Operations: Y Scale Add 50 | Import

s2: 3.5:1 of solid/liquid ratio that cured at 60°C and immersed in H_2SO_4 at 26°C sample

s4: 2.5:1 of solid/liquid ratio that curde at 26°C and immersed in H_2SO_4 at 26°C sample