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THESIS

ACID RESISTANCE OF GEOPOLYMER PASTE

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This research presents the results of experiments to study the durability of geopolymer materials. Geopolymer specimens were manufactured from Class C fly ash (60, 65 and 70 percent content) and Class F fly ash (50, 55 and 60 percent content) activated by sodium hydroxide (at the concentration at 6M, 10M and 14M) and sodium silicate solution at room temperature, then exposed to 5% solutions of acetic and sulfuric acids. The parameters studied were the weight change, appearance of specimens, change in compressive strength and porosity which were tested by Mercury Intrusion Porosimetry (MIP). The deterioration of geopolymer paste specimens from both classes of fly ash showed varying degrees of deterioration when prepared with different concentrations of NaOH exposed to acetic and sulfuric acids

In acidic environment, the geopolymer paste specimens with high concentration of NaOH and fly ash content did better than others. For the class C fly ash specimens and 70% fly ash content did well in weight loss. Strength loss was moderate after exposure to acetic attack but was significantly higher under sulfuric. The specimens with 14 M NaOH and 70% type C fly ash showed the best acid resistance, but the strength was too low. The class F fly ash geopolymer paste specimens had high performance when the content of fly ash was higher. The best resistance to acidic attack was obtained from 60% type F fly ash specimens when submersed in acetic and sulfuric acids in terms of strength and weight loss.

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TABLE OF CONTENTS

Page

TABLE OF CONTENTS i
LIST OF TABLES ii
LIST OF FIGURES iv
LIST OF ABBREVIATIONS ix
INTRODUCTION 1
OBJECTIVES 3
LITERATURE REVIEW 5
MATERIALS AND METHODS 20
    Materials 20
    Methods 22
RESULTS AND DISCUSSION 30
CONCLUSION AND RECOMMENDATIONS 63
    Conclusion 63
    Recommendations 64
LITRATURE CITED 65
APPENDICES 70
    Appendix A Tables 71
    Appendix B Figures 84
CURRICULUM VITAE 100

LIST OF TABLES

Table **Page**

1 Classification of fly ash by ASTM C618 standards 6
2 Chemical concentrations (%wt) by XRF 20
3 Proportion of NaOH 23
4 The mixture formulations 24
5 Weight changes of class C fly ash geopolymer specimens exposed to 5% acetic and sulfuric acid solutions at 18 weeks 38
6 Weight changes of the class F fly ash geopolymer specimens exposed to 5% solutions of acetic and sulfuric acids at 18 weeks 47
7 Ratio mixture of geopolymer paste soaking in 5% solutions of acetic and sulfuric acid solutions after 18 weeks for MIP experimentation 54

Appendix Table

A1 Mixture composition in terms of molar ratio of class C fly ash specimens 72
A2 Mixture composition in terms of molar ratio of class F fly ash specimens 72
A3 The compressive strength of class C fly ash at 28 days 73
A4 The compressive strength of class F fly ash at 28 days 74
A5 The compressive strength of class C fly ash at 28 days after immersion in 5% of acetic acid after 18 weeks 75
A6 The compressive strength of class C fly ash at 28 days after immersion in 5% of sulfuric acid after 18 weeks. 76
A7 The compressive strength of class F fly ash at 28 days after immersion in 5% of acetic acid after 18 weeks 77

LIST OF TABLES (Continued)

Appendix Table **Page**

A8 The compressive strength of class F fly ash at 28 days after immersion in 5% of sulfuric acid after 18 weeks 78

A9 Weight change of specimens for class C fly ash after immersion in 5% of acetic acid period 18 weeks 79

A10 Weight change of specimens for class C fly ash after immersion in 5% of sulfuric acid period 18 weeks 79

A11 Weight change of specimens for class F fly ash after immersion in 5% of acetic acid period 18 weeks 80

A12 Weight change of specimens for class F fly ash after immersion in 5% of sulfuric acid period 18 weeks 80

A13 Compressive strength change of specimens for class C fly ash after immersion in 5% of acetic and sulfuric acid after 18 weeks 81

A14 Compressive strength change of specimens for class F fly ash after immersion in 5% of acetic and sulfuric acid after 18 weeks 81

A15 Weight change of specimens for class C fly ash after immersion in various concentrations of acetic acid at 14 days 82

A16 Weight change of specimens for class C fly ash after immersion in various concentrations of sulfuric acid at 14 days 82

A17 Weight change of specimens for class F fly ash after immersion in various concentrations of acetic acid at 14 days 83

A18 Weight change of specimens for class F fly ash after immersion in various concentrations of sulfuric acid at 14 days 83

LIST OF FIGURES

Figure **Page**

1 Compressive strength evolution of Portland cement and alkali-activated fly ash (with NaOH (FAN) and sodium silicate (FASS)) specimens exposed to 5% acetic acid solution and 5% sulfuric acid solution. 18
2 Equipment for experiments 21
3 Acetic acid and Sulfuric Acid 22
4 Fly Ash Class F , Fly Ash Class C 22
5 Sodium-hydroxide solution preparation 23
6 Sodium-silicate solution preparation 24
7 (a) Sodium-hydroxide solution added to the fly ash
(b) The materials mixed together 25
8 Sodium-silicate added to the mixture 26
9 (a) The moulds prepared (Ø 40 x80 mm)
(b) Paste poured into the moulds 26
10 Cylindrical samples kept for testing. 27
11 Effect of NaOH concentration of class C fly ash on compressive strength after 28 days 32
12 Effect of NaOH concentration of class F fly ash on compressive strength after 28 days 32
13 Effect of class C fly ash content on compressive strength at 28 days 33
14 Effect of class F fly ash content on compressive strength at 28 days 34
15 Effect of SiO_2/Al_2O_3 mole ratio of class C fly ash on compressive strength at 28 days 35

LIST OF FIGURES (Continued)

Figure **Page**

16 Effect of SiO_2/Al_2O_3 mole ratio of class F fly ash on compressive strength at 28 days 35
17 Percentage weight change of class C fly ash geopolymer specimens immersed in 5% of acetic acid 38
18 Percentage weight change of class C fly ash geopolymer specimens immersed in 5% of sulfuric acid 39
19 Change in compressive strength of class C fly ash geopolymer before and after immersion in 5% acetic and sulfuric acid after 18 weeks 43
20 Weight loss of class C fly ash geopolymer after immersion in acetic acid solution for 14 days 45
21 Weight loss of class C fly ash geopolymer after immersion in sulfuric acid solution for 14 days 45
22 Percentage weight change of class F fly ash geopolymer specimens immersed in 5% of acetic acid solution 47
23 Percentage weight change of class F fly ash geopolymer specimens immersed in 5% of sulfuric acid solution 48
24 Change in compressive strength of class F fly ash geopolymer before and after immersed in 5% acetic and sulfuric acid solutions after 18 weeks 51
25 Weight loss of class C fly ash geopolymer after immersed in solutions of acetic acid solution for 14 days 53
26 Weight loss of class C fly ash geopolymer after immersed in sulfuric acid solution for 14 days 53
27 Volume intruded distribution of class C fly ash geopolymer before and after 18 weeks in 5% acetic and sulfuric acid solutions 56

LIST OF FIGURES (Continued)

Figure **Page**

28 Total porosity of class C fly ash geopolymer before and after immersed in 5% acetic and sulfuric acid solutions after 18 weeks 57

29 Pore classification of class C fly ash geopolymer before and after immersed in 5% acetic and sulfuric acid solutions after 18 weeks 58

30 Volume intruded distribution of class F fly ash geopolymer before and after immersed in 5% acetic and sulfuric acid solutions after 18 weeks 60

31 Total porosity of class F fly ash geopolymer before and after immersed in 5% acetic and sulfuric acid solutions after 18 weeks 61

32 Pore classification of class F fly ash geopolymer before and after immersed in 5% acetic and sulfuric acid solutions after 18 weeks 62

Appendix Figure

B1 Visual appearance of 10C-70 geopolymer paste for class C fly ash before and after immersed in 5% solutions of acetic and sulfuric acids after 18 weeks 85

B2 Visual appearance of 14C-70 geopolymer paste for class C fly ash before and after immersed in 5% solutions of acetic and sulfuric acids after 18 weeks 85

B3 Visual appearance of 10F-60 geopolymer paste for class F fly ash before and after immersed in 5% solutions of acetic and sulfuric acids after 18 weeks. 86

LIST OF FIGURES (Continued)

Appendix Figure **Page**

B4 Visual appearance of 14F-60 geopolymer paste for class F fly ash before and after immersed in 5% solutions of acetic and sulfuric acids after 18 weeks 86
B5 Pore size distribution in the MIP tests for 10C-70 geopolymer paste of class C fly ash 87
B6 Pore size distribution in the MIP tests for 10C-70 geopolymer paste of class C fly ash after immersed in 5% acetic acid solution for 18 weeks. 88
B7 Pore size distribution in the MIP tests for 14C-70 geopolymer paste of class C fly ash 89
B8 Pore size distribution in the MIP tests for the 14C-70 geopolymer of class C fly ash 90
B9 Pore size distribution in the MIP tests for the 14C-70 geopolymer of class C fly ash after exposure to 5% acetic acid for 18 weeks 91
B10 Pore size distribution in the MIP tests for the 14C-70 geopolymer of class C fly ash after exposure to 5% sulfuric acid for 18 weeks 92
B11 Pore size distribution in the MIP tests for the 10F-60 geopolymer of class F fly ash 93
B12 Pore size distribution in the MIP tests for the 10F-60 geopolymer of class F fly ash after exposure to 5% acetic acid for 18 weeks. 94
B13 Pore size distribution in the MIP tests for the 10F-60 geopolymer of class F fly ash after exposure to 5% sulfuric acid for 18 weeks 95

LIST OF FIGURES (Continued)

Appendix Figure **Page**

B14 Pore size distribution in the MIP tests for the 14F-60 geopolymer of class F fly ash 96

B15 Pore size distribution in the MIP tests for the 14F-60 geopolymer of class F fly ash after exposure to 5% acetic acid for 18 weeks. 97

B16 Pore size distribution in the MIP tests for the 14F-60 geopolymer of class F fly ash after exposure to 5% sulfuric acid for 18 weeks. 98

B17 Acetic acid and sulfuric acid test 99

B18 Compression strength machine 99

LIST OF ABBREVIATIONS

Al = Aluminum

ASTM = American Standard Testing Method

Ca = Calcium

FA = Fly ash

ksc = Kilogram per square centimeter

Kg. = Kilogram

M = Molarity

MIP. = Mercury Intrusion Porosimetry

min = Minute

mm = Millimeter

NaOH = Sodium Hydroxide

Na_2SiO_3 = Sodium Silicate

Si = Silicon

SiO_2 = Silica

Std = Standard

XRF = X-Ray Fluorescence

w/b = Water to binder ratio

ACID RESISTANCE OF GEOPOLYMER PASTE

INTRODUCTION

Demand for Portland cement concrete, one of the most widely used construction materials, has always been on the increase. The cement industry is held responsible for the large amount of CO_2 emissions that have been blamed for climate change. The production of one ton of Portland cement emits approximately one ton of CO_2 into the atmosphere. In this respect, geopolymers show considerable promise to construction industry with applications as an alternative binder to Portland cement (Duxson *et al*., 2007). With consideration of global warming, geopolymer technology could significantly reduce CO_2 emissions caused by cement production (Gartner, 2004).

The chemical composition of geopolymers has been explained by several researchers (Davidovits, 1994; van Jaarsveld *et al*., 1997), and is also explained in other parts of this research. With regard to the engineering properties of geopolymer concrete, it must be noted that water is released during the chemical reaction which occurs in the formation of geopolymers. This water -expelled from the geopolymer matrix during curing and further drying periods- leaves behind discontinuous nano-pores in the matrix, which provide benefits to the performance of geopolymer concrete. The water in a geopolymer concrete mixture, therefore, plays no role in the chemical reaction that takes place; it merely provides workability to the mixture during handling. This contrasts to the chemical reaction of water in a Portland Cement-concrete mixture during the hydration process. In industrialized countries cement may be considered a cheap product suitable for concrete mass production. To compete with such an optimized system, cheap raw materials, such as blast furnace slag and coal fly ash, may be used for geopolymer production.

Geopolymers are a class of alkali-activated aluminosilicate materials that are currently finding increasing use worldwide, either as a substitute for Portland-based cements or as a room temperature means of synthesizing aluminosilicate ceramics.

The binder phase in geopolymers is the activation of a solid aluminosilicate source material with a highly-alkaline activating solution, aided by thermal curing.

Geopolymers can be considered to be a mesoporous material, where the pore structure is dependent on the reaction temperature, water content, alkali concentration, and silicate concentration of the activating solution. Leaching in acid has been shown to have some effects on a geopolymer's network structure, although their acid resistance is in most cases significantly superior to that of ordinary Portland cements. In the past few decades, geopolymer binders have emerged a possible alternative to OPC binders due to their reported high early strength and resistance against acid and sulfate attack, this apart from their environmental friendliness.

Fly ash-based geopolymers are one branch of the geopolymer family, and have attracted more attention since the 1990s. As a novel binder, the performance of fly ash-based geopolymers is promising, especially in some aggressive situations where Portland cement concretes are vulnerable. Geopolymer binders appear a promising alternative in the development of acid-resistant concrete. Since geopolymers are a novel binder, which rely on alumina-silicate rather than calcium-silicate hydrate bonds for structural integrity, they have been reported as being acid resistant: Davidovits *et al*., (1990) found that metakaoline-based geopolymer has very low mass loss when samples were immersed in 5% sulphuric acid solutions for 4 weeks; Bakharev (2005) studied the resistance of geopolymer materials prepared from fly ash against 5% sulfuric acid, with up to 5 months exposure, and concluded that geopolymer materials have better resistance than ordinary cement counterparts; Song *et al*. (2006) conducted an accelerated test to assess the durability of geopolymer concrete in a 10% sulfuric acid solution for 56 days and reported its good durability. Wallah and Rangan (2007) showed that geopolymer composites possess excellent durability in a study conducted to evaluate the long term properties of fly ash-based geopolymers; and Allahverdi Ali and Skavara (2007) conducted tests to study the corrosion mechanisms of geopolymer cements in high and low concentrations of sulfuric acid.

OBJECTIVES

The geopolymer samples for this analysis were made up from among potential applications of class F and class C fly ash-based geopolymers, the development and characterisation for acetic and sulfuric acid resistance offering the greatest attraction. The findings of the present study shall be useful in determining the applicability of geopolymer materials for use in acidic environments. The choice of acid solutions and their concentrations was based on the practical utilization of concrete as a construction material in sewage pipes, mining and the food processing industry.

The present study is aimed at evaluating the response of different fly ash-based geopolymer pastes to 5% solutions of acetic and sulfuric acid, using physico-mechanical indicators as degrees of deterioration in accelerated test conditions.

This study will use a combined determination, including changes in weight, porosity, compressive strength, presence of the products of degradation and micro-structural changes as a measure of resistance against acetic and sulfuric acid. The objectives for this study are therefore:

1. To assess the performance properties and characteristics of the microstructure of the geopolymer mixtures-each synthesized from different molarities of sodium hydroxide and fly ash contents and types- before and after acid exposure, for high-performance strength and durability.

2. To investigate sample durability in terms of acetic and sulfuric acid resistance over an 18 weeks exposure period by evaluating changes in appearance, weight, strength and porosity, and comparing these results with pre-immersion geopolymer samples.

Scope of Study

It is well known that geopolymers show better resistance to acid than Portland cement mortars or concrete. The acid concentration and immersion time are the most critical parameters affecting the compressive strength of geopolymer-paste samples. Trial mixes demonstrating good initial compressive strength at 28 days will, therefore, be selected in this study for microstructure analysis. This is especially important considering geopolymer's proposed use for structural purposes, in that the geopolymer must have sufficient residual strength -- more would indicate it would be more durable after acid attack -- when exposed to acid to avoid structural failure. This research study will be performed with the scope listed below:

1. At 28 days the specimens were immersed in 5% solutions of acetic and sulfuric acid for a quick examination.

2. The concentrations of NaOH solution, 6, 10 and 14M NaOH were used

3. The fly ash used in this study was of classes C and F.

4. Geopolymer-paste samples were immersed in acetic and sulfuric acid for 18 weeks.

5. Mercury intrusion porosimetry (MIP) was performed on the fly ash-based geopolymer.

LITERATURE REVIEW

1. Fly Ash

The fine powder known as fly ash is formed from the mineral matter in coal transported from combustion chambers by exhausts gases, and is comprised of the noncombustible matter in coal, plus a small amount of carbon that remains from incomplete combustion. Fly ash is characteristically a light tan color, and consists mainly of silt and clay-particle-sized glassy spheres, somewhat like talcum powder. The properties of fly ash vary according to coal composition and plant-operating conditions.

Classifying coal ash depending on generating place and product type:

1.1 Fly ash, as collected from burning gas by electrical precipitator: These fly ash particles are mostly spherical in shape and range in size from 0.5 µm to 100 µm. The ash is composed of silicon-dioxide (SiO_2), alminium oxide (Al_2O_3) and iron-oxide (Fe_2O_3), which is present in two forms: amorphous (rounded and smooth), and crystalline (sharp, pointed and hazardous). These fly ashes are generally highly heterogeneous, consisting of a mixture of glassy particles with various identifiable crystalline phases, such as quartz, mullite, and various iron-oxides.

1.2 Boiler slag -a black granular material- is coarser than conventional fly ash and formed in cyclone boilers that produce a molten ash cooled with water.

1.3 Bottom ash, which, as the name suggests, is collected from furnace bottoms, is a coarse granular, incombustible byproduct of burning coal for the generation of steam, electric power or both. It is coarser than fly ash, with a particle size of approximately 0.50-10.00mm. Coal bottom ash and fly ash are quite different physically, mineralogically and chemically.

This research study into fly ash-based geopolymers is good for economical, social and environmental reasons, as when compared to coal ash, fly ash gave better results. The two classes of fly ash, as classified by ASTM C618, are shown in Table 1.

Table 1 Classification of fly ash by ASTM C618 standards.

Properties of fly ash	**Classification of fly ash**	
	Class F	**Class C**
$SiO_2 + Al_2O_3 + Fe_2O_3$	70.0	50.0
SO_3, max, %	5.0	5.0
Moisture content, max, %	3.0	3.0
Loss on ignition, max, %	6.0	6.0
Strength activity index, min percentage of control	75.0	75.0

2. Geopolymer

2.1 Introduction to geopolymer

Geopolymer is an inorganic polymer formed when an alkali activator is mixed with source material, such as silica (SiO_2), compound material and alumina (Al_2O_3) compound material. Some examples of this are fly ash, metakaolin and husk ash. The synthesis mechanism in these source materials is activated by a high pH solution, dissolving the silica and alumina. The Aluminum (Al) and silicon (Si) particles form new species bonds as part of this dissolution process. The sodium silicate solution bonding compound then reacts with the new bond of polymer (Si-O-Al), turning it into something like $(Si_2O_5, Al_2O_2)_n$, with a three-dimensional macromolecular structure. (Davidovits, 1988b)

Geopolymer and Zeolite materials are similar, but geopolymer is short-range-ordered and Zeolite long-range-ordered, making them different in the amorphous phase. However, the duplicitous bonding in geopolymer is not countable because it is in the non-crystalline phase, as analyzed by either normal or synchrotron XRD.

Zeolites are classified in the same group as geopolymers due to their source material origins and them having the same three-dimensional structure. Zeolite, however, requires 150-200^{o}C for synthesis and more hydrothermal conditioning; and it takes a long time in the crystalline phase. Geopolymer uses room temperature, up to 90^{o}C, for synthesis, and the covalent bonding between molecules is strong and stable; also, due to a greater surface area between molecules, the characteristic mechanism is good. Geopolymer is acid-resistant because its molecules are chain-linked and more difficult to weaken and destroy.

Geopolymers are amorphous, aluminosilicate binders that can be synthesized utilizing sol-gel chemistry at room temperature. The geo-polymerization process starts with the dissolution of aluminosilicate precursors, by the breaking of Si-O-Si or Si-O-Al bonds from their solid particles, and the subsequent step of solid surfacing through the exchange of H^+ with mono-valent cation (K^+ or Na^+) from the bulk solution. This reaction produces $Si(OH)^4$ and $Al(OH)^4$ solution. The dissolution step occurs concomitantly with precipitation on the solid surface, which is known as the reorganization of silicates and aluminates. There follows polymerization, which occurs through the condensation of Si and Al, expelling water and leaving un-reacted excess alkali in the liquid phase.

Davitovits suggested that certain synthesis limits exist for the formation of strong products, saying that satisfactory compositions lay in the ranges (Davidovits, 1994).

$$0.2 < M_2O/SiO_2 < 0.48$$

$$3.3 < SiO_2/Al_2O_3 < 4.5$$

$$10 < H_2O/M_2O < 25$$

where M is either sodium or potassium. Accordingly, the best geopolymer will be produced when ratios are contained within these ranges.

2.2 Geopolymer composition

Geopolymer consists of two main materials, namely “source materials” and “alkaline liquids”. Those based on alumina-silicate should be rich in silicon (Si) and aluminum (Al), and could include natural minerals, such as kaolinite, clays, etc. Alternatively, it can come from by-product materials, such as fly ash, silica fume, slag, rice-husk ash, red mud etc. Only alumina (Al), however, gives a negative charge. Electric neutrality in the matrix should be balanced with cations such as Na^+ and K^+. Alkali metals are usually Sodium (Na) or Potassium (K) based. The most common alkaline liquid used in polymerization is a combination of sodium-hydroxide (NaOH), or potassium-hydroxide (KOH), and sodium-silicate or potassium-silicate. Construction materials have significant strength and stability requirements, and careful consideration should be given to geopolymers for this reason. Their composition can be represented by the formula:

$$M_n[-(Si\text{-}O)_z\text{-}Al\text{-}O]_n wH_2O \tag{1}$$

2.3 The Geopolymer Polymerization Process

Geopolymer is a mineral based on a poly-sialate (Si-O-Al-O) framework structure, with alternating SiO_4 and AlO_4 tetrahedrons joined together in three directions through sharing all the oxygen atoms. The replacement of Al^{3+} (four-fold coordination) for Si^{4+} causes a negative charge, which needs alkalis or alkali-earths to balance them, such as Na^+, K^+,Ca^2+ or $Mg^{2+.}$ Davidovits (1979).

According to Davidovits (1991), geopolymers comprised several fundamental poly-sialate units, as follows:

- Polysialate (-Si-O-Al-O-)
- Polysialate-siloxo (-Si-O-Al-O-Si-O-)
- Polysialate-disilaxo (-Si-O-Al-O-Si-O-Si-O-)

Davidovits (1994) stated “The polymerization process involves a substantially fast chemical reaction under alkaline conditions on Si-Al minerals that results in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds”. There follows the process and result of the reaction of alumino-silicate material in a strong alkaline environment: Initially comes the breakdown of Si-O-Si bonds; then a new reaction-phase occurs, with the dominant process being the penetration of the Al atoms into the original Si-O-Si structure.

“Currently geopolymer reaction takes place through 3 main steps.” (Gokhale, 2001; Van Jaarsveld *et al*., 2002).

2.3.1 Dissolution (described by the complexing action of hydroxide species)

$$\text{Solid Al} - \text{Si} + \text{MOH} = \text{M}\ {}^{+}\text{OSi(OH)}^{-}{}_{3} + \text{M}^{+}\ \text{Al(OH)}_4 \qquad (2)$$

Where M = slightly alkali cation Na^+ or K^+

Room temperature is a good dissolution condition for the exothermic process; but one must be mindful during the filling of alkaline solutions or sodium-silicate activators and sodium-hydroxide (NaOH) so that activator dissolution does not disturb the system process. The solution then undergoes heat exchange called the induction period which is the beginning of the next step of geopolymer synthesis. Temperature change is pivotal for the induction period, being shorter if the temperature is raised; the induction period is longer if the sodium-hydroxide intensity is raised, and will be stable if an intensity of more than 12 molar is used. Clearly, there are many variables specific to the dissolution step and induction period.

The complexion reaction shown in Equation 2 illustrates the intricate reaction mechanism in the geopolymer system. But this equation does not fully explain the complexities of the mechanism. Lee and van Deventer (2003) have proposed that the amount of soluble silicates in the system promote the dissolution process of the aluminosilicate species. Keyte *et al*. (2005) suggested that the soluble silicates increase the initial concentration of dissolved silicon, consequently causing more dissolution. Xu *et al*. (2004) suggested that the OH^- species hydrolyze the oxide bonds at the surface of the aluminosilicate source, promoting the aqueous products shown in Equation 2.

2.3.2 Hydrolysis-Polycondensation (Partial orientation and partial internal restructuring of the species.)

$$\mathrm{OSi\ OH^- + M^+OSi\ OH^- + M^+ = M +OSi\ OH^-\ OSi\ OH + MOH} \qquad (3)$$

The next step in the synthesis of geopolymer is hydrolysis-polycondensation, or geopolymerization, of the dissolved ions from source material resulting from the exothermic reaction. This starts from the bonding of monomer or molecules that are the core of the geopolymer, with continued bonding, producing chain bonding, which then become solid particles via the hydrolysis mechanism. In other words, Al and Si ions are dissolved from source material, and the ions then link between hydroxyl particles as hydrolysis occurs. The system also has alkaline ions because hydrolysis reactions require anionic species by using the system in high pH. As an aside, anionic species pull cationic species in to join in the same structure. While alkaline species are surrounded by H_2O (like water hull) and positioned nearby anionic species, they are not linked by strength bonding. The H_2O molecular structure is hexagonal and there is enough space for small ions such as Na and K. H_2O molecules, and those ions are attracted via ion-dipole interaction. The studies of

J.G.S. van Jaarsveld *et. al*., (2002), suggests that a lot of attraction means water hull in high order.

Xu and Van Deventer (2002a) suggest that this is mainly a physical electrostatic reaction, where the M^+ cation reacts in a cation-anion pair condensation, with the divalent-orthosilic acid and trivalent-orthosilic acid ions balancing the resulting coulombic electrostatic repellence.

2.3.3 Polycondensation or Re-precipitation (hardening of the geopolymer matrix).

In Hydrolysis-polycondensation, H_2O will occur due to the reaction of hydroxyl (OH^-) and anionic species from the hydrolysis reaction.

This segment of the geopolymer reaction describes the gel formation and the final hardening process. It is believed that dissolution of the raw materials continues simultaneously with the condensation reaction from the formed species in the gel (Lee and van Deventer, 2003). This is followed by the evaporation of water from the matrix and the final, hardened, geopolymer (Lee and van Deventer, 2002c).

The binding properties of the matrix are believed to be those of a three-dimensional amorphous silica network, with the general formula (Yip and van Deventer, 2005)

$$M_n[-(Si\text{-}O)_z\text{-}Al\text{-}O]_n wH_2O \qquad (4)$$

Where z is 1,2 , 3 or >3 ;

M was an alkali cation, such as Potassium (K) or Sodium (Na).

n was degree of polymerization.

w was the water in the matrix

The intrinsic ordering present within aluminosilicate is mainly considered for the stability of geopolymer material in aggressive environments. More crystalline geopolymer material prepared with sodium-hydroxide was stable in the aggressive environment of sulfuric and acetic acid solutions than amporphous geopolymers prepared with sodium-silicate activators. Chemical instability would also depend on the presence of the active sites on the aluminosilicate-gel surface, which appeared to increase K ions (Bakharev, 2003).

2.4 Effect of source materials on geopolymerisation

The source materials used in geopolymer synthesis were widely researched in the past. Duxson *et al*. (2005b) found that Na+ affects geopolymerisation by increasing the initial dissolution rate, while K+ accelerates the polycondensation/gelation reactions. Xu and van Deventer (2003b) suggested that by mixing one part calcinated source with one part non-calcinated one would find a product with both high initial and final strengths. Xu *et al*. (2005) completed a study on the activation of different starting materials. The result determined that GGBFS is successfully activated by both high-alkaline solution and high-alkaline soluble silicate solution, Class C fly ash activated by a high alkaline soluble silicate solution, and Class F fly ash only properly activated by a very high-alkaline soluble silicate solution, where a high-alkaline solution is defined by [OH-]>5M. It should be noted that in these cases a calcinated source can be added to assist the geopolymerisation process.

2.5 Effect of temperature and alkalinity on geopolymerisation

The previous research period time for curing was 24 hours, dependent on temperature. But there is no clear statement as to an optimum temperature and heating period. It is clear that curing conditions vary from one case to another, but elevated temperature curing for 24 hours is generally accepted: Hardjito *et al*. (2002) temperature is 60^{o}C, Sindhunata *et al*. (2004) temperature is 75^{o}C, Baharev (2005c) temperature is 95^{o}C, Fernandez-Jimenez and Palomo (2002) temperature is 85^{o}C. The negative effects of continuous high temperature curing for strength development cannot be overlooked. Van Jaarsveld *et al.* (2002) questioned prolonged curing at high temperature, i.e. 70°C for 24 hours, suggesting it could break down the gel structure in the geopolymer matrix, resulting in the lower strength with increased curing hours.

Palomo *et al.* (1992) found that the most important elements affecting compressive strength were curing temperature, curing time, and type of activator; the solution to fly ash ratio was not deemed a relevant parameter. It was determined that an increase in curing temperature increased compressive strength. Alkaline activators that contained soluble silicates resulted in a higher reaction rate than when only hydroxides were used as activators. Van Jaarsveld *et al.* (2002) also confirmed the importance of curing at elevated temperature for fly ash based geopolymers and that a longer period of time at elevated temperature weakened the micro structure, producing semi-crystalline or polycrystalline products when synthesised at higher temperatures.

Fewer nuclei will produce larger crystals and induce slower solidification. The larger crystals will not be able to pack as densely in the binder phase as their smaller counterparts, thus producing a more porous geopolymer (Provis *et al.*, 2005a). To solve this problem and produce optimum end strengths Van Jaarsveld *et al.* (2002) recommended a curing temperature of 40 to 60°C. This recommendation is largely based on a consideration of the significance of crystal water within the matrix, which in turn reduces structural cracking.

Generally it was found that a higher pH contributes to geopolymer with higher compressive strength (Xu *et al.*, 2001; Phair *et al.*, 2001a).

2.6 Geopolymer Properties

2.6.1 High early strength

Geopolymers harden rapidly and gain compressive strength in the range of 20 MPa after only 4 hours at 20^{o}C, and 70-100 MPa after 28 days (Hardjito, 2002).

In the first few hours of setting, in most cases, geopolymers gain an additional 20-30% to their final strength. The high early strength of geopolymer cement, designed particularly for waste containment, could be enhanced with the adjunction of microwave preheating devices, which can raise the temperature of the waste by 30-35^{o}C (Davidovits, 1988a).

2.6.2 Low Shrinkage

Geopolymetric cement has a higher expansion in water than Portland cement. But it also has extremely low shrinkage in air after drying, thereby preventing crack formation (Davidovits, 1988b).

2.6.3 Strong alkali and acid corrosion resistance

Davidovits (1994) studies, based on the ASTMC227 bar expansion set, indicate that while geopolymer cements have a much higher alkali content compared to ordinary Portland cement (OPC), geopolymer does not generate any dangerous alkali-aggregate reactions, while ordinary Portland cement did.

Comrie *et al.* (1998) presented geopolymer as a medium for the encapsulation of toxic wastes. They hailed Geopolymers as efficient for the encapsulation of toxic wastes because the mineral compound in geopolymer has a low response to acid erosion.

2.7 Geopolymer's compressive strength characteristics

Van Jaarsveld *et al.* (2003) stated that compressive strength was a factor in the evaluation of geopolymerization because the compressive strength test was easy and cheap. The compressive strength test is a basic way to promote geopolymer for construction industry. The compressive strength of geopolymer depends on:

2.7.1 Strength of gel phase.

2.7.2 The ratio of the gel phase/un-dissolved Al–Si particles.

2.7.3 The distribution and hardness of the undissolved Al–Si, and their particle sizes.

2.7.4 The amorphous nature of geopolymer and how crystalline it is.

2.7.5 The surface reaction between the gel phase and the un-dissolved Al–Si particles.

2.7.6 The % CaO, % K2O and the type of alkali.

2.7.7 The significance of the molar Si/Al ratio during alkaline dissolution of individual minerals.

Yip and van Deventer (2005) studied the effect of adding calcium to the basic geopolymer matrix. They found that early in the process the coexistence of gel and the CSH phase, geopolymer activation was a major contributor to the physical strength of the samples. Adding too much Ca to a geopolymer-gel led to the formation of CSH, which contributed to a more brittle, weaker matrix. Amorphous CSH could be the major component, if correctly controlled, which contributes to the overall strength, and its formation can significantly affect the geopolymer's properties, such as overall acid resistance (Lloyd and van Deventer, 2005).

Lee and van Deventer (2004) presented that a higher amount of aqueous silica available during polymerization directly influenced higher compressive strength in the matrix. A matrix with a silica concentration below 2.5M had the lowest compressive strength, regardless of the alkali concentration. An important consideration is that an optimum limit exists for the addition of soluble silica; the addition of too much aqueous silica might inhibit the dissolution of the Si-ions (Lee and van Deventer, 2002c). A suitable SiO_2 concentration of between 5 and 10M is suggested (Lee and van Deventer, 2003).

In fly ash systems, another interesting aspect was observed, as Keyte *et al*. (2005) discovered that the removal of iron from the fly ash source did not improve the compressive strength of the matrix.

3. The importance of analytical techniques in geopolymer

Pore size and pore size distribution by Mercury Intrusion Porosimetry (MIP): Mercury Intrusion Porosimetry is generally used to study pore size and pore distribution in materials. During the early stage, Ritter and Drake used it to determine pore size in ceramic material including, rock, clay, and cement paste because it is an easy and comfortable test. It is also useful in determining other results, such as volume and size of pore, pore size distribution, total porosity, threshold diameter, theoretical pore diameter, maximum continuous pore diameter and mean pore diameter. The results of MIP tests showed the pore size distribution of concrete. Usually cumulative pore size distribution is shown using the cumulative pore size distribution curve.

4. Acid attack

An interesting property of geopolymer cement is its relatively high resistance to acidic media. The resistance of alkali activated metakaolin, or fly ash, to chemical attack by acids such as nitric, sulfuric, or hydrochloric has been claimed to be far better than of Portland cement mortar or concrete.

Davidovits *et al*. (1999), for example, indicated that metakaolin-based geopolymer ‘K-PSS’ showed only 7 % mass loss when samples were immersed in a 5

% sulfuric acid solution for 4 weeks. According to Silverstrim *et al*., (1997), an alkali-activated fly ash specimen exposed to 70% vol of nitric acid for 3 months retained its dense microstructure.

Bakharev (2005b) studied the durability of geopolymer manufactured using fly ash and alkaline activators when exposed to 5% solutions of acetic (pH=2.4) and sulfuric acids (pH=0.8). In this case, as was the case in the presence of sodium-sulphate solution, it was found that the best performance in both tests was achieved by samples activated with NaOH solution (see Fig. 1), instead of by sodium-silicate solution or a mixture of sodium-hydroxide and potassium-hydroxide solution. The deterioration observed was connected to the depolymerisation of aluminosilicate polymers with liberation of silicic acid, replacement of Na and K cations by hydrogen or hydronium ions, and dealumination of the geopolymer structure. It was also connected to the condensation of siliceous polymers and zeolites, which in some cases led to a significant strength loss. When prepared with sodium-hydroxide, the more crystalline of the geopolymer material was greater stability than amorphous geopolymer prepared with a sodium-silicate activator in aggressive environments like sulfuric and acetic acid solutions.

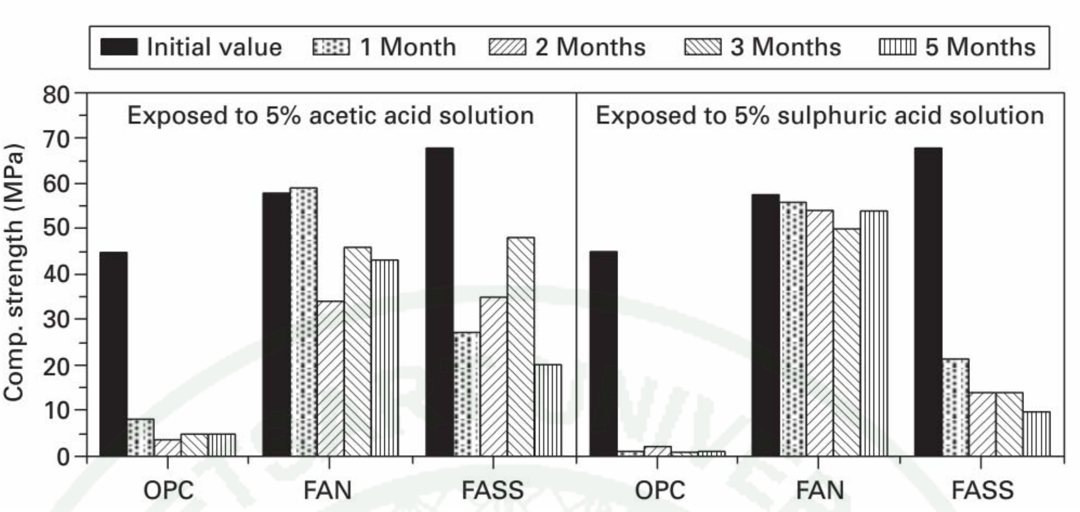


Figure 1 Compressive strength evolution of Portland cement and alkali-activated fly ash (with NaOH (FAN) and sodium silicate (FASS)) specimens exposed to 5% acetic acid solution and 5% sulfuric acid solution.

Source: Bakharev (2005b)

Interaction of geopolymer with a concentrated weak acid (e.g., acetic acid) can cause replacement of the exchangeable cations (Na, K) in the geopolymer framework by hydrogen or hydronium ions. However, treatment of geopolymer with a strong acid (e.g., sulfuric acid) results in a direct attack on the aluminosilicate framework, and dealumination. This attack will cause breakage of Si-O-Al bonds, an increased number of Si-OH and Al-OH groups in the geopolymer, and an increased amount of silicic acid ions in the solution. Consequently, this process leads to loss of mass in geopolymer materials. Inorganic polymer structures with a Si/Al ratio of 1 are more affected by acid attack than more siliceous polymers. In other cases, the dealumination process caused an increase in the Si/Al ratio and polymer chain length in the gel (Bakharev, 2005b).

OPC specimens did not perform well in either sulfuric or acetic acid solution, deteriorating completely within the first month of testing (Figure 1). This severe deterioration of OPC samples in acidic environments is connected with the material's high calcium content.

In acidic environments, high-performance geopolymer materials deteriorate via the formation of fissures in the amorphous polymer matrix, while low-performance geopolymers deteriorate through crystallization of zeolites and the formation of fragile grainy structures.

Wallah *et al*. (2005) indicated that geopolymer concrete derived from alkali-activated fly ash showed pitting and erosion on the surface of specimens soaked in sulfuric acid solution (2% concentration) after one year of exposure. Also, a significant decrease in compressive strength was observed. However, the reduction in compressive strength was significantly smaller in the case of 1% and 0.5% concentrations of sulfuric acid.

Suresh Thokchom *et al*. (2009) study was conducted to assess the resistance of fly ash-based geopolymer mortar specimens in sulfuric acid. Samples with various percentages of Na_2O, and with from 5% to 8 % of fly ash, were placed in a 10% sulfuric acid solution for a period of up to 18 weeks, to evaluate its resistance in terms of visual appearance, residual alkalinity, changes in weight and compressive strength at regular intervals. Visual inspection of geopolymer mortar samples did not show obvious color changes and remained structurally intact, though the exposed surface softened slightly. A corroded surface was observed using an optical microscope, which increased with exposure time. After 18 weeks exposure in the acid solution, the specimen showed minor weight loss, in the rage of 0.14% to 1.23% of its initial weight. In high percentages of Na_2O, weight loss in samples was higher. For specimens with 5% Na_2O, compressive strength loss was 52%, while those with 8% Na_2O showed a strength loss of 28%. The results obtained in the study indicated that geopolymers are highly resistant to sulfuric acid.

In summary, the acid attack mechanism was very similar in all cases, and the small differences between investigations depended on acid strength, solution concentration, exposure time, as well as the physicochemical and mineralogical composition of specimens. In general, fly ash that was alkali-activated with NaOH solution had the best acid resistance. Significantly, however, all geopolymers, regardless of type, outperformed OPC mortars.

MATERIALS AND METHODS

Materials

1. The chemical and mineral compositions of fly ash are shown in Table 2. The Class F fly ash used was sourced from Rayong province in the east of Thailand; the Class C fly ash used was sourced from Mae Moh Power Station in the north of Thailand (ASTM C618).

Table 2 Chemical concentrations (%wt) by XRF

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	P_2O_5	MnO	SO_3	LOI *
class F	66.07	22.68	3.28	1.23	0.57	0.94	0.33	1.3	0.31	0.03	0.21	2.61
class C	33.55	19.01	14.50	20.78	1.88	2.37	1.36	0.46	0.22	0.11	4.79	0.27

* Loss on ignition

2. Sodium silicate solution (Na_2O = 10.12%, SiO_2 = 31.19% and 58.69% water) with silicate modulus ~ 3.18.

3. Laboratory-grade sodium hydroxide in pellet form (98% purity).

4. Water: tap water.

5. Plastic cylindrical molds Ø 40x80 mm.

6. Glass bowl.

7. 5% Acetic acid solution.

8. 5% Sulfuric acid solution.

9. Compressive strength testing machine.

10. Glass bowl.

11. Digital scale, 2kg capacity, with an accuracy of 0.01 kg; and 5kg capacity, with an accuracy of 0.001 kg

12. Mercury Intrusion Porosimetry (MIP).

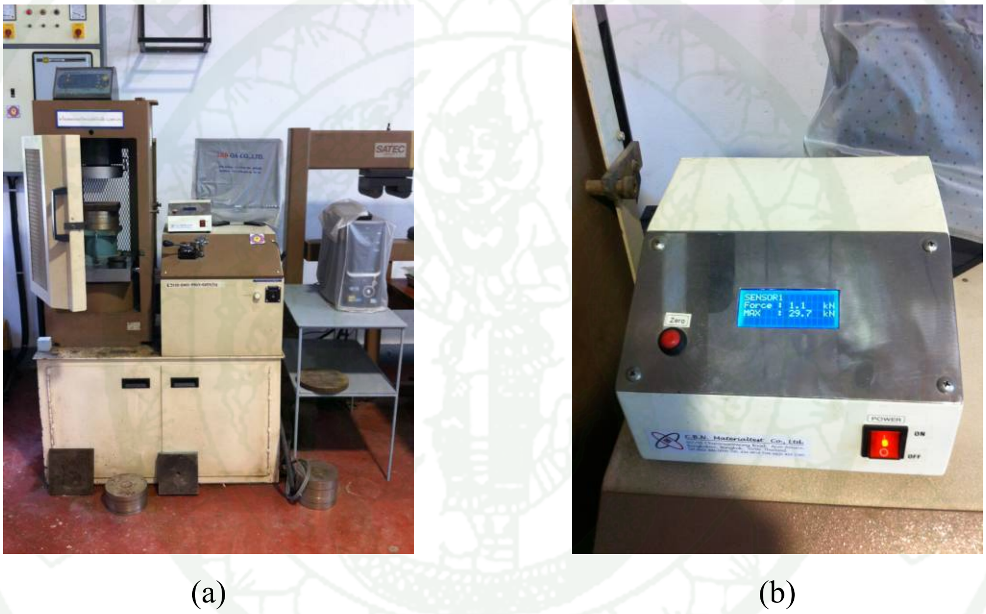

(a) (b)

Figure 2 (a)-(b) Equipment for experiments

Figure 3 Acetic acid and Sulfuric Acid

Methods

1. Preparation of geopolymer specimens

1.1 The chemical compositions of the fly ashes were determined by means of X-ray fluorescence spectroscopy (XRF) and are given in Table 2. The fly ashes used in this thesis are classified as Class F and Class C, according to the requirement of ASTM C618, and are shown in Figure 4.

(a) (b)

Figure 4 (a) Fly Ash Class F (b) Fly Ash Class C

1.2 A sodium-hydroxide solution was prepared by dissolving sodium hydroxide pellets in tap water, as shown in Figure 5. The degree of purity of the pellets was 98%. The proportion of NaOH is shown in Table 3.

Table 3 Proportion of NaOH

NaOH concentration (M)	98% purity (g/L)
6	240
10	400
14	560

(a) (b)

Figure 5 Sodium-hydroxide solution preparation

1.3 The Sodium-silicate solution, with silicate modulus of 3.18, is shown in Figure 6 and was used with both fly ashes.

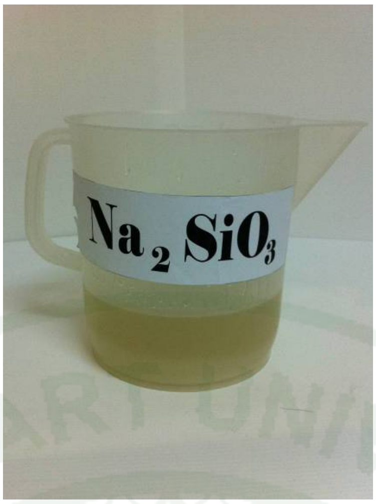


Figure 6 Sodium-silicate solution preparation

2. Test procedures:

2.1 A combination of sodium-hydroxide (NaOH) and sodium-silicate ($Na_2O.SiO_3$) solution was used as the alkaline activator in this study, as were both types of fly ash, F and C. The geopolymer mixture proportions are given in Table 4 and include data for both classes of fly ash, as used in laboratory studies.

Table 4 The mixture formulations

Mix No.	Fly ash (gm.)	Alkali solution (gm.)			Ms
		Na_2SiO_3	NaOH	Total	
6F-50	500	250	250	500	1.09
6F-55	550	225	225	450	1.09
6F-60	600	200	200	400	1.09
10F-50	500	250	250	500	0.76
10F-55	550	225	225	450	0.76
10F-60	600	200	200	400	0.76
14F-50	500	250	250	500	0.58
14F-55	550	225	225	450	0.58
14F-60	600	200	200	400	0.58

Table 4 (Continued)

Mix No.	**Fly ash (gm.)**	**Alkali solution (gm.)**			**Ms**
		Na_2SiO_3	**NaOH**	**Total**	
6C-60	600	200	200	400	1.09
6C-65	650	175	175	350	1.09
6C-70	700	150	150	300	1.09
10C-60	600	200	200	400	0.76
10C-65	650	175	175	350	0.76
10C-70	700	150	150	300	0.76
14C-60	600	200	200	400	0.58
14C-65	650	175	175	350	0.58
14C-70	700	150	150	300	0.58

2.2 Fly ash mixed with sodium-hydroxide solution for 10 minutes, as shown in Figure 7.

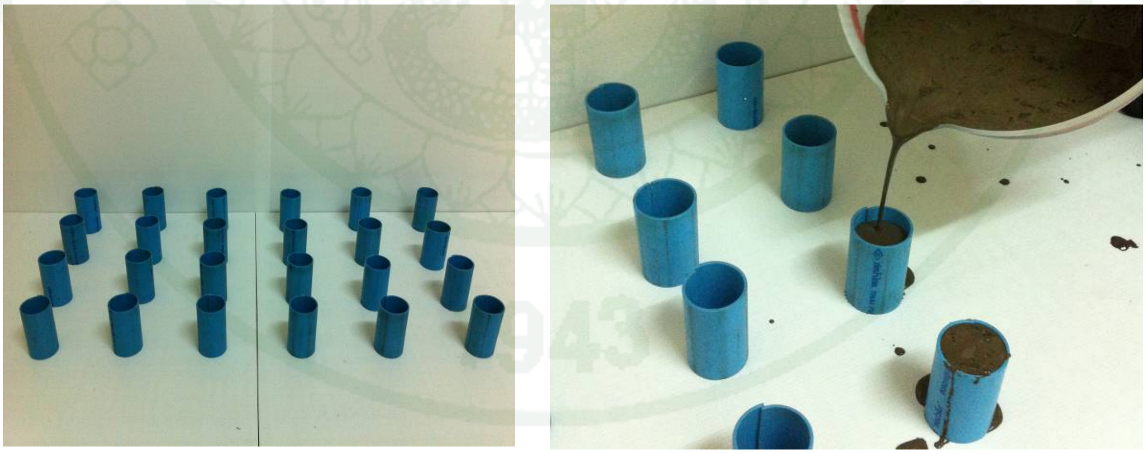
(a) (b)

Figure 7 (a) Sodium-hydroxide solution added to the fly ash (b) The materials mixed together

2.3 The sodium-silicate solution added to the mixture and mixed until a homogeneous paste is obtained, as shown Figure 8.

Figure 8 Sodium-silicate added to the mixture

2.4 The samples cast in Ø 40 x80mm moulds. Specimens cured in the moulds for a period of 24 hours at room temperature as shown in Fig.9



(a)

(b)

Figure 9 (a) The moulds prepared (Ø 40 x80 mm) (b) Paste poured into the moulds

2.5 After 24 hours curing the samples were removed from their moulds. The cylindrical specimens were kept in zip-lock bags to avoid evaporation. All specimens were kept at room temperature (30°C) until testing, as shown Figure 10.

Figure 10 The cylindrical samples kept for testing.

3. Acid Resistance Tests

Geopolymer paste was used to study the degradation mechanism of the aluminosilicate structure under both acetic acid and sulfuric acid attack. Since there are, as yet, no specific testing standards to determine the acid resistance of geopolymer, a modification of ASTM C267 was adopted for acetic and sulfuric acid resistance tests. The test steps were as follows:

- Specimen size: Cylindrical molds of size Ø 40x80mm were used so that appearance, mass loss and compressive strength reduction could be evaluated in geopolymer-paste test samples.

- Acid concentration: Geopolymer-paste cylinders were exposed to 5% (by weight) acetic and sulfuric acid solutions for 18 weeks. The results obtained in these two cases were compared with unexposed samples.

- Acid volume: The ratio of the acetic and sulfuric acid volume (ml) and the samples surface area (cm^2) was fixed at a ratio of eight and replaced monthly with fresh solutions (Chang *et al*. 2005).

- Appearance: This was the most convenient way to observe whether geopolymer paste was acid resistant. Spalling and cracking are clear and visible signs of deterioration.

- Mass change: When geopolymer paste samples were exposed to acetic and sulfuric acids, the alkali and aluminium ions in the geopolymer matrix leached out or were exchanged with hydrogen ions in the solution. The mass change with exposure time was, therefore, expected (Bakharev 2005c).

- Compressive strength change: Changes in compressive strength generally reflect aspects of deterioration after exposure to acetic and sulfuric acid, and include degradation of the gel and increased porosity of the geopolymer paste matrix.

- Porosity change: Geopolymer paste samples became porous after acetic and sulfuric acid attack (Wastiels *et al*., 1993). The porosity change of the corroded geopolymer paste was assessed using the Mercury Intrusion Porosimetry (MIP) testing method.

Testing for acid resistance was done to evaluate the acetic and sulfuric acid resistance of the geopolymer-paste specimens. This method was divided into two parts, as follows:

3.1 In the first part, to study the effects of exposure to acidic environments, the post-28-day samples were immersed in 5% solutions of acetic acid and 5% solutions of sulfuric acid for a further period of 18 weeks. The effects of acid on the specimens were constantly monitored through visual inspection, for weight change, compressive-strength and porosity, as mentioned above.

3.2 In the second part, after the 18 weeks, the specimens were removed from the acid solutions, rinsed in tap water and dried at room temperature (30°C) for two days. They then underwent micro-structural analysis of the corroded geopolymer, of different durability, in order to assess porosity changes, along with changes to microstructure and chemical composition.

Microstructure changes analysis

The MIP tests to study porosity and average-pore-diameter before and after samples were exposed to acid were conducted in order to understand its effect on mechanical strength.

RESULTS AND DICUSSION

We aim to investigate the durability of geopolymer pastes in terms of acid resistance. By using fly ash and a concentrated NaOH solution in geopolymer paste, we created different ratios of class C fly ash and class F fly ash. These results will help determine the effects of fly ash and NaOH on the geopolymer's acidity and therefore provide valuable insights into the material's durability.

For this study, we tested several different compositions of geopolymer pastes. The ratios of fly ash and NaOH concentrations are listed as follows. The class C fly ash concentrations were 60%, 65%, and 70% by weight and class F fly ash concentrations were 50%, 55%, and 60% by weight. For the NaOH solution, we tested the durability of geopolymer pastes when interacting with concentrations of 6, 10, and 14 Molars. Therefore we attempted to test three main objectives in this study as listed below:

Part 1 Testing the durability of geopolymer paste before immersion in acid solution

Part 2 Testing the durability of geopolymer paste after immersion in acid solution

Part 3 Testing the pore volume intrusion of geopolymer paste before and after immersion in acid solution

Part 1. The Result of the testing of geopolymer paste's durability before being soaked in acid solution

1. Compressive Strength

The compressive strength of different geopolymer pastes after 28 days yielded results that vary based on the concentrations of fly ash or NaOH solutions.

Appendix tables 1, 2, 3, and 4 summarize the results of testing the compressive strength of geopolymer pastes after 28 days for both class C and class F fly ash. Additionally, the results are summarized in Figures 11-14. The details follow in the next section.

1.1 Effect of Concentration of NaOH Solution

Figure 11 and 12 demonstrate the effects of the concentration of sodium hydroxide solution and the compressive strength of geopolymer paste made from class C and class F fly ash. By increasing the concentration of NaOH solution from 6M to 14M, the compressive strength rose from 212.54 to 326.15 ksc (C-60), 284.33 to 371.89 ksc (C-65), from 345.25 to 509.93 ksc (C-70) and from 226.39 to 363.86 ksc(F-60) respectively. The only exception to the demonstrated trend of increasing NaOH concentration with compressive strength was geopolymer paste made from class F fly ash comprised of 50% and 55% of fly ash by weight. Raising the concentration from 6 M to 14 M caused the compressive strength to reduce from 295.15 to 98.01 ksc and from 276.76 to 127.98 ksc, respectively.

Hardjito and Rangan (2005) found that higher molar concentrations of sodium hydroxide solution results in a higher compressive strength of geopolymer concrete. Similarly, Thanatkit and Prinya (2005) found increasing compressive strength in geopolymer when the concentration of sodium hydroxide liquid increased.

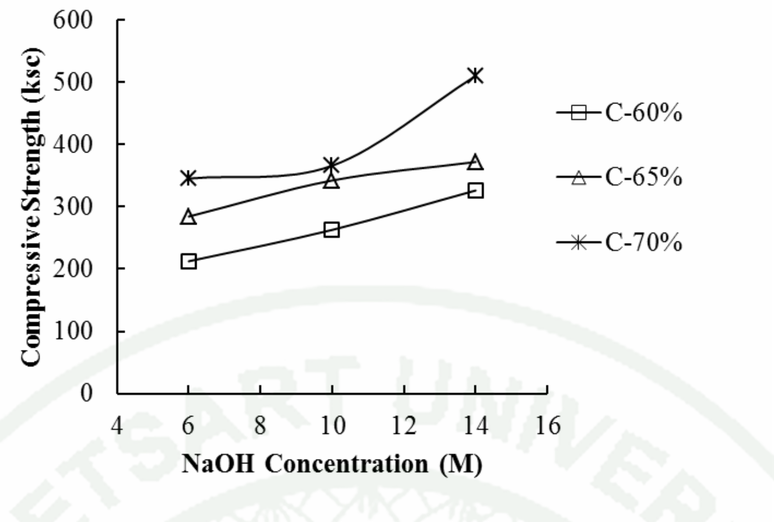


Figure 11 Effect of NaOH concentration of class C fly ash on compressive strength after 28 days

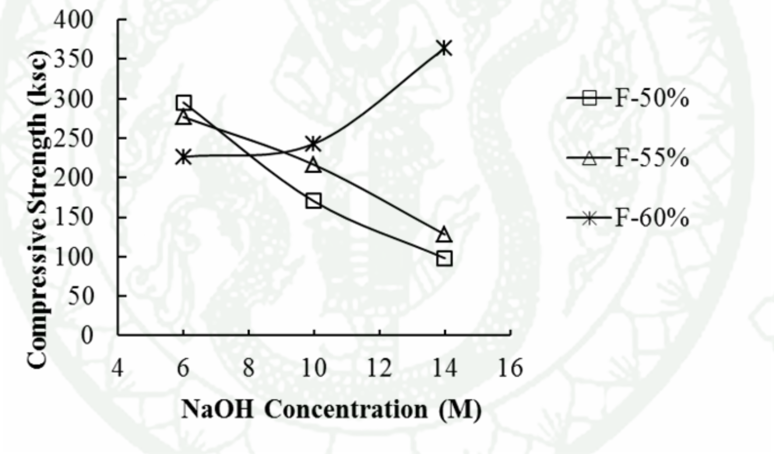


Figure 12 Effect of NaOH concentration of class F fly ash on compressive strength after 28 days

1.2 Effect of Fly Ash Content

Figures 13 demonstrates that the increased content of class C fly ash affects the compressive strength of geopolymer pastes. Class C fly ash pastes resulted in compressive strength increases from 212.54 to 345.25 ksc, from 262.93 to 365.90 ksc and from 326.15 to 509.93 ksc with fly ash composition percentage increasing from 60 to 70%. Also, for NaOH concentrations of 10M and 14 M, the class F geopolymer paste resulted in the fly ash composition percentage increase, the compressive strength also increased. In contrast, lowest NaOH solutions resulted in a decrease of compressive strength with increased content of class F fly ash as summarized in Figure 14.

Geopolymer paste made from class C fly ash (70% fly ash content by weight) in 14 M NaOH solution after 28 days yielded the best results for the highest correlation between fly ash content and compressive strength. This represents a general trend for fly ash content and should be noted for increasing the durability of geopolymer paste.

Regarding geopolymer paste made from class F fly ash with the fly ash content of 60% (F-60) by weight, higher concentrations of NaOH solution resulted in a concurrent rise of compressive strength. Also, the higher concentrations of NaOH solutions at 10M and 14M, resulted in a similar increase of compressive strength and therefore, durability. However, when using at lower than 60% (F-60) fly ash content by weight at lower concentrations NaOH solution (6M), the increased concentration of NaOH and fly ash results in an overall decrease in compressive strength. Such results clearly indicate that the most appropriate ratio for geopolymer paste occurs with a 60% fly ash content and 14 M concentration of NaOH. These conditions clearly yielded the highest compressive strength of 363.86 ksc, which also generates a positive trend for fly ash content and compressive strength.

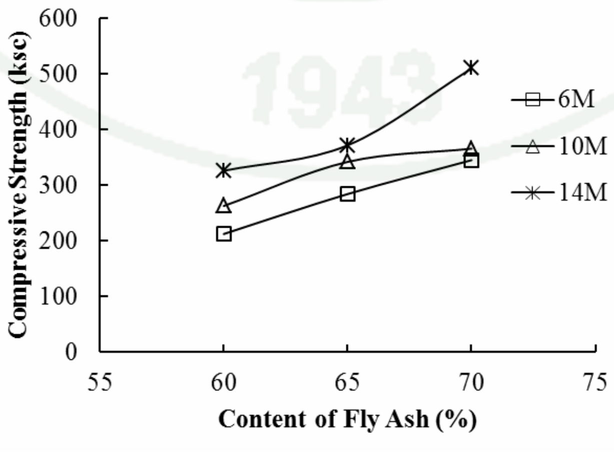


Figure 13 Effect of class C fly ash content on compressive strength at 28 days

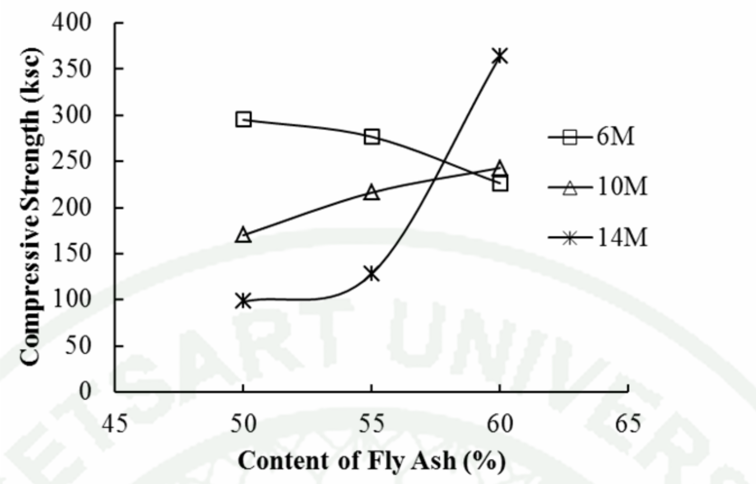


Figure 14 Effect of class F fly ash content on compressive strength at 28 days

1.3 Effect of SiO_2/Al_2O_3 mole ratio

Figures 15 and 16 represent the effects of increasing the SiO_2/Al_2O_3 mole ratio on compressive strength for geopolymer pastes. The increase of SiO_2/Al_2O_3 mole ratio resulted in a general decrease in compressive strength, while a higher concentration of NaOH (14M) yielded the same compressive strength as class C fly ash specimens. The molar ratio of SiO_2/Al_2O_3 did not change during this trial. These results are in accordance with Hardjito (2004a), who found a positive trend between the compressive strength of pastes with fly ash containing silica (Si) and aluminum (Al) and sodium hydroxide solution concentrations. Furthermore, this study confirms Smith Songpiriyakij *et al.* (2010) finding that compressive strength increases alongside an increased SiO_2/Al_2O_3 ratio.

Class F fly ash specimens with 10M and 14 M concentration of NaOH was similar result to class C fly ash specimens. Solutions of 6M NaOH decrease and therefore follow a negative trend with the SiO_2/Al_2O_3 ratio.

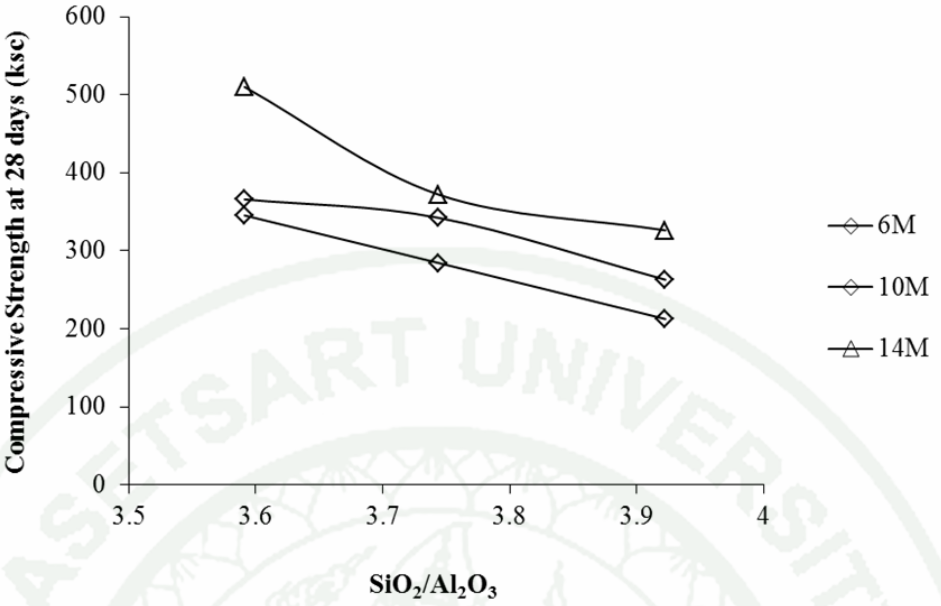


Figure 15 Effect of SiO_2/Al_2O_3 mole ratio of class C fly ash on compressive strength at 28 days

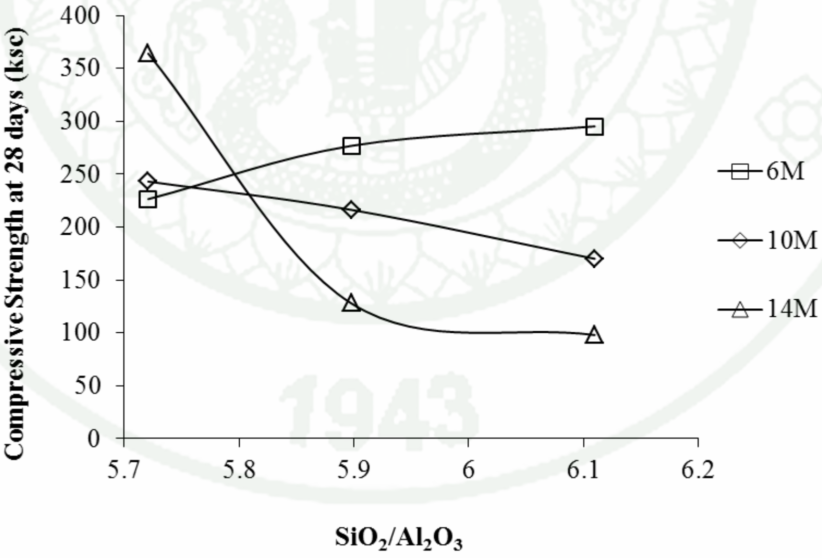


Figure 16 Effect of SiO_2/Al_2O_3 mole ratio of class F fly ash on compressive strength at 28 days

Part 2: Testing the durability of geopolymer paste after immersion in acetic and sulfuric acid solution

1. Resistance properties for acetic and sulfuric solutions of geopolymer paste made from class C fly ash.

The following section summarizes the class C fly ash geopolymer paste results when soaking in 5% solutions of acetic and sulfuric acid for 18 weeks.

1.1 Average percent weight change of specimens immersed in 5% solutions of acetic acid and sulfuric acid for 18 weeks

Table 5 and Figure 17 show the change in weight of Class C fly ash geopolymer specimen with a curing period of 28 days when soaked in a 5% solution of acetic acid for 18 weeks. Additionally, this information can be observed in Appendix Table 9. The 60% of fly ash content mixture with 10M concentration gradually lost approximately 12.0% of its weight over 18 weeks. The 10M concentration reduced nearly 14.7%, additionally. After the severe weight loss following week 12, the 14 M concentration specimen could not be tested for its weight while maintaining accuracy.

The 65% of class C fly ash specimens lost 11.362% and 10.532% of their weight respectively at 6M and 10M. The 14C-65 specimens gained weight by week 3 and deteriorated until testing could not continue at week 6, losing 2.2% of their weight, respectively.

The 70% of class C fly ash specimens gained weight by week 2 at 6M and week 6 at 10M, 14M concentration of NaOH. The specimens, in order of increasing concentrations (6M, 10M, 14M), each lost 6.424%, 5.382% and 4.712% of their weight by week 18.

Figure 17 clearly higlights the highest performance and most durability for 70% of class C fly ash specimens. Additionally, the 14M concentration lost the least amount of weight due to immersion in acetic acid.

Table 5 and Figure 18 summarize the weight gains for 60% class C fly ash specimens, which gained weight for twelve consecutive weeks and the results are listed in Appendix Table 10.

Following the course of the 60% class C fly ash specimens, the 65% fly ash group continued to gain weight. The most durable specimens took the least amount of time to deteriorate. For instance, the rate of change for exposure to higher concentrations of NaOH solution tended to increase and deteriorate after 12 weeks, however with a lower concentration, severe deterioration occurred after 15 weeks. The overall trend was deterioration after 15 to 18 weeks that followed an initial period of weight gain.

The test results demonstrate that geopolymer paste specimens made of class C fly ash shared similar results with specimens soaked in acetic acid solutions. The acetic acid solution slowed the deterioration process of the paste. Furthermore, the increased concentration of NaOH solution present reduced the percentage of the weight lost. Sulfuric acid expedited the deterioration process.

In summary, 14C-70 specimens had the best durability performance for both acetic and sulfuric acid.

Table 5 Weight changes of class C fly ash geopolymer specimens exposed to 5% acetic and sulfuric acid solutions at 18 weeks

Specimens	**Weight loss (%)**	
	Acetic acid	**Sulfuric acid**
6C-60	-11.988	0.159**
10C-60	-14.699	1.405**
14C-60	-11.794**	4.735*
6C-65	-11.362	5.539***
10C-65	-10.532	-14.505
14C-65	-2.257*	5.351*
6C-70	-6.424	0.965***
10C-70	-5.382	-14.375
14C-70	-4.712	-7.096

Complete deterioration
* at 6 weeks
** at 12 weeks
*** at 15 weeks

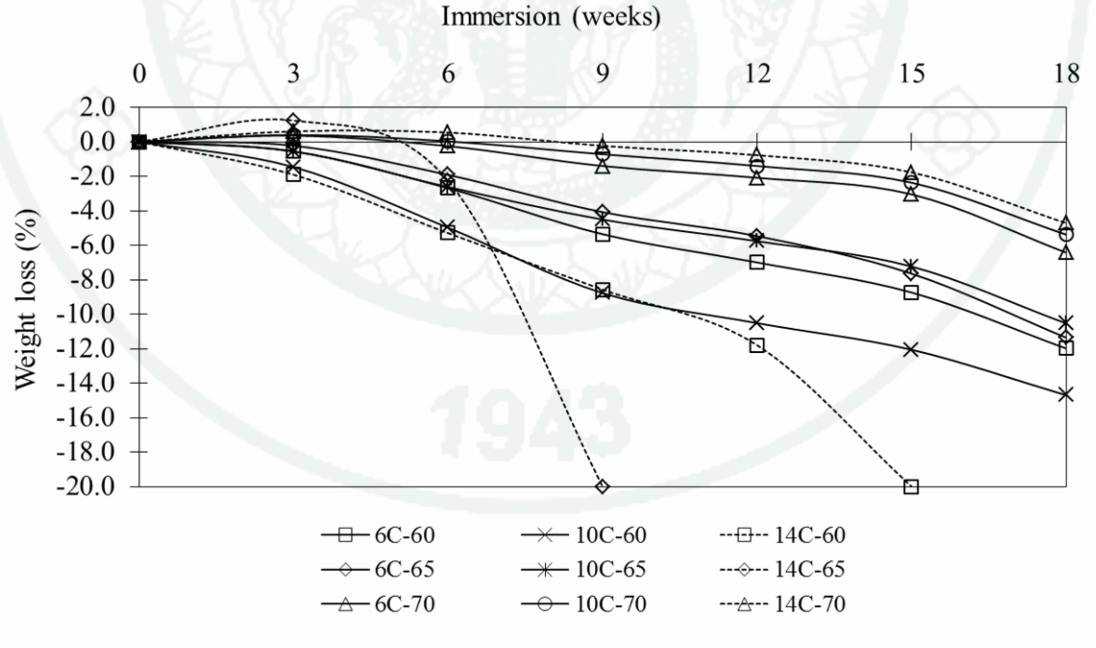


Figure 17 Percentage weight change of class C fly ash geopolymer specimens immersed in 5% of acetic acid

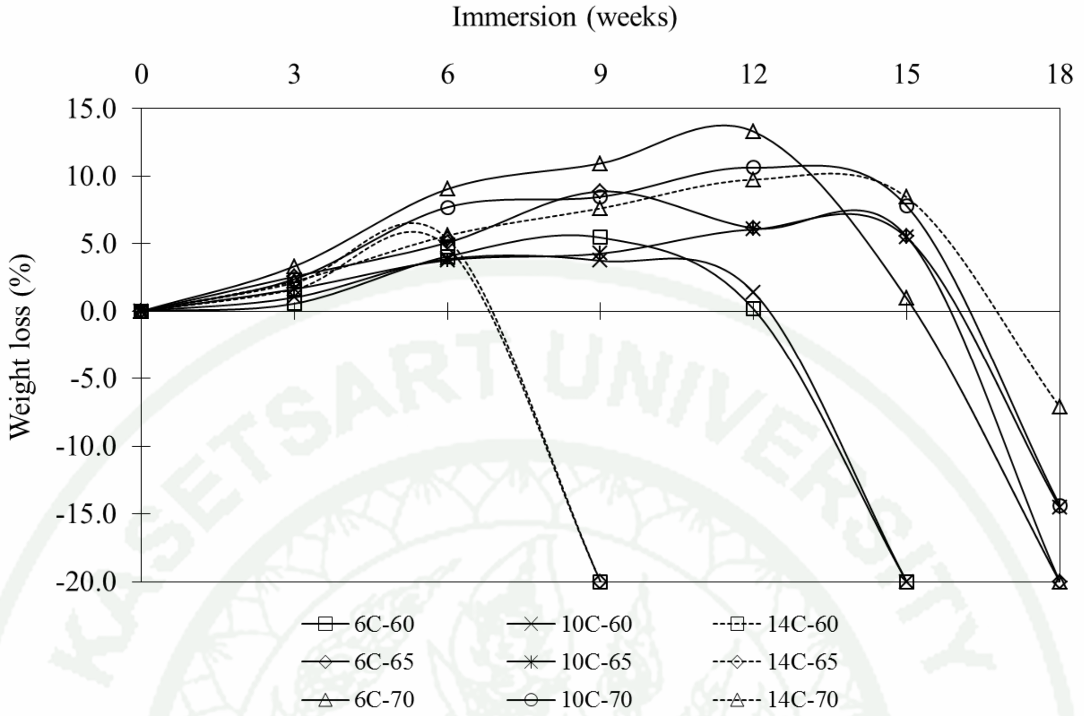


Figure 18 Percentage weight change of class C fly ash geopolymer specimens immersed in 5% of sulfuric acid

1.2 Appearance of the specimens after immersion in 5% solutions of acetic acid and sulfuric acid for 18 weeks

As seen in both Appendix Figure 1 and Figure 2, the visual examination of specimens exposed to 5% of acetic and sulfuric acid solutions after 18 weeks showed very fine cracks on the surface. This compares with the appearance of geopolymer paste specimens when exposed to acetic acid solution, resulting in severe deterioration of the specimens. Within a week, the specimens immersed in sulfuric acid made with Class C fly ash formed a thick layer of white paste on the surface. These results confirm the findings of Wallah and Rangan (2006), who suggest a slight damaged appearance on the surface of specimens following exposure to sulfuric acid.

1.3 Average percentage in the compressive strength of the specimens after immersion in 5% solutions of acetic acid and sulfuric acid for 18 weeks

In series of C-60, the specimens greatly reduce the residual compressive strength. The increase of NaOH solution concentration helps reduce compressive strength losses. Specimens with a concentration of 14M NaOH solution could not resist the loss of compressive strength when soaked in acetic acid. Increasing the concentration of NaOH tends to increase the residual compressive strength for 6M and 10M in series of C-65. All specimens in series of C-70 increased residual compressive strength with the increased concentration of NaOH solution. This occurred in a nearly linear fashion.

According to the test result of geopolymer paste made of Class C fly ash after being soaked in 5% solutions of acetic acid for 18 weeks, shown in Figure 19 and Appendix Table 5 and Appendix Table 13, it was found that C-60 specimens, in which concentration of NaOH solution increased from 6M to 10M and 14M, tended to greatly reduce the residual compressive strength respectively. Increasing concentration of NaOH from 6M to 10M as specimens with proportion of mixture by 6C-60 and 10C-60 effected reduction of the residual compressive strength from 168.31 ksc to 88.69 ksc and strength loss was increased from 20.81% to 66.27% respectively. Regarding specimens with concentration of NaOH for 14M, it was found that it had no capacity to resist strength when soaking in solution of acetic acid.

At C-65 specimens, increasing concentration of NaOH from 6M to 10M tended to slightly increase the residual compressive strength as 6C-65 and 10C-65 specimens. The residual compressive strength was increased from 168.87 ksc to 175.28 ksc and strength loss was increased from 40.61% to 48.77% respectively. Based on using concentration of NaOH for 14M and proportion of mixture by 14C-65, the residual compressive strength could not be found when soaking in solution of acetic acid as 14C-60 specimens.

Meanwhile, C-70 specimens, with increased concentration of NaOH solution from 6M to 10M and 14M tended to increase significantly the residual compressive strength all specimens. The residual compressive strength was increased from 229.44 ksc to 239.84 ksc and 316.89 ksc respectively. Strength loss was continually increased from 33.54% to 34.45% and 37.86% respectively.

The increased concentration of fly ash aids in increasing residual compressive strength in addition to NaOH solution. Therefore, most of the relationships seen in the increase of fly ash concentration and NaOH solution result in positive associations with durability for the geopolymer pastes.

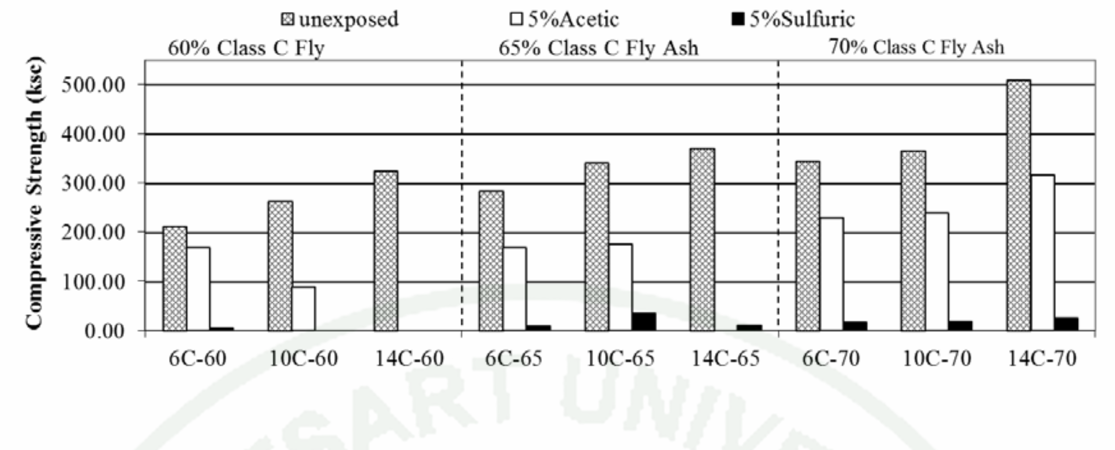
Geopolymer paste after immersed in sulfuric acid solutions of 5% for 18 weeks, shown in Figure 19, Appendix Table 6 and Appendix Table 13 with increased concentration of NaOH solution from 6M to 10M and 14M of C-60 specimens, specimens tended to greatly reduce the residual compressive strength respectively. The character of specimens emerged swell and cracks and a decreased strength more than soaking in acetic acid for all specimens. The 6C-60 specimens highly reduced the residual compressive strength which was 4.6 ksc and 97.83% strength loss. However, when increasing concentration of NaOH solution from 6M to 10M and 14M as specimens with proportion of mixture by 10C-60 and 14C-60 respectively, it was found severe deterioration of specimens until strength loss could not be found.

Increased concentration of NaOH solution from 6M to 10M of C-65 specimens tended to increase the residual compressive strength from 7.60 ksc to 35.27 ksc. Meanwhile strength loss tended to reduce from 97.33% to 89.69% respectively. When increasing concentration of NaOH solution up to 14M, it resulted in the decrease of the residual compressive strength for 10.61 ksc and the increase of strength loss by 97.15 % when comparing with using concentration of NaOH solution for 10M.

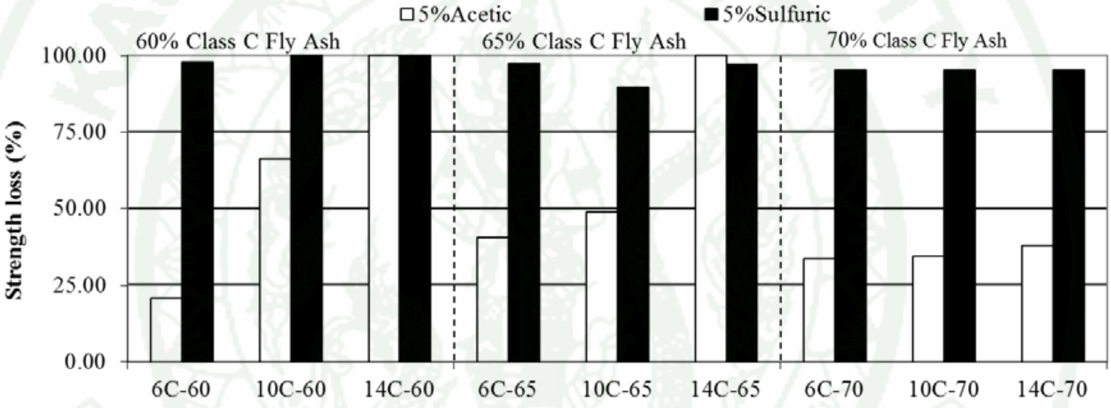
Increased concentration of C-70 specimens from 6M to 10M and 14M tended to increase the residual compressive strength the same as soaking in acetic acid solution. Specimens with proportion of mixture by 6C-70, 10C-70 and 14C-70 slightly increased the residual compressive strength from 229.44 ksc to 239.84 ksc and 316.89 ksc, respectively and strength loss tended to closely reduce from 95.19% to 95.13% and 95.12% respectively. In case of increasing content of fly ash from 60% to 56% and 70%, this tended to increase the residual compressive strength and reduce percentage of strength loss when using same concentration of NaOH solution and the least strength loss was found from geopolymer paste at proportion of mixture by 10C-65.

According to the test result, it was shown that specimens of geopolymer paste made of Class C fly ash after being soaked in acetic acid solution for 18weeks and increased concentration of NaOH solution C-60 and C-65 specimens tended to increase percentage of strength loss. On the contrary, using C-70 specimens resulted in the opposite and increased concentration of NaOH resulted in the decrease of strength loss percentage. The increase of fly ash in mixture with same concentration of NaOH solution resulted in the decrease of strength loss percentage respectively except lowest concentration of NaOH (6M) and increased fly ash tended to increase strength loss percentage.

In sulfuric acid solution with C-60 specimens, increase concentration of NaOH solution in strength loss percentage was more than using C-65 and C-70 specimens respectively. At C-60 specimens, using concentration of NaOH solution for more than 6M resulted in severe deterioration of the specimens and strength loss could not be found. At C-65 specimens using concentration of NaOH solution for 10M showed the least strength loss percentage. Using C-70 specimens and increased concentration of NaOH solution resulted in a slight decrease of strength loss and the increase of 60% fly ash to 65% and 70% tended to reduce strength loss percentage respectively when using same concentration of NaOH.



a) Compressive strength of mixture



b) Strength loss of mixture

Figure 19 Change in compressive strength of class C fly ash geopolymer before and after immersion in 5% acetic and sulfuric acid after 18 weeks

1.4 Average percentage of weight change of the specimens when immersed in various solutions of acetic acid and sulfuric acid for 14 days

Figures 20-21 and Appendix Tables 15-16, show percentage weight loss of C-60, C-65 and C-70 geopolymer specimens immersed in acetic and sulfuric acid. The weight changes of the class C fly ash specimens at different exposure durations are related to concentration of NaOH when immersed in 1%, 2%, 3%,4% and 5% solutions of acetic and sulfuric acid.

In acetic acid, C-60, C-65 and C-70 specimens had strength loss decrease when concentration of NaOH increased except 10C-70 specimens which had 8.079% weight loss, which was more weight loss than 6C-70 specimens had 6.391%.

In sulfuric acid, all specimens had loss in weight over the entire duration of exposure. The 6M of NaOH had the lowest weight loss and 10M had the highest weight loss in terms of C-60, C-65 and C-70 specimens.

The best performance of acid attack was 14C-70 in both acids corresponding with the results of specimens immersed in 5% acetic and sulfuric acid solutions period 18 weeks. It can be deduced from the results that the lower concentration of NaOH had more effect on the deterioration of specimens than higher concentration of NaOH.

Wallah and Rangan (2006) confirmed that surface damage to specimens increases along with the concentration of the acid solution (Appendix tables 13-14). Breck (1974) suggested that increased acid would break Si–O–Al bonds, increasing the number of Si–OH and Al– OH groups in geopolymers and increasing the amount of silicic acid ions and dimers in solution. This process leads to weight loss of the geopolymer paste. Polymer structures with higher Si/Al ratios remain more at risk to acid attacks than siliceous polymers.

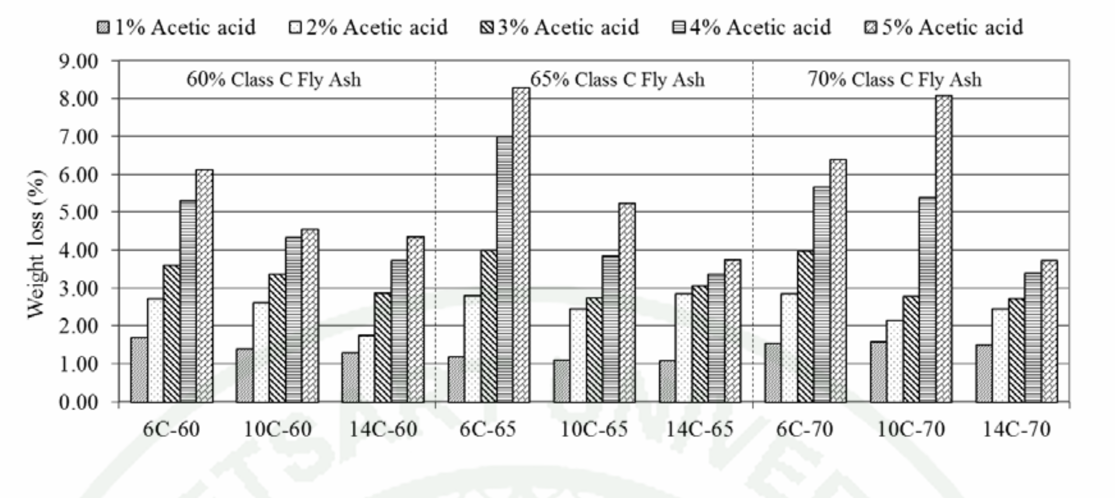


Figure 20 Weight loss of class C fly ash geopolymer after immersion in acetic acid solution for 14 days

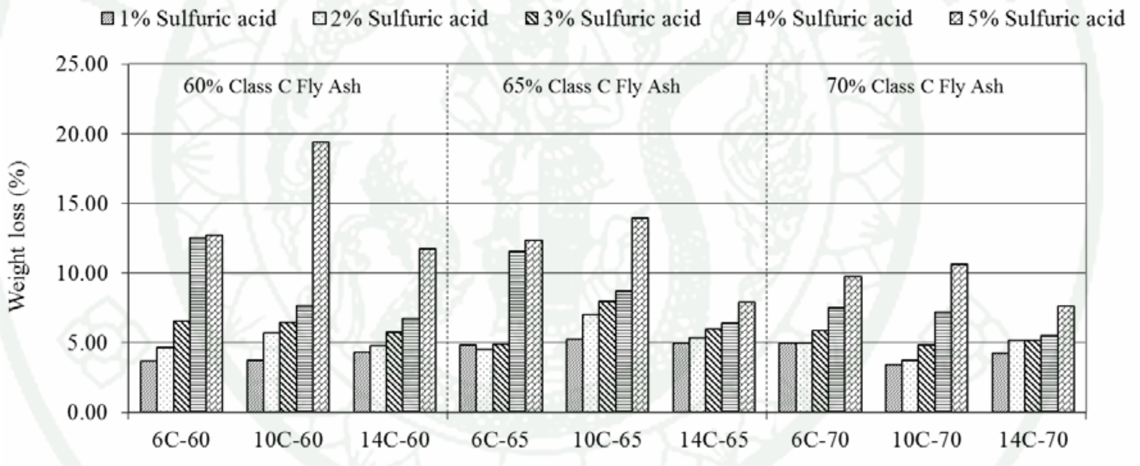


Figure 21 Weight loss of class C fly ash geopolymer after immersion in sulfuric acid solution for 14 days

2. The results of resistance properties for acetic and sulfuric solutions of geopolymer paste made of Class F fly ash.

The result of geopolymer paste made of fly ash type F when immersed in 5% solutions of Acetic acid and Sulfuric acid for 18 weeks were as follows:

2.1 Average percentage of weight change of the specimens when immersed in 5% solutions of acetic acid and sulfuric acid for 18 weeks

Table 6, Figure 22 and Appendix Table 11 display weight change of geopolymer paste specimens based on class F fly ash in 5% of acetic acid solution for a period of 18 weeks. It was found that F-50 specimens lost strength after the first week and continued to deteriorate until week 18. At F-60, the specimen groups displayed good durability in terms of strength loss with percent changes of -5.807%, -4.455% and -5.843% respectively.

The results demonstrate that class F fly ash specimens with a medium concentration of NaOH solution have the least strength loss followed by the highest concentration of NaOH resulting in the most strength loss.

Table 6, Figure 23 and Appendix Table 12 displayed the weight change of geopolymer paste based on class F fly ash in 5% of sulfuric acid solution period 18 weeks. It was found that all ratios tended to lose weight from the first week except 6F-60 and 14F-60 specimens which gained weight until after 12 and 9 weeks. All specimens increase weight loss with an increased concentration of NaOH as witnessed earlier.

Table 6 Weight changes of class F fly ash geopolymer specimens exposed to 5% solutions of acetic and sulfuric acids at 18 weeks

Specimens	**Weight loss (%)**	
	Acetic acid	**Sulfuric acid**
6F-50	-9.147	-7.655
10F-50	-9.030	-7.741
14F-50	-12.771	-20.088
6F-55	-7.615	-5.292
10F-55	-5.675	-5.485
14F-55	-8.597	-13.489
6F-60	-5.807	-2.216
10F-60	-4.455	-4.219
14F-60	-5.843	-5.532

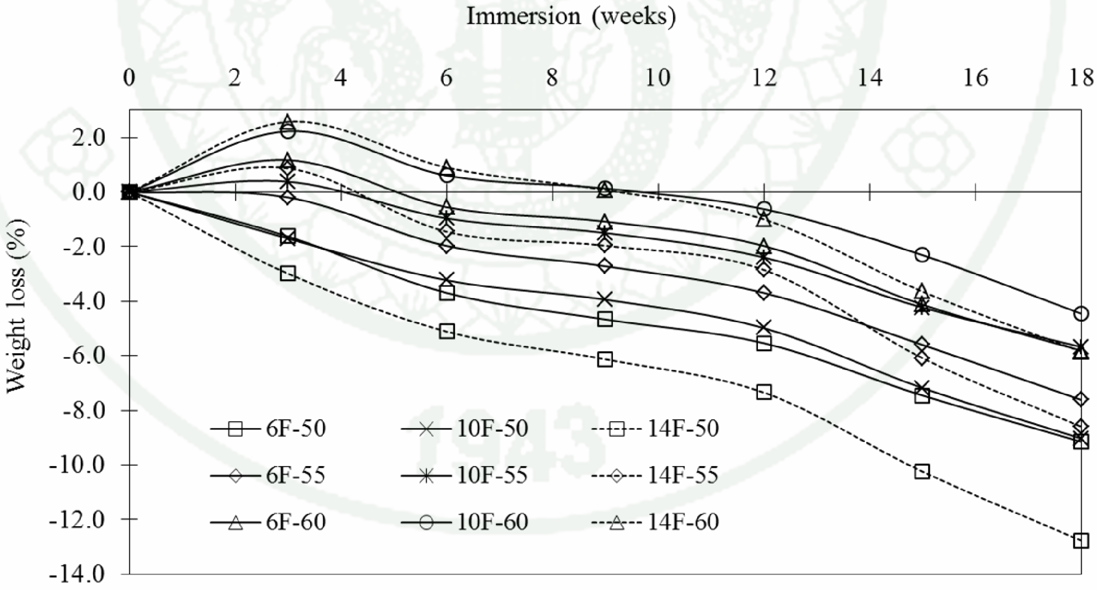


Figure 22 Percentage weight change of class F fly ash geopolymer specimens immersed in 5% of acetic acid solution

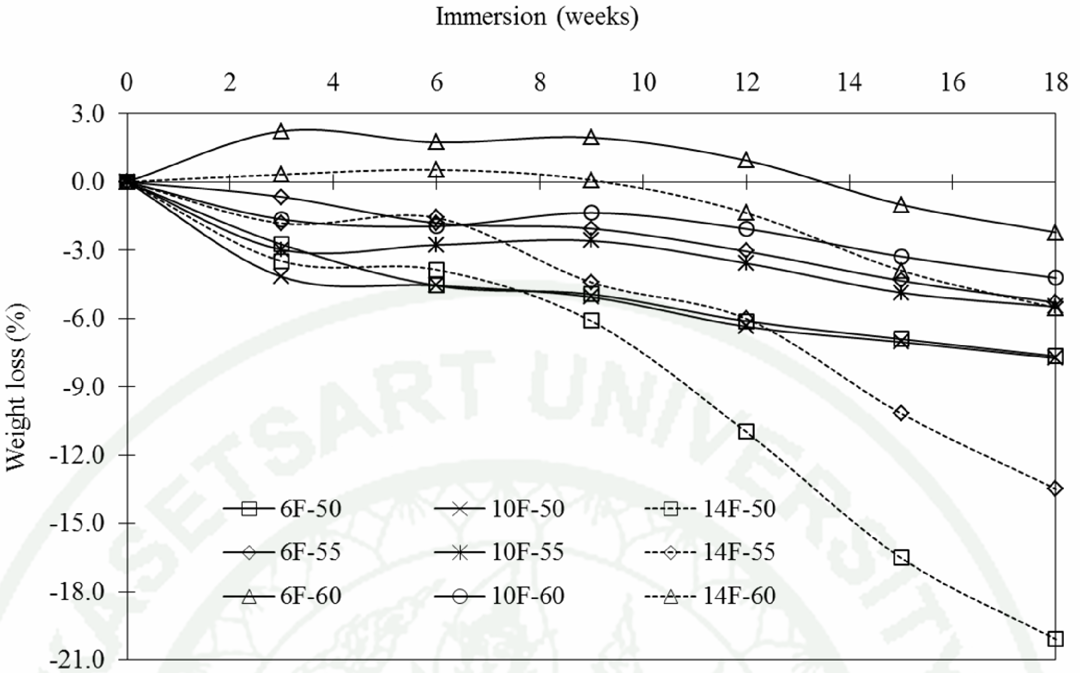


Figure 23 Percentage weight change of class F fly ash geopolymer specimens immersed in 5% of sulfuric acid solution

2.2 Appearance of the specimens after immersion in 5% solutions of acetic acid and sulfuric acid for 18 weeks

Appendix Figure 3 and Figure 4 show that after 18 weeks of exposure to acetic and sulfuric acid solutions, there were very few visual changes for class F fly ash. In sulfuric acid solutions, there were many cracks on the surface and some softening of the surface cover specimens. The appearance of the geopolymer paste was swollen and cracked as explained by Figure 3 and Figure 4. Wallah and Rangan, (2006) confirm the damage and cracks due to exposure to acid solution.

2.3 Average percentage in the compressive strength of the specimens when immersed in 5% solutions of acetic acid and sulfuric acid for 18 weeks

Increasing the NaOH concentration from 6M to 10 M for class F fly ash, the residual compressive strength also increased when soaked in both acetic acid and sulfuric acid. Similar results occurred for specimens in class F-50, F-55 and F-60 as summarized in Figure 24, Appendix Table 7 and Appendix Table 17.

When immersed geopolymer paste strength made of fly ash Class F in 5% solution of acetic acid for 18 weeks, shown in figure 24 and appendix table 7 and 17, it was found that using F-50 specimens and increased concentration of NaOH solution from 6M to 10 M tended to slightly increase the residual compressive strength. When soaking in acetic acid solution, the residual compressive strength increased from 104.74 ksc to 117.44 ksc at proportion of 6F-50 to 10F-50 and strength loss reduced from 64.51% to 30.95% compared with Geopolymer paste strength before being soaked, while concentration of NaOH solution at 14M tended to reduce the residual compressive strength as 34.09 ksc at proportion of 14F-50 and slightly increased strength loss percentage by 65.22%.

In using F-55 specimens and increased concentration of NaOH solution from 6M to 10M displayed the result were the same as using F-50 specimens but it tended to slightly increase the residual compressive strength as specimens of mixture proportion by 6F-55 from 121.72 ksc to 171.59 ksc at proportion of 10F-55 and strength loss reduced from 56.02% to 20.09% compared with geopolymer paste strength before being soaked, whereas increased concentration of NaOH solution from 6M to 14M tended to reduce the residual compressive strength for 35.79 ksc of 14F-55 and strength loss increased by 72.04%.

Using F-60 specimens had result in same direction as with using F-50 and F-55 specimens. It could be said that increased concentration of NaOH solution from 6M to 10M also resulted in increased residual compressive strength as specimens of mixture proportion by 6F-60 which resulted in increased residual compressive strength from 82.08 ksc to 235.06 ksc at proportion by 10F-60 causing the most residual compressive strength and strength loss reduced from 63.74% to 3.26%, which was the least strength loss compared with strength of specimens before soaking. Meanwhile increased concentration of NaOH at 14M tended to increase the residual compressive strength for 212.54 ksc and 41.59% strength loss of 14F-60. The increased fly ash from 50% to 55% and 60% also tended to increase the residual compressive strength as well as strength loss percentage, which tended to reduce when using concentration of NaOH solution for 10M and 14M.

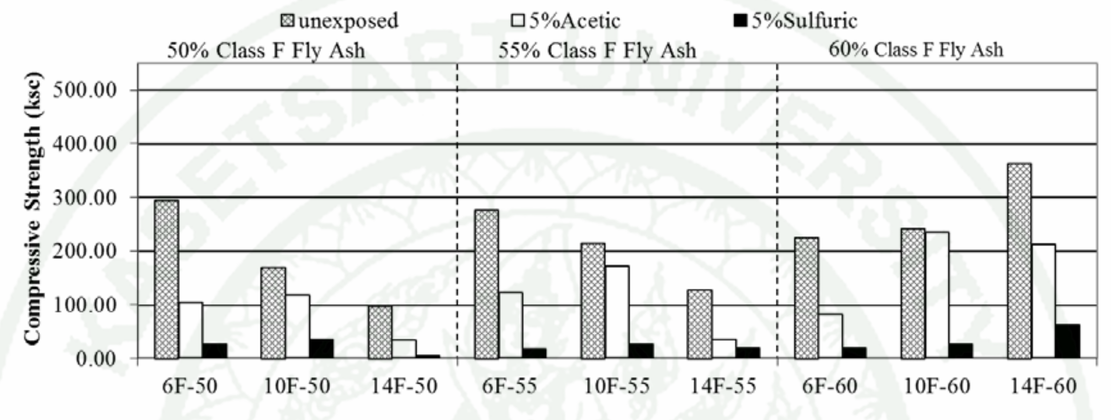
Class F specimens soaked in acetic acid demonstrated the lower losses of strength when using concentration of NaOH at 10M and increasing the percentage of fly ash. The strength loss also decreased, the result which was consistent with the weight loss.

When soaking in 5% solution of sulfuric acid for 18 weeks, shown in figure 24 and appendix table 8 and 14, the results of geopolymer strength of all mixture ratios reduced strength more than when it was soaked in acetic acid solution as in figure 3a. With increased concentration of NaOH solution of F-50 specimens, residual compressive strength was 26.84 ksc, 34.78 ksc and 4.84 ksc and 90.91%, 79.55% and 95.03% strength loss of 6F-50, 10F-50 and 14F-50 respectively.

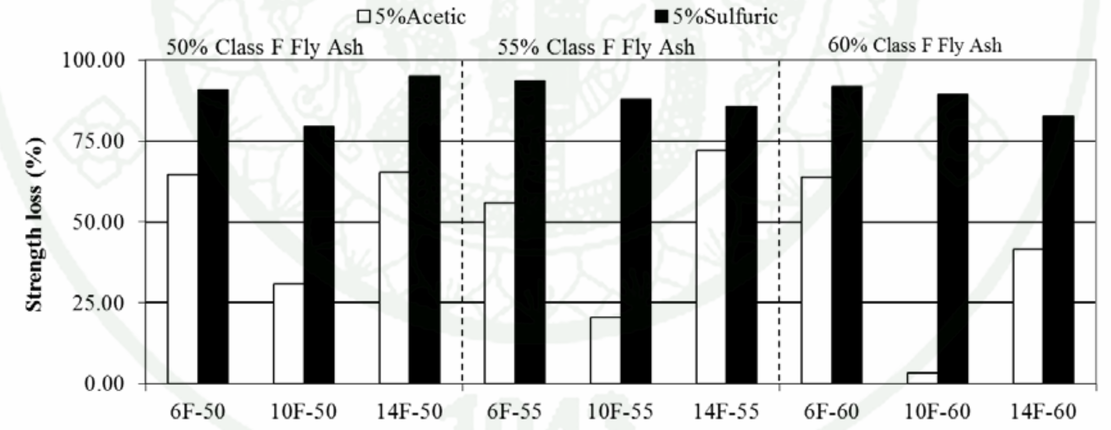
For F-55 specimens, with increased concentration of NaOH, strength loss decreased as shown table 6. The residual compressive strength of 6F-55, 10F-55 and 14F-55 were 17.52 ksc, 26.41 ksc, and 18.22 ksc and 93.67%, 87.79% and 85.76% strength loss respectively.

Using F-60 specimens and increased concentration of NaOH from 6M to 10M and 14M tended to continuously increase the residual compressive strength as specimens of mixture ratios by 6F-60, 10F-60 and 14F-60 with the residual compressive strength for 18.22 ksc , 25.78 ksc and 62.95 ksc respectively including strength loss which tended to continuously be reduced by 91.95%, 89.39% and 82.70% at the mixture ratios by 6F-60, 10F-60 and 14F-60 respectively.

When soaked in sulfuric acid specimens, demonstrated poor durability and experienced strength loss when increasing the NaOH concentration. Strength loss decreased for F-55 and F-60. With F-50, when increasing the NaOH concentration from 6M to 10 M, strength loss decreased, but when increasing the NaOH concentration to 14 M that showed maximum strength loss.



a) Compressive strength of mixture



b) Strength loss of mixture

Figure 24 Change in compressive strength of class F fly ash geopolymer before and after immersed in 5% acetic and sulfuric acid solutions after 18 weeks

2.4 Average percent weight change of the specimens when immersed in solutions of acetic and sulfuric acid for 14 days

Figures 25-26 exhibit the percent weight loss of F-60, F-65 and F-70 geopolymer specimens immersed in acetic and sulfuric acid. The weight decreases for each class C fly ash specimens at different exposure durations. There is a negative relationship with NaOH concentration as observed in Appendix Table 17-18 and confirmed by Wallah and Rangan (2006).

When immersed in acetic acid, the specimens lost less weight when the concentration of NaOH also decreased, which corresponds with Suresh Thokchom et. al. Those with higher alkali content recorded higher weight loss also. Not only did 10F-60 specimens have the lowest weight loss at -3.291%, but they shared similar results of immersed specimens to 5% acetic acid period after 18 weeks.

In sulfuric acid, there were many trends resulting in weight loss. Figure 16 explains that the good acid attack was for F-60 specimens, which had greater lower weight loss than F-50 and F-55 specimens, which corresponds with the previous experiment.

Sreevidya *et al.* (2010) suggested that an acid attack would break Si–O–Al bonds, and increase the number of Si–OH and Al– OH groups in geopolymers. This results in an increased amount of silicic acid ions and dimers in solution because interactions with geopolymers and acid solutions can also cause replacement of the exchangeable cations (Na, K) in polymers by hydrogen or hydronium ions. However, treatment of a geopolymer with a strong acid might result in a direct attack on the aluminosilicate framework and dealumination. The increment of acid concentration also lead to the increment of percentage weight change loss of geopolymer paste.

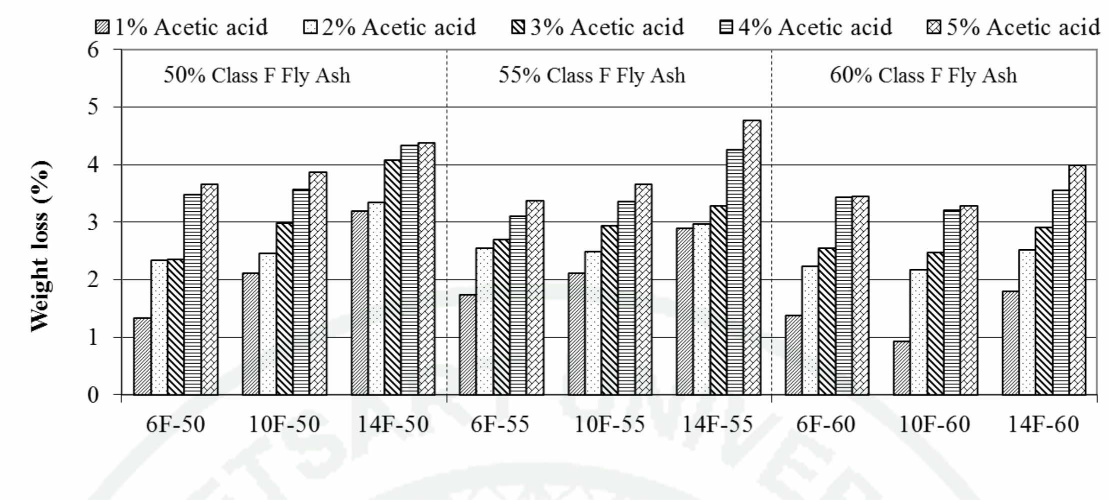


Figure 25 Weight loss of class C fly ash geopolymer after immersed in solutions of acetic acid solution for 14 days

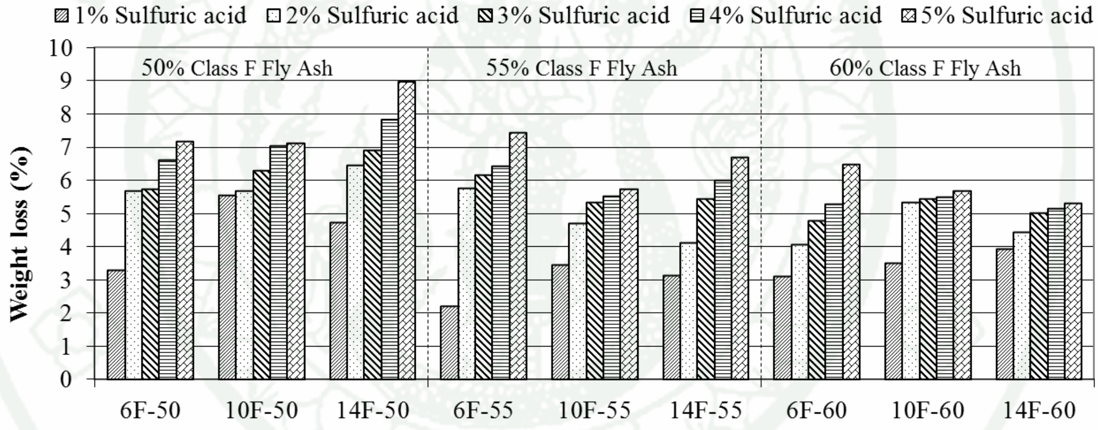


Figure 26 Weight loss of class C fly ash geopolymer after immersed in sulfuric acid solution for 14 days

Part 3 Testing the pore volume intrusion of geopolymer paste before and after immersion in acid solution

The strongest geopolymer pastes after making fly ash occurred at 10M and 14M as summarized in Table 7. The pore volume intrusion aids as a way to measure durability.

Table 7 Ratio mixture of geopolymer paste soaking in 5% solutions of acetic and sulfuric acid solutions after 18 weeks for MIP experimentation

Mix. No.	**Compressive strength (ksc)**	**Weight changes (%)**		**Strength losses (%)**	
		Acetic acid	**Sulfuric acid**	**Acetic acid**	**Sulfuric acid**
10C-70	365.90	-5.38	-14.37	34.45	95.13
14C-70	509.93	-4.71	-7.10	37.86	95.12
10F-60	242.98	-7.01	-3.00	3.26	89.39
14F-60	363.86	-8.54	-7.31	41.59	82.70

1. Pore volume of geopolymer paste specimens made of Class C fly ash

Figure 27 shows connected pore diameters and volume intrusions of geopolymer pastes with varying NaOH concentrations. Before soaking, increased concentration of NaOH solution from 10M to 14M resulted in a reduced rate of cumulative volume intrusion as specimens of mixture ratio by 14C-70 with the increase of volume intrusion lower than specimens of mixture ratio 10C-70, which resulted in more early strength, as displayed in Table 5. After being soaked in acetic and sulfuric acid solutions, it was found that the increase of volume intrusion was higher when compared with specimens before soaking the paste in acid solution. This resulted in an increased pore volume intrusion in sulfuric solution.

Using the concentration of NaOH solution for 10M at mixture ratio by 10C-70 as demonstrated in figure 27(a), it was found that the largest increase of volume intrusion in sulfuric acid solution was 0.433 cc/g. The second largest increase was the volume intrusion in acetic acid solution for 0.291 cc/g when compared with specimens before soaking. The increase of volume intrusion in sulfuric acid solution was lower than acetic acid solution with a pore size higher than 2 μm. Meanwhile since the pores were larger than 7μm, the increase of volume intrusion in sulfuric acid solution was lowest when comparing with specimens before and after soaking in acetic acid solution.

When using concentration of NaOH solution with 14M at mixture ratio by 14C-70, the increase of volume intrusion in sulfuric acid solution was highest for 0.504 cc/g including the increase of volume intrusion in acetic acid solution which was 0.157 cc/g when comparing with specimens before soaking with the increase of volume intrusion for 0.093 cc/g as displayed in figure 27(b). It was found that a pore size was bigger that 4 µm and the increase of volume intrusion in acetic acid solution before soaking was quite similar, but it tended to reduce when comparing with the increase of volume intrusion in sulfuric acid solution.

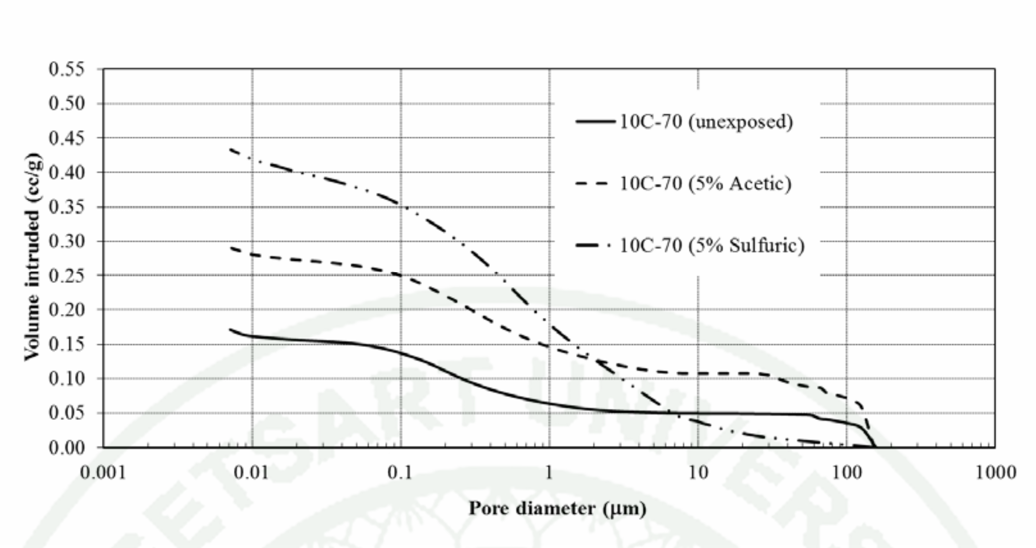
The test results demonstrated that increased concentration of NaOH solution from 10M to 14M resulted in the decrease of volume intrusion in acetic acid solution, but increase of sulfuric acid solution.

Figure 28 shows the total porosity of geopolymer paste made of Class C fly ash. It was found that before soaking specimens with increased concentration of NaOH solutions from 10M to 14M they had reduced total porosity.

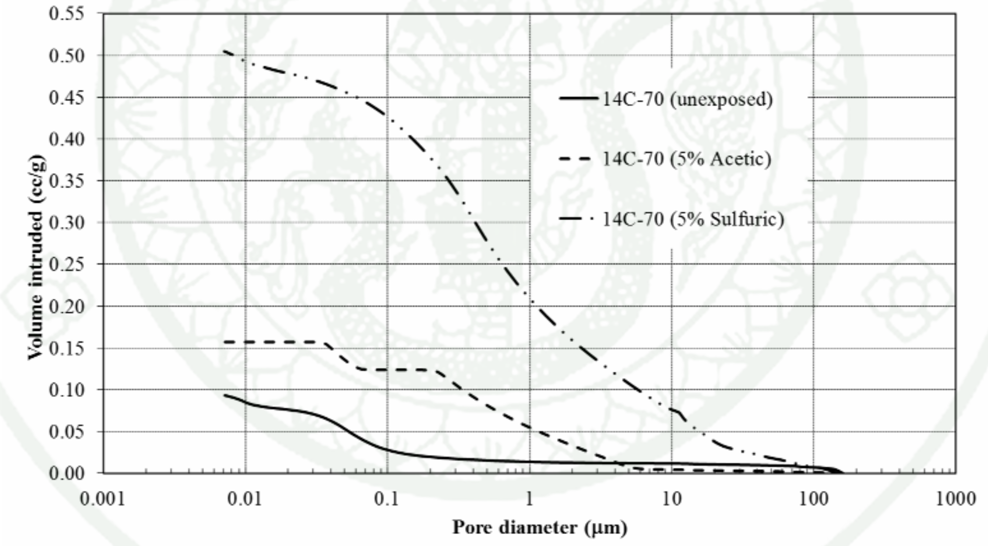
After soaking specimens in acetic acid solution, it was found that specimens of the mixture ratio by 10C-70 reduced total porosity while specimens of the mixture ratio by 14C-70 increased total porosity by 19.88% and 33.67% respectively. Similar results were found for sulfuric acid.

Increasing the concentration of NaOH solution from 10M to 14M resulted in a total porosity increase from 19.88% to 33.67% in acetic and sulfuric acid solutions. It was found that they slightly increased from 42.86% to 45.51%.

The total porosity was lower as displayed in the Figure 28 after the test results. Geopolymer paste soaked in sulfuric acid and increased concentration of NaOH solution from 10M to 14M increased the total porosity and resulted in a similar strength loss percentage.



a) The concentration of NaOH solution is 10M



b) The concentration of NaOH solution is 14M

Figure 27 Volume intruded distribution of the geopolymer class C fly ash before and after 18 weeks in 5% acetic acid and sulfuric acid

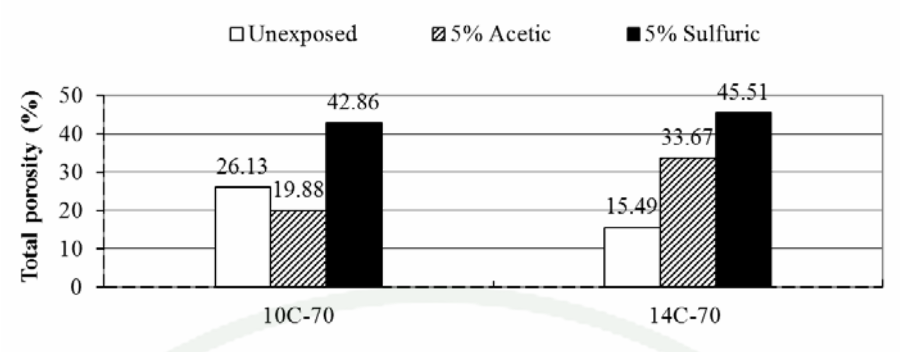


Figure 28 Total porosity of the geopolymer class C fly ash before and after immersed in 5% acetic acid and sulfuric acid after 18 weeks

From the pore analysis of geopolymer paste made of fly ash Class C before and after soaking in solution at the mixture ratio by 10C-70 and 14C-70, it was found that all ratios consisted of capillary pores that ranged from 8.52%-23.27% of total porosity. The second largest pore type was an air void that ranged from 5.00%-15.77% and gel pores were the lowest ranging from 1.97%-6.46% of total porosity, respectively.

Figure 29 displays the separation of the pore diameter of geopolymer paste made of class C fly ash. Geopolymer specimens with lower concentration of solutions had more air void, gel pores, and capillary pores. According to specimens of the mixture ratio by 10C-70, there were more air voids, gel pores, and capillary pores than specimens of the mixture ratio by 14C-70. Capillary pores directly resulted in permeability and had great influence against durability of geopolymer including that it had the most pores compared to others.

After soaking geopolymer in solution, it was found that all diameters of pore had decreased their pore volume intrusion in acetic acid solution but all diameters of pore had the increase of volume intrusion in sulfuric acid solution for specimens of the mixture ratio by 10C-70. Specimens soaked in sulfuric solution increased significantly of volume intrusion.

Increasing concentrations of NaOH solution from 10M to 14M resulted in the volume intrusion increase of capillary pores from 10.19% to 17.75% and 21.77% to 23.27% in acetic and sulfuric acid solutions respectively. Such results conformed to specimens after being soaked in acetic acid solution at the mixture ratio by 10C-70 with lower strength loss percentages than specimens at the mixture ratio by 14C-70. This occured because of lower capillary pore, so there was a property of low permeability as displayed in Table 6. Strength loss percentage was not different for increased NaOH solutions. However, weight loss percentage decreased when increasing concentration of NaOH solution from 10M to 14M. As shown from geopolymer paste at the mixture ratio by 14C-70, weight loss percentage was lower due to the increase of gel pore and slightly increased capillary pore as displayed in Table 6.

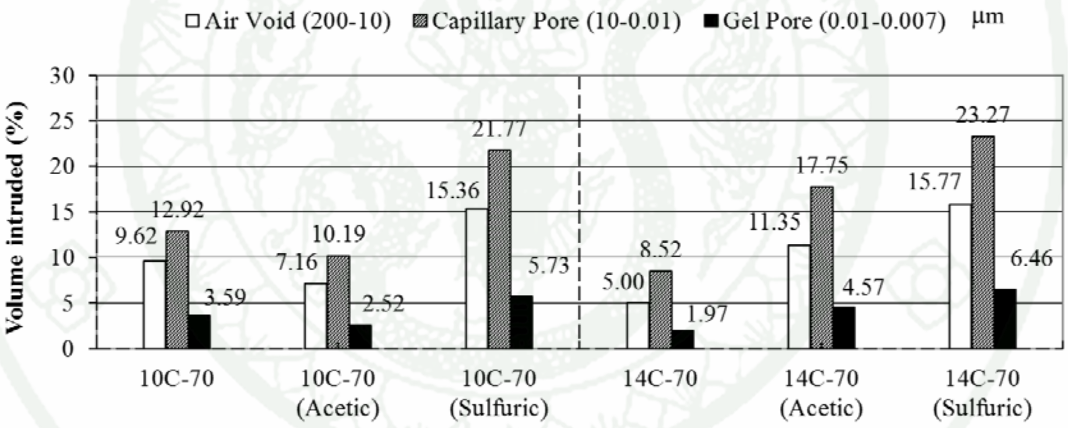


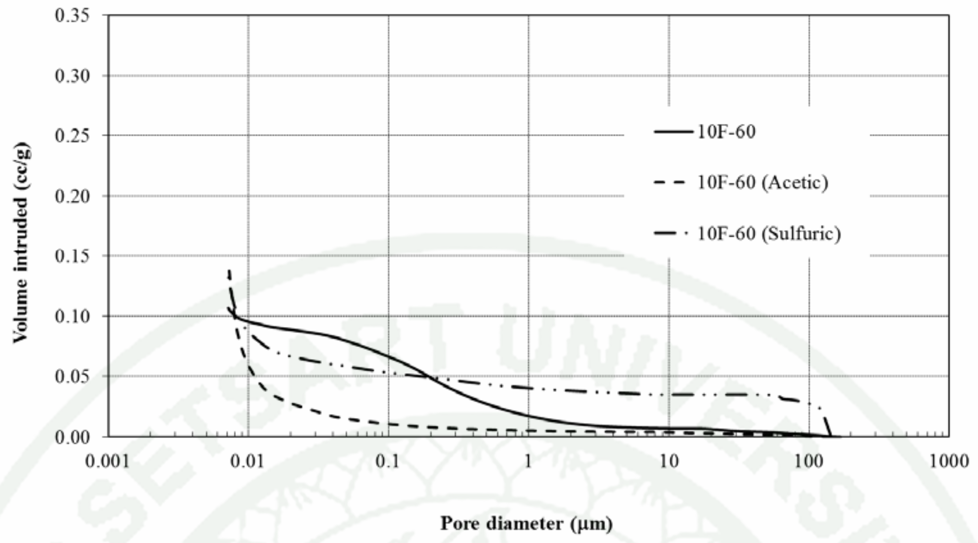
Figure 29 Pore classification of the geopolymer class C fly ash before and after immersed in 5% acetic acid and sulfuric acid after 18 weeks

2. Volume intrusion of Geopolymer paste made of Class F fly ash

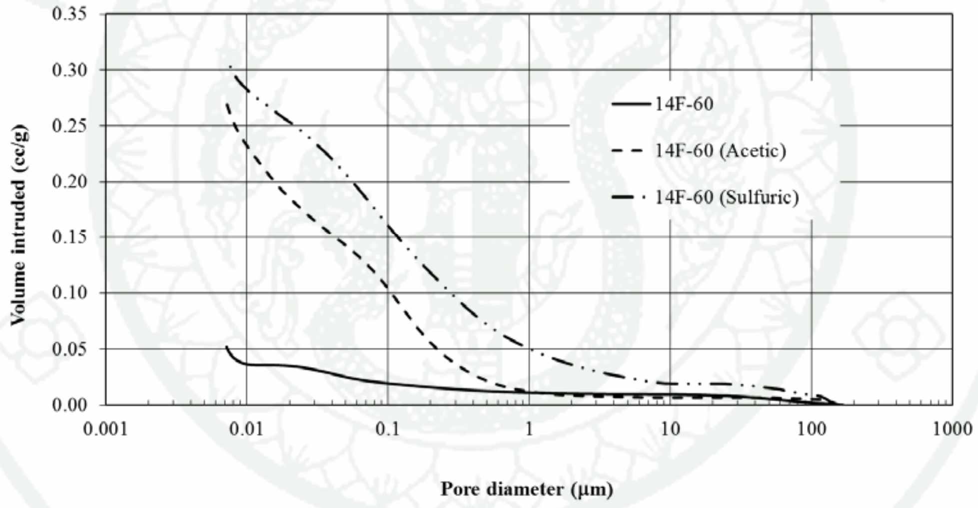
Figure 30 highlights the connection of pore diameter and volume intrusion of Geopolymer paste made of fly ash with increased concentration of NaOH from 10M to 14M. After soaking in acetic and sulfuric acid solutions, it was found that the increase of volume intrusion in sulfuric acid solution was more than in acetic acid solution. For specimens at the mixture ratio by 14F-60, it was found that the increase of volume intrusion in sulfuric acid solution greatly increased by 0.312 cc/g and 0.272 cc/g in sulfuric and acetic acid respectively with a pore smaller than 1 μm when comparing with other specimens before soaking. In addition, it was found that the increase of volume intrusion in acetic acid solution with specimens before soaking was not different when the pore was larger than 1 μm.

Using concentration of NaOH solution for 10M at the mixture ratio by 10F-60, it was found that when a pore diameter was 0.2μm - 0.01μm, specimens before soaking had more increase of volume intrusion than specimens soaked in acetic and sulfuric acid solutions. When a pore was more than 0.2 μm, the increase of volume intrusion in sulfuric acid solution was higher while the increase of volume intrusion in acetic acid solution was lowest. However, when the diameter was smaller than 0.01μm, the increase of volume in acetic acid solution was highest at 0.147 cc/g. The second was the increase of volume intrusion in sulfuric acid solution for 0.136 cc/g and 0.107 cc/g for specimens before soaking.

The test result demonstrated that an increased concentration of NaOH from 10M to 14M. In case of before soaking, it resulted in the increase of volume intrusion being reduced and displayed good result for early strength. After soaking acetic and sulfuric acid solutions, increased concentration of NaOH solution greatly increased volume intrusion and resulted in higher strength loss including weight loss.



a) The concentration of NaOH solution is 10 M



b) The concentration of NaOH solution is 14 M

Figure 30 Volume intruded distribution of the geopolymer class F fly ash before and after immersed in 5% acetic acid and sulfuric acid after 18 weeks

Figure 31 demonstrates the total porosity in geopolymer paste made of Class F. It found an increased concentration of NaOH solution from 10M to 14M resulted in the decrease of total porosity from 17.18% to 7.1% at the mixture ratio.

After soaking in acid solution, it was found that specimens at the mixture ratio by 10F-60 with concentration of NaOH solution for 10M tended to reduce total porosity by 15.98% in acetic solution and 16.22% in sulfuric solution. However, using the concentration of NaOH solution for 14M at the mixture ratio by 14F-60 resulted a higher increase of total porosity for 30.98% and 35.33% in acetic and sulfuric acid solution respectively.

In sulfuric solution, increased concentration of NaOH solution from 10M to 14M tended to increase total porosity more than in acetic acid solution and resulted in the increase of strength loss, but the result of weight loss was fluctuation.

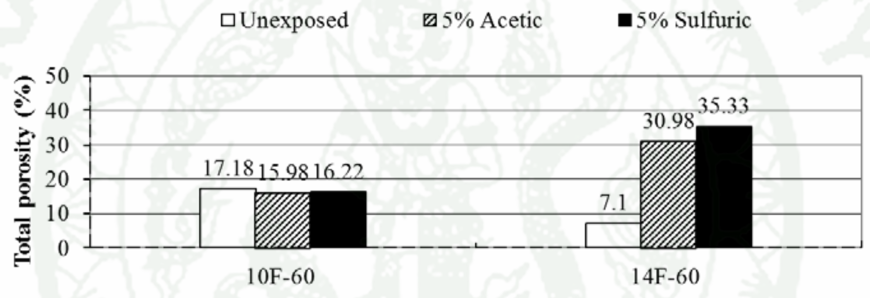


Figure 31 Total porosity of the geopolymer class F fly ash before and after immersed in 5% acetic acid and sulfuric acid after 18 weeks

Figure 32 demonstrated the separation of the pore diameter of geopolymer paste made of fly ash Class F. From the analysis results of pore types in geopolymer paste before and after being soaked in solution at the mixture ratio by 10F-60 and 14F-60, it was found that the air void volume, capillary pore, and gel pore shared the same tendency before and after being soaked. All mixture ratios consisted of capillary pores, which had the most porosity by volume. The second was air void followed by gel pores.

Air void volume, capillary pore, and gel pore had similar volume intrusions when compared with specimens before soaking. For specimens using concentrations of NaOH solution for 14M, it was found that air void volume, capillary pore, and gel pore had higher pore volumes, particularly in sulfuric acid solution which highly increased volume intrusion when comparing with specimens before soaking which resulted in the increase percentage of strength loss and weight loss as displayed in the Table 7.

Increasing the concentration of NaOH solution from 10M to 14M before soaking resulted in the decrease of air void volume, capillary pore, and gel pore from 6.05% to 2.61%, 8.79% to 3.62% and 2.34% to 0.87% respectively. Such results conform to the strength of specimens with a mixture ratio of 14F-60 and higher strength due to the decrease of air void, capillary pore, and gel pore volume as displayed in Table 7. On the contrary, increased concentration of NaOH solution after soaking in solution resulted in the increase of air void volume, capillary pore, and gel pore and the higher strength loss and weight loss.

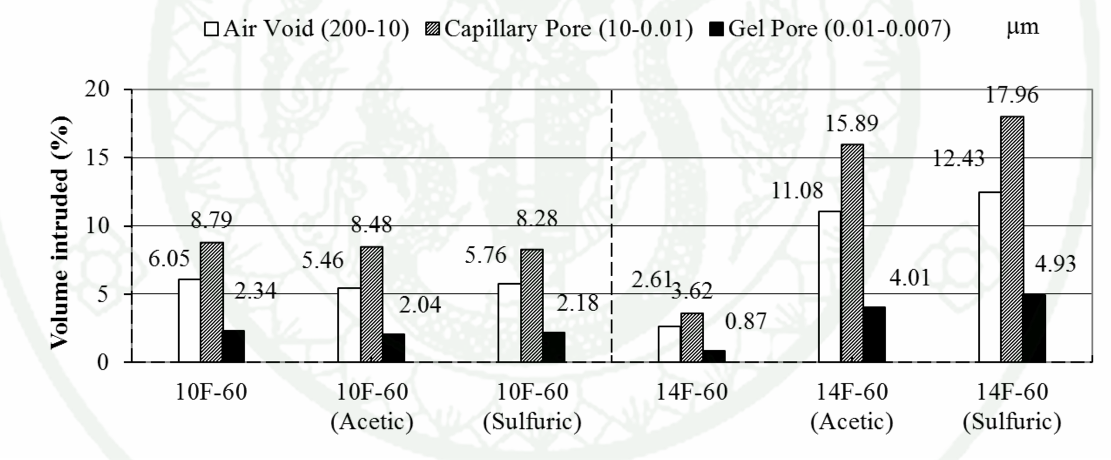


Figure 32 Pore classification of the geopolymer class F fly ash before and after immersed in 5% acetic acid and sulfuric acid after 18 weeks

CONCLUSION AND RECOMMENDATIONS

Conclusion

Based on the previous results, the effect of NaOH solution and fly ash on acid resistance of geopolymer paste depends on the composition NaOH solution and fly ash. The following were concluded:

1. The experimental results of class C and class F fly ash specimens with higher concentration of NaOH solution results in higher compressive strength of geopolymer specimens and the increased content of fly ash, and compressive strength also rose with a variable SiO_2/Al_2O_3 ratio. All samples increased except F-50 and F-55 specimens which showed the opposite trend.

2. Specimens showed deterioration, corrosion, and cracks when observed for visual damage after immersion in acid.

3. In terms of durability, the mixture ratios of 14C-70 made using class C fly ash-based geopolymer specimens had the most strength for acetic and sulfuric acid. It still had substantial residual compressive strength.

4. The investigation of durability of Class F fly ash-based geopolymer paste found lower concentrations of NaOH to be more resistant than higher concentrations of NaOH solution. The 10F-60 specimens performed well in terms of weight loss and strength loss in acetic acid and F-60 specimens group exhibited good durability for weight loss but was not very successful in achieving high strength coefficients.

5. The addition of NaOH concentration to fly ash based geopolymers improves the early strength, which reduces total porosity before soaking in acid. But the addition of NaOH concentration after soaking in acetic and sulfuric acid solutions associate with difference of the pore diameter and total porosity which had greater durability for both acids leading to an increase in strength and weigh loss.

Recommendations

1. In the future, the test may be suggested to use concentration of acid less than 5% because specimens collapse faster than a reasonable time.

2. The test should be concerned about water to geopolymer solids ratio by mass and micro-cracks of geopolymer to understand more clearly about the durability.

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APPENDICES

Appendix A

Tables

Appendix Table A1 Mixture composition in terms of molar ratio of class C fly ash specimens

Specimens	Concentration of NaOH	Molar ratio			
	(Molar)	SiO_2/Al_2O_3	Na_2O/SiO_2	Na_2O/Al_2O_3	sol/FA
6C-60	6	3.92	0.24	0.95	0.67
10C-60	10	3.92	0.33	1.3	0.67
14C-60	14	3.92	0.42	1.66	0.67
6C-65	6	3.74	0.21	0.79	0.54
10C-65	10	3.74	0.29	1.08	0.54
14C-65	14	3.74	0.36	1.36	0.54
6C-70	6	3.59	0.18	0.65	0.43
10C-70	10	3.59	0.25	0.88	0.43
14C-70	14	3.59	0.31	1.11	0.43

Appendix Table A2 Mixture composition in terms of molar ratio of class F fly ash specimens

Specimens	Concentration of NaOH	Molar ratio			
	(Molar)	SiO_2/Al_2O_3	Na_2O/SiO_2	Na_2O/Al_2O_3	sol/FA
6F-50	6	6.11	0.17	1.07	1.00
10F-50	10	6.11	0.25	1.51	1.00
14F-50	14	6.11	0.32	1.96	1.00
6F-55	6	5.90	0.15	0.88	0.82
10F-55	10	5.90	0.21	1.24	0.82
14F-55	14	5.90	0.27	1.61	0.82
6F-60	6	5.72	0.13	0.72	0.67
10F-60	10	5.72	0.18	1.02	0.67
14F-60	14	5.72	0.23	1.32	0.67

Appendix Table A3 The compressive strength of class C fly ash at 28 days

Symbol	Sample no.	Height (cm.)	Dai. (cm.)	Weight (g.)	Area (cm^2)	Load (kN.)	Compressive strength (ksc.)	Average (ksc.)
6C-60	1	7.175	4.300	181.50	14.52	30.52	214.29	212.54
	2	7.556	4.320	192.50	14.66	30.60	212.87	
	3	7.024	4.334	177.50	14.75	30.45	210.46	
10C-60	1	7.392	4.355	190.50	14.90	38.24	261.76	262.92
	2	7.259	4.300	185.00	14.52	38.24	268.46	
	3	7.502	4.378	196.00	15.05	38.17	258.54	
14C-60	1	7.450	4.287	201.00	14.43	47.25	333.77	326.15
	2	7.570	4.353	204.00	14.88	47.18	323.25	
	3	7.669	4.349	207.50	14.85	46.83	321.44	
6C-65	1	7.800	4.325	200.50	14.69	40.49	281.02	284.33
	2	7.500	4.269	189.00	14.31	40.45	288.15	
	3	7.820	4.295	200.00	14.49	40.33	283.83	
10C-65	1	7.733	4.390	207.00	15.14	49.40	332.78	342.15
	2	7.654	4.291	204.50	14.46	47.90	337.73	
	3	7.716	4.313	206.50	14.61	51.00	355.93	
14C-65	1	7.357	4.381	203.50	15.07	54.84	370.91	371.89
	2	7.477	4.355	204.00	14.90	54.44	372.65	
	3	7.154	4.371	196.00	15.01	54.76	372.10	
6C-70	1	7.682	4.362	202.50	14.94	51.42	350.85	345.25
	2	7.502	4.364	199.50	14.96	50.25	342.55	
	3	7.833	4.350	207.50	14.86	49.90	342.36	
10C-70	1	7.535	4.338	202.50	14.78	53.19	366.92	365.90
	2	7.558	4.339	208.50	14.79	52.89	364.69	
	3	7.646	4.338	206.50	14.78	53.07	366.09	
14C-70	1	7.530	4.368	210.00	14.98	79.30	539.59	509.93
	2	7.417	4.305	205.50	14.56	62.70	439.22	
	3	7.270	4.366	203.00	14.97	80.90	550.98	

Appendix Table A4 The compressive strength of class F fly ash at 28 days

Symbol	Sample no.	Height (cm.)	Dai. (cm.)	Weight (g.)	Area (cm^2)	Load (kN.)	Compressive strength (ksc.)	Average (ksc.)
6F-50	1	7.185	4.090	409.00	13.14	37.87	293.90	295.15
	2	7.004	4.102	410.20	13.22	38.23	294.96	
	3	7.042	4.064	406.40	12.97	37.73	296.58	
10F-50	1	6.768	3.810	381.00	11.40	19.90	177.98	170.08
	2	6.883	3.785	378.50	11.25	20.80	188.49	
	3	6.874	3.782	378.20	11.23	15.84	143.77	
14F-50	1	7.018	3.787	378.70	11.26	10.95	99.12	98.01
	2	6.660	3.795	379.50	11.31	10.77	97.08	
	3	7.310	3.807	380.70	11.38	10.92	97.82	
6F-55	1	7.126	4.057	405.70	12.93	36.33	286.59	276.76
	2	7.231	4.060	406.00	12.95	34.78	273.93	
	3	7.372	4.054	405.40	12.91	34.15	269.76	
10F-55	1	7.459	4.087	408.70	13.12	24.70	191.97	216.26
	2	7.605	4.113	411.30	13.29	27.70	212.58	
	3	7.504	4.108	410.80	13.25	31.75	244.23	
14F-55	1	7.514	4.098	409.80	13.19	16.16	124.93	127.98
	2	7.317	4.110	411.00	13.27	16.30	125.27	
	3	7.513	4.119	411.90	13.33	17.48	133.74	
6F-60	1	7.406	4.092	409.20	13.15	29.46	228.42	226.39
	2	7.557	4.098	409.80	13.19	28.54	220.63	
	3	7.455	4.083	408.30	13.09	29.55	230.12	
10F-60	1	7.770	4.112	411.20	13.28	33.15	254.52	242.98
	2	7.702	4.114	411.40	13.29	31.91	244.74	
	3	7.702	4.111	411.10	13.27	29.90	229.69	
14F-60	1	7.639	4.105	410.50	13.23	49.65	382.51	363.86
	2	7.694	4.129	412.90	13.39	48.00	365.52	
	3	7.541	4.142	414.20	13.47	45.40	343.55	

Appendix Table A5 The compressive strength of class C fly ash at 28 days after immersion in 5% of acetic acid after 18 weeks

Symbol	Sample no.	Height (cm.)	Dai. (cm.)	Weight (g.)	Area (cm^2)	Load (kN.)	Compressive strength (ksc.)	Average (ksc.)
6C-60	1	8.018	4.351	435.10	14.87	26.11	179.06	168.31
	2	7.767	4.355	435.50	14.90	23.60	161.54	
	3	7.865	4.318	431.80	14.64	23.60	164.33	
10C-60	1	7.774	4.275	427.50	14.35	15.35	109.01	88.69
	2	7.945	4.355	435.50	14.90	9.70	66.40	
	3	7.863	4.321	432.10	14.66	13.04	90.67	
14C-60	1	7.846	4.353	435.30	14.88	-	-	0.00
	2	7.987	4.459	445.90	15.62	-	-	
	3	8.091	4.980	498.00	19.48	-	-	
6C-65	1	7.602	4.304	430.40	14.55	24.45	171.37	168.87
	2	7.327	4.368	436.80	14.98	23.48	159.78	
	3	7.622	4.301	430.10	14.53	25.00	175.45	
10C-65	1	7.826	4.341	434.10	14.80	37.42	257.83	175.28
	2	7.626	4.332	433.20	14.74	20.50	141.82	
	3	7.600	4.339	433.90	14.79	18.30	126.19	
14C-65	1	8.112	5.011	501.10	19.72	-	-	0.00
	2	8.234	5.213	521.30	21.34	-	-	
	3	8.456	5.331	533.10	22.32	-	-	
6C-70	1	7.932	4.378	437.80	15.05	30.18	204.44	229.44
	2	7.600	4.370	437.00	15.00	38.10	259.01	
	3	7.945	4.345	434.50	14.83	32.70	224.87	
10C-70	1	7.757	4.309	430.90	14.58	38.03	265.88	239.84
	2	7.866	4.351	435.10	14.87	30.00	205.73	
	3	7.880	4.372	437.20	15.01	36.50	247.91	
14C-70	1	7.908	4.278	427.80	14.37	35.50	251.83	316.89
	2	7.953	4.321	432.10	14.66	52.00	361.57	
	3	7.994	4.334	433.40	14.75	48.80	337.29	

Appendix Table A6 The compressive strength of class C fly ash at 28 days after immersion in 5% of sulfuric acid after 18 weeks

Symbol	Sample no.	Height (cm.)	Dai. (cm.)	Weight (g.)	Area (cm^2)	Load (kN.)	Compressve strength (ksc.)	Average (ksc.)
6C-60	1	7.31	5.20	185.00	21.27	1.20	5.75	4.60
	2	7.54	5.28	192.50	21.88	1.02	4.74	
	3	7.53	5.24	189.50	21.55	0.70	3.31	
10C-60	1	8.31	5.11	224.50	20.51	-	-	0.00
	2	8.62	5.21	242.30	21.34	-	-	
	3	8.83	5.23	253.40	21.47	-	-	
14C-60	1	8.45	5.01	235.50	19.73	-	-	0.00
	2	8.55	5.01	223.10	19.71	-	-	
	3	8.75	5.03	245.80	19.84	-	-	
6C-65	1	7.36	5.09	192.50	20.37	1.28	6.40	7.60
	2	7.38	5.29	203.50	21.96	2.20	10.21	
	3	7.18	5.23	190.50	21.44	1.30	6.18	
10C-65	1	7.69	4.95	215.00	19.28	5.08	26.88	35.27
	2	8.18	4.96	228.00	19.28	6.30	33.31	
	3	8.20	4.98	229.00	19.45	8.70	45.62	
14C-65	1	7.55	5.30	222.30	22.08	2.50	11.54	10.61
	2	6.53	5.37	174.50	22.61	1.70	7.67	
	3	7.47	5.34	218.00	22.37	2.77	12.62	
6C-70	1	8.49	5.15	233.50	20.80	2.50	12.26	16.60
	2	8.50	5.20	239.00	21.22	4.03	19.36	
	3	8.88	5.21	242.50	21.30	3.80	18.19	
10C-70	1	8.35	4.90	243.00	18.82	3.50	18.96	17.83
	2	7.93	4.95	243.50	19.27	3.30	17.46	
	3	8.53	4.94	246.50	19.13	3.20	17.06	
14C-70	1	8.22	5.01	238.50	19.74	5.00	25.83	24.86
	2	8.18	5.00	242.50	19.63	3.66	19.02	
	3	8.08	5.03	234.50	19.89	5.80	29.73	

Appendix Table A7 The compressive strength of class F fly ash at 28 days after immersion in 5% of acetic acid after 18 weeks

Symbol	Sample no.	Height (cm.)	Dai. (cm.)	Weight (g.)	Area (cm^2)	Load (kN.)	Compressve strength (ksc.)	Average (ksc.)
6F-50	1	7.34	4.33	163.50	14.70	11.40	79.08	104.74
	2	7.37	4.32	163.00	14.64	16.40	114.19	
	3	7.48	4.32	166.00	14.63	17.35	120.94	
10F-50	1	7.61	4.34	175.50	14.77	19.32	133.33	117.44
	2	7.50	4.32	171.00	14.64	16.10	112.10	
	3	7.53	4.33	172.00	14.69	15.40	106.88	
14F-50	1	7.55	4.35	174.50	14.85	4.10	28.16	34.09
	2	7.45	4.37	170.00	15.01	4.70	31.92	
	3	7.60	4.33	175.00	14.75	6.10	42.19	
6F-55	1	7.53	4.35	173.00	14.85	20.50	140.78	121.72
	2	7.86	4.32	180.50	14.64	19.10	133.06	
	3	7.84	4.30	180.50	14.52	13.00	91.32	
10F-55	1	7.45	4.37	178.50	14.98	22.00	149.77	171.95
	2	7.71	4.30	183.00	14.52	25.90	181.82	
	3	7.74	4.32	179.50	14.66	26.50	184.26	
14F-55	1	7.52	4.36	187.50	14.95	12.03	82.02	82.08
	2	7.57	4.29	166.00	14.45	13.30	93.82	
	3	7.78	4.34	188.00	14.77	10.20	70.40	
6F-60	1	7.77	4.34	184.50	14.78	17.10	117.97	82.08
	2	7.70	4.36	185.50	14.94	11.75	80.23	
	3	7.39	4.32	176.00	14.64	6.90	48.04	
10F-60	1	7.59	4.35	188.00	14.87	39.05	267.80	235.06
	2	7.69	4.34	188.50	14.76	33.12	228.83	
	3	7.57	4.33	186.50	14.71	30.09	208.55	
14F-60	1	7.70	4.34	196.00	14.81	36.40	250.54	212.54
	2	7.72	4.33	198.00	14.75	34.22	236.61	
	3	7.72	4.31	197.00	14.57	21.50	150.47	

Appendix Table A8 The compressive strength of class F fly ash at 28 days after immersion in 5% of sulfuric acid after 18 weeks

Symbol	Sample no.	Height (cm.)	Dai. (cm.)	Weight (g.)	Area (cm^2)	Load (kN.)	Compressve strength (ksc.)	Average (ksc.)
6F-50	1	7.43	4.35	170.50	14.84	3.90	26.81	26.84
	2	7.46	4.37	172.50	15.03	3.91	26.56	
	3	7.46	4.35	171.50	14.84	3.95	27.15	
10F-50	1	7.69	4.37	178.00	14.97	4.40	29.97	34.78
	2	7.42	4.35	170.00	14.86	5.00	34.30	
	3	7.37	4.29	170.50	14.47	5.69	40.07	
14F-50	1	8.03	4.64	178.00	16.88	0.70	4.23	4.87
	2	8.01	4.67	179.00	17.14	0.91	5.43	
	3	8.00	4.58	167.00	16.47	0.80	4.95	
6F-55	1	7.82	4.49	188.00	15.83	3.25	20.91	17.52
	2	7.85	4.45	187.50	15.57	2.36	15.43	
	3	7.47	4.35	197.00	14.87	2.37	16.22	
10F-55	1	7.61	4.44	185.50	15.46	3.72	24.56	26.41
	2	7.66	4.43	185.50	15.42	4.41	29.13	
	3	7.77	4.38	187.50	15.09	3.78	25.54	
14F-55	1	8.02	4.61	199.50	16.69	2.60	15.88	18.22
	2	7.84	4.60	192.00	16.58	3.39	20.84	
	3	8.07	4.66	199.00	17.06	3.00	17.94	
6F-60	1	7.63	4.39	184.00	15.15	2.64	17.77	18.22
	2	8.00	4.53	193.50	16.08	2.82	17.88	
	3	8.11	4.34	193.50	14.79	2.76	19.02	
10F-60	1	7.92	4.48	197.50	15.73	3.80	24.64	25.78
	2	7.61	4.59	192.50	16.54	4.75	29.27	
	3	7.67	4.53	193.00	16.10	3.70	23.44	
14F-60	1	7.87	4.49	202.00	15.82	9.78	63.01	62.95
	2	7.99	4.54	205.00	16.19	9.59	60.43	
	3	7.94	4.51	202.50	15.94	10.23	65.41	

Appendix Table A9 Weight change of specimens for class C fly ash after immersion in 5% of acetic acid period 18 weeks

Specimens	**Weight loss (%)**						
	0	**3**	**6**	**9**	**12**	**15**	**18**
6C-60	0.000	-0.552	-2.690	-5.355	-6.996	-8.741	-11.988
10C-60	0.000	-1.462	-4.979	-8.774	-10.518	-12.062	-14.699
14C-60	0.000	-1.904	-5.294	-8.573	-11.794	-100.000	-100.000
6C-65	0.000	-0.224	-1.919	-4.076	-5.469	-7.646	-11.362
10C-65	0.000	-0.560	-2.620	-4.509	-5.762	-7.233	-10.532
14C-65	0.000	1.235	-2.257	-100.000	-100.000	-100.000	-100.000
6C-70	0.000	0.359	-0.249	-1.402	-2.082	-3.044	-6.424
10C-70	0.000	0.402	0.028	-0.720	-1.403	-2.368	-5.382
14C-70	0.000	0.607	0.539	-0.235	-0.774	-1.770	-4.712

Appendix Table A10 Weight change of specimens for class C fly ash after immersion in 5% of sulfuric acid period 18 weeks

Specimens	**Weight loss (%)**						
	0	**3**	**6**	**9**	**12**	**15**	**18**
6C-60	0.000	0.558	4.048	5.451	0.159	-100.000	-100.000
10C-60	0.000	1.015	3.864	3.734	1.405	-100.000	-100.000
14C-60	0.000	1.610	4.735	-100.000	-100.000	-100.000	-100.000
6C-65	0.000	2.562	5.079	8.865	6.148	5.539	-100.000
10C-65	0.000	1.632	3.774	4.260	6.058	5.455	-14.505
14C-65	0.000	2.089	5.351	-100.000	-100.000	-100.000	-100.000
6C-70	0.000	3.322	9.036	10.937	13.304	0.965	-100.000
10C-70	0.000	2.378	7.697	8.465	10.633	7.799	-14.375
14C-70	0.000	2.217	5.601	7.609	9.740	8.436	-7.096

Appendix Table A11 Weight change of specimens for class F fly ash after immersion in 5% of acetic acid period 18 weeks

Specimens	Weight loss (%)						
	0	3	6	9	12	15	18
6C-60	0.000	-1.626	-3.696	-4.666	-5.535	-7.453	-9.147
10C-60	0.000	-1.702	-3.231	-3.940	-4.984	-7.183	-9.030
14C-60	0.000	-2.989	-5.111	-6.127	-7.345	-10.244	-12.771
6C-65	0.000	-0.196	-1.971	-2.716	-3.695	-5.599	-7.615
10C-65	0.000	0.378	-0.964	-1.498	-2.394	-4.234	-5.675
14C-65	0.000	0.868	-1.452	-1.965	-2.849	-6.083	-8.597
6C-70	0.000	1.171	-0.554	-1.081	-1.972	-4.124	-5.807
10C-70	0.000	2.228	0.603	0.133	-0.623	-2.309	-4.455
14C-70	0.000	2.572	0.897	0.074	-0.996	-3.647	-5.843

Appendix Table A12 Weight change of specimens for class F fly ash after immersion in 5% of sulfuric acid period 18 weeks

Specimens	Weight loss (%)						
	0	3	6	9	12	15	18
6C-60	0.000	-2.751	-4.559	-4.938	-6.115	-6.902	-7.655
10C-60	0.000	-4.166	-4.553	-5.060	-6.361	-7.036	-7.741
14C-60	0.000	-3.480	-3.859	-6.114	-10.983	-16.513	-20.088
6C-65	0.000	-0.661	-1.812	-2.048	-3.050	-4.344	-5.292
10C-65	0.000	-2.975	-2.768	-2.588	-3.574	-4.865	-5.485
14C-65	0.000	-1.818	-1.575	-4.414	-6.014	-10.169	-13.489
6C-70	0.000	2.221	1.734	1.945	0.943	-1.002	-2.216
10C-70	0.000	-1.642	-1.947	-1.358	-2.060	-3.279	-4.219
14C-70	0.000	0.323	0.530	0.081	-1.364	-3.895	-5.532

Appendix Table A13 Compressive strength change of specimens for class C fly ash after immersion in 5% of acetic and sulfuric acid after 18 weeks

Specimens	Compressive strength (ksc)			strength loss (%)	
	before	acetic	sulfuric	acetic	sulfuric
6C-60	212.54	168.31	4.60	20.81	97.83
10C-60	262.92	88.69	0.00	66.27	100.00
14C-60	326.15	0.00	0.00	100.00	100.00
6C-65	284.33	168.87	7.60	40.61	97.33
10C-65	342.15	175.28	35.27	48.77	89.69
14C-65	371.89	0.00	10.61	100.00	97.15
6C-70	345.25	229.44	16.60	33.54	95.19
10C-70	365.90	239.84	17.83	34.45	95.13
14C-70	509.93	316.89	24.86	37.86	95.12

Appendix Table A14 Compressive strength change of specimens for class F fly ash after immersion in 5% of acetic and sulfuric acid after 18 weeks

Specimens	Compressive strength (ksc)			strength loss (%)	
	before	acetic	sulfuric	acetic	sulfuric
6F-50	295.15	104.74	26.84	64.51	90.91
10F-50	170.08	117.44	34.78	30.95	79.55
14F-50	98.01	34.09	4.87	65.22	95.03
6F-55	276.76	121.72	17.52	56.02	93.67
10F-55	216.26	171.95	26.41	20.49	87.79
14F-55	127.98	35.79	18.22	72.04	85.76
6F-60	226.39	82.08	18.22	63.74	91.95
10F-60	242.98	235.06	25.78	3.26	89.39
14F-60	363.86	212.54	62.95	41.59	82.70

Appendix Table A15 Weight change of specimens for class C fly ash after immersion in various concentrations of acetic acid at 14 days

Specimens	Weight loss (%)				
	1%	2%	3%	4%	5%
6C-60	-1.703	-2.728	-3.609	-5.314	-6.137
10C-60	-1.407	-2.631	-3.370	-4.338	-4.563
14C-60	-1.296	-1.765	-2.875	-3.736	-4.367
6C-65	-1.202	-2.811	-4.005	-6.993	-8.288
10C-65	-1.113	-2.459	-2.757	-3.862	-5.242
14C-65	-1.089	-2.857	-3.064	-3.379	-3.751
6C-70	-1.553	-2.865	-3.979	-5.655	-6.391
10C-70	-1.593	-2.158	-2.797	-5.393	-8.079
14C-70	-1.513	-2.458	-2.722	-3.399	-3.737

Appendix Table A16 Weight change of specimens for class C fly ash after immersion in various concentrations of sulfuric acid at 14 days

Specimens	Weight loss (%)				
	1%	2%	3%	4%	5%
6C-60	-3.628	-4.604	-6.530	-12.519	-12.718
10C-60	-3.711	-5.649	-6.428	-7.644	-19.369
14C-60	-4.247	-4.739	-5.711	-6.739	-11.722
6C-65	-4.818	-4.505	-4.874	-11.526	-12.365
10C-65	-5.206	-7.018	-7.909	-8.698	-13.923
14C-65	-4.913	-5.321	-5.934	-6.337	-7.864
6C-70	-4.917	-4.912	-5.842	-7.454	-9.729
10C-70	-3.412	-3.693	-4.798	-7.176	-10.583
14C-70	-4.210	-5.124	-5.167	-5.480	-7.579

Appendix Table A17 Weight change of specimens for class F fly ash after immersion in various concentrations of acetic acid at 14 days

Specimens	Weight loss (%)				
	1%	2%	3%	4%	5%
6F-50	-1.341	-2.336	-2.360	-3.487	-3.660
10F-50	-2.119	-2.465	-2.988	-3.572	-3.871
14F-50	-3.197	-3.345	-4.076	-4.335	-4.386
6F-55	-1.737	-2.550	-2.696	-3.100	-3.374
10F-55	-2.119	-2.493	-2.943	-3.354	-3.661
14F-55	-2.890	-2.969	-3.283	-4.267	-4.765
6F-60	-1.378	-2.232	-2.554	-3.432	-3.450
10F-60	-0.931	-2.181	-2.472	-3.210	-3.291
14F-60	-1.795	-2.514	-2.917	-3.551	-3.984

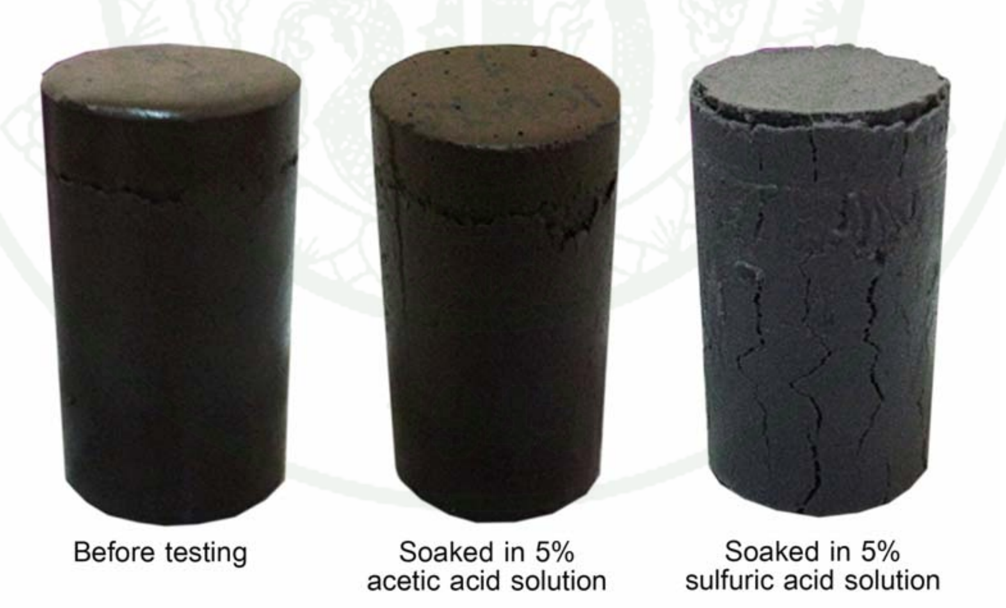
Appendix Table A18 Weight change of specimens for class F fly ash after immersion in various concentrations of sulfuric acid at 14 days

Specimens	Weight loss (%)				
	1%	2%	3%	4%	5%
6F-50	-3.285	-5.693	-5.735	-6.619	-7.167
10F-50	-5.551	-5.696	-6.290	-7.041	-7.126
14F-50	-4.723	-6.453	-6.913	-7.856	-8.979
6F-55	-2.186	-5.768	-6.162	-6.428	-7.434
10F-55	-3.436	-4.695	-5.347	-5.518	-5.740
14F-55	-3.121	-4.123	-5.442	-6.006	-6.697
6F-60	-3.098	-4.072	-4.787	-5.287	-6.483
10F-60	-3.496	-5.343	-5.456	-5.498	-5.678
14F-60	-3.922	-4.444	-5.016	-5.160	-5.302

Appendix B

Figures

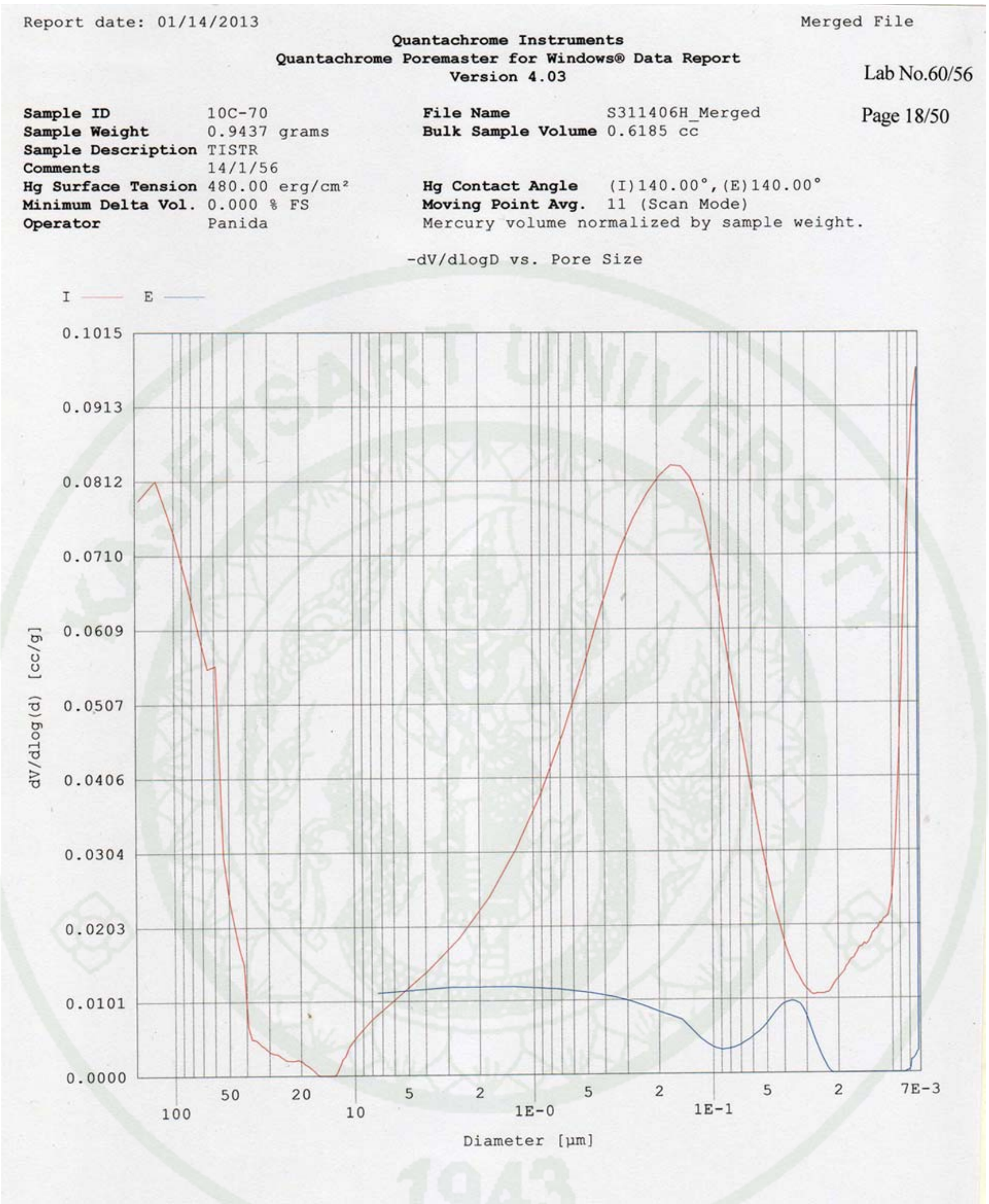
Appendix Figure B1 Visual appearance of 10C-70 geopolymer paste for class C fly ash before and after exposure to 5% solutions of acetic and sulfuric acids after 18 weeks



Appendix Figure B2 Visual appearance of 14C-70 geopolymer paste for class C fly ash before and after exposure to 5% solutions of acetic and sulfuric acids after 18 weeks.

Appendix Figure B3 Visual appearance of 10F-60 geopolymer paste for class F fly ash before and after exposure to 5% solutions of acetic and sulfuric acids after 18 weeks.

Appendix Figure B4 Visual appearance of 14F-60 geopolymer paste for class F fly ash before and after exposure to 5% solutions of acetic and sulfuric acids after 18 weeks.



Report date: 01/14/2013 Merged File

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Version 4.03

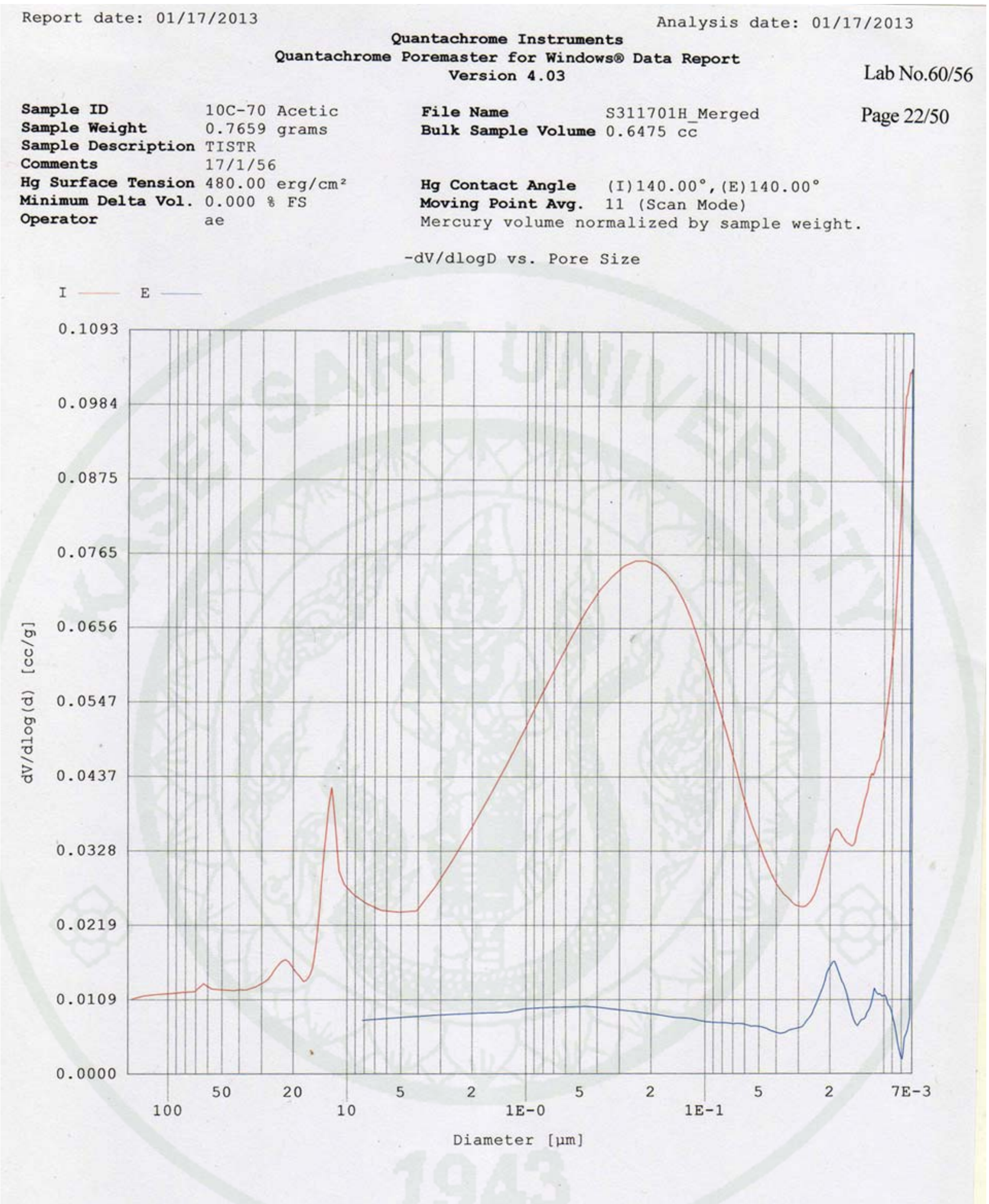
Lab No.60/56

Page 18/50

Sample ID 10C-70
Sample Weight 0.9437 grams
Sample Description TISTR
Comments 14/1/56
Hg Surface Tension 480.00 erg/cm²
Minimum Delta Vol. 0.000 % FS
Operator Panida

File Name S311406H_Merged
Bulk Sample Volume 0.6185 cc
Hg Contact Angle (I)140.00°,(E)140.00°
Moving Point Avg. 11 (Scan Mode)
Mercury volume normalized by sample weight.

Appendix Figure B5 Pore size distribution in the MIP tests for the 10C-70 geopolymer of class C fly ash.



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Report date: 01/17/2013                                    Analysis date: 01/17/2013
                              Quantachrome Instruments
                   Quantachrome Poremaster for Windows® Data Report
                                    Version 4.03

Sample ID           10C-70 Acetic          File Name           S311701H_Merged
Sample Weight       0.7659 grams           Bulk Sample Volume  0.6475 cc
Sample Description  TISTR
Comments            17/1/56
Hg Surface Tension  480.00 erg/cm²         Hg Contact Angle    (I)140.00°,(E)140.00°
Minimum Delta Vol.  0.000 % FS             Moving Point Avg.   11 (Scan Mode)
Operator            ae                     Mercury volume normalized by sample weight.
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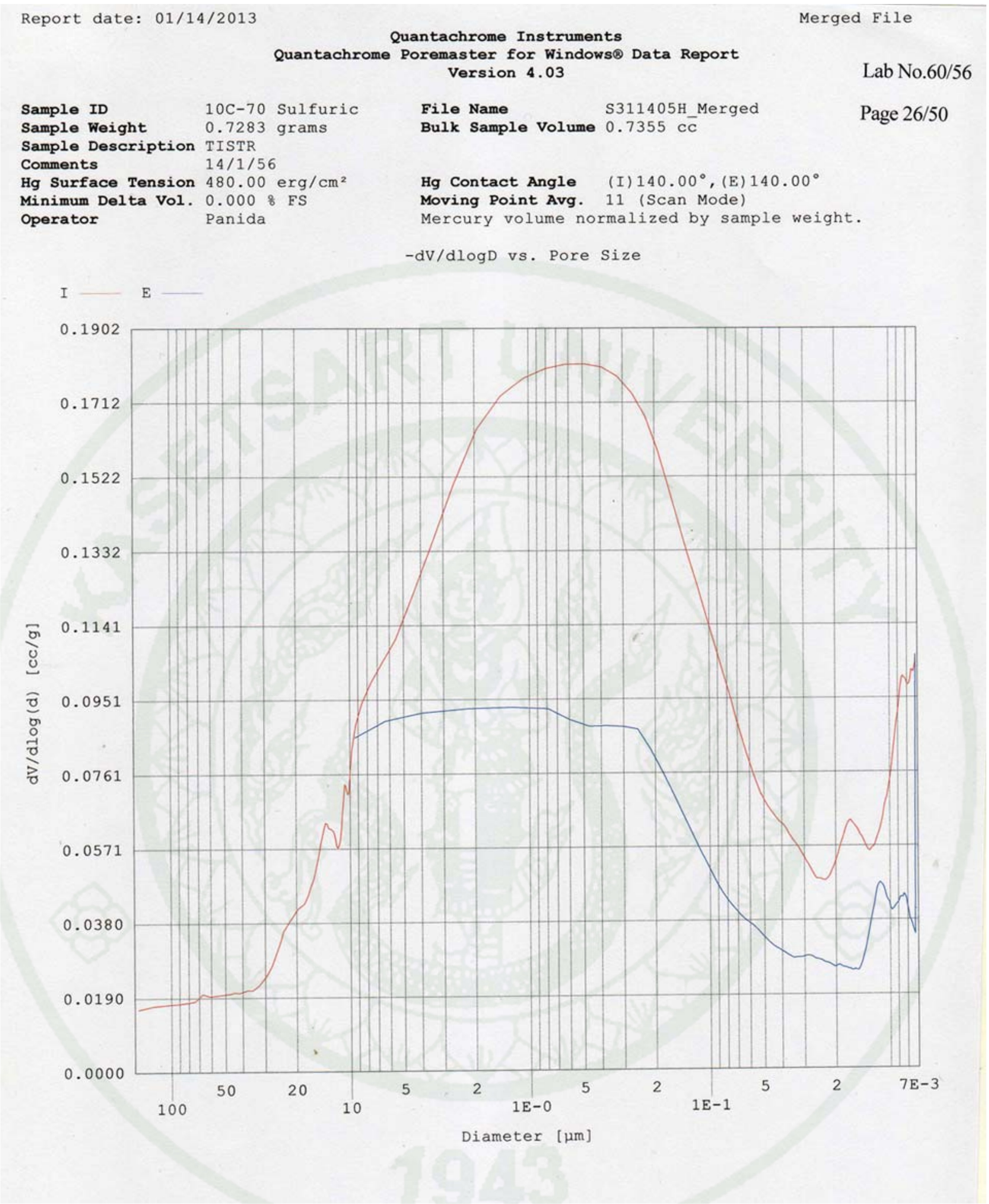
Lab No.60/56

Page 22/50

Appendix Figure B6 Pore size distribution in the MIP tests for the 10C-70 geopolymer of class C fly ash after exposure to 5% acetic acid for 18 weeks.



Report date: 01/14/2013 Merged File

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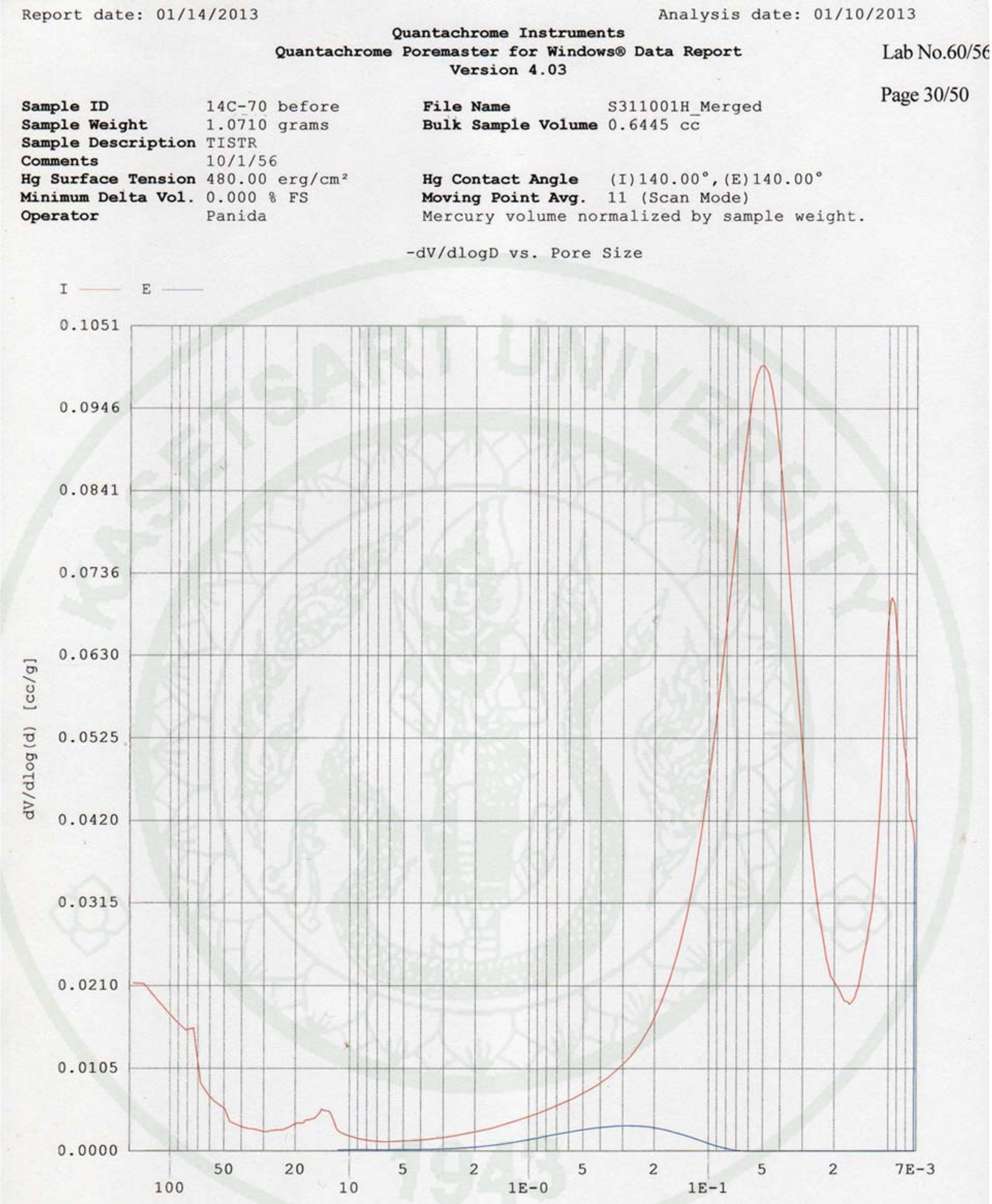
Version 4.03

Lab No.60/56
Page 26/50

Sample ID	10C-70 Sulfuric	**File Name**	S311405H_Merged
Sample Weight	0.7283 grams	**Bulk Sample Volume**	0.7355 cc
Sample Description	TISTR		
Comments	14/1/56		
Hg Surface Tension	480.00 erg/cm²	**Hg Contact Angle**	(I)140.00°,(E)140.00°
Minimum Delta Vol.	0.000 % FS	**Moving Point Avg.**	11 (Scan Mode)
Operator	Panida		Mercury volume normalized by sample weight.

-dV/dlogD vs. Pore Size

Appendix Figure B7 Pore size distribution in the MIP tests for the 10C-70 geopolymer of class C fly ash after exposure to 5% sulfuric acid for 18 weeks.



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Report date: 01/14/2013                                          Analysis date: 01/10/2013
                                     Quantachrome Instruments
                          Quantachrome Poremaster for Windows® Data Report
                                          Version 4.03

Sample ID            14C-70 before            File Name            S311001H_Merged
Sample Weight        1.0710 grams             Bulk Sample Volume   0.6445 cc
Sample Description   TISTR
Comments             10/1/56
Hg Surface Tension   480.00 erg/cm²           Hg Contact Angle     (I)140.00°,(E)140.00°
Minimum Delta Vol.   0.000 % FS               Moving Point Avg.    11 (Scan Mode)
Operator             Panida                   Mercury volume normalized by sample weight.
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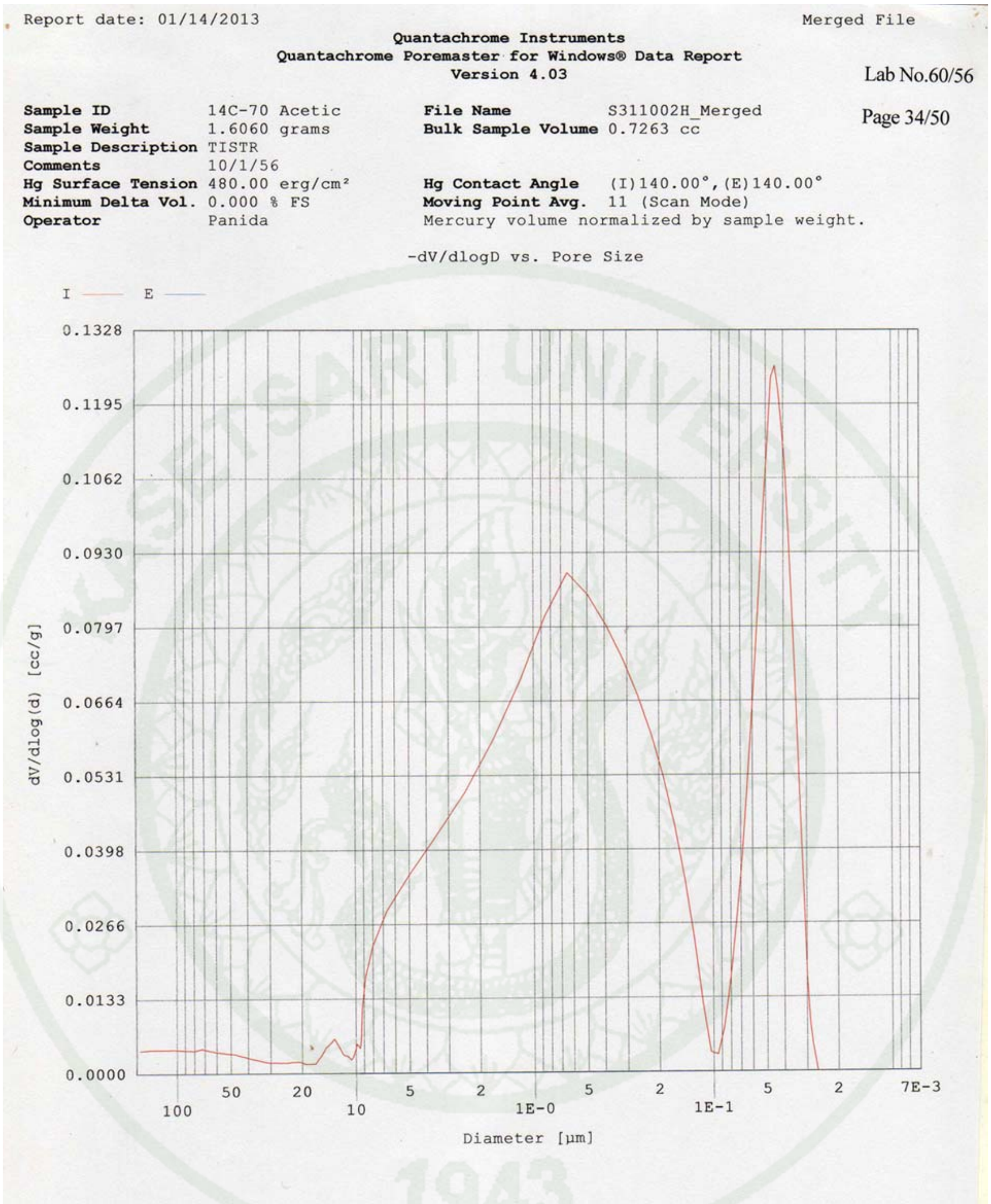
Lab No.60/56

Page 30/50

Appendix Figure B8 Pore size distribution in the MIP tests for the 14C-70 geopolymer of class C fly ash



Report date: 01/14/2013 Merged File

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Version 4.03

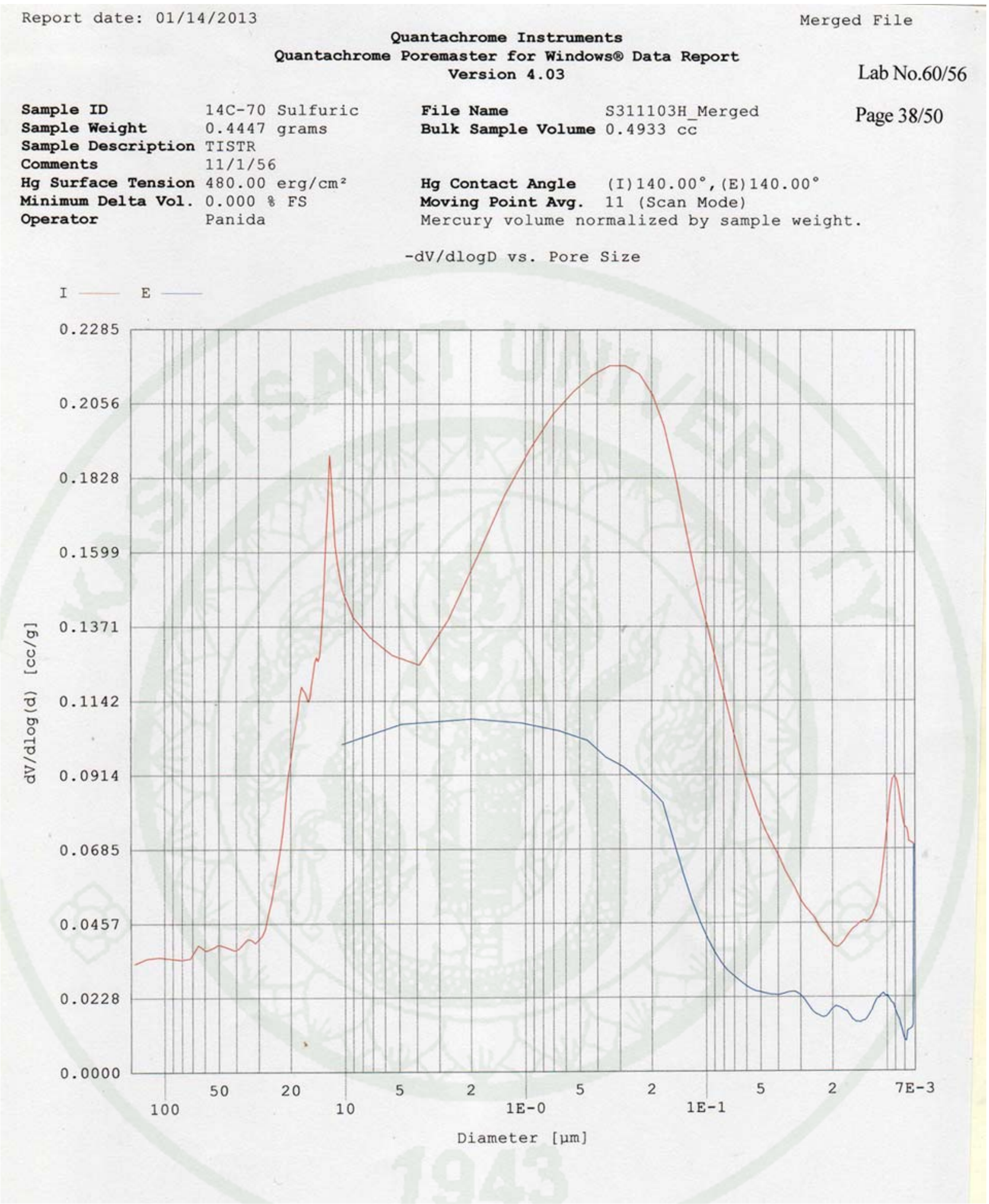
Lab No.60/56

Page 34/50

Sample ID	14C-70 Acetic	**File Name**	S311002H_Merged
Sample Weight	1.6060 grams	**Bulk Sample Volume**	0.7263 cc
Sample Description	TISTR		
Comments	10/1/56		
Hg Surface Tension	480.00 erg/cm²	**Hg Contact Angle**	(I)140.00°,(E)140.00°
Minimum Delta Vol.	0.000 % FS	**Moving Point Avg.**	11 (Scan Mode)
Operator	Panida		Mercury volume normalized by sample weight.

-dV/dlogD vs. Pore Size

Appendix Figure B9 Pore size distribution in the MIP tests for the 14C-70 geopolymer of class C fly ash after exposure to 5% acetic acid for 18 weeks



Report date: 01/14/2013 Merged File

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Version 4.03

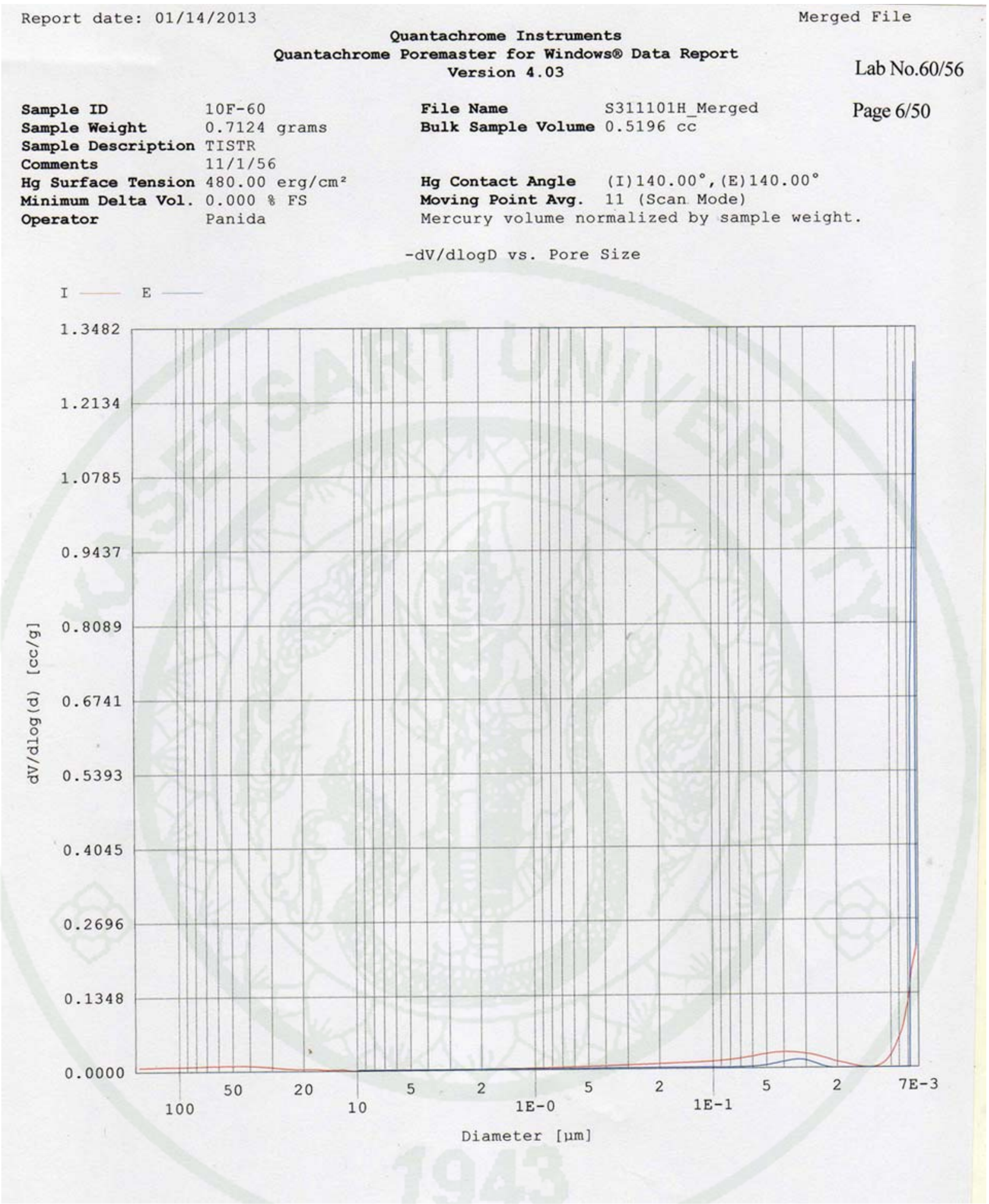
Lab No.60/56
Page 38/50

Sample ID 14C-70 Sulfuric
Sample Weight 0.4447 grams
Sample Description TISTR
Comments 11/1/56
Hg Surface Tension 480.00 erg/cm²
Minimum Delta Vol. 0.000 % FS
Operator Panida

File Name S311103H_Merged
Bulk Sample Volume 0.4933 cc
Hg Contact Angle (I)140.00°,(E)140.00°
Moving Point Avg. 11 (Scan Mode)
Mercury volume normalized by sample weight.

-dV/dlogD vs. Pore Size

Appendix Figure B10 Pore size distribution in the MIP tests for the 14C-70 geopolymer of class C fly ash after exposure to 5% sulfuric acid for 18 weeks



Report date: 01/14/2013

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Version 4.03

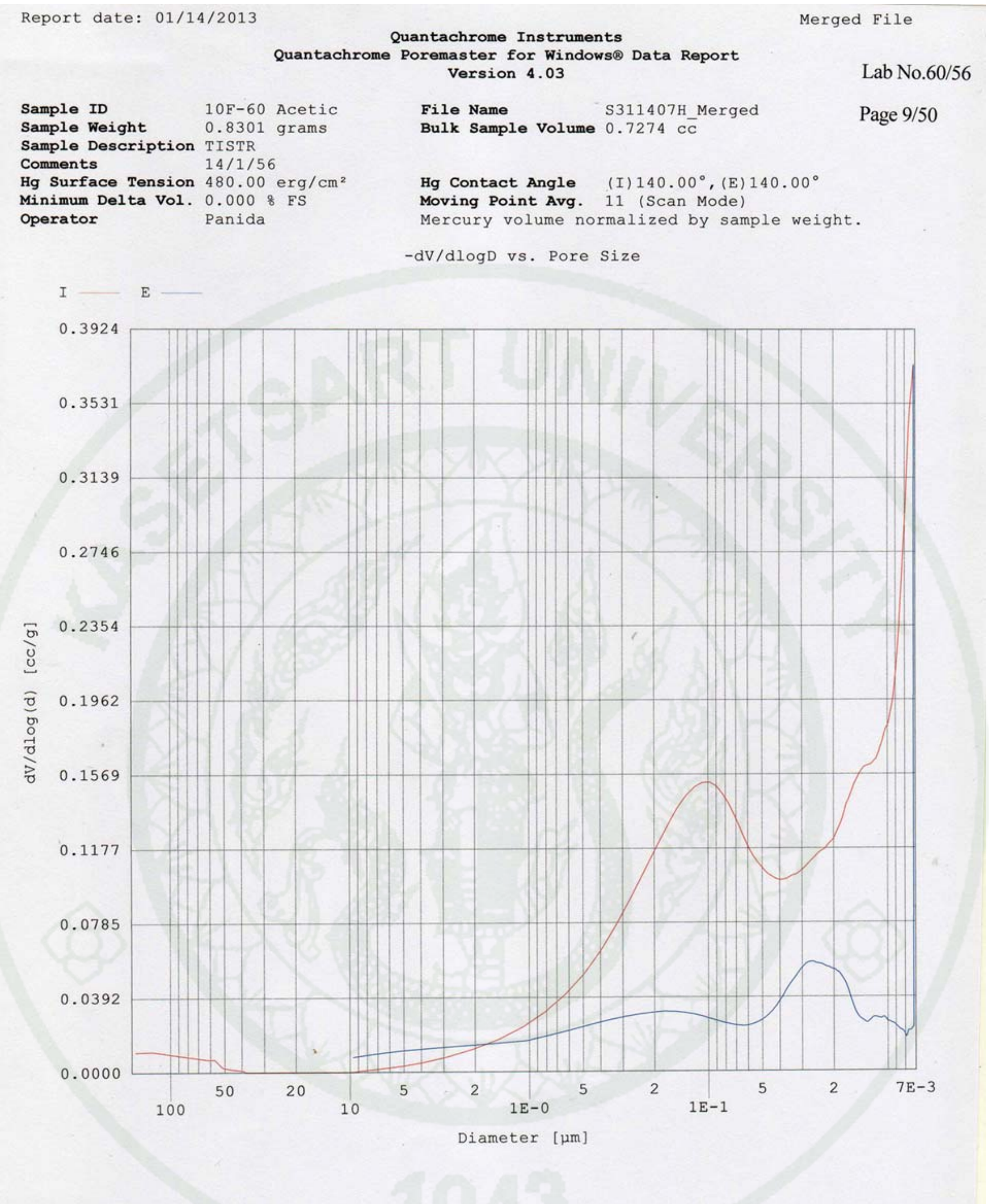
Lab No.60/56

Page 6/50

Sample ID 10F-60
Sample Weight 0.7124 grams
Sample Description TISTR
Comments 11/1/56
Hg Surface Tension 480.00 erg/cm²
Minimum Delta Vol. 0.000 % FS
Operator Panida

File Name S311101H_Merged
Bulk Sample Volume 0.5196 cc
Hg Contact Angle (I)140.00°,(E)140.00°
Moving Point Avg. 11 (Scan Mode)
Mercury volume normalized by sample weight.

Appendix Figure B11 Pore size distribution in the MIP tests for the 10F-60 geopolymer of class F fly ash



Report date: 01/14/2013 Merged File

Quantachrome Instruments
Quantachrome Poremaster for Windows® Data Report
Version 4.03

Lab No.60/56

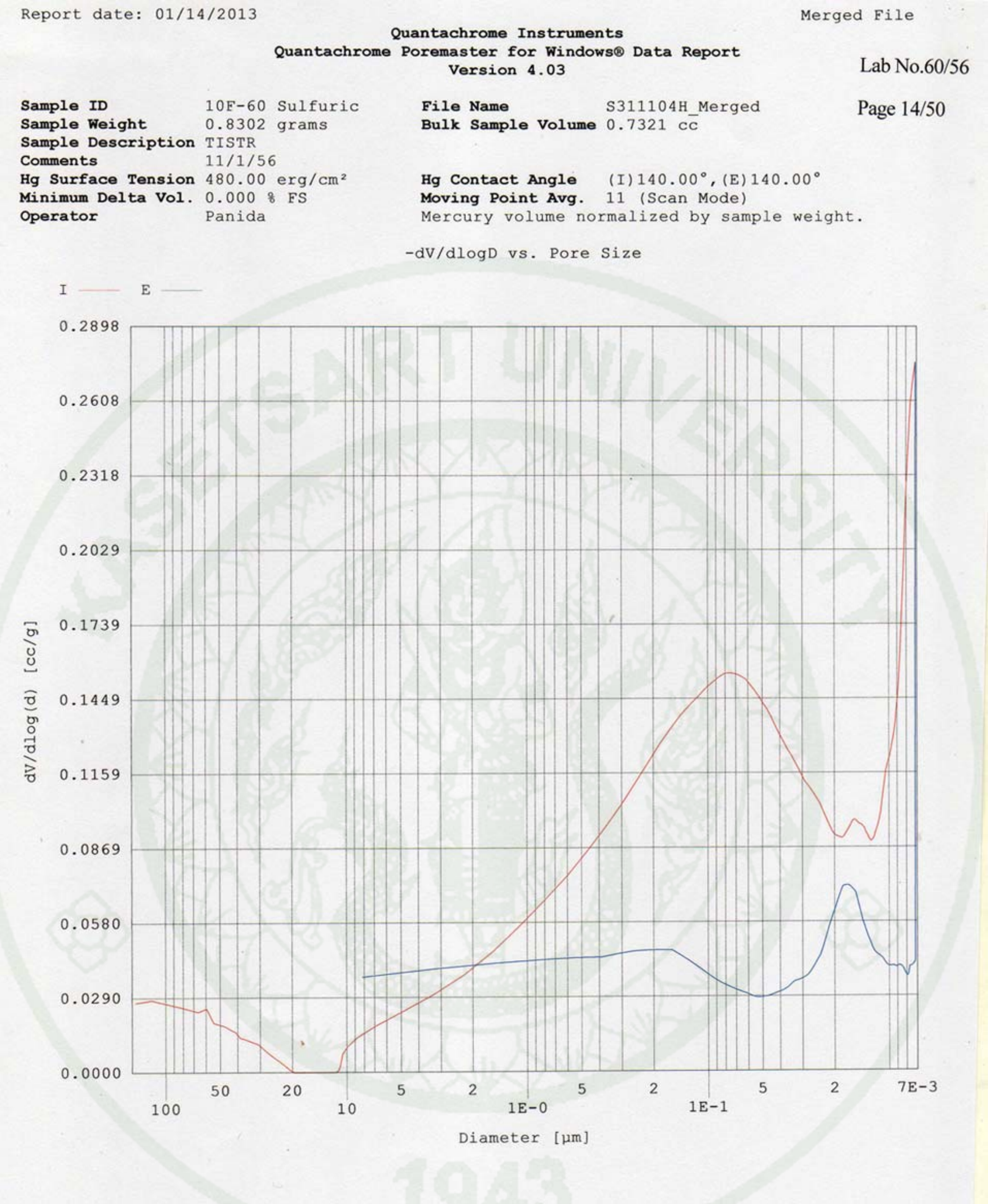
Page 9/50

Sample ID 10F-60 Acetic
Sample Weight 0.8301 grams
Sample Description TISTR
Comments 14/1/56
Hg Surface Tension 480.00 erg/cm²
Minimum Delta Vol. 0.000 % FS
Operator Panida

File Name S311407H_Merged
Bulk Sample Volume 0.7274 cc
Hg Contact Angle (I)140.00°,(E)140.00°
Moving Point Avg. 11 (Scan Mode)
Mercury volume normalized by sample weight.

-dV/dlogD vs. Pore Size

Appendix Figure B12 Pore size distribution in the MIP tests for the 10F-60 geopolymer of class F fly ash after exposure to 5% acetic acid for 18 weeks.



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Quantachrome Instruments
Quantachrome Poremaster for Windows® Data Report
Version 4.03

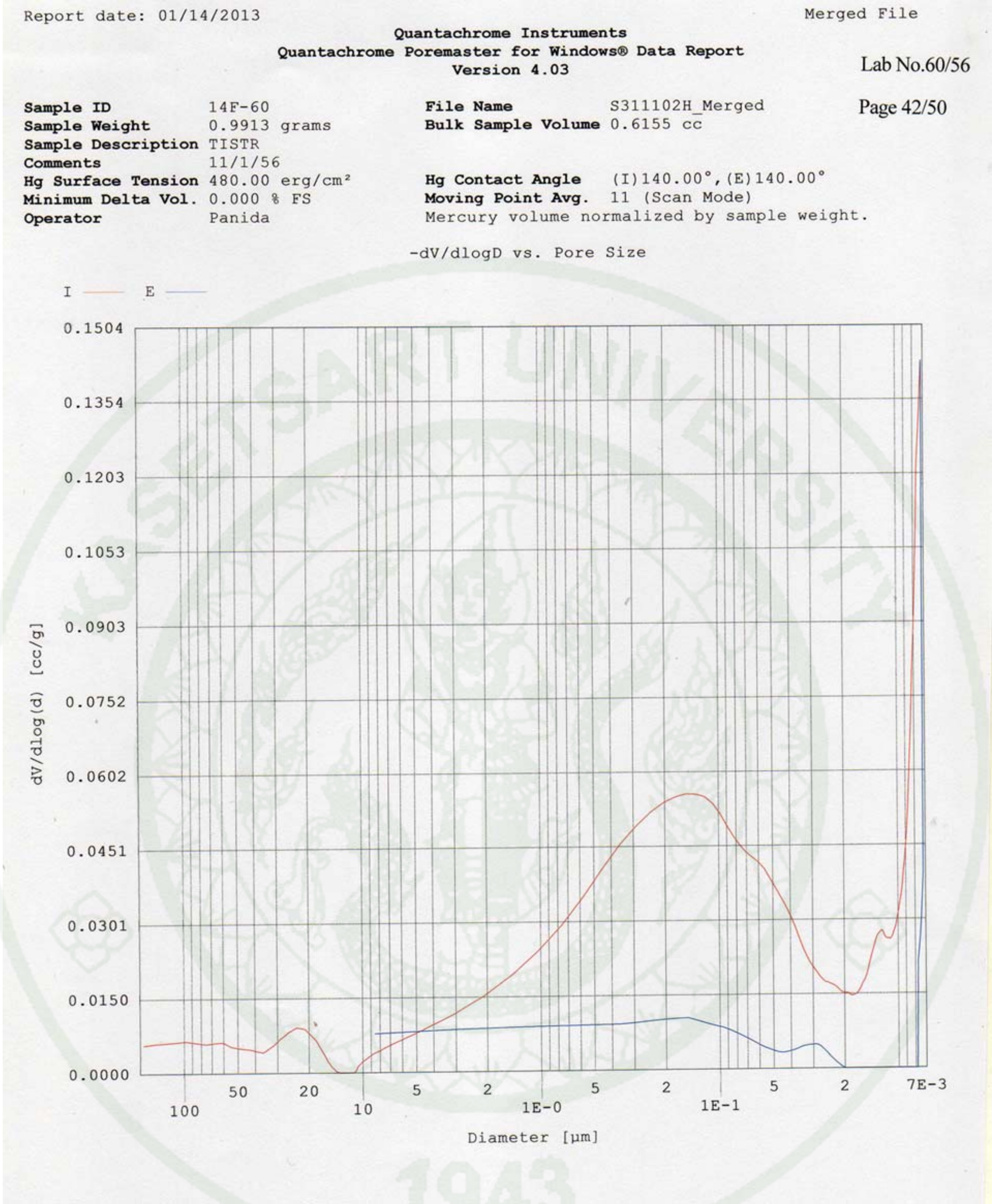
Lab No.60/56

Page 14/50

Sample ID 10F-60 Sulfuric
Sample Weight 0.8302 grams
Sample Description TISTR
Comments 11/1/56
Hg Surface Tension 480.00 erg/cm²
Minimum Delta Vol. 0.000 % FS
Operator Panida

File Name S311104H_Merged
Bulk Sample Volume 0.7321 cc
Hg Contact Angle (I)140.00°,(E)140.00°
Moving Point Avg. 11 (Scan Mode)
Mercury volume normalized by sample weight.

Appendix Figure B13 Pore size distribution in the MIP tests for the 10F-60 geopolymer of class F fly ash after exposure to 5% sulfuric acid for 18 weeks



Report date: 01/14/2013 Merged File

Quantachrome Instruments
Quantachrome Poremaster for Windows® Data Report
Version 4.03

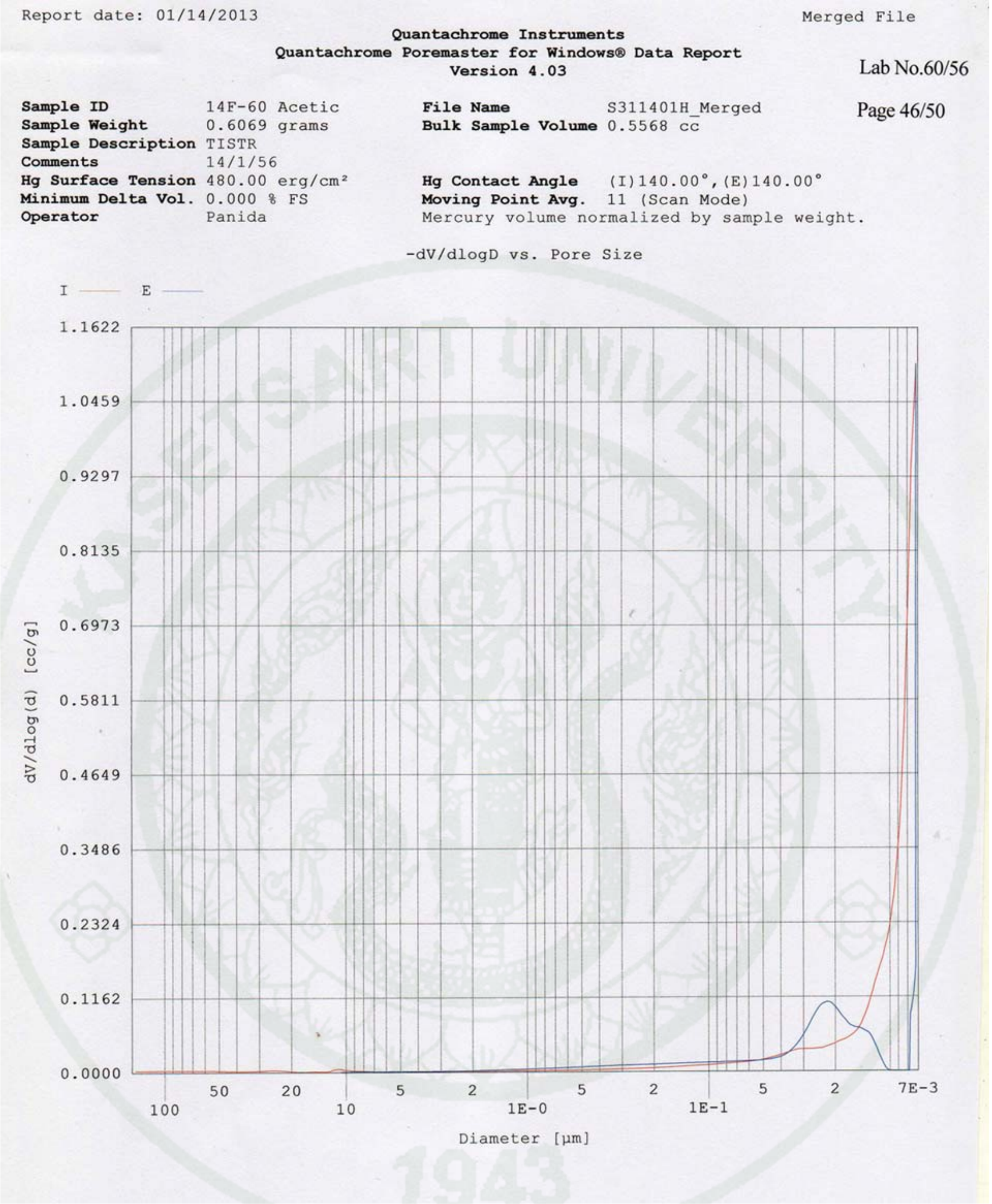
Lab No.60/56
Page 42/50

Sample ID 14F-60
Sample Weight 0.9913 grams
Sample Description TISTR
Comments 11/1/56
Hg Surface Tension 480.00 erg/cm²
Minimum Delta Vol. 0.000 % FS
Operator Panida

File Name S311102H_Merged
Bulk Sample Volume 0.6155 cc
Hg Contact Angle (I)140.00°,(E)140.00°
Moving Point Avg. 11 (Scan Mode)
Mercury volume normalized by sample weight.

-dV/dlogD vs. Pore Size

Appendix Figure B14 Pore size distribution in the MIP tests for the 14F-60 geopolymer of class F fly ash



Report date: 01/14/2013

Merged File

Quantachrome Instruments
Quantachrome Poremaster for Windows® Data Report
Version 4.03

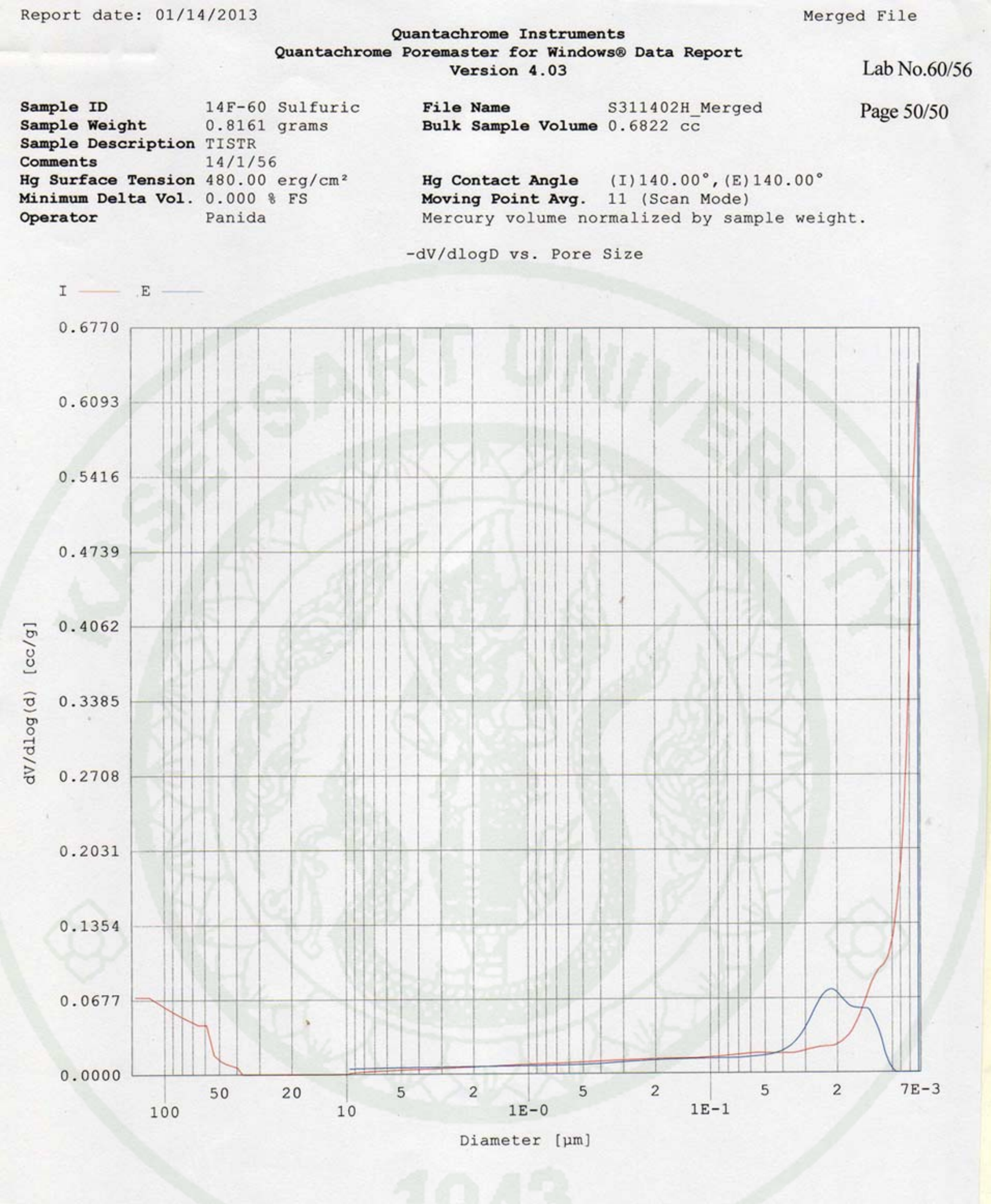
Lab No.60/56

Page 46/50

Sample ID	14F-60 Acetic	**File Name**	S311401H_Merged
Sample Weight	0.6069 grams	**Bulk Sample Volume**	0.5568 cc
Sample Description	TISTR		
Comments	14/1/56		
Hg Surface Tension	480.00 erg/cm²	**Hg Contact Angle**	(I)140.00°,(E)140.00°
Minimum Delta Vol.	0.000 % FS	**Moving Point Avg.**	11 (Scan Mode)
Operator	Panida		Mercury volume normalized by sample weight.

-dV/dlogD vs. Pore Size

Appendix Figure B15 Pore size distribution in the MIP tests for the 14F-60 geopolymer of class F fly ash after exposure to 5% acetic acid for 18 weeks.



Report date: 01/14/2013

Merged File

Quantachrome Instruments
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Version 4.03

Lab No.60/56

Page 50/50

Sample ID 14F-60 Sulfuric
Sample Weight 0.8161 grams
Sample Description TISTR
Comments 14/1/56
Hg Surface Tension 480.00 erg/cm²
Minimum Delta Vol. 0.000 % FS
Operator Panida

File Name S311402H_Merged
Bulk Sample Volume 0.6822 cc
Hg Contact Angle (I)140.00°,(E)140.00°
Moving Point Avg. 11 (Scan Mode)
Mercury volume normalized by sample weight.

-dV/dlogD vs. Pore Size

Appendix Figure B16 Pore size distribution in the MIP tests for the 14F-60 geopolymer of class F fly ash after exposure to 5% sulfuric acid for 18 weeks.

5% Acetic acid solution

5% Sulfuric acid solution

Appendix Figure B17 Acetic acid and sulfuric acid test

Appendix Figure B18 Compression strength machine

CURRICULUM VITAE

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