สกว
TRF

สำนักงานคณะกรรมการการอุดมศึกษา

รายงานวิจัยฉบับสมบูรณ์

โครงการการพัฒนากำลังอัดและโครงสร้างจุลภาคของดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์จีโอโพลีเมอร์

โดย ผู้ช่วยศาสตราจารย์ ดร.เชิดศักดิ์ สุขศิริพัฒนพงศ์

เมษายน 2562

สัญญาเลขที่ MRG6080073

รายงานวิจัยฉบับสมบูรณ์

โครงการการพัฒนากำลังอัดและโครงสร้างจุลภาคของดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์จีโอโพลีเมอร์

ผู้ช่วยศาสตราจารย์ ดร.เชิดศักดิ์ สุขศิริพัฒนพงศ์
มหาวิทยาลัยเทคโนโลยีราชมงคลอีสาน

สนับสนุนโดยสำนักงานกองทุนสนับสนุนการวิจัยและ
สำนักงานคณะกรรมการการอุดมศึกษา

บทคัดย่อ

Project Code : MRG6080073

Project Title : การพัฒนากำลังอัดและโครงสร้างจุลภาคของดินเหนียวอ่อนผสม
เถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์

Investigator : ผู้ช่วยศาสตราจารย์ ดร. เชิดศักดิ์ สุขศิริพัฒนพงศ์
มหาวิทยาลัยเทคโนโลยีราชมงคลอีสาน

E-mail Address : Cherdsak.su@rmuti.ac.th

Project Period : 24 เดือน

งานวิจัยนี้ศึกษาความเป็นไปได้ในการใช้เถ้าลอย และโพลิไวนิลแอลกอฮอล์ (PVA) จีโอโพลีเมอร์ เพื่อปรับปรุงกำลังของดินเหนียวอ่อนสำหรับวัสดุก่อสร้าง ปัจจัยที่มีผลต่อกำลังประกอบด้วยปริมาณเถ้าลอย, ปริมาณสารกระตุ้น, อัตราส่วน Na_2SiO_3/NaOH, ความเข้มข้นของ NaOH, ปริมาณ PVA, ความเข้มข้นของ PVA และอายุบ่ม โดยศึกษาโครงสร้างทางจุลภาคของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ เพื่อเข้าใจถึงอิทธิผลควบคุมต่อการพัฒนากำลังอัด ทดสอบการชะโลหะหนักของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ เพื่อตรวจสอบคุณสมบัติด้านสิ่งแวดล้อม ท้ายสุดศึกษาการปล่อยก๊าซ CO_2 ของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ และเปรียบเทียบกับตัวอย่างที่ใช้ปูนซีเมนต์ ผลการทดสอบแสดงให้เห็นว่ากำลังอัดที่อายุบ่ม 7 วันของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย จีโอโพลีเมอร์เพิ่มขึ้นกับการเพิ่มปริมาณ FA และ L/FA เนื่องจากตัวอย่างเกิดผลิตภัณฑ์จีโอโพลีเมอร์ไรเซชั่นมากกว่า อย่างไรก็ตามปริมาณความชื้นที่มากเกินไป (มากกว่าขีดจำกัดเหลว) ทำให้ความเข้มข้นของ NaOH เจือจาง และทำให้เกิดช่องว่างขนาดใหญ่ อัตราส่วน Na_2SiO_3/NaOH มีผลกระทบต่อกำลังอัดที่อายุบ่ม 7 วันเพียงเล็กน้อย กำลังอัดสูงสุดของตัวอย่างมีค่าเท่ากับ 1,990 kPa ที่ปริมาณความชื้นเท่ากับขีดจำกัดเหลว, อัตราส่วน Na_2SiO_3/NaOH=1 และอายุบ่มเท่ากับ 90 วัน อัตราส่วนผสมที่เหมาะสมเมื่อพิจารณาด้านความสามรถในการทำงานและราคาของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย จีโอโพลีเมอร์ พบที่ปริมาณความชื้นเท่ากับ 1.5 เท่าของขีดจำกัดเหลว, อัตราส่วน Na_2SiO_3/NaOH=1 ปริมาณเถ้าลอบเท่ากับร้อยละ 30 และอัตราส่วน L/FA ซึ่งมีกำลังอัดที่อายุบ่มเท่ากับ 28 วัน เท่ากับ 682 kPa และผ่านเกณฑ์มาตรฐานของกรมทางหลวงซึ่งระบุกำลังอัดไว้ไม่น้อยกว่า 500 kPa สำหรับเสาเข็มดิน กำลังอัดสูงสุดที่อายุบ่ม 7 วันของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ มีค่าเท่ากับ 600 kPa ที่ ปริมาณ PVA เท่ากับร้อยละ 15 และความเข้มข้นของ PVA เท่ากับร้อยละ 2 ตามที่ค่ามาตรฐานน้ำดื่มที่ U.S. Environmental Protection Agency กำหนดไว้ พบว่าปริมาณสารที่ตรวจพบในตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ มีค่าไม่

เกินเกณฑ์ที่กำหนด การปล่อยก๊าซ CO_2 ของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ (FA, Na_2SiO_3, NaOH และ PVA) มีค่าเท่ากับ 221 kg CO_2-e/ton เมื่อเปรียบเทียบกับการปล่อยก๊าซ CO_2 ของปูนซีเมนต์ (246 kg CO_2-e/ton) พบว่าการปล่อยก๊าซ CO_2 ของปูนซีเมนต์มีค่าสูงกว่าการปล่อยก๊าซ CO_2 ของตัวอย่างดินเหนียวอ่อนผสมเถ้าลอย และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ร้อยละ 10.16 การพัฒนากำลังอัดและโครงสร้างจุลภาคของดินเหนียวอ่อนผสมเถ้าจากเกษตรกรรม (เถ้าชานอ้อย เถ้าแกลบ หรือเถ้าปาล์มน้ำมัน) และโพลิไวนิลแอลกอฮอล์ จีโอโพลีเมอร์ควรศึกษาต่อในอนาคต

Keywords : กำลังอัด โครงสร้างจุลภาค ดินเหนียวอ่อน เถ้าลอย จีโอโพลีเมอร์
โพลิไวนิลแอลกอฮอล์

Abstract

Project Code : MRG6080073

Project Title : Strength Development and Microstructure of Soft Clay-Fly ash Geopolymer Modified with Polyvinyl Alcohol (PVA)

Investigator : Assistant professor Dr. Cherdsak Suksiripattanapong
Rajamangala University of Technology Isan

E-mail Address : Cherdsak.su@rmuti.ac.th

Project Period : 24 months

This research investigates the possibility of using fly ash (FA) and polyvinyl alcohol (PVA) geopolymer to improve strength of soft clay for using as construction materials. The influential factors include mixing ingredient (FA content, water content, liquid alkaline activation content, Na_2SiO_3/NaOH ratio, concentration of NaOH, PVA content, concentration of PVA) and curing time. The microstructural analyses of soft clay stabilized with FA and PVA geopolymer is undertaken using Scanning Electron Microscopy (SEM) to understand the role of influential factors controlling the strength development. The leaching test was undertaken on the soft clay stabilized with FA and PVA geopolymer in order to understand the environmental effects of using the soft clay stabilized with FA and PVA geopolymer in construction materials needs to be ascertained. Moreover, the carbon footprints of soft clay stabilized with FA and PVA geopolymer are calculated and compared with soft clay stabilized with cement. The 7-day UCS of SC-FA geopolymer increases with increasing FA and L/FA content, due to higher geopolymerization products. However, the excessive water content (more than LL) causes a diluted NaOH concentration (less dissolved Si and Al from FA) and a large pore space. The Na_2SiO_3/NaOH ratios were found to have a small effect on 7-day strength. The maximum UCS was found to be 1,990 kPa at LL and Na_2SiO_3/NaOH = 1 and 90 days of curing. The optimum ingredient considering workability and cost of SC-FA geopolymer was found to be at 1.5LL, Na_2SiO_3/NaOH = 1, FA = 30% and L/FA = 0.6, which gave the 28-day compressive strength of 682 kPa and meets the minimum UCS requirement of Departmant of Highways Thailand (> 5 00 kPa) for soil cement column. The highest 7-day compressive strength was found at PVA content of 15%, where the maximum compressive strengths are 600 kPa for PVA concentrations of 2 %. According to benchmark mandated by the U.S. Environmental Protection Agency (EPA) for storm-water sampling, these results indicate that the SC-HFA-PVA geopolymer sample are within allowable limits. The emission CO2-e of HFA-PVA geopolymer (FA, Na2SiO3, NaOH and PVA) is 221 kg CO2-e/ton compared with the emission CO2-e of cement of 246 kg CO2-e/ton. The emission CO2-e of cement is higher than that of HFA-PVA geopolymer which is about 10.16%. The strength and microstructure development in soft clay stabilized with agricultural ash and polyvinyl alcohol (PVA) geopolymer should be further studied.

Keywords : Strength, Microstructure, Soft Clay, Fly ash Geopolymer, Polyvinyl Alcohol

แบบสรุปผู้บริหาร

[Executive Summary]

Project Code : MRG6080073

Project Title : Strength Development and Microstructure of Soft Clay-Fly ash Geopolymer Modified with Polyvinyl Alcohol (PVA)

Investigator : Assistant professor Dr. Cherdsak Suksiripattanapong
Rajamangala University of Technology Isan

E-mail Address : Cherdsak.su@rmuti.ac.th

Project Period : 24 months

1. Introduction to the research problem and its significance

Bangkok clay is one of the well-known soft clay deposits. It possesses high water content close to its liquid limit with large potential for settlement and low inherent shear strength. Several ground improvement techniques dealing with soft soil foundation have been developed over the past three decades (Bouazza et al., 2006; Bergado et al., 2003; Du et al., 2013 and 2014; Chai et al., 2014; Bo et al., 2015; Wu et al., 2015). In situ deep mixing (DM) is an effective means. The DM method has been developed during last over two decades primarily to effect columnar inclusions into the soft ground to transform such whole soft ground to composite ground. The DM technology was simultaneously developed in Sweden and Japan using quicklime as a hardening agent. Later on, ordinary Portland cement slurry was used. Portland cement is commonly used as a cementing agent because it is readily available at reasonable cost. The influential factors, controlling the field strength of deep mixing columns such as penetration and withdrawal rates, water to cement ratio, and rate of blade rotation were extensively investigated by Horpibulsuk et al., (2004a) (Ariake clay, Japan) and Horpibulsuk et al. (2011b and 2012c) (Bangkok clay, Thailand). However, the manufacturing of Portland cement is a resource exhausting and energy intensive process that releases large amount of the CO_2 into the atmosphere, which cause the greenhouse gases (Davidovits 1991; Davidovits and Davidovics 2008). Therefore, the development of a new cementing agent and development of recycled construction materials derived from waste materials with low carbon dioxide release is considered an interesting issue. Commercial and industrial utilization of alkali-activated alumino-silicate cements, known as ‘geopolymers’ has been increasingly well-known over the past several decades as the search for high-performance and an environmentally maintainable alternative for ordinary Portland cement (J. Davidovits 1991; Phetchuay et al., 2016).

Geopolymers are a group of cementitious materials that has garnered increasing interest as an alternative stabilizing agent (Sukmak et al. 2013a; Suksiripattanapong et al. 2015a, 2015b; Horpibulsuk et al., 2015) to replace portland cement. The chemical process to produce geopolymers involves the co-polymerization of alumina and silica components whereby aluminosilicate-rich materials are dissolved by highly alkaline solutions such as sodium hydroxide (NaOH). Sodium silicate (Na_2SiO_3) can further increase the strength of the geopolymer (Palomo et al. 1999) because of the gel-like product derived from the aluminosilicate-sodium silicate reaction (Xie and Xi 2001). The silica-rich materials such as clay or kaolin (Buchwald and Kaps 2002), fly ash, and bottom ash (Davidovits et al., 1999) can be used as a precursor to react with the liquid alkaline activator. Fly Ash (FA) provides the greatest opportunity for commercial utilization of this technology due to the plentiful worldwide raw material supply, which is derived from coal-fired electricity generation (Mohapatra and Rao, 2001; Van Jaarsveld et al., 1998).

This research investigates the possibility of using fly ash (FA) and polyvinyl alcohol (PVA) geopolymer to improve strength of soft clay for using as construction materials. The role of influential factors on strength development in soft clay stabilized with FA and PVA geopolymer is also examined. The influential factors include mixing ingredient (FA content, water content, liquid alkaline activation content, Na_2SiO_3/NaOH ratio, concentration of NaOH, PVA content, concentration of PVA) and curing time. The microstructural analyses of soft clay stabilized with FA and PVA geopolymer is undertaken using Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) techniques to understand the role of influential factors controlling the strength development. The leaching test was undertaken on the soft clay stabilized with FA and PVA geopolymer in order to understand the environmental effects of using the soft clay stabilized with FA and PVA geopolymer in construction materials needs to be ascertained. Moreover, the carbon footprints of soft clay stabilized with FA and PVA geopolymer are calculated and compared with soft clay stabilized with cement. The outcome of this research can reduce the use of traditional Portland cement that releases large amount of the CO_2 into the atmosphere, which cause the greenhouse gases by using FA geopolymer modified with PVA as an alternative and effective green soil stabilizer.

2. Objectives

The objectives of this research are as follows:

1 To study a possibility of using fly ash geopolymer modified with polyvinyl alcohol (PVA) to improve the strength of soft clay through the examinations of the strength properties and microstructure analyses.

2 To calculate the carbon footprints of soft clay stabilized with fly ash (FA) and polyvinyl alcohol (PVA) geopolymer and compare with soft clay stabilized with cement.

3 To investigate the leaching analysis of soft clay stabilized with FA and PVA geopolymer.

3. Scope of research

In this research, the possibility of using fly ash geopolymer modified with polyvinyl alcohol (PVA) to improve the strength of soft clay through the examinations of the strength properties and microstructure analyses. The index properties (liquid limit, plastic limit, shrinkage limit and plasticity index) of soft clay stabilized with FA and PVA geopolymer is investigated. The microstructural analysis of soft clay stabilized with FA and PVA geopolymer is observed through Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) analyses for understanding the role of influential factors controlling the strength properties. The influential factors studied in this study include fly ash content, water content, PVA content, liquid alkali activator, Na_2SiO_3/NaOH ratio and curing time at ambient room temperature. The scope of this research is as follows: The water content of soft Bangkok clay varied from1 to 2 times LL to simulate the variation of in-situ water content. The FA contents were 10, 20, 30, 40 and 50% by weight of dry soil. The Na_2SiO_3/NaOH ratios were 1, 2, 3 and 4. The NaOH concentrations were 1, 3, 5 and 7 molars. The PVA contents were 0, 2, 4, 6, 8 and 10. The PVA concentrations were 5, 10, 15, 20, 25 and 30. The L/FA ratios were 0.4, 0.5, 0.6, 0.7 and 0.8. The curing temperature were room temperature. The UCS tests were carried out on the samples after 7, 14, 28, 60 and 90 days of curing in accordance with ASTM C69-09. The carbon footprint, defined as CO_2-e emitted (kg CO_2-e/ton), of FA geopolymer and FA–PVA geopolymer stabilized soft clay is calculated and compared with that of cement stabilized soft clay at the same UCS. The CO_2 emission calculation considered the mining, processing and manufacturing of raw materials. The growth of geopolymerization structures on the samples was illustrated using X-ray Diffraction

(XRD) and Scanning Electron Microscope (SEM). The XRD traces were used to provide fundamental information on geopolymerization structures.

4. Results

This research investigates the possibility of using fly ash (FA) and polyvinyl alcohol (PVA) geopolymer to improve strength of soft clay for using as construction materials. The influential factors include mixing ingredient (FA content, water content, liquid alkaline activation content, Na_2SiO_3/NaOH ratio, concentration of NaOH, PVA content, concentration of PVA) and curing time. The 7-day UCS of SC-FA geopolymer increases with increasing FA and L/FA content, due to higher geopolymerization products. However, the excessive water content (more than LL) causes a diluted NaOH concentration (less dissolved Si and Al from FA) and a large pore space. The Na_2SiO_3/NaOH ratios were found to have a small effect on 7-day strength. The maximum UCS was found to be 1,990 kPa at LL and Na_2SiO_3/NaOH = 1 and 90 days of curing. The optimum ingredient considering workability and cost of SC-FA geopolymer was found to be at 1.5LL, Na_2SiO_3/NaOH = 1, FA = 30% and L/FA = 0.6, which gave the 28-day compressive strength of 682 kPa and meets the minimum UCS requirement of Departmant of Highways Thailand (> 500 kPa) for soil cement column. The highest 7-day compressive strength was found at PVA content of 15%, where the maximum compressive strengths are 600 kPa for PVA concentrations of 2 %. According to benchmark mandated by the U.S. Environmental Protection Agency (EPA) for storm-water sampling, these results indicate that the SC-HFA-PVA geopolymer sample are within allowable limits. The emission CO2-e of HFA-PVA geopolymer (FA, Na2SiO3, NaOH and PVA) is 221 kg CO2-e/ton compared with the emission CO2-e of cement of 246 kg CO2-e/ton. The emission CO2-e of cement is higher than that of HFA-PVA geopolymer which is about 10.16%. The strength and microstructure development in soft clay stabilized with agricultural ash and polyvinyl alcohol (PVA) geopolymer should be further studied.

5. The outcome of this research

The outcome of this research can reduce the use of traditional Portland cement that releases large amount of the CO_2 into the atmosphere, which cause the greenhouse gases by using FA geopolymer modified with polyvinyl alcohol as an alternative and effective green soil stabilizer.

TABLE OF CONTENTS

Page

CHAPTER

TABLE OF CONTENTS (Continued)

Page

LIST OF TABLES

Table **Page**

LIST OF FIGURES

Figure **Page**

LIST OF FIGURES (continued)

Figure **Page**

LIST OF FIGURES (continued)

Figure **Page**

LIST OF FIGURES (continued)

Figure **Page**

LIST OF FIGURES (continued)

Figure **Page**

CHAPTER I

INTRODUCTION

1.1 Introduction to the research problem and its significance

Bangkok clay is one of the well-known soft clay deposits. It possesses high water content close to its liquid limit with large potential for settlement and low inherent shear strength. Several ground improvement techniques dealing with soft soil foundation have been developed over the past three decades (Bouazza et al., 2006; Bergado et al., 2003; Du et al., 2013 and 2014; Chai et al., 2014; Bo et al., 2015; Wu et al., 2015). In situ deep mixing (DM) is an effective means. The DM method has been developed during last over two decades primarily to effect columnar inclusions into the soft ground to transform such whole soft ground to composite ground. The DM technology was simultaneously developed in Sweden and Japan using quicklime as a hardening agent. Later on, ordinary Portland cement slurry was used. Portland cement is commonly used as a cementing agent because it is readily available at reasonable cost. The influential factors, controlling the field strength of deep mixing columns such as penetration and withdrawal rates, water to cement ratio, and rate of blade rotation were extensively investigated by Horpibulsuk et al., (2004a) (Ariake clay, Japan) and Horpibulsuk et al. (2011b and 2012c) (Bangkok clay, Thailand). However, the manufacturing of Portland cement is a resource exhausting and energy intensive process that releases large amount of the CO_2 into the atmosphere, which cause the greenhouse gases (Davidovits 1991; Davidovits and Davidovics 2008). Therefore, the development of a new cementing agent and development of recycled construction materials derived from waste materials with low carbon dioxide release is considered an interesting issue. Commercial and industrial utilization of alkali-activated alumino-silicate cements, known as ‘geopolymers’ has been increasingly well-known over the past several decades as the search for high-performance and an environmentally maintainable alternative for ordinary Portland cement (J. Davidovits 1991; Phetchuay et al., 2016).

Geopolymers are a group of cementitious materials that has garnered increasing interest as an alternative stabilizing agent (Sukmak et al. 2013a; Suksiripattanapong et al. 2015a, 2015b; Horpibulsuk et al., 2015) to replace portland cement. The chemical process to produce geopolymers involves the co-polymerization of alumina and silica components whereby aluminosilicate-rich materials are dissolved by highly alkaline solutions such as sodium hydroxide (NaOH). Sodium silicate (Na_2SiO_3) can further increase the strength of the geopolymer (Palomo et al. 1999) because of the gel-like product derived from the aluminosilicate-sodium silicate reaction (Xie and Xi 2001). The silica-rich materials such as clay or kaolin (Buchwald and Kaps 2002), fly ash, and bottom ash (Davidovits et al., 1999) can be used as a precursor to react with the liquid alkaline activator. Fly Ash (FA) provides the greatest opportunity for commercial utilization of this technology due to the plentiful worldwide raw material supply, which is derived from coal-fired electricity generation (Mohapatra and Rao, 2001; Van Jaarsveld et al., 1998).

This research investigates the possibility of using fly ash (FA) and polyvinyl alcohol (PVA) geopolymer to improve strength of soft clay for using as construction materials. The role of influential factors on strength development in soft clay stabilized with FA and PVA geopolymer is also examined. The influential factors include mixing ingredient (FA content, water content, liquid alkaline activation content,

Na_2SiO_3/NaOH ratio, concentration of NaOH, PVA content, concentration of PVA) and curing time. The microstructural analyses of soft clay stabilized with FA and PVA geopolymer is undertaken using Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) techniques to understand the role of influential factors controlling the strength development. The leaching test was undertaken on the soft clay stabilized with FA and PVA geopolymer in order to understand the environmental effects of using the soft clay stabilized with FA and PVA geopolymer in construction materials needs to be ascertained. Moreover, the carbon footprints of soft clay stabilized with FA and PVA geopolymer are calculated and compared with soft clay stabilized with cement. The outcome of this research can reduce the use of traditional Portland cement that releases large amount of the CO_2 into the atmosphere, which cause the greenhouse gases by using FA geopolymer modified with PVA as an alternative and effective green soil stabilizer.

1.2 Objectives

The objectives of this research are as follows:

1.2.1 To study a possibility of using fly ash geopolymer modified with polyvinyl alcohol (PVA) to improve the strength of soft clay through the examinations of the strength properties and microstructure analyses.

1.2.2 To calculate the carbon footprints of soft clay stabilized with fly ash (FA) and polyvinyl alcohol (PVA) geopolymer and compare with soft clay stabilized with cement.

1.2.3 To investigate the leaching analysis of soft clay stabilized with FA and PVA geopolymer.

1.3 Scope of research

In this research, the possibility of using fly ash geopolymer modified with polyvinyl alcohol (PVA) to improve the strength of soft clay through the examinations of the strength properties and microstructure analyses. The index properties (liquid limit, plastic limit, shrinkage limit and plasticity index) of soft clay stabilized with FA and PVA geopolymer is investigated. The microstructural analysis of soft clay stabilized with FA and PVA geopolymer is observed through Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) analyses for understanding the role of influential factors controlling the strength properties. The influential factors studied in this study include fly ash content, water content, PVA content, liquid alkali activator, Na_2SiO_3/NaOH ratio and curing time at ambient room temperature. The scope of this research is as follows: The water content of soft Bangkok clay varied from1 to 2 times LL to simulate the variation of in-situ water content. The FA contents were 10, 20, 30, 40 and 50% by weight of dry soil. The Na_2SiO_3/NaOH ratios were 1, 2, 3 and 4. The NaOH concentrations were 1, 3, 5 and 7 molars. The PVA contents were 0, 2, 4, 6, 8 and 10. The PVA concentrations were 5, 10, 15, 20, 25 and 30. The L/FA ratios were 0.4, 0.5, 0.6, 0.7 and 0.8. The curing temperature were room temperature. The UCS tests were carried out on the samples after 7, 14, 28, 60 and 90 days of curing in accordance with ASTM C69-09. The carbon footprint, defined as CO_2-e emitted (kg CO_2-e/ton), of FA geopolymer and FA–PVA geopolymer stabilized soft clay is calculated and compared with that of cement stabilized soft clay at the same UCS. The

CO_2 emission calculation considered the mining, processing and manufacturing of raw materials. The growth of geopolymerization structures on the samples was illustrated using X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM). The XRD traces were used to provide fundamental information on geopolymerization structures.

CHAPTER II

THEORETICAL BACKGROUND AND LITERATURE REVIEW

2.1 Geopolymer

The Cordi-Géopolymère private research laboratory in Saint-Quentin in 1972 discovered a fresh inorganic material called as ‘geopolymer'. Geopolymer can be formed at low temperature and small amount of time and based on the alkaline activation of easily obtainable natural and by-product silica and alumina materials happening as a network of the alumino-silicates (Davidovits 1988c). After shorter setting and hardening time, geopolymer with tightly packed poly-crystalline structure is formed showing better mechanical properties Geopolymer is extremely environmentally attractive for various reasons. Its performance as construction materials can be compared with Portland cement in lots of ways but the geopolymer needs no heat in its manufacturing process. This implies a substantial benefit with regards to reducing global CO_2 emissions. Furthermore, the utilization of industrial by-products meets the increasing trend towards waste re-utilization (Jiminez et al. 2004). Geopolymer is definitely an alumino-silicate material which includes excellent physical and chemical properties of numerous applications (Komnitsas and Zaharaki 2007).

2.2 Chemistry of geopolymer

Three sources are used to form the geopolymer, i.e. raw materials, inactive filler and geopolymer liquor. Raw materials might be natural (alumino-silicate) minerals or industrial wastes e.g. fly ash, slag, and waste glass. Inactive filler, mainly kaolinite or meta-kaolinite, is employed for supplying Al^{3+} ions (Ikeda 1998). Geopolymer liquor is definitely an alkali hydroxide solution required for dissolving raw materials while sodium (or potassium) silicate solution acts as a binder, alkali activator and dispersant or plasticizer (Phair 2001). Geopolymer (poly (sialates)) contains silicate (SiO_4) and aluminate oxides (AlO_4) tetrahedral alternately linked where all oxygen atoms are exchanged (Davidovits 1976). Positive ions (Na^+, K^+ and Ca^{2+}) must certainly be contained in the framework voids to balance the negative charge of Al^{3+}.Poly (sialates) is chain polymers with Si^{4+} and Al^{3+} in 4-fold coordination with oxygen and their empirical formula could be expressed as:

$$M_n(-(\mathrm{SiO}_2)_z - AlO_2)_n wH_2O$$

Where z is 1, 2, 3, or higher

M is a monovalent cation such as K^+ or Na^+

n is the degree of poly-condensation

The forms of polysialates distinguished are illustrated in Figure 2.1 (Davidovits 1988a). The complex geopolymer structure thus contains chains, sheet-like and three-dimensional networks composed of various unit kinds of connected SiO_4 and AlO_4 tetrahedral (Singh et al. 2005).

Geopolymerization can be an exothermic procedure that is conducted through oligomers dimer which provide the specific unit structures for the three dimensional macromolecular edifice. Among several hardening mechanisms could be the chemical reaction of alumino-silicate oxides with alkalis and alkali–polysilicates, leading to polymeric Si–O–Al bonds with a $(Si_2O_5, Al_2O_2)n$ formula, which may be accomplished by calcining alumino-silicate hydroxides $(Si_2O_5,Al_2(OH)_4)$ through the reaction (Davidovits 1988a).

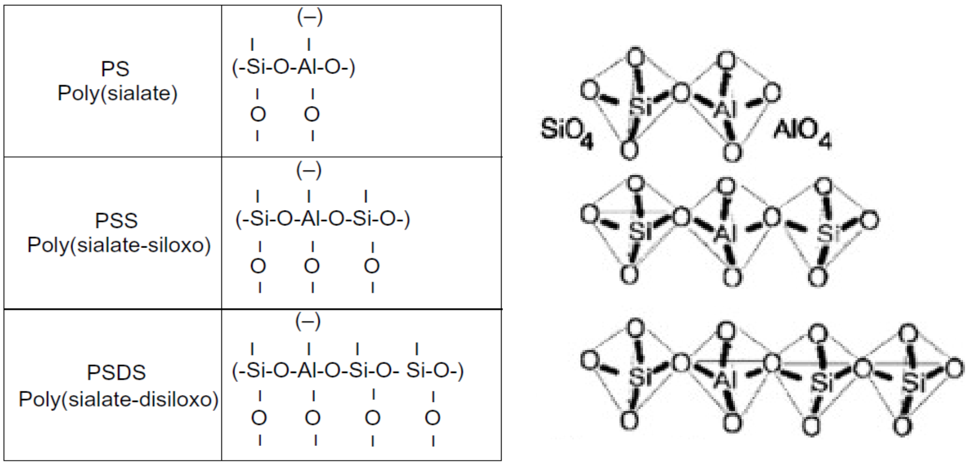


Figure 2.1 The types of polysialates (Davidovits 1988a).

Primary steps of geopolymerization involve dissolution of solid alumino-silicate oxides in *M*OH solution (*M*: alkali metal), diffusion or transportation of dissolved Al and Si complexes from the particle surface to the inter-particle space, formation of a gel phase caused by the polymerization between added silicate solution and Al and Si complexes, and finally hardening of the gel phase (Van Jaarsveld 2000; Xu 2001) for the poly-condensation occurring during geopolymerization of minerals:

$$\text{Al–Si material}_{(s)} + M\text{OH}_{(aq)} + \text{Na}_2\text{SiO}_{3\,(s)\ \text{or}\ (aq)} \quad (1)$$

$$\downarrow$$

$$\text{Al–Si material}_{(s)} + [M_z\,(\text{AlO}_2)_x\,(\text{SiO}_2)_y \cdot nM\text{OH} \cdot m\text{H}_2\text{O}]\ \text{gel} \quad (2)$$

$$\downarrow$$

$$\text{Al–Si material}_{(s)} + [M_a\,((\text{AlO}_2)_a\,(\text{SiO}_2)_b)\ nMOH \cdot m\text{H}_2\text{O}] \quad (3)$$

In reactions (1) and (2), the amount of Al–Si material used is dependent upon the particle size, the extent of dissolution of Al–Si materials, and the concentration of the alkaline solution. The forming of $[M_z(\text{AlO}_2)_x(\text{SiO}_2)_y nM\text{OH}m\text{H}_2\text{O}]$ gel is basically on the basis of the extent of dissolution of alumino-silicate materials, while geopolymer with amorphous structure is formed during the reaction (3). Enough time necessary for the alumino-silicate solution to create a continuous gel is dependent upon raw material processing conditions (Ivanova et al. 1994).

Dissolution of the starting materials could be the major step that has a twofold role, i.e. firstly, poly-sialate forming species are liberated from the starting materials (Duxson et al. 2007a; Grutzeck and Siemer 1997), and secondly, dissolution activates the top and binding reactions occur significantly supporting the ultimate strength of the structure. The extent of the dissolution part of geopolymerization is not fully clear whilst the extent to which other factors complement or no dissolution must be further investigated (Phair 2001).

Under alkaline conditions, alumino-silicates are changed into extremely reactive materials, and generally speaking it is thought that the dissolution process is initiated by the clear presence of hydroxyl ions. Higher levels of hydroxyl ions facilitate the dissociation of different silicate and aluminate species, promoting thus further polymerization (Davidovits 1988b; Phair et al. 2000). However, in case a quite high alkaline environment (>30 mol% overall Na_2O content) is employed, the connectivity of silicate anions might be reduced, causing poor polymerization (Singh et al. 2005).

The analysis of the dissolution on different alumino-silicate industrial minerals and by-products indicated that the extent of dissolution is higher when NaOH is employed in place of KOH (Panagiotopoulou et al. 2007) due to the smaller size of Na^+ that may better stabilize the silicate monomers and dimmers contained in the solution. This thus enhances the minerals dissolution rate (Xu and Van Deventer 2000a). Additionally, it absolutely was reported that Si and Al appear to possess a synchronized leaching behavior in both alkaline media.

Furthermore, there have been the studies of the dissolution on various kinds of fly ash in caustic soda solutions as much as 15 M at 25 and 80°C. It had been reported that high dissolution of Al_2O_3 is seen in 80°C, while the increased dissolution of SiO_2 occurs during leaching with increasing NaOH concentrations in 25°C.

2.3 Geopolymer Development

Geopolymer cements develop through a series of several distinct reaction processes from initial pozzolanic activation to final microstructure development. The benefits of using pozzolans as a matrix binder for agglomeration is that they tend to be economical, environmentally-friendly, more absorbent of liquids and produce a highly durable product (Jiminez et al. 2005). The major processes are dissolution of the aluminosilicate species within a highly basic, alkaline environment, polymerization of the dissolved minerals into short-lived structural gel, precipitation of formed hydration products similar to natural zeolites and final hardening of the matrix by excess water exclusion and the growth of crystalline structures (Petermann et al. 2010). Figure 2.2 illustrates the overall polymerization process in alkali-activated geopolymers.

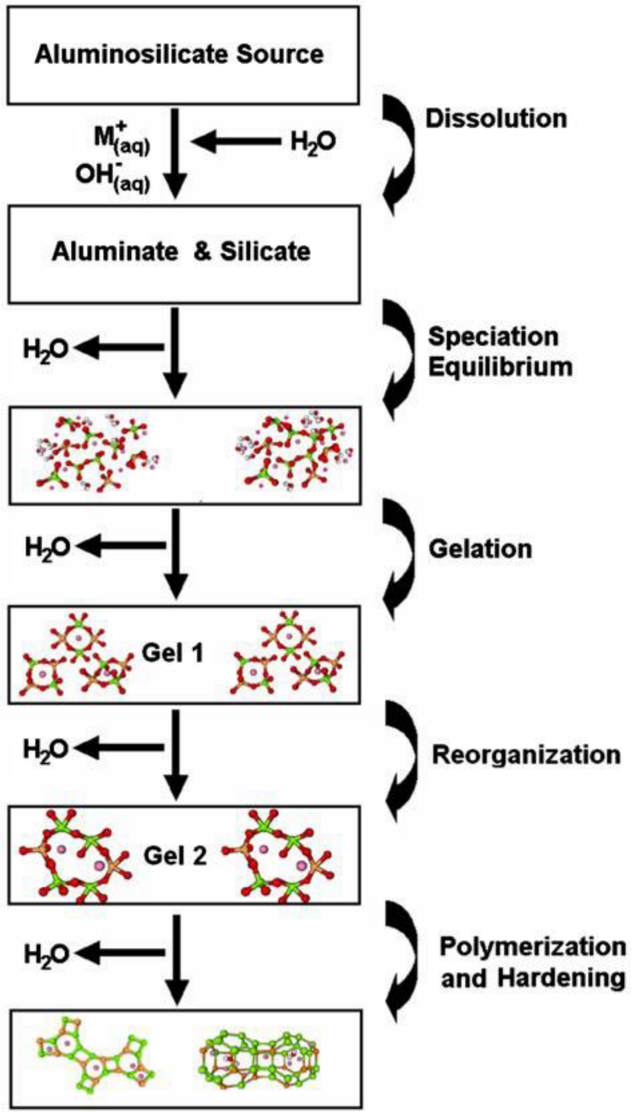


Figure 2.2 Geopolymer Development Model (Vijaya Rangan).

2.3.1 Dissolution

Dissolution occurs immediately upon contact between the alkaline solution and the pozzolanic material and allows for ionic interface between species and the breaking of covalent bonds between silicon, aluminum and oxygen atoms. Similarly to PCC, this process generates rapid and intense heat and is directly proportional to the pH level of the activating solution. The rate of dissolution is relevant to the amount and composition of the ashes and the pH of the activating solution (Jiminez et al. 2006: Xie and Xi. 2001:Petermann et al. 2010).

2.3.2 Polymerization

The polymerization process involves a substantially fast chemical reaction under alkaline condition on Si-Al minerals, resulting in a three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds. The formed gel product contains alkaline cations which compensate for the deficit charges associated with the aluminum-for-silicon substitution (Xie and Xi. 2001:Petermann et al. 2010). An intermediate, aluminum-rich phase is first formed which then gives way to a more stable, silicon-rich three-dimensional gel product of form Q^4(nAl), which is dependent upon curing conditions and activator type (Jiminez et al. 2006:Petermann et al. 2010).

2.3.3 Growth

During this process, the slow growth of crystalline structures become evident as the nuclei of the polymerized gel reaches critical size. The matrix crystallinity is relative to the rate by which precipitation occurs: fast reactions between alkali and ash do not allow time for growth of a well-structured crystalline environment (representative of typical zeolites). Therefore, most hardened geopolymer cements are referred to as zeolitic precursors rather than actual zeolites. The final product of geopolymerization is an amorphous, semi-crystalline cementitious material. (Petermann et al. 2010).

2.4 Raw materials

Theoretically, any material composed of silica and aluminium can be alkali-activated. So far the investigations performed have used the following raw materials:

(a) kaolinitic clays (Barbosa et al. 2000; Davidovits 1979; Davidovits and Sawyer 1985);

(b) metakaolin (Alonso and Palomo 2001a; Alonso and Palomo 2001b; Davidovits 1999);

(c) fly ashes (Fernandez-Jimenez and Palomo 2005; Palomo et al. 1999);

(d) blast furnace slag (Fernandez-Jimenez et al. 1999; Purdon 1940; Wang and Scrivener 1995);

(e) mixtures of fly ashes and slag (Puertas and Fernandez-Jimenez 2003; Puertas et al. 2000);

(f) mixtures of fly ashes and metakaolin (Swanepoel and Strydom Appl Geochem 2002);

(g) mixtures of slag and metakaolin (Cheng and Chiu 2003);

(h) mixtures of slag and red mud (Zhihua et al. 2002; Zhihua et al. 2003);

(i) mixtures of fly ashes and non-calcined materials like kaolin and stilbite (Xu et al. 2002).

In 1974, Kaolinite and other clays were initially utilized in as fillers for the encapsulation of radioactive wastes right into a monolithic solid. Davidovits primarily used kaolinite and calcined kaolinite (metakaolin) as the supply of alumino-silicate oxides to be able to synthesize geopolymer (Davidovits 1984). A number of other researchers also have centered on the manufacture of geopolymer products and their industrial applications by utilizing either kaolinite or metakaolinite as the key reactant (Barbosa and MacKenzie 2003a; Barbosa and MacKenzie 2003b; Cioffi et al. 2003; Xu and Van Deventer 2002).

Kaolinite provides structure forming species to the entire geopolymerization process. Xu and Van Deventer (Xu and Van Deventer 2000b) stated that the addition of kaolinite is essential because the rate of Al dissolution from the raw materials is insufficient to generate a gel of the required composition. The lower reactivity of kaolinite requires sufficient time for interactions among the original materials or the source material and the gel phase to form. Little is known in regards to the behavior with this gel phase and the extent to which the character of the starting materials and the specific concentrations in solution are affecting its formation and setting (Xu and Van Deventer 2002). However, a massive amount added kaolinite might not take part in the synthesis reaction at all (Van Jaarsveld et al. 2002; Zaharaki et al. 2006).

Some authors (Xu and Van Deventer 2000b) studied sixteen different alumino-silicate minerals with the addition of kaolinite to be able to synthesize geopolymer and observed that for the majority of the alumino- silicate minerals the addition of kaolinite is required for the forming of gel. Only if kaolinite is utilized without the presence of other alumino-silicates, a poor structure is formed, which means synergy between different aluminosilicates is apparently quite important (Xu and Van Deventer 1999).

Furthermore, other authors (Palomo et al. 1999; Xu and Van Deventer 1999) studied the usage of natural minerals and wastes (in particular fly ash) in geopolymerization, as well as the immobilization of toxic metals was investigated extensively in recent years Also, some authors (Van Jaarsveld et al. 1998) have used black coal fly ash and construction wastes as the foundation for geopolymerization.

Some authors (Xu et al. 2002) used fly ash, kaolinite and albite (Na-rich end member of the Albite-Anorthite Series.) in several combinations for the synthesis of geopolymer. It is thought that the larger reactivity of fly ash and albite, the interaction between the original materials and the gel phase along with the reinforcing effect induced by the large unreacted albite particles are accountable for an ideal mechanical behavior (high compressive strength and low cracking probability).

Some authors (Davidovits 2005a) studied the 15 types of combustion fly ashes were tested for suitability in geopolymer cements. The samples were cured at room temperature and the compressive strength was measured after 28 days. A sizable variation in the behavior of the fly ashes was noticed which range from unworkable situations to strength of 95 MPa.

Some authors (Xu and Van Deventer 2000b) proved that calcined materials for instance slag, fly ash and metakaolinite which are generally amorphous, usually display a greater reactivity during geopolymerization in comparison to non-calcined materials. This is explained by the truth that calcination activates materials by changing their crystalline into amorphous structure with subsequent storage of the additional energy (Xu et al. 2002).

Moreover, the production of consistent geopolymer from heterogeneous industrial waste sources is really a challenging issue for the minerals industry, since raw material suitability cannot be fully guaranteed by elemental composition analysis (Fernandez-Jimenez and Palomo 2003). So far as fly ash based geopolymers are involved, the mechanical strength increases as a result of the formation of an Al-rich alumino-silicate gel during the initial stage of alkaline activation of ash particles, and may further increase as a result of the Si enrichment of the material (Fernandez-Jimenez et al. 2006). Therefore, it is important to control Al and Si dissolution from the raw materials, through pre-processing and/or utilization of combinations of raw materials with various reactivities (Duxson et al. 2007a)**.**

2.5 Factors affecting of compressive strength

Measurements of compressive strength are employed by many researchers as an instrument to measure the success of geopolymerization due to the low cost and simplicity of compressive strength testing, along with of the truth that strength development is really a primary way of measuring the utility of the materials found in different applications of the construction industry (Provis et al. 2005).

The compressive strength of geopolymer is dependent upon many factors such as the gel phase strength, the ratio of the gel phase/undissolved Al–Si particles, the distribution and the hardness of the undissolved Al–Si particle sizes, the amorphous nature of geopolymer or the amount of crystallinity, and the surface reaction between the gel phase and the undissolved Al–Si particles (Van Jaarsveld et al. 2003; Xu 2001)

Furthermore, factors such as % CaO, % K_2O and the kinds of alkali have an important correlation with compressive strength. The significance of the molar Si/Al ratio during the alkaline dissolution of the individual minerals suggests that compressive strength is obtained by complex reactions between the mineral surface, kaolinite and the concentrated sodium silicate solution.

After geopolymerization, the undissolved particles remain bonded in the matrix, so the hardness of the minerals correlates positively with the last compressive strength (Xu and Van Deventer 1999; Xu and Van Deventer 2000b). During geopolymerization of natural minerals, it is known that after adding aggregate for instance granular sand to the geopolymer mixture, the compressive strength increases (Xu et al. 2002).

The quantity of metakaolinite added to the geopolymer matrix, along with the KOH concentration and the addition of sodium silicate, also play an important role on the ultimate compressive strength. some authors (Swanepoel et al. 1999) proved that the strength increases with increasing addition of metakaolinite. The main reason might be that the more metakaolinite added, the more Al gel forms in the system, causing a higher level of polymerization. Some authors (Wang et al. 2005) proved experimentally that the compressive strength, along with the apparent density and the content of the amorphous phase of metakaolinite-based geopolymer, increase with the increase of NaOH concentration within the range 4–12 mol/L. This is caused by the enhanced dissolution of the metakaolinite particulates and therefore the accelerated condensation of the monomer in the clear presence of higher NaOH concentration.

Some authors (Luz Granizo et al. 2007) supported the proven fact that the alkali activation of metakaolin using sodium silicate and NaOH containing solutions results in the production of material exhibiting higher mechanical strength compared to the activation with only NaOH. Furthermore, the flexural strength increases once the activator volume decreases or the concentration of Na increases.

Compressive strength of metakaolin based geopolymer increased linearly by approximately 400% from Si/ Al = 1.15 to Si/Al = 1.90, where it obtained its maximum value, before decreasing again at the greatest Si/Al ratio of 2.15 (Duxson et al. 2005) (Figure 2.3).

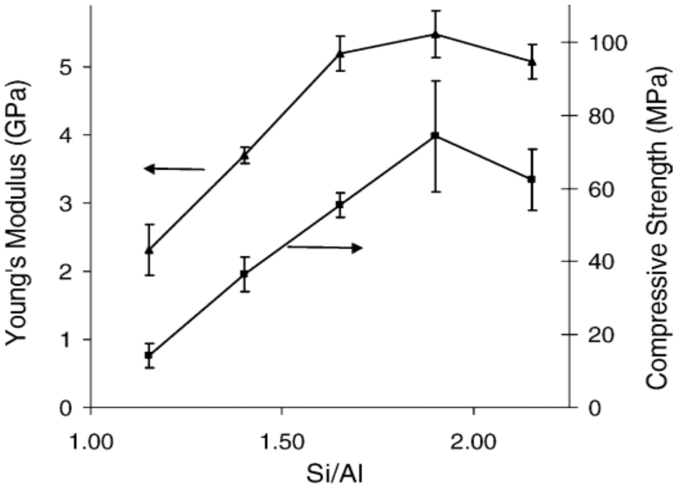


Figure 2.3 Young's moduli (▲) and ultimate compressive strengths (■) of geopolymers. (Duxson et al. 2005).

It is thought that the quantity of unreacted materials in specimens with higher silica content acts as defect site and includes a negative impact on strength. Higher strength was recorded once the ratios SiO_2/Al_2O_3 and Na_2O/Al_2O_3 were 3.0–3.8 and approximately 1, respectively (Duxson et al. 2005; Stevenson and Sagoe-Crentsil 2005). However, these initial ratios might be changed based on the total amount of the raw materials used as Al_2O_3 and SiO_2 source (Silva et al. 2007).

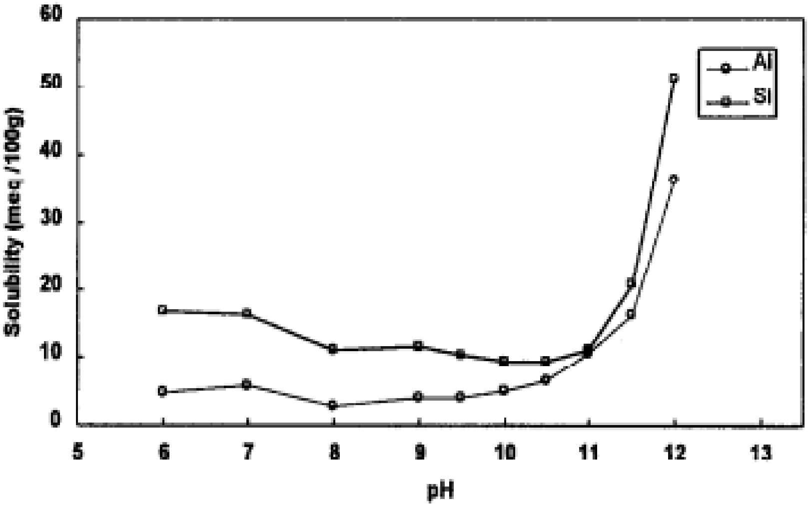


Figure 2.4 Dissolution of Al Si from metakaolin as a function of pH over a 5 h period (Phair and Van Deventer 2001).

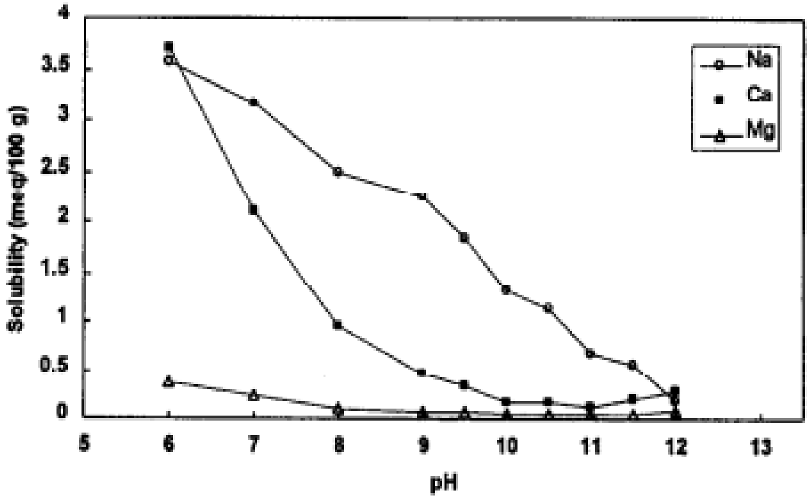


Figure 2.5 Dissolution of Na, Ca, and Mg metakaolin as a function of pH over a 5 h period. Ti and Fe concentration were negligible (Phair and Van Deventer 2001).

Based on some authors (Phair and Van Deventer 2001), probably the most significant factor that controls the compressive strength of fly ash-based geopolymer may be the pH of the original alkali metal silicate precursor. When working with cement as a setting additive in the geopolymer matrix, the compressive strength increases almost exponentially with increasing pH (Figure 2.4 and 2.5).

The larger alkali content was found to enhance solid dissolution but also to cause alumino-silicate gel precipitation at very early stages, causing lower compressive strength (Lee and Van Deventer 2002). Additionally, the calcium content in fly ash based geopolymer, along with the water/fly ash ratio, is apparently highly important (Van Jaarsveld et al. 2003).

Some authors (Palomo et al. 2007) proved that alkali activation of highly blended cements containing 30% Portland cement clinker and 70% fly ash results in acceptable mechanical strength, which will be strongly affected by the sort of the alkaline activator put into the system. Some authors (Kumar et al. 2005) indicated that mechanically activated fly ash based geopolymer exhibits higher compressive strength due to the formation of a tight microstructure. Mechanical activation of fly ash appears to favor geopolymerization, since the reaction requires less time and occurs at lower temperature.

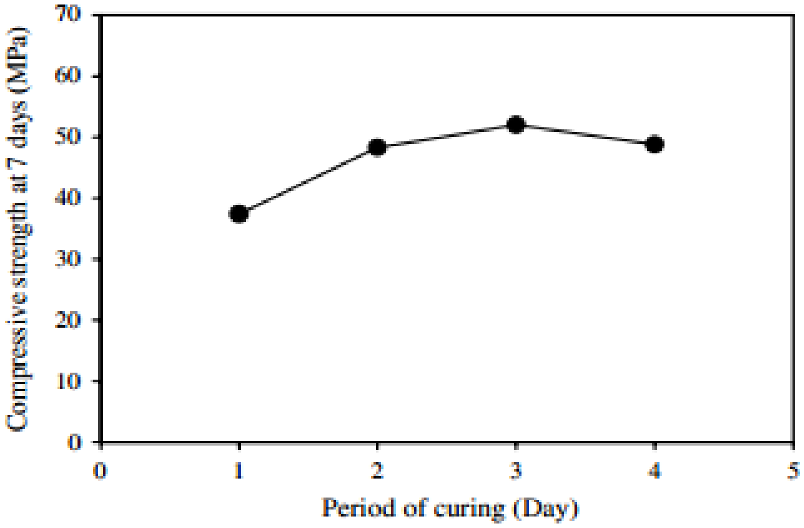


Figure 2.6 Duration of heat curing and strength cured at 60°C with delay time (Chindaprasirt et al. 2007).

The influence of curing temperature and time on the flexural properties of geopolymer centered on class C fly ash has been investigated by some authors . (Miller et al. 2005). It had been discovered that the curing regime includes a very significant effect on the physical and chemical properties of fly ash-based geopolymer. Indeed, the mere escalation in synthesis temperature is sufficient to boost the degree of long-range ordering in geopolymer binders (Duxson et al. 2007a).

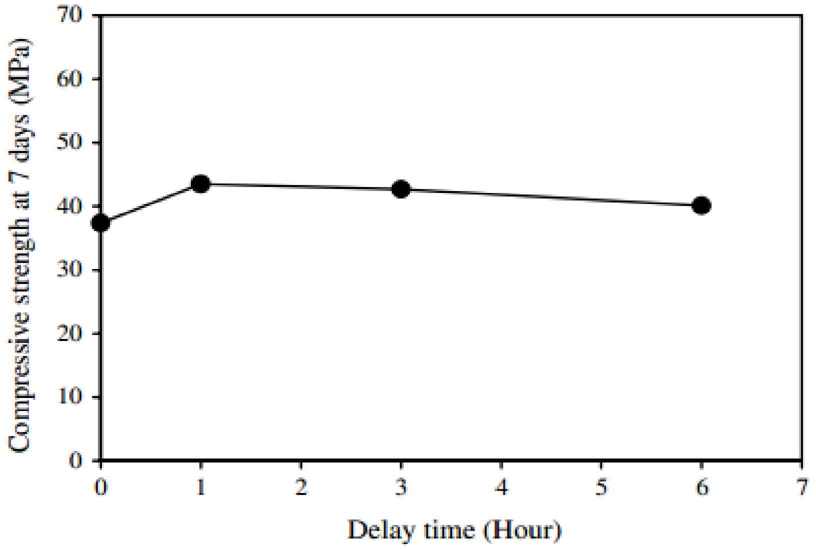


Figure 2.7 Strength and delay time of geopolymer mortar with 60°C heat curing for 24 h (Chindaprasirt et al. 2007).

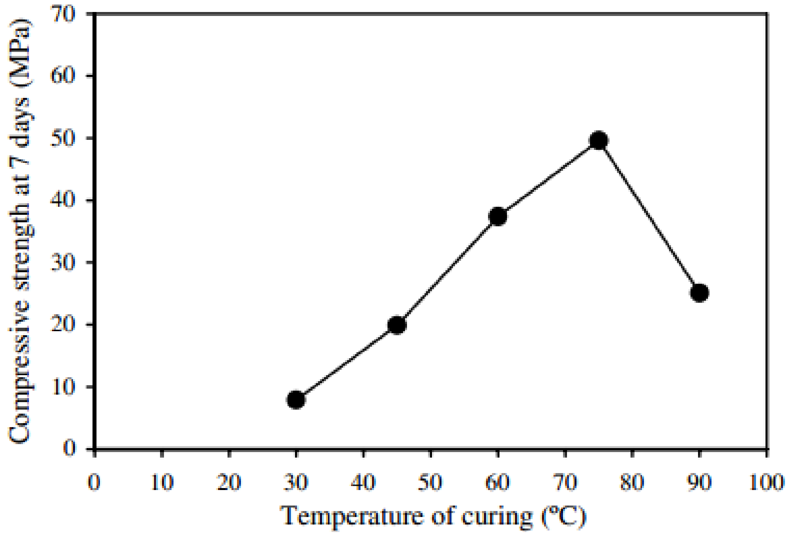


Figure 2.8 Strength and temperature of curing of geopolymer with no delay time before curing (Chindaprasirt et al. 2007).

Geopolymers include relatively massive amount water in large pores readily available for evaporation, which does not end up in capillary strain. This fact may account for the low temperature region of dimensional stability (Barbosa and MacKenzie 2003b). Once the freely evaporable water is taken from pores, the top part of the gel structure increases as water is liberated from the outer lining of the gel, and small pores leading to shrinkage are observed. In this instance, the gel contraction might be correlated with the decrease in surface area (Duxson et al. 2007b).

Moisture evaporation results in deterioration of the geopolymer product which cannot develop satisfactory strength. Furthermore, the addition of water improves the workability of the mortar (Chindaprasirt et al. 2007) (Figure 2.6, 2.7 and 2.8).

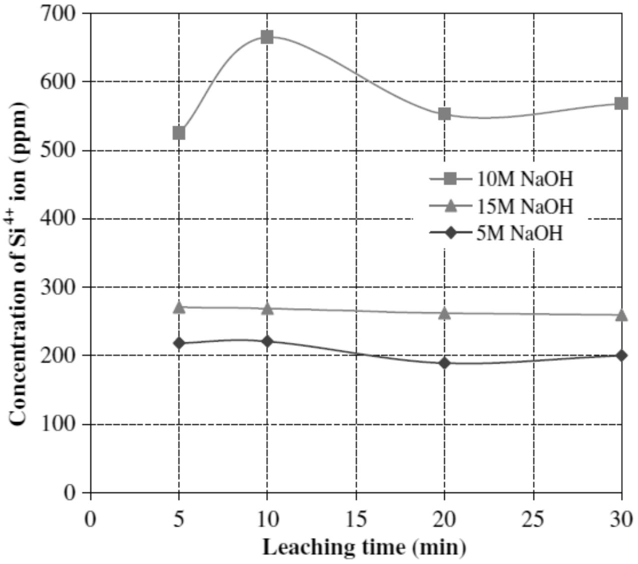


Figure 2.9 Si^{4+} ion concentration with fly ash/NaOH = 3:1 in 5, 10, and 15 M NaOH (Rattanasak and Chindaprasirt 2009).

Furthermore, some authors (Rattanasak and Chindaprasirt 2009) studied the influence of NaOH solution on the synthesis of fly ash geopolymer. To produce geopolymer paste, separate mixing and normal mixing were used. For separate mixing, NaOH solution was blended with fly ash for the initial 10 min; the subsequently sodium silicate solution was added to the mixture. For normal mixing, fly ash, sodium hydroxide and sodium silicate solution were incorporated and mixed at the same time. Geopolymer were cured at 65°C for 48 h. At 5 M NaOH, the dissolution was low because of the relatively low base condition. For 10 M NaOH, the base condition was higher and the dissolution was, therefore, increased. For the 15 M NaOH, the dissolution was again reduced owing primarily to a rise in coagulation of silica (Bergna and Roberts 2006) (Figure 2.9 and 2.10).

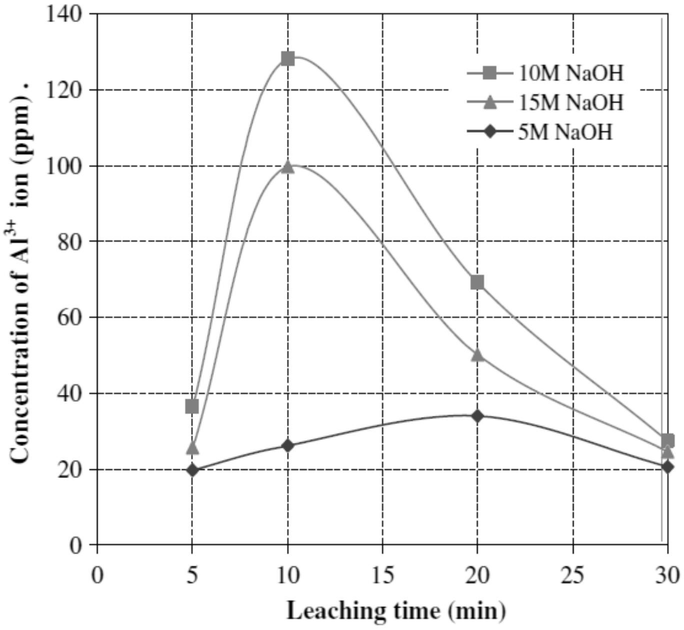


Figure 2.10 Al^{3+} ion concentration with fly ash/NaOH = 3:1 in 5, 10, and 15 M NaOH (Rattanasak and Chindaprasirt 2009).

The water within geopolymer and its subsequent removal by evaporation plays a significant role in obtaining a crack-free geopolymer, which means that rapid drying during curing ought to be avoided, while curing at less relative humidity (e.g. 30%) is advised (Perera et al. 2004). Also, it had been found that when the curing temperature is high (approximately 90°C), the geopolymer will substantially lose the moisture (Bakharev 2005b).

Other researches (Khalil and Merz 1994; Van Jaarsveld et al. 2002) proved that curing for longer amounts of time at elevated temperature generally seems to weaken the structure, suggesting that small levels of structural water must be retained to be able to eliminate cracking and maintain structural integrity. It appears that prolonged curing at elevated temperatures breaks down the gelular structure of the geopolymer synthesis mixture, leading to dehydration and excessive shrinkage, while long procuring at room temperature is good for strength development when utilizing fly ash as a raw material (Bakharev 2005b). Furthermore, J.G.S Van Jaarsveld et al.2002 (Table 1) stated that initial curing at higher temperatures (above 50–80°C) does not increase compressive strength substantially above that accomplished by curing at room temperature.

Generally speaking, it is needed to adequately cure to be able to achieve advanced mechanical and durability performance. Additionally, the setting time is practically important as it defines enough time necessary for transport, placing and compaction (Teixeira-Pinto et al. 2002). In order to raise the acceptance of geopolymerization by the industry, curing conditions ought to be just like those utilized in OPC production.

Table 1 The effect of curing conditions on the compressive strength of a matrix containing Macquarie fly ash with compositional variables: clay (kaolinite) content = 15% (mass) (J.G.S. Van Jaarsveld et al. 2002).

Time (h)	Temperature (°C)					
	30	50	70	30 B	50 B	70 B
6	6	-	14	19	-	28
12	15	26	34	7	22	21
24	20	12	33	19	24	29
48	19	-	28	21	-	15
Average (12/24 h samples)	17	19	34	13	23	25

[a] Samples were cured in an oven, open to the atmosphere except for samples denoted by “B” which were cured in sealed plastic bags under the same conditions. Compressive strength values in MPa.

Based on researches (Hardjito et al. 2004) collected the consequence of geopolymer concrete development. The binder in this concrete, the geopolymer paste, is formed by activating by-product materials, for example low-calcium (Class F) fly ash. he found that numerous variables for instance curing temperature, curing time, concentration of alkali solution, and delay time effected to compressive strength of geopolymer concrete as follows;

- The curing temperature in the range of 30 to 90°C increases, the compressive strength of geopolymer concrete also increases. Higher curing temperature triggered larger compressive strength, although a rise in the curing temperature beyond 60°C did not raise the compressive strength substantially (Figure 2.11).

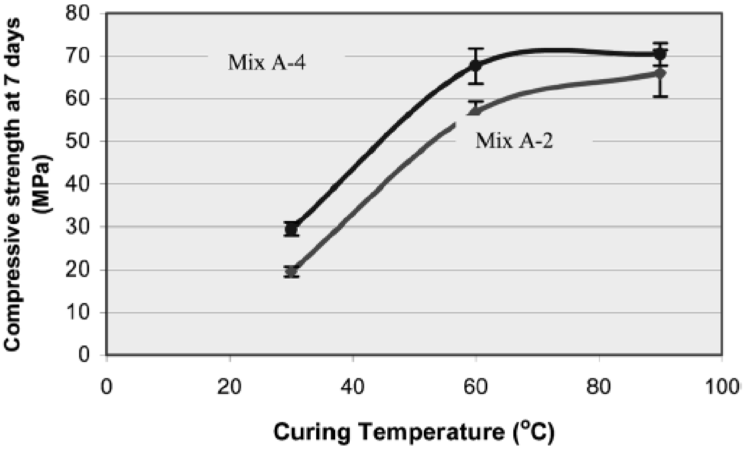


Figure 2.11 Effect of curing temperature on compressive strength (Hardjito et al. 2004).

- The influence of curing time on the compressive strength indicate that a longer did not produce weaker material as claimed by researches (Van Jaarsveld et al. 2002). However, the escalation in strength for curing periods beyond 48 h is not significant (Figure 2.12).

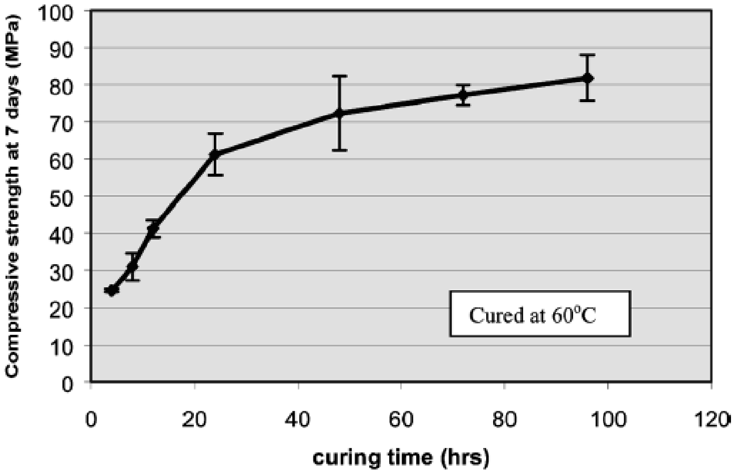


Figure 2.12 Influence of curing time on compressive strength (Hardjito et al. 2004).

- The concentration of sodium hydroxide as measured by Molarity (second column). Mixture A-3 with higher concentration of NaOH yielded higher compressive strength than Mixture A-1. The same trend can also be observed for the Mixtures A-2 and A-4 (Table 2).

Table 2 Effect of parameter on compressive strength (Hardjito et al. 2004).

Mixture	Concentration of NaOH liquid in molarity (M)	Sodium silicate/NaoH liquid ratio by mass	7-day compressive strength after curing at 60°C for 24 h. (MPa)
A-1	8 M	0.4	17.3
A-2	8 M	2.5	56.8
A-3	14 M	0.4	47.9
A-4	14 M	2.517.3	67.6

- The new concrete was permitted to stand at room temperature after mixing and just before being put into molds for a particular amount of time. The new geopolymer concrete is easily handled as much as 120 min without the sign of the setting and the degradation in the compressive strength (Figure 2.13).

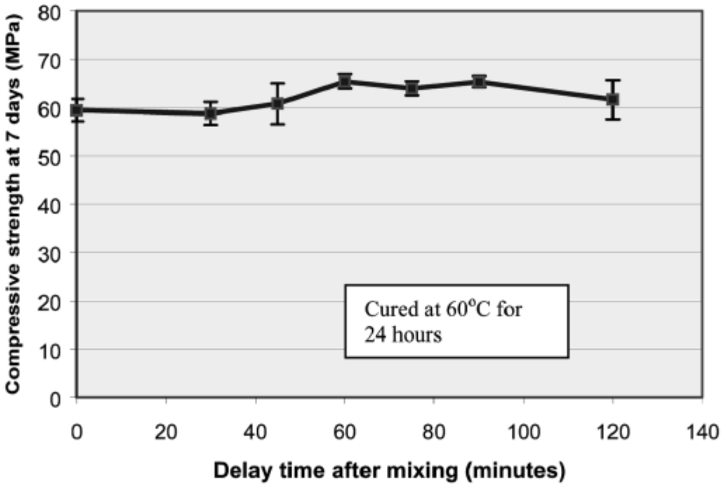


Figure 2.13 Influence of delay time on compressive strength (Hardjito et al. 2004).

- The compressive strength does not vary with the age of concrete, when cured for 24 h. This observation is in contrast to the well-known behavior of OPC concrete, which undergoes a hydration process and hence gains strength over time (Figure 2.14).

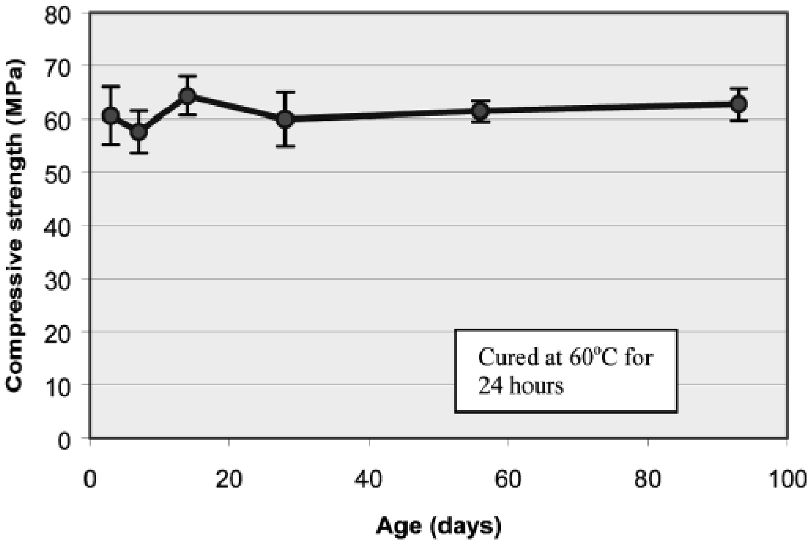


Figure 2.14 Compressive strength at different ages (Hardjito et al. 2004).

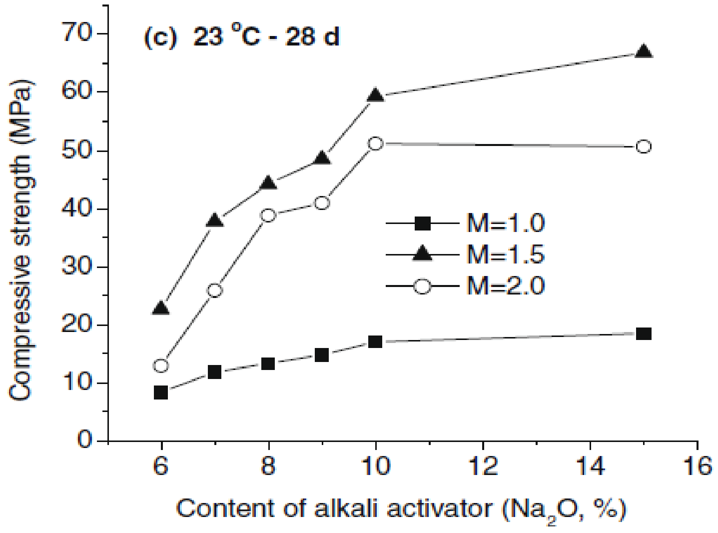


Figure 2.15 Effects of modulus (M) and content of the mixed alkali activator on the compressive strength of fly ash geopolymer cured at room temperature of 23°C for 28 days (Guo et al. 2010).

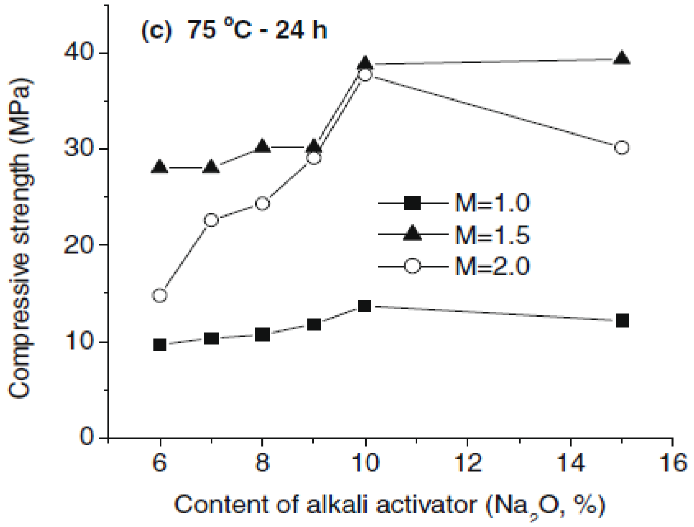


Figure 2.16 Effects of modulus (M) and content of the mixed alkali activator on the compressive strength of fly ash geopolymer cured at room temperature of 75°C for 4, 8, and 24 h (Guo et al. 2010).

Some authors (Guo et al. 2010) indicated that the high compressive strength was obtained once the class C fly ash (CFA) was activated by the mixed alkali activator (sodium hydroxide and sodium silicate solution) with the optimum modulus viz., molar ratio of SiO_2/Na_2O of 1.5. The appropriate content of the mixed activator was 50% as evaluated by the mass proportion of Na_2O to CFA. The compressive strength of those samples was 63.4 MPa when these were cured at 75°C for 8 h accompanied by curing at 23°C for 28 days (Figure 2.15 and 2.16).

2.6 Effect of Polyvinyl Alcohol (PVA) geopolymer on compressive strength

Zhang et al. (2004) studied the novel modification method for inorganic geopolymer by using water soluble organic polymers. The ‘‘organic polymers modified kaolinite Na-PSs’’ were called ‘‘OMPSs’’. The compressive strength and weight loss of PVA–OMPS were 5.6% and 59.6 higher than those of kaolinite Na-PS when PVA–OMPS samples were mixed with modification agent of 0.2% (Figure 2.17).

Kou and Poon (2010) investigated properties of concrete prepared with PVA-impregnated recycled concrete aggregates. It was found that the 10% fines value of the PI-RCA was higher, and the water absorption of the PI-RCA were lower when compared to the untreated RCA (Figure 2.18).

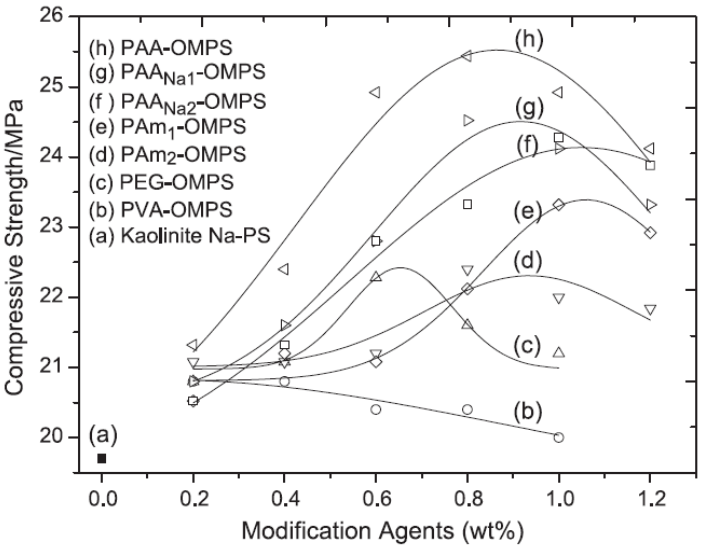


Figure 2.17 Comparison of compressive strength between OMPSs with the following modification agents: (a) none, (b) PVA, (c) PEG, (d) PAm2, (e) PAm1, (f) PAANa2, (g) PAANa1, (h) PAA. (Zhang et al. 2004).

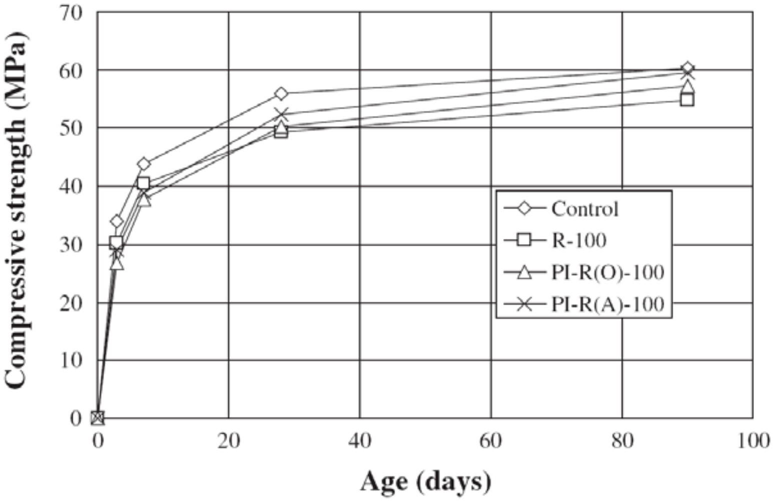


Fig. 2. Development of compressive strength of the concrete mixtures.

Figure 2.18 Development of compressive strength of the concrete mixtures (Kou and Poon, 2010).

2.7 Microstructural techniques

Several easy or advanced techniques can be utilized to acquire maximum information and elucidate geopolymerization mechanisms. The capability of Al–Si minerals to undergo geopolymerization might be predicted by specific surface area measurements, which offer an indication of how much surface area participates in heterogeneous reactions inside a solid–fluid system (Van Jaarsveld et al. 2002).

Optical microscopy provides a visible description of the microstructure because it is shown in scale the physical size and model of the different aspects of geopolymer. X-ray fluorescence (XRF) spectrometry works extremely well for elemental analysis of Al–Si minerals. X-ray diffraction (XRD) might be also a helpful tool although the quantity of information which may be obtained is restricted as a result of substantial amorphous nature of geopolymer. However it will provide information concerning the extent to which crystalline starting materials have reacted (Van Jaarsveld et al. 2002). Figure 2.19 presents the XRD pattern of geopolymer when fly ash is employed as raw materials and activated employing a NaOH (8M) solution and cured at 85°C (20 h) (Fernandez Jiminez et al. 2004).

Scanning electron microscopy (SEM) allows visual examination of a product from millimeters to micrometers to yield definitive topographical information along with good physical and mechanical description of the microstructure of crystalline and amorphous materials, which may not be detected by other techniques (Duxson et al. 2006; Lee 2002). Some authors (Jiminez et al. 2004) provided the geopolymer microstructures (Figure 2.20 – 2.23) are characterized by way of a dispersion of distinctive morphologies in a large of predominantly featureless hydration product (alumino-silicate gel). Occasionally, cracking in the item is observed. This might be because of the thermal treatment carried out within the activation process, mechanical damage during sample preparation or to drying shrinkage in the vacuum of the electron microscope. The relatively low magnification images (Figure 2.20 and 2.21) offer a summary of the distribution of numerous constituent phases with an increase of local detail being provided in Figure. 22 and 23.

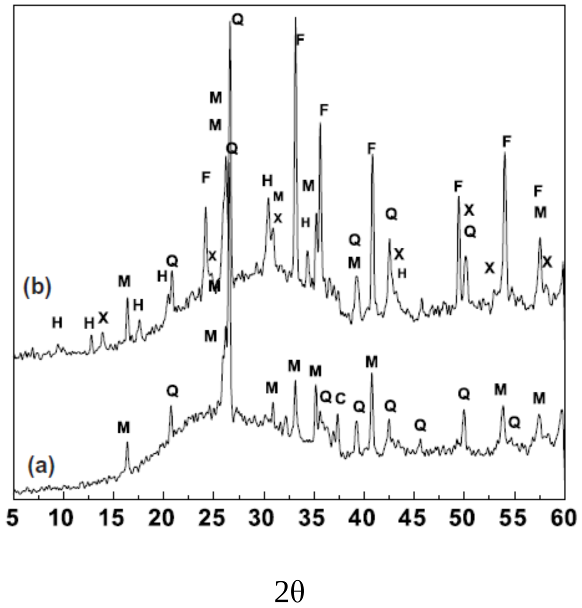


Figure 2.19 XRD spectra (a) un-reacted fly ash; (b) alkali-activated fly ash 20 h at 85°C Q=Quartz; M= Mullite; F=Hematite; C=CaO; H=Herschelite; X=Hydroxysodalite (Fernandez Jiminez et al. 2004).

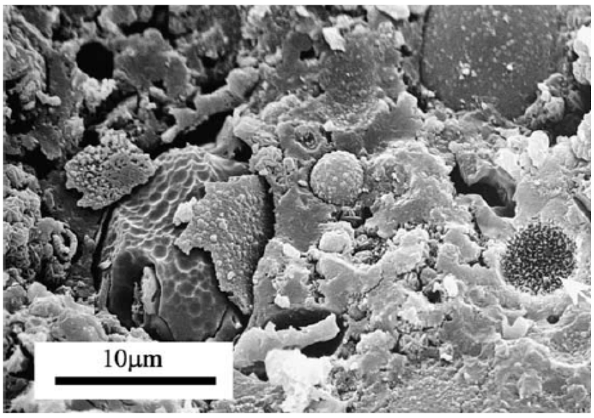


Figure 2.20 SEM micrograph of fracture surface of alkali-activated PFA geopolymer. Fe_2O_3 is arrowed (Jiminez et al. 2004).

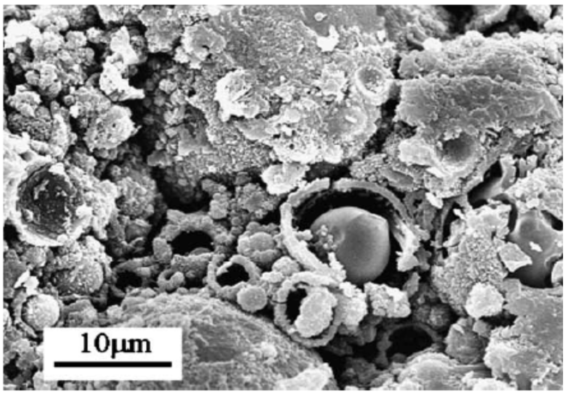


Figure 2.21 SEM micrograph of fracture surface of alkali-activated PFA geopolymer (Jiminez et al. 2004).

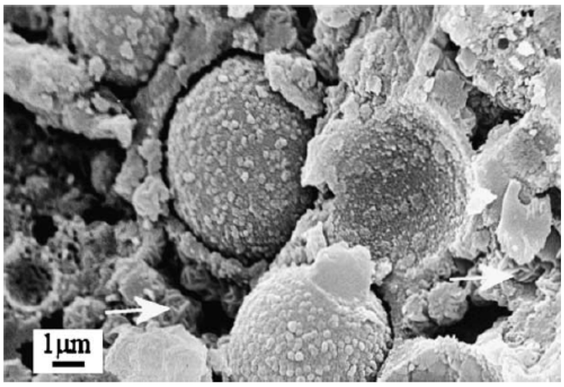


Figure 2.22 SEM micrograph of fracture surface of alkali-activated PFA geopolymer showing PFA particle with reaction shells and also unidentified spherical assemblages (arrowed) (Jiminez et al. 2004).

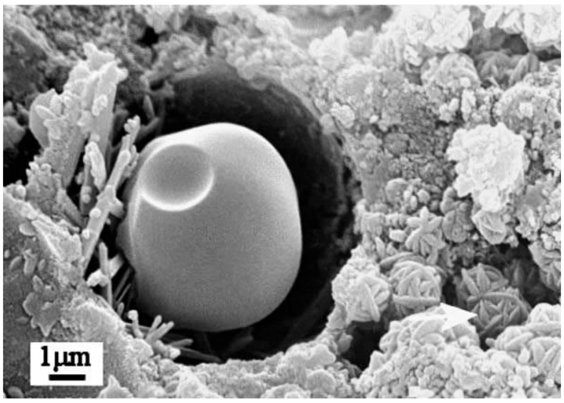


Figure 2.23 SEM micrograph of fracture surface of alkali-activated PFA geopolymer showing considerably eroded PFA particle and also unidentified spherical assemblages (arrowed) (Jiminez et al. 2004).

CHAPTER III

METHODOLOGY

In this research, the research methodology consists of 5 parts. The testing program is summarized in Figure 3.1

3.1 Index and strength properties of soft clay stabilized with FA and PVA Geopolymer

The influential factors include mixing ingredient (fly ash content, water content, liquid alkaline activation content, Na_2SiO_3/NaOH ratio, concentration of NaOH, PVA content, concentration of PVA) and curing time. The sample preparation started with mixing soft clay and FA for 5 min in a mixer to ensure homogeneity of the mixture. The mixer was stopped and the mixture was mixed with the liquid alkaline activator (L) and polyvinyl alcohol (PVA) for an additional 5 minutes. The uniform soft clay–FA–L-PVA mixtures were transferred to cylinder molds of 38 mm in diameter and 76 mm in height for UCS test. The FA-PVA geopolymer stabilized soft clay samples were dismantled, wrapped within vinyl sheet and then cured at room temperature for the required curing duration. The UCS tests were carried out on the samples after 7, 14, 28, 60 and 90 days of curing in accordance with ASTM C69-09. For each curing time and ingredient, at least five samples were tested under the same conditions to assure test result consistency. In most cases, the results under the same testing condition were reproducible with lowmean standard deviation, SD (SD/x b 10%, where x is mean strength value).

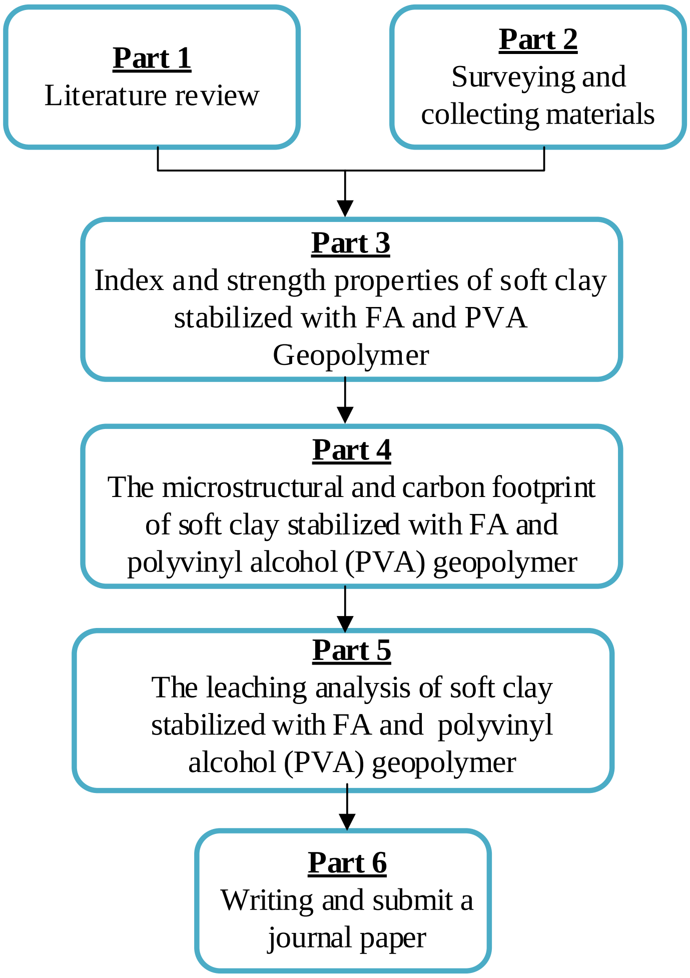


Figure 3.1 testing program

3.2 The microstructure and carbon footprint of soft clay stabilized with FA and polyvinyl alcohol (PVA) geopolymer

The growth of geopolymerization structures on the samples was illustrated using X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM). The XRD traces were used to provide fundamental information on geopolymerization structures.The FA geopolymer and FA–PVA geopolymer stabilized soft clay samples were carefully broken and small fragments were taken from the center for microstructure tests. The

SEM samples were frozen at -195 ^{o}C by immersion in liquid nitrogen for 5 min and evacuated at a pressure of 0.5 Pa at -40 ^{o}C for 5 days. All samples were coated with gold before SEM (JOEL JSM-6400) analysis. The XRD analysis using Cu X-ray tube was done on powdered samples and patterns were obtained by scanning at 0.1^{o} (2h) per min and at steps of 0.05^{o} (2h). The carbon footprint, defined as CO_2-e emitted (kg CO_2-e/ton), of FA geopolymer and FA–PVA geopolymer stabilized soft clay is calculated and compared with that of cement stabilized soft clay at the same UCS. The CO_2 emission calculation considered the mining, processing and manufacturing of raw materials.

3.3 The leaching analysis of the soft clay stabilized with FA and PVA geopolymer

In order to use soft clay stabilized with FA and PVA geopolymer in construction material, the environmental effects of using soft clay stabilized with FA and PVA geopolymer in construction material needs to be ascertained. The leachate analysis of soft clay stabilized with FA and PVA geopolymer was compared with the U.S. Environmental Protection Agency (EPA, 1999).

CHAPTER IV

RESULTS AND DISCUSSIONS

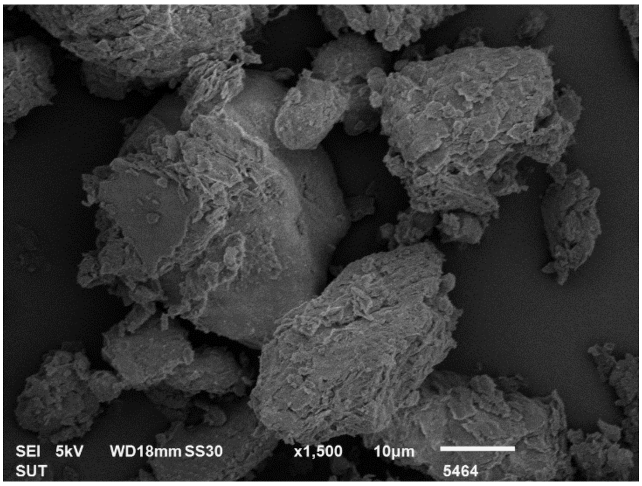
4.1 Laboratory investigation

4.1.1 Materials

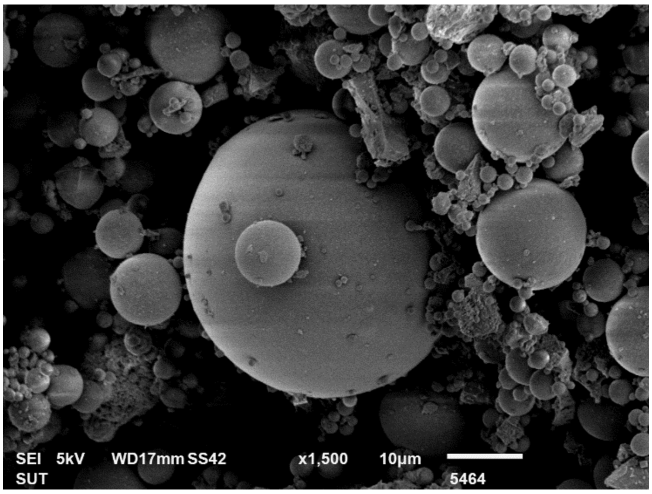
Soft Bangkok clay (SC) was collected at 3 m depth from Nong Chok, Bangkok, Thailand. The particle size distribution of SC obtained from a laser diffraction analysis is shown in Figure 4.1. The average grain size (D_{50}) of SC was 3.5 µm. The liquid limit (LL) and plastic limit (PL) were 70% and 31%, respectively and the specific gravity followed ASTM D854-14 (2014) was 2.67. According to the Unified Soil Classification System (ASTM D2487-10, 2011), the SC was classified as highly plastic clay (CH). Scanning electron microscope (SEM) image of SC was shown in Fig. 2(a) indicating that the SC particles were irregular in shape.

Table 4.1 Chemical composition of SC and HFA.

Chemical Composition (%)	SC	HFA
SiO_2	67.87	43.10
Al_2O_3	19.51	18.95
Fe_2O_3	7.3	15.10
CaO	0.89	16.09
MgO	1.25	N.D.
SO_3	0.74	3.66
Na_2O	0.17	0.73
K_2O	2.22	1.76
LOI	0.05	0.61



(a) SC



(b) FA

Figure 4.1 SEM images of: (a) SC, and (b) FA.

High calcium fly ash (FA) was collected from the Mae Moh power station, northern of Thailand. Table 4.1 shows the chemical properties of FA determined by X-Ray Fluorescence (XRF) test. The sum of main chemical composition (SiO_2, Al_2O_3 and Fe_2O_3) was 77.15% and the CaO content was 16.09%. This FA was therefore classified as class C FA, high calcium FA. Figure 4.2 shows the particle size distribution of FA, which was undertaken using laser diffraction analysis. The particle size of FA was larger than that of SC. The average grain size (D_{50}) of FA was 13.25 µm and the specific gravity was 2.35. Figure 4.1(b) shows SEM image of FA particle, indicating fine and spherical shape. The liquid alkaline activator (L) contained sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH). The Na_2SiO_3 was composed of Na_2O = 9% and SiO_2 = 30% by weight. The NaOH with 98% purity was mixed with water to attain NaOH concentration of 8 molars.

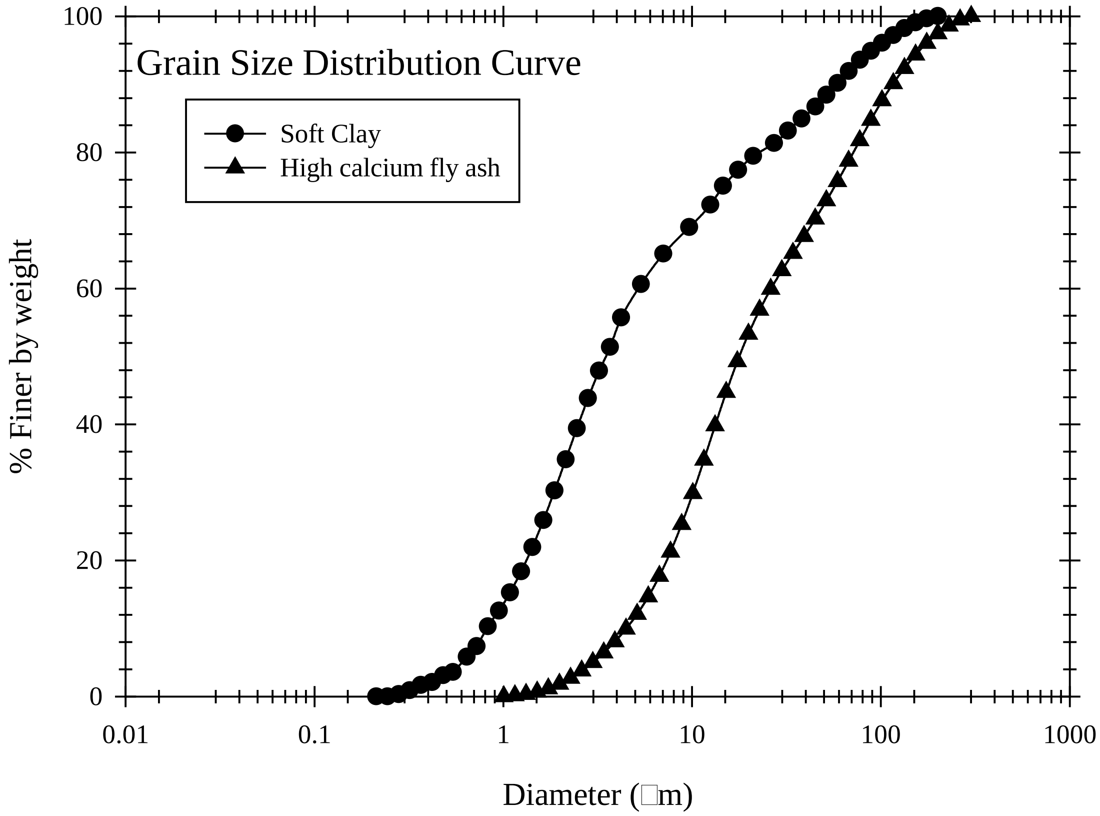


Figure **4.2** Grain size distribution curves of SC and FA.

4.1.2 Methodology

SC was prepared at water content (w) of LL, 1.5LL and 2.0LL to represent the in-situ water content (about 1 to 1.5 LL) and the increased water content caused by the wet mixing process before injection of liquid alkaline activator. The SC was mixed with FA at 10, 20, 30, 40 and 50 % by weight for 5 minutes and then mixed with L at various ingredients for another 5 minutes. The studied Na_2SiO_3/NaOH ratios were 1, 2, 3 and 4 and the L/FA ratios were 0.6, 0.9 and 1.2 because L/FA ratios of 0.6 to 1.2 were previously recommended for researches on soft marine clay-fly ash and calcium carbide residue based geopolymer (Phetchuay et al. 2016). The SC-FA geopolymer mixture was next transferred to a cylindrical mold with 36 mm diameter and 72 mm height for unconfined compression strength (UCS) test. After 24 hours of curing, the UCS samples were wrapped within clean vinyl and cured at a room temperature. The UCS of SC-FA geopolymer samples at 7, 28, 60, and 90 days was measured (ASTM, 2007b).

The PVA-FA geopolymer binder was a combination of PVA, FA and liquid alkaline activator (Na_2SiO_3 and NaOH). The FA content was fixed at 30% by weight of dry soil. The Na_2SiO_3/NaOH ratio was 50:50. The concentration of NaOH was 8 molars. The L/FA ratio was 0.6. The initial water content (*w*) of the SC was 1.0, 1.5 and 2 times LL. The PVA contents were 0, 5, 10, 15, 20, 25 and 30% by weight of dry soil. The PVA concentrations were 2, 4, 6, 8 and 10%. The mixtures were transferred to cylinder molds of 38 mm in diameter and 76 mm in height for UCS test. The FA geopolymer stabilized CIS samples were dismantled, wrapped within vinyl sheet and then cured at room temperature. UCS tests were carried out on the samples after 7 and 28 days of curing in accordance with ASTM C69-09.

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure. The SEM is also capable of performing analyses of selected areas ranging from approximately 1 cm to 5 microns in width and can be imaged in a scanning mode (magnification ranging from 20X to approximately 30,000X). Therefore, The growth of calcium silicate hydrate (CSH) and sodium aluminosilicate hydrate (NASH) gel on the samples was evaluated using scanning electron microscope (SEM). The SC-FA geopolymer samples were carefully broken and small fragments were taken from the center for microstructure test. The SC-FA geopolymer samples were frozen at -195^{o}C by immersion in liquid nitrogen for 5 minutes and evacuated at a pressure of 0.5 Pa at -40^{o}C for 5 days (Suksiripattanapong et al. 2015). All samples were coated with gold before tested by using SEM (JOEL JSM-6400, gold coating) tests (Suksiripattanapong et al. 2015a, 2015b).

4.2 Test results and discussion

Figures 4.3-4.5 show the 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4), FA contents (10, 20, 30, 40 and 50%) and L/FA ratios (0.6, 0.9 and 1.2). The SC-FA geopolymer sample with FA = 10%, w = 2.0LL, Na_2SiO_3/NaOH = 4 and L/FA = 0.6 possesses the lowest 7-day and 28-day UCS. Typically, the 7-day and 28-day UCS values of SC-FA geopolymer increase with increasing FA content, due to higher silica and alumina oxides in amorphous phase for geopolymerization reaction (Phummiphan et al. 2016), expect the sample at LL and L/FA = 0.6. The UCS values of the sample at LL and L/FA = 0.6 increase as FA content increases until FA content of 40% and then decreases with an increase in FA content because the water content and L are insufficient for reacting with high FA content. The maximum UCS is 1525 kPa at 1.0LL, Na_2SiO_3/NaOH = 4, FA = 50% and L/FA = 1.2. The optimum ingredient considering workability and cost of SC-FA geopolymer is found to be at 1.5LL, Na_2SiO_3/NaOH = 1, FA = 30% and L/FA = 0.6. Its 28-day UCS of 682 kPa meets the strength requirement of Departmant of Highways Thailand (>500 kPa) (DOH 2007), Swedish Transport Administration (>200 kPa) (Sundqvist 2015) and Federal Highway Administration (FHWA) (typical range about 400-1500 kPa) (Elias et al. 2006) for soil-cement column.

The effect of water content and L/FA ratio on UCS of SC-FA geopolymer sample is shown in Figures 4.3-4.5 The 7-day and 28-day UCS values decrease as the water content increases due to a diluted NaOH concentration (Suksiripattanapong 2015b) and excessive water content, which result in a large amount void between clay particles (Horpibulsuk 2011a). However, the 7-day UCS increases as L/FA increases because of higher geopolymerization products (Suksiripattanapong et al. 2015a, 2015b) similar to the finding by Phetchuay et al. (Phetchuay et al. 2016) in which the L/FA = 1 was the optimal ratio.

Figures 4.3-4.5 illustrate the effect of Na_2SiO_3/NaOH ratio on 7-day and 28-day UCS of SC-FA geopolymer sample. For L/FA ratio of 0.6 (Fig. 4.3), the SC-FA geopolymer sample with Na_2SiO_3/NaOH = 1 outperforms than other Na_2SiO_3/NaOH ratios. It is because the UCS of SC-FA geopolymer samples is influenced by high NaOH content, which leaches silica and alumina from FA and finally form the geopolymerization products (Phummiphan 2016). However, at L/FA > 0.6 (L/FA = 0.9

and 1.2), the higher Na_2SiO_3/NaOH ratio results in higher early strength due to sufficient liquid alkaline content.

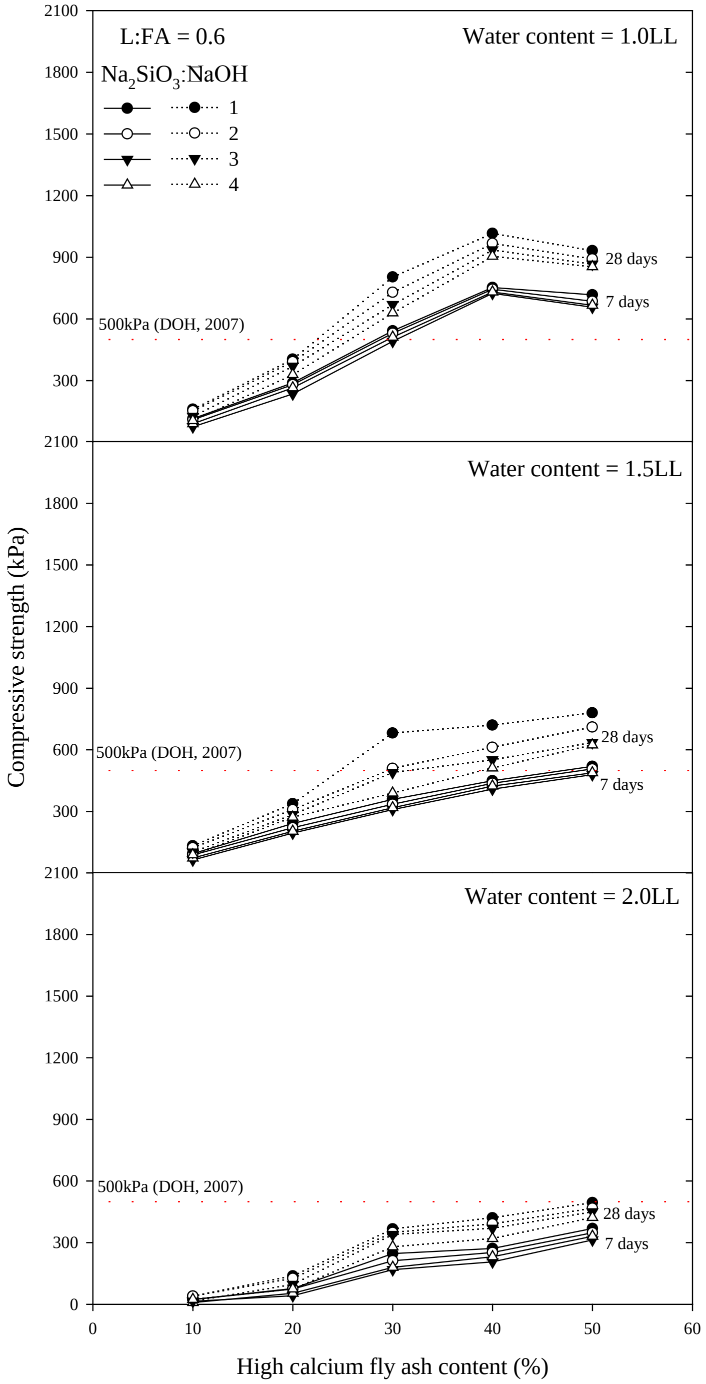


Figure 4.3 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 0.6.

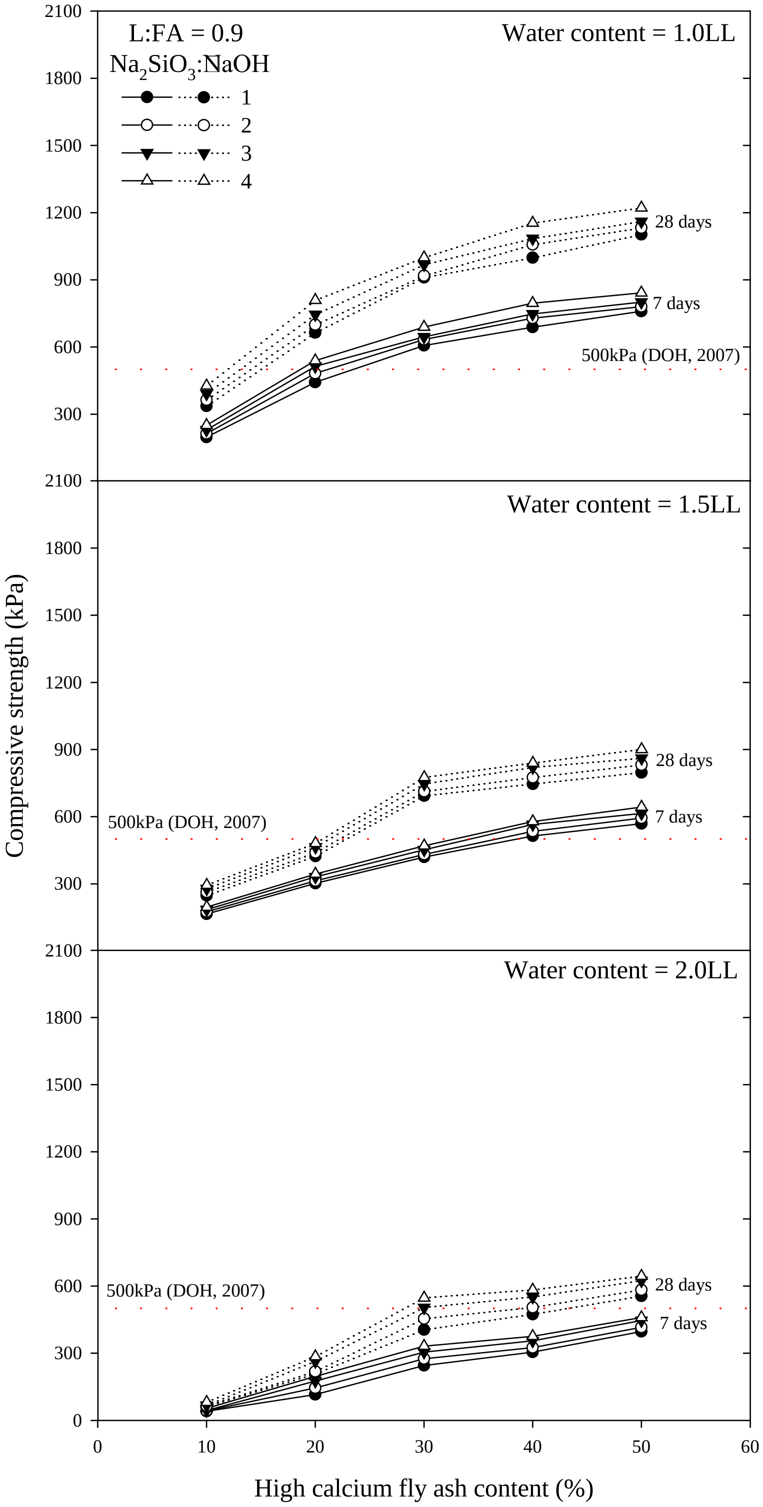


Figure 4.4 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 0.9.

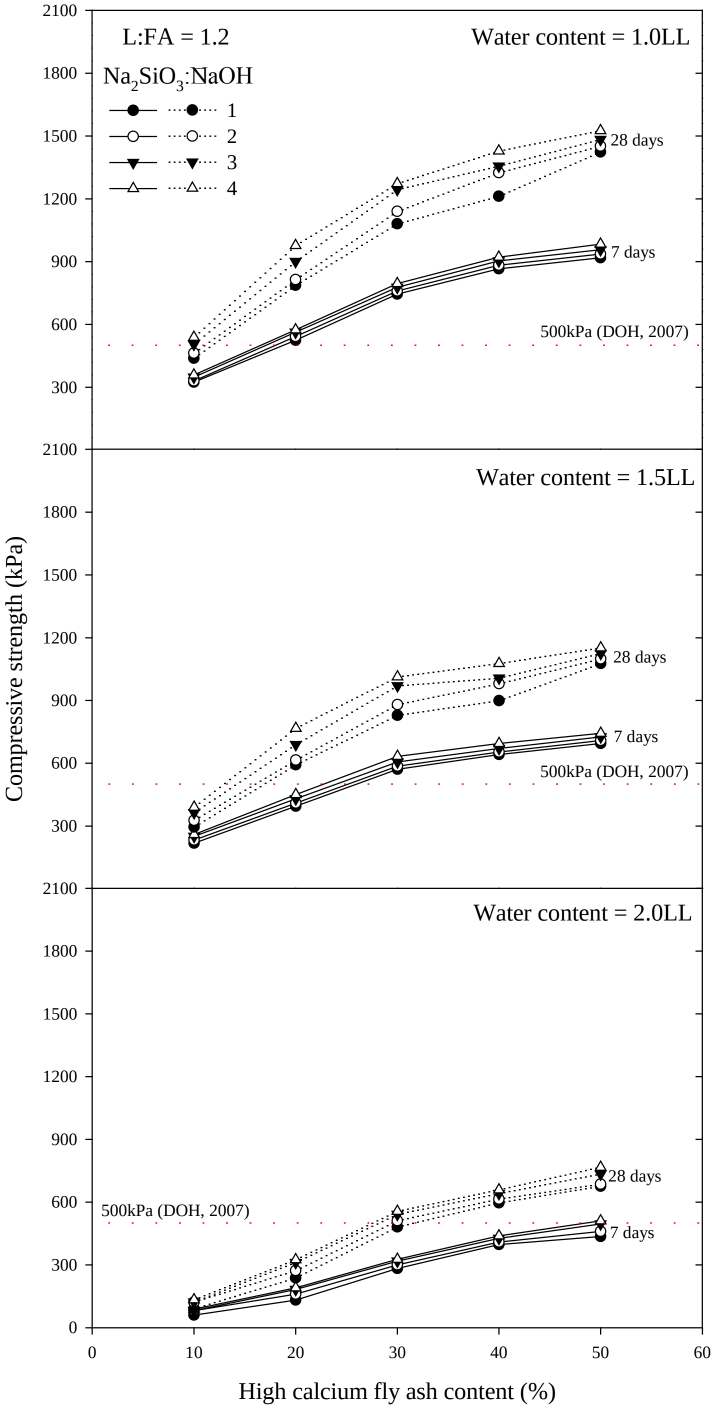


Figure 4.5 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 1.2.

Figure 4.6 shows the strength development over time of the SC-FA geopolymer at FA content of 30%, L/FA = 0.6, w = LL, 1.5LL and 2.0LL and Na_2SiO_3/NaOH = 1, 2, 3 and 4. For Na_2SiO_3/NaOH < 2, the UCS of SC-FA geopolymer increases gradually as curing time increases up to 28 days. After 28 days, the UCS of SC-FA geopolymer increases rapidly with increasing curing time because the silica and alumina in FA is continuously leached by NaOH solution, which results in the CSH formation. Unlike for Na_2SiO_3/NaOH > 2, the rate of UCS development with time is slow because the higher Na_2SiO_3 results in fast setting, which hinders the chemical reaction (Phummiphan 2016). Therefore, the maximum UCS is found to be 1,990 kPa at LL and Na_2SiO_3/NaOH = 1 and 90 days of curing.

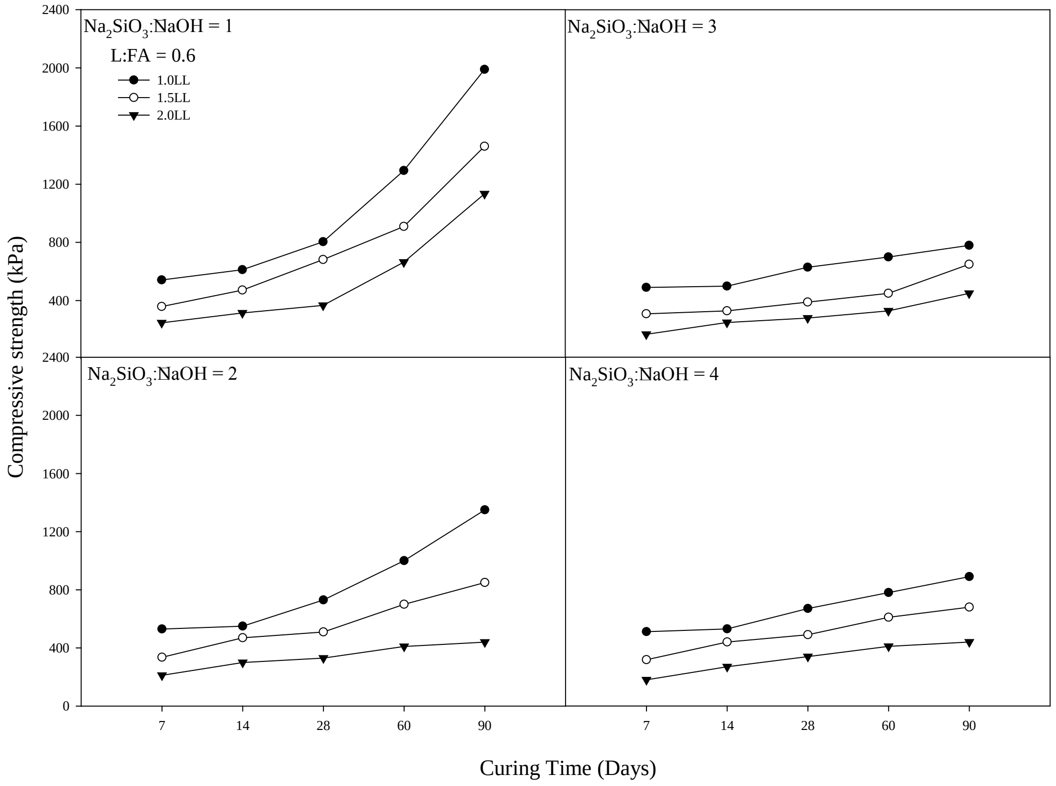
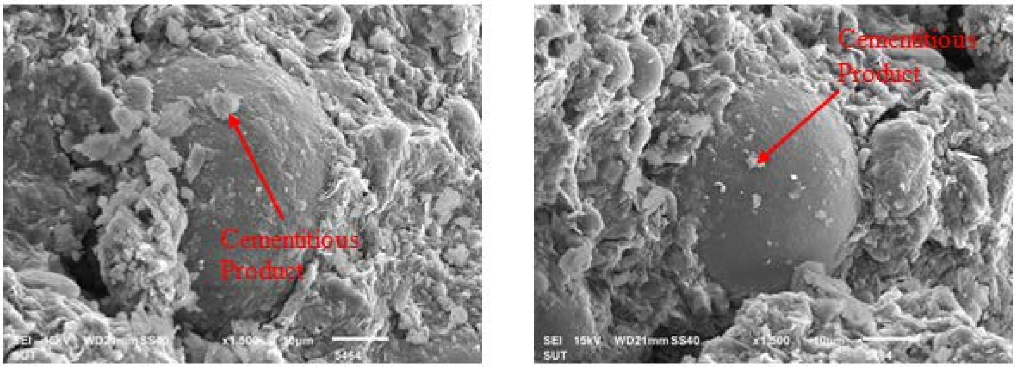
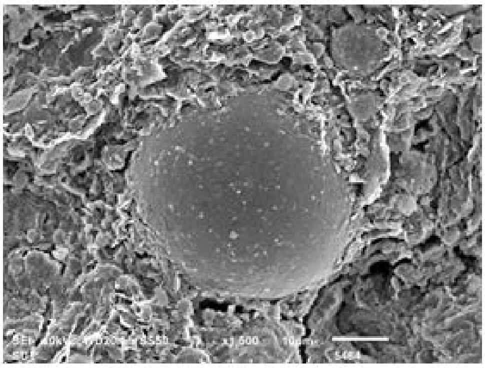


Figure 4.6 Strength development of the SC-FA geopolymer at FA content of 30%, L/FA = 0.6, water content = LL, 1.5LL and 2.0LL and Na_2SiO_3/NaOH = 1, 2, 3 and 4.

The SEM images of 7-day SC-FA geopolymer samples at L/FA = 0.6, Na_2SiO_3/NaOH = 1, FA = 30% and w = LL, 1.5LL, and 2.0LL are presented in Figure 4.7 The geopolymerization products are evidently found on FA surface (Figure 4.7a), indicating that the L is enough to react with FA. The increase in water content has a significant effect on the formation of sodium aluminosilicate gel in the matrix. The geopolymerization products on FA surface decrease as water content increases (Figure 4.7c) due to the diluted NaOH concentration; hence, less dissolved Si and Al from FA.



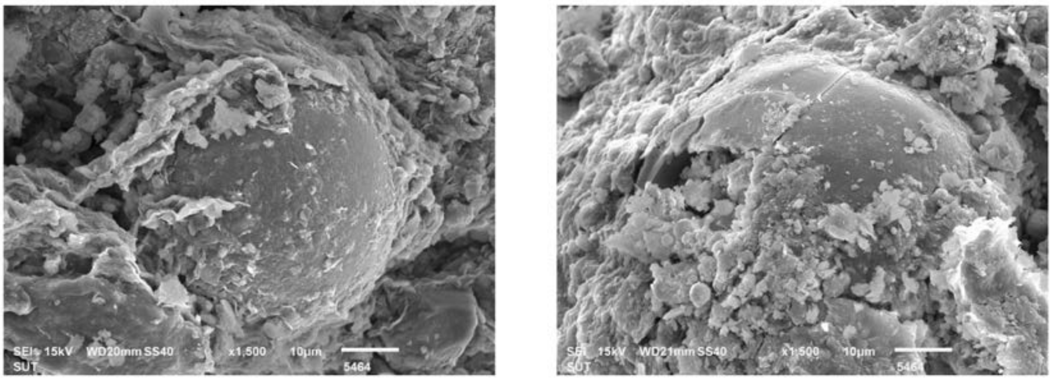
(a) LL (b) 1.5LL



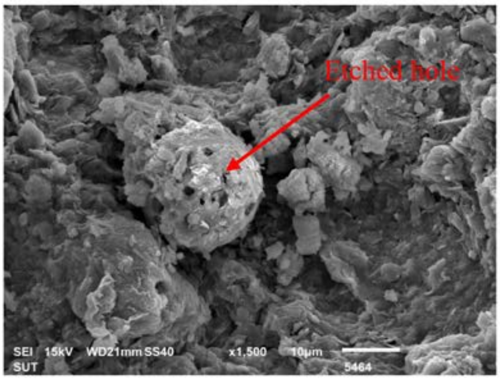
(c) 2.0LL

Figure 4.7 SEM images of 7-day SC-FA geopolymer samples at L/FA = 0.6, Na_2SiO_3/NaOH = 1, FA = 30% and w = (a) LL, (b) 1.5LL, and (c) 2.0LL.

Figure 4.8 presents SEM images of SC-FA geopolymer at w = LL, Na_2SiO_3/NaOH = 2, FA = 30% and L/FA = 0.6, 0.9, and 1.2. The etched hole on the FA surface increase as L/FA increases. Due to the sufficient L content to leach soluble silica and alumina available from high calcium FA for the chemical reaction with Ca, the cementitious products are due to the formation of sodium aluminosilicate hydrate (N-A-S-H) product coexisted with calcium silicate hydrate (C-S-H) and/or calcium aluminosilicate hydrate (C-A-S-H) products (Yaghoubi et al 2018, ASTM D854-1440-2014). Therefore, the 7-day UCS of SC-FA geopolymer increases with the increase of L/FA ratio.



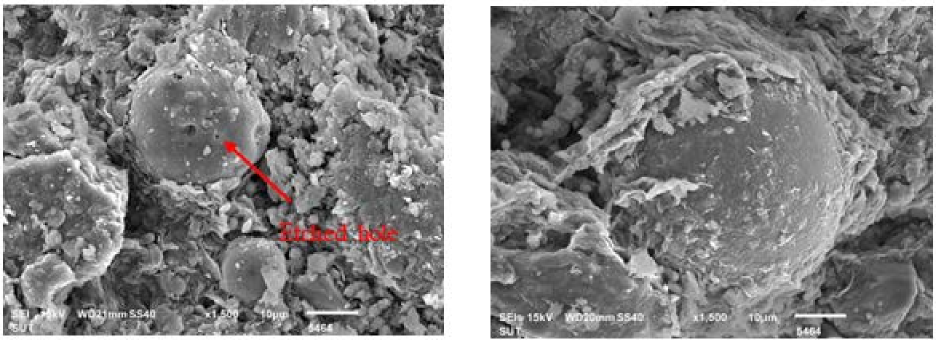
(a) L/FA=0.6 (b) L/FA=0.9



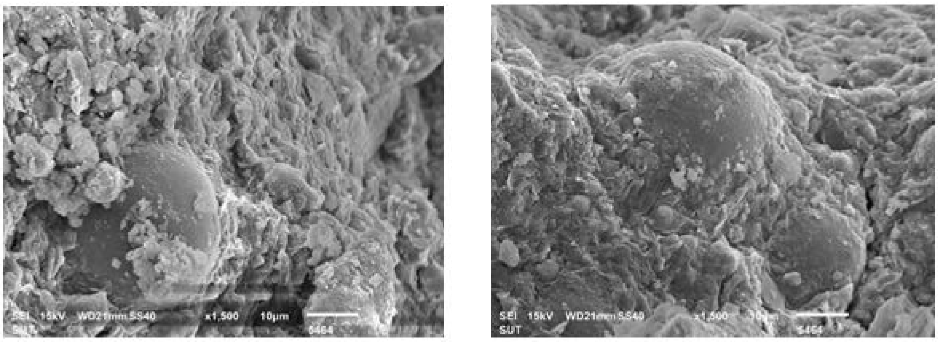
(c) L/FA=1.2

Figure 4.8 SEM images of 7-day SC-FA geopolymer samples at w = LL, Na_2SiO_3/NaOH = 2, FA = 30% and L/FA = (a) 0.6, (b) 0.9, and (c) 1.2.

The effect of Na_2SiO_3/NaOH ratios on geopolymerization products of the 7-day SC-FA geopolymer sample at Na_2SiO_3:NaOH = 1, 2, 3 and 4 is indicated in Figure 4.9 The cementitious products with several etched holes on the FA surface are clearly observed at the excessive amount of NaOH (Na_2SiO_3:NaOH ratio 1) which is associated with the highest strength. Whereas the unreacted FA particles are noticed at Na_2SiO_3:NaOH ratio of 4 because the NaOH in the liquid alkaline activator for Na_2SiO_3:NaOH ratios of 4 is insufficient to dissolve silica and alumina oxide from FA for producing geopolymerization products (Fig.4.9d). Hence, the UCS of SC-FA geopolymer decreases as Na_2SiO_3:NaOH ratio increases.



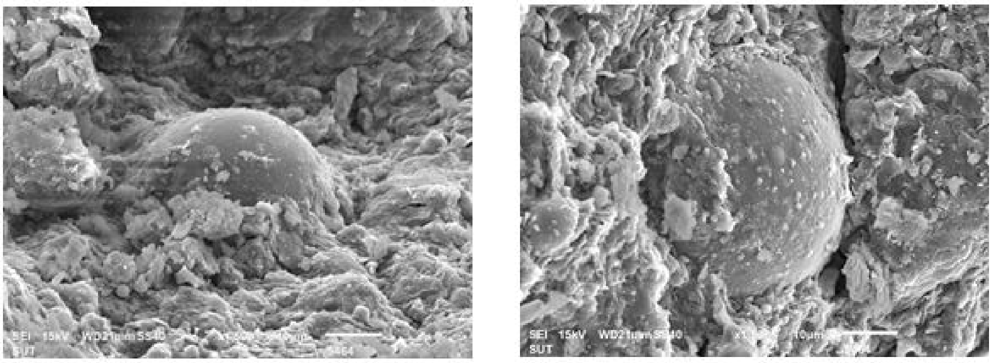
(a) Na_2SiO_3/NaOH=1 (b) Na_2SiO_3/NaOH=2



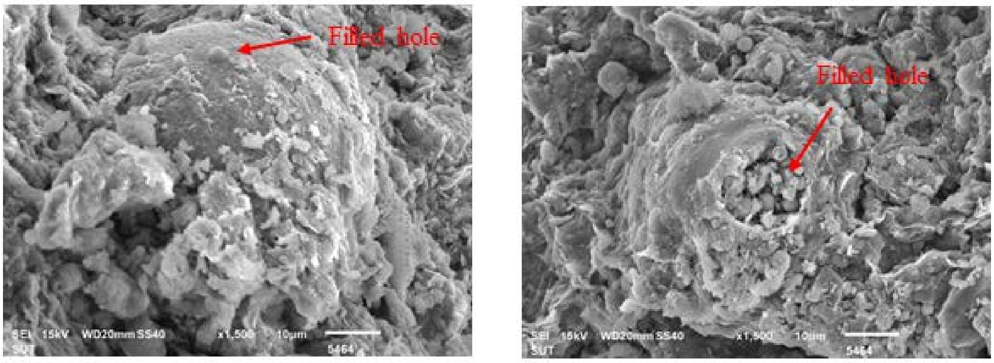
(c) Na_2SiO_3/NaOH=3 (d) Na_2SiO_3/NaOH=4

Figure 4.9 SEM images of 7-day SC-FA geopolymer samples at w = LL, FA = 30% and L/FA = 0.6, (a) Na_2SiO_3/NaOH = 1, (b) Na_2SiO_3/NaOH = 2, (c) Na_2SiO_3/NaOH = 3 and (d) Na_2SiO_3/NaOH = 4.

Figure 4.10 shows the growth of geopolymerization products of SC-FA geopolymer samples at the optimal ingredient of FA = 30%, w = LL, L/FA ratio of 0.6, Na_2SiO_3/NaOH = 1 and curing time = 14, 28, 60 and 90 days. This shows clearly the geopolymerization products (N-A-S-H) on FA particles for all curing time. Figure 4.10d shows the etched holes in FA particles, which are filled with smaller FA particles, indicating the complete degree of chemical reaction (Phummiphan et al. 2016,2017). Consequently, highest UCS is found at this ingredient as shown in Figure 4.6



(a) 14 days (b) 28 days



(c) 60 days (d) 90 days

Figure 4.10 SEM images of 7-day SC-FA geopolymer samples at w = LL, Na_2SiO_3/NaOH = 1, FA = 30%, L/FA = 0.6 and curing time = (a) 14 days, (b) 28 days, (c) 60 days and (d) 90 days.

In practice, FA geopolymer based cementitious agent is discharged, mixed and churned with in-situ soft soil by rotating mixing blades, at the same time the FA geopolymer slurry is jetted from the nozzle located at the tip of the mixing blade. The procedure for installation of soil-FA geopolymer column is suggested by the following steps:

1. Check initial water content of soil: initial water content of soil > 1.5LL would be better mixed. The in-situ water content < 1.5LL is not sufficient for mixing.

2. Drill and drive: if the in-situ water content < 1.5LL, it will be adjusted by water injected during the penetration through soft soil.

3. Withdrawal with jetting and mixing: FA geopolymer slurry will be injected during the withdrawal.

4. Complete the installation of the mixed column until the mixing shaft reaches the desired elevation (typically at the ground surface).

Fig. 4.11-4.13 show the relationships between 7-day compressive strength and PVA content at different PVA concentrations. It can be seen that the highest 7-day compressive strength was found at PVA content of 15%, where the maximum compressive strengths are 600 kPa for PVA concentrations of 2 %. This result indicates that the compressive strength reduces with increasing of PVA concentrations. According to the strength requirements, the 28-day strength of soft clay stabilized with PVA-FA geopolymer at PVA content of 15%, PVA concentration of 2% is acceptable as soil column material. The strength of SC-PVA-FA geopolymer sample increases with increasing PVA content. Beyond this optimum PVA content, the strength of SC-PVA-FA geopolymer sample decreases as PVA content increases.

Figure 4.14 presents SEM images of 7-day SC-PVA-FA geopolymer samples at L/FA = 0.6, Na_2SiO_3/NaOH = 1, FA = 40% and PVA content = (a) 0%, (b) 5%, (c) 15%, and (d) 25%. It can be seen that the PVA films are clearly observed at PVA content of 25% which is associated with the lowest strength. Whereas the dense matrix can be seen in the sample at PVA of 15%, which are the formation of PVA and geopolymerization.

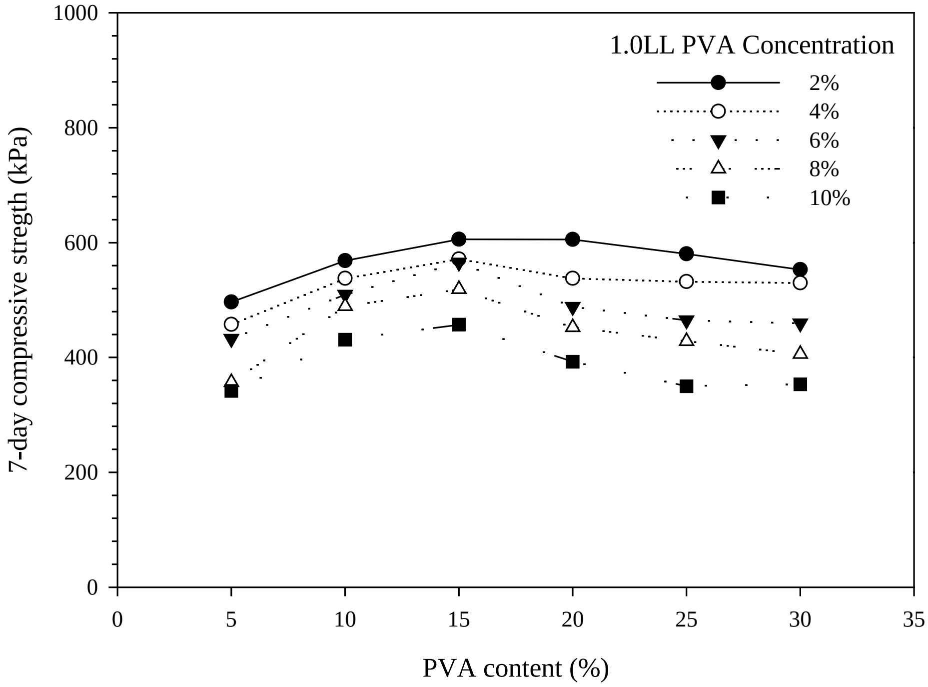


Figure 4.11 7-day compressive strength of SC-PVA-FA geopolymer at various PVA content (0, 5, 10, 15, 20, 25, and 30) and PVA concentrations (2, 4, 6, 8 and 10).

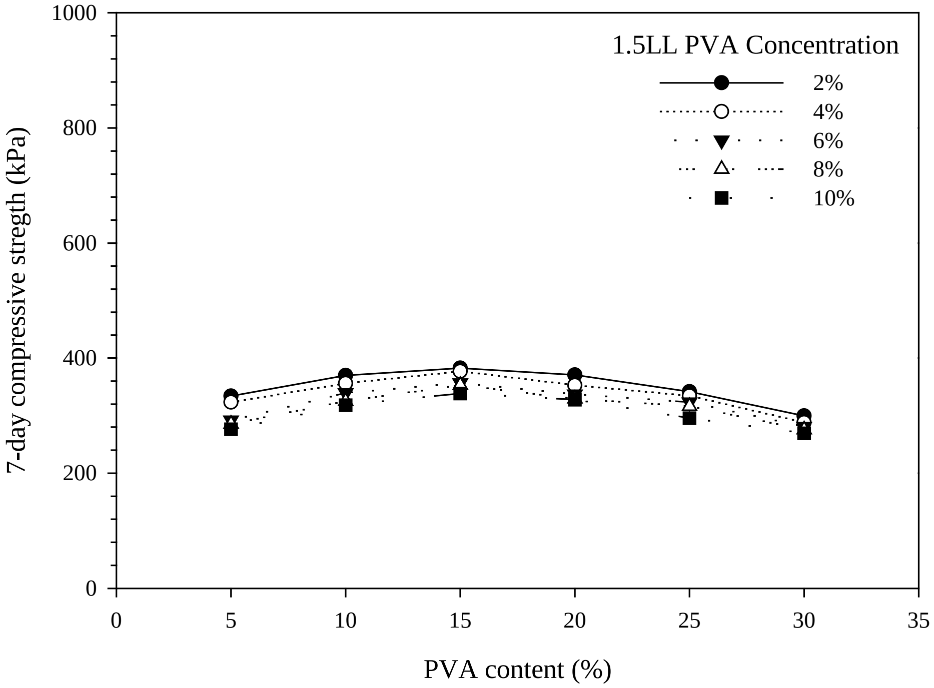


Figure 4.12 7-day compressive strength of SC-PVA-FA geopolymer at various PVA content (0, 5, 10, 15, 20, 25, and 30) and PVA concentrations (2, 4, 6, 8 and 10).

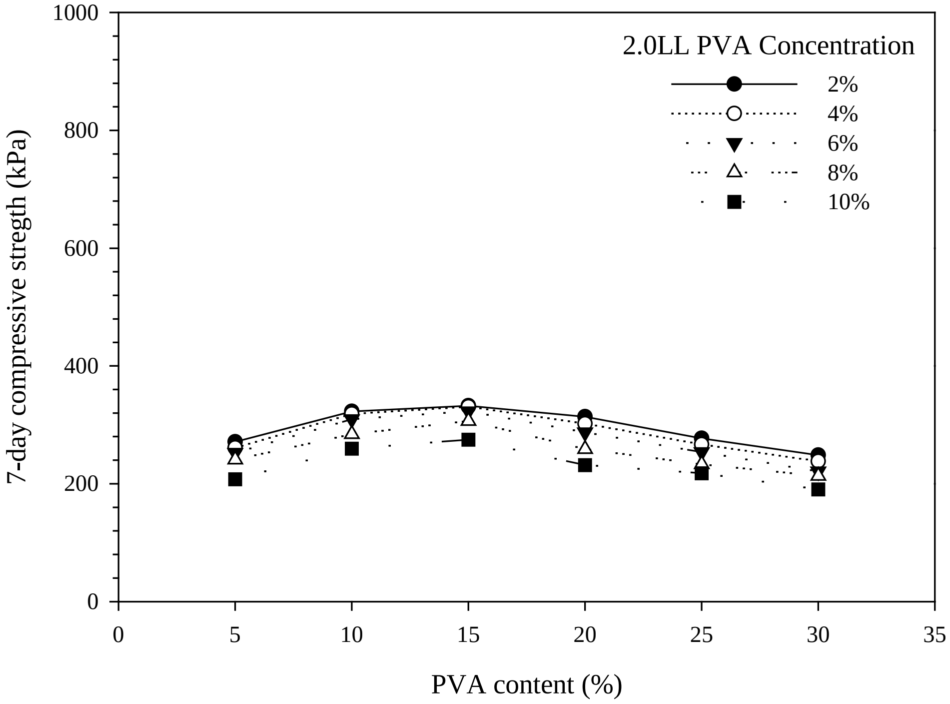
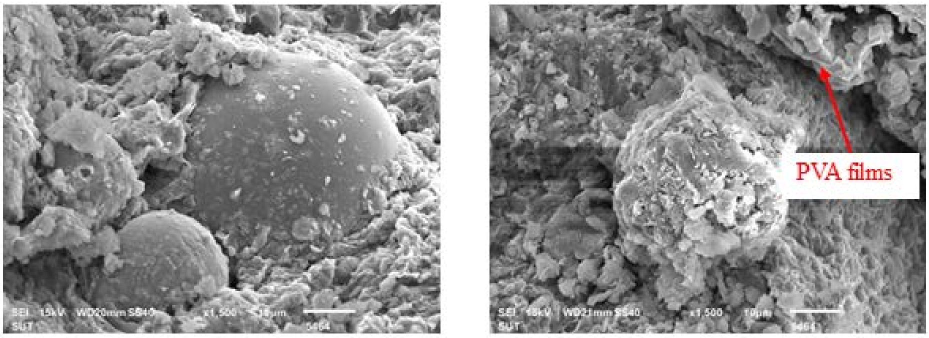
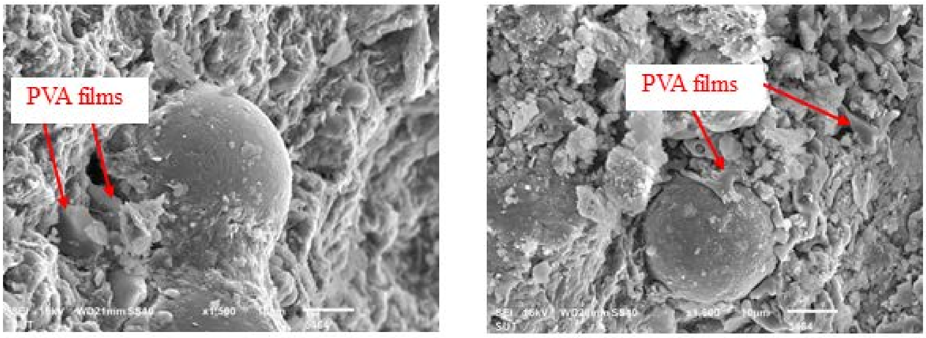


Figure 4.13 7-day compressive strength of SC-PVA-FA geopolymer at various PVA content (0, 5, 10, 15, 20, 25, and 30) and PVA concentrations (2, 4, 6, 8 and 10).



(a) PVA content=0% (b) PVA content=5%



(c) PVA content=15% (d) PVA content=25%

Figure 4.14 SEM images of 7-day SC-PVA-FA geopolymer samples at L/FA = 0.6, Na_2SiO_3/NaOH = 1, FA = 40% and PVA content = (a) 0%, (b) 5%, (c) 15%, and (d) 25%

Toxicity Characteristic Leaching Procedure (TCLP) test is the method prescribed by the US EPA guidelines to determine if the solid waste is hazardous. The TCLP tests were evaluated on SC-HFA-PVA geopolymer. The crushed sample was extracted using an acetic acid solution (pH=4) in a volume with a solid to liquid ratio of 1:20. The mixture was agitated for 18 h in the extraction vessels, which were rotated in an end-over-end manner at 30 rpm. The leachate was then filtered through a 0.45-μm membrane filter to remove suspended solids and divided into two portions. One portion was used for a pH measurement, and the other was used for the determination of the metals present in the leachate by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES).

Table 4.2 shows the leachate analysis of SC-HFA-PVA geopolymer and U.S. EPA requirements. According to benchmark mandated by the U.S. Environmental Protection Agency (EPA) for storm-water sampling, these results indicate that the SC-HFA-PVA geopolymer sample are within allowable limits.

Table 4.2. Leachate analysis of SC-HFA-PVA geopolymer and U.S. EPA requirements

Parameter	SC-HFA-PVA geopolymer	Drinking water standards (EPA, 1999) (mg/L)
Arsenic	0.033	0.05
Cadmium	BDL	0.005
Chromium	0.064	0.1
Lead	0.011	0.015
Mercury	BDL	0.002
Barium	0.548	2.0
Selenium	0.03	0.05

Silver	0.029	0.05

Note: BDL = Below Detection Limit.

The Functional Unit, defined as CO_2-e emitted (kg CO_2-e/ton), is the unit constant, used in this study to undertake the environmental assessment between Original Portland Cement (OPC) and geopolymer binders. The CO_2 emission of cement stabilized soil and geopolymer stabilized soil, which associated with the mining, processing and manufacturing of raw materials and cement production was examined. **Table 4.3** shows the emission factor of material. It is found that the PVA is the highest emission factor for the geopolymer, while fly ash has the lowest emission factor. The energy spending relating to fly ash capture, milling and grinding, drying, and transport causes an emission factor of 0.027 kg CO_2-e/ton. **Table 4.4** shows the total CO_2-e of cement and geopolymer. From the calculation show that the emission CO_2-e of HFA-PVA geopolymer (FA, Na_2SiO_3, NaOH and PVA) is 221 kg CO_2-e/ton compared with the emission CO2-e of cement of 246 kg CO_2-e/ton (Chayakrit et al., 2016). The emission CO_2-e of cement is higher than that of HFA-PVA geopolymer which is about 10.16%.

Table 4.3. The emission factor of material (Turner and Collins, 2013)

Material	Emission factor (kg CO_2-e/ton)
Fly ash (kg)	0.027
Sodium hydroxide (kg)	1.915
Sodium silicate (kg)	1.514
Cement (kg)	0.82

PVA (kg)	2.06

Table 4.4. The comparison between PVA-FA geopolymer samples and cement

Binder	emission CO2-e (kg CO_2-e/ton)
PVA-FA geopolymer	221
cement	246

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Summary and conclusions

This project studied the strength and microstructure development in soft clay stabilized with fly ash and polyvinyl alcohol (PVA) geopolymer. The conclusions can be drawn as follows:

1. The optimum ingredient considering workability and cost of SC-FA geopolymer was found at water content = 1.5LL, $Na_2SiO_3/NaOH = 1$, FA = 30% and L/FA = 0.6 which gives the 28-day UCS of 682 kPa.
2. The 7-day UCS of sample decreases as water content increases. This decreased strength was due to a diluted NaOH concentration and excessive water content resulting in a large amount void between the clay particles.
3. For L/FA ratio of 0.6, the SC-FA geopolymer sample with $Na_2SiO_3/NaOH$ ratio of 1 outperforms than other $Na_2SiO_3/NaOH$ ratios. It is because the UCS of SC-FA geopolymer samples is influenced by high NaOH content, which leaches silica and alumina from FA and finally form the geopolymerization products. However, at L/FA > 0.6 (L/FA = 0.9 and 1.2), the higher $Na_2SiO_3/NaOH$ ratio results in higher early strength due to sufficient liquid alkaline content.
4. For $Na_2SiO_3/NaOH < 2$, the UCS increases as curing time increases gradually until curing time of 28 days. Then the UCS increases rapidly with increasing curing time. It is because the silica and alumina from FA is continuously

leached by NaOH solution to react with Ca oxide and subsequently form CSH. The same is not true for Na_2SiO_3/NaOH > 2, the UCS increases slowly as the curing time increases.

5. For FA = 30%, w = LL and Na_2SiO_3/NaOH=2, the cementitious products on the FA surface increase as L/FA increases. Due to the sufficient L content to leach soluble silica and alumina available from high calcium FA for the chemical reaction with Ca, the cementitious products are due to the formation of sodium aluminosilicate hydrate (N-A-S-H) product coexisted with calcium silicate hydrate (C-S-H) and/or calcium aluminosilicate hydrate (C-A-S-H) products.
6. The highest 7-day compressive strength was found at PVA content of 15%, where the maximum compressive strengths are 600 kPa for PVA concentrations of 2 %.
7. According to benchmark mandated by the U.S. Environmental Protection Agency (EPA) for storm-water sampling, these results indicate that the SC-HFA-PVA geopolymer sample are within allowable limits.
8. The emission CO_2-e of HFA-PVA geopolymer (FA, Na_2SiO_3, NaOH and PVA) is 221 kg CO_2-e/ton compared with the emission CO2-e of cement of 246 kg CO_2-e/ton. The emission CO_2-e of cement is higher than that of HFA-PVA geopolymer which is about 10.16%.

5.2 Recommendations for future work

The strength and microstructure development in soft clay stabilized with fly ash and polyvinyl alcohol (PVA) geopolymer are investigated. The strength and microstructure development in soft clay stabilized with agricultural ash and polyvinyl alcohol (PVA) geopolymer should be further studied.

REFERENCES

Arulrajah, A., Kua, T.A., Horpibulsuk, S., Phetchuay, C., Suksiripattanapong, C., Du, Y.J., 2016. Strength and microstructure evaluation of recycled glass-fly ash geopolymer as low-carbon masonry units. Construction and Building Materials. 114, 400-406.

Arulrajah, A., Mohammadinia, A., Horpibulsuk, S., Wisanukorn, S., 2016. Influence of class F fly ash and curing temperature on strength development of fly ash-recycled concrete aggregate blends. Construction and Building Materials. 127:743–750.

Arulrajah, A., Kua, T.A., Phetchuay, C., Horpibulsuk, S., Maghool, F., Disfani, M.M., 2016. Spent coffee grounds–fly ash geopolymer used as an embankment structural fill material. Journal of Materials in Civil Engineering, 28(5).

Arulrajah, A., Mohammadinia, Phummiphan, I., Horpibulsuk, S., Wisanukorn, S., 2017. Stabilization of Recycled Demolition Aggregates by Geopolymers comprising Calcium Carbide Residue, Fly Ash and Slag precursors. Construction and Building Materials. 114, 864–873.

Arulrajah, A., Kua, T.A., Suksiripattanapong, C., Horpibulsuk, S., J.S. Shen., 2017. Compressive strength and microstructural properties of spent coffee grounds-bagasse ash based geopolymers with slag supplements. Journal of Cleaner Production. 162, 1491-1501

ASTM. 2011. D2487-10: ‘Standard practice for classification of soils for engineering purposes (Unified Soil Classification System), ASTM International. West Conshohocken, PA, USA.

ASTM. 2014. D854-14: 'Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer', ASTM International. West Conshohocken, PA, USA.

Bakria, A.M.M.A., Kamarudin, H., BinHussain, M., Nizar, I.K., Zarina, Y., Rafiza, A.R. 2011. The Effect of Curing Temperature on Physical and Chemical Properties of Geopolymers. Physics Procedia, 22, 286-291.

Buchwald, A., Kaps, Ch., 2002. Peroperty controlling influences on the generation of geopolymeric binders based on clay. Geopolymer (Melbournd Australia).

Chen, J., Shen, S-L., Yin, Z-Y., Ye-Shuang Xu, Y-S., Horpibulsuk, S., 2016. Evaluation of effective depth of PVD improvement in soft clay deposit: a field case study. Marine Georesources and Geotechnology. 34(5), 532-541.

Chindaprasirt, P. Jaturapitakkul, C., Chalee, W., Rattanasak, U., 2009. Comparative study on the characteristics of fly ash and bottom ash geopolymers. Waste Management, 29(2), 539-543.

Chindaprasirt, P.C., T. Hatanaka, S. Cao, T. 2011. High-Strength Geopolymer Using Fine High-Calcium Fly Ash. Journal of Materials in Civil Engineering, 23(3), 264-270.

Chindaprasirt, P., Rattanasak, U., 2017. Characterization of the high-calcium fly ash geopolymer mortar with hot-weather curing systems for sustainable application. Advanced Powder Technology 28: 2317–2324.

Davidovits, J., Buzzi, L., Rocher, R., Gimeno, D., Marini, C., Tocco, S., 1999. Geopolymeric cement based on low cost geologic material, results from the European Research project GEOCIS-TEM. In: Davidovits, et al. (Eds.), Proceedings of the second international conference Geopolymere '99, 83–96.

DOH (Departmant of Highways). (2007). Manual of highway construction, Thailand.

Elias, V., Welsh, J., Warren, J., Lukas, R., Collin, J.G., and Berg, R.R. (2006). Ground Improvement Methods. FHWA NHI-06-020, Federal Highway Administration, Washington, DC.

Hamidiab, S., Marandiab, S.M., 2018. Clay concrete and effect of clay minerals types on stabilized soft clay soils by epoxy resin. Applied Clay Science, 151, 92-101.

Horpibulsuk, S., Miura, N., Nagaraj, T.S., 2003. Assessment of strength development in cement-admixed highwater content clays with Abrams' law as a basis. Geotechnique, 53 (4), 439–444.

Horpibulsuk, S., Miura, N., Koga, H., Nagaraj, T.S., 2004. Analysis of strength development in deep mixing – a field study. Ground Improv. 8 (2), 59–68.

Horpibulsuk, S., Rachan, R., Raksachon, Y., 2009. Role of fly ash on strength and microstructure development in blended cement stabilized silty clay. Soils Found. 49 (1), 85–98.

Horpibulsuk, S., Rachan, R., Suddeepong, A., 2011a. Assessment of strength development in blended cement admixed Bangkok clay. Constr. Build. Mater. 25 (4), 1521–1531.

Horpibulsuk, S., Rachan, R., Suddeepong, A., Chinkulkijniwat, A., 2011b. Strength development in cement admixed Bangkok clay: laboratory and field investigations. Soils Found. 51 (2), 239–251.

Hoy, M., Horpibulsuk, S., Rachan, R., Chinkulkijniwat, A., Arulrajah, A., 2016. Environmental impact on recycled asphalt pavement-fly ash geopolymers a a sustainable pavement base material, Science of The Total Environment 573:19-26

Hoy, M., Horpibulsuk, S., Arulrajah, A., 2016. Strength development of Recycled Asphalt Pavement – fly ash geopolymer as a road construction material, Constr. Build. Mater. 117: 209–219.

Hoy, M., Rachan, R., Horpibulsuk, S., Arulrajah, A., Mirzababaei, M., 2017. Effect of wetting–drying cycles on compressive strength and microstructure of recycled asphalt pavement – Fly ash geopolymer. Constr. Build. Mater. 144:624–634

Huiskes, D.M.A., Keulen, A., Yu, Q.L., Brouwers, H.J.H. 2016. Design and performance evaluation of ultra-lightweight geopolymer concrete. Materials & Design, 89, 516-526.

Jamsawang, P., Poorahong, H., Yoobanpot, N., Songpiriyakij, S., Jongpradist, P., 2017. Improvement of soft clay with cement and bagasse ash waste. Construction and Building Materials. 154, 61-71.

Oh, J.E., Monteiro, P.J.M., Jun, S.S., Choi, S., Clark, S.M. 2010. The evolution of strength and crystalline phases for alkali-activated ground blast furnace slag and fly ash-based geopolymers. Cement and Concrete Research, 40(2), 189-196.

Phetchuay, C., Horpibulsuk, S., Suksiripattanapong, C., Chinkulkijniwat, A., Arulrajah, A., Disfani, M.M. 2014. Calcium carbide residue: Alkaline activator for clay–fly ash geopolymer. Construction and Building Materials, 69, 285-294.

Phetchuay, C., Horpibulsuk, S., Arulrajah, A., Suksiripattanapong, C., Udomchai, A., 2016. Strength development in soft marine clay stabilized by fly ash and calcium carbide residue based geopolymer. Applied Clay Science, 127–128, 134-142.

Phummiphan, I., Horpibulsuk, S., Sukmak, P., Chinkulkijniwat, A., Arulrajah, A., Shen, S.L., 2016. Stabilization of marginal lateritic soil using high calcium fly ash-based geopolymer. Road Materials and Pavement Design, 17(4), 877–891.

Phummiphan, I., Horpibulsuk, S., Phoo-ngernkham, T., Arulrajah, A., Shen, S.L., 2017. Marginal lateritic soil stabilized with calcium carbide residue and fly ash geopolymers as a sustainable pavement base material. Journal of Materials in Civil Engineering, 29(2).

Shen, S.L., Wang, Z.F., Horpibulsuk, S., Kim, Y.H., 2013a. Jet grouting with a newly developed technology: the twin-jet method. Eng. Geol. 152 (1), 87–95.

Sukpunya, A., Jotisankasa, A., 2016. Large simple shear testing of soft Bangkok clay stabilized with soil–cement-columns and its application. Soils and Foundations, 56(4):640–651.

Suksiripattanapong, C., Horpibulsuk, S., Chanprasert, P., Sukmak, P., Arulrajah, A., 2015. Compressive strength development in fly ash geopolymer masonsy units manufactured from water treatment sludge. Constr. Build. Mater. 82, 20–30.

Suksiripattanapong, C., Horpibulsuk, S., Boongrasan, S., Udomchai, A., Chinkulkijniwat, A., Arulrajah, A., 2015. Unit weight, strength and microstructure of a water treatment sludge–fly ash lightweight cellular geopolymer. Construction and Building Materials. 94, 807-816.

Suksiripattanapong, C., Kua, T.A., Arulrajah, A., Maghool, F., Horpibulsuk, S., 2017. Strength and microstructure properties of spent coffee grounds stabilized with rice husk ash and slag geopolymers. Construction and Building Materials. 146, 312-320.

Suksiripattanapong, C., Horpibulsuk, S., Phetchuay, C., Suebsuk, J., Phoo-ngernkham,, T., Arulrajah, A., 2017. Water Treatment Sludge–Calcium Carbide Residue Geopolymers as Nonbearing Masonry Units. Journal of Materials in Civil Engineering, 29(9).

Sundqvist, T., (2015). Stabilization of sulphide soil with lime-cement columns by the river Keräsjoki. Proceedings of the 24th European Young Geotechnical Engineers Conference (EYGEC), Durham, UK

Teerawattanasuk, C., Voottipruex, P., Horpibulsuk, S. 2015. Mix design charts for lightweight cellular cemented Bangkok clay. Applied Clay Science, 104, 318-323.

Wu, H-N., Shen, S-L., Ma, L., Yin, Z-Y., Horpibulsuk, S., 2015. Evaluation of the strength increase of marine clay under staged embankment loading: a case study. Marine Georesources and Geotechnology. 33(6), 532-541.

Yaghoubi, M., Arulrajah, A., Disfani, M.M., Horpibulsuk, S., Bo, M.W. and Darmawan, S., 2018. Effects of industrial by-product based geopolymers on the strength development of a soft soil. Soils and Foundations, doi: 10.1016/j.sandf.2018.03.005

Yoobanpot, N., Jamsawang, P., Horpibulsuk, S., 2017. Strength behavior and microstructural characteristics of soft clay stabilized with cement kiln dust and fly ash residue. Applied Clay Science, 141, 146-156.

Zhang, N., Shen, S.L., Wu, H.N., Chai, J.C., Xu, Y.S., Yin, Z.Y. 2015. Evaluation of effect of basal geotextile reinforcement under embankment loading on soft marine deposits. Geotextiles and Geomembranes, 43(6), 506-514.

Revised manuscript: 8087095

Compressive strength and microstructure properties of soft clay stabilized with high calcium fly ash geopolymer

Cherdsak Suksiripattanapong
Assistant Professor, Rajamangala University of Technology Isan, Nakhon Ratchasima, 30000, Thailand.

Suksun Horpibulsuk
Professor and Chair, School of Civil Engineering, and Director of Center of Excellence in Innovation for Sustainable Infrastructure Development, Suranaree University of Technology, Nakhon Ratchasima, Thailand.

Wisanukhorn Samingthong
Post-graduate researcher, Center of Excellence in Innovation for Sustainable Infrastructure Development, Suranaree University of Technology,
Email: khorn_civil_11@hotmail.com

Corresponding Authors:

Cherdsak Suksiripattanapong
E-mail : Cherdsak.su@rmuti.ac.th
Tel: +66-81-760-7722 and +66-4423-3000 -3211;
Fax: +66-4423-3000 x3211

Suksun Horpibulsuk
E-mail : suksun@g.sut.ac.th
Tel: +66-44-22-4322 and +66-89-767-5759;
Fax: +66-44-22-4607

Compressive strength and microstructure properties of soft clay stabilized with high calcium fly ash geopolymer

ABSTRACT

Soft Bangkok clay (SC) is world recognized as a problematic soil of Thailand. High calcium fly ash (FA) is a by-product obtained from the Mae Moh power plant, Thailand. This paper investigates the possibility of using high calcium fly ash (FA) geopolymer to improve strength of soft Bangkok clay for deep mixing applications to be a construction material, through the liquid alkaline activation composed of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH). The studied influence factors included FA content, water content, liquid alkaline activation (L) content, Na_2SiO_3/NaOH ratio and curing time. Strength development in SC-FA geopolymer specimen at 7, 28, 60, and 90 days of curing was evaluated using the unconfined compressive strength (UCS) test. The 7-day UCS of SC-FA geopolymer increases with increasing FA and L/FA content, due to higher geopolymerization products. However, the excessive water content (more than LL) causes a diluted NaOH concentration (less dissolved Si and Al from FA) and a large pore space. The Na_2SiO_3/NaOH ratios were found to have a small effect on 7-day strength. The maximum UCS was found to be 1,990 kPa at LL and Na_2SiO_3/NaOH = 1 and 90 days of curing. The optimum ingredient considering workability and cost of SC-FA geopolymer was found to be at 1.5LL, Na_2SiO_3/NaOH = 1, FA = 30% and L/FA = 0.6, which gave the 28-day compressive strength of 682 kPa and meets the minimum UCS requirement of Departmant of Highways Thailand (> 500 kPa), Swedish Transport Administration (> 200 kPa) and Federal Highway Administration (FHWA) (typical range about 400-1500 kPa) for soil cement column. This SC-FA geopolymer material can therefore be used as a low-carbon soil-FA geopolymer column.

Keywords: soft Bangkok clay; high calcium fly ash; geopolymer; construction material.

1. INTRODUCTION

Soft Bangkok clay is the world-wide well-known as a soft clay deposit. It possesses a low shear strength, high water content (about liquid limit) and compressibility index. Ground improvement techniques have been employed to stabilize soft soil over the past three decades such as prefabricated vertical drain (PVD) [1,2], grouting [3] and deep mixing (DM) [4-15]. The DM method has been used worldwide including Thailand, China, Sweden, Japan and Singapore. Horpibulsuk et al. [8] have investigated the laboratory and field strengths of soil-cement columns in Bangkok. The strength of soil-cement column was classified into 2 groups: the low strength column with a 28-day strength less than 1500 kPa and high strength column with a 28-day strength higher than 1500 kPa. The required strength of soil-cement column is based on its application (roads, railways, embankment and temporary works), which is governed by equipment types, mixing methods, binder types, site characterization, binder content, etc. Portland cement slurry is typically used to execute the soil-cement columns in Thailand due to its available material at reasonable cost. However, the manufacturing of Portland cement is a resource exhausting and energy intensive process, which releases large amount of carbon dioxide into the atmosphere and causes the greenhouse gases [16,17]. Therefore, the use of new cementing agent with low carbon dioxide release is an interesting issue.

The geopolymer is an environmentally low-carbon cementing agent, alternative to Portland cement. Geopolymerization results from the alkaline activation of silicon (Si) and aluminum (Al) from precursors such as kaolin [18], fly ash [19-23], bottom ash [24], rice husk ash [25], bagasse ash [26] and slag [27]. Several researchers have used geopolymers to stabilize recycled waste materials and marginal soils in civil applications: pavement base/subbases [28-33], embankment fills [34], building masonry units [35-37] and deep mixing improved soft soils

[38]. Recently, Phetchuay et al. [15] investigated strength development of calcium carbide residue (CCR) and fly ash (FA) based geopolymer stabilized marine clay. The optimal ingredient, providing the highest compressive strength, was found at Na_2SiO_3/NaOH ratio of 2.33, L/FA ratio of 1.0 and heat temperature of 40°C. As the low calcium FA was used in their research, the heat curing and CCR additive were needed [22] to accelerate the early strength. With the use of high calcium FA, the moderate to high early strength can be developed at ambient condition [39]. Even though there is available research on the use of high calcium FA to stabilize problematic soil, they are for the compacted material with relative low water content close to optimum water content. As per the authors' knowledge, the research on the high calcium FA stabilized soft clay whose water content is very close to or higher than liquid limit has not been available yet.

This research attempts to investigate the viability of using high calcium FA geopolymer to improve strength of soft Bangkok clay for DM applications. The sodium hydroxide (NaOH) and sodium silicate solution (Na_2SiO_3) were used to create liquid alkaline activator (L). The role of influence factors, which were FA content, water content, L content, Na_2SiO_3/NaOH ratio and curing time, on strength development in soft clay stabilized with FA geopolymer were examined. The microstructural analyses of FA geopolymer stabilized clay via scanning electron microscopy (SEM) were carried out to enlighten the role of influence factors. The outcome of this research will promote the usage of FA geopolymer as an alternative green soil stabilizer alternative to Portland cement for a DM improved soft clay.

2. MATERIALS AND METHODS

Soft Bangkok clay (SC) was collected at 3 m depth from Nong Chok, Bangkok, Thailand. The particle size distribution of SC obtained from a laser diffraction analysis is shown in **Figure**

1. The average grain size (D_{50}) of SC was 3.5 µm. The liquid limit (LL) and plastic limit (PL) were 70% and 31%, respectively and the specific gravity followed ASTM D854-14, 2014 [40] was 2.67. According to the Unified Soil Classification System [41], the SC was classified as highly plastic clay (CH). Scanning electron microscope (SEM) image of SC was shown in Fig. 2(a) indicating that the SC particles were irregular in shape.

High calcium fly ash (FA) was collected from the Mae Moh power station, northern of Thailand. **Table 1** shows the chemical properties of FA determined by X-Ray Fluorescence (XRF) test. The sum of main chemical composition (SiO_2, Al_2O_3 and Fe_2O_3) was 77.15% and the CaO content was 16.09%. This FA was therefore classified as class C FA, high calcium FA. **Figure 1** shows the particle size distribution of FA, which was undertaken using laser diffraction analysis. The particle size of FA was larger than that of SC. The average grain size (D_{50}) of FA was 13.25 µm and the specific gravity was 2.35. **Figure 2(b)** shows SEM image of FA particle, indicating fine and spherical shape. The liquid alkaline activator (L) contained sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH). The Na_2SiO_3 was composed of Na_2O = 9% and SiO_2 = 30% by weight. The NaOH with 98% purity was mixed with water to attain NaOH concentration of 8 molars.

SC was prepared at water content (w) of LL, 1.5LL and 2.0LL to represent the in-situ water content (about 1 to 1.5 LL) and the increased water content caused by the wet mixing process before injection of liquid alkaline activator. The SC was mixed with FA at 10, 20, 30, 40 and 50 % by weight for 5 minutes and then mixed with L at various ingredients for another 5 minutes. The studied Na_2SiO_3/NaOH ratios were 1, 2, 3 and 4 and the L/FA ratios were 0.6, 0.9 and 1.2 because L/FA ratios of 0.6 to 1.2 were previously recommended for soft marine clay stabilized geopolymer [15]. The SC-FA geopolymer mixture was next transferred to a cylindrical mold with 36 mm diameter and 72 mm height for unconfined compression strength (UCS) test. After 24 hours of curing, the UCS samples were wrapped within clean vinyl and cured at a room temperature. The UCS of SC-FA geopolymer samples at 7, 28, 60, and 90 days was measured (ASTM, 2007b).

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure. The SEM is also capable of performing analyses of selected areas ranging from approximately 1 cm to 5 microns in width and can be imaged in a scanning mode (magnification ranging from 20X to approximately 30,000X). Therefore, the growth of calcium silicate hydrate (CSH) and sodium aluminosilicate hydrate (NASH) gel on the samples was evaluated using scanning electron microscope (SEM). The SC-FA geopolymer samples were carefully broken and small fragments were taken from the center for SEM test. The SC-FA geopolymer samples were frozen at -195^{o}C by immersion in liquid nitrogen for 5 minutes and evacuated at a pressure of 0.5 Pa at -40^{o}C for 5 days [17]. All samples were coated with gold before tested by using SEM (JOEL JSM-6400, gold coating) tests [17,35].

3. RESULTS AND DISCUSSIONS

Figures 3-5 show the 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4), FA contents (10, 20, 30, 40 and 50%) and L/FA ratios (0.6, 0.9 and 1.2). The SC-FA geopolymer sample with FA = 10%, w = 2.0LL, Na_2SiO_3/NaOH = 4 and L/FA = 0.6 possesses the lowest 7-day and 28-day UCS. Typically, the 7-day and 28-day UCS values of SC-FA geopolymer increase with increasing FA content, due to higher silica and alumina oxides in amorphous phase for geopolymerization reaction [28], expect the sample at LL and L/FA = 0.6. The UCS values of the sample at LL and L/FA = 0.6 increase as FA content increases until FA content of 40% and then decreases with an increase in FA content because the water content and L are insufficient for reacting with high FA content. The maximum UCS is 1525 kPa at 1.0LL, Na_2SiO_3/NaOH = 4, FA = 50% and L/FA = 1.2. The optimum ingredient considering workability and cost of SC-FA geopolymer is found to be at 1.5LL, Na_2SiO_3/NaOH = 1, FA = 30% and L/FA = 0.6. Its 28-day UCS of 682 kPa meets the strength requirement of Departmant of Highways Thailand (> 500 kPa) [42], Swedish Transport Administration (> 200 kPa) [43] and Federal Highway Administration (FHWA) (typical range about 400-1500 kPa) [44] for soil-cement column.

The effect of water content and L/FA ratio on UCS of SC-FA geopolymer sample is shown in **Figures 3-5**. The 7-day and 28-day UCS values decrease as the water content increases due to a diluted NaOH concentration [35] and excessive water content, which result in a large amount void between clay particles [7]. However, the 7-day UCS increases as L/FA increases because of higher geopolymerization products [17,35] similar to the finding by Phetchuay et al. [15] in which the L/FA = 1 was the optimal ratio.

Figures 3-5 illustrate the effect of Na_2SiO_3/NaOH ratio on 7-day and 28-day UCS of SC-FA geopolymer sample. For L/FA ratio of 0.6 (**Fig. 3**), the SC-FA geopolymer sample with Na_2SiO_3/NaOH = 1 outperforms than other Na_2SiO_3/NaOH ratios. It is because the UCS of SC-FA geopolymer samples is influenced by high NaOH content, which leaches silica and alumina from FA and finally form the geopolymerization products [28]. However, at L/FA > 0.6 (L/FA = 0.9 and 1.2), the higher Na_2SiO_3/NaOH ratio results in higher early strength due to sufficient liquid alkaline content.

Figure 6 shows the strength development over time of the SC-FA geopolymer at FA content of 30%, L/FA = 0.6, w = LL, 1.5LL and 2.0LL and Na_2SiO_3/NaOH = 1, 2, 3 and 4. For Na_2SiO_3/NaOH < 2, the UCS of SC-FA geopolymer increases gradually as curing time increases up to 28 days. After 28 days, the UCS of SC-FA geopolymer increases rapidly with increasing curing time because the silica and alumina in FA is continuously leached by NaOH solution, which results in the CSH formation. Unlike for Na_2SiO_3/NaOH > 2, the rate of UCS development with time is slow because the higher Na_2SiO_3 results in fast setting, which hinders the chemical reaction [28]. Therefore, the maximum UCS is found to be 1,990 kPa at LL and Na_2SiO_3/NaOH = 1 and 90 days of curing.

The SEM images of 7-day SC-FA geopolymer samples at L/FA = 0.6, Na_2SiO_3/NaOH = 1, FA = 30% and w = LL, 1.5LL, and 2.0LL are presented in **Figure 7**. The geopolymerization products are evidently found on FA surface (**Figure 7a**), indicating that the L is enough to react with FA. The increase in water content has a significant effect on the formation of sodium aluminosilicate gel in the matrix. The geopolymerization products on FA surface decrease as water content increases **(Figure 7c)** due to the diluted NaOH concentration; hence, less dissolved Si and Al from FA.

Figure 8 presents SEM images of SC-FA geopolymer at w = LL, Na_2SiO_3/NaOH = 2, FA = 30% and L/FA = 0.6, 0.9, and 1.2. The etched hole on the FA surface increase as L/FA increases. Due to the sufficient L content to leach soluble silica and alumina available from high calcium FA for the chemical reaction with Ca, the cementitious products are due to the formation of sodium aluminosilicate hydrate (N-A-S-H) product coexisted with calcium silicate hydrate (C-S-H) and/or calcium aluminosilicate hydrate (C-A-S-H) products [38,40]. Therefore, the 7-day UCS of SC-FA geopolymer increases with the increase of L/FA ratio.

The effect of Na_2SiO_3/NaOH ratios on geopolymerization products of the 7-day SC-FA geopolymer sample at Na_2SiO_3:NaOH = 1, 2, 3 and 4 is indicated in **Figure 9.** The cementitious products with several etched holes on the FA surface are clearly observed at the excessive amount of NaOH (Na_2SiO_3:NaOH ratio 1) which is associated with the highest strength. Whereas the unreacted FA particles are noticed at Na_2SiO_3:NaOH ratio of 4 because the NaOH in the liquid alkaline activator for Na_2SiO_3:NaOH ratios of 4 is insufficient to dissolve silica and alumina oxide from FA for producing geopolymerization products (**Fig. 9d**). Hence, the UCS of SC-FA geopolymer decreases as Na_2SiO_3:NaOH ratio increases.

Figure 10 shows the growth of geopolymerization products of SC-FA geopolymer samples at the optimal ingredient of FA = 30%, w = LL, L/FA ratio of 0.6, Na_2SiO_3/NaOH = 1 and curing time = 14, 28, 60 and 90 days. This shows clearly the geopolymerization products (N-A-S-H) on FA particles for all curing time. **Figure 10d** shows the etched holes in FA particles, which are filled with smaller FA particles, indicating the complete degree of chemical reaction [28,29]. Consequently, highest UCS is found at this ingredient as shown in Figure 6.

In practice, FA geopolymer based cementitious agent is discharged, mixed and churned with in-situ soft soil by rotating mixing blades, at the same time the FA geopolymer slurry is jetted from the nozzle located at the tip of the mixing blade. The procedure for installation of soil-FA geopolymer column is suggested by the following steps:

1. Check initial water content of soil: initial water content of soil > 1.5LL would be better mixed. The in-situ water content < 1.5LL is not sufficient for mixing.
2. Drill and drive: if the in-situ water content < 1.5LL, it will be adjusted by water injected during the penetration through soft soil.
3. Withdrawal with jetting and mixing: FA geopolymer slurry will be injected during the withdrawal.
4. Complete the installation of the mixed column until the mixing shaft reaches the desired elevation (typically at the ground surface).

4. CONCLUSIONS

This paper studied the compressive strength and microstructure of soft clay stabilized with high calcium fly ash geopolymer. The conclusions are drawn as follows:

1. The optimum ingredient considering workability and cost of SC-FA geopolymer was found at water content = 1.5LL, $Na_2SiO_3/NaOH = 1$, FA = 30% and L/FA = 0.6 which gives the 28-day UCS of 682 kPa.
2. The 7-day UCS of sample decreases as water content increases. This decreased strength was due to a diluted NaOH concentration and excessive water content resulting in a large amount void between the clay particles.
3. For L/FA ratio of 0.6, the SC-FA geopolymer sample with $Na_2SiO_3/NaOH$ ratio of 1 outperforms than other $Na_2SiO_3/NaOH$ ratios. It is because the UCS of SC-FA geopolymer samples is influenced by high NaOH content, which leaches silica and alumina from FA and finally form the geopolymerization products. However, at L/FA > 0.6 (L/FA = 0.9 and 1.2), the higher $Na_2SiO_3/NaOH$ ratio results in higher early strength due to sufficient liquid alkaline content.
4. For $Na_2SiO_3/NaOH < 2$, the UCS increases as curing time increases gradually until curing time of 28 days. Then the UCS increases rapidly with increasing curing time. It is because the silica and alumina from FA is continuously leached by NaOH solution to react with Ca oxide and subsequently form CSH. The same is not true for $Na_2SiO_3/NaOH > 2$, the UCS increases slowly as the curing time increases.
5. For FA = 30%, w = LL and $Na_2SiO_3/NaOH=2$, the cementitious products on the FA surface increase as L/FA increases. Due to the sufficient L content to leach soluble silica and alumina available from high calcium FA for the chemical reaction with Ca, the cementitious products are due to the formation of sodium aluminosilicate hydrate

(N-A-S-H) product coexisted with calcium silicate hydrate (C-S-H) and/or calcium aluminosilicate hydrate (C-A-S-H) products.

ACKNOWLEDGEMENTS

The first author acknowledges the financial support from the Thailand Research Fund and the Office of Higher Education Commission under the Research Grant for New Scholar program Grant No. MRG6080073 and Rajamangala University of Technology Isan. The first and second authors are grateful the financial support from the Thailand Research Fund under the TRF Senior Research Scholar program Grant No. RTA5980005 and Suranaree University of Technology.

REFERENCES

[1] Wu, H-N., Shen, S-L., Ma, L., Yin, Z-Y., Horpibulsuk, S., 2015. Evaluation of the strength increase of marine clay under staged embankment loading: a case study. Marine Georesources and Geotechnology. 33(6), 532-541.

[2] Chen, J., Shen, S-L., Yin, Z-Y., Ye-Shuang Xu, Y-S., Horpibulsuk, S., 2016. Evaluation of effective depth of PVD improvement in soft clay deposit: a field case study. Marine Georesources and Geotechnology. 34(5), 532-541.

[3] Shen, S.L., Wang, Z.F., Horpibulsuk, S., Kim, Y.H., 2013a. Jet grouting with a newly developed technology: the twin-jet method. Eng. Geol. 152 (1), 87–95.

[4] Horpibulsuk, S., Miura, N., Nagaraj, T.S., 2003. Assessment of strength development in cement-admixed highwater content clays with Abrams' law as a basis. Geotechnique, 53 (4), 439–444.

[5] Horpibulsuk, S., Miura, N., Koga, H., Nagaraj, T.S., 2004. Analysis of strength development in deep mixing – a field study. Ground Improv. 8 (2), 59–68.

[6] Horpibulsuk, S., Rachan, R., Raksachon, Y., 2009. Role of fly ash on strength and microstructure development in blended cement stabilized silty clay. Soils Found. 49 (1), 85–98.

[7] Horpibulsuk, S., Rachan, R., Suddeepong, A., 2011a. Assessment of strength development in blended cement admixed Bangkok clay. Constr. Build. Mater. 25 (4), 1521–1531.

[8] Horpibulsuk, S., Rachan, R., Suddeepong, A., Chinkulkijniwat, A., 2011b. Strength development in cement admixed Bangkok clay: laboratory and field investigations. Soils Found. 51 (2), 239–251.

[9] Hamidiab, S., Marandiab, S.M., 2018. Clay concrete and effect of clay minerals types on stabilized soft clay soils by epoxy resin. Applied Clay Science, 151, 92-101.

[10] Yoobanpot, N., Jamsawang, P., Horpibulsuk, S., 2017. Strength behavior and microstructural characteristics of soft clay stabilized with cement kiln dust and fly ash residue. Applied Clay Science, 141, 146-156.

[11] Jamsawang, P., Poorahong, H., Yoobanpot, N., Songpiriyakij, S., Jongpradist, P., 2017. Improvement of soft clay with cement and bagasse ash waste. Construction and Building Materials. 154, 61-71.

[12] Teerawattanasuk, C., Voottipruex, P., Horpibulsuk, S. 2015. Mix design charts for lightweight cellular cemented Bangkok clay. Applied Clay Science, 104, 318-323.

[13] Sukpunya, A., Jotisankasa, A., 2016. Large simple shear testing of soft Bangkok clay stabilized with soil–cement-columns and its application. Soils and Foundations, 56(4):640–651.

[14] Zhang, N., Shen, S.L., Wu, H.N., Chai, J.C., Xu, Y.S., Yin, Z.Y. 2015. Evaluation of effect of basal geotextile reinforcement under embankment loading on soft marine deposits. Geotextiles and Geomembranes, 43(6), 506-514.

[15] Phetchuay, C., Horpibulsuk, S., Arulrajah, A., Suksiripattanapong, C., Udomchai, A., 2016. Strength development in soft marine clay stabilized by fly ash and calcium carbide residue based geopolymer. Applied Clay Science, 127–128, 134-142.

[16] Phetchuay, C., Horpibulsuk, S., Suksiripattanapong, C., Chinkulkijniwat, A., Arulrajah, A., Disfani, M.M. 2014. Calcium carbide residue: Alkaline activator for clay–fly ash geopolymer. Construction and Building Materials, 69, 285-294.

[17] Suksiripattanapong, C., Horpibulsuk, S., Chanprasert, P., Sukmak, P., Arulrajah, A., 2015. Compressive strength development in fly ash geopolymer masonsy units manufactured from water treatment sludge. Constr. Build. Mater. 82, 20–30.

[18] Buchwald, A., Kaps, Ch., 2002. Peroperty controlling influences on the generation of geopolymeric binders based on clay. Geopolymer (Melbournd Australia).

[19] Davidovits, J., Buzzi, L., Rocher, R., Gimeno, D., Marini, C., Tocco, S., 1999. Geopolymeric cement based on low cost geologic material, results from the European Research project GEOCIS-TEM. In: Davidovits, et al. (Eds.), Proceedings of the second international conference Geopolymere '99, 83–96.

[20] Huiskes, D.M.A., Keulen, A., Yu, Q.L., Brouwers, H.J.H. 2016. Design and performance evaluation of ultra-lightweight geopolymer concrete. Materials & Design, 89, 516-526.

[21] Oh, J.E., Monteiro, P.J.M., Jun, S.S., Choi, S., Clark, S.M. 2010. The evolution of strength and crystalline phases for alkali-activated ground blast furnace slag and fly ash-based geopolymers. Cement and Concrete Research, 40(2), 189-196.

[22] Bakria, A.M.M.A., Kamarudin, H., BinHussain, M., Nizar, I.K., Zarina, Y., Rafiza, A.R. 2011. The Effect of Curing Temperature on Physical and Chemical Properties of Geopolymers. Physics Procedia, 22, 286-291.

[23] Chindaprasirt, P.C., T. Hatanaka, S. Cao, T. 2011. High-Strength Geopolymer Using Fine High-Calcium Fly Ash. Journal of Materials in Civil Engineering, 23(3), 264-270.

[24] Chindaprasirt, P. Jaturapitakkul, C., Chalee, W., Rattanasak, U., 2009. Comparative study on the characteristics of fly ash and bottom ash geopolymers. Waste Management, 29(2), 539-543.

[25] Suksiripattanapong, C., Kua, T.A., Arulrajah, A., Maghool, F., Horpibulsuk, S., 2017. Strength and microstructure properties of spent coffee grounds stabilized with rice husk ash and slag geopolymers. Construction and Building Materials. 146, 312-320.

[26] Arulrajah, A., Kua, T.A., Suksiripattanapong, C., Horpibulsuk, S., J.S. Shen., 2017. Compressive strength and microstructural properties of spent coffee grounds-bagasse ash based geopolymers with slag supplements. Journal of Cleaner Production. 162, 1491-1501

[27] Arulrajah, A., Mohammadinia, Phummiphan, I., Horpibulsuk, S., Wisanukorn, S., 2017. Stabilization of Recycled Demolition Aggregates by Geopolymers comprising Calcium Carbide Residue, Fly Ash and Slag precursors. Construction and Building Materials. 114, 864–873.

[28] Phummiphan, I., Horpibulsuk, S., Sukmak, P., Chinkulkijniwat, A., Arulrajah, A., Shen, S.L., 2016. Stabilization of marginal lateritic soil using high calcium fly ash-based geopolymer. Road Materials and Pavement Design, 17(4), 877–891.

[29] Phummiphan, I., Horpibulsuk, S., Phoo-ngernkham, T., Arulrajah, A., Shen, S.L., 2017. Marginal lateritic soil stabilized with calcium carbide residue and fly ash geopolymers as a sustainable pavement base material. Journal of Materials in Civil Engineering, 29(2).

[30] Hoy, M., Horpibulsuk, S., Rachan, R., Chinkulkijniwat, A., Arulrajah, A., 2016. Environmental impact on recycled asphalt pavement-fly ash geopolymers a a sustainable pavement base material, Science of The Total Environment 573:19-26

[31] Hoy, M., Horpibulsuk, S., Arulrajah, A., 2016. Strength development of Recycled Asphalt Pavement – fly ash geopolymer as a road construction material, Constr. Build. Mater. 117: 209–219.

[32] Hoy, M., Rachan, R., Horpibulsuk, S., Arulrajah, A., Mirzababaei, M., 2017. Effect of wetting–drying cycles on compressive strength and microstructure of recycled asphalt pavement – Fly ash geopolymer. Constr. Build. Mater. 144:624–634

[33] Arulrajah, A., Mohammadinia, A., Horpibulsuk, S., Wisanukorn, S., 2016. Influence of class F fly ash and curing temperature on strength development of fly ash-recycled concrete aggregate blends. Construction and Building Materials. 127:743–750.

[34] Arulrajah, A., Kua, T.A., Phetchuay, C., Horpibulsuk, S., Maghool, F., Disfani, M.M., 2016. Spent coffee grounds–fly ash geopolymer used as an embankment structural fill material. Journal of Materials in Civil Engineering, 28(5).

[35] Suksiripattanapong, C., Horpibulsuk, S., Boongrasan, S., Udomchai, A., Chinkulkijniwat, A., Arulrajah, A., 2015. Unit weight, strength and microstructure of a water treatment sludge–fly ash lightweight cellular geopolymer. Construction and Building Materials. 94, 807-816.

[36] Suksiripattanapong, C., Horpibulsuk, S., Phetchuay, C., Suebsuk, J., Phoo-ngernkham,, T., Arulrajah, A., 2017. Water Treatment Sludge–Calcium Carbide Residue Geopolymers as Nonbearing Masonry Units. Journal of Materials in Civil Engineering, 29(9).

[37] Arulrajah, A., Kua, T.A., Horpibulsuk, S., Phetchuay, C., Suksiripattanapong, C., Du, Y.J., 2016. Strength and microstructure evaluation of recycled glass-fly ash geopolymer as low-carbon masonry units. Construction and Building Materials. 114, 400-406.

[38] Yaghoubi, M., Arulrajah, A., Disfani, M.M., Horpibulsuk, S., Bo, M.W. and Darmawan, S., 2018. Effects of industrial by-product based geopolymers on the strength development of a soft soil. Soils and Foundations, doi: 10.1016/j.sandf.2018.03.005

[39] Chindaprasirt, P., Rattanasak, U., 2017. Characterization of the high-calcium fly ash geopolymer mortar with hot-weather curing systems for sustainable application. Advanced Powder Technology 28: 2317–2324.

[40] ASTM. 2014. D854-14: 'Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer', ASTM International. West Conshohocken, PA, USA.

[41] ASTM. 2011. D2487-10: ‘Standard practice for classification of soils for engineering purposes (Unified Soil Classification System), ASTM International. West Conshohocken, PA, USA.

[42] DOH (Departmant of Highways). (2007). Manual of highway construction, Thailand.

[43] Sundqvist, T., (2015). Stabilization of sulphide soil with lime-cement columns by the river Keräsjoki. Proceedings of the 24th European Young Geotechnical Engineers Conference (EYGEC), Durham, UK

[44] Elias, V., Welsh, J., Warren, J., Lukas, R., Collin, J.G., and Berg, R.R. (2006). Ground Improvement Methods. FHWA NHI-06-020, Federal Highway Administration, Washington, DC.

LIST OF TABLES

Table 1. Chemical composition of SC and HFA.

Chemical Composition (%)	SC	HFA
SiO_2	67.87	43.10
Al_2O_3	19.51	18.95
Fe_2O_3	7.3	15.10
CaO	0.89	16.09
MgO	1.25	N.D.
SO_3	0.74	3.66
Na_2O	0.17	0.73
K_2O	2.22	1.76
LOI	0.05	0.61

LIST OF FIGURES

Figure 1. SEM images of: (a) SC, and (b) FA.

Figure 2. Grain size distribution curves of SC and FA.

Figure 3. 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 0.6.

Figure 4. 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 0.9.

Figure 5. 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 1.2.

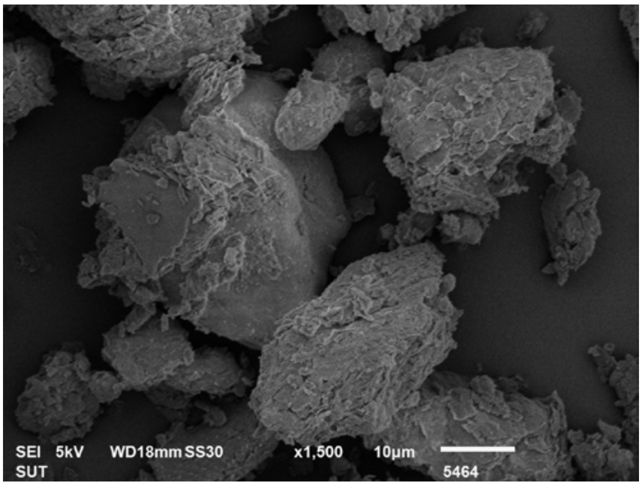
Figure 6. Strength development of the SC-FA geopolymer at FA content of 30%, L/FA = 0.6, water content = LL, 1.5LL and 2.0LL and Na_2SiO_3/NaOH = 1, 2, 3 and 4.

Figure 7. SEM images of 7-day SC-FA geopolymer samples at L/FA = 0.6, Na_2SiO_3/NaOH = 1, FA = 30% and w = (a) LL, (b) 1.5LL, and (c) 2.0LL.

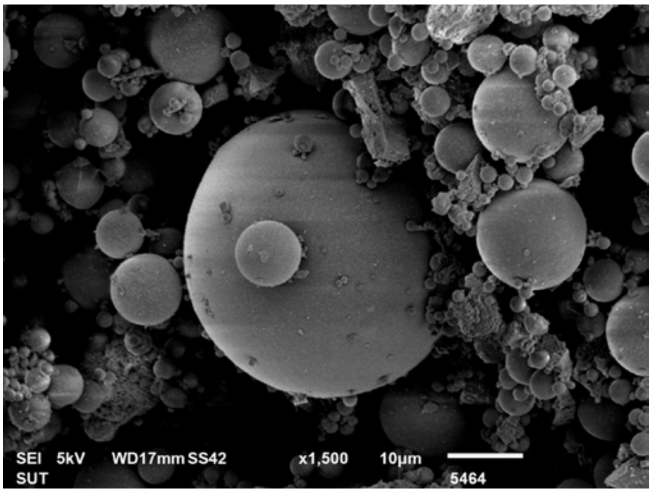
Figure 8. SEM images of 7-day SC-FA geopolymer samples at w = LL, Na_2SiO_3/NaOH = 2, FA = 30% and L/FA = (a) 0.6, (b) 0.9, and (c) 1.2.

Figure 9. SEM images of 7-day SC-FA geopolymer samples at w = LL, FA = 30% and L/FA = 0.6, (a) Na_2SiO_3/NaOH = 1, (b) Na_2SiO_3/NaOH = 2, (c) Na_2SiO_3/NaOH = 3 and (d) Na_2SiO_3/NaOH = 4.

Figure 10. SEM images of 7-day SC-FA geopolymer samples at w = LL, Na_2SiO_3/NaOH = 1, FA = 30%, L/FA = 0.6 and curing time = (a) 14 days, (b) 28 days, (c) 60 days and (d) 90 days.



(a) SC



(b) FA

Figure 1. SEM images of: (a) SC, and (b) FA.

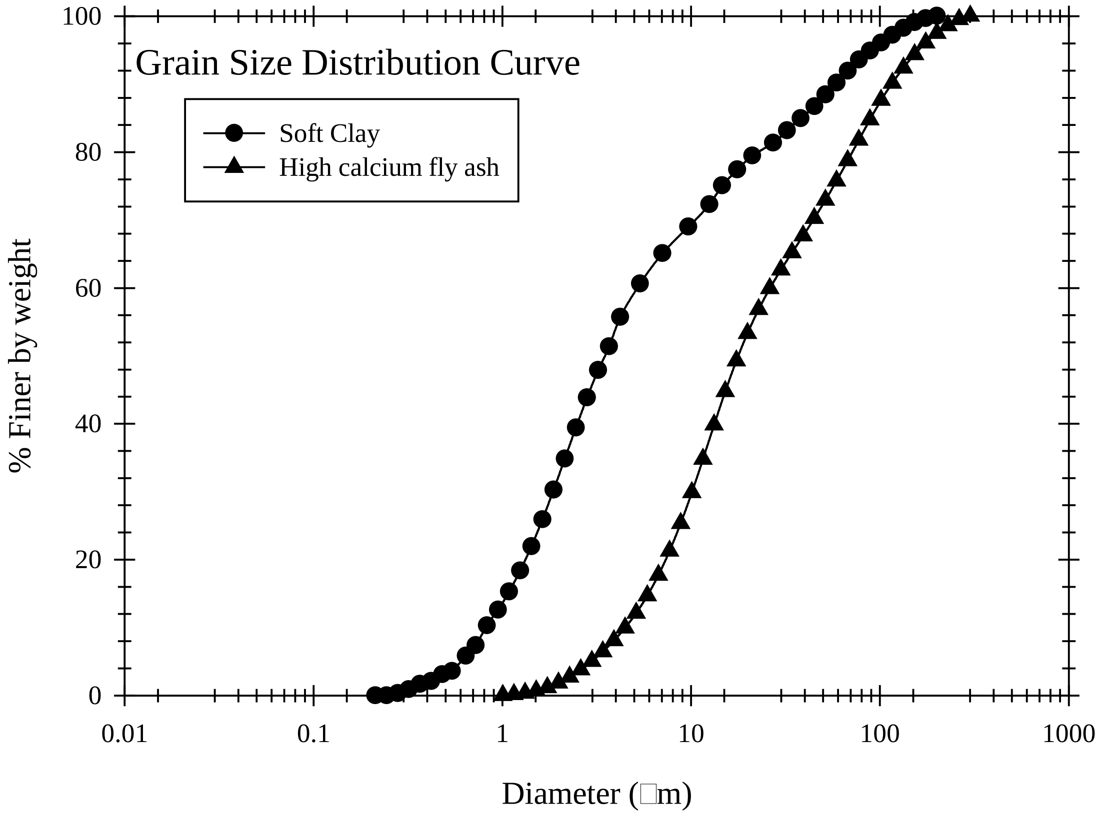


Figure 2. Grain size distribution curves of SC and FA.

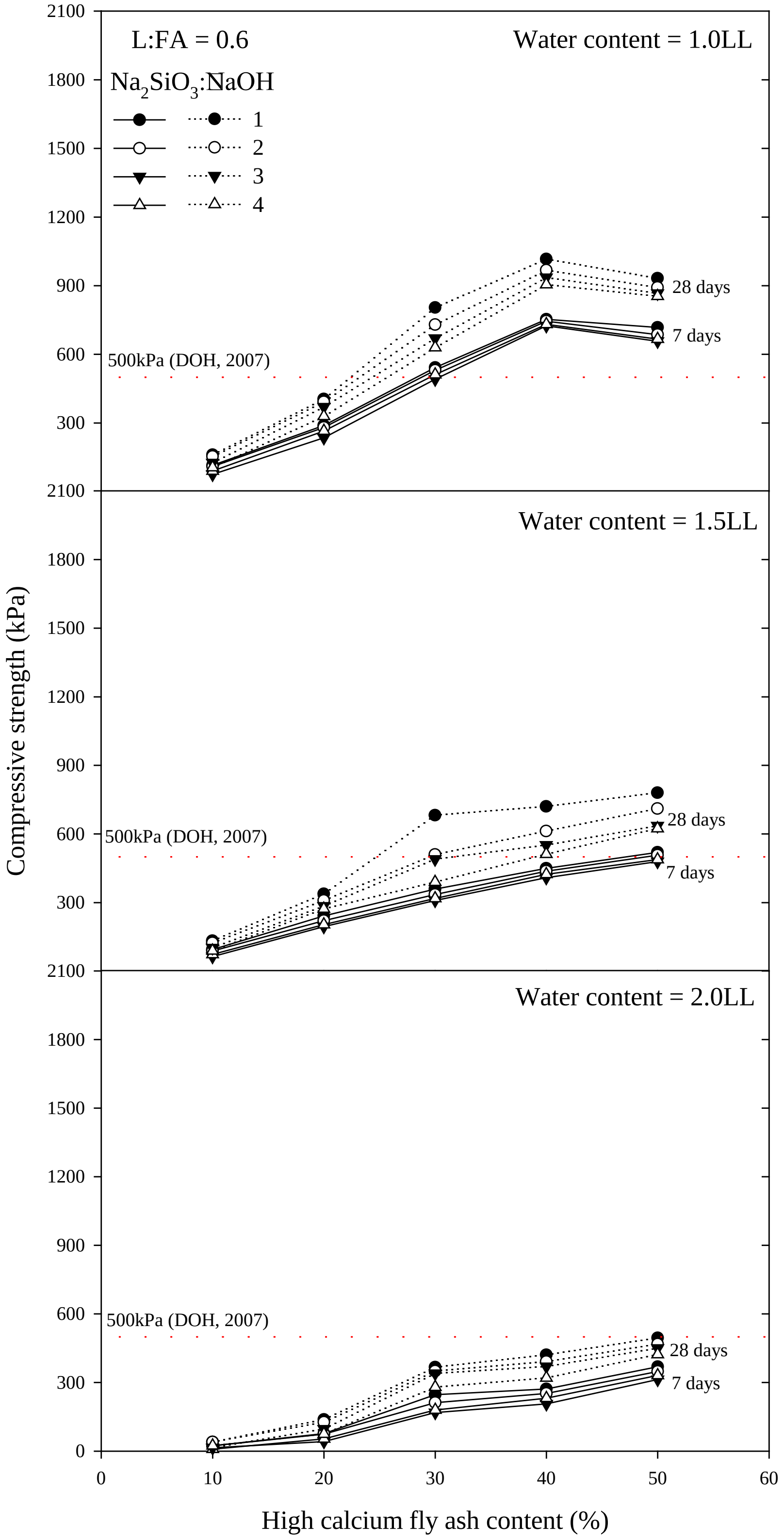


Figure 3. 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 0.6.

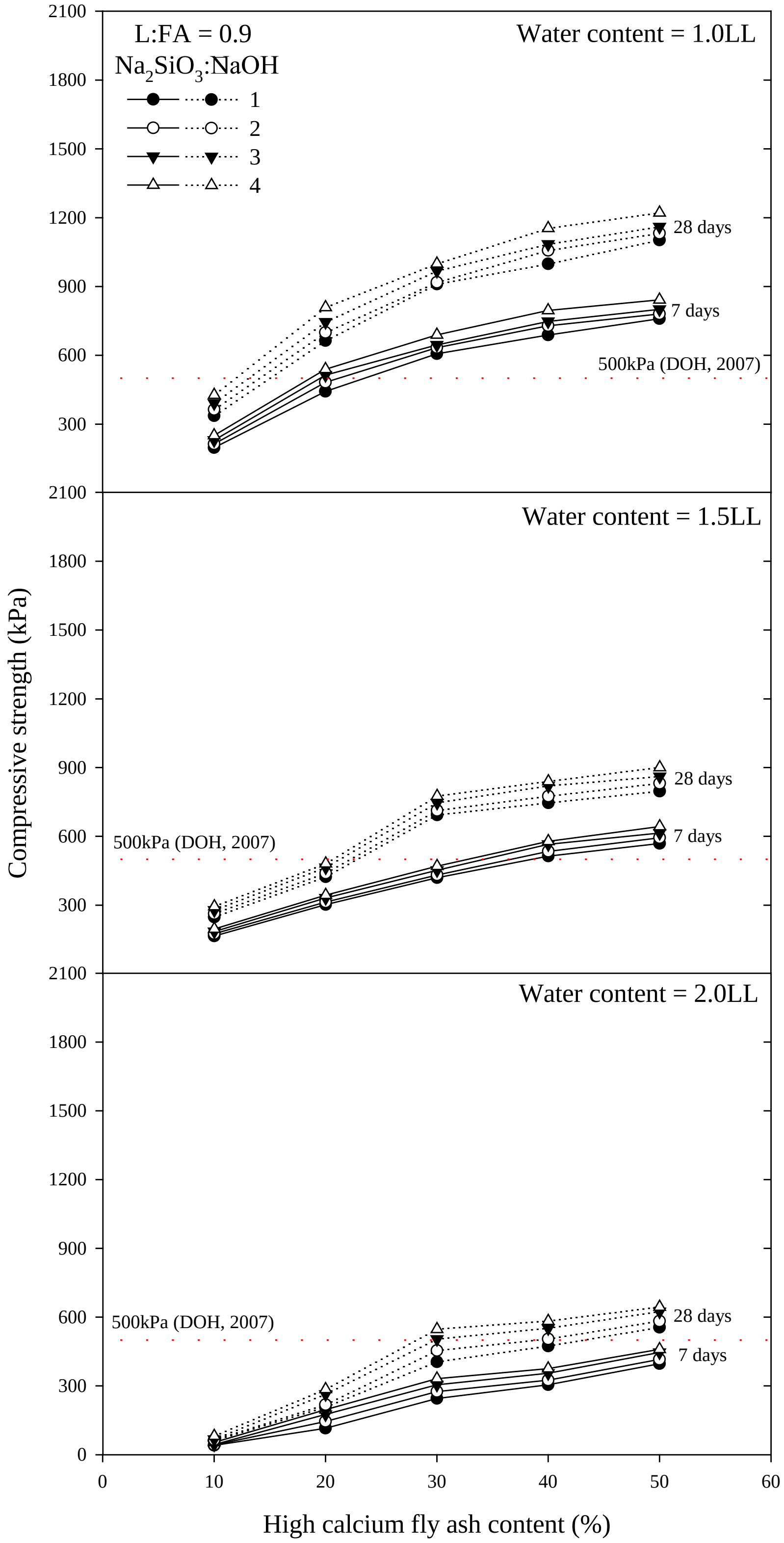


Figure 4. 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 0.9.

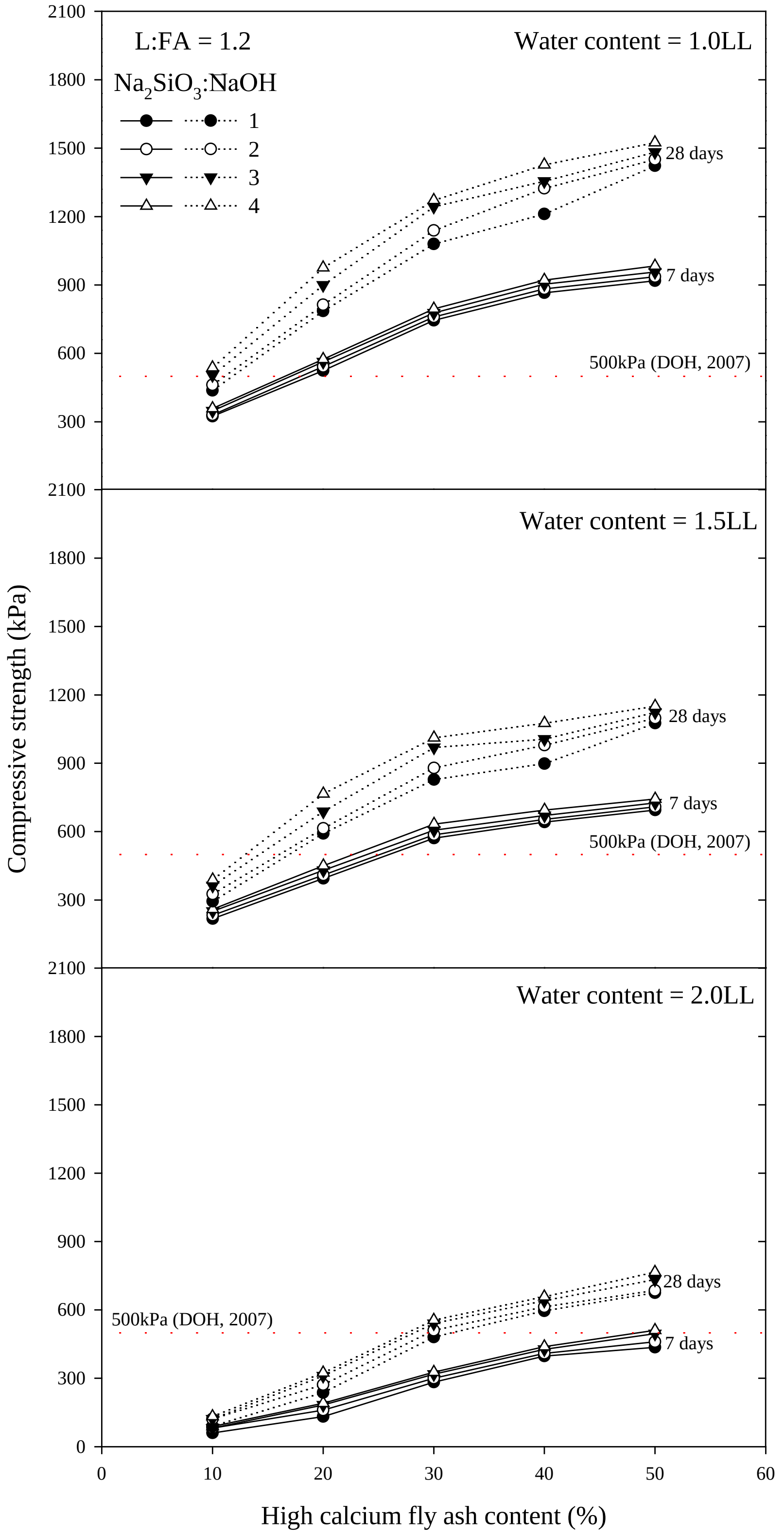


Figure 5. 7-day and 28-day compressive strengths of SC-FA geopolymer at various water contents (LL, 1.5LL and 2.0LL), Na_2SiO_3/NaOH ratios (1, 2, 3 and 4) and FA contents (10, 20, 30, 40 and 50%) and L/FA ratio 1.2.

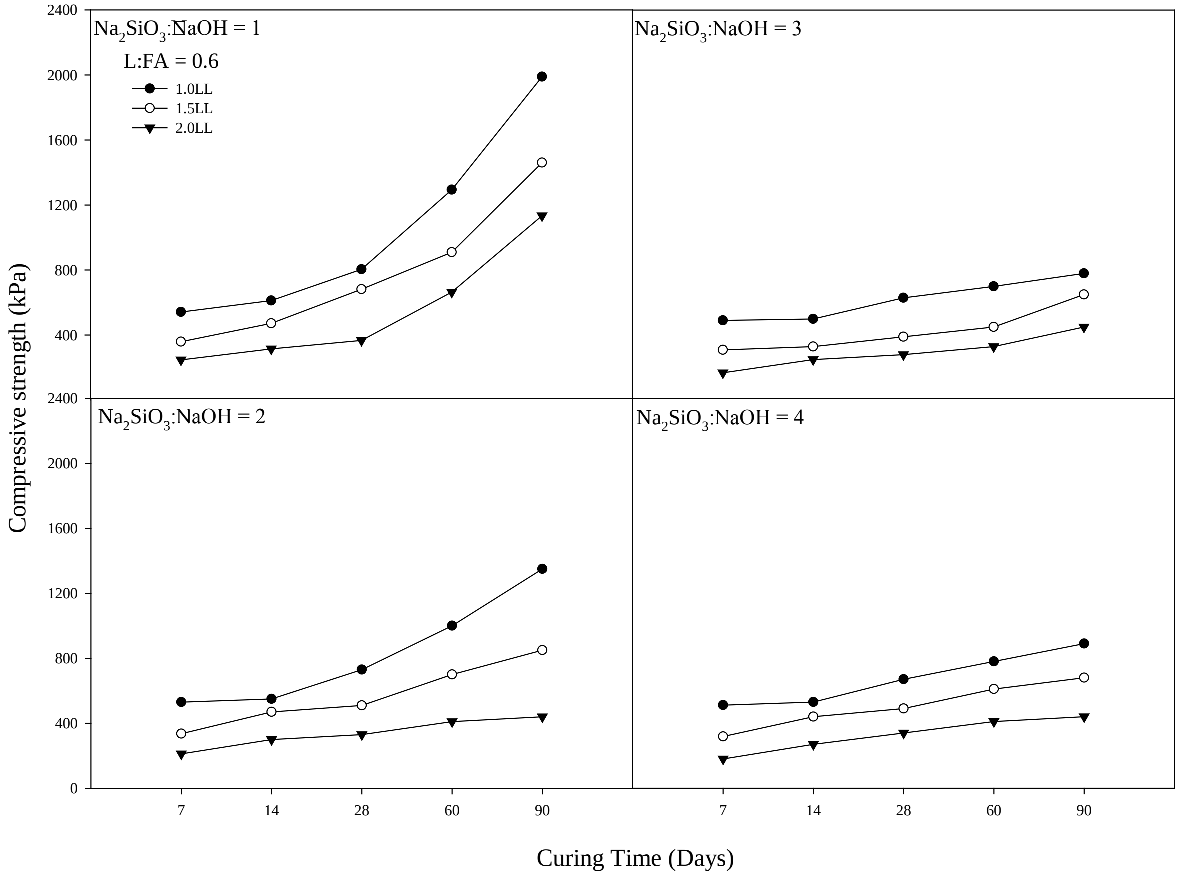
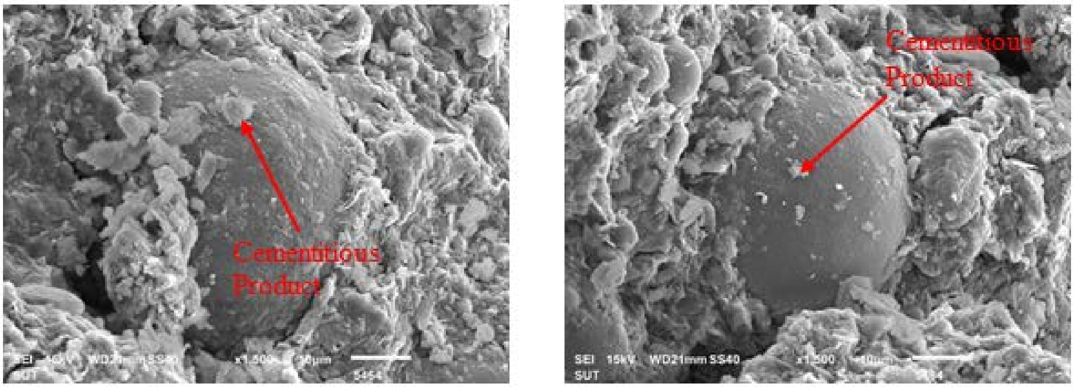
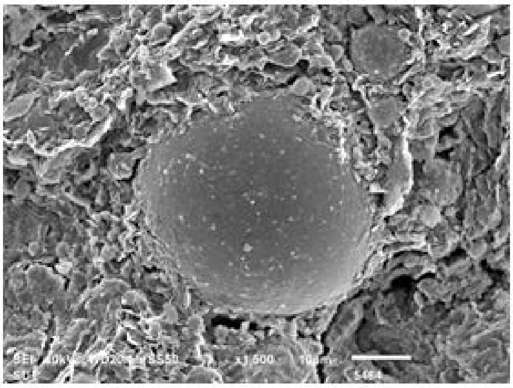


Figure 6. Strength development of the SC-FA geopolymer at FA content of 30%, L/FA = 0.6, water content = LL, 1.5LL and 2.0LL and Na_2SiO_3/NaOH = 1, 2, 3 and 4.



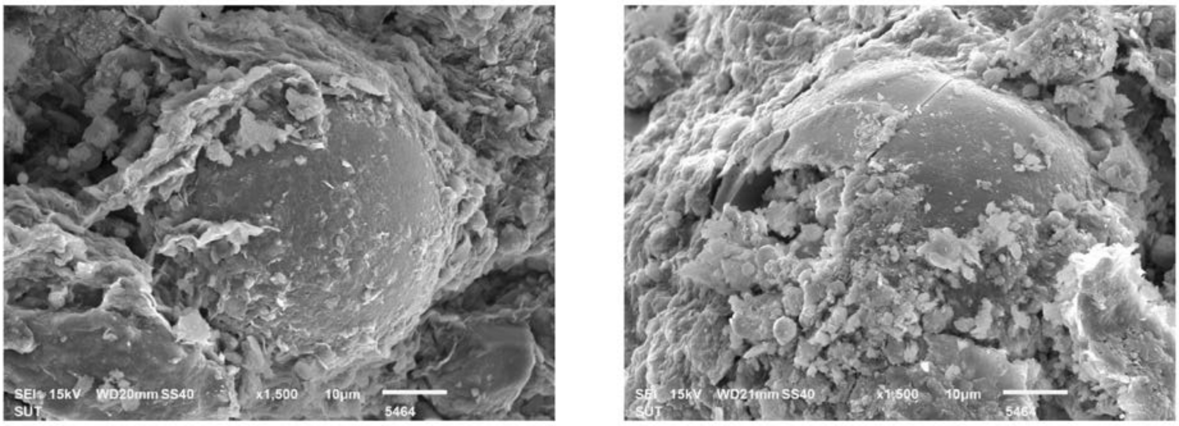
(a) LL

(b) 1.5LL

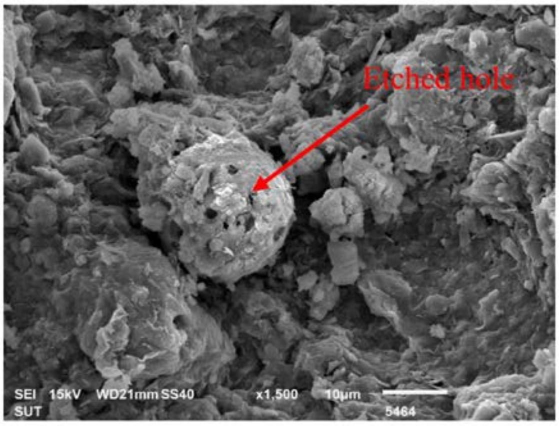


(c) 2.0LL

Figure 7. SEM images of 7-day SC-FA geopolymer samples at L/FA = 0.6, Na_2SiO_3/NaOH = 1, FA = 30% and w = (a) LL, (b) 1.5LL, and (c) 2.0LL.

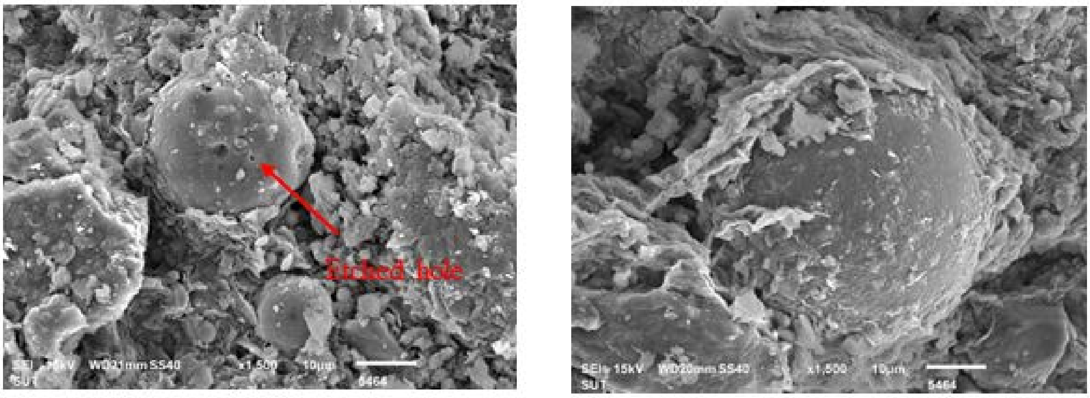


(a) L/FA=0.6 (b) L/FA=0.9



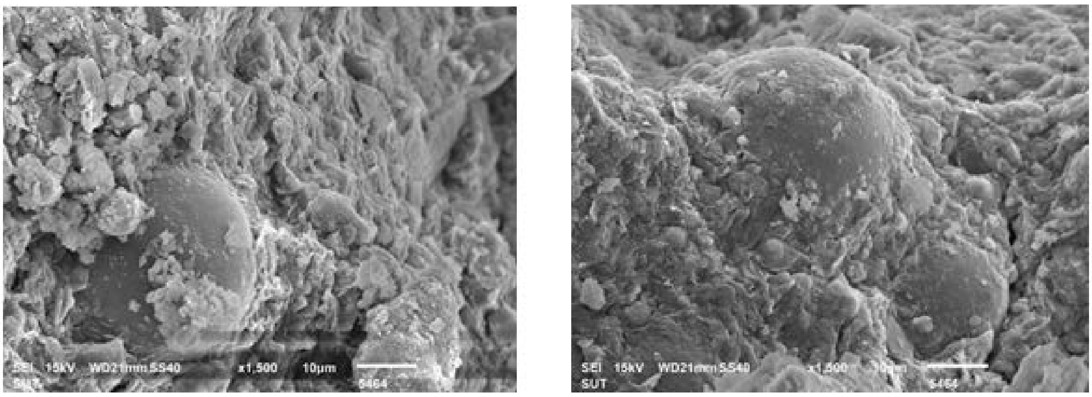
(c) L/FA=1.2

Figure 8. SEM images of 7-day SC-FA geopolymer samples at w = LL, Na_2SiO_3/NaOH = 2, FA = 30% and L/FA = (a) 0.6, (b) 0.9, and (c) 1.2.



(a) Na_2SiO_3/NaOH=1

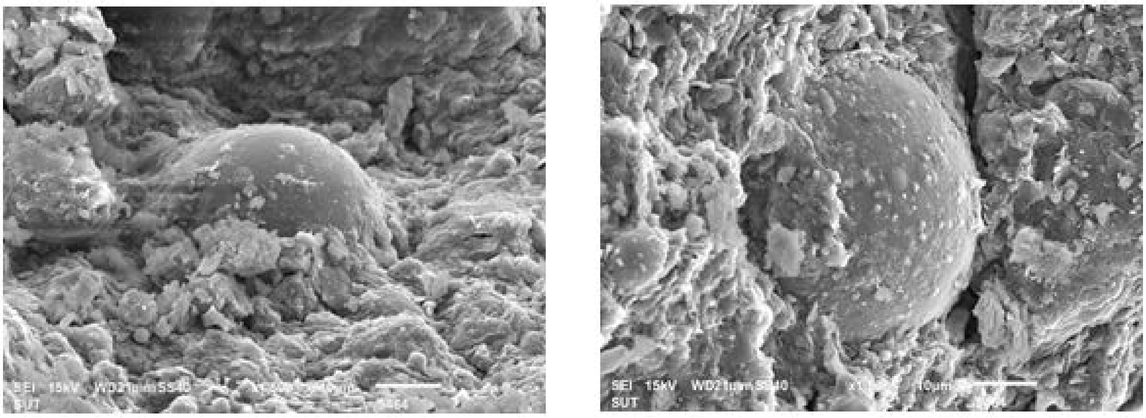
(b) Na_2SiO_3/NaOH=2



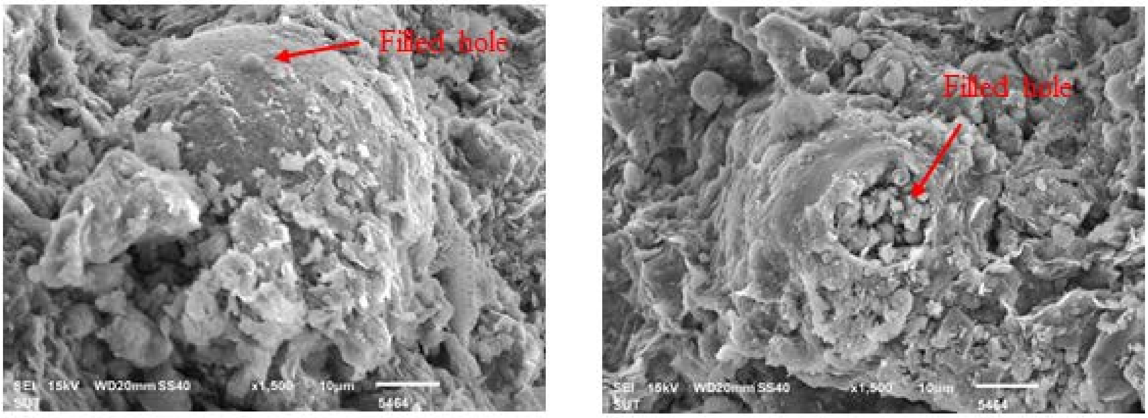
(c) Na_2SiO_3/NaOH=3

(d) Na_2SiO_3/NaOH=4

Figure 9. SEM images of 7-day SC-FA geopolymer samples at w = LL, FA = 30% and L/FA = 0.6, (a) Na_2SiO_3/NaOH = 1, (b) Na_2SiO_3/NaOH = 2, (c) Na_2SiO_3/NaOH = 3 and (d) Na_2SiO_3/NaOH = 4.



(a) 14 days (b) 28 days



(c) 60 days (d) 90 days

Figure 10. SEM images of 7-day SC-FA geopolymer samples at w = LL, $Na_2SiO_3/NaOH = 1$, FA = 30%, L/FA = 0.6 and curing time = (a) 14 days, (b) 28 days, (c) 60 days and (d) 90 days.