

**DEVELOPMENT OF THE CEMENT REPLACEMENT
MATERIAL FROM LOW QUALITY FLY ASH**



**A Thesis Submitted in Partial Fulfillment of the Requirements for the
Degree of Master of Engineering in Material Engineering**

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การพัฒนาวัสดุทดแทนปูนซีเมนต์จากเถ้าลอยที่มีคุณภาพต่ำ



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต

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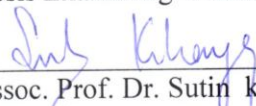
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ปีการศึกษา 2563

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MATERIAL FROM LOW QUALITY FLY ASH**


Suranaree University of Technology has approved this thesis submitted
in partial fulfillment of the requirements for a Master's Degree.

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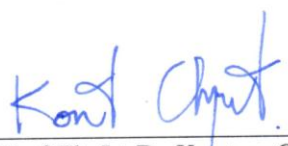
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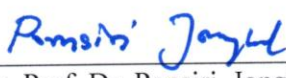
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วรรณเพชร ศิริพงษ์พรรณ : การพัฒนาวัสดุทดแทนปูนซีเมนต์จากเถ้าลอยที่มีคุณภาพต่ำ
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หน้า.

ปูนซีเมนต์ปอร์ตแลนด์ (Portland cement) ถูกใช้เป็นวัสดุก่อสร้างอย่างกว้างขวางทั่วโลก
อย่างไรก็ตาม กระบวนการผลิตปูนซีเมนต์ปอร์ตแลนด์ต้องใช้พลังงานในการผลิตสูง และมีการ
ปลดปล่อยก๊าซคาร์บอนไดออกไซด์ออกมาในจำนวนมาก เป็นที่ทราบกันดีว่าก๊าซคาร์บอนได-
ออกไซด์เป็นสาเหตุหลักที่ทำให้เกิดสภาวะโลกร้อน เมื่อไม่นานมานี้มีนักวิจัยจำนวนหนึ่งพยายาม
หาวัสดุสำหรับใช้ทดแทนปูนซีเมนต์ จีโอพอลิเมอร์ถูกคาดหวังว่าจะสามารถนำมาใช้เป็นวัสดุ
ทดแทนปูนซีเมนต์ได้ เนื่องจากเป็นวัสดุที่เป็นมิตรต่อสิ่งแวดล้อม งานวิจัยนี้มีวัตถุประสงค์เพื่อ
พัฒนาวัสดุจีโอพอลิเมอร์โดยใช้เถ้าลอยคุณภาพต่ำ (Low quality fly ash) ผสมกับดินขาวเผา (Calcined
kaolinite clay) เป็นวัตถุดิบ

ผลการศึกษาพบว่าเพื่อให้ได้ความแข็งแรงที่เพียงพอต่อการใช้งาน จำเป็นต้องใช้วิธีการ
ผสมแบบแยกส่วน การผสมแบบปกติจะทำให้ได้วัสดุจีโอพอลิเมอร์ที่มีความแข็งแรงต่ำ เนื่องจาก
เกิดฟองอากาศขึ้นระหว่างกระบวนการแข็งตัวของจีโอพอลิเมอร์ ฟองอากาศดังกล่าวเกิดจากการทำ
ปฏิกิริยาระหว่างสารละลายอัลคาไลน์ และสิ่งเจือปนในเถ้าลอยคุณภาพต่ำ ดินขาวเผาเป็นวัตถุดิบที่
จำเป็นสำหรับการพัฒนาวัสดุจีโอพอลิเมอร์จากเถ้าลอยคุณภาพต่ำ การใช้เถ้าลอยคุณภาพต่ำเพียง
อย่างเดียวจะทำให้วัสดุจีโอพอลิเมอร์มีความแข็งแรงต่ำ เนื่องจากเถ้าลอยคุณภาพต่ำเกิดปฏิกิริยากับ
สารละลายอัลคาไลน์ได้น้อย กำลังรับแรงอัดของจีโอพอลิเมอร์หลังการบ่มที่อุณหภูมิห้องมีค่าสูง
กว่าการบ่มที่อุณหภูมิ 60 องศาเซลเซียส การบ่มที่อุณหภูมิ 60 องศาเซลเซียสจะส่งผลให้เกิดรอยร้าวที่
ผิวของจีโอพอลิเมอร์ซึ่งเป็นสาเหตุทำให้จีโอพอลิเมอร์มีความแข็งแรงต่ำ ปัจจัยสำคัญที่ส่งผลต่อ
กำลังรับแรงอัดของจีโอพอลิเมอร์ถูกตรวจสอบด้วยการออกแบบการทดลอง (Design of experiment)
พบว่า ปัจจัยที่สำคัญที่สุดคืออัตราส่วนระหว่างเถ้าลอยคุณภาพต่ำต่อดินขาวเผา

สาขาวิชา วิศวกรรมวัสดุ
ปีการศึกษา 2563

ลายมือชื่อนักศึกษา อรรณ บพขร
ลายมือชื่ออาจารย์ที่ปรึกษา

VANHNAPHET SIRIPHONGPHANH : DEVELOPMENT OF THE
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GEOPOLYMER/FLY ASH/CALCINED KAOLINITE CLAY

Ordinary Portland cement (OPC) is widely used as a construction material around the world. However, the production process of PC requires high energy, and, release large amounts of the carbon dioxide (CO_2). As well-known, CO_2 is the main cause of global warming. Recently, several researchers have been investigating for a sustainable cement replacement material. Geopolymer is expected to use as a cement replacement material because it is an environmental friendly material. The aim of this thesis is to develop the geopolymer material by using low quality fly ash (LQFA) blended with calcined kaolinite clay (CK) as the raw materials.

The results show that to obtain sufficient strength, the sequence mixing method (SM) is required. Using normal mixing method (NM), geopolymer has low strength because the bubbles is generated from the reaction between alkaline solution and the metal impurity in LQFA during hardening process. CK is essential for development of geopolymer from LQFA. Using LQFA as a single precursor, geopolymer has low strength because LQFA has low reactivity with an alkali solution. Compressive strength of the cured-geopolymer at room temperature is higher than at the curing at 60°C . Curing at 60°C , the surface cracks are formed which is the causes of low strength. The significant factor that influence on the compressive strength of

the geopolymer is determined by Design of experiment (DOE). The result indicates that the most important factor is the ratio of LQFA to CK.



School of Material Engineering

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Student's Signature Vanhnaphet

Advisor's Signature [Signature]

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Vahnaphet Siriphongphanh

มหาวิทยาลัยเทคโนโลยีสุรนารี

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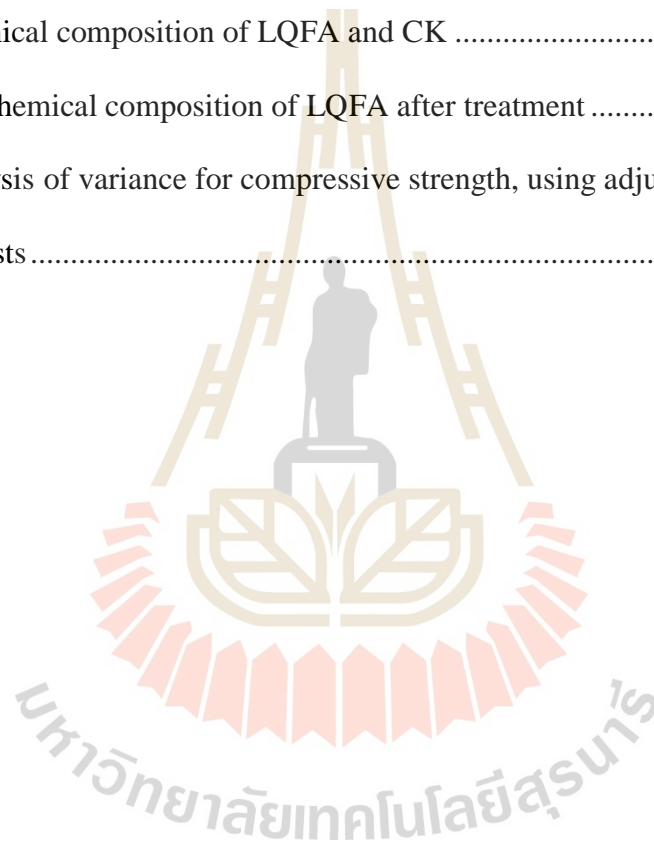
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LIST OF ABBREVIATIONS

OPC	=	Ordinary Portland cement
LQFA	=	Low quality fly ash
HQFA	=	High quality fly ash
CK	=	Calcined kaolinite clay
P/S	=	Powder/solution
LQFA/CK	=	Low quality/calcined kaolinite clay
DOE	=	Design of experiment
M	=	Molar
NM	=	Normal mixing method
SM	=	sequence mixing method
XRD	=	X-ray diffraction
XRF	=	X-ray fluorescence
SEM	=	Scanning electron microscope
UTM	=	Universal Testing Machine
f_m	=	Compressive strength
A	=	Area of load surface
P	=	Total maximum load
W_D	=	Dry Weight
W_s	=	Submerged Weight
W_{ss}	=	Saturated Weigh

CHAPTER I

INTRODUCTION

1.1 General Introduction

Ordinary Portland cement (OPC) is widely used as a construction material around the world. However, the production process of PC requires high energy and release large amounts of carbon dioxide (CO₂) (Kong and Sanjayan, 2010). Carbon dioxide is released into an atmosphere around 1 ton for producing 1 ton of OPC (Imbabi, M.S., et al. 2012). As well-known, CO₂ is the main cause of global warming. Recently, several researchers have been investigated for a sustainable cement replacement material.

Geopolymer is expected to use as a cement replacement material because it is an environmental friendly material. Production process of geopolymer requires low energy consumption and no releasing of CO₂. Geopolymer is synthesized via a reaction between alumino-silicate precursors and alkali solution. There are variety materials that can be used as alumino-silicate precursors such as meta-kaolin, slag, and fly ash.

Fly ash is a waste from the coal-fired power plant. It is widely used as an pozzolanic material for the building construction. However, not all fly ashes are suitable for the construction. The properties of fly ashes depend on the fuel type and the furnace used (Wesche, K. 1991). Numerous standard including ASTM C618, BS 3892, and JIS A6201 are used to classify types of fly ash. High quality fly ash (HQFA) is described as a high reactive ash which is a highly desirable commodity in

the construction industry. HQFA is obtained from a high quality coal with a high temperature combustion system. Morphology of HQFA is mainly vitreous spherical particles. According to ASTM C618-15, HQFA is classified in either Class F or Class C depending on the SiO_2 , Al_2O_3 and Fe_2O_3 contents of the pozzolanic compounds (Gomaa, E., et al. 2017). Class F fly ash contains at least 70% of pozzolanic compounds, while Class C fly ash contains at least 50% of the pozzolanic compounds. Another major difference between both types is the calcium content which the fly ash Class C contains higher content than Class F (Gomaa, E., et al. 2017).

However, the most of fly ash is classified into the low quality fly ash (LQFA) due to the chemical compositions are out of the usual standards. Wesche (1991) has described that LQFA has a high percentage of irregular crystalline particles with low reactivity. LQFA is a hazardous waste, which is undesirable for construction works. Therefore, LQFA is abandoned in the landfills, leading to a serious environmental problem with the heavy metal leachate from. In addition, the number of LQFA tend to increase significantly year by year. The sustainable ways to solve the problem is to treat the LQFA as a valuable precursor for geopolymer application. Although several researchers have studied development of geopolymer from fly ash, there are a few reports on the development of geopolymer from LQFA.

Therefore, this thesis aims to develop the geopolymer material by the using LQFA mixed with the calcined kaolinite clay (CK) as the alumino-silicate precursor.

1.2 Research Objectives

The objective of research will be as follows;

- (i) To develop the cement replacement material from LQFA blended with CK.

(ii) To examine the mixing procedure for improving the compressive strength of LQFA-geopolymer.

(iii) To determine the significant factors that influence on the compressive strength of LQFA-geopolymer by using the experimental design method.

1.3 Scope of the Research

Investigation will be carried out as follows;

(i) The compositions of LQFA and CK will be characterized by X-ray diffraction (XRD) and X-ray fluorescence (XRF).

(ii) Morphology of LQFA will be examined by scanning electron microscope (SEM).

(iii) Optimal properties of the alkali solution to obtain the usable LQFA-geopolymer will be investigated.

(iv) The effect of the mixing procedures on the compressive strength of LQFA-geopolymer will be examined.

(v) Optimum ratios of LQFA/CK to obtain the usable LQFA-geopolymer will be investigated.

(vi) The significant factors that influence on compressive strength of LQFA-geopolymer will be determined by using the analysis of variance (ANOVA) technique.

CHAPTER II

LITERATURE REVIEW

2.1 Introduction

Ordinary Portland cement (OPC) is widely used as a construction material around the world. It is produced by burning calcium oxide (CaO), silica (SiO₂) and alumina (Al₂O₃) at high temperature around of 1450°C (K.Svinning 2006). The carbon dioxide (CO₂) gas releasing into the atmosphere is around 1 ton for the production of 1 ton of PC (Imbabi et al.,2012). As well-known, CO₂ is the main cause of global warming. Recently, several researchers have been investigated a sustainable cement replacement material. Geopolymer is expected to use as a cement replacement material.

Geopolymer is an environmental friendly material. Its production process requires low energy consumption without CO₂ emission. Geopolymer can be synthesized via a reaction between alumino-silicate precursors and alkali solution (potassium/sodium hydroxide and potassium/sodium silicate) (Duxson, et al., 2007). There are several materials that can be used as alumino-silicate precursors such as meta-kaolin, slag, and fly ash. Geopolymer process are shown in Figure 2.1.

In the 1950s, Viktor Glukovsky who is originally developed concrete materials under the names soil silicate concretes and soil cement. Meanwhile, geopolymer was the name given by Davidovits.

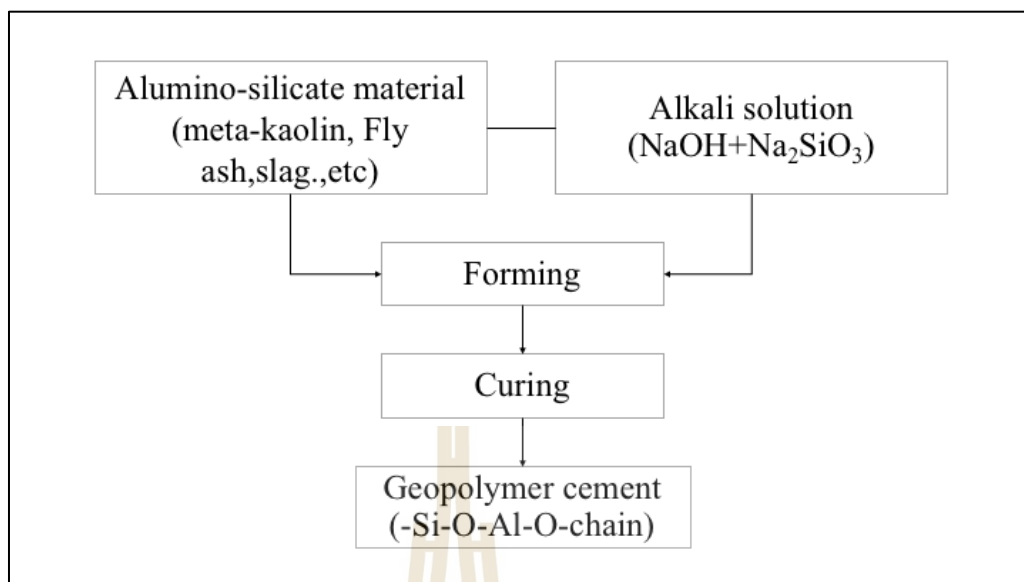
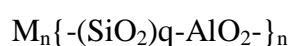


Figure 2.1 Geopolymer process.

2.1.1 Geopolymer cement

Geopolymer cement is alternative material replacement materials. When inorganic aluminosilicate-based materials react with alkaline solutions, polycondensation reaction forming geopolymers occurs. Geopolymer structure is divided into three basic forms which depend on Si/Al ratios. Three basic units are poly (sialate), poly (sialate-siloxo) and poly (sialate-disiloxo) (Yun-Ming et al., 2016) as shown in Figure 2.2.

The chemical structure is written as:



Where M is alkali cation, n is the degree of poly-condensation, and q is Si/Al ratio. During the chemical reaction under alkali conditions, a three-dimensional polymeric chain structure is developed. The concentration of alkali solutions and the

composition of raw materials control the microstructure and the mechanical properties of the geopolymers.

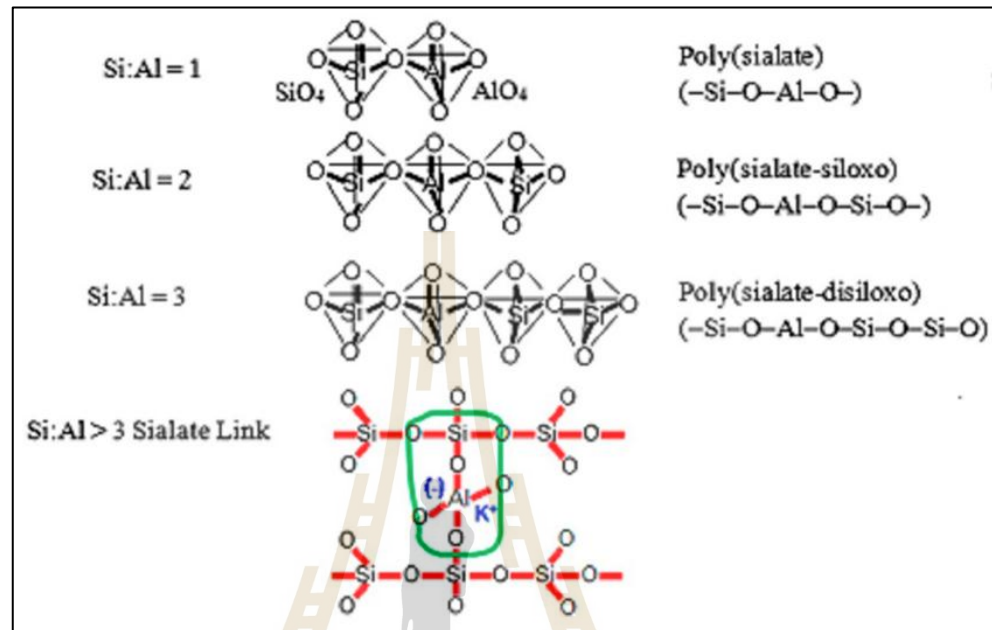


Figure 2.2 Structure of polysialates (Progress in Materials Science 2016).

2.1.2 Sodium Hydroxide (NaOH) solution

The alkaline activator used in geopolymerisation is a combination of sodium silicate or potassium silicate and sodium hydroxide (NaOH) or potassium hydroxide (KOH). The concentration of alkali solution affects the dissolution of fly ash. The function of alkali solution is leaching of Al^{3+} and Si^{4+} ions from fly ash particles. Leaching is dependent on the concentration of alkali solution and leaching time (U.Rattanasak et al., 2009). The NaOH in the activating solution made the reaction process more rapidly and gel is less smooth. High NaOH solution concentration is high dissolution of fly ash particles.

2.1.3 Sodium silicate solution

The sodium silicate (Na_2SiO_3) solution to the NaOH solution as the alkaline activator increase the reaction between the fly ash and the mixed solution. The function of sodium silicate activator is to rapidly dissolves and initiate bonding of fly ash particles. Thus the compressive strength enhance when NaOH: Na_2SiO_3 ratios is increased (Hardjito et al.,2005)

2.1.4 Powder to solution ratio

The powder to solution (P/S) ratio is important to increase the compressive strength of geopolymer. High P/S ratios result in low viscosity of the slurry, mean while the lower P/S ratios increase the geopolymerization time (Z.Zuhua et al., 2009). However, the low P/S ratios could rapidly dissolve the source materials Meanwhile, it is not important to poly-condensation process at high NaOH concentration. Very high P/S ratios can not improve the strength because low extent of binder formation (JL.Provis et al., 2009).

2.1.5 Fly ash

Fly ash can be defined as a waste from the coal-fired power plant. It is mainly used as the pozzolanic materials for the building construction. The fly ash consists of oxide compound of aluminium, silica, iron and some calcium. The fly ash particle varies in chemical and physical due to the source and the furnace used (Wesche, K. 1991). However, not all the fly ashes are suitable for construction works. Numerous standards are used to classify types of fly ash, for example, ASTM C618, BS 3892, and JIS A6201. High quality fly ash (HQFA) is described as high reactive ash, which is a highly desirable commodity in the construction industry. HQFA is obtained from high quality coal with high temperature combustion system.

Morphology of HQFA is mainly vitreous spherical particles. According to ASTM C618-15, HQFA is classified as either in Class F and Class C depending on the contents of the pozzolanic compounds (SiO_2 , Al_2O_3 and Fe_2O_3) (Gomaa, E., et al. 2017). Class F fly ash contains at least 70% of pozzolanic compounds, while Class C fly ash contains at least 50% of the pozzolanic. Another major difference between both types is the calcium content in which a higher calcium content is found in class C fly ash (Gomaa, E., et al. 2017).

2.1.6 Kaolinite clay

Kaolinite is the most common clay mineral used in geopolymer as alumino-silicate precursors. Thermal treatment of kaolinite leads to the transformation of crystalline phases into amorphous phases, which can improve the strength of geopolymer. The thermal treatment at around $500\text{-}800^\circ\text{C}$ which results in dehydroxylation of strong bounded hydroxyl ion on the Al-constitutive layer, and therefore, kaolinite is transformed into meta-kaolin.

2.1.7 Design of Experiment

Design of experimental (DOE) is an effective way to determine the significant factor for reducing the research budget and research time. The input factors are changed to consider the causal variables actors involved with changes in responses in the process of interest (Montgomery, 2005).

2.2 Literature review

Hardjito, D., and Rangan. (2005) developed the low-calcium fly ash based geopolymer by mixing fly ash with the different ratio of sodium silicate to NaOH (0.4 to 2.5) and the concentration of NaOH is 8 M to 14 M. The results showed that

strength of geopolymer is increased with increasing sodium silicate to NaOH ratio and the ratio of 2.5 with 14 M NaOH obtained the highest strength of 68 MPa.

Gomaa, E., et al. (2017) have studied the compressive strength of high calcium fly ash mixed with the different ratios of sodium silicate to NaOH, the studied ratios were 0.5, 1, 1.5 and 2.5. The NaOH concentration was fixed at 10 M. Researchers concluded that the optimum ratio is 1.0. The compressive strength of a geopolymer increase with increasing the curing temperature. The strength is 21 MPa and 41 MPa after curing at room temperature and 70°C, respectively. Likewise, Skvara, F., et al. (2005) have concluded that a high curing temperature (between 40°C to 95°C) was important for the formation of interconnected network within the fly ash geopolymer. However, an extremely high curing temperature is the cause of reducing in compressive strength. This is because the high temperature curing increase the water evaporation which is required for geopolymerization process. Bakkali, H., et al. (2016) have developed the fly ash based geopolymer using the class F fly ash mixed with NaOH solution. The effects of NaOH concentration and the curing temperature on compressive strength of geopolymer were examined. The results showed that the compressive strength fly ash-geopolymer was developed by using NaOH as an activated solution. Compressive strength was also increased by increasing concentrations of NaOH. Compressive strength of 30 MPa was obtained by using 14M NaOH with curing temperature at 80°C. The strength of geopolymer was however, decreased when increasing the curing temperature from 80°C to 100°C.

Chen, L., et al. (2016) investigated the influence of curing temperature (20°C, 40°C, 60°C, 80°C and 100°C) and curing times (24, 72 and 168 hours) on the properties of metakaolin-based geopolymer. They found that the strength of

geopolymer is increased with increasing curing temperature and curing time. The highest strength (52 MPa) is obtained after curing at 60°C for 168 hours. However, the strength is reduced when curing at temperature over 60°C.

Bascarevevic, Z., et al. (2015) have studied the impact of sulfate solution on mechanical properties and structure of fly ash based geopolymer. The fly-ash-geopolymer samples were immersed in the sodium sulfate (Na_2SO_4) solution for the period of 365 days. After immersion, the -Si-O-Si- bonds were broken and the small cracks were appeared. As a result, the strength of geopolymer was slightly decreased around 10%. Skvara, F., et al. (2005) reported that the fly ash based geopolymer has an excellent durability in the corrosive environment of salt solution, and withstand at high temperature up to 600°C. Another advantage of the fly ash geopolymer is the high early strength. GuO, X., et al. (2010) reported that the compressive strength of 63.4 MPa was obtained after curing the class C fly ash geopolymer at 75°C for 8 hours.

Mary, B., et al. (2016) synthesized the geopolymer from fly ash blended with a calcined clay. They found that the strength of the class F fly ash geopolymer was increased more than two times after adding calcined clay. By adding 25 weight% of calcined clay, the maximum strength of 37 MPa was obtained. Likewise, Gorhon. G., et al. (2016) also reported that the strength of the fly ash geopolymer was enhanced by blend fly ash with meta-kaolin.

Zhang, Z., et al. (2014) reported the effect of fly ash on setting time of the metakaolin-geopolymer. Although, the replacement of meta-kaolin by small amount of fly ash (10 weight%) can increase for the compressive strength of the metakaolin-

geopolymer, setting time of metakaolin-geopolymer is also increased. This is due to the contact surface area of solid particles with alkaline solution is decreased.

Diaz, O.B., et al. (2012) investigated the effects of the chemical composition, curing temperature, aging time and strength of a low-purity metakaolin geopolymers. A coarse mineral compose of 70% kaolinite and 30% of quartz was calcined at 750°C for 3 hours to induce the formation of reactive metakaolin. The researchers concluded that the calcined low-purity kaolin can be used to produce the high strength geopolymer. The obtained strengths were 30 MPa and 40 MPa after curing at 20°C for 7 days and 28 days, respectively.

Wang, M.R., et al. (2010) have studied the effect of calcined temperature on compressive strength of the metakaolin-geopolymer. Kaolinite clay was calcined at 800°C and 900°C for 4 hours. The results demonstrated that the calcination temperature effected aluminum atoms more than silicon atoms. By calcining clay at 900°C, the obtained strength of geopolymer was higher than that of 800°C.

Rattanasak, U., and Chindaprasirt P. (2009) have proposed the separate mixing procedure for synthesizing geopolymer. In the separate mixing method, fly ash was mixed with NaOH for 10 min, then a sodium silicate solution was added to the mixture and mixed for 1 minute. The results showed that the mixing sequence had small effects on compressive strength of geopolymer. By using a separate mixing procedure, the strength of geopolymers was around 60-70 MPa, while the strength for the normal mixing was 65 MPa.

Topark-Ngarm, P., et al. (2014) developed the rebar-geopolymer composites from high-calcium fly ash. The mixing process was similar to the separate mixing procedure of Rattanasak U., and Chindaprasirt P. (2009). Researchers conclude that

high-calcium fly ash geopolymer had high bond strength with rebar. In the additions geopolymer concrete had a short setting time due to the presence of high calcium content.

Although, numerous research study on the fly ash based geopolymer, the development of geopolymer from LQFA have never been reported so far. Most researchers have developed the geopolymer from high quality fly ash.



CHAPTER III

Experimental procedure

3.1 Experimental procedure

Details of the LQFA-geopolymer developing procedure are illustrated in Figure 3.1. The details are as follows:

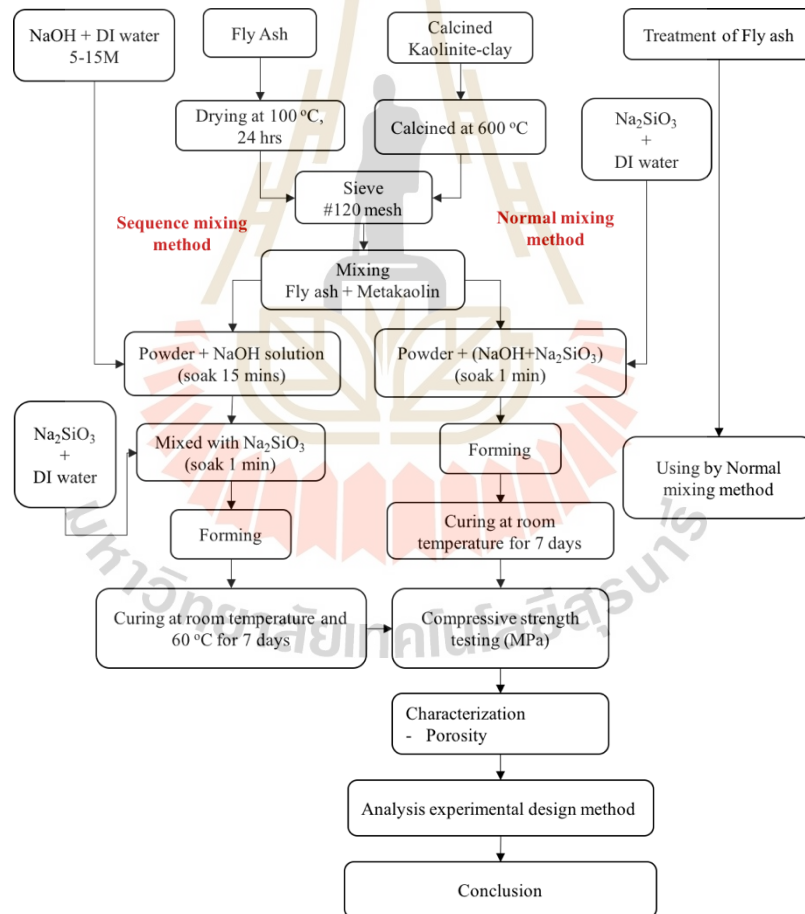


Figure 3.1 Experimental procedure.

3.1.1 Preparation of alkali solution

Sodium hydroxide pellets and distilled (DI) water was mixed to obtain a concentration of 5 M, 10 M and 15 M, then allowed to cool down at a room temperature. Na_2SiO_3 was mixed with DI water to reduce viscosity. The used ratio of Na_2SiO_3 to distilled water is 17:3 by weight.

3.1.2 Precursor preparation

LQFA was obtained from Siam Kraft Industry Co., Ltd., Thailand. To remove moisture and the impurities, LQFA was passed through a 120-mesh sieve and oven-dried at 100°C for 24 hours.

To obtain calcined kaolinite clay (CK) powder, kaolinite clay from Ranong province, Thailand, was grinded by mortar and passed through a sieve before calcined at 600°C for 3 hours.

LQFA and CK powder was blended in the LQFA to CK ratios of 100:0, 70:30, and 50:50 by weight.

3.1.3 Sample preparation

For normal mixing method (NM), NaOH and Na_2SiO_3 solution were mixed in the NaOH to Na_2SiO_3 ratios of 1:1, 1:1.5 and 1:2 by weight then storage for 24 hours. The blended powder (topic 3.1.2) was mixed with an alkali solution for 1 min, then poured into the 50 mm x 50 mm x50 mm steel mold.

For sequence mixing method (SM), the blended powder was mixed with NaOH solution for 15 minutes. Then, added Na_2SiO_3 and mixed for 1 minute. The used ratios of NaOH/ Na_2SiO_3 are 1:1, 1:1.5 and 1.2 by weight. After that, the paste will be poured into the 50 mm x 50 mm x50 mm steel mold.

All samples were sealed with the vinyl sheet before curing at room temperature or 60°C for 7 days.

3.1.4 Treatment of LQFA

The 20 g-LQFA powder in 200 ml of 2 M-NaOH was prepared in the treatment process. The LQFA was treated by NaOH solution. First step, LQFA was mixed with NaOH solution, and then stirred using magnetic stirrer at 60°C for 30 min. Once the reaction complete keep storage the mixture for 24 hours. Then washing LQFA with distilled water (DI water) and dried.

3.2 Characterization techniques

3.2.1 X-ray diffraction

The mineral compositions of LQFA and CK was evaluated by X-ray diffraction (XRD, Bruker D5000) with $\text{CuK}\alpha$ radiation as shown in Figure 3.2.



Figure 3.2 X-ray diffraction (XRD, Bruker D5000).

3.2.2 X-ray fluorescence

X-ray fluorescence (XRF) was used to analyze chemical compositions of LQFA and CK as shown in Figure 3.3.



Figure 3.3 (X-ray Fluorescence XRF).

3.2.3 Setting time

Setting time of geopolymer paste was measured following ASTM C266 2013 shown in Figure 3.4. The apparatus consists of two horizontal arms which carry two weighted steel. The initial needle 2.12 mm dimension, weighs 113.4 g and the final needle 1.06 mm dimension weighs 453.6 g. The formation time was recorded when both of the needle were unable to press.



Figure 3.4 Gilmore needle.

3.2.4 Scanning electron microscope

The LQFA morphology was examined by Scanning electron microscope (SEM, JOEL JSM-6010L V). The signals used to produce an image result need electron reflection that hit the surface of the sample. The reflection of that electron produces secondary electron. The electron can be reflected only when the sample has metal surface. Therefore, it is necessary to coat the sample surface with gold metal before testing shown in Figure 3.5.



Figure 3.5 Scanning electron microscope (SEM, JOEL JSM-6010LV).

3.2.5 Compressive strength

The compressive strength test was examined by Universal Testing Machine Capacity 100 kN (Model: MUL-125 TTR/THAI) as shown in Figure 3.6.



Figure 3.6 Universal Testing Machine Capacity 100 kN (Model: MUL-125 TTR/THAI).

The method for calculating the compressive strength (MPa) is shown in equation (3.1).

$$f_m = \frac{P}{A} \quad (3.1)$$

Where

- f_m is Compressive strength [MPa]
- A is Area of loaded surface [mm^2]
- P is Total maximum load [N]

3.2.6 Porosity

Porosity of geopolymer was determined by Archimedes method. The cured-geopolymers were vacuumed for 45 min. in distilled water to fill up distilled water in open pores. Open porosity is calculated by equations 3.2.

$$\% \text{ Porosity} = \frac{(W_s - W_D)}{(W_s - W_{ss})} \times 100 \quad (3.2)$$

Where W_D , W_s and W_{ss} are dry weight, saturated weight and immersed weight in distilled water, respectively.

3.2.7 Design of Experiment

Design of experiment in which the input factors are changed to consider the causal variables factors involved with changes in responses in the process of interest. Input variables can be divided into 2 types: controllable variables and uncontrollable variables. The first experiment is determined key variables from all controllable variables in the experiment. The next experiment is aimed to determine a suitable level of variables affecting the outcome nearest to target. The impact analysis of the main factors and interactions is analysis of variance (ANOVA) by consideration of the interactions.

ANOVA can be used to analysis two factors of the factorial experiment, in which two characteristics of two factor are A and B. ANOVA using adjusted sum of square (SS) for tests shown in table 3.1 Then put all value in the ANOVA model (program).

Table 3.1 Analysis of variance for compressive strength, using adjusted SS for tests.

Source	DF	Seq. SS	Adjust SS	Adjust MS	F	P
A	a-1	SS_A	SS_A	$MS_A = \frac{SS_A}{a-1}$	$F_o = \frac{SS_A}{MS_E}$	<i>P – Value</i>
B	b-1	SS_B	SS_B	$MS_B = \frac{SS_B}{b-1}$	$F_o = \frac{SS_B}{MS_E}$	<i>P – Value</i>
AB	(a-1)(b-1)	SS_{AB}	SS_{AB}	$MS_{AB} = \frac{SS_{AB}}{(a-1)(b-1)}$		<i>P – Value</i>
Error	ab(n-1)	SS_E	SS_E	$MS_E = \frac{SS_E}{ab(n-1)}$		
Total	Abn-1	SS_T				

Where

DF is Degrees of freedom

Seq.SS is Sequence of Sum of square

Adjust SS is Sum of square

Adjust MS is Mean squares

F is F-value

P is P-value

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CHAPTER IV

Results and discussion

4.1 Chemical compositions of LQFA and CK

Chemical composition of LQFA and CK characterized by X-ray fluorescence (XRF) is shown in Table 4.1. The main compositions of LQFA are CaO (30 wt%), SiO₂ (33wt%), Al₂O₃ (17 wt%) and SO₃ (11 wt%). Chemical compositions of CK compose of SiO₂ (57 wt%) and Al₂O₃ (33wt%).

Table 4.1 Chemical composition of LQFA and CK.

Raw materials	Chemical composition (wt %)								
	Al ₂ O ₃	SiO ₂	CaO	K ₂ O	TiO ₂	MnO ₂	Fe ₂ O ₃	SO ₃	Etc.
LQFA	17	33	30	1	1	-	7	11	1
CK	33	57	-	5	-	-	3	-	1

The X-ray diffraction (XRD) of LQFA is demonstrated in Figure 4.1. Quartz, anhydrite-gypsum (CaSO₄), lime and calcium hydroxide are the main phases.

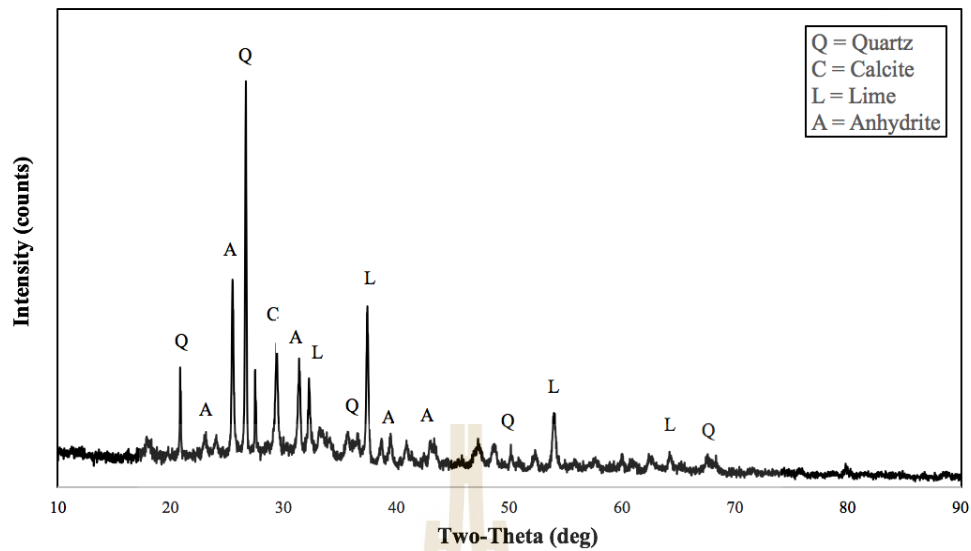


Figure 4.1 X-ray diffraction (XRD) of LQFA.

4.2 Morphology of LQFA

Morphology of LQFA is shown in Figure 4.2. All particles are irregular in shape. Although, LQFA are out of the usual standards, Wesche (1991) has reported that low-quality fly ash is described by a low reactivity and has high percentage of irregular crystalline particles.

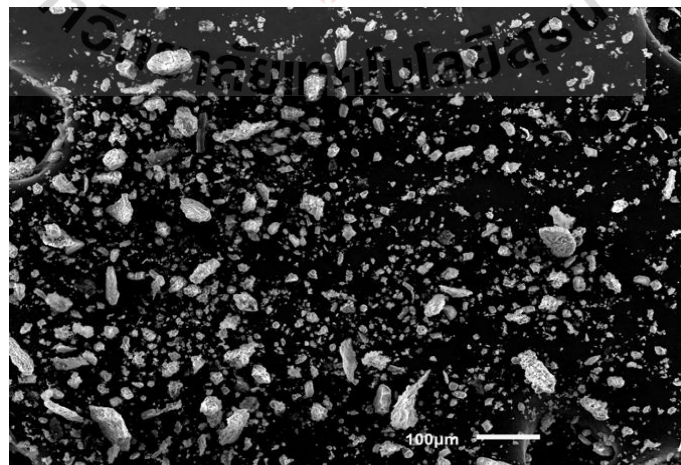


Figure 4.2 SEM image of LQFA.

4.3 Properties of the alkali solution on compressive strength of geopolymer

All samples were prepared by using of LQFA and CK in the ratios of 1:1, 1:1.5, and 1:2, respectively, and the P/S ratio is 0.6. All samples were cured at room temperature for 7 days.

Figure 4.3 shows the compressive strength of LQFA-geopolymer with various concentration of NaOH and NaOH to Na_2SiO_3 ratios. Compressive strength of geopolymer reach a maximum at NaOH to Na_2SiO_3 ratio of 1:2 with 15-M NaOH. The use of low concentration of 5-M NaOH give low strength geopolymer less than 9 MPa. Low concentration of NaOH is low solubility due to relatively low base condition (R. ubolluk et al., 2009). For 10 M of NaOH, the compressive strength is slightly increase around 11 MPa. By using 15 M of NaOH, the result is shows a high compressive strength compared to that of 5 M and 10M of NaOH. Due to a high of dissolution of LQFA in NaOH solution as well as an increase of the Al and Si ion concentration (Ubolluk and Prinya, 2009). The role of Na_2SiO_3 is binder in the geopolymerization process. An increase in Na_2SiO_3 content enhance silica gel and increase the occur of geopolymerization reaction. By using 15 M of NaOH with 1:2 ratio of NaOH: Na_2SiO_3 shows high strength of geopolymer up to 15 MPa. However, the strength of LQFA-geopolymer is still lower than the minimum requirement of PC (19 MPa).

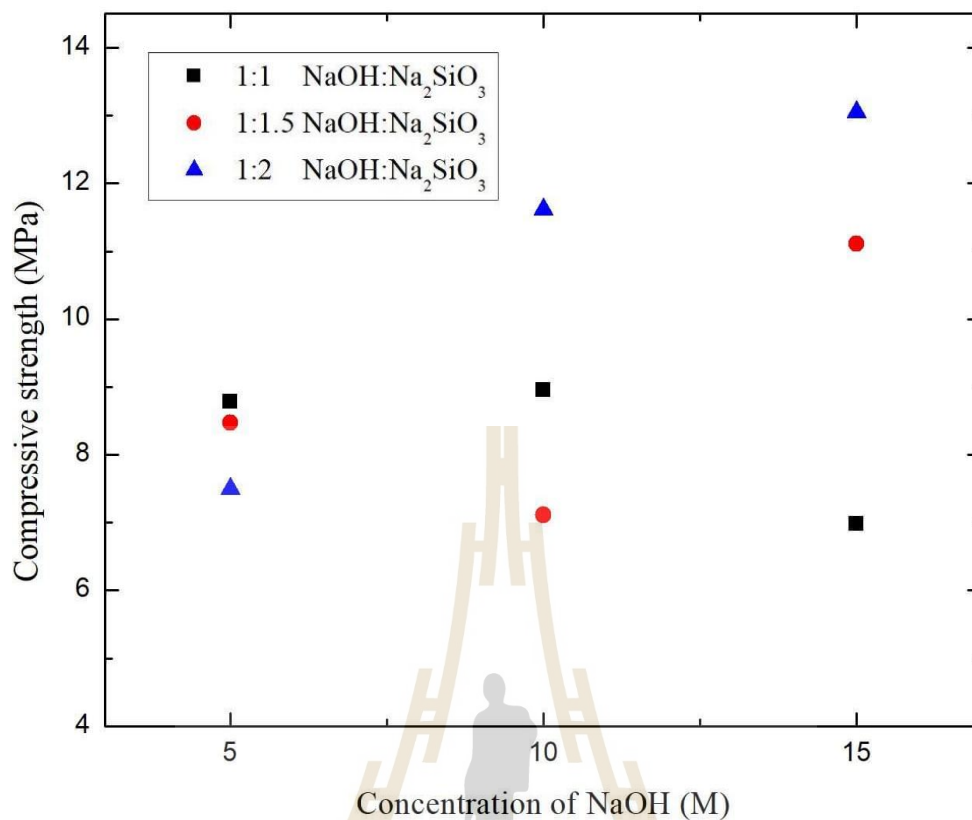


Figure 4.3 The compressive strength of LQFA-geopolymer with different NaOH concentration and NaOH:Na₂SiO₃ ratio.

4.4 Effect of powder to solution ratio (P/S) on properties of geopolymer

All samples were prepared by using 15M of NaOH, the ratio of NaOH/Na₂SiO₃ of 1:2 by weight and the LQFA to CK ratio of 0.6, 0.8 and 1 by weight. All samples were cured at room temperature for 7 days.

Figure 4.4 shows effect of the P/S ratio on the compressive strength of geopolymer. Strength of geopolymer increases when P/S ratio increases from 0.6 to 0.8 because the excess solution causes the increased porosity (Luna, Y., et al. 2016). However, the strength of geopolymer reduces when the P/S ratio is above 0.8 due to the sufficient solution is required for geopolymerization reaction (Luna, Y., et al. 2016).

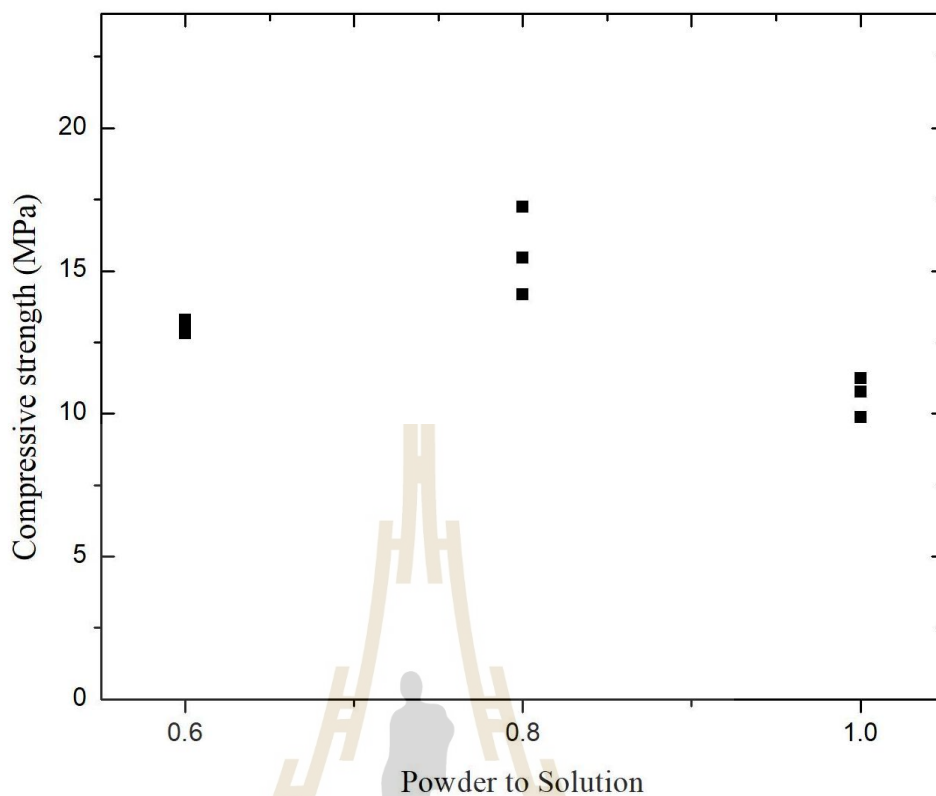


Figure 4.4 Compressive strength of LQFA-geopolymer with different powder/solution (P/S).

4.5 Effect of ratios of LQFA to CK on compressive strength and porosity of geopolymer

All samples were prepared by using 15 M NaOH solution, the ratio of NaOH/Na₂SiO₃ of 1:2 by weight, the P/S ratio of 0.8 by weight. All samples were cured at room temperature for 7 days.

The compressive strength of geopolymer at different replacement of LQFA with CK is shown in Figure 4.4. Compressive strength of 0 wt%, 30 wt% and 50 wt% of CK replacements are 3 MPa, 3MPa and 5 MPa, respectively. LQFA has low reactivity with the alkali activator solution. By using 100% LQFA, the compressive strength of geopolymer is lower than 5 MPa which is not enough to use in engineering

applications. Compressive strength of geopolymer increase with increasing the CK replacement because the reactivity of CK is higher than that of LQFA.

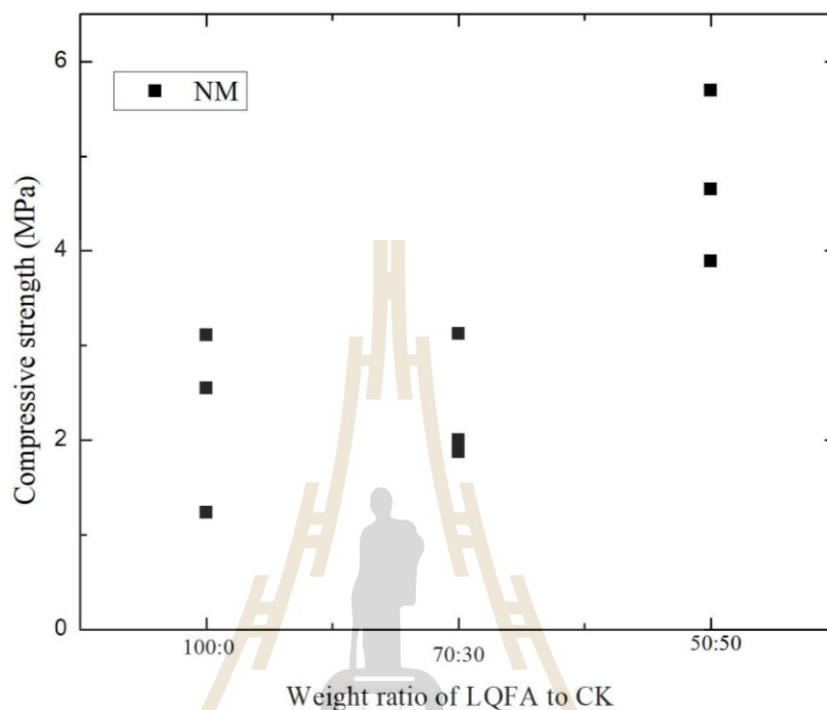


Figure 4.5 Compressive strength of LQFA-geopolymer after 7 days curing.

Figure 4.6 shows porosity of geopolymer after curing for 7 days. Porosity of 0 wt%, 30 wt% and 50 wt% CK replacement is 50%, 34% and 25%, respectively. Porosity of geopolymer relates to reactivity of precursor. Low porous geopolymer is obtained by using a high reactivity precursor. Therefore, the porosity of geopolymer reduce with increasing of CK replacement.

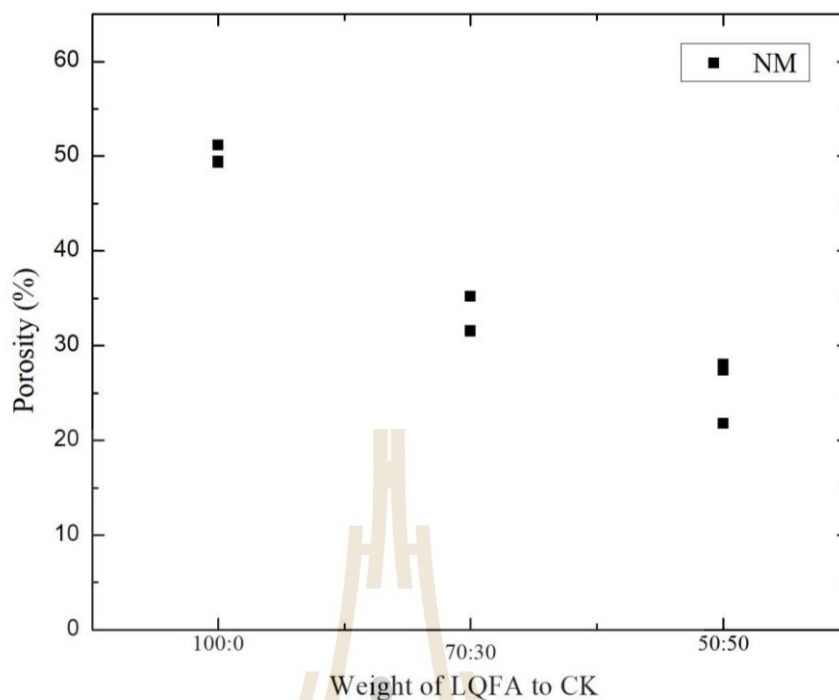


Figure 4.6 Porosity of LQFA-geopolymer by using NM.

4.6 Sequence mixing on the properties of geopolymer

All samples were prepared by using 15 M NaOH, the ratio of NaOH/Na₂SiO₃ of 1:2 by weight, the P/S ratio of 0.8 by weight. All samples were cured at room temperature for 7 days.

The compressive strengths of LQFA-geopolymer fabricated by SM and NM are shown in Figure 4.7. Maximum strength of geopolymer fabricated by NM is 5 MPa. By using SM and replacing LQFA by 50-wt%, the compressive strength of geopolymer is around 4 MPa, which is sufficient for engineering applications. The compressive strength of LQFA-geopolymer fabricated by SM is significantly higher than that of NM. This is because, the porosity of LQFA-geopolymer is reduced by SN fabrication as show in Figure 4.8. Porosity of 0%, 30% and 50% CK replacement is 16%, 22% and 8%, respectively. Also, the bubbles gas is generated from the reaction

between alkaline solution and the metal impurity in LQFA. The generated bubbles are left in the sample during geopolymer hardening process. As a result, geopolymer has high porosity after curing. Deconstruction and condensation steps of geopolymerization process are separated by the SM. The generated bubbles are leaved from the sample before it is hardened. The porosity is reduced, and therefore the strength is increased by using the SM.

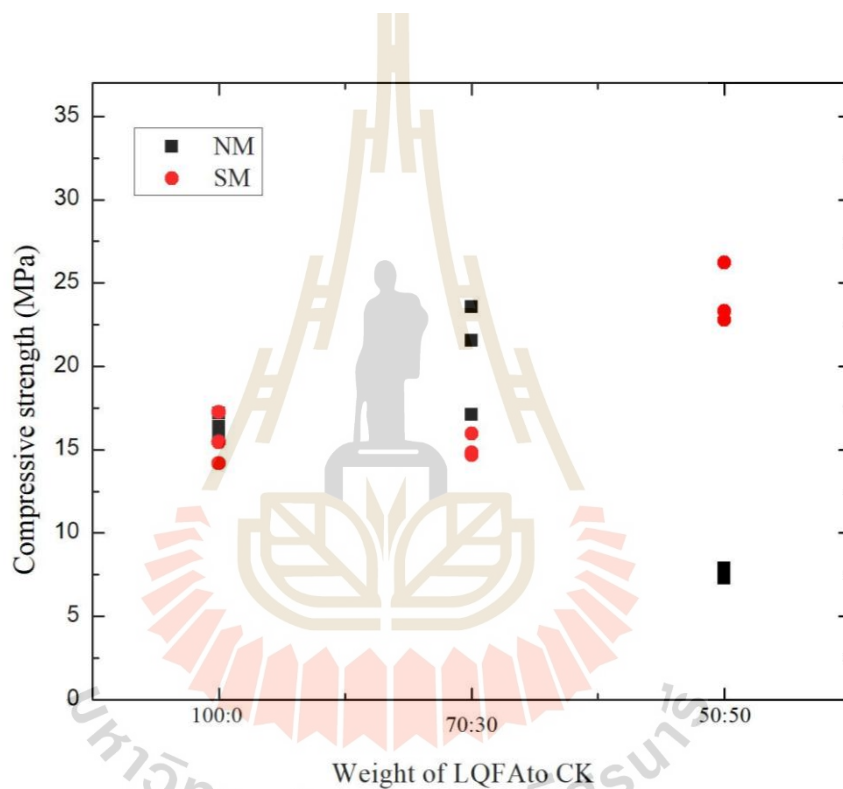


Figure 4.7 Compressive strength of LQFA-geopolymer with different mixing method.

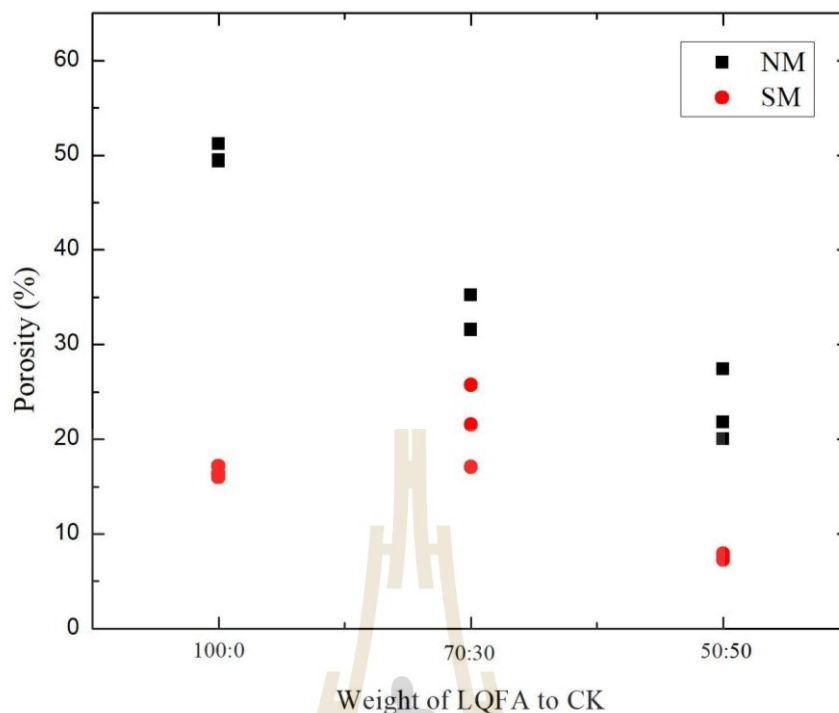


Figure 4.8 The porosity of LQFA-geopolymer by using SM and NM.

4.7 The treated-LQFA (T-LQFA) on properties of geopolymer

All samples were prepared by using 15 M of NaOH, NaOH/Na₂SiO₃ ratio of 1:2 by weight, the P/S ratio of 0.8 by weight and by using NM. All samples were cured at room temperature for 7 days.

The treated fly ash (T-LQFA) is used to eliminate the bubble gas which is generated from the reaction between reactive solution and metal-impurity in fly ash. Chemical composition of the treated fly ash is shown in Table 4.3. After the chemical treatment process, most of the metal impurities are removed. The content of CaO increases. In the literature review, using high CaO fly ash mixed with alkali-activated, the compressive strength and the setting time are reduced because high CaO content of LQFA has low Al₂O₃ content (P.chindaprasirt et al, 2018). Figure 4.10 shows the compressive strength of T-LQFA-geopolymer. The strength of all samples is lower

than 19 MPa. It was due to, the essential components for the strength development., i.e., Si ion and Al ion, are washed away from T-LQFA.

Table 4.2 The chemical composition of LQFA after treatment.

	Chemical composition (wt %)								
	Al ₂ O ₃	SiO ₂	CaO	K ₂ O	TiO ₂	MnO ₂	Fe ₂ O ₃	SO ₃	Other component
LQFA-T	8.57	19.66	51.41	1.25	1.17	0.46	11.29	1.16	5.026

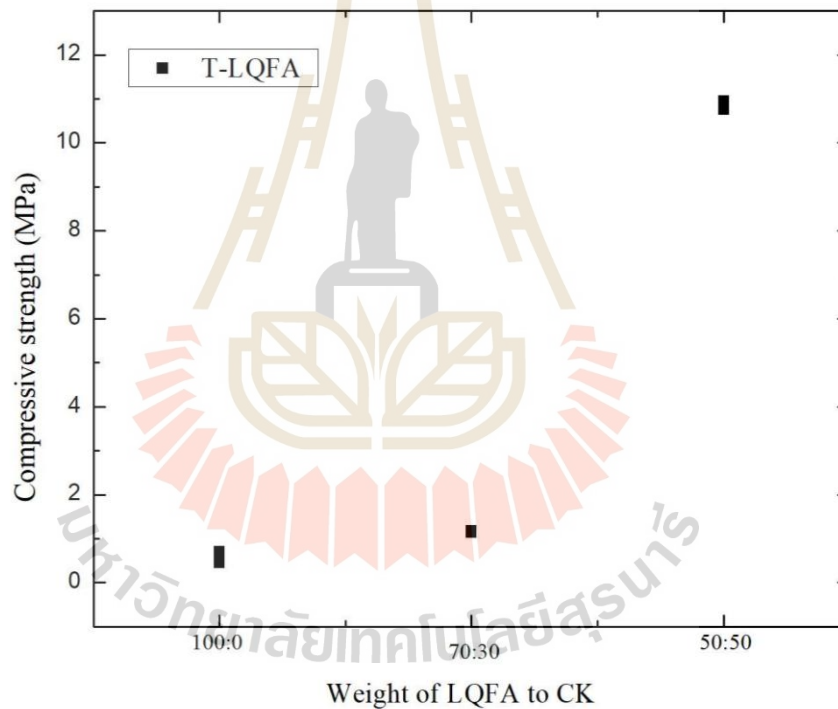


Figure 4.9 Compressive strength of LQFA-geopolymer after treatment of LQFA curing 7 days.

4.8 Effect of curing temperature on compressive strength and porosity of geopolymer

All samples were prepared by using 15 M NaOH solution, the ratio of NaOH/Na₂SiO₃ ratio of 1:2 by weight and the P/S ratio of 0.8 by weight. All samples were cured at room temperature for 7 days.

Figure 4.10 shows the strength of LQFA-geopolymer after curing at room temperature and at 60°C. The results show that the strength of curing at room temperature is higher than curing at 60°C, because the cracks are developed on the surface.

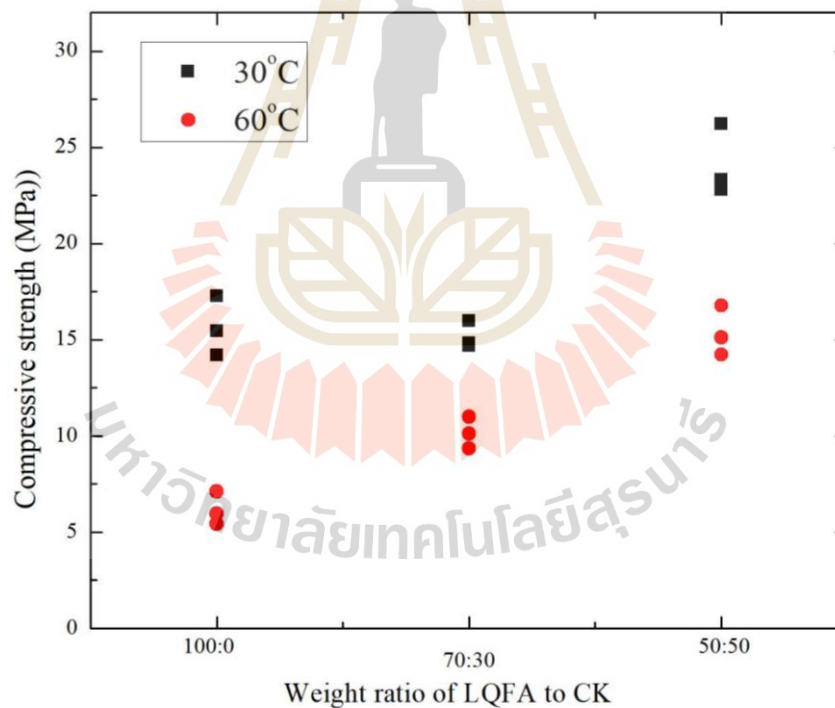


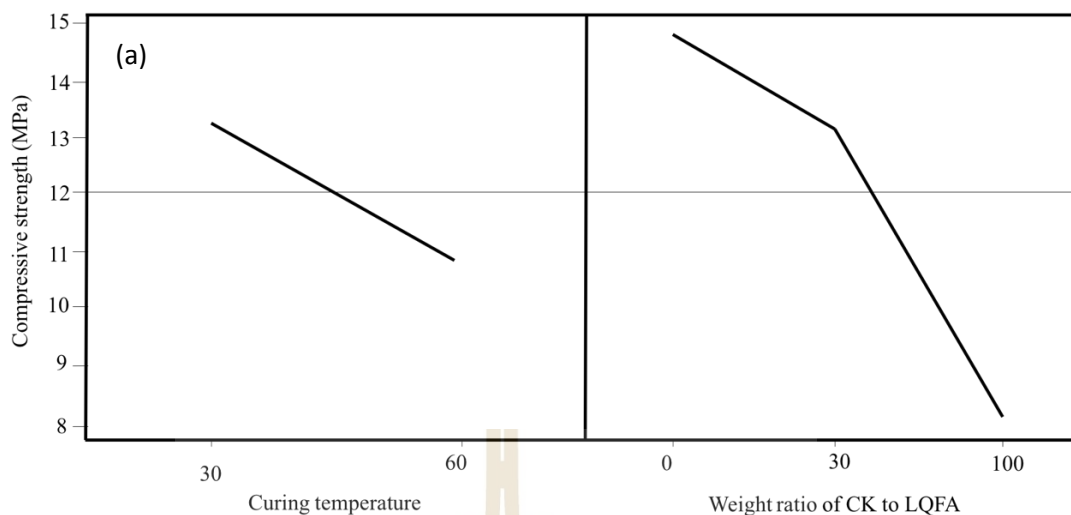
Figure 4.10 The compressive strength of LQFA-geopolymer by curing at room temperature and 60°C.

4.9 Significant factor determined by DOE

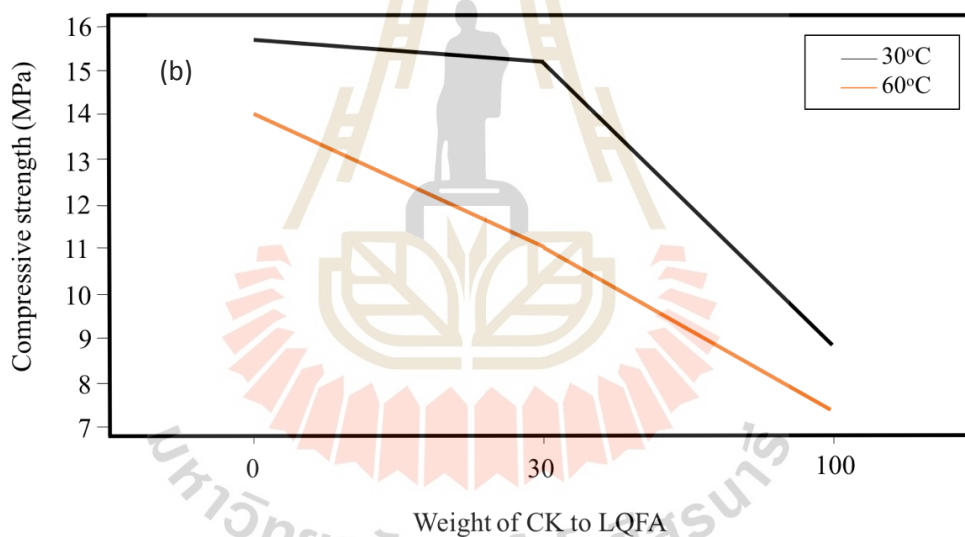
The data obtained from the experiment were analyzed using the Minitab Release 15 program. A factor with a p-value < 0.05 was considered statistically with a 95% confidence interval. The ANOVA test showed that the most significant factor was represented by CK to LQFA ratio. The ANOVA test is shown in Table 4.2.

Table 4.3 Analysis of variance for compressive strength, using adjusted SS for tests.

Source	DF	Seq.SS	Adjust SS	Adjust MS	F	P
Curing Temp.	1	26.298	26.298	26.298	4.29	0.061
Ratio of CK to LQFA	2	146.049	146.049	73.024	11.91	0.001
Curing temp.*Ratio of CK to LQFA	2	6.139	6.139	3.070	0.50	0.618
Error	12	73.560	73.560	6.130		
Total	7	252.047				



a) Main effects Plot for compressive strength (MPa)



b) interaction Plot for compressive strength

Figure 4.11 The main effect plot and interaction plot for compressive strength.

The results from effect plot and interaction plot are shown in Figure 4.11. The interaction plot presented that the 30°C curing temperature achieved a better result of compressive strength than the 60°C curing temperature. For the main effect plot, the 30°C curing temperature also obtained a better result of compressive strength than the

60°C curing temperature shown in Figure 4.11(a). The interaction plot showed that the 30°C curing temperature is appropriate for further use in the experiment. The results of further testing at the curing temperature of 30°C with different percentages of the CK to LQFA ratio showed that the most outstanding result was with the 50:50 of CK to LQFA ratio as shown in Figure 4.11(b).



CHAPTER V

Conclusion

In this thesis, cement replacement material, i.e., geopolymer has been developed. Geopolymer is fabricated by using low-quality-fly-ash (LQFA) and calcined-kaolinite-clay (CK) as precursors. To obtain sufficient strength for engineering applications, the sequence mixing method (SM) is required. Using normal mixing method (NM), geopolymer has low strength because, the bubbles are generated from the reaction between alkaline solution and the metal impurity in LQFA during hardening process. By using SM method, the generated bubbles are released before the sample was hardened. As a result, the porosity of the sample is reduced and the strength is significantly increased.

The chemical treatment procedure is used to eliminate the metal impurity in LQFA. However, the strength of geopolymer still low due to Si ions and Al ions are leached from LQFA during the treatment process.

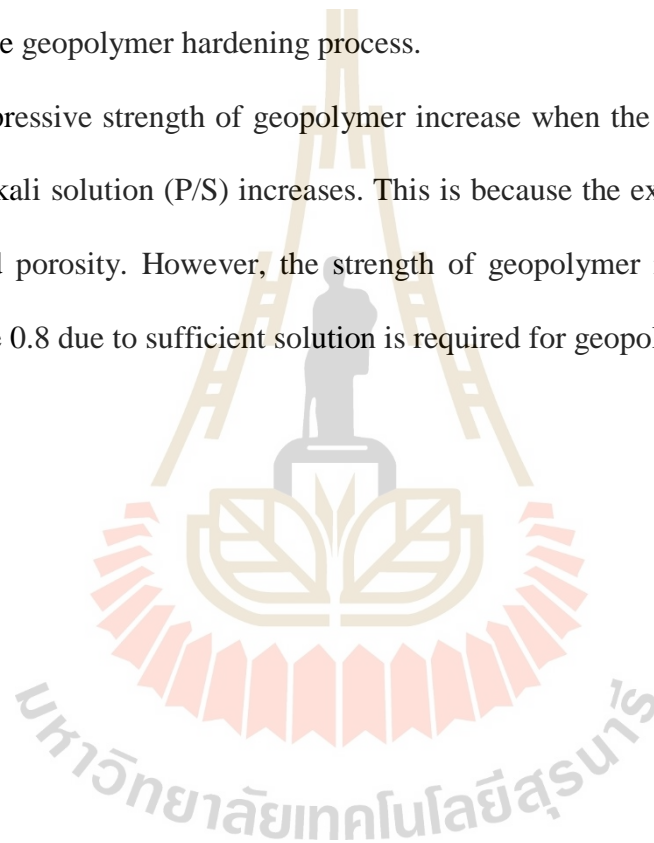
Design of experiment (DOE) is used to determine the significant factors which influence on the compressive strength of the geopolymer. The results indicate that the most important factor is the ratio of LQFA to CK. Moreover, the curing temperature at 30°C achieved a higher compressive strength than the curing temperature at 60°C.

CK is essential for development of geopolymer from LQFA. Using LQFA as a single precursor, geopolymer has low strength because LQFA has low reactivity with an alkali solution. Reactivity of precursor is improved by blending LQFA with CK. The ratio of LQFA to CK of 1.0 by weight is the optimum ratio.

Compressive strength of the cured-geopolymer at room temperature is higher than at the curing at 60°C. Curing at 60°C, the surface cracks are formed which is the causes of low strength.

High concentration of NaOH solution and enough Na₂SiO₃ binder are required for fabrication of the high strength geopolymer. High concentration of NaOH is required for leaching of Al and Si ions in LQFA. Meanwhile, Na₂SiO₃ solution act as a binder in the geopolymer hardening process.

Compressive strength of geopolymer increase when the ratio of the precursor powder to alkali solution (P/S) increases. This is because the excess solution leads to the increased porosity. However, the strength of geopolymer reduces when the p/s ratio is above 0.8 due to sufficient solution is required for geopolymerization reaction.



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