STRENGTH DEVELOPMENT OF GEOPOLYMER MORTARS USING CALCIUM CARBONATE SLUDGE AND FLY ASH PRECURSORS

SOE THANDAR

A Thesis Submitted in Partial Fulfillment of the Requirements for the Degree of
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การพัฒนากำลังของจีโอพอลิเมอร์มอร์ตาร์ที่ใช้ตะกอนแคลเซียมคาร์บอเนตและเถ้าลอยเป็นวัสดุตั้งต้น

นางโซ ทันดาร์

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต
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งานวิจัยนี้ศึกษาผลของการกระตุ้นด้วยด่างต่อมอร์ตาร์จีโอพอลิเมอร์ที่ผลิตจากเถ้าลอยและตะกอนแคลเซียมคาร์บอเนต (CCS) โดยพิจารณาตัวแปรสำคัญ ได้แก่ ความเข้มข้นของ NaOH (5M, 10M, 15M), อัตราส่วนโซเดียมซิลิเกตต่อโซเดียมไฮดรอกไซด์ (0:100–90:10), อัตราส่วนเถ้าลอยต่อ CCS (100:0–70:30) และอัตราส่วนของเหลวต่อวัสดุประสาน (0.3–0.5) ผลการทดสอบพบว่าอัตราส่วนของเหลวต่อวัสดุประสานที่ 0.4 ให้สมรรถนะที่เหมาะสมที่สุด โดยหลายส่วนผสมมี กำลังอัดสูงกว่าค่ากำลังอัดอ้างอิงของมอร์ตาร์ซีเมนต์ที่ 24.5 MPa ความเข้มข้น NaOH ที่สูงขึ้น (15M) ช่วยลดระยะเวลาการก่อตัวและเพิ่มกำลังอัดอย่างต่อเนื่อง การผสม CCS ซึ่งเป็นผลพลอยได้จากอุตสาหกรรมที่มีแคลเซียมสูง ส่งผลให้เกิดระบบสารยึดเหนี่ยวแบบผสมระหว่างเจล N-A-S-H และ C-A-S-H ทำให้ได้โครงสร้างจุลภาคที่หนาแน่นและสม่ำเสมอยิ่งขึ้น การวิเคราะห์โครงสร้าง จุลภาคและแร่ธาตุยืนยันการก่อตัวของเฟสอสัณฐานที่ชัดเจนมากขึ้น โดยเฉพาะที่ความเข้มข้น NaOH สูง นอกจากนี้ยังมีการประยุกต์ใช้การวิเคราะห์เชิงเครื่องเรียนรู้ (XGBoost ร่วมกับ SHAP analysis) ซึ่งให้ความแม่นยำสูงในการทำนายกำลังอัด และชี้ให้เห็นว่าอัตราส่วนโซเดียมซิลิเกตต่อ NaOH เป็นตัวแปรที่มีอิทธิพลมากที่สุด รองลงมาคือความเข้มข้นของ NaOH ผลการศึกษานี้แสดงให้เห็นถึงศักยภาพของจีโอพอลิเมอร์จาก CCS–เถ้าลอยในการพัฒนาเป็นวัสดุก่อสร้างที่ยั่งยืนผ่านการออกแบบส่วนผสมและกระบวนการที่เหมาะสม

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ลายมือชื่อนักศึกษา ....
ลายมือชื่ออาจารย์ที่ปรึกษา ....

SOE THANDAR: STRENGTH DEVELOPMENT OF GEOPOLYMER MORTARS USING
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Keyword: Geopolymer mortar, fly ash, calcium carbonate sludge, alkaline activation, compressive strength, microstructural analysis, machine learning

This study examines the effects of alkaline activation on geopolymer mortars produced from fly ash and calcium carbonate sludge (CCS) precursors. Key parameters included NaOH molarity (5M, 10M, 15M), sodium silicate-to-sodium hydroxide ratios (0:100–90:10), fly ash-to-CCS ratios (100:0–70:30), and liquid-to-binder ratios (0.3–0.5). Optimal performance was achieved at an L/B ratio of 0.4, with several mixes surpassing the reference cement mortar strength of 24.5 MPa. Higher NaOH concentrations (15M) consistently shortened setting times and improved compressive strength. The addition of CCS, a calcium-rich industrial by-product, promoted the formation of hybrid N-A-S-H and C-A-S-H gels, leading to denser and more homogeneous microstructures. Microstructural and mineralogical analyses confirmed enhanced amorphous phases and compact matrices, especially at higher NaOH levels. Machine learning models (XGBoost with SHAP analysis) achieved high predictive accuracy for compressive strength and identified the sodium silicate-to-NaOH ratio as the most influential factor, followed by NaOH molarity. Overall, the results highlight the potential of CCS–fly ash geopolymers as sustainable construction materials through optimized mix design and processing conditions.

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LIST OF ABBREVIATIONS

AdaBoost = Adaptive Boosting

AI = Artificial Intelligence

Al_2O_3 = Aluminum Oxide

ASTM = American Society for Testing and Materials

C-A-S-H = Calcium Aluminate Silicate Hydrate

$Ca(OH)_2$ = Calcium Hydroxide

CaO = Calcium Oxide

CatBoost = Categorical Boosting

CCS = Calcium Carbonate Sludge

CO2 = Carbon Dioxide

FA = Fly Ash

Fe_2O_3 = Iron Oxide

GGBFS = Ground Granulated Blast Furnace Slag

H_2O = Water

l/b = Liquid-to-Binder Ratio

LCA = Life Cycle Analysis

MgO = Magnesium Oxide

MSE = Mean Squared Error

LIST OF ABBREVIATIONS (Continued)

N-A-S-H = Sodium Alumino-Silicate Hydrate

Na_2O = Sodium Oxide

NaOH = Sodium Hydroxide

Na_2SiO_3 = Sodium Silicate

OPC = Ordinary Portland Cement

RMSE = Root Mean Squared Error

R^2 = R-squared

SEM = Scanning Electron Microscopy

SH = Sodium Hydroxide

SHAP = SHapley Additive exPlanations

SiO_2 = Silicon Dioxide

SO_3 = Sulfur Trioxide

SS = Sodium Silicate

SVM = Support Vector Machine

XGBoost = Extreme Gradient Boosting

XRD = X-ray Diffraction

CHAPTER I

INTRODUCTION

1.1 Introduction

Cement production plays a crucial role in modern infrastructure development as the primary binder in concrete and other construction materials. However, it is also one of the most significant contributors to global carbon dioxide (CO2) emissions, accounting for nearly 8% of total anthropogenic CO2 emissions (Andrew, 2018; Olivier et al., 2017). The calcination of limestone, an energy-intensive process, is the primary source of these emissions, releasing nearly 900 kg of CO2 per ton of cement produced (Barcelo et al., 2014; Miller et al., 2018). This environmental impact, combined with extensive natural resources use and high energy consumption in cement manufacturing, makes the transition toward sustainable alternatives imperative (Barcelo & Kline, 2012; Elshabrawy, 2025; Environment et al., 2018).

Geopolymer-based materials have emerged as promising alternatives to traditional Portland cement by harnessing alkali-activated polymerization reactions, offering significant environmental benefits while maintaining or exceeding conventional mechanical properties (Barcelo et al., 2014; Provis, 2018). Through the elimination of energy-intensive limestone calcination and the strategic utilization of industrial by-products, these materials can reduce CO2 emissions by up to 80% compared to Portland cement-based products, representing a significant step toward sustainable construction practice (Habert et al., 2011; McLellan et al., 2011). Recent studies have expanded the application of geopolymers to lightweight and high-strength concrete, including performance under elevated temperatures and the use of advanced additives like nanomaterials, fibers, and industrial by-products (Amin et al., 2022; Amin et al., 2021; Tayeh et al., 2022; Tayeh et al., 2021; Zeyad et al., 2024; Zeyad, Bayagoob, et al., 2025; Zeyad, Magbool, et al., 2025).

Geopolymer synthesis occurs through alkali activation of aluminosilicate materials at lower temperatures (typically 60-80oC), significantly reducing energy consumption. The process involves the dissolution of industrial by-products like fly ash or slag in an alkaline solution, followed by polycondensation reactions that form three-dimensional aluminosilicate networks. Beyond their environmental advantages, geopolymers have demonstrated superior performance characteristics under aggregative conditions, including enhanced durability with compressive strength exceeding 70 MPa and stability at temperature up to 800oC (Tanya Bakharev, 2005; Kong & Sanjayan, 2010).

Various industrial by-products have been investigated as potential precursors for geopolymer synthesis, with fly ash (FA) establishing itself as a cornerstone material due to its unique chemical composition and widespread accessibility (Davidovits, 2008; Provis, 2018). The effectiveness of FA in geopolymer systems stems from its high concentrations of reactive silica (SiO2, 40-60%) and alumina (Al2O3, 20-30%), spherical particle morphology, and optimized particle size distribution, creating ideal conditions for geopolymerization reactions (Fernández-Jiménez & Palomo, 2003; Kutchko & Kim, 2006). Recent research has indicated the critical influence of reactive Si/Al ratios on geopolymers' mechanical performance and consistency. Liu et al. (2022) offer a comprehensive review linking Si/Al ratios to strength across various industrial residues, while Liu et al. (2023) correlate reactive Si, Al, and Fe dissolution to compressive strength in fly ash–based systems. However, the accelerating global shift away from coal-fired power generation threatens both the availability and consistency of FA supply (Clark et al., 2020; Oberschelp et al., 2019), necessitating exploration of alternative and complementary precursor materials to ensure continued development of sustainable construction technologies.

Calcium carbonate sludge (CCS) from the sugar industry presents a particularly promising solution, offering both environmental and technical advantages. As a waste product from sugarcane juice clarification, CCS is generated in large quantities globally and typically discarded in landfills or waste ponds, creating environmental pollution and waste management challenges for the sugar industry (Kumar et al., 2021). The incorporation of CCS into geopolymer systems thus addresses two sustainability challenges simultaneously: reducing cement-related CO2 emissions while diverting an industrial waste stream from landfills (Gopinath et al., 2018; Kumari et al., 2024).

While various Ca-rich precursors, such as ground granulated blast furnace slag, cement kiln dust, and limestone powder, have been widely studied for enhancing geopolymer performance through C-A-S-H gel formation, CCS remains underexplored. CCS is primarily a calcite-based waste material generated during sugar clarification processes. It contains over 90% CaO but lacks aluminosilicates, requiring hybridization with sources like fly ash.

1.2 Problem Statement

Previous research has shown that calcium carbide slag with high calcium oxide (CaO) content, similar to CCS, promoted the formation of calcium aluminosilicate hydrate (C-A-S-H) gels, which significantly enhanced early strength development and durability in geopolymer systems (Hanifa et al., 2025; Qiu et al., 2022). When combined with FA, the calcium-rich nature of this material complemented FA's silica and alumina contents, enabling the formation of a sophisticated hybrid binding system containing both sodium aluminosilicate hydrate (N-A-S-H) and C-A-S-H gels (Narani & Siddiqua, 2024; Yang et al., 2024; Zhang et al., 2023). This dual gel formation mechanism enhanced mechanical properties and promoted microstructural homogeneity in the final material.

The successful development of geopolymer systems demanded understanding the complex interplay between alkaline activation parameters and precursor characteristics (Krivenko et al., 2014). The activation process, involving aluminosilicate dissolution and subsequent gel formation (Duxson & Provis, 2008), remains inadequately explored for hybrid FA-CCS systems despite existing research on traditional FA-based geopolymers. Additionally, the liquid-to-binder (l/b) ratio critically influences workability and mechanical properties, with optimal ranges typically between 0.3-0.5 (Chindaprasirt & Rattanasak, 2023; Li et al., 2019; Xie et al., 2020), requiring careful control to balance performance and practical application requirements.

While previous research has established the influence of alkaline activator concentration and composition on traditional FA-based geopolymers, the interaction between activator parameters and hybrid FA-CCS systems remains largely unexplored. This research addressed these knowledge gaps by investigating the effects of alkaline activation and precursor proportions on hybrid FA-CCS geopolymer mortars. This study examined the influence of NaOH molarity (5M, 10M, and 15M), sodium silicate-to-sodium hydroxide (SS:SH) ratio (0:100 to 90:10), FA:CCS ratio (100:0 to 70:30), and liquid-to-binder (l/b) ratio (0.3, 0.4, 0.5) on setting time and compressive strength development. In recent years, machine learning has emerged as a powerful tool in materials science for uncovering complex relationships among input parameters and predicting mechanical properties of advanced materials (Khaled & Singla, 2025). In this study, four machine learning algorithms (XGBoost, CatBoost, AdaBoost, and Support Vector Machine) were systematically evaluated based on their established capabilities for handling complex, non-linear materials science data. These algorithms were employed to quantify feature influence on strength predictions, providing a mechanistic interpretation of compositional effects on geopolymer properties. This research hypothesized that calcium-rich CCS will accelerate setting times and enhance early strength when optimally combined with FA and appropriate alkaline activators. Our objectives are to determine the optimal FA-CCS combination and most effective alkaline activator composition for these hybrid systems, thereby establishing fundamental relationships between composition, processing, and performance for sustainable construction materials.

1.3 Purpose of the Research

This study contributes to the advancement of sustainable construction materials by introducing a novel hybrid geopolymer system using FA and CCS, an underutilized industrial by-product. Furthermore, the integration of machine learning provides a predictive framework and mechanistic insight into parameter sensitivity, which enhances mix optimization and promotes data-driven material design. These findings bridge experimental material science and AI-based modeling to support the development of high-performance, low-carbon construction materials aligned with circular economy goals.

Specifically, the research seeks to achieve the following objectives:

1) To determine the optimal mix proportions of fly ash and calcium carbonate sludge in geopolymer mortars that result in improved setting times, compressive strength, and durability. By investigating different ratios of these materials, the study aims to identify formulations that provide the best performance characteristics for various construction applications.

2) To investigate the effects of varying molarities of sodium hydroxide (NaOH) and different ratios of NaOH to sodium silicate on the properties of the geopolymer mortars. Understanding how these alkaline solutions influence setting times, compressive strength, and workability will provide valuable insights into developing high-performance geopolymer mortars.

3) To compare the performance of different geopolymer mortars in terms of their mechanical properties, such as setting time and 7-day compressive strength. This comparison will help evaluate the effectiveness of various binder ratios and alkaline solutions, allowing for the identification of the most suitable formulations for practical use.

4) To assess the feasibility of using fly ash and calcium carbonate sludge-based geopolymer mortars in real-world construction scenarios. The study will explore

the potential of these materials to replace or partially substitute traditional cement-based mortars in infrastructure development, precast concrete products, and the repair and rehabilitation of existing structures.

5) To contribute to the advancement of sustainable construction practices by demonstrating the viability of geopolymers as an alternative to OPC. The research aims to provide evidence that geopolymers can offer comparable or superior performance while significantly reducing the environmental impact of construction activities.

1.4 Scope of the Research

This research addresses the growing demand for sustainable construction materials by investigating the development of geopolymer mortars as an alternative to conventional Ordinary Portland Cement (OPC) binders. The study focuses on the valorization of industrial by-products, systematically evaluating the performance of a geopolymer system synthesized from fly ash (FA), a primary aluminosilicate precursor, and calcium carbonate sludge (CCS), a secondary additive. The core of this investigation is a comprehensive parametric study designed to elucidate the influence of key mix design variables on the material's behavior. The experimental program meticulously examines the effects of varying the FA-to-CCS replacement level to understand the synergistic interactions between the precursors. Concurrently, the composition of the alkaline activator solution is systematically adjusted by varying the sodium hydroxide (NaOH) molarity at 5 M, 10 M, and 15 M, as well as the mass ratio of sodium silicate (Na_2SiO_3) to the NaOH solution.

Performance assessment will encompass the characterization of both fresh properties, primarily setting time, and the development of hardened properties, particularly the compressive strength at various curing ages. The ultimate aim is to establish robust correlations between the mix proportions and the resultant engineering properties. This will provide a foundational understanding of the governing mechanisms and deliver the technical data necessary to assess the viability of FA-CCS based geopolymers for specific construction applications, including precast elements and materials for structural repair and rehabilitation.

CHAPTER II

LITERATURE REVIEWS

2.1 Introduction

This chapter reviews the relevant literature to establish the scientific foundation for this thesis. The review is organized to cover several key areas. First, it explores the use of industrial by-products—specifically fly ash and calcium carbonate sludge as precursor materials for the synthesis of geopolymer mortars. This section analyzes how these materials influence the mortar's mechanical strength and durability. Following this, the chapter examines the critical role of alkaline activator solutions in driving the geopolymerization process and determining the final properties of the mortar. This entire discussion is framed within the broader context of sustainable construction materials. Finally, the review concludes by synthesizing the current research to identify a distinct knowledge gap, which provides the central motivation and justification for this study.

2.2 A Review of Fly Ash and Calcium Carbonate Sludge in Geopolymer Mortars: Impact on Strength and Durability

The demand for sustainable construction materials has driven the exploration of alternative cementitious binders. Geopolymer mortars are promising in this respect, particularly when industrial waste by-products, such as fly ash and calcium carbonate sludge (CCS), are used as partial replacements for traditional materials. This literature review evaluates the use of fly ash and CCS in geopolymer mortars, with a focus on their impact on strength and durability. Fly ash, a by-product of coal combustion, is rich in silica and alumina and is commonly used as a precursor in geopolymer formulations. It is particularly valued for its potential to improve both the mechanical properties and durability of geopolymer mortars. The high pozzolanic activity of fly ash leads to the formation of strong alumina-silicate bonds when activated with alkaline solutions, such as sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). For instance, a study by Mohana and Bharathi (2022) demonstrated that pre-treated fly ash geopolymer mortars could reach significant compressive strengths and resist chloride ion penetration, which is crucial for durability in marine environments. Additionally, Ozcelikci et al. (2023) found that geopolymer mortars with a blend of fly ash and slag exhibited enhanced compressive strength and durability, especially under thermal cycling conditions. Calcium carbonate sludge, a by-product of sugarcane processing, has been increasingly explored as an additive in geopolymer mortars. While less reactive than fly ash, CCS contributes to improved workability and may enhance certain mechanical properties. However, it has been observed that adding $CaCO_3$ can decrease the overall compressive strength when used in large proportions.

Lekshmi et al. (2022) explored the durability of geopolymer mortars incorporating both fly ash and CCS and found that while $CaCO_3$ increased the setting time, it marginally reduced compressive strength. However, the presence of calcium compounds like $CaCO_3$ in the matrix improved the resistance to sulfate attack, thus enhancing durability in aggressive environments. Another study indicated that nano-calcium carbonate could improve the microstructure of geopolymer mortars, leading to increased strength and reduced porosity. Combining fly ash and CCS in geopolymer mortars has been shown to yield both positive and negative effects on strength and durability. This mixture also exhibited improved workability, which is beneficial for practical construction applications. Additionally, Ozcelikci et al. (2023) reported that the inclusion of calcium carbonate in geopolymer mortars positively impacted the long-term durability, particularly in terms of resistance to efflorescence, a common issue in fly ash-based mortars.

However, the addition of higher proportions of CCS can negatively impact the overall compressive strength of the geopolymer mortars. For example, an experimental study found that while $CaCO_3$ improves thermal resistance and workability, it can lower the strength properties of mortars when used at levels exceeding 30% by weight. Durability is a critical factor in the application of geopolymer mortars. Fly ash-based geopolymers have demonstrated excellent durability properties, particularly in environments where resistance to chemical attacks is crucial. The high alumino-silicate content in fly ash forms a dense matrix that resists the ingress of harmful chemicals like chlorides and sulfates, which can degrade conventional Portland cement-based materials. A study by Qaidi et al. (2022) emphasized that incorporating calcium carbonate can help mitigate efflorescence issues, further enhancing the durability of fly ash-based geopolymer mortars.

The use of industrial by-products like fly ash and CCS not only improves the performance of geopolymer mortars but also contributes significantly to sustainability in construction. Geopolymer mortars reduce the environmental impact of traditional cement production by utilizing waste materials that would otherwise contribute to landfill or pollution. This aligns with global efforts to reduce CO_2 emissions and promote sustainable development. The combination of fly ash and calcium carbonate sludge in geopolymer mortars offers significant benefits for strength and durability, though careful consideration of mixed proportions is required. Fly ash contributes to high compressive strength and chemical resistance, while calcium carbonate improves workability and enhances certain durability aspects like sulfate resistance. Overall, the use of these materials in geopolymer mortars aligns with sustainability goals, providing a low-carbon alternative to conventional cementitious binders.

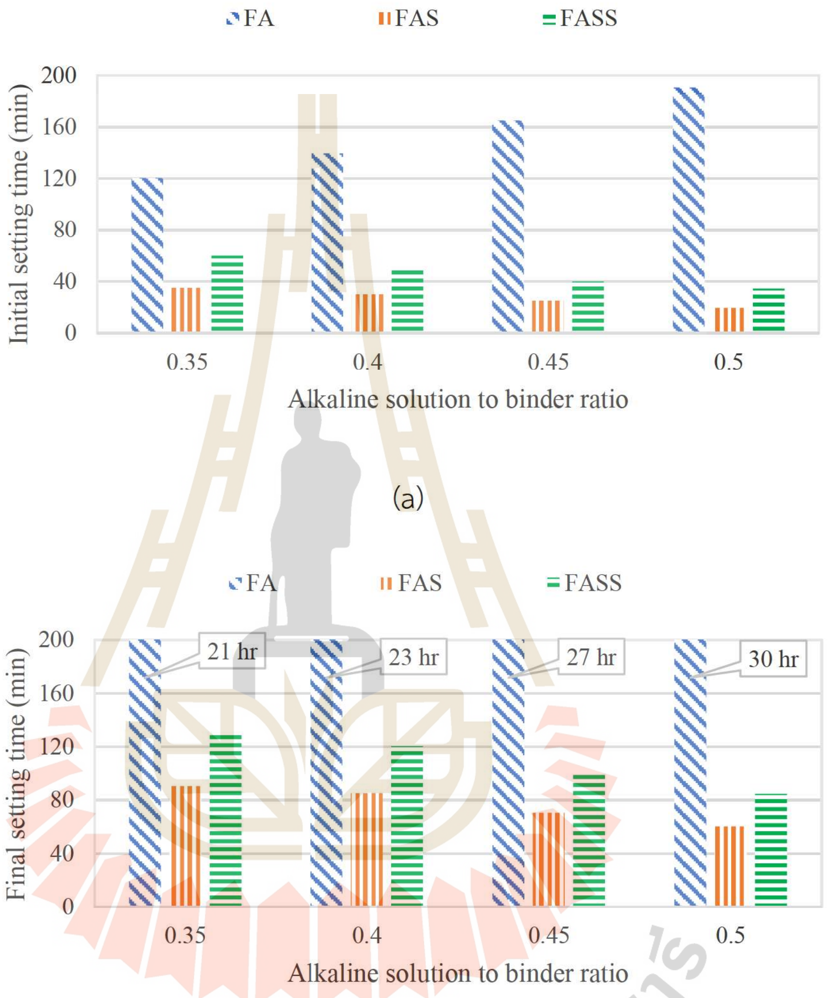
2.3 Previous Studies Exploring Alkaline Solutions' Role in Geopolymer Mortar Performance

Geopolymers are recognized as sustainable, eco-friendly alternatives to traditional Portland cement, contributing to significant reductions in carbon emissions. The use of geopolymer mortars, which utilize industrial by-products such as fly ash and calcium carbonate sludge, has gained attention for its potential to mitigate environmental degradation while offering robust mechanical properties, including high compressive strength and chemical resistance. A crucial factor in the performance of geopolymer mortars is the composition and concentration of alkaline solutions, which act as activators to enhance the dissolution and polymerization of aluminosilicate materials. This literature review explores previous studies on the role of different alkaline solutions, particularly sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3), in the synthesis and performance of geopolymer mortars.

NaOH is a commonly used activator in geopolymer systems due to its effectiveness in dissolving aluminosilicate sources such as fly ash. Several studies have demonstrated that increasing the molarity of NaOH enhances the compressive strength of geopolymer mortars. For example, an experimental study conducted by Palomo et al. (1999) showed that higher NaOH concentrations (up to 15M) significantly improved the mechanical performance of fly ash-based geopolymer mortars. Similarly, research by Hardjito et al. (2005) found that the optimum molarity of NaOH in geopolymer concrete is between 10M and 15M, depending on the specific mix proportions.

The addition of sodium silicate, commonly referred to as water glass, further boosts the polymerization process in geopolymer systems. The combination of NaOH and Na_2SiO_3 is particularly effective in creating a robust geopolymer matrix. Duxson, Provis, et al. (2007) reported that the ratio of Na_2SiO_3 to NaOH significantly impacts the compressive strength, workability, and setting time of geopolymer mortars. Ratios between 0.5:1 to 2:1 Na_2SiO_3 to NaOH have been explored, with results indicating that a balanced ratio optimizes both mechanical performance and setting time. **Figure 2.1** shows the effect of sodium hydroxide solution molarity on the setting time of geopolymer mortars.

The molarity of NaOH not only influences the compressive strength but also significantly affects the setting time of geopolymer mortars. Research by Samarakoon et al. (2019) observed that lower molarity NaOH solutions result in slower setting times, whereas higher molarity solutions accelerate the setting process, which is desirable in certain construction applications. However, the balance between compressive strength and workability must be carefully managed, as high NaOH molarity may result in less workable mixes.



(a)

(b)

Figure 2.1 Effect of sodium hydroxide solution molarity on setting time of geopolymer mortar with various binders at ambient temperature and A/B of 0.35 (a) initial setting time (b) final setting time (Elyamany et al., 2018).

Figure 2.2 shows the impact of sodium hydroxide molarity on 7-day compressive strength. The use of fly ash as the primary aluminosilicate source in geopolymer mortars is well-documented. **Figure 2.3** illustrates the effect of varying NaOH to Na_2SiO_3 ratios on the 7-day compressive strength of geopolymer mortars. However, the integration of calcium carbonate sludge (CCS), a by-product of the sugar industry, presents a novel approach to enhancing the sustainability of geopolymer mortars. Studies by Davidovits (2008) and Rattanasak and Chindaprasirt (2009) suggest that the addition of CCS can influence the setting time, density, and compressive strength of geopolymer mortars.

The performance of geopolymer mortars is highly dependent on the composition and concentration of alkaline activators. Sodium hydroxide and sodium silicate, in various combinations, play critical roles in determining the mechanical properties, setting time, and durability of geopolymer mortars. While higher NaOH molarity generally enhances compressive strength, the addition of sodium silicate improves workability and accelerates the polymerization process. Future research should continue to explore the optimal ratios of these alkaline solutions in combination with supplementary materials like calcium carbonate sludge to further enhance the sustainability and performance of geopolymer mortars.

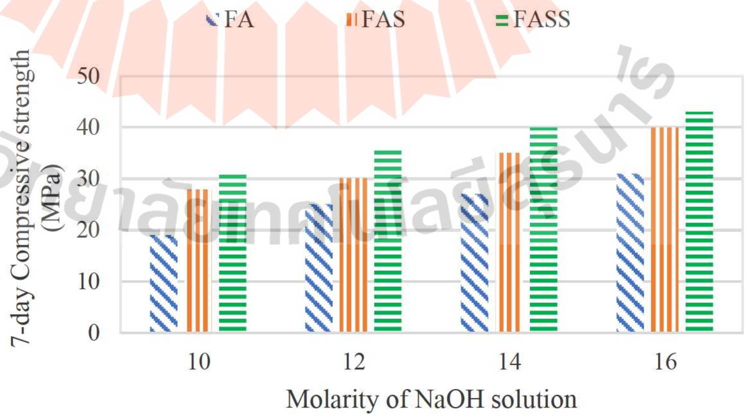


Figure 2.2 Effect of sodium hydroxide solution molarity on 7- day compressive strength of geopolymer mortar with various binders at curing temperature of 60 °C and A/B of 0.35 (Elyamany et al., 2018).

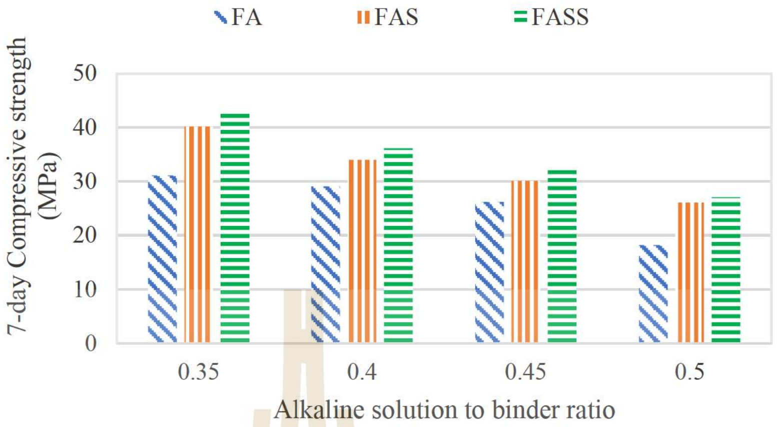


Figure 2.3 Effect of alkaline solution to binder ratio on 7- day compressive strength of geopolymer mortar with various binders at curing temperature of 60 C and NaOH molarity of 16 M (Elyamany et al., 2018).

2.4 Sustainable Alternatives in Construction: A Review of Geopolymer Mortars Using Industrial By-Products

The environmental impact of traditional construction materials, particularly ordinary Portland cement (OPC), has led to an urgent need for sustainable alternatives. Geopolymer mortars, a class of inorganic polymers, have emerged as a promising solution due to their lower carbon footprint, high mechanical performance, and utilization of industrial by-products. These materials are synthesized by activating aluminosilicate materials such as fly ash and ground granulated blast furnace slag (GGBFS) with alkaline solutions, offering enhanced durability and resistance to aggressive environments. This literature review explores the use of industrial by-products, including fly ash and calcium carbonate sludge, in the production of geopolymer mortars, evaluating their mechanical properties, environmental benefits, and potential for large-scale application. Geopolymers have garnered attention due to their potential to replace conventional cement-based materials, which are responsible for approximately 8% of global CO_2 emissions. By utilizing industrial by-products, geopolymers provide a low-energy and low-emission alternative, aligning with global sustainability goals. Davidovits (2008) pioneered the concept of geopolymers, emphasizing their ability to form stable, inorganic polymeric networks through the activation of aluminosilicate precursors with alkaline solutions such as sodium hydroxide and sodium silicate.

Fly ash, a waste product of coal combustion in thermal power plants, is one of the most used industrial by-products in geopolymer mortar synthesis. Studies have shown that fly ash-based geopolymer mortars exhibit excellent compressive strength, low shrinkage, and resistance to chemical attack. According to Hardjito et al. (2005), fly ash-based geopolymer concrete can achieve compressive strengths exceeding 40 MPa, comparable to traditional OPC concrete, with the added benefit of reducing greenhouse gas emissions by up to 80%.

The primary environmental advantage of geopolymer mortars lies in their ability to significantly reduce CO_2 emissions compared to traditional cement-based materials. A life cycle analysis conducted by Habert et al. (2011) found that geopolymers emit up to 80% less CO_2 than Portland cement, largely due to the avoidance of calcination, which is a major contributor to greenhouse gas emissions in cement production. Fly ash and other industrial by-products used in geopolymer mortars are typically waste materials that would otherwise contribute to environmental pollution. By utilizing these by-products, geopolymer mortars not only reduce carbon footprints but also address the issue of industrial waste disposal. Geopolymer mortars contribute to resource efficiency by replacing conventional cement with industrial waste materials. This reduces the reliance on virgin raw materials such as limestone and clay, which are heavily mined for cement production. Moreover, the lower energy demand for the synthesis of geopolymers compared to the energy-intensive clinker production process in Portland cement further underscores their sustainability.

Several studies have demonstrated that geopolymer mortars can achieve compressive strengths comparable to or even higher than conventional cement mortars. Research by Palomo et al. (1999) and Nath and Sarker (2014) revealed that the compressive strength of fly ash-based geopolymers could range from 30 MPa to 50 MPa, depending on the type and concentration of the alkaline activator used. The addition of GGBFS or CCS can further enhance the compressive strength, making geopolymer mortars suitable for structural applications. Geopolymer mortars have demonstrated excellent resistance to chemical attack, particularly in aggressive environments such as marine and industrial settings. Studies by T Bakharev (2005) and Fernández-Jiménez et al. (2005) show that geopolymer mortars exhibit superior resistance to sulfate attack, acid exposure, and chloride penetration compared to traditional cement mortars as shown in **Figure 2.4-2.6**. The low permeability of geopolymer mortars, combined with their dense microstructure, makes them ideal for applications where durability is critical. Furthermore, their resistance to high temperatures and fire makes them suitable for fireproofing materials and applications in high temperature environments.

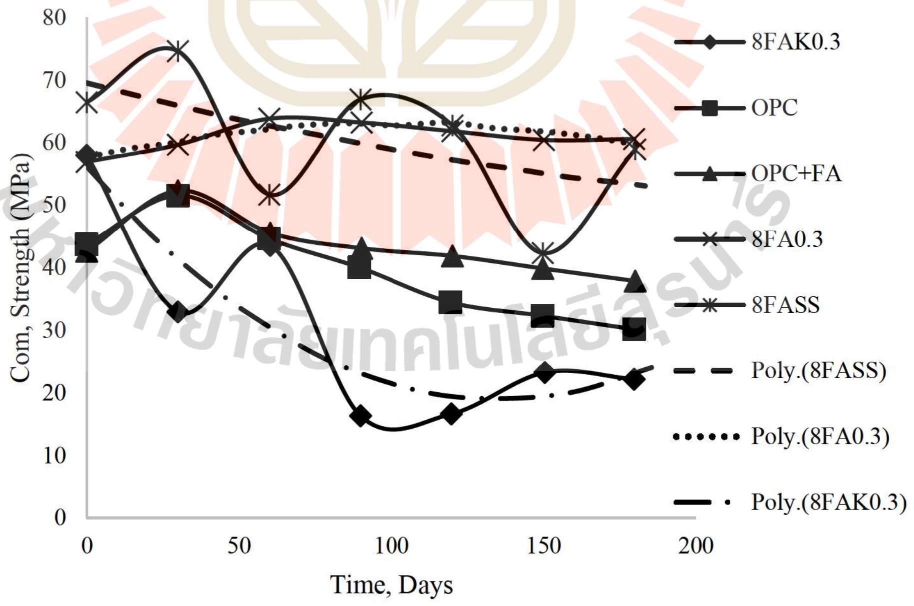


Figure 2.4 Compressive strength evolution of the geopolymer and Portland cement specimens exposed to 5% sodium sulfate solution (T Bakharev, 2005).

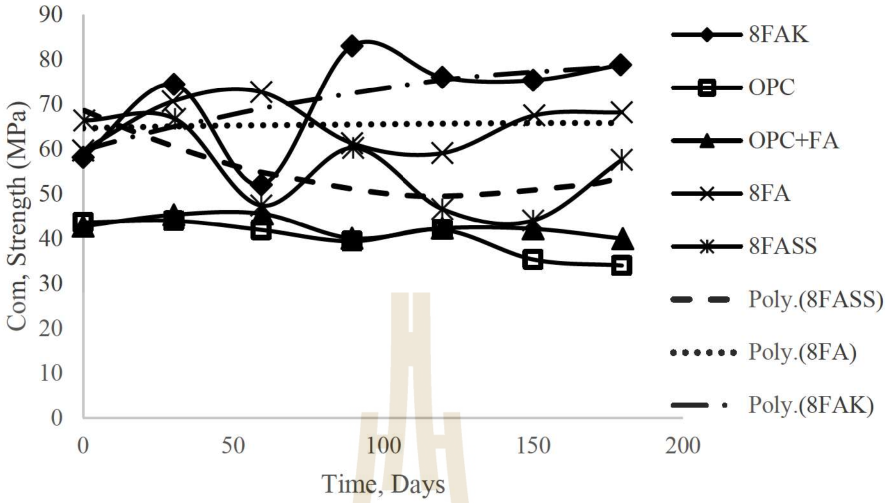


Figure 2.5 Compressive strength evolution of the geopolymer and Portland cement specimens exposed to 5% magnesium sulfate solution (T Bakharev, 2005).

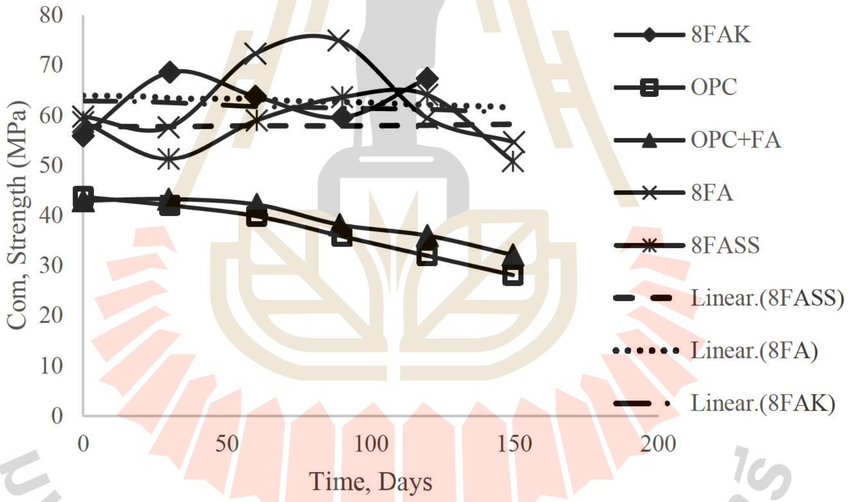


Figure 2.6 Compressive strength evolution of the geopolymer and Portland cement specimens exposed to a solution of 5% sodium sulfate+5% magnesium sulfate (T Bakharev, 2005).

Geopolymer mortars provide a sustainable alternative to conventional cement-based materials by utilizing industrial by-products such as fly ash, GGBFS, and CCS. These materials not only reduce CO_2 emissions but also address waste management issues by repurposing industrial waste. Geopolymers exhibit excellent mechanical properties, chemical resistance, and durability, making them viable for various construction applications. As research continues to optimize their mix proportions and activator compositions, geopolymer mortars are poised to play a significant role in the future of sustainable construction.

2.5 Identifying Gaps in Geopolymer Mortar Research

Geopolymer mortars have garnered significant attention as eco-friendly alternatives to traditional Portland cement mortars. The utilization of industrial by-products such as fly ash, calcium carbonate sludge (CCS), and other aluminosilicate materials for geopolymer production offers environmental benefits, including reduced carbon emissions and improved waste management. However, despite the numerous studies on geopolymer mortars, there remain several gaps in the existing research, particularly in the areas of material optimization, long-term durability, and the feasibility of large-scale adoption. This literature review identifies key gaps in the current research and suggests areas for further exploration.

While significant research has been conducted on using fly ash, ground granulated blast furnace slag (GGBFS), and other industrial by-products in geopolymer mortars, the role of calcium carbonate sludge (CCS) remains underexplored. CCS is a by-product of the sugar industry and has the potential to enhance the sustainability of geopolymers by reducing the demand for conventional materials and mitigating environmental issues related to its disposal. However, there is limited research on the optimal mix proportions and the long-term performance of geopolymer mortars that incorporate CCS. Existing studies, such as those by Rattanasak and Chindaprasirt (2009), have demonstrated that the addition of CCS can improve the workability of geopolymer mortars. However, the specific impact of CCS on the setting time, compressive strength, and durability of geopolymer mortars in various environmental conditions is not well-documented. Further research is needed to fully understand the role of CCS as a sustainable alternative and its potential to replace or supplement other aluminosilicate materials.

Geopolymer synthesis relies heavily on the activation of aluminosilicate materials with alkaline solutions, typically sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). Although various studies have explored the influence of alkaline activators on the performance of geopolymer mortars, there is no clear consensus on the optimal ratios of NaOH and Na_2SiO_3 for different by-products, particularly when new materials like CCS are introduced. For instance, the current research proposal investigates various NaOH-to- Na_2SiO_3 ratios in geopolymer mortars made from fly ash and CCS. However, there is a lack of standardized guidelines on how to determine these ratios for the best mechanical performance, workability, and durability. Studies such as those by Duxson, Provis, et al. (2007) have indicated that the ratios of alkaline solutions play a critical role in the polymerization process, but further research is required to develop tailored solutions for different industrial by-products.

Curing conditions, particularly temperature and humidity, have a significant influence on the performance of geopolymer mortars. Many studies focus on curing geopolymers at elevated temperatures (60°C to 80°C) to enhance early strength development. However, the feasibility of large-scale applications where ambient temperature curing is the norm remains a critical research gap. For example, research by Bernal et al. (2011) indicated that ambient temperature curing results in lower compressive strengths for geopolymer mortars compared to those cured at elevated temperatures. Moreover, the variability in curing practices across different studies has led to inconsistent results, making it difficult to compare performance data across studies. More standardized research on ambient temperature curing, particularly in real-world environments, is necessary to ensure the widespread adoption of geopolymer mortars in construction.

While short-term mechanical properties such as compressive strength are well-documented in geopolymer mortar research, there is a dearth of studies on the long-term durability of these materials. Factors such as freeze-thaw cycles, sulfate attack, carbonation, and chloride ion penetration can significantly affect the longevity of geopolymer mortars in different environments. For instance, research by Duxson, Provis, et al. (2007) highlighted the importance of durability studies but acknowledged that long-term testing under real environmental conditions is still lacking. There is little information available on how geopolymer mortars incorporating CCS behave over extended periods when exposed to aggressive environments such as marine or wastewater conditions. Long-term studies are essential to ensure that geopolymer mortars can offer the same level of durability as traditional OPC mortars.

Although geopolymer mortars have demonstrated significant promise in laboratory settings, there are considerable challenges associated with scaling up these materials for practical use in large-scale construction projects. Issues such as the availability of raw materials (e.g., fly ash and GGBFS), consistency in material quality, and the economic feasibility of producing alkaline activators on a large scale have not been sufficiently addressed in the literature. Furthermore, the development of standardized manufacturing processes and quality control measures for geopolymer mortars is crucial for their adoption by the construction industry. Currently, the lack of established regulations and industry standards is a significant barrier to the widespread use of geopolymer mortars. Addressing these issues through large-scale pilot projects and collaboration with industry stakeholders will be necessary for the successful implementation of geopolymer technologies in the construction sector.

While geopolymer mortars have been extensively studied as sustainable alternatives to traditional cement-based materials, several gaps remain in the research. The role of CCS in geopolymer mortars, optimal alkaline solution ratios, curing conditions, long-term durability, and large-scale implementation challenges are areas that require further investigation. Addressing these gaps will be critical for advancing geopolymer technology and ensuring its practical adoption in the construction industry.

CHAPTER III

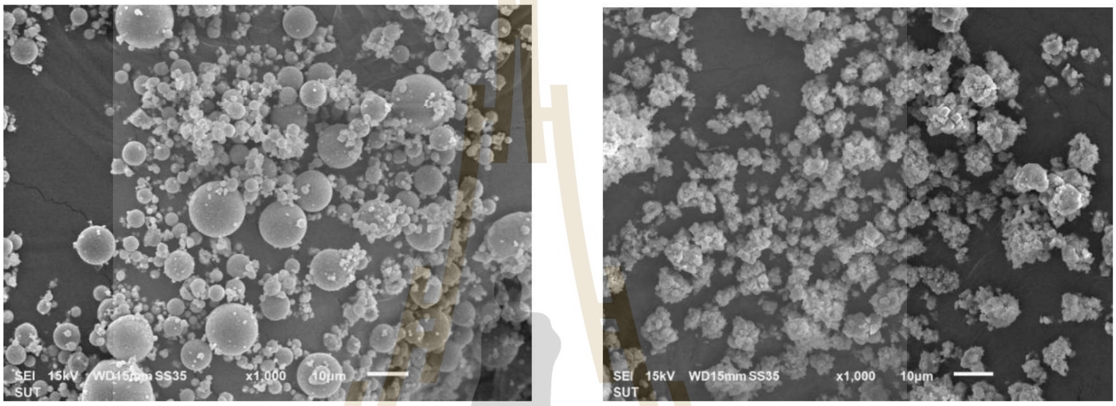
RESERCH METHODOLOGY

3.1 Materials

3.1.1 Precursor Materials

This study investigated calcium carbonate sludge (CCS) as a potential alternative precursor to partially replace conventional fly ash (FA) in geopolymer systems. The FA used in this study was obtained from Mae Moh Power Plant, Lampang Province, Thailand, which is a lignite coal-fired power plant. The FA was collected from electrostatic precipitators and classified as Class C fly ash according to ASTM C618 due to its high calcium content (CaO: 28.72%). The CCS was collected as a by-product from a sugar refinery plant in Nakhon Ratchasima, Thailand. This CCS was generated during the sugarcane juice clarification process, where lime ($Ca(OH)_2$) was added to remove impurities through precipitation. The CCS was air-dried for 3 days, followed by oven-drying at 105°C for 24 hours to remove residual moisture. The morphological characteristics of both materials were examined using scanning electron microscopy (SEM) as shown in **Figure 3.1**. FA displayed the smooth and spherical particle characteristic typical of coal combustion by-products (**Figure 3.1a**); a morphology that typically facilities reactivity due to high surface area and enhanced dissolution kinetics. In contrast, CCS exhibited irregular and agglomerated particles with rough surface textures (**Figure 3.1b**), reflecting its origin as a precipitated waste material from sugar processing.

X-ray diffraction (XRD) analysis provided critical insights into the crystalline composition of both precursors. The FA pattern (**Figure 3.2a**) revealed primary crystalline phases of quartz, mullite, and hematite, with a pronounced amorphous hump between 20° and 30° at 2θ, indicating substantial reactive aluminosilicate content essential for geopolymerization. The CCS diffractogram (**Figure 3.2b**) showed strong peaks for calcium oxide with minor quartz and alumina phase, indicating its potential role as a calcium-enriching additive rather than a direct aluminosilicate source.

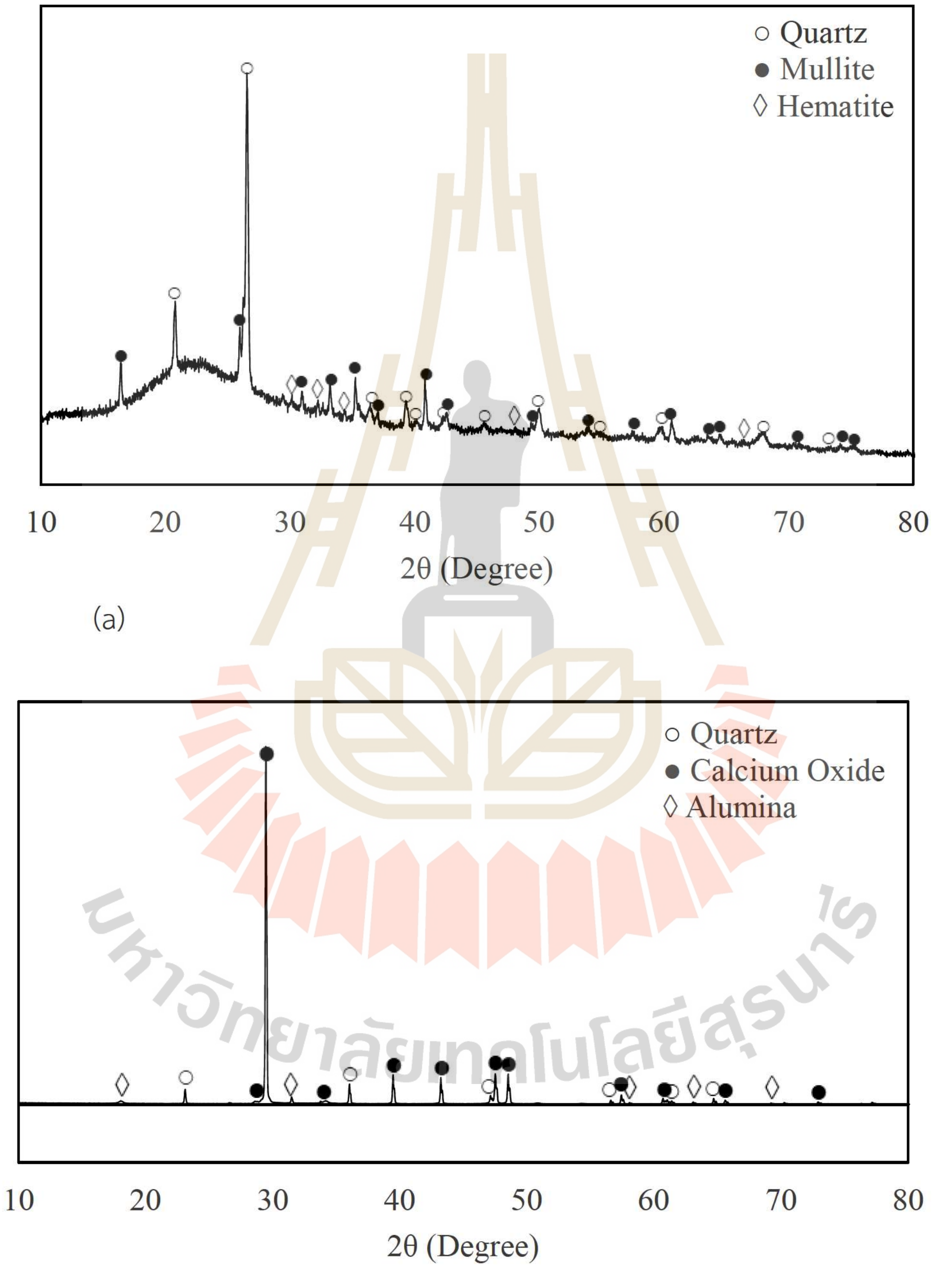


(a) (b)

Figure 3.1 SEM image of (a) FA and (b) CCS.

Chemical composition analysis (**Table 3.1**) quantified the fundamental differences between these materials. FA contained significant proportions of silicon dioxide (29.31%) and aluminum oxide (11.01%), which are the primary network-forming components in geopolymer systems. The notable calcium oxide content (28.72%) classified this as a high-calcium fly ash, potentially contributing to additional reaction pathways. CCS demonstrated an exceptionally high calcium oxide concentration (94.64%) with minimal silica (3.11%) and alumina (1.36%), indicating its potential to modify reaction kinetics and final product characteristics when used as a partial replacement for FA.

Particle size distribution analysis (**Figure 3.3**) revealed that CCS possessed a finer overall particle distribution than FA, with 100% of CCS particles passing through approximately 100 μm compared to 300 μm for FA. This comparative fineness of CCS indicated potential benefits for matrix densification and enhanced reactivity when incorporated into geopolymer systems, potentially offsetting some limitations of its chemical composition when used as a partial substitute for FA.



(a)

(b)

Figure 3.2 X-ray diffraction patterns of (a) FA and (b) CCS.

Table 3.1 Chemical composition of fly ash and calcium carbonate sludge.

Chemical Composition	Fly ash (%)	CCS (%)
Silicon dioxide (SiO_2)	29.31	3.11
Iron oxide (Fe_2O_3)	14.87	0.28
Aluminum oxide (Al_2O_3)	11.01	1.36
Calcium oxide (CaO)	28.72	94.64
Magnesium oxide (MgO)	3.42	-
Sulfur trioxide (SO_3)	10.48	0.79

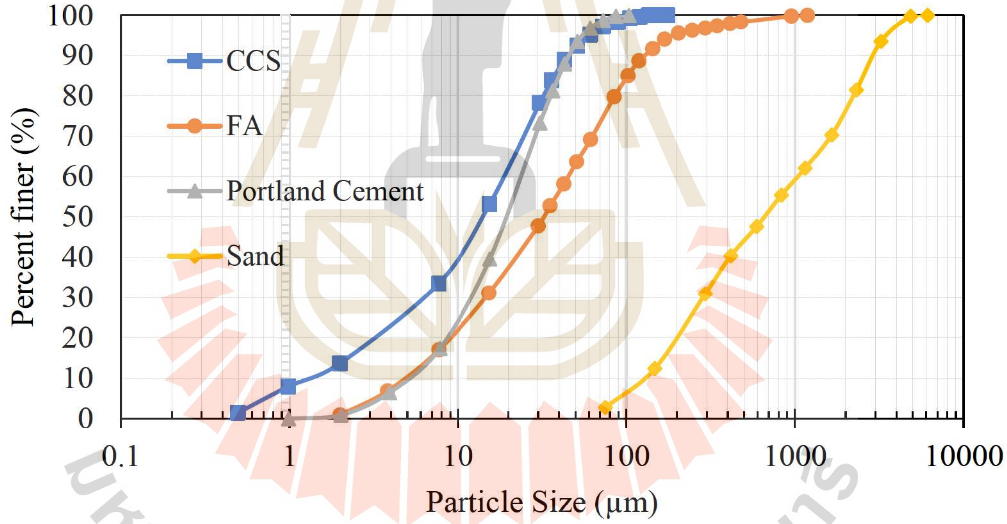


Figure 3.3 Grain size distribution of CCS, FA, Portland Cement, and Sand.

3.1.2 Fine Aggregate

Standardized fine aggregate (river sand) was employed across all mixtures to isolate the effects of precursor substation. The aggregate properties (**Table 3.2**) included a saturated surface dry specific gravity of 2.65, dry specific gravity of 2.60, dry unit density of 1604 kg/m3, and a fineness modulus of 2.7. **Figure 3.3** shows the grain size distribution of sand.

Table 3.2 Basic physical and engineering properties of fine aggregate.

Properties	Value
Saturated surface dry specific gravity	2.65
Dry specific gravity	2.6
Percentage of voids (%)	38.2
Dry unit density (kg.m^{-3})	1604
Absorption (%)	0.74
Moisture content (%)	2.46
Fineness modulus	2.7

3.1.3 Alkaline Activators

The alkaline activation system comprised sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solutions. Sodium hydroxide of analytical grade (98% purity) was dissolved in distilled water to prepare solutions of three different molarities: 5M, 10M, and 15M. These solutions were prepared 24 hours prior to use to allow for complete dissolution and cooling to ambient temperature.

Commercial-grade sodium silicate solution with a density of 1.45 g/cm³ was used as the silicate source in the activator system. The sodium silicate possessed a modulus ratio (SiO_2/Na_2O) of 2.5, with chemical composition comprising 30.1% SiO_2, 12.0% Na_2O, and 57.9% H_2O by weight. The viscosity of the solution measured 400-600 mPa·s at 20°C, providing appropriate flow characteristics for mixing while ensuring adequate silica availability for geopolymer network formation.

These activator solutions were systematically combined in ten different sodium silicate-to-sodium hydroxide ratios, ranging from 0:100 to 90:10 SS:SH (**Table 3**). This variation enabled precise control of both the overall alkalinity (pH) and the dissolved silica content of the activation solution, two critical factors influencing geopolymerization mechanisms, particularly when investigating precursors with different chemical compositions.

3.1.4 Mix Design

The experimental program was designed to systematically evaluate CCS as a partial replacement for FA across a comprehensive range of activation parameters. As detailed in **Table 3.3**, the study included: (1) Control cement mortars at three liquid-to-binder (l/b) ratios (0.3, 0.4, 0.5) to establish baseline performance metrics. (2) Reference FA-based geopolymer mortars with no CCS substitution, tested across ten sodium silicate-to-sodium hydroxide ratios (0:100 to 90:10 SS:SH), three NaOH concentrations (5M, 10M, 15M), and three l/b ratios (0.3, 0.4, 0.5). (3) FA+CCS blended geopolymer mortars with three CCS replacement levels (10%, 20%, 30%), systematically evaluated across all activator combinations and l/b ratios. The selected FA:CCS ratios of 100:0, 90:10, 80:20, and 70:30 were chosen to systematically investigate the effect of gradual replacement of aluminosilicate-rich FA with calcium-rich CCS. Prior studies have demonstrated that incorporating moderate levels of calcium-rich additives such as CCS or calcium carbide residue into FA-based geopolymers can promote C-A-S-H gel formation and improve early-age strength and microstructural densification (Hanifa et al., 2025). However, high levels of calcium can interfere with N-A-S-H gel formation. Therefore, this study limited the CCS content to a maximum of 30% to avoid excessive dilution of reactive aluminosilicates while enabling hybrid gel formation. The l/b ratios were selected based on previous literature (Wang et al., 2023), which suggests that geopolymer mortars typically achieve optimal workability and strength within this range.

3.1.5 Sample Preparation

Sample preparation followed a rigorous protocol designed to ensure consistency across all test specimens. Precursor materials (FA and CCS) were first proportioned by weight according to the designated replacement levels and thoroughly blended with the standardized fine aggregate in a laboratory mixer at low speed for 2 minutes to achieve homogeneous distribution.

Table 3.3 Mix proportion of geopolymer mortars.

Mix Ingredients	Mix ID	Na_2SiO_3 /NaOH	NaOH (M)	l/b	Fly Ash (FA)	Calcium carbonate sludge (CCS)	Sand (g)
Cement Mortar	Control	-	-	0.3,0.4,0.5	-	-	400
FA	0SS:100SH	0:100	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	10SS:90SH	10:90	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	20SS:80SH	20:80	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	30SS:70SH	30:70	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	40SS:60SH	40:60	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	50SS:50SH	50:50	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	60SS:40SH	60:40	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	70SS:30SH	70:30	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	80SS:20SH	80:20	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA	90SS:10SH	90:10	5M,10M,15M	0.3,0.4,0.5	100%	-	400
FA+CCS	0SS:100SH	0:100	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	10SS:90SH	10:90	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	20SS:80SH	20:80	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	30SS:70SH	30:70	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	40SS:60SH	40:60	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	50SS:50SH	50:50	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	60SS:40SH	60:40	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	70SS:30SH	70:30	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	80SS:20SH	80:20	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400
FA+CCS	90SS:10SH	90:10	5M,10M,15M	0.3,0.4,0.5	90%,80%,70%	10%,20%,30%	400

The pre-prepared alkaline activator solution, having been formulated 24 hours in advance to ensure thermal equilibrium and complete dissolution, was then gradually introduced into the dry mixture under continuous agitation. Following complete addition of the activator, mixing continued at medium speed for an additional 3 minutes to ensure uniform incorporation of all components and initiation of the geopolymerization reaction. The fresh mortar was then transferred to 50mm cubic molds for compressive strength testing and appropriate configurations for setting time evaluation, with all specimens subjected to vibration compaction to eliminate entrapped air and ensure uniform density throughout. To prevent moisture loss during the critical initial setting period, molds were immediately sealed with impermeable plastic film following casting. The sealed specimens were then subjected to standardized curing conditions optimized for geopolymer formation, with controlled temperature and humidity maintained throughout the maturation period.

3.2 Testing Methods

3.2.1 Setting Time Test

The setting time of geopolymer mortars was determined using the Vicat needle apparatus in accordance with ASTM-C191 . The procedure involves measuring the initial and final setting times to evaluate the effects of varying mix proportions and activator compositions. For each mixture composition, three replicate specimens were tested to ensure reliability and repeatability of results. The reported values represent the mean of three measurements with error bars indicating standard deviation.

3.2.2 Compressive Strength Test

The compressive strength of the mortar specimens was evaluated at 7 days using a universal testing machine following ASTM-C109/C109M . Prior to testing, each specimen was measured for dimensional accuracy and inspected for defects. The compression testing was performed using a calibrated universal testing machine with a maximum capacity of 250 kN. The loading rate was maintained at 1.0 ± 0.2 MPa/s in accordance with the standard. The load was applied until failure occurred, and the maximum load was recorded. For each mixture composition, three replicate specimens were tested, and the average compressive strength was calculated. Statistical variation was assessed through standard deviation calculations, which were represented as error bars in all strength development figures.

3.2.3 Scanning Electron Microscopy (SEM)

SEM analysis was performed to examine the microstructural characteristics of the geopolymer mortars. Samples were sputter-coated with gold to ensure conductivity and minimize charging effects. Imaging was conducted at accelerating voltages of 15 kV under high vacuum conditions, with working distances optimized for each magnification range (1000x for raw materials and 3500x for geopolymer mortars).

3.2.4 X-ray Diffraction (XRD)

Phase identification and crystallinity assessment were conducted via XRD analysis on powdered samples passing the 45 μm sieve. Diffraction patterns were collected over a 2θ range of 10° to 80° with a step size of 0.02° and a counting time of 1 second per step following ASTM-C1365 .

3.2.5 Machine Learning Analysis

The experimental dataset comprising 45 geopolymer mortar mix designs was prepared for machine learning analysis with input features including FA:CCS ratio, SS:SH ratio, NaOH molarity, and l/b ratio. The dataset included mean values from triplicate testing, ensuring that the machine learning models were trained on statistically representative data rather than individual test variations. These four parameters were selected based on their demonstrated influence on geopolymer properties in prior research. All features were normalized using StandardScaler prior to model development to ensure equal weighting during the learning process.

Four machine learning algorithms were systematically evaluated based on their established capabilities for handling complex, non-linear materials science data:

XGBoost, CatBoost, AdaBoost, and Support Vector Machine (SVM). Despite the relatively modest dataset size (n = 45), gradient boosting methods were included due to their demonstrated effectiveness in materials optimization problems with limited samples. Bayesian optimization identified optimal hyperparameters for each algorithm, while 5-fold cross-validation ensured model robustness against overfitting. Models were evaluated using R^2, MSE, and RMSE metrics to comprehensively assess predictive performance.

The highest-performing model underwent SHapley Additive exPlanations (SHAP) analysis to quantify feature influence on strength predictions, providing mechanistic interpretation of compositional effects on geopolymer properties. All implementations utilized Python 3.8 with appropriate machine learning libraries (scikit-learn, XGBoost, CatBoost, SHAP).

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Setting Time

4.1.1 Effect of Liquid-to-Binder Ratio on Setting Characteristics

The initial setting time of geopolymer mortars was significantly influenced by the l/b ratio, exhibiting a systematic pattern across the experimental matrix. As illustrated in **Figures 4.1-4.3**, increasing the l/b ratio from 0.3 to 0.5 consistently extended the setting times across all mixture compositions. This phenomenon can be attributed to the dilution effect, where higher liquid content reduces the effective concentration of reactive species in the system, thereby decelerating the geopolymerization reaction kinetics (Chindaprasirt et al., 2007; Deb et al., 2014).

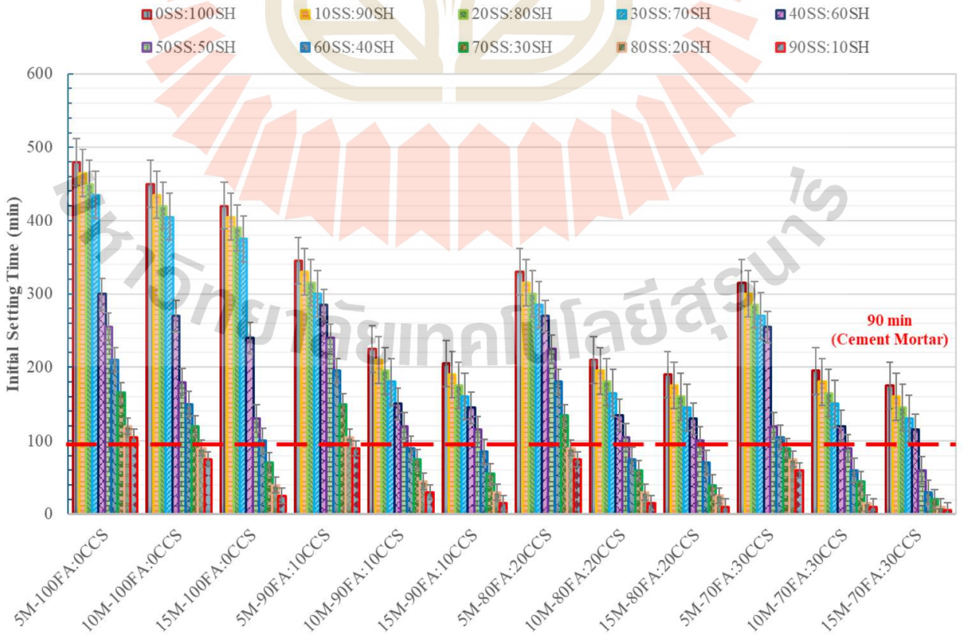


Figure 4.1 Initial setting time of geopolymer mortars with various alkaline solution at l/b=0.3 for different precursors ratios and NaOH concentrations.

At l/b = 0.3 (**Figure 4.1**), the geopolymer mortars demonstrated notably accelerated setting behavior compared to the 90-minute baseline established by the reference cement mortar. This acceleration was particularly pronounced in mixtures with higher sodium silicate-to-sodium hydroxide ratios (SS:SH = 60:40 to 90:10) activated with 15M NaOH concentration. The rapid setting observed under these conditions can be attributed to the enhanced dissolution of aluminosilicate species in the concentrated alkaline environment, facilitating expedited gel formation and subsequent hardening (Fernandez-Jimenez et al., 2006).

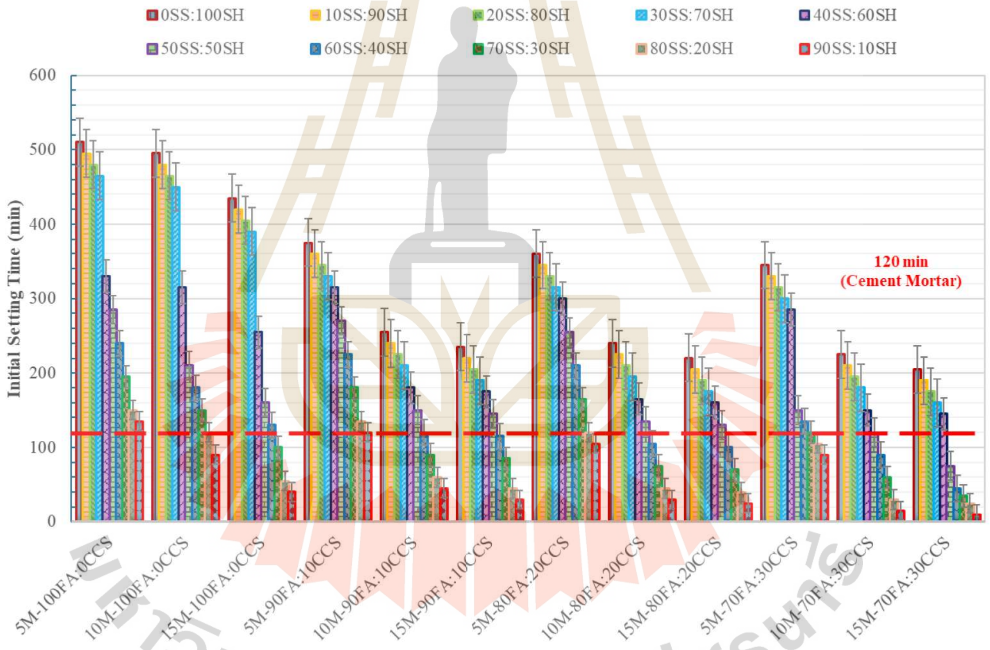


Figure 4.2 Initial setting time of geopolymer mortars with various alkaline solution at l/b=0.4 for different precursors ratios and NaOH concentrations.

As the l/b ratio increased to 0.4 (**Figure 4.2**), a moderate extension of setting times was observed across all mixture compositions, though the general trends regarding the effects of activator composition remained consistent. This intermediate l/b ratio represents an optimal balance between workability and setting characteristics for most practical applications, aligning with findings reported by Li et al. (2019) and Xie et al. (2020) in their investigations of geopolymer systems.

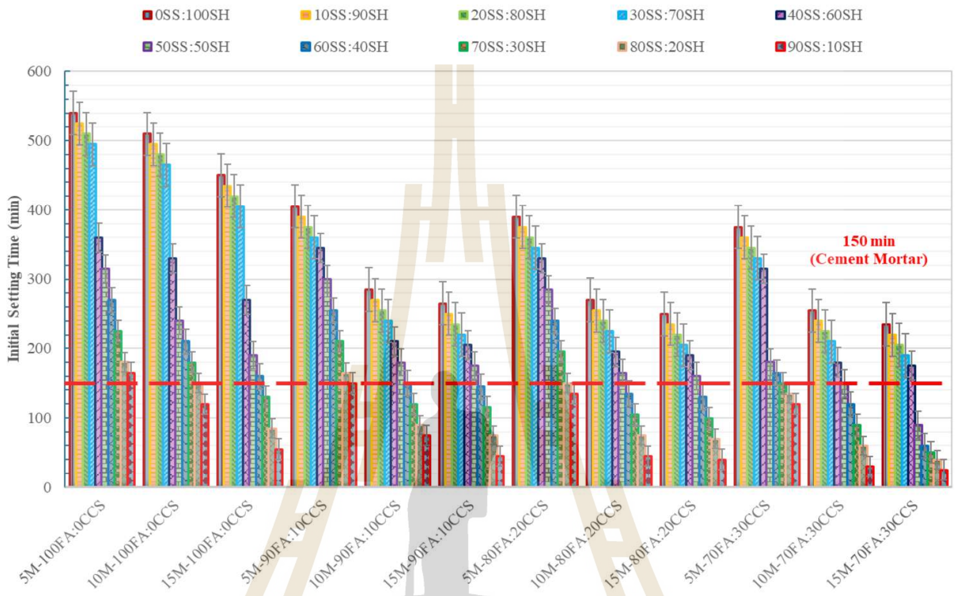


Figure 4.3 Initial setting time of geopolymer mortars with various alkaline solution at l/b=0.5 for different precursors ratios and NaOH concentrations.

Further increasing the l/b ratio to 0.5 (**Figure 4.3**) resulted in substantially prolonged setting times, particularly evident in mixtures with lower NaOH concentrations (5M). This extension of setting time at higher l/b ratios can be attributed to two primary mechanisms: (1) the aforementioned dilution effect reducing reaction kinetics, and (2) the increased spatial separation between reactive particles, necessitating longer diffusion pathways for reaction product formation (Provis, 2018).

4.1.2 Influence of Alkaline Activator Composition on Setting Behavior

The composition of the alkaline activator, particularly the SS:SH ratio and NaOH concentration, exhibited profound effects on the setting characteristics of geopolymer mortars. Higher NaOH concentrations (15M) consistently produced shorter setting times compared to lower molarities (5M and 10M) across all precursor combinations and l/b ratios. This acceleration can be attributed to enhanced dissolution of aluminosilicate species and accelerated geopolymerization in more concentrated alkaline environments (Duxson, Fernández-Jiménez, et al., 2007).

The SS:SH ratio demonstrated a non-linear relationship with setting time, revealing complex interactions within the geopolymerization mechanism. At intermediate SS:SH ratios (40:60 to 70:30), optimum setting times were generally observed, suggesting that a balanced supply of silicate species from the sodium silicate solution and hydroxide ions from NaOH creates favorable conditions for network formation. This observation aligns with the findings of Criado et al. (2007), who identified the critical role of silicate speciation in controlling geopolymerization kinetics.

Interestingly, at very high SS:SH ratios (80:20 to 90:10), a slight retardation in setting was observed, particularly at a lower NaOH concentration (5M). This effect can be attributed to the increased viscosity of the activator solution at high silicate contents, potentially impeding the mobility of reactive species and delaying the formation of three-dimensional networks (Fernández-Jiménez et al., 2005).

4.1.3 Effect of Calcium Carbonate Sludge Incorporation on Setting Dynamics

The incorporation of CCS into the FA system introduced significant modifications to the setting behavior, attributable to the high calcium oxide content (CaO-94.64%) as shown in **Table 2.1**. Across all l/b ratios and activator compositions, increasing the CCS content from 0% to 30% progressively accelerated the setting process, with the effect becoming more pronounced at higher replacement levels.

At 10% CCS replacement, modest reductions in setting time were observed, typically in the range of 10-15% compared to the corresponding 100FA:0CCS formulation. This modest effect suggests that at lower replacement levels, the calcium content remains insufficient to substantially alter the dominant sodium aluminosilicate hydrate (N-A-S-H) gel formation mechanism characteristic of fly ash-based geopolymers (Yip et al., 2008).

As the CCS content increased to 20%, more substantial reductions in setting time became evident, particularly in systems activated with higher NaOH concentrations (15M). This synergistic effect between calcium content and alkaline concentration indicates a cooperative mechanism, where the calcium ions from CCS enhance the dissolution of aluminosilicate species from FA in the highly alkaline environment, promoting accelerated gel formation (Lee & Van Deventer, 2002).

At the maximum CCS replacement level (30%), the most dramatic acceleration of setting was observed, with some formulations achieving initial set in less than 30 minutes when activated with 15M NaOH at l/b = 0.3. This pronounced effect can be attributed to the formation of a sophisticated hybrid binding system containing both N-A-S-H and calcium aluminosilicate hydrate (C-A-S-H) gels. The calcium ions from CCS participate in rapid precipitation reactions, forming calcium-rich phases that accelerate the overall hardening process (Garcia-Lodeiro et al., 2011).

The acceleration effect of CCS was most prominent in systems with lower SS:SH ratios (0:100 to 40:60), suggesting that in these hydroxide-dominated environments, calcium ions can more readily participate in the reaction mechanisms without interference from the complexing effects of silicate species present in sodium silicate solution (Phair & Van Deventer, 2001).

4.1.4 Integration of Setting Time Behavior with Practical Applications

The systematic investigation of setting characteristics across the experimental matrix provides valuable insights for tailoring geopolymer formulations to specific application requirements. For applications demanding rapid setting, such as emergency repairs or precast elements with high production turnover, formulations incorporating 20-30% CCS activated with 15M NaOH at l/b = 0.3 offer optimal performance, with setting times generally under 45 minutes.

Conversely, for applications requiring extended workability, such as large-scale castings or situations with complex formwork, formulations with lower CCS content (0-10%), activated with 5M or 10M NaOH at l/b = 0.5, provide more manageable setting windows exceeding 90 minutes in most cases.

The observed setting time characteristics also correlate with strength development patterns discussed in subsequent sections, where more rapid setting generally corresponds with enhanced early-age strength but may influence ultimate strength development through densification mechanisms and reaction product distribution. This integrated understanding of setting behavior and mechanical performance enables the strategic design of geopolymer formulations optimized for specific construction applications.

4.2 Compressive Strength Development

The 7-day compressive strength results revealed complex relationships between mixture composition, processing parameters, and mechanical performance. Through systematic evaluation of key variables, this study identified critical factors influencing the development of high strength geopolymer mortars.

4.2.1 Effect of NaOH Molarity on Strength Development

The NaOH concentration significantly influenced compressive strength across all mixture compositions, as illustrated in **Figures 4.4-4.6**. Increasing NaOH molarity from 5M to 15M consistently enhanced mechanical performance, with the effect becoming more pronounced in formulations containing both FA and CCS.

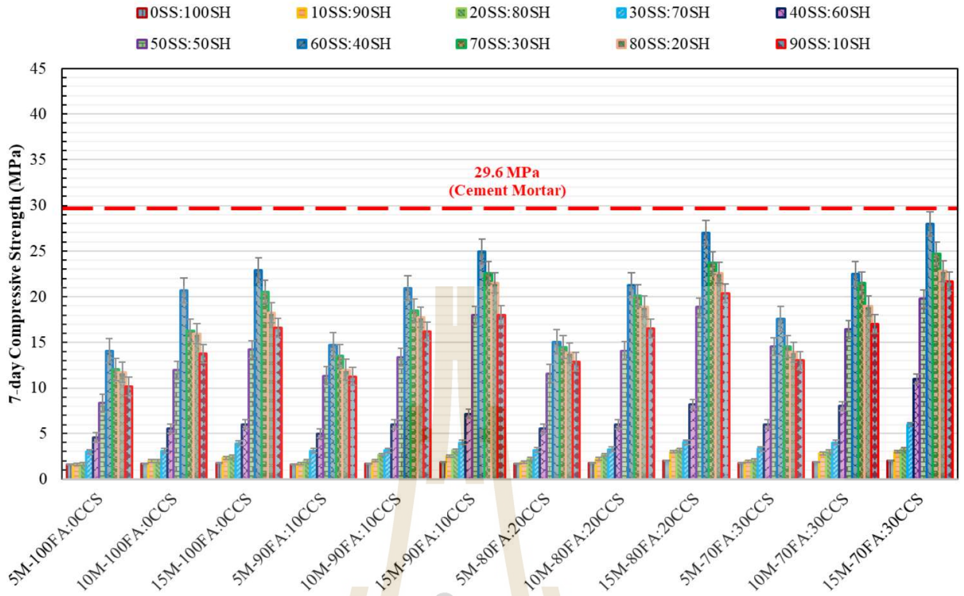


Figure 4.4 7-day compressive strength development of geopolymer mortars with various alkaline solution at l/b=0.3 for different precursors ratios and NaOH concentrations.

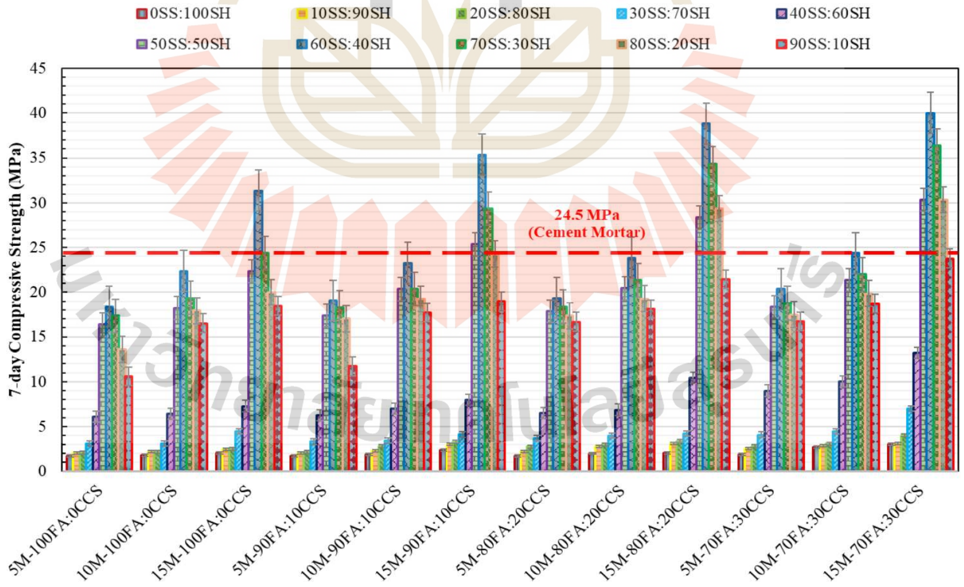


Figure 4.5 7-day compressive strength development of geopolymer mortars with various alkaline solution at l/b=0.4 for different precursors ratios and NaOH concentrations.

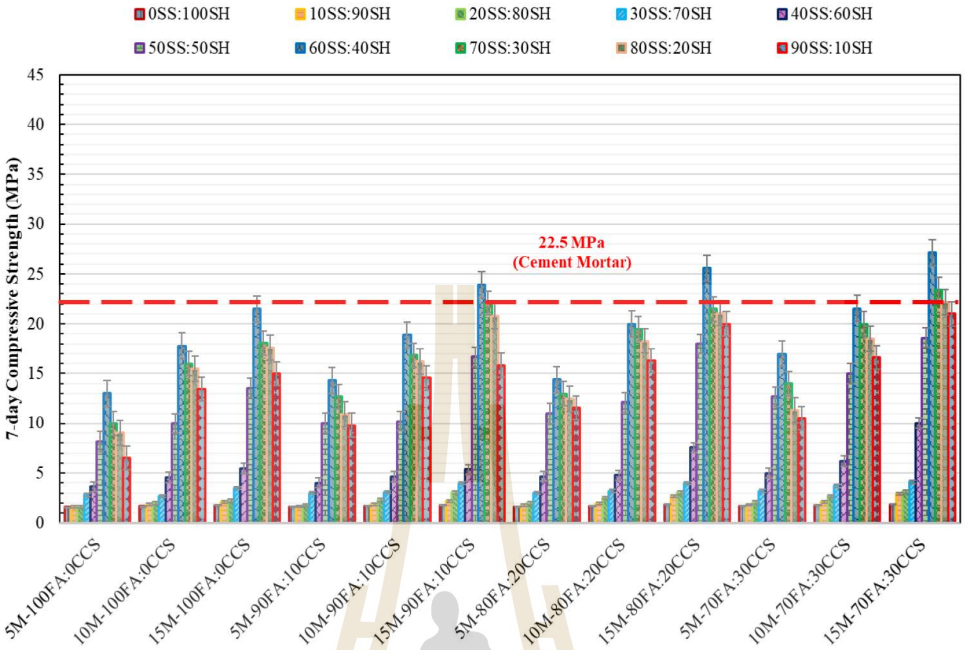


Figure 4.6 7-day compressive strength development of geopolymer mortars with various alkaline solution at l/b=0.5 for different precursors ratios and NaOH concentrations.

At 5M NaOH concentration, most formulations achieved modest strength values ranging from 15-20 MPa, regardless of other mixture parameters. This limited performance can be attributed to insufficient alkalinity for effective dissolution of aluminosilicate species, resulting in incomplete geopolymerization reactions (Kutchko & Kim, 2006). Intermediate NaOH concentration (10M) generated moderate improvements, with typical strength values between 20-25 MPa depending on other formulation variables.

The most substantial strength gains were observed at 15M NaOH concentration, where several formulations exceeded 30 MPa at optimal SS:SH ratios. This enhanced performance stems from comprehensive dissolution of aluminosilicate precursors and accelerated reaction kinetics, leading to the formation of more robust and densely packed geopolymeric networks (Senior & Johnson, 2005). The superior strength at higher NaOH concentrations demonstrates the critical role of alkalinity in maximizing the reaction potential of aluminosilicate precursors.

4.2.2 Influence of Sodium Silicate-to-Sodium Hydroxide Ratio

The SS:SH ratio emerged as a pivotal parameter influencing strength development. Analysis of **Figures 4.4-4.6** reveals that a SS:SH ratio of 60:40 consistently produced the highest compressive strength across all experimental conditions, regardless of NaOH concentration, FA:CCS ratio, or l/b ratio. This consistent pattern identifies 60:40 as the optimal balance between sodium silicate and sodium hydroxide for maximizing strength development in geopolymer mortars (Fernández-Jiménez & Palomo, 2003).

At lower SS:SH ratios (0:100 to 50:50), insufficient soluble silica limited the formation of silica-rich gel networks, resulting in suboptimal mechanical performance. Conversely, at higher SS:SH ratios (70:30 to 90:10), excessive silicate species potentially impeded precursor dissolution through surface coating effects, leading to strength reduction. The 60:40 ratio represents the ideal balance between silicate species and hydroxide ions, creating favorable conditions for network formation and structural densification (Rostami et al., 2012).

The sensitivity to variations from this optimal 60:40 ratio was most pronounced at higher NaOH concentrations (15M) and in formulations incorporating both FA and CCS. This enhanced sensitivity indicates a sophisticated interplay between silicate speciation, hydroxide concentration, and calcium availability in determining the reaction mechanisms and product formation pathways (Van der Merwe et al., 2014).

4.2.3 Effect of Fly Ash and Calcium Carbonate Sludge Proportions

The incorporation of CCS as a partial replacement for FA significantly altered strength development patterns across the experimental matrix. As evident in **Figures 4.4-4.6**, increasing CCS content from 0% to 30% generally enhanced compressive strength, particularly when combined with higher NaOH concentrations and the optimal 60SS:40SH ratio.

The strength enhancement with CCS incorporation can be attributed to the formation of a hybrid binding system containing both sodium aluminosilicate hydrate (N-A-S-H) and calcium aluminosilicate hydrate (C-A-S-H) gels (Environment et al., 2018; Provis, 2018). The calcium-rich environment provided by CCS promotes the formation of C-A-S-H phases, which complements the traditional N-A-S-H networks characteristic of FA-based geopolymers. This dual gel formation mechanism creates a more sophisticated binding system with enhanced mechanical properties (Phair & Van Deventer, 2001).

The optimal CCS replacement level appeared to be 20-30%, depending on the NaOH concentration. At this replacement level, formulations typically demonstrated 15-25% higher strength compared to the corresponding 100FA:0CCS reference mixtures. The synergistic effect between FA and CCS was most pronounced at higher NaOH concentrations (15M), indicating that aggressive alkaline conditions facilitate optimal interaction between the precursors (Barcelo et al., 2014).

4.2.4 Comprehensive Analysis of Liquid-to-Binder Ratio Effects

Figure 4.7 presents a comprehensive analysis of how the liquid-to-binder (l/b) ratio affects compressive strength at the optimal SS:SH ratio of 60:40 across various precursor combinations and NaOH concentrations. The relationship between l/b ratio and compressive strength exhibits a consistent parabolic pattern across all mixture compositions, with peak strength occurring at l/b = 0.4 (Duxson, Provis, et al., 2007; Hardjito et al., 2004).

This optimal l/b ratio represents the ideal balance between sufficient liquid content for complete dissolution and reaction of precursors while maintaining adequate particle proximity for effective network formation. For instance, the 15M-70FA:30CCS mixture increased in strength from approximately 18 MPa at l/b = 0.3 to 40 MPa at l/b = 0.4, followed by a decline to about 27 MPa at l/b = 0.5. This substantial variation (approximately 122% increase from l/b = 0.3 to 0.4) reveals the critical importance of precise l/b ratio control in high-performance geopolymer formulations (Tanya Bakharev, 2005).

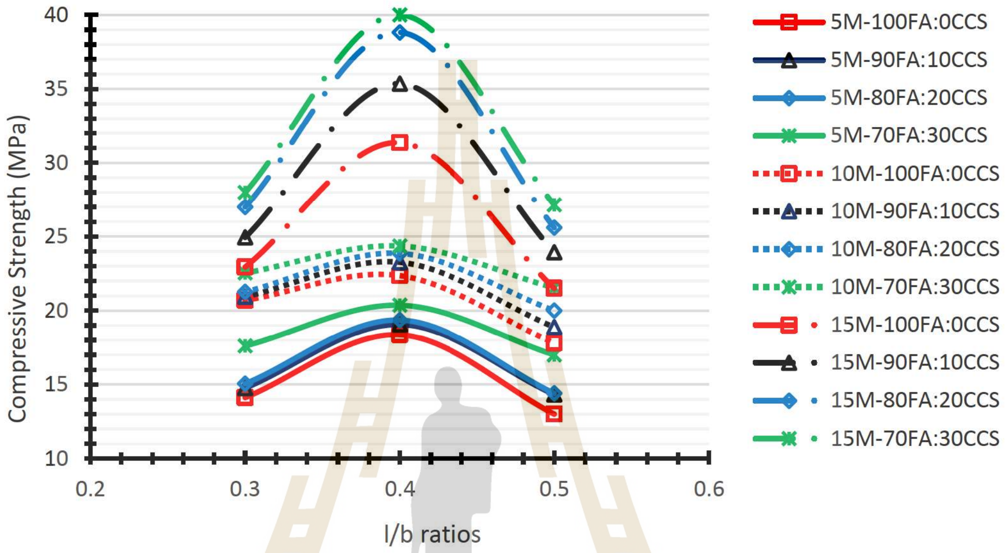


Figure 4.7 Effect of l/b ratio on 7-day compressive strength of geopolymer mortars with 60SS:40SH at different precursors ratios and NaOH concentrations.

The strength reduction observed when increasing the l/b ratio from 0.4 to 0.5 is consistent across all mixtures but more pronounced in 15M NaOH activated systems. This decline in strength (approximately 32-35% for 15M systems) can be attributed to the dilution effect of excess liquid, which creates a more porous microstructure in the hardened matrix and reduces the concentration of reactive species during geopolymerization (Duxson, Provis, et al., 2007).

Similarly, the lower strength observed at l/b = 0.3 compared to l/b = 0.4 indicates insufficient liquid for complete dissolution and reaction of precursors, resulting in incomplete geopolymerization and consequently reduced mechanical performance (Kong & Sanjayan, 2010). This effect is particularly evident in high-strength formulations (15M NaOH with CCS incorporation), suggesting that as performance potential increases, so does the sensitivity to l/b ratio optimization.

4.2.5 Comparison with Conventional Cement Mortar

A critical evaluation criterion for any alternative binder system is its performance relative to conventional cement-based materials. **Figures 4.4-4.6** include benchmark values for conventional cement mortar, providing a direct comparison with the geopolymer formulations across different l/b ratios.

At l/b = 0.4 (**Figure 4.5**), several geopolymer formulations surpassed the reference cement mortar strength of 24.5 MPa. Particularly noteworthy were the 15M NaOH activated mixtures with the optimal 60SS:40SH ratio, where all FA:CCS combinations achieved strengths exceeding the cement benchmark. The 15M-70FA:30CCS formulation demonstrated the most impressive performance, reaching approximately 40 MPa at the optimal parameter combination (i.e., representing a 63% strength enhancement over conventional cement mortar).

Even at l/b = 0.3 and l/b = 0.5 (**Figures 4.4 and 4.5**), select geopolymer formulations with 15M NaOH activation and optimal SS:SH ratio outperformed the reference cement mortar. This consistent superior performance across various l/b ratios demonstrates the robustness of optimized geopolymer systems and their potential as viable alternatives to conventional cement (Kutchko & Kim, 2006).

The 10M NaOH activated geopolymer mortars with 60:40 SS:SH ratio achieved strengths comparable to the cement reference, particularly when incorporating CCS at higher replacement levels. In contrast, the 5M NaOH activated systems generally underperformed relative to conventional cement mortar, indicating that sufficient alkalinity is essential for geopolymer systems to compete with traditional cementitious materials (Škvára et al., 2009).

This benchmarking confirms that properly optimized geopolymer mortars incorporating industrial by-products can match or exceed the mechanical performance of conventional cement-based systems. Given the significant environmental benefits associated with geopolymer production, including substantial reductions in carbon emissions and the valorization of industrial waste streams, these results demonstrate the practical viability of geopolymer technology as a sustainable alternative to conventional cement in construction applications (Andrew, 2018; Olivier et al., 2017).

It is important to note that the comparison presented here between geopolymer and cement mortar is intended as a practical performance benchmark rather than a rigorous chemical equivalence. The fundamental differences in chemistry between geopolymers (alkalinity, silica modulus, polycondensation) and cement (hydration) make direct comparison inherently limited. A more rigorous comparison would require aligning not only mix proportions but also parameters such as heat release, cumulative reaction degree, and workability under identical curing conditions.

4.2.6 Analysis of Compressive Strength Trends Across Multiple Variables

The relationships between compressive strength and sodium silicate (SS) content across various experimental conditions are systematically illustrated in **Figures 4.8-4.10**, providing deeper insights into the complex interaction of multiple variables in geopolymer systems. They reveal distinctive patterns in how compressive strength responds to sodium silicate content across different formulations. These patterns can be interpreted through the lens of reaction mechanisms and critical threshold effects.

The consistent bell-shaped response curves across **Figures 4.8-4.10** suggest the existence of critical thresholds governing geopolymerization efficiency. At SS contents below approximately 40%, insufficient soluble silicate species limit network formation, resulting in structures dominated by hydroxide-mediated reactions rather than silicate-based polymerization. This "silicate-deficient zone" produces incomplete geopolymerization and consequently reduced mechanical properties (Fernández-Jiménez & Palomo, 2003; Phair & Van Deventer, 2001).

Conversely, at SS contents exceeding approximately 70%, the systems enter a "hydroxide-limited zone" where the high concentration of silicate species without adequate hydroxide ions to catalyze reactions leads to inefficient polymerization and potentially interference effects. This upper threshold becomes more pronounced at higher NaOH concentrations (**Figure 4.10**), suggesting that excessive silicate species may coat precursor particles and inhibit dissolution when combined with aggressive alkaline conditions (Rostami et al., 2012).

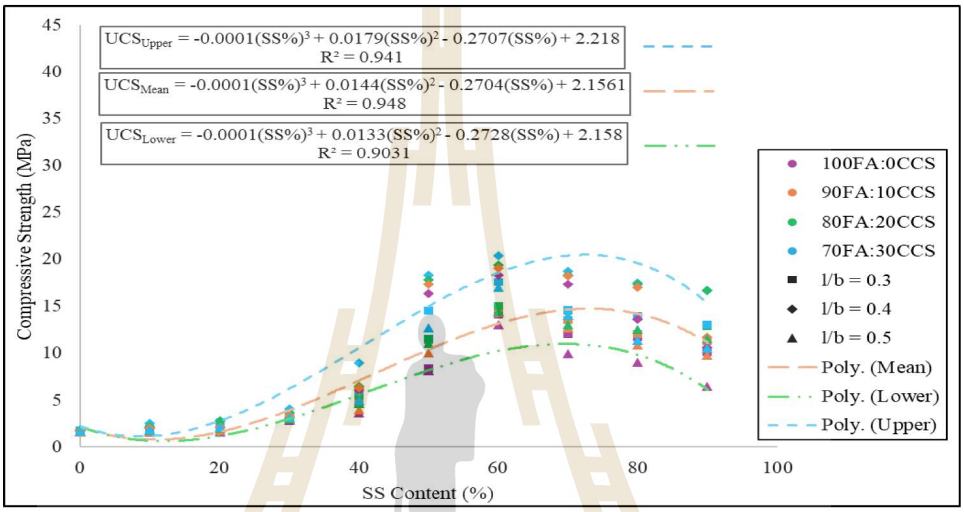


Figure 4.8 Relationship between compressive strength (MPa) and SS content (%) for different alkaline solution with 5M NaOH at different binder ratios and l/b ratios.

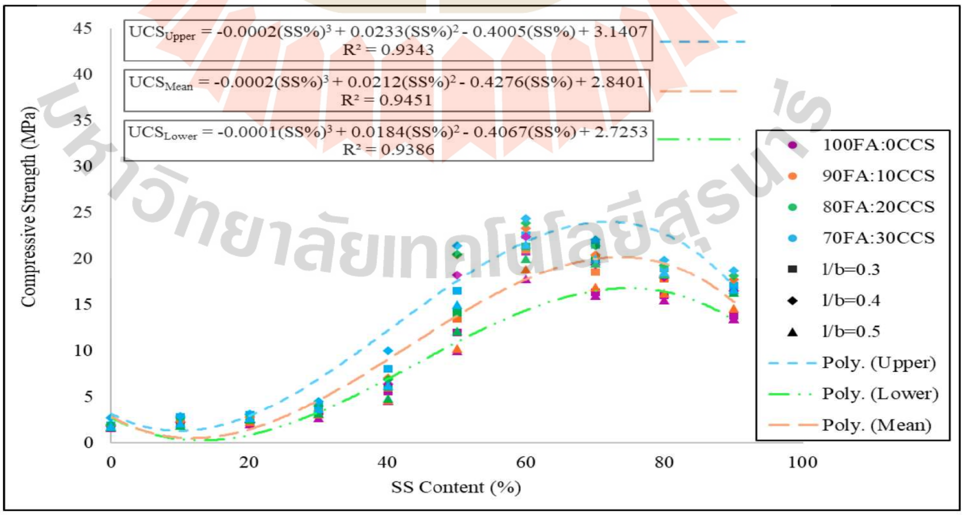


Figure 4.9 Relationship between compressive strength (MPa) and SS content (%) for different alkaline solution with 10M NaOH at different binder ratios and l/b ratios.

The data consistently identify an "optimum zone" between approximately 50:50 and 70:30 SS:SH ratios where balanced dissolution and polymerization mechanisms operate synergistically. Within this range, sufficient hydroxide ions facilitate effective precursor dissolution while adequate silicate species enable robust network formation (Škvára et al., 2009).

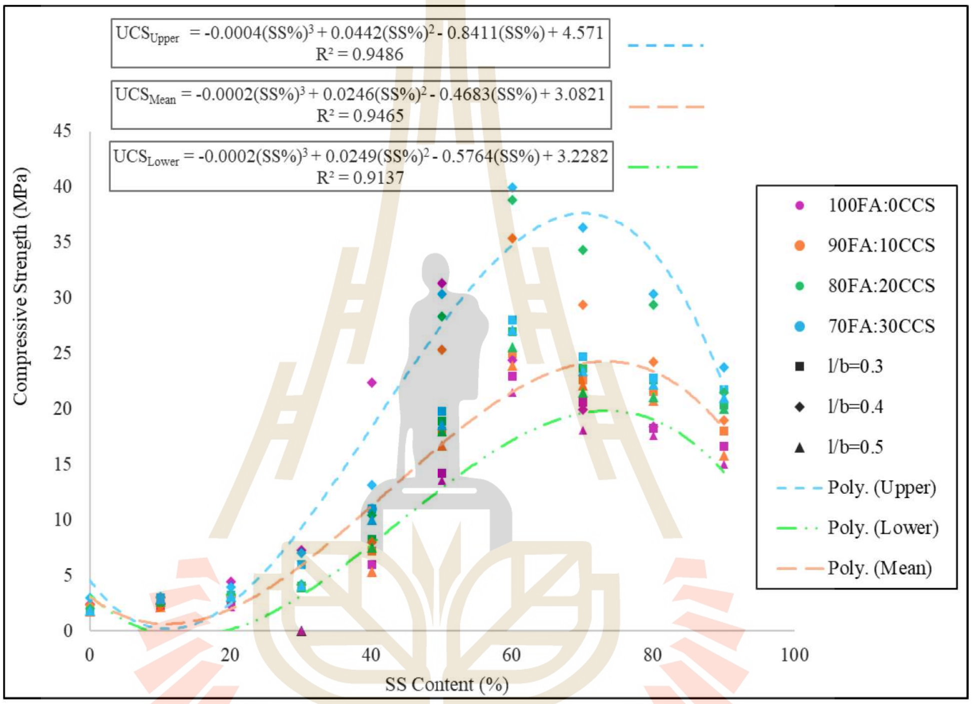


Figure 4.10 Relationship between compressive strength (MPa) and SS content (%) for different alkaline solution with 15M NaOH at different binder ratios and l/b ratios.

Notably, this optimum zone shifts slightly depending on other parameters. At higher NaOH concentrations (**Figure 4.10**), the optimum narrows and shifts toward lower SS content (50:50 to 60:40), likely because the increased hydroxide concentration enhances silicate species formation from precursor dissolution, reducing the need for additional silicate from the activator (Environment et al., 2018).

The presence of CCS also modulates the optimum zone, with higher CCS contents generally performing better at slightly lower SS content ranges. This shift can be attributed to calcium's ability to modify silicate speciation and create additional reaction pathways through C-A-S-H gel formation, reducing dependence on silicate species from the activator solution (Provis, 2018; Van der Merwe et al., 2014).

Figure 4.11 provides a comprehensive comparative analysis of compressive strength development in 5M and 15M NaOH systems across various SS ratios, FA proportions, and l/b ratios. This multivariable comparison reveals several significant trends that illuminate the fundamental geopolymerization mechanisms under different activation conditions.

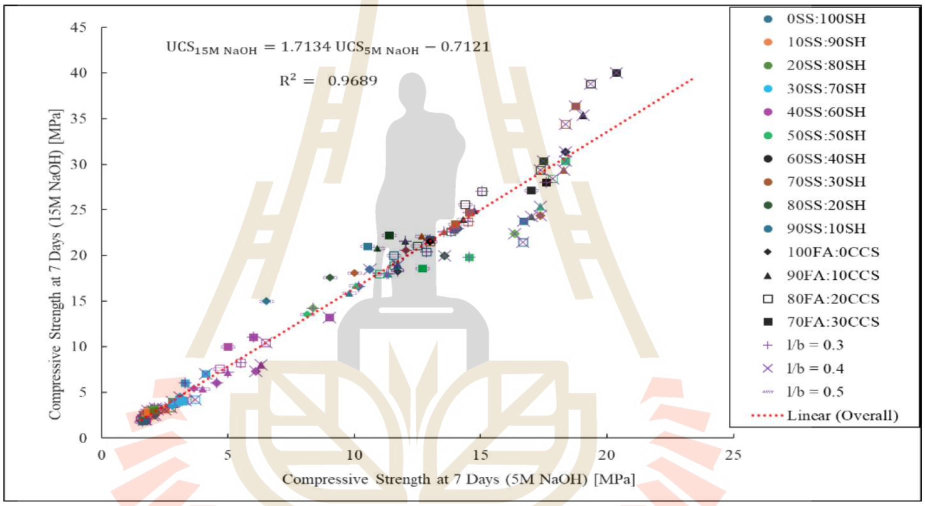


Figure 4. 11 Relationship between 7-day compressive strength of various FA: CCS geopolymer mortars at 5M and 15M NaOH concentrations at different SS:SH and l/b ratios.

The strength difference between 5M and 15M systems is consistently substantial, with 15M systems demonstrating 40-100% higher strength values depending on other formulation parameters. This enhancement is most pronounced at intermediate SS contents (40-60%) and in formulations incorporating higher CCS proportions, suggesting a synergistic effect between high alkalinity and calcium availability, consistent with observations by García-Lodeiro et al. (2011). The enhanced dissolution of aluminosilicate species under more aggressive alkaline conditions creates a more reactive environment for Ca^{2+} incorporation into the developing gel structure.

The influence of l/b ratio is particularly evident in 15M systems, where the optimal l/b ratio of 0.4 consistently produces superior performance compared to either 0.3 or 0.5. The sensitivity to l/b ratio variation is more pronounced in high-alkalinity systems, indicating that as reaction kinetics accelerate under more aggressive alkaline conditions, the balance between reactant concentration and mobility becomes increasingly critical, as demonstrated by Zhang et al. (2023).

The enhancement effect of CCS incorporation is magnified at higher NaOH concentrations, with 70FA:30CCS formulation showing the most substantial performance improvement in 15M systems compared to their 5M counterparts. This observation suggests that the calcium-rich environment provided by CCS can be more effectively leveraged under highly alkaline conditions, leading to more sophisticated hybrid binding systems incorporating both N-A-S-H and C-A-S-H gels, as described by Hanifa et al. (2025) and Yang et al. (2024).

These comprehensive analyses of compressive strength trends across multiple variables provide valuable insights for optimizing geopolymer formulations for specific strength requirements and processing conditions. The identified patterns demonstrate the complex interplay between alkaline activation parameters, precursor characteristics, and mixing proportions in determining the ultimate mechanical performance of geopolymer mortars.

4.3 Microstructural Analysis

4.3.1 SEM Analysis

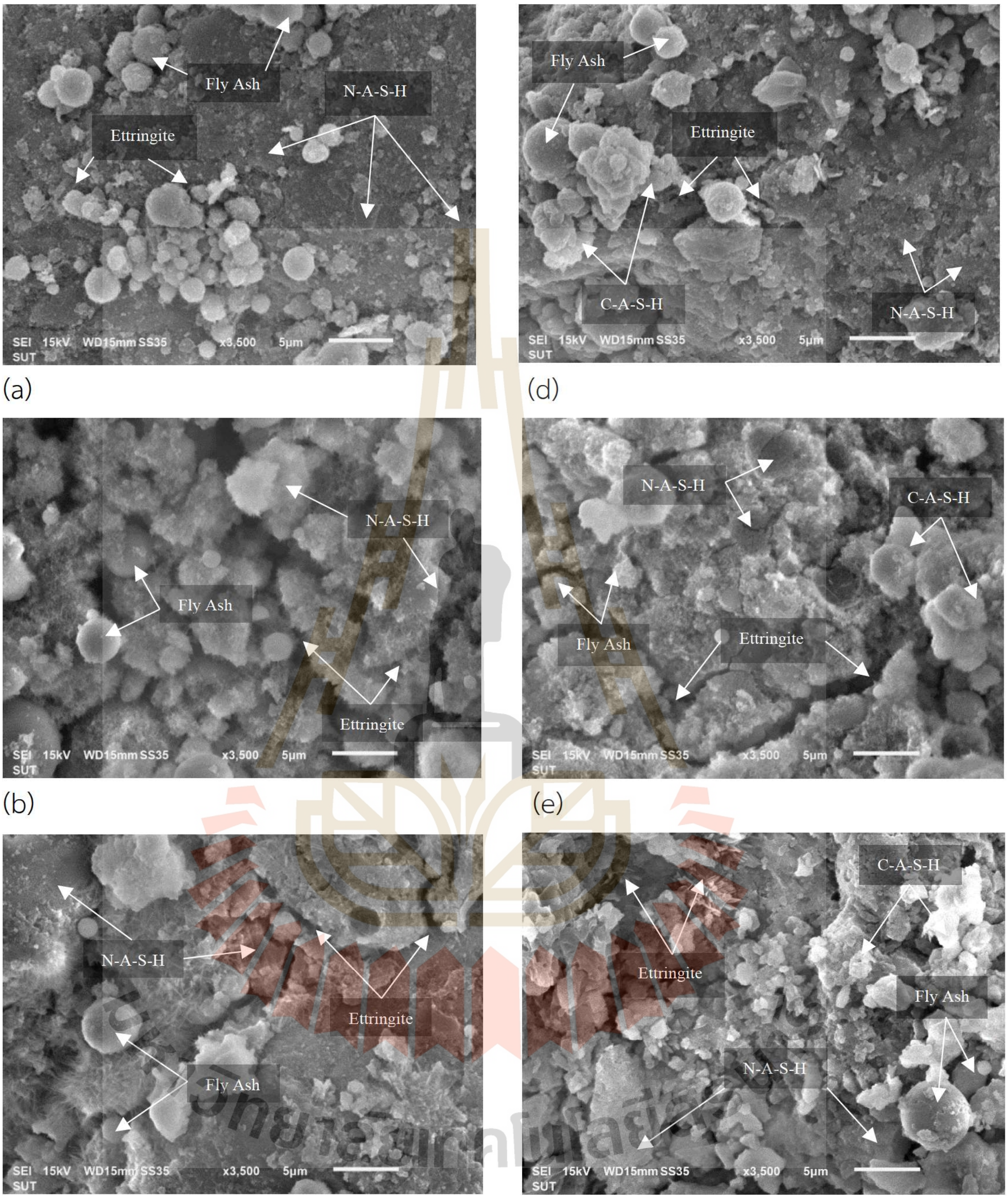
The microstructural evolution of geopolymer mortars with varying NaOH concentrations and precursor compositions was systematically investigated using SEM analysis. **Figure 4.12** presents SEM micrographs of specimens with different NaOH concentrations (5M, 10M, and 15M) and precursor ratios (100FA:0CCS and 70FA:30CCS) at the optimal liquid-to-binder ratio of 0.4.

In the 100FA:0CCS system activated with 5M NaOH (**Figure 4.12a**), the microstructure exhibits partially reacted fly ash particles embedded in a relatively loose matrix. The geopolymeric gel formation was incomplete, with visible boundaries between particles and gel phases, indicating limited dissolution of aluminosilicate species under low alkaline conditions. This microstructural observation corresponded directly with the modest compressive strength values (15-20 MPa) as reported in compressive strength test results.

As the NaOH concentration increased to 10M (**Figure 4.12b**), a notable densification of the microstructure was observed, with increased gel formation and reduced interparticle porosity. Partially reacted fly ash spheres remained visible but showed signs of surface etching and dissolution, indicating more effective alkaline activation. The enhanced gel formation creates stronger particle-matrix interfaces, explaining the moderate increase in compressive strength (20-25 MPa) observed in these systems.

At 15M NaOH concentration (**Figure 4.12c**), the pure fly ash system demonstrated substantially improved microstructural characteristics with extensive gel formation. Most fly ash particles show advanced dissolution, with reaction products filling interparticle spaces and creating a more homogeneous matrix. This densified microstructure provides the structural foundation for the enhanced compressive strength (25-31 MPa) recorded in these high-alkali formulations, as detailed in compressive strength test results.

The progressive microstructural refinement with increasing NaOH concentration aligns with the reaction mechanisms proposed by Duxson, Provis, et al. (2007), where higher alkalinity enhances the dissolution of aluminosilicate species, leading to more extensive N-A-S-H gel formation and consequently improved mechanical properties.



(a) (d)

(b) (e)

(c) (f)

Figure 4.12 Scanning electron microscope images of (a) 5M-100FA:0CCS (b) 10M-100FA:0CCS (c) 15M-100FA:0CCS (d) 5M-70FA:30CCS (e) 10M-70FA:30CCS (f) 15M-70FA:30CCS at l/b ratio of 0.4.

The incorporation of 30%CCS replacement of FA introduces significant microstructural modifications across all NaOH concentrations. At 5M NaOH (**Figure 4.12d**), the 70FA:30CCS system exhibited a more compact microstructure compared to its pure fly ash counterpart, with visible calcium-rich reaction products filling interstitial spaces. The microstructure remains somewhat heterogeneous, with distinct regions of varying morphology, revealing the simultaneous formation of different gel types, likely N-A-S-H and C-A-S-H phases as reported by García-Lodeiro et al. (2011).

When activated with 10M NaOH (**Figure 4.12e**), the 70FA:30CCS system demonstrated enhanced microstructural integration, with reaction products effectively binding particle clusters into a more cohesive matrix. The improved interfacial transition zones between particles and binding phases corresponded to the increased compressive strength observed in these formulations.

The most significant microstructural refinement was observed in the 15M NaOH activated 70FA:30CCS system (**Figure 4.12f**), which exhibited remarkably dense geopolymeric gels. The microstructure shows characteristics of advanced geopolymerization, with extensive reaction product formation creating a sophisticated binding network. The formation of this hybrid binding system, containing both N-A-S-H and C-A-S-H gels as suggested by Hanifa et al. (2025) provides the microstructural foundation for the exceptional compressive strength (approximately 40 MPa) recorded in these formulations.

The SEM observations further validate the proposed mechanisms for CCS incorporation effects, where calcium ions from CCS participate in the formation of C-A-S-H gels in addition to N-A-S-H gels typical of fly ash-based geopolymers. This dual gel formation creates a more sophisticated binding system with enhanced mechanical properties.

4.3.2 XRD Analysis

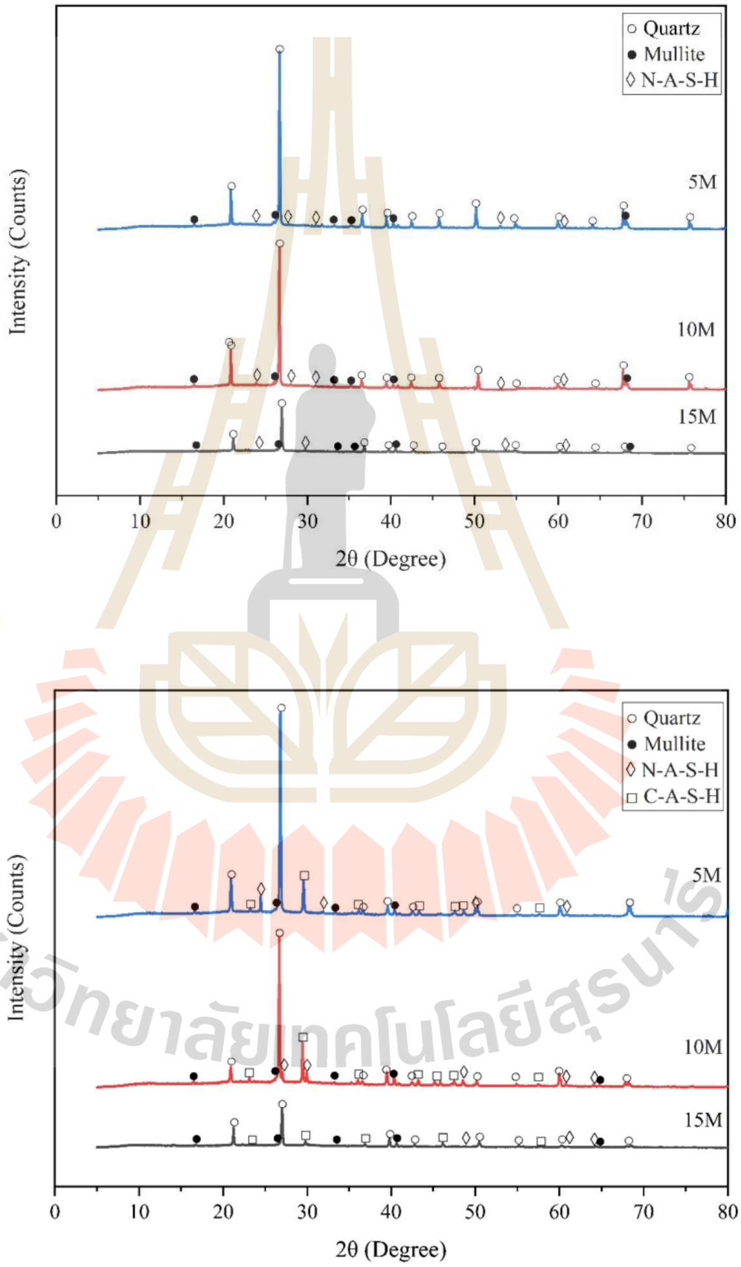
XRD analysis was conducted to investigate the crystalline phases and mineralogical transformations in geopolymer mortars with varying NaOH concentrations and precursor compositions. **Figure 4.13** presents the XRD patterns for 100FA:0CCS and 70FA:30CCS specimens with different NaOH molarities (5M, 10M, and 15M) at the optimal liquid-to-binder (l/b) ratio of 0.4.

The XRD patterns of 100FA:0CCS specimens (**Figure 4.13a**) reveal several characteristic crystalline phases across all NaOH concentrations. Primary crystalline phases include quartz (SiO_2) identified by characteristic peaks at approximately 2θ = 26.6° and 20.8°, and mullite ($Al_6Si_2O_{13}$) indicated by peaks at approximately 2θ = 16.4°, 25.9°, and 33.2°. These phases represent the crystalline components of the original fly ash that remain largely unreacted during the geopolymerization process, as similarly observed by Criado et al. (2016).

A prominent broad hump observed between 20° and 35° 2θ indicates the formation of amorphous geopolymeric gel, primarily N-A-S-H. The intensity and breadth of this amorphous hump increase with higher NaOH concentrations, indicating more extensive dissolution of aluminosilicate species and subsequent formation of amorphous reaction products. This observation aligns with the SEM findings, where higher NaOH concentrations resulted in more advanced geopolymerization and densified microstructures.

The relative intensity of crystalline peaks, particularly quartz and mullite, illustrated slight but progressive reduction with increasing NaOH concentration. This reduction indicated partial dissolution of the crystalline phases under more aggressive alkaline conditions, contributing additional reactive species to the geopolymerization process. These findings supported the mechanical performance trends, where higher NaOH concentrations consistently produced enhanced compressive strength.

The XRD patterns of 70FA:30CCS specimens (**Figure 4.13b**) exhibit notable differences compared to the pure fly ash systems, reflecting the influence of calcium incorporation on reaction product formation. While the primary crystalline phases (quartz and mullite) from the original fly ash remain present, their relative intensities appear diminished compared to the 100FA:0CCS systems, revealing enhanced dissolution in the calcium-rich environment.



(a)

(b)

Figure 4.13 X-ray diffraction patterns of (a) 100FA:0CCS and (b) 70FA:30CCS with different molarities of NaOH at l/b ratio of 0.4.

Additional peaks corresponding to calcium-bearing phases can be observed in the 70FA:30CCS patterns. These include potential C-S-H and C-A-S-H phases, which were characteristic of systems containing both calcium and aluminosilicate sources. The formation of these calcium-bearing phases aligns with the proposed hybrid binding mechanism, where calcium ions from CCS participate in reaction pathways parallel to traditional geopolymerization.

The amorphous hump between 20° and 35° 2θ in the 70FA:30CCS patterns appears broader and potentially incorporates multiple overlapping amorphous phases compared to the 100FA:0CCS systems. This broader amorphous feature likely represented the coexistence of N-A-S-H and C-A-S-H gels, as suggested by García-Lodeiro et al. (2011) in their investigation of compatibility between these gel types.

The influence of NaOH concentration on mineralogical composition is evident in both precursor systems. Higher NaOH molarity (15M) consistently results in more pronounced amorphous characteristics and reduced crystallinity compared to lower molarities (5M and 10M). This trend indicates more extensive dissolution of precursor materials and formation of reaction products under more aggressive alkaline conditions, supporting microstructural observations.

The most significant mineralogical transformations were observed in the 15M-70FA:30CCS specimen, which exhibited the most pronounced amorphous features and the least distinct crystalline peaks. This enhanced transformation aligns with the exceptional mechanical performance of this formulation, which achieved the highest compressive strength (approximately 40 MPa) among all tested compositions.

These findings validated the optimization strategies developed through the systematic parameter evaluation and provided mineralogical evidence for the microstructural characteristics observed in the SEM analysis. The XRD results confirmed that optimal performance was achieved when reaction conditions facilitated extensive amorphous gel formation while maintaining appropriate phase compatibility between N-A-S-H and C-A-S-H gels in hybrid systems.

It is noted that the presence of quartz sand in the mortar samples may contribute to dominant crystalline peaks, potentially masking signals from amorphous or weak crystalline geopolymer gel phases. Future mineralogical analysis should be performed on paste samples to minimize aggregate interference and improve the detection of reaction products.

4.4 Machine Learning Model Performance and Analysis

The comparative analysis of machine learning algorithms revealed substantial differences in predictive capability for geopolymer strength estimation. XGBoost demonstrated superior performance with an exceptional R^2 value of 0.998 (on a scale of 0 to 1), indicating it explained nearly all variance in the compressive strength data. This was complemented by the lowest error metrics (MSE=0.142, RMSE=0.377 MPa), representing predictions within approximately 0.4 MPa of actual strength values across the entire range of experimental results.

CatBoost achieved the second-best performance (R^2=0.992, MSE=0.620, RMSE=0.787 MPa), maintaining strong predictive power while showing moderately higher error values. Both gradient boosting algorithms substantially outperformed traditional approaches, with SVM (R^2=0.946, MSE=4.045, RMSE=2.011 MPa) and AdaBoost (R^2=0.929, MSE=5.198, RMSE=2.280 MPa) exhibiting considerably higher prediction errors.

Given its superior performance metrics, XGBoost was selected for further interpretability analysis using SHAP. The XGBoost model demonstrated exceptional predictive capability for geopolymer strength estimation as evidenced by **Figure 4.14**. The scatter plot reveals a remarkably strong linear relationship between actual and predicted compressive strength values spanning the entire experimental range (2-40 MPa). The fitted regression line ($y = 0.9971x + 0.0306$) exhibits a slope approaching unity and a negligible y-intercept, confirming the model's extraordinary accuracy across all strength levels.

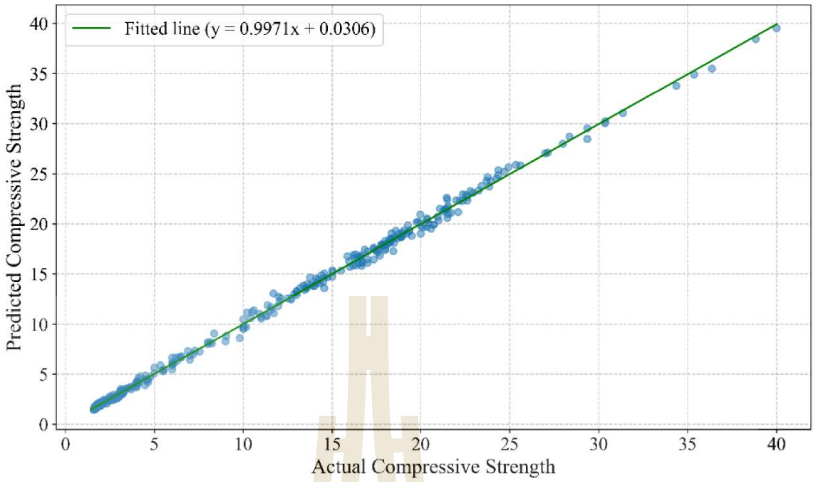
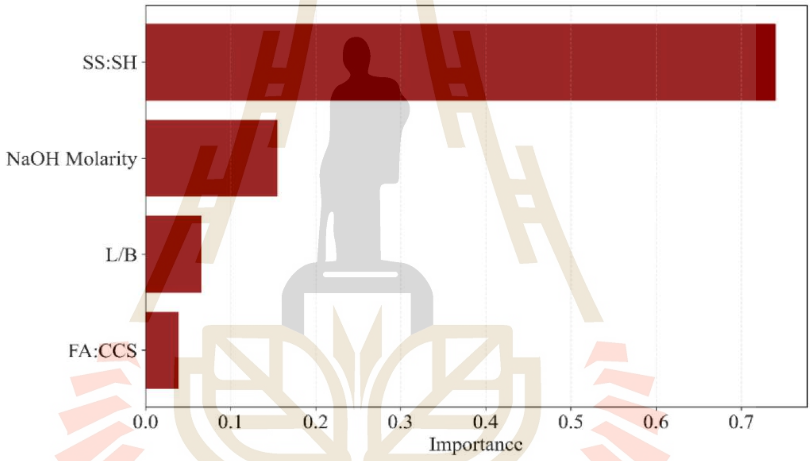


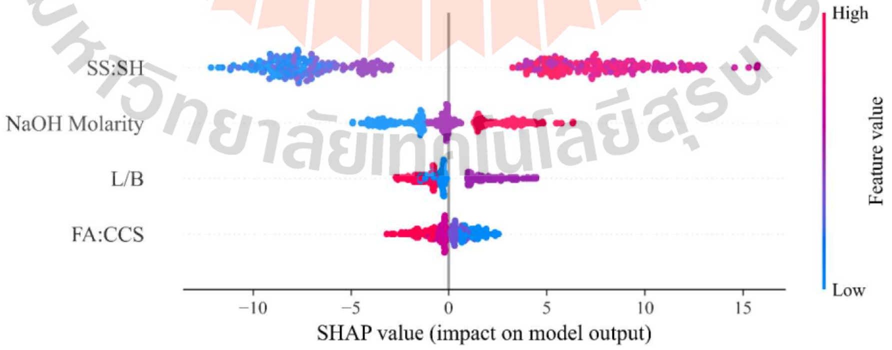
Figure 4.14 A relationship between actual and predicted compressive strength based on machine learning models.

The XGBoost-SHAP analysis presented in **Figure 4.15** provides critical insights into the relative importance and specific impacts of compositional parameters on geopolymer strength development. **Figure 4.15a** clearly establishes a hierarchical ranking of parameters that influence compressive strength prediction. The SS:SH ratio emerged as the most influential parameter with a normalized importance value of approximately 0.7 (on a scale of 0 to 1), significantly higher than all other parameters. NaOH molarity ranks second in importance (approximately 0.15), showing moderate influence on strength predictions but significantly less than the SS:SH ratio. l/b ratio and FA ratio show substantially lower importance values (approximately 0.05 each), suggesting these traditionally emphasized mix design parameters exert less influence than activator composition in determining final strength properties.

The SS:SH ratio's predominant influence stems from its critical role in regulating silicate network formation during geopolymerization, fundamentally shaping the molecular architecture of binding gels. This parameter directly controls the availability of soluble silica species necessary for forming the three-dimensional aluminosilicate framework that provides structural integrity to the hardened material. The precise balance between silicate and hydroxide ions determines reaction pathway efficiency, network connectivity, and ultimate mechanical properties. NaOH molarity emerges as the secondary influence by governing precursor dissolution kinetics and subsequent gel formation. The hydroxide concentration dictates the rate at which aluminosilicate species are released from raw materials into the reaction medium, while also affecting pH-dependent condensation reactions that form the geopolymer backbone. Optimized molarity ensures efficient precursor activation without disrupting critical polymerization processes.



(a)



(b)

Figure 4.15 XGBoost-SHAP analysis: (a) Features importance ranking, (b) Features impact on the model output (compressive strength).

Figure 4.15b expands on the importance ranking by revealing how specific values of each parameter positively or negatively impact strength predictions. SS:SH ratio displays a distinct pattern where higher values (represented by red points, generally SS:SH ratios above 60:40) predominantly appear on the positive side of the SHAP value axis, indicating that increased sodium silicate proportions generally enhance compressive strength. Lower SS values (blue points, SS:SH ratios below 40:60) consistently show negative impacts, suggesting insufficient soluble silicate undermines strength development.

NaOH molarity exhibits a more complex non-linear relationship. Moderate molarity values (purple points, particularly in the 10-12M range) tend to contribute positively to strength predictions, while both very low concentrations (below 7M, blue points) and very high concentrations (above 13M, red points) often show negative impacts. This supports the existence of an optimal alkalinity range for geopolymerization.

l/b ratio demonstrates a clear inverse relationship with strength, where lower l/b values (blue points, particularly ratios below 0.4) consistently show positive contributions to the predicted strength, while higher values (red points, ratios above 0.45) exhibit negative impacts. This confirms the detrimental effect of excessive liquid content on mechanical properties. FA ratio shows a more nuanced distribution where certain specific ratios (particularly in the 70:30 to 80:20 FA:CCS range) exhibit stronger positive contributions than others, suggesting optimal replacement levels rather than a simple linear trend.

These SHAP analysis findings reveal that activator composition parameters (particularly SS:SH ratio) exert significantly greater influence on geopolymer strength development than precursor proportions or water content. This insight challenges conventional mix design approaches that often prioritize solid precursor ratios and suggests that optimizing the alkaline activator should be the primary focus for enhancing geopolymer performance. For practical implementation, this indicates that practitioners should first establish the optimal SS:SH ratio (around 60:40) and NaOH concentration (10-12M) before fine-tuning precursor ratios and water content. The non-linear relationships identified between specific parameter values and strength impacts further highlight the complexity of geopolymer systems and demonstrate the value of machine learning approaches in capturing these intricate interactions.

It is of interest to note that the machine learning models developed in this study are based on a focused dataset of 45 mix designs. While this represents comprehensive coverage of the parameter space investigated, model predictions outside the studied ranges should be approached with caution. The models are most reliable for interpolation within the investigated parameter ranges rather than extrapolation beyond them.

CHAPTER V

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This comprehensive study on geopolymer mortars using fly ash (FA) and calcium carbonate sludge (CCS) has yielded several significant findings regarding the relationships between composition, processing parameters, and performance characteristics:

1) The setting time of geopolymer mortars was significantly influenced by the liquid-to-binder ratio, alkaline activator composition, and precursor materials. Higher NaOH concentrations (15M) consistently produced shorter setting times compared to lower molarities (5M and 10M), attributed to enhanced dissolution of aluminosilicate species and accelerated geopolymerization. The incorporation of CCS further accelerated setting times, with 30% CCS replacement showing the most pronounced effect.

2) Compressive strength development showed optimal performance at a liquid-to-binder ratio of 0.4, with several mix designs exceeding the reference cement mortar strength of 24.5 MPa. The most effective compositions featured a sodium silicate to sodium hydroxide ratio of 60:40, achieving strengths of 25-40 MPa. This optimal ratio represented the ideal balance between silicate species and hydroxide ions for maximizing network formation and structural densification.

3) The incorporation of CCS with FA resulted in the formation of a sophisticated hybrid binding system containing both N-A-S-H and C-A-S-H gels. This synergistic effect was most pronounced at 20-30% CCS replacement and 15M NaOH concentration, where formulations demonstrated up to 63% higher strength compared to conventional cement mortar (40 MPa vs 24.5 MPa).

4) Microstructural analysis revealed that higher NaOH concentrations (15M) produced denser and more compact matrices, particularly in specimens containing both FA and CCS. The presence of both N-A-S-H and C-A-S-H gels in hybrid systems resulted in more homogeneous microstructures compared to systems with FA alone, providing the structural foundation for enhanced mechanical properties.

5) XRD analysis confirmed the formation of complex reaction products, with higher NaOH molarities leading to more pronounced amorphous characteristics, indicating more extensive dissolution of precursor materials. The incorporation of CCS introduced additional calcium-bearing phases, supporting the formation of C-A-S-H gel observed in microscopic analysis.

6) Machine learning analysis using XGBoost demonstrated exceptional predictive accuracy (R^2=0.998) for compressive strength based on compositional parameters. SHAP analysis revealed that the SS:SH ratio was the most influential parameter (approximately 70% importance), followed by NaOH molarity (15%), with liquid-to-binder ratio and FA ratio showing substantially lower importance (approximately 5% each). These finding challenges conventional mix design approaches that often prioritize solid precursor ratios and suggests that optimizing the alkaline activator should be the primary focus for enhancing geopolymer performance. While the machine learning models demonstrated exceptional predictive accuracy within the studied parameter ranges, future research should validate these models with expanded datasets or external data from different raw material sources to confirm their broader applicability and generalizability across various geopolymer systems.

From a practical implementation perspective, this research provides valuable guidelines for developing geopolymer mortars tailored to specific applications. For rapid-setting applications, formulations with 20-30% CCS activated with 15M NaOH at l/b=0.3 offer optimal performance. For applications requiring higher strength, the 70FA:30CCS formulation with 15M NaOH and 60:40 SS:SH ratio at l/b=0.4 provides exceptional mechanical properties.

5.2 Recommendation

Future research should comprehensively address several critical aspects for practical implementation:

1) Long-term durability characteristics, including resistance to aggressive environments (sulfate and acid attack, freeze-thaw cycling), dimensional stability (drying shrinkage), and carbonation resistance under various exposure conditions;

2) Quantitative environmental and economic assessments through life cycle analysis (LCA) and detailed cost comparisons with conventional cement, considering raw material costs, processing energy, and carbon credits;

3) Scale-up challenges, including raw material variability between different sources of FA and CCS, industrial-scale mixing procedures, quality control protocols, and optimization of curing conditions for large-scale production.

Addressing these aspects systematically would provide a complete evaluation of the commercial viability and further advance the practical implementation of this sustainable construction technology. Future research should focus on long-term durability characteristics, including resistance to aggressive environments, dimensional stability, and performance under varying environmental conditions. Additionally, scaling these optimized formulations for commercial applications and evaluating their economic feasibility compared to conventional cementitious materials would further advance the practical implementation of this sustainable construction technology.

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BIOGRAPHY

Mrs. Soe Thandar, born on July 2, 1996, in Pathein township, Myanmar, began her educational journey in High School, where she successfully passed her matriculation examination in 2012 with four distinctions out of six subjects. She went on to pursue a bachelor's degree in Civil Engineering at Technological University (Pathein), graduating in November 2018 with an excellent GPAX of 4.8 out of 6.0.

Following her undergraduate studies, Mrs. Thandar began her postgraduate journey as a Master of Geotechnical Engineering Candidate at Yangon Technological University from June 2019 to January 2021. Her passion for the field led her to further her education abroad, and in December 2022, she began a Master of Civil, Transportation and Geo-resources Engineering at Suranaree University of Technology, Thailand, supported by the Vithedbundit Scholarship.

During her master's studies at Suranaree University of Technology, she has served as a Research and Teaching Assistant, gaining practical experience in consolidation analysis using PLAXIS, slope stability analysis, and soil stabilization research. Concurrently, she has amassed three years of experience as a Founder for the Geotechnical Engineering Knowledge Centre, teaching basic and advanced soil mechanics to over 460 professionals, candidates, and students from around the world.

Her research efforts resulted in the publication of a paper in the Case Studies in Construction Materials journal, titled "Strength and Microstructural Evaluation of Sustainable Geopolymer Mortars using Calcium Carbonate Sludge and Fly Ash as Precursors." She also presented her work at the Geomate Conference in November 2024. With a strong educational foundation, a passion for geotechnical engineering, and a commitment to research and teaching, she aspires to pursue a Ph.D. and become a professional engineer who contributes significantly to society.