Influence of Mix Proportions, Pressing Force, Aging and Sintering for Acid-Resistant Pressed Geopolymers

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LIST OF ABBREVIATIONS

AA	Alkali activator
Al	Aluminium
ANOVA	Analysis of variance
AS	Aluminosilicate source
ASTM	American Society of Testing and Materials
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
Ca	Calcium
CaO	Calcium oxide
CO_2	Carbon dioxide
Cs_2SiO_3	Cesium silicate
C-S-H	Calcium-silicate-hydrate
Cu	Copper
ECI	Embodied carbon index
ECO_2	Embodied carbon dioxide emission
EE	Embodied energy
Fe_2O_3	Iron oxide
FTIR	Fourier transform infrared
H_2SO_4	Sulfuric acid
HCl	Hydrochloric acid
HNO_3	Nitric acid
I	Cumulative water absorption
ICP	Inductively coupled plasma
IOPC	Inorganic-organic polymer composites
ISO	International organization for standardization
k	Sorptivity coefficient
K	Potassium
K_2SiO_3	Potassium silicate
KOH	Potassium hydroxide
Mg	Magnesium
MIP	Mercury intrusion porosimetry
Na	Sodium
Na_2SiO_3	Sodium silicate

NaOH	Sodium hydroxide
N-A-S-H	Sodium-aluminate-silicate-hydrate
NMR	Nuclear magnetic resonance
OPC	Ordinary Portland cement
pH	Potential of hydrogen
S	Sulfur
SEM	Scanning electron microscopy
Si	Silicon
SiO_2	Silicon dioxide
SO_2	Sulfur dioxide
TGA	Thermogravimetric
XRD	X-ray diffraction
XTM	X-ray tomography

LIST OF SYMBOLS

%	Percentage (mathematical symbol)
<	Smaller than
>	Greater than
°	Degree (unit of angle)
℃	Degree Celsius (unit of temperature)
μm	Micron (unit of length)
2θ	2-theta (physical symbol)
cm^{-1}	Per centimeter (unit of wavenumber)
day	Day (unit of period)
g/cm^3	Gram per cubic centimeter (unit of bulk density)
h	Hours (unit of time)
kg CO_2/kg	Kilogram of carbon dioxide equivalent per kilogram (unit of embodied carbon dioxide emission)
kg/m^3	Kilogram per cubic meter (unit of density)
M	Molar (unit of concentration)
mg/L	Milligrams per liter (unit of concentration)
min	Minute (unit of time)
MJ/kg	Megajoules per kilogram (unit of embodied energy)
mm	Millimeter (unit of length)
mm^3	Cubic millimeter (unit of volume)
mm^3/g	Cubic millimeter per gram (unit of volume)
MPa	Megapascal (unit of strength)
nm	Nanometer (unit of length)
ppm	Parts per million (unit of concentration)
ton	Ton (unit of pressing force)
wt.%	Weight percentage (unit of mass as percentage of the total mass)

Pengaruh Nisbah Campuran, Daya Tekan, Penuaan dan Pensinteran terhadap Geopolimer Tekan Tahan Asid

ABSTRAK

Geopolimer adalah alternatif kepada simen Portland biasa (OPC). Namun, geopolimer tuangan memerlukan pengaktif alkali yang tinggi dan menyebabkan pembentukan liang dan mengehadkan pembangunan kekuatannya. Kajian ini bertujuan untuk mengoptimumkan nisbah sumber aluminosilikat/pengaktif alkali (AS/AA), kepekatan NaOH, nisbah Na_2SiO_3/NaOH dan daya tekanan bagi geopolimer tekanan. Geopolimer tekanan yang dioptimumkan telah dibandingkan dengan geopolimer tuangan dari segi sifat bahan dan kesan alam sekitar. Selain itu, kesan tempoh penuaan (1 - 28 hari) dan suhu pensinteran (700 - 1000°C) terhadap prestasi geopolimer tekanan telah diperiksa. Kajian ini juga menilai ketahanan asid geopolimer tekanan yang telah dan belum disinter dengan merendamnya dalam pelbagai jenis (HNO_3 dan H_2SO_4) dan kepekatan (3% dan 8%) asid. Sifat-sifat yang dinilai bagi geopolimer tekanan termasuk penampilan fizikal, pengecutan linear, jisim, ketumpatan, kcliangan, penyerapan air, kekuatan mampat, kekuatan lenturan, "sorptivity" dan tingkah laku pelepasan. Pencirian bahan merangkumi mikrostruktur, komposisi unsur, evolusi struktur, ciri-ciri komposisi dan pengenalan struktur Si. Keputusan menunjukkan bahawa konfigurasi optimum untuk geopolimer tekanan melibatkan nisbah AS/AA sebanyak 5.5, kepekatan NaOH sebanyak 14 M, nisbah Na_2SiO_3/NaOH sebanyak 2.0 dan daya tekanan sebanyak 5 tan, mencapai kekuatan mampat dan lenturan sebanyak 114.2 MPa dan 29.9 MPa, masing-masing, selepas 28 hari penuaan. Geopolimer tekanan mengatasi geopolimer tuangan, menunjukkan peningkatan kekuatan mampat dan lenturan sebanyak 90% dan 382% masing-masing. Selain itu, geopolimer tekanan menunjukkan penurunan kekuatan mampat yang lebih rendah sebanyak 32% selepas ujian peroi, berbanding dengan penurunan sebanyak 60% dalam geopolimer tuangan. Selain itu, geopolimer tekanan dapat mengurangkan tenaga terbenam (EE), pelepasan karbon dioksida terbenam (ECO_2) dan indeks karbon terbenam (ECI) sebanyak 50%, 59% dan 21%, masing-masing. Kekuatan mekanikal geopolimer tekanan dipertingkatkan lagi dengan penuaan selama 7 hari dan pensinteran pada suhu 1000°C. Analisis fasa mengesahkan pembentukan nefelin yang ketara, meningkatkan sifat seramik geopolimer tekanan. Geopolimer tekanan yang telah disinter mengekalkan kekuatan mekanikal yang unggul berbanding dengan yang belum disinter, terutamanya selepas 28 hari perendaman dalam 3% HNO_3 di mana sampel yang telah disinter menunjukkan peningkatan kekuatan mampat. Kedua-dua sampel yang telah dan belum disinter membentuk gypsum selepas perendaman H_2SO_4, tetapi hanya sampel yang telah disinter mencegah pembentukan retak disebabkan oleh struktur silang yang padat. Kajian ini mengesyorkan pensinteran geopolimer tekanan selama 7 hari pada suhu 1000°C untuk meghasilkan bahan binaan yang tahan terhadap asid dan sesuai untuk aplikasi pengkapsulan sisa. Hasil kajian ini menunjukkan potensi geopolimer yang ditekan sebagai alternatif yang kukuh dan mesra alam kepada OPC untuk aplikasi pembinaan, sejajar dengan Matlamat Pembangunan Mampan (SDG) melalui promosi infrastruktur lestari dan pengurangan kesan alam sekitar melalui pelepasan karbon dan penggunaan tenaga yang lebih rendah.

Influence of Mix Proportions, Pressing Force, Aging and Sintering for Acid-Resistant Pressed Geopolymers

ABSTRACT

Geopolymer is an alternative to ordinary Portland cement (OPC). However, traditional cast geopolymers require highly alkali activators, leading to pores formation and limiting its strength development. The study aimed to optimize the aluminosilicate source-to-alkali activator (AS/AA) ratios, NaOH concentrations, Na_2SiO_3/NaOH ratios and pressing forces of fly ash-based pressed geopolymer. The optimized pressed geopolymer was compared to cast geopolymer in terms of material properties and environmental impacts. Besides, the dual effects of aging periods (1 – 28 days) and sintering temperatures (700 – 1000°C) on the performance of pressed geopolymer were evaluated. The study also assessed the acid resistance of sintered and unsintered pressed geopolymers by immersing them in different types (HNO_3 and H_2SO_4) and concentrations (3% and 8%) of acids. The evaluated properties of the pressed geopolymer included physical appearance, linear shrinkage, mass, density, porosity, water absorption, compressive strength, flexural strength, sorptivity and leaching behavior. Materials characterizations encompassed microstructure, elemental composition, structural evolution, composition characteristics and Si structure identification. Results indicated that the optimal configuration for pressed geopolymer involved AS/AA ratio of 5.5, NaOH concentration of 14 M, Na_2SiO_3/NaOH ratio of 2.0 and pressing force of 5 tons, achieving compressive and flexural strengths of 114.2 MPa and 29.9 MPa, respectively, after 28 days of aging. The pressed geopolymer significantly surpassed cast geopolymer in performance, showing increases in compressive and flexural strengths by 90% and 382%, respectively. Not only that, the pressed geopolymer demonstrated a lower declinein compressive strength of 32% after efflorescence testing, compared to a 60% reduction in cast geopolymer. Besides, pressed geopolymers reduce embodied energy (EE), embodied carbon dioxide emission (ECO_2) and embodied carbon index (ECI) by 50%, 59% and 21%, respectively. The mechanical strengths of pressed geopolymer were further improved by aging for 7 days and sintering at 1000°C. Phase analysis confirmed significant nepheline formation, enhancing the ceramic-like properties of pressed geopolymer. The sintered pressed geopolymers maintained superior mechanical strength over their unsintered counterpart, especially notable after a 28-day immersion in 3% HNO_3 where sintered samples showed increased compressive strength. Both sintered and unsintered samples developed gypsum after H_2SO_4 immersion, but only the sintered samples prevented crack formation due to their dense cross-linked structure. This study recommended sintering the 7-day pressed geopolymer at 1000°C for developing acid-resistant building materials suitable for waste encapsulation applications. These findings demonstrate the potential of pressed geopolymer as a robust, eco-friendly alternative to OPC for construction applications, aligning with the Sustainable Development Goals (SDGs) by promoting sustainable infrastructure and reducing environmental impact through lower carbon emissions and energy consumption.

CHAPTER 1 : INTRODUCTION

1.1 Research Background

Electricity generation from pulverized coal produces fly ash, which poses environmental challenges due to its high concentrations of heavy metals. These contaminants can be environmentally hazardous if not managed properly. A promising approach to address this issue is to repurpose fly ash in geopolymer production, offering a sustainable alternative for waste utilization. Geopolymers represent a class of inorganic polymers that have garnered significant attention in various sectors, especially construction and environmental engineering, due to their unique properties and potential sustainability benefits. The genesis of the term "geopolymer" can be traced back to the innovative work of Joseph Davidovits in the 1970s (Davidovits, 1989). The etymology of "geopolymer" is rooted in "geo" from the Greek word for earth, reflecting the geological origin of its raw materials, while "polymer" indicates a chain of molecules produced from the same unit. This term aptly describes a synthetic aluminosilicate binder system characterized by networks of inorganic molecules (Younis et al., 2020).

Developed as a viable substitute for ordinary Portland cement (OPC), geopolymers offer a promising avenue toward more eco-friendly construction practices (Ren et al., 2021). Unlike OPC which relies on the hydration of calcium silicates, geopolymers form through a chemical reaction between an aluminosilicate source, such as fly ash, metakaolin or slag, and an alkali activator solution. This reaction reorganizes the silica (SiO_2) and alumina (Al_2O_3) species into a dense, three-dimensional framework, endowed with notable mechanical strength and durability (Roopchund et al., 2022). The production of geopolymer significantly reduces carbon dioxide (CO_2) emissions and

often incorporates industrial by-products, thereby contributing to waste reduction and resource conservation (Zhao et al., 2021).

The formation of geopolymers, known as geopolymerization, is a chemical process that transforms loose aluminosilicate powders into a solid and cohesive structure (Shehata et al., 2021). This transformation involves the dissolution of aluminosilicate source in an alkali activator solution, followed by the reorganization of the dissolved species into a polymeric network through a series of gelation, condensation and hardening stages (Ranjbar et al., 2020b). The versatility of this process allows for the tailoring of the properties of geopolymers by adjusting factors such as the type and concentration of the alkaline activator (Atabey et al., 2020), aluminosilicate source composition (Aouan et al., 2023) and curing conditions (Sajan et al., 2021).

Geopolymers, characterized by their excellent mechanical properties and low environmental impact, have emerged as a promising alternative to conventional construction materials. However, the widespread adoption of geopolymers faces challenges related to production methods, which significantly affect the material's performance and application scope. The widely-used geopolymer fabrication method, casting method, have limitations that may hinder the optimal exploitation of geopolymer properties. These limitations include inconsistent density, potential for void formation, longer setting times and increased shrinkage (Filipponi et al., 2023; Prasanphan et al., 2020; Ranjbar et al., 2020a). These factors can compromise the mechanical strength and durability of the final product, impacting its suitability for certain applications.

The pressing method presents an innovative approach to geopolymer production, promising to address some of these challenges by offering better control over the material's microstructure and improved density, thus enhancing mechanical properties. Research has been done on the optimization of the pressing method for geopolymer production. This includes understanding the influence of aluminosilicate source-to-alkali activator (AS/AA) ratio (Wei et al., 2022), concentration of sodium hydroxide (NaOH) (Wongsa et al., 2019), sodium silicate-to-sodium hydroxide (Na_2SiO_3/NaOH) ratio (Wang et al., 2019) and pressing force (Filipponi et al., 2022) on the microstructure, mechanical properties and durability of geopolymers. Yet, comprehensive studies on the optimization of pressed geopolymer are scarce, particularly concerning its influence on the degree of reaction, stiffness and flexural strength. Additionally, comparisons between casting and pressing methods on the mechanical properties, microstructure and environmental benefits have not been fully quantified, leaving a gap in understanding its potential as a sustainable manufacturing process.

Lengthening the aging period has been shown to improve the properties of geopolymer, especially within 28 days. After 28 days of the aging period, the rate of compressive strength increase diminished, eventually plateauing (Yadollahi et al., 2015). The strength enhancement over the aging period is attributed to ongoing geopolymerization, which minimizes unreacted raw materials and increases the reaction products of geopolymer. This subsequently strengthens the bond between the geopolymer paste and aggregates, thereby elevating the overall strength (Ghasemzadeh Mousavinejad & Gashti, 2021).

Apart from aging, sintering has been identified as a process that strengthens the geopolymer structure. Subjecting geopolymers to sintering temperatures between 800 and 1000 ℃ increased the chemical reaction rate, improving the density and compressive strength of the material (Sawan et al., 2020). Besides, the formation of new phases at 800 ℃ contributes to pore refinement and increase in material strength, while the transformation of geopolymer into ceramic-like materials maximizes the compressive strength at 1000 ℃ (Zawrah et al., 2020). The change in the microstructure of geopolymer after sintering, such as the formation of neck connections and a decrease in porosity, underscores the structural changes that occur (Ahmad et al., 2022b). However, current literature provides limited insight into the optimal aging periods and sintering temperatures necessary to maximize the performance of geopolymers across various applications. This knowledge gap hinders the ability to consistently produce geopolymers with tailored properties for specific construction needs.

Exposure to acidic environments can significantly degrade materials, affecting their structural integrity and reducing their lifespan. For instance, immersion of geopolymer in a sulphuric acid (H_2SO_4) solution can lead to the precipitation of white and yellow crystals, attributed to the generation of gypsum and ettringite (Qu et al., 2021). These crystals induce cracking due to their size and volumetric expansion, thus impairing the surface and mechanical integrity of the geopolymer (Bai et al., 2023). Not only that, the leaching of the sodium-aluminate-silicate-hydrate (N-A-S-H) gel from the geopolymer structure during acid immersion weakens the material, with the degree of strength reduction intensifying over time (Valencia-Saavedra et al., 2020). The acid resistance of geopolymers is affected by their composition, microstructure and processing conditions like sintering. This is a critical area where existing research falls short,

especially in offering insights into optimal processing conditions for enhancing the acid resistance of geopolymer. A thorough comparative analysis of sintered versus unsintered geopolymers in acidic environments is crucial for understanding degradation mechanisms and identifying compositions resilient against such conditions.

Therefore, this study aims to bridge gaps by developing fly ash-based geopolymers through the pressing method, with a focus on optimizing mixing proportion and pressing force. Subsequently, the performance and environmental impacts of optimized pressed geopolymer were compared with cast geopolymer. The dual effects of aging periods and sintering temperatures were evaluated to improve the properties of pressed geopolymer. Furthermore, this research serves the purpose of examining the acid resistance of both sintered and unsintered pressed geopolymers to assess the impact of sintering on durability in acidic environments. The physical, mechanical, microstructural, elemental composition, structural evolution and composition characteristics of pressed geopolymer were investigated. This study provided significant insights into the influence of mix proportions, pressing force, aging period and sintering temperature on the performance of pressed geopolymer, and the effect of sintering on acid resistance. In short, this study paves the way for developing high-strength and acid-resistant cementitious materials.

1.2 Problem Statement

The combustion of pulverized coal for electricity generation produces various waste products, including fly ash. Disposing of fly ash remains an unresolved environmental concern, as it contains high concentrations of heavy metals that can be harmful to the environment. An effective solution to this issue is to utilize fly ash in

geopolymer production. While recent studies have predominantly employed the casting method to fabricate geopolymers, this approach often leads to increased porosity and limits the material's long-term durability. Current research has mainly focused on the compressive strength development of pressed geopolymers, with limited attention given to flexural strength, stiffness, and the degree of reaction (Cao et al., 2023; Ranjbar et al., 2020a). These parameters are critical, as flexural strength and stiffness are essential for tile production, while the degree of reaction is fundamental to achieving comprehensive geopolymerization.

Unlike the conventional casting method, which requires a high liquid content, numerous studies have shown that pressure compaction with reduced liquid content effectively eliminates the porosity of the resulting structure (Ahmad & Rashid, 2022; Nishikawa et al., 2022). However, comprehensive comparative studies between the pressing and casting methods for geopolymer production are scarce in the literature. Existing comparative research has primarily focused on microstructure evolution and compressive strength, neglecting critical aspects such as the degree of reaction, efflorescence and ecological analysis. Evaluating the performance of pressed geopolymer compared to cast geopolymer provides valuable insights into the effectiveness of these methods for producing durable construction materials.

The fabrication of traditional ceramics typically requires high-temperature sintering, often exceeding 1600 ℃, which is energy-intensive and results in significant greenhouse gas emissions (Binner et al., 2020). On the other hand, geopolymers formed stable crystalline phases at lower temperatures (< 1000 ℃), endowing them with ceramic-like properties (He et al., 2010; Xiang et al., 2021). Cesium silicate (Cs_2SiO_3) and

potassium silicate (K_2SiO_3) are the common alkaline activators used for the preparation of geopolymers with ceramic-like properties. However, the large-scale production of these materials is financially demanding due to the high costs of the raw materials used. While the effect of sintering temperature on geopolymer properties has been extensively studied, the combined influence of aging period and sintering temperature is still unexplored. Since the aging period significantly impacts the polymerization reaction, manipulating both aging and sintering conditions could substantially affect the microstructure, phase composition and mechanical properties of sintered geopolymers.

Acid attacks on construction materials commonly originate from sources such as acid rain and organic acids present in sewage and wastewater systems. Structures immersed in acidic environments, such as those involving hydrochloric (HCl) acid, H_2SO_4 acid and nitric (HNO_3) acid, face significant damage during their service life. This acid infiltration severely compromises cementitious materials, leading to environmental concerns and substantial maintenance costs. Although previous studies have shown that geopolymeric materials perform well in acidic conditions, the acid resistance of pressed geopolymers remains largely unexplored (Khan et al., 2020; Nnaemeka & Singh, 2020; Saif et al., 2022). Given the changes in geopolymer microstructure with high temperatures, it is essential to investigate how the sintering process and pressure compaction enhance geopolymer durability in aggressive acidic environments. Therefore, the acid resistance of sintered and unsintered pressed geopolymer in different types and concentrations of acids has to be evaluated discretely.

1.3 Objectives

This study served the purpose of elucidating the properties of fly ash-based pressed geopolymer with various mix proportions, pressing force, sintering temperature and aging period, as well as the acid resistance of sintered and unsintered fly ash-based pressed geopolymer. The objectives of the study are listed below:

i. To elucidate the influences of mix proportions (AS/AA ratio, NaOH concentration, Na_2SiO_3/NaOH ratio) and pressing force on the physical and mechanical properties of fly ash-based pressed geopolymer.
ii. To evaluate and compare the material properties of optimized fly ash-based pressed geopolymer and cast geopolymer.
iii. To examine the combined influence of aging period and sintering temperature on the properties of fly ash-based pressed geopolymer with optimized mix proportion and pressing force.
iv. To validate the acid resistance of sintered and unsintered fly ash-based pressed geopolymer with optimized aging period and sintering temperature.

1.4 Scope of Study

In this study, fly ash was used as the aluminosilicate source, with NaOH and Na_2SiO_3 solutions serving as alkali activator. This study was divided into four distinct stages. In the first stage, the focus was on optimizing the mix proportions and pressing force for the pressed geopolymer. Various samples were tested with different AS/AA ratios (4.5, 5.5, 6.5 and 7.0), NaOH concentrations (10, 12, 14 and 16 M), Na_2SiO_3/NaOH

ratios (1.0, 1.5, 2.0 and 2.5) and pressing forces (3, 4, 5 and 6 tons). The physical and mechanical properties of the fly ash-based pressed geopolymer were determined through analysis of bulk density, porosity, water absorption, compressive strength, flexural strength, stiffness and degree of reaction. Moreover, the microstructure of the pressed geopolymer was revealed by scanning electron microscopy (SEM), while the functional group was examined using Fourier transform infrared (FTIR) spectroscopy. The phase evolution of the pressed geopolymers was carried out using X-ray diffraction (XRD). Additionally, an analysis of variance (ANOVA) was performed to identify the significance of mix proportions and pressing force on the engineering properties.

The second stage involved a comparative study of the properties of cast and pressed geopolymers. The optimum mix proportion and pressing force of pressed geopolymer obtained from the first stage were used, while the mix proportion for cast geopolymer was based on findings from related studies. Various physical analyses (bulk density, porosity and water absorption), mechanical analysis (compressive and flexural strengths) and characterizations (SEM, elemental composition (EDX), FTIR, XRD and X-ray tomographic microscopy (XTM)) were conducted. The degree of reaction and tendency for efflorescence in both cast and pressed geopolymers were examined. The embodied energy (EE), embodied carbon dioxide emission (ECO_2) and embodied carbon index (ECI) of cast and pressed geopolymers were calculated and compared.

In the third stage, the optimized pressed geopolymer from the first stage was subjected to sintering. The combined effects of the aging period and sintering temperature on the performance of pressed geopolymer were evaluated by aging the samples for different periods (1, 7 and 28 days) before sintering at various temperatures (700, 800,

900 and 1000 ℃). The engineering properties of sintered pressed geopolymer, including linear shrinkage, mass loss, density loss, porosity, water absorption, compressive strength and flexural strength, were investigated. Characterizations were conducted using SEM, FTIR, XRD and XTM.

The fourth stage focused on the acid resistance of sintered and unsintered pressed geopolymer by immersing the samples in HNO_3 and H_2SO_4 solutions with different concentration levels (3 and 8%) for 7 and 28 days. The aging period and sintering temperature of sintered pressed geopolymer were selected based on the outcomes from the third stage. The acid resistance of sintered and unsintered pressed geopolymer was compared by carried out using various tests and analysis, including sorptivity, mass loss, compressive strength, flexural strength, Inductively coupled plasma (ICP) spectroscopy, Brunauer-Emmett-Teller (BET), SEM, EDX, XRD and Nuclear magnetic radiation (NMR) spectroscopy.