



**PROPERTIES OF ZEOLITE-BASED GEOPOLYMER FOAM REINFORCED
NANOCELLULOSE PREPARED IN LOW ALKALINE MEDIA**

By

TAY CHAI HUA

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
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January 2024

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The growing interest in geopolymer foam, driven by its exceptional properties and environmental benefits, presents promising prospects for diverse industrial applications. However, its reliance on highly concentrated alkaline solutions and fresh water poses significant limitations. Concentrated alkaline solutions are expensive, low in supply, and corrosive, while fresh water is becoming scarce globally. This study developed geopolymer foams using low molarity alkaline solutions and seawater to address these issues. The geopolymer consisted of aluminosilicate zeolite, a mixture of Potassium Silicate (KSi), below 2M Potassium Hydroxide (KOH), Potassium Chloride (KCl), and seawater as the alkaline solution, with Sodium Lauryl Ether Sulphate (SLES) and Benzalkonium Chloride (BAC) as surfactants to stabilize the foam produced by Hydrogen Peroxide (H_2O_2). Nanocellulose (NC) was used as reinforcement.

Geopolymerisation validation revealed successful depolymerization, reticulation, networking, and solidification of aluminosilicates, indicating that low molarity alkaline solution and seawater can effectively produce geopolymers. Response Surface Methodology (RSM) was used to statistically analyze the impact of each material on properties such as density, porosity, water absorption, and compressive strength. All four models displayed high R^2 values of more than 0.85, indicating that the chosen factors (SW/KSiI, KOH/KCl, SLES/BAC, and H_2O_2 /NC) effectively explain the variability in the tested properties. Optimization yielded a density of 1.691 g/cm³, porosity of 52.86%, water absorption of 43.106%, and compressive strength of 0.677 MPa, each with an average error below 15%. This study is the first to report on low molarity alkaline solution and seawater-based geopolymer foam, highlighting its potential as an eco-friendly alternative for various applications.

Keywords: Low molarity; Geopolymer Concrete, Nanocellulose, Response Surface Methodology, Seawater

SDG: GOAL 12: Responsible Consumption and Production

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**SIFAT-SIFAT GEOPOLIMER BERASASKAN ZEOLIT BERTETULANG
NANOSELULOSA YANG DISEDIAKAN DALAM MEDIA BERALKALI
RENDAH**

Oleh

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Minat yang semakin meningkat dalam geopolimer berbuis, didorong oleh sifat luar biasa dan faedah alam sekitar, memberikan prospek yang menjanjikan untuk pelbagai aplikasi industri. Walau bagaimanapun, kebergantungannya pada larutan alkali yang sangat pekat dan air tawar menimbulkan had yang ketara. Larutan beralkali pekat adalah mahal, rendah bekalnya, dan mengakis, manakala air tawar semakin berkurangan di seluruh dunia. Kajian ini menghasilkan geopolimer berbuis berasaskan larutan alkali kemolaran rendah dan air laut. Geopolimer terdiri daripada zeolit aluminosilika, campuran Kalium Silikat (KSil), Kalium Hidroksida (KOH) di bawah 2M, Kalium Klorida (KCl), dan air laut sebagai larutan alkali, dan surfaktan Natrium Lauril Eter Sulfat (SLES) and Benzalkonium Klorida (BAC) untuk menstabilkan busa yang dihasilkan oleh Hidrogen Peroksida (H_2O_2). Nanoselulosa (NC) digunakan sebagai tetulang.

Pengesahan geopolimerisasi mendedahkan kejayaan penyahpolimeran, retikulasi, rangkaian, dan pemejalan aluminosilikat, menunjukkan bahawa larutan alkali kemolaran rendah dan air laut boleh menghasilkan geopolymer dengan berkesan. Metodologi Permukaan Tindak Balas (RSM) digunakan untuk menganalisis secara statistik kesan setiap bahan terhadap sifat geopolimer seperti ketumpatan, keliangan, penyerapan air, dan kekuatan mampatan. Keempat-empat model memaparkan nilai R^2 yang tinggi iaitu lebih daripada 0.85, menunjukkan bahawa faktor yang dipilih (SW/KSi, KOH/KCl, SLES/BAC, dan H_2O_2 /NC) menerangkan dengan berkesan kebolehubahan dalam sifat yang diuji. Pengoptimuman menghasilkan ketumpatan 1.691 g/cm^3 , keliangan 52.86%, penyerapan air 43.106%, dan kekuatan mampatan 0.677 MPa, setiap satu dengan ralat purata di bawah 15%. Kajian ini adalah yang pertama melaporkan tentang geopolymer berbusar berasaskan larutan alkali kemolaran rendah dan air laut, menonjolkan potensinya sebagai alternatif mesra alam untuk pelbagai aplikasi.

Kata Kunci: Kemolaran rendah, Konkrit geopolimer, nanoselulosa, Metodologi permukaan tindak balas, air laut

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LIST OF ABBREVIATIONS

RSM	Response Surface Methodology
SW	Seawater
KSil	Potassium Silicate
KOH	Potassium Hydroxide
KCl	Potassium Chloride
SLES	Sodium Lauryl Ether Sulphate
BAC	Benzalkonium Chloride
H ₂ O ₂	Hydrogen Peroxide
NC	Nanocellulose
FTIR	Fourier Transform Infrared Spectroscopy
XRF	X-ray Fluorescence
XRD	X-ray Diffraction
FESEM-EDX	Field Emission Scanning Electron with Energy Dispersive X-Ray Spectroscopy
Na	Sodium
K	Potassium
Ca	Calcium
Al ₂ O ₃	Aluminium Oxide
NaOH	Sodium Hydroxide
Na ₂ O ₃	Sodium Silicate
K ₂ SiO ₃	Potassium Silicate
Si	Silica
Al	Alumina

CHAPTER 1

INTRODUCTION

1.1 Research Background

Geopolymers are networks of mineral molecules linked by covalent bonds (Davidovits, 2017; Davidovits, 2018). This inorganic polymer is produced mainly by mixing aluminosilicates with alkaline solution, forming a ceramic-like structure (Davidovits, 2017). By varying its main composition of aluminosilicates (Esparham et al., 2020) and alkaline solutions (Sore et al., 2020), and additional components such as water content (Vu et al., 2020) and reinforcement (Dheyaaldin et al., 2023), the properties of geopolymer can be tailored accordingly.

Initially, geopolymer was invented to develop a nonflammable and noncombustible plastic material (Davidovits, 2015a). Over time, geopolymer has evolved to exhibit similar or higher compressive strength compared to Ordinary Portland Cement (OPC) (Chowdhury et al., 2021), making it a greener, potential alternative to OPC, which emits high energy during its production (Amran et al., 2020). This is because the preparation of geopolymer avoids the “two grinding and one burning” process in the current OPC production (which are the raw material grinding, clinker calcination, and cement grinding) (Zhao et al., 2021). Governed by its environmentally friendly approach of using low embodied energy aluminosilicates, and low processing

energy, geopolymer has been developed as other materials such as ceramics (Mohd Mortar et al., 2022), coatings (Jiang et al., 2020), adhesives (He et al., 2011), and resin for composites (Ranjbar and Zhang, 2020), for potential applications in construction, manufacturing, and environmental engineering.

Geopolymer foam is an emerging material for similar applications mentioned beforehand but with higher porosity and therefore lower compressive strength. This is conducted by introducing porosity in the material through methods such as the addition of foaming agent (Hajimohammadi et al., 2018). Thus, geopolymer foam exhibits additional properties such as low density (Shakouri et al., 2020) and high-water absorption (Alnahhal et al., 2022) which expands its potential applications to lightweight concrete (Dhasindrakrishna et al., 2021) and evaporative cooling construction material (Emdadi et al., 2017). As such, this drives the escalating studies on the effect of variation in composition of geopolymer foam and its relation to the material's properties, such as density (Dhasindrakrishna et al., 2021), porosity (Gu et al., 2020), water absorption (Wang et al., 2020; Alnahhal et al., 2022) and compressive strength (Jaya et al., 2020; Polat and Güden, 2021). This reinforces geopolymer and its derivative, geopolymer foam, as a green and versatile material that deserves intensive research.

1.2 Problem Statement

Despite the mentioned environmentally friendly traits of geopolymer and significant research performed on the material, the commercialisation of the

technology is still in infancy (Shamsaei et al., 2021). This is due to the utilization of concentrated alkaline solution in geopolymer preparation (Abdollahnejad et al., 2015; Assi et al., 2018). Review papers have reported on high concentration of alkaline solution in conventional geopolymers ranging between 8M to 16M (Nakum and Arora, 2022) and 8M to 14M (Ng et al., 2018a). These alkaline solutions are selected for its ability to dissolve more aluminosilicate, leading to higher geopolymerisation and subsequently higher strength (Shilar et al., 2022; Farhan et al., 2020; John et al., 2021). This raises several issues: the deficit in current alkaline solution supply, the high cost in geopolymer preparation and the adverse effects on both human health and the environment due to their corrosive nature.

Currently, there is a deficit supply of alkaline solution worldwide. If the common geopolymer prepared in concentrated alkaline solution were to replace OPC globally, only 7.3% could be replaced due to the deficit in current Sodium Hydroxide (NaOH) supply (Assi et al., 2020). Additionally, the utilisation of current concentrated alkaline solution incurs high cost in geopolymer preparation, up to 139% of OPC (McLellan et al., 2011). According to Abdollahnejad, 80% of this cost is caused by the alkaline solution (Abdollahnejad, 2015). Moreover, the utilisation of such high concentration alkaline solutions posed risk both to humans and the environment. Potassium Hydroxide (KOH) solution with concentration exceeding 1.78M already qualifies as corrosive to humans (Statlab, 2024). This indicates that the preparation of conventional geopolymers presents a potential hazard to workers. In the environmental aspect, while the Carbon Dioxide (CO₂)

emission of geopolymer concrete is lower than OPC, its impact in categories such as ozone layer depletion, fresh water ecotoxicity, and photochemical oxidation surpasses OPC. These issues are caused by the production process of alkaline solution. This summation was made by comparing the 16M based geopolymer concrete to OPC (Habert et al., 2011).

Fresh water is a component of the geopolymer mixture, serving as the medium in which the alkaline solution is mixed and produced. However, according to the United Nations, the world is facing an imminent water crisis, with demand expected to outstrip the fresh water supply by 40% by the end of 2030 (United Nations, 2024). The building sector solely consumes up to 20% of the annual global water usage. This issue forces the United Nation Environment Programme (UNEP) to seek and identify opportunities and best practices for achieving greater water resource efficiency in the construction supply chain (UNEP, 2024). Additionally, the cost of construction in sea islands and remote coastal areas increases significantly due to the transportation expense for the major constituents such as fresh water, which are only available at a distance from the construction site (Thanh et al., 2022). Together, this highlights the issue of fresh water scarcity, exacerbating the challenges faced by the construction industry, particularly in regions where fresh water is limited. The combination of these two issues, the utilisation of concentrated alkaline solutions and the scarcity of fresh water pushes the need to innovate the current geopolymer material prepared in concentrated alkaline solution and fresh water.

To tackle the issue of concentrated alkaline solution, geopolymer foam may be prepared in low molarity alkaline solution, preferably KOH below 1.78M to eliminate the issue of corrosiveness on humans. However, there is limited research on the use of low molarity alkaline solution (Qin et al., 2022). In relation to the scarcity of fresh water, geopolymer foam may be prepared using seawater instead. Approximately 97% of the Earth's water is seawater. This suggests that the vast supply of seawater could serve as a substitute for fresh water, potentially reducing the dependency on the fresh water. While there is reported work on seawater based geopolymer (Sun et al., 2023), to the best of author's knowledge, there is no work reported on the combination of seawater based geopolymer foam prepared in low molarity alkaline solution. As a result, there is insufficient data on the properties of such geopolymers, which include density, porosity, water absorption, and compressive strength. Additionally, this leads to lack of studies on its microstructural and elemental analysis as well. Therefore, the use of statistical methods such as Response Surface Methodology (RSM) which facilitates statistical analysis, modelling of large data, optimization of responses and reduction of experimental run, would be great to contribute to a large dataset in the material domain at a lower material cost.

1.3 Research Objectives

The main objective is to develop a low molarity alkaline solution and seawater based geopolymer foam. In this study, the concentration of alkaline solution

used is below 1.62M, defining the term low molarity from hereon. Along with the main objective, the supporting objectives include:

1. To design experimental works for low molarity alkaline solution and seawater based geopolymer foam using RSM and statistically analyse the experimental data.
2. To conduct experiments on the density, porosity, water absorption and compressive strength of low molarity alkaline solution and seawater based geopolymer foam.
3. To analyse the physical, morphology, and chemical characteristics of low molarity alkaline solution and seawater based geopolymer foam.
4. To investigate the optimum range of composition for low molarity alkaline solution and seawater based geopolymer foam that produces the lowest density, highest porosity, highest water absorption and highest compressive strength.

1.4 Research Scope and Limitation

There are several scopes in this study. Firstly, the seawater used is collected in the foreshore area of Dataran 1 Malaysia, Melaka, Malaysia. Since the nominal composition of seawater varies globally (Ali et al., 2016), the discussion on the effect of seawater on the properties of low molarity alkaline solution and seawater based geopolymer foam is limited to only the seawater collected from this specific location.

Secondly, the design of the experiment is based on four factors, involving eight materials which are seawater/Potassium Silicate (SW/KSil), Potassium Hydroxide/Potassium Chloride (KOH/KCl), Sodium Lauryl Ether Sulphate/Benzalkonium Chloride (SLES/BAC) and Hydrogen Peroxide/Nanocellulose (H_2O_2 /NC). Each of the factors are designed with their respective low and high levels, which scopes their range of loading. The ratio of SW/KSil is designed at 1 to 1.2, KOH/KCl is designed at 20%/80% to 100/0% of 10 wt% of KSil, SLES/BAC is designed at 0%/100% to 100%/0% of 0.05 wt% Basic Geopolymer Slurry (BGS) and H_2O_2 /NC is designed at 0.4 wt% of BSG. As a result, the concentration of KOH solution studied is between 0.32M and 1.62M.

Thirdly, the mechanical characterization of samples is focused on only the compressive test. Given the anticipated low strength of the material, the characterization is focused on physical properties such as density, porosity, and water absorption, which are significant for application of low strength geopolymer foam.

In this study, there is a limitation in the porosity analysis conducted using water immersion method. Since there is a minimum pore size of which water can pass through, there may be tiny pores that are not measurable through this method, thereby limiting this analysis to only pores that are accessible to water. Assumption also has to be made, such that there are no pores that are inaccessible to water.

1.5 Thesis Organization

This thesis consists of five chapters. Chapter One underlines the research background, problem statement, research objectives, research scope and limitation, and thesis organisation.

Chapter Two presents the literature review on the fundamentals of geopolymer material, encompassing the main materials used for the preparation, as well as the processes involved in the preparation. Additionally, the issue with using concentrated alkaline solution and fresh water in the preparation of geopolymer is highlighted here, followed by the research gap in the current research area, thereby paving way for this study.

Chapter Three presents the properties of all the raw materials used in the geopolymer foam fabrication, describes the experimental design under RSM, explains the sample preparation set-up, and presents the methods in material characterization.

Chapter Four presents the results and discussion of the study. It begins with a scoping analysis for RSM to identify suitable factors and parameters. Subsequently, a geopolymerization validation analysis was conducted on all RSM samples. This was followed by a statistical analysis of each response: density, porosity, water absorption, and compressive strength, supplemented by microscopic images. Optimization was then performed to find the composition that resulted in the lowest density, highest porosity, highest water

absorption, and highest compressive strength. Finally, superimposition was carried out to determine the composition that achieved the desired combination of responses.

Chapter Five concludes the whole study and suggests recommendations for future research.



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