Development of One-Part Geopolymer Using Fly Ash and Ladle Furnace Slag for Microwave Absorption

by

Hang Yong Jie
(2140413428)

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LIST OF ABBREVIATIONS

5G	5th generation mobile network
Al	Aluminum
Al_2O_3	Aluminum oxide
ANOVA	Analysis of variance
ASTM	American Society of Testing and Materials
BOFS	Blast oxygen furnace slag
Ca	Calcium
CaO	Calcium oxide
C-A-S-H	Calcium-aluminate-silicate-hydrate
CO_2	Carbon dioxide
C-S-H	Calcium-silicate-hydrate
CST-MWS	Computer Simulation Technology-Microwave Studio
EAFS	Electric arc furnace slag
FA	Fly ash
Fe	Iron
Fe_2O_3	Iron oxide
FTIR	Fourier Transform Infrared
GGBS	Ground granulated blast furnace slag
H	Hydrogen
ITZ	Interfacial transition zone
K	Potassium
LFS	Ladle furnace slag
Mg	Magnesium
MgO	Magnesium oxide
Na	Sodium
Na_2CO_3	Sodium carbonate
Na_2O	Sodium oxide
Na_2SiO_3	Sodium metasilicate
$Na_2SiO_3 \cdot 5H_2O$	Sodium metasilicate pentahydrate
$Na_2SiO_3 \cdot 9H_2O$	Sodium metasilicate hexahydrate
$NaAlO_2$	Sodium aluminate
NaOH	Sodium hydroxide

N-A-S-H	Sodium-aluminate-silicate-hydrate
NMR	Nuclear magnetic resonance
O	Oxygen
OH	Hydroxide
OPC	Ordinary Portland cement
OPG	One-part geopolymer
pH	Potential of hydrogen
Pt	Platinum
S_{11}	Reflective coefficient
S_{21}	Transmission coefficient
SEM	Scanning Electron Microscopy
Si	Silicon
SiO_2	Silicon oxide
UTM	Universal Testing Machine
Wi-Fi	Wireless Fidelity
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
XTM	X-ray Tomography
µXRF	Micro X-ray Fluorescence

LIST OF SYMBOLS

%	Percentage (mathematical symbol)
°	Degree (unit of angle)
°C	Degree Celsius (unit of temperature)
2θ	2-theta (physical symbol)
cm	Centimeter (unit of diameter or length)
cm^{-1}	Per centimeter (unit of wavenumber)
dB	Decibel (unit of power or signal intensity)
f	Frequency
g	Gram (unit of mass)
g/cm^{3}	Gram per cubic centimeter (unit of bulk density)
GHz	Gigahertz (unit of frequency)
h	Hours (unit of time)
Hz	Hertz (unit of frequency)
kHz	Kilohertz (unit of frequency)
kV	Kilovolt (unit of voltage)
mA	Milliampere (unit of current)
MHz	Megahertz (unit of frequency)
min	Minute (unit of time)
mm	Millimeter (unit of distance or length)
mm^{3}	Cubic millimeter (unit of volume)
MPa	Megapascal (unit of strength)
ppm	Parts per million (unit of concentration)
rpm	Revolution per minute (unit of rotational speed)
$\tan \delta\varepsilon$	Loss tangent
wt.%	Weight percentage (unit of mass as a percentage of the total mass)
ε'	Dielectric constant
ε''	Dielectric loss
μ'	Magnetic constant
μ''	Magnetic loss
μm	Micrometer (unit of diameter or length)
λ	Wavelength
σ	Conductivity

Pembangunan Geopolimer Satu-Bahagian Menggunakan Abu Terbang dan Sanga Relau Senduk untuk Penyerapan Gelombang Mikro

ABSTRAK

Bahan bangunan yang menyerap gelombang mikro adalah penting pada zaman moden disebabkan oleh pembangunan teknologi elektronik dalam persekitaran bandar. Komposit simen sering digunakan; namun, pengeluarannya melibatkan penggunaan tenaga dan pelepasan karbon yang tinggi, menyebabkan isu-isu alam sekitar. Penambahan sisa industri juga menjadi cabaran kritikal pada masa kini. Oleh itu, geopolimer satu-bahagian, yang disintesis daripada abu terbang (FA) dan sanga relau senduk (LFS), digunakan untuk mengatasi masalah ini. Kesan daripada nisbah campuran seperti pengaktif beralkali/aluminosilikat (AA/AS), natrium metasilikat/natrium hidroksida (SM/SH), dan air/pepejal (W/B) ke atas prestasi geopolimer telah disiasat menggunakan teknik faktorial penuh 3^3. Geopolimer yang optimum diperolehi pada AA/AS 0.2, SM/SH 5.0, dan W/B 0.25. Analisis varians menunjukkan bahawa nisbah AA/AS, W/B, dan AA/AS-W/B mempunyai kesan yang paling ketara kepada sifat-sifat fizikal and mekanikal, manakala nisbah SM/SH kurang mempegaruhi. Analisis mikrostruktur dan fasa memaparkan kehadiran kalsium-silikat-hidrat (C-S-H), menyumbangkan integriti struktur dan kekuatan. Selain itu, pengaruh frekuensi, masa penuaan, dan nisbah campuran terhadap sifat dielektrik dan penyerapan gelombang mikro telah dinilai. Sifat dielektrik geopolimer berkurangan dengan peningkatan frekuensi (2 – 18 GHz), menghasilkan penyerapan daripada 50.0% – 90.0%. Secara khusus, penyerapan gelombang mikro meningkat dan mencapai tahap tetap selepas 8 GHz. Apabila geopolimer melalui penuaan hingga 28 hari, sifat dielektrik berkurangan, namun penyerapan gelombang mikro meningkat melebihi 80.0%. Dengan nisbah campuran yang optimum, geopolimer mempamerkan kekuatan dan penyerapan gelombang mikro yang tinggi. Seterusnya, kesan ketebalan dan pemisahan antena terhadap penyerapan gelombang mikro telah diperiksa untuk memaksimumkan kemampuan geopolimer. Geopolimer dengan ketebalan 100.0 mm dan diletakkan pada pemisahan antena 20.0 mm meningkatkan penyerapan gelombang mikro dari 60.0% ke 80.0%, disebabkan oleh peningkatan pantulan berganda dan pelepasan tenaga dalam struktur. C-S-H menggalakkan kesan polarisasi, mengurangkan sifat dielektrik dan menggalakkan penyerapan gelombang mikro geopolimer satu-bahagian. Tambahan pula, kesan kandungan agregat terhadap kekuatan dan penyerapan gelombang mikro geopolimer telah dikaji. Pasir sungai dan kerikil digunakan untuk menghasilkan mortar dan konkrit. Geopolimer mortar dengan 60.0% agregat dan geopolimer konkrit dengan 75.0% agregat mencapai kekuatan maksimum 41.7 dan 52.9 MPa. Ini dikaitkan dengan kesan *interlock* mekanikal dan pengisian liang oleh agregat. Walaupun agregat meningkatkan sifat dielektrik, mereka menggalakkan padanan impedans, kesan polarisasi, dan penyebaran gelombang, menghasilkan penyerapan gelombang mikro ~90.0%. Kajian ini menyediakan pandangan untuk pengoptimuman geopolimer satu-bahagian FA-LFS dan meningkatkan pemahaman dalam komuniti geopolymer. Kajian ini juga menawarkan pilihan lestari untuk membina bahan bangunan yang mampu menyerap gelombang mikro, terutamanya sesuai untuk kawasan dengan rangkaian Wi-Fi dan 5G yang tinggi.

Development of One-Part Geopolymer Using Fly Ash and Ladle Furnace Slag for Microwave Absorption

ABSTRACT

Microwave-absorbing building materials are essential in modern times due to the proliferation of electronic technologies in urban environments. Cement composites are commonly used; however, their production involves high carbon emissions and energy consumption, leading to environmental issues. Besides, the abundance of industrial waste has become a critical challenge today. Hence, one-part geopolymers, which were synthesized by fly ash (FA) and ladle furnace slag (LFS), were employed to overcome these issues. The effects of mixing ratios such as alkali activator/aluminosilicate source (AA/AS), sodium metasilicate/sodium hydroxide (SM/SH), and water/binder (W/B) on the performances of one-part geopolymer were investigated using 3^3 full factorial design. An optimal one-part geopolymer was obtained at AA/AS of 0.2, SM/SH of 5.0, and W/B of 0.25. Analysis of variance indicated that AA/AS, W/B, and AA/AS-W/B ratios had the most significant impact on the physical and mechanical properties, while the SM/SH ratio was less influential. Microstructural and phase analyses revealed the presence of calcium-silicate-hydrate (C-S-H), contributing to structural integrity and strength development. Besides, the influences of frequency level, ageing time, and mixing ratios on dielectric properties and microwave absorption were evaluated. The dielectric properties of the geopolymers were reduced with increasing frequency levels (2 – 18 GHz), resulting in favorable microwave absorptions of 50.0% – 90.0%. Specifically, the microwave absorption increased and reached a plateau beyond 8 GHz. As the geopolymers aged 28 days, the dielectric properties diminished, yet microwave absorption rose by over 80.0%. With the optimal mixing ratios, the one-part geopolymers excelled in mechanical strength and microwave absorption. Additionally, the effects of thickness and antenna separation on microwave absorption were examined to further maximize the geopolymers' capabilities. The geopolymers with a thickness of 100.0 mm and antenna separation of 20.0 mm improved microwave absorption from 60.0% to 80.0% due to the increased multiple reflections and energy dissipation within the structure. The C-S-H enhanced polarization effects, reducing the dielectric properties and boosting the microwave absorption of one-part geopolymers. Moreover, the impact of aggregate content on compressive strength and microwave absorption of the geopolymers was explored. River sand and gravel were used as aggregates to produce one-part geopolymer mortars and concretes. The geopolymer mortar with 60.0% aggregates and the geopolymer concrete with 75.0% aggregates obtained maximum strengths of 41.7 and 52.9 MPa, respectively. This was attributed to the mechanical interlocking and pore-filling effects of the aggregates. Although the aggregate increased the dielectric properties, they promoted impedance matching, polarization effects, and wave dispersion, resulting in a ~90.0% microwave absorption. This research provided valuable insights for optimizing FA-LFS one-part geopolymers and enhanced the understanding within the geopolymer community. This work also offers a sustainable option for constructing building materials capable of absorbing microwaves, especially suitable for areas with extensive Wi-Fi and 5G networks.

CHAPTER 1 : INTRODUCTION

1.1 Research Background

The worldwide demand for ordinary Portland cement (OPC) has resulted in a substantial increase in the release of carbon dioxide (CO_2), contributing approximately 5.0% – 8.0% to global CO_2 emissions (Andrew, 2019). In the context of Malaysia, 20.0 million tons of OPC produced 17.8 million tons of CO_2 (Mohammed et al., 2023). Furthermore, the manufacturing process of OPC is energy-intensive, consuming 2.9 – 4.6 GJ/ton of OPC produced (Wolde et al., 2024). These statistics have raised impacts regarding the environmental (such as global warming and climate change) and economic (such as cost volatility and increased regulatory costs) on the construction sector. In response, the United Nations proposed 17 Sustainable Development Goals (SDGs) in 2015, particularly SDG 12 (Responsible Consumption and Production) and SDG 13 (Climate Action), to promote sustainable practices in the construction sector (Shehata et al., 2022). The adoption of strategies such as repurposing industrial waste materials and minimizing CO_2 emissions throughout the manufacturing process could significantly contribute to the fulfillment of SDGs 12 and 13.

Numerous scholars have advocated a sustainable approach using geopolymer technology as a substitute for OPC, highlighting its potential to reduce CO_2 emissions and energy consumption by 60.0% to 80.0% (Chen et al., 2023b; Pasupathy et al., 2023). Traditional geopolymer, or two-part geopolymer, is generally composed of aluminosilicate sources mixed with alkaline solutions. The aluminosilicate sources are typically derived from industrial waste materials like fly ash (FA) and ground granulated blast slag (GGBS), whereas the alkaline solutions include alkali hydroxide and alkali silicate (Krishna et al., 2021). Extensive research has confirmed the exceptional mechanical and durability properties of two-part geopolymers (Nuaklong et al., 2021; Pasupathy et al., 2021; Saludung et al., 2023). Nevertheless, due to safety concerns, the corrosive nature of alkaline solutions used in two-part geopolymers limits their applications to smaller scales. This has led to the exploration of one-part geopolymers, which require only the introduction of water to a dry mix of aluminosilicate sources and alkali activators. The one-part geopolymers offer safer handling and broader application potential (Oderji et al., 2019a).

Researchers have explored various aluminosilicate sources and alkali activators in synthesizing one-part geopolymer, tailoring its mixing compositions to adapt diverse applications like structural and non-structural building components, road bases, and absorbents for heavy metal removal (Mohammed et al., 2019; Shah et al., 2021). Specifically, the mixture of FA and GGBS was introduced in the development of one-part geopolymer (Ma et al., 2023; Wang et al., 2021). The higher calcium (Ca) content of GGBS promoted the chemical interaction with FA in an alkaline environment, enhancing the mechanical performance of the one-part geopolymers. Ladle furnace slag (LFS), with even greater Ca content than GGBS and less frequently used in geo-synthesis, facilitates the creation of hydration gels known as calcium-silicate-hydrate (C-S-H). These gels can refine the morphology and enhance the mechanical strength of the geopolymers (Hui-Teng et al., 2021; Yong-Sing et al., 2021). Moreover, prior research discovered that optimal alkali activator/aluminosilicate source (AA/AS), sodium metasilicate/sodium hydroxide (SM/SH), and water/binder (W/B) ratios were crucial and indispensable, making them significant to discuss (Sadeghian et al., 2022; Wan-En et al., 2021; Zhang et al., 2021).

Numerous researchers have explored the application of design of experiment (DOE) methodologies in geopolymer optimization (Driouich et al., 2023; Maaze & Shrivastava, 2023). DOE represents an applied statistical approach employing mathematical models to systematically conduct the experiments, enabling the examination of data to reach a robust conclusion. Various DOE techniques are commonly utilized, including full factorial design, response surface methodology, and Taguchi method (Bingol et al., 2010; Hadi et al., 2017; Rivera et al., 2019). DOE offers several advantages, such as cost-effectiveness and time efficiency due to reduced experimental runs, and it facilitates the examination among the factors (Bingol et al., 2010). Furthermore, DOE is particularly valuable for preliminary studies which involve multiple factors. It incorporates several statistical tools to analyze results, including analysis of variance (ANOVA), main and interaction effect plots, as well as response optimization.

The extensive use of electronic equipment in wireless networking and communication technologies, such as Wireless-Fidelity (Wi-Fi) and 5th generation (5G) mobile networks, has contributed to a remarkable increase in microwave interference within a building space. Past literature has demonstrated several effective methods for tackling microwave interferences, which include the utilization of microwave-shielding and microwave-absorbing materials (Bai et al., 2022; Sun et al., 2023). Microwave-shielding material acts as a barrier to restrict the transmission of microwaves, but it causes a secondary reflection of microwaves, implying that it is ineffective at minimizing or eliminating microwave interferences (Xie et al., 2020). Besides, microwave-absorbing material is highly efficient for attenuating microwave waves; it operates by dissipating the microwaves within the structure, causing a substantial decrease in microwave interferences (Elmahaishi et al., 2022). Therefore, a building material with microwave-absorbing ability can minimize microwave interference and improve the microwave environment in an enclosed area, reducing the potential risks to human health and electrical equipment.

One-part geopolymer is recognized as a microwave-absorbing building material owing to its unique dielectric properties. The relationship between the dielectric properties and microwave absorption garners considerable interest, noting that greater microwave absorption is associated with a lower dielectric constant (ε') and a higher dielectric loss (ε") (Ayeni et al., 2022). Malkawi et al. (2018) reported that the dielectric properties of FA-based two-part geopolymers are influenced by frequencies (1 kHz – 30 MHz), time after mixing (24 hours), and mixing ratios (solid-to-liquid ratio and alkaline molarity). The key findings revealed that the porous nature of the two-part geopolymers introduced more air gaps, influencing the dielectric properties. Despite these, research has yet to explore the effects of higher frequencies (GHz), prolonged ageing times (7 – 28 days), and one-part geopolymer mixing ratios on the dielectric properties and microwave absorption. This underscores the importance of understanding the influence of these aspects on the dielectric properties and microwave absorption of one-part geopolymers in the current study.

Thickness has emerged as a crucial study in the investigation of microwave absorption in the realm of one-part geopolymer. Shi et al. (2022) and Wang et al. (2023) were revealed that altering the thicknesses significantly affects microwave absorption due to impedance matching and elongation of the transmission path. An optimal impedance matching enabled maximum microwave propagation, while an extended transmission path enhanced microwave attenuation. Additionally, antenna separation plays a significant role in microwave absorption, reflecting the source-receiver distance in real-world scenarios. The electromagnetic absorption rate is inversely related to the distance between the transmitter and receiver, drawing attention in optimizing the distance to minimize public microwave exposure (Bhargava et al., 2019; Ramachandran et al., 2019). However, past research only provided very limited insight into the impacts of thickness and antenna separation on microwave absorption, leaving a significant knowledge gap in understanding their potential in one-part geopolymers.

Previous research has demonstrated the inclusion of aggregates on the mechanical performance of geopolymers (Prusty & Pradhan, 2022; Stefaniuk et al., 2022), but there is a notable lack of research on the effect of aggregates on geopolymers' microwave absorption. Earlier investigations have mainly explored the incorporation of titanium-oxide (TiO_2) aggregate, carbon-based materials, and ferrite to boost microwave absorption, particularly geopolymers (Bai et al., 2022; Lu et al., 2017; Ma et al., 2020b; Sun et al., 2023). However, the geopolymers with such aggregates cause the disadvantages of the laborious and expensive preparation procedure. To address these challenges, this study aimed to utilize locally available aggregates, such as river sand and gravel, in the one-part geopolymer mortars and concretes. This aspect represents a gap in current research, especially in providing detailed guidance on the optimal aggregate content for maximizing microwave absorption. Thus, it is essential to understand the microwave absorption of the one-part geopolymer mortars and concretes and to determine the most effective aggregate content for microwave absorption.

To recap, this study focused on the development of FA-LFS one-part geopolymers by utilizing FA and LFS, as their existing incorporation is still limited. The dielectric properties were vital for examining microwave absorption; hence, the dielectric properties of the FA-LFS one-part geopolymers were evaluated in relation to frequency, ageing time, and mixing ratios. The influences of thickness and antenna separation were studied to determine their effectiveness on microwave absorption of the FA-LFS one-part geopolymers. Lastly, the effect of aggregate content is important to evaluate the mechanical properties and microwave absorption of the FA-LFS one-part geopolymers. In short, all of the experimental efforts were aimed at developing high-performance, sustainable microwave-absorbing building materials and directed towards expanding knowledge within the geopolymer community.

1.2 Problem Statements

The AA/AS ratio is a key part of the geopolymerization reaction. The alkali cations undergo ion exchange with aluminosilicate sources to generate a stable geopolymer network. The efficacy of the one-part geopolymers is governed by the alkali concentration since higher alkalinity may promote the dissolution rate of the aluminosilicate sources. Besides, the SM/SH ratio affects the polycondensation and has a direct relationship to the mechanical strength of the one-part geopolymers. The optimal silicon (Si) increases the rate of geopolymer network formation within a system. In addition, the W/B ratio is important in regulating the workability of the mixture to achieve the desired performance of the one-part geopolymers. Additionally, the water influences the flowability of the geopolymer mixtures. Here, studies on the effects of AA/AS, SM/SH, and W/B ratios in one-part geopolymer are essential for further improvement of its effectiveness in building and construction applications.

The significance of the frequency level lies in its varied interactions within the geopolymer structure, which in turn alters its dielectric properties. Moreover, ageing time and mixing ratios (AA/AS, SM/SH, and W/B) contribute to moisture content and microstructural evolution, both of which are critical to the dielectric properties of the one-part geopolymers. Exploring the effects of frequency, ageing time, and mix ratios on the dielectric properties can significantly impact the microwave-absorbing ability of the one-part geopolymers. Therefore, it is important to understand the influence of frequency, ageing time, and mixing ratios on dielectric properties and microwave absorption to advance their application in microwave-absorbing building and construction materials.

In addition, the thickness and the antenna separation are two important criteria for the practicality of microwave-absorbing building materials. The thickness of the one-part geopolymers limits their microwave-absorbing capabilities. Thin geopolymers provide better impedance matching but lack effectiveness in microwave absorption. This reveals that the thin geopolymers have higher uncertainty, which leads to limitations in practical applications. Antenna separation has greatly influenced the microwave absorption of the one-part geopolymers. The longer the distance, the lower the microwave absorption rate, which is due to the electromagnetic radiation distributed to the surrounding area and the signal loss along the path. Hence, understanding the microwave absorption of the one-part geopolymers with various parameters is a good approach for practical use in the building and construction field.

The introduction of functional aggregates into the one-part geopolymers is to increase their microwave absorption. TiO_2-based aggregate, carbon-based dopant, and ferrite are the most commonly utilized, attributed to their excellent electromagnetic transparency. Nonetheless, the high cost of these aggregates has restrained their large-scale applications in one-part geopolymer production. In this sense, it is a necessity to determine an alternative for one-part geopolymer technology from a construction perspective. The locally available aggregates like river sand and gravel appear as an alternative cost-effective incorporation for the dielectric dopants due to their electromagnetic performance and high availability. Besides, the aggregate content will affect the physical, mechanical, and microstructural properties of one-part geopolymers. Thus, the exploration of different aggregate contents is important for effective microwave absorption and superior mechanical strength in microwave-absorbing building and construction materials.

1.3 Research Objectives

The primary purpose of this study is to evaluate the mechanical strength and microwave absorption of one-part geopolymer synthesized from FA and LFS. Additionally, this study encompasses a comprehensive analysis of various parameters affecting the performance of the FA-LFS one-part geopolymer. The research objectives are listed below:

i) To investigate the effects of various mixing ratios (AA/AS, SM/SH, and W/B) on FA-LFS one-part geopolymer using a 3^3 full factorial approach.

ii) To examine the effects of frequency, ageing time, and mixing ratios on dielectric properties and microwave absorption of FA-LFS one-part geopolymer.

iii) To evaluate the effects of thickness and antenna separation on microwave absorption of FA-LFS one-part geopolymer.

iv) To elucidate the effect of aggregate content on the physical properties, mechanical properties and microwave absorption of FA-LFS one-part geopolymer, with a comparative study among the geopolymer paste, mortar, and concrete.

1.4 Research Scopes

In this research, FA and LFS were utilized as the aluminosilicate sources, and sodium metasilicate (Na_2SiO_3) and sodium hydroxide (NaOH) were used as the alkali activators for the synthesization of FA-LFS one-part geopolymers. The study was divided into four main stages. Firstly, the parameter study was performed to obtain the optimal mixing ratios of FA-LFS one-part geopolymers. The experiment was conducted through a 3^3 full factorial design. The parameters included the AA/AS (0.10, 0.15, and 0.20), SM/SH (3.0, 4.0, and 5.0), and W/B (0.20, 0.25, and 0.30) ratios. The physical and mechanical properties of the FA-LFS one-part geopolymers, such as bulk density, water absorption, apparent porosity, and compressive strength, were determined. Besides, a series of statistical techniques, including ANOVA, main effect, interaction effect, and response optimization, were performed to identify the significance of the mixing ratios on the performance of the FA-LFS one-part geopolymers. Moreover, the microstructure of the FA-LFS one-part geopolymers was revealed by Scanning Electron Microscope (SEM). The examinations of the phase and functional group of the FA-LFS one-part geopolymers were carried out using X-ray Diffraction (XRD) and Fourier Transform Infrared (FTIR).