MECHANICAL, DURABILITY AND MICROSTRUCTURAL PERFORMANCE OF KENAF FIBRE-REINFORCED GEOPOLYMER COMPOSITES

By

ALGHAZALI NOOR ABBAS NAJM

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December 2022

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Ordinary Portland Cement (OPC) is the main ingredient in concrete, which is commonly used in construction applications because of its excellent mechanical and durability performances. However, concrete and cement-based products produce greenhouse gas emissions, which gives a bad impact on the environment. Hence the development of greener construction materials is in need. Recently, many researchers have focused on geopolymer materials, which improve the greenness of conventional concrete with comparable or better characteristics.

Geopolymers have good mechanical properties, are non-flammable, acid-resistant, long-lasting, and have fewer CO_2 emissions than OPC. However, when subjected to flexure and tension stresses, the brittleness of geopolymer composites (GPC) is inherent. Therefore, natural fibres, kenaf is introduced to improve the brittleness, control crack propagation, and enhance flexural and tensile strength. This composite is abbreviated as Kenaf Fibre-Reinforced Geopolymer Concrete Composites (KFRGC) in this thesis. This research studied the KFRGC with kenaf fibre volume fractions of 0.75, 1.0, 1.25, and 1.5%, and fibre lengths of 20, 30, and 40mm. The fibres are mixed with 40 MPa geopolymer composite with fly ash (FA) and ground granulated blast slag (GGBS) as binders. A series of standard material tests were conducted to determine the impact of kenaf fibres on the fresh, mechanical, durability and microstructural properties of KFRGC.

The results demonstrated that the workability and unit weight of KFRGC reduced as the fibre volume fraction and length increased. The inclusion of higher volume fractions and longer lengths of kenaf fibres did not improve the compressive strength and modulus of elasticity at all ages but give higher tensile strength and flexural strength. The KFRGC prepared with 1.25% fibre volume fraction (V_f) and 30 mm long obtained the highest

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tensile and flexural strength with an improvement of about 20% and 27%, respectively, compared to the plain geopolymer. This improvement was justified by the strong fibre-matrix interfacial adhesion properties, which transferred a high amount of stress from the matrix to the fibre, as evidenced by the FESEM images. The specimens showed a triangular failure pattern with oblique cracks on brittle geopolymer without fibres, while retaining their original shape with a few vertical cracks and fissures on KFRGC samples. This shows that, although natural fibres have lower tensile strength than synthetic fibres, it is sufficient to alter the failure mode of GPC from brittle to ductile.

In terms of durability, KFRGC showed that increasing the fibre length or volume will impact their performance. All durability and water resistance tests showed that longer and higher V_f of KF caused fibre balling during mixing and resulted in lower properties of KFRGC. In conclusion, the optimum kenaf fibre length and V_f recommended for KFRGC is 30 mm and 1.0%, respectively for use as indoor composite green materials. This research paves the way for kenaf addition in geopolymer concrete as a better option of a potential green material natural fibre than coconut, which has less tensile strength. This hopefully contributes to an option of green concrete in construction industries.

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

PRESTASI MEKANIKAL, KETAHANAN DAN MIKROSTRUKTUR KOMPOSIT GEOPOLIMER BERTULANG-KENAF GENTIAN

Oleh

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Simen Portland Biasa (OPC) adalah bahan utama dalam konkrit, yang biasa digunakan dalam aplikasi pembinaan kerana prestasi mekanikal dan ketahanan yang sangat baik. Walau bagaimanapun, produk berasaskan konkrit dan simen menghasilkan pelepasan gas rumah hijau, yang memberi kesan buruk kepada alam sekitar. Justeru pembangunan bahan binaan hijau amat diperlukan. Pada masa kini, ramai penyelidik memberi tumpuan kepada bahan geopolimer, yang meningkatkan kehijauan konkrit konvensional dengan ciri yang setanding atau lebih baik.

Geopolimer mempunyai sifat mekanikal yang baik, tidak mudah terbakar, tahan asid, tahan lama, dan mempunyai kurang pelepasan CO_2 daripada OPC. Walau bagaimanapun, apabila ia dikenakan dengan tegasan lentur dan tegangan, GPC akan menjadi rapuh. Oleh itu, gentian asli, iaitu kenaf, diperkenalkan untuk memperbaiki kerapuhan, mengawal perambatan retak, dan meningkatkan kekuatan lentur dan tegangan. Komposit ini disingkatkan sebagai Komposit Geopolimer Bertetulang Gentian Kenaf (KFRGC) dalam tesis ini. Penyelidikan ini mengkaji KFRGC dengan penggunaan pecahan isipadu (V_f) yang berbeza iaitu sebanyak 0.75, 1.0, 1.25%, dan 1.5%, serta panjang adalah 20, 30, dan 40mm. Gentian dicampur dengan komposit geopolimer 40 MPa dengan abu terbang (FA) dan sanga bagas butiran tanah (GGBS) sebagai pengikat. Satu siri ujian piawai bahan telah dijalankan untuk menentukan kesan gentian kenaf terhadap sifat baru, mekanikal, ketahanan dan mikrostruktur bagi KFRGC.

Keputusan menunjukkan bahawa kebolehkerjaan dan berat unit KFRGC berkurangan apabila V_f gentian meningkat, manakala kesan panjang terhadap ketinggian slump adalah tidak ketara. Peningkatan V_f serta panjang gentian kenaf tidak meningkatkan kekuatan mampatan dan modulus keanjalan pada semua peringkat umur tetapi, meningkatkan kekuatan tegangan dan kekuatan lenturannya. KFRGC yang mengandungi 1.25% V_f dan 30 mm panjang memperoleh kekuatan tegangan dan

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lenturan tertinggi dengan peningkatan masing-masing kira-kira 20% dan 27% berbanding dengan geopolimer biasa. Peningkatan ini diwajarkan lagi oleh sifat lekatan antara muka gentian-matriks yang kuat, yang memindahkan jumlah tegasan yang tinggi dari matriks ke gentian, seperti yang dibuktikan oleh imej FESEM. Spesimen menunjukkan corak kegagalan segi tiga dengan rekahan serong pada GPC rapuh, dengan mengekalkan bentuk asalnya dengan beberapa retakan menegak dan rekahan pada sampel KFRGC. Ini menunjukkan bahawa, walaupun gentian asli mempunyai kekuatan tegangan yang lebih rendah daripada gentian sintetik, ia memadai untuk mengubah mod kegagalan daripada rapuh kepada mulur.

Dari segi ketahanan, KFRGC menunjukkan peningkatan panjang dan V_f gentian akan memberi impak kepada prestasinya. Semua ujian ketahanan dan rintangan air menunjukkan lebih panjang dan lebih tinggi kandungan V_f gentian akan menyebabkan bebola gentian semasa pengadunan dan mengakibatkan penurunan prestasi ketahanan KFRGC. Kesimpulannya, panjang dan kandungan V_f optimum gentian kenaf yang disyorkan untuk KFRGC ialah 30 mm dan 1.0%, masing-masing untuk digunakan sebagai bahan hijau komposit dalaman. Penyelidikan ini membuka jalan kepada penggunaan kenaf sebagai bahan tambahan dalam konkrit geopolimer sebagai pilihan yang lebih baik daripada gentian semulajadi lain seperti kelapa, yang mempunyai kekuatan tegangan yang lebih rendah. Ini diharapkan dapat menyumbang kepada pilihan konkrit hijau dalam industri pembinaan.

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LIST OF ABBREVIATIONS

OPC	Ordinary Portland Cement
CO_2	Carbon Dioxide
KFRGC	Kenaf Fibre-Reinforced Geopolymer Concrete Composites
GPC	Geopolymer Composite
KF	Kenaf Fibre
NF	Natural Fibre
FRGC	Fibre-Reinforced Geopolymer Composite
KFRC	Kenaf Fibre-Reinforced Cement-Based Concrete
ST	Steel Fibre
SY	Synthetic Fibre
Inor	Inorganic Fibre
PPF	Polypropylene
PVA	Polyvinyl alcohol
GF	Glass Fibre
SEM	Scanning Electron Microscopy
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-ray spectroscopy
ITZ	Interfacial Transition Zone
V_f	Volume fraction
µm	Micron

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CHAPTER 1

INTRODUCTION

1.1 Background

Cement industries, which produce the main component of concrete continue to be one of the biggest challenges facing the world, as it contributes significantly to climate change and possesses a negative impact on the earth. According to Lemougna et al. (2020), above 4 billion metric tons of ordinary Portland cement (OPC) is produced annually, and for every ton of OPC, 1 ton of Carbon dioxide (CO_2) emits to the atmosphere. Besides, the production of OPC consumes 10-11 EJ of energy yearly, which is about 3% of global prime energy use (Riofrio et al., 2022; Narayanan & Shanmugasundaram, 2018).

CO_2 is one of the greenhouse gases that act like a blanket wrapped around the earth, trapping the sun's heat, and raising temperatures. Currently, the greenhouse gas levels are at the highest in 2 million years and are continuously increasing. As a result, the world has warmed by around 1.1°C since the 1800s according to the report of the United nation (2022). The previous ten years were the warmest on record. The increase in temperature will influence changes in all other aspects of the Earth system in which everything is connected. Major fires, melting polar ice, rising sea levels, flooding, droughts, water scarcity, and declining biodiversity are all now the effects of climate change. It affects our health, food supply, housing, safety, and work. Hence, the emissions from the construction industry as well as energy consumption need to be reduced, and an alternative binder to OPC is crucial to develop, which should be environmentally sustainable and display high performance (Manjunath & Narasimhan, 2018).

Geopolymer composites (GPC) are inorganic polymers produced chemically from an aluminosilicate source and an alkaline solution and are currently the material to replace OPC (Saloni et al., 2020). Waste materials and industrial by-products like fly ash (FA), palm oil fuel ash (POFA), ground granulated blast slag (GGBS), silica fume (SF) etc., are binders used to make geopolymers. According to the studies, the manufacture of GPC produces 80% less CO_2 and uses 60% less energy than the production of OPC (Charkhtab et al., 2021; Walbrück et al., 2021; Nematollahi et al., 2016). This can produce a promising and environmentally friendly solution for construction and building materials. Numerous studies have shown the remarkable characteristics of GPC like high early strength (Xie et al., 2019), high surface hardiness (Ramamohana et al., 2019), durability against chemical attacks (Gopalakrishnan & Chinnaraju, 2019) and high fire resistance (Rafeet et al., 2017).

In addition to the GPC, natural fibres are attracting huge attention due to increasing interest in ensuring long-term sustainability and environmental concerns. That has made the use of more renewable sources in the building and construction industry a necessity. Natural fibres are abundant, lightweight and cost-effective materials (Jawaid et al., 2022;

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Castañeda et al., 2020). One of them is kenaf fibre (KF), which is a well-known natural fibre in Malaysia that has been used in bio-composite materials in the industry, including textile, automotive components (dashboard and insulator), rope and yarn, non-woven and geotextile and building materials (Solahuddin, 2022; Akil et al., 2011).

Kenaf planting, cultivation and harvesting have become the subject of interest in Malaysia and are supported by the government to replace the tobacco plant in the future. Government agencies like the National Kenaf and Tobacco Board are undertaking intensive research and development (R&D) on natural fibres, especially kenaf plants (Sim & Nyam, 2021). Moreover, Malaysia is towards eco-friendly degradable materials, particularly for domestic applications. About RM25 mil has been allocated under the Ninth Malaysia Plan for the kenaf crop (Suhairil et al., 2012). KF is one of the fibres introduced by the Malaysians in Expo 2020 Dubai, referring to the numerous potentials of KF in various industries such as building materials for construction, biocomposites, textiles, and many more.

KF is much cheaper than synthetic fibres like glass fibres. Glass fibres cost $3.25 per kg, compared to $0.53 per kg for KF. In addition, kenaf fibres absorb the highest level of carbon dioxide among other types of natural fibres and use a low amount of energy during production. Around 15 moles per joule are needed to make 1 kg of KF, which is less energy than the 54 moles per joule to produce the same amount of glass fibres (Adole et al., 2019). Additionally, kenaf bast fibre has been recognized as a viable reinforcing element in composite materials due to its high mechanical characteristics. The tensile strength of 423 MPa can be attained by a single kenaf fibre that is 10 mm long and 83.24 μm in diameter (Esmaeilpourshirvani & Taghavighalesari, 2019). Kenaf fibres have great potential to replace glass fibre (Pirmohammad et al., 2020).

The use of kenaf fibres to strengthen geopolymer concrete is expected to provide an environmentally friendly replacement for traditional concrete. However, to date, no research has been conducted on the development of geopolymer concrete containing FA and GGBS from kenaf fibres and how the fibre content and length can affect their performance. Such developments would create a stable pathway for kenaf fibre composites and provide new materials for structural applications.

1.2 Problem Statement

There is high demand for clean, high-performance, and long-lasting building materials for environmentally friendly structures all around the world. Cement production pollutes the environment, uses a lot of natural resources and energy and does not meet the sustainable development criteria of today's building materials. In recent years, geopolymers have become more popular as cement substitutes. Geopolymers have attractive properties such as low energy consumption and carbon footprint, high compressive strength, fire and flame resistance, and durability. However, these materials suffer from low tensile and flexural strength. Besides, the cracking resistance of geopolymer concrete is lower than that of conventional concrete (Meng et al., 2022). Hence, fibre reinforcement is a well-known method to enhance the tensile and flexural strength of GPC and enhance its durability (Li et al., 2022). There are two types of fibre

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reinforcement: continuous and short. Short ones are preferable for civil engineering applications because of their cost effectiveness, ease of handling, and manufacture (Payakaniti et al., 2018).

The utilization of fibres in geopolymer creates composites called fibre-reinforced geopolymer composites (FRGC). The type of fibre, aspect ratio (fibre length/fibre diameter), volume fraction, and matrix properties determine the performance of the FRGC. According to relevant studies, the general relationship between mechanical properties and fibre parameters is crucial for the development of FRGC. For instance, the inclusion of fibres to improve the hardened characteristics could decrease workability, and this reduction determines by the type, length, and volume fraction of the fibres in addition to the matrix characteristics. The uniform dispersion of fibres will be affected by poor workability. Bhutta et al. (2019) stated that only good distribution might produce the best fibre effects. Silva et al. (2020) identified an optimum fibre volume fraction at which the FRGC can no longer flow and a fibre balling forms. Hence, optimising the fibre parameters is essential to obtain uniform fibre distribution and satisfactory mechanical properties of FRGC.

Research in the production of fibre-reinforced geopolymer concrete has stepped up in recent times but compared to fibre-reinforced concrete (FRC), documentation of the influences of natural fibre reinforcement in geopolymer concrete is still in its infancy.

Figure 1.1 presents the yearly published papers on FRGC on ScienceDirect. The studies done on FRGC before 2010 are considerably limited. The number of research papers increased significantly from the year 2018, indicating that global scholars are paying much attention to this area. The Figure shows the number of publications has dropped in 2020, this could be attributed to the lockdown due to the Covid 19 virus. Based on these studies, the fibre type is one of the most important factors that should be considered to achieve FRGC with the required properties. Steel, synthetic, inorganic, and natural fibres are the types of fibres that are the most widely used in the FRGC.

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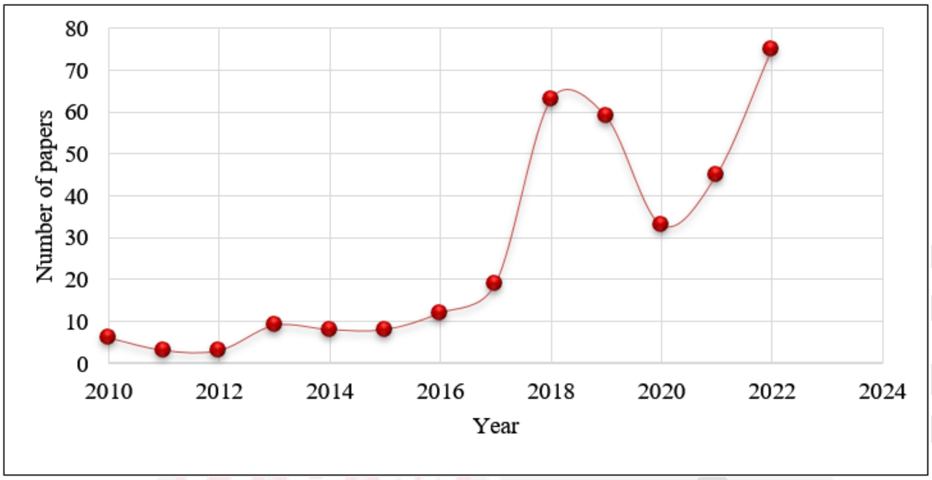

Figure 1.1 : Scientific papers published per year on FRGC from 2010 up to 2022 (data from ScienceDirect)

Lately, extensive studies have been conducted on the incorporation of different types of natural fibres in the construction industry as an environmentally friendly alternative to steel and synthetic fibres. Previous research has focused on the effect of the properties of natural fibres like jute, sisal, cotton, hemp, flax, and coconut on the properties of geopolymer composites (Silva et al., 2020; Poletanovic et al., 2020; Assaedi et al., 2016; Korniejenko et al., 2016). The authors found that using the appropriate length and volume fraction of natural fibres is an effective solution to overcome the brittleness of GPC. Hence, these findings pave the way to explore kenaf fibre addition in geopolymer concrete composite.

The major obstacles to the use of natural fibres as a reinforcing material are the weak compatibility between the fibre and the matrix in addition to the inherent high absorption of moisture, which could lead to dimensional changes in the fibre that could result in the microcracking of the composites and deterioration of the mechanical properties. Surface modification by using alkaline treatment is an effective method for minimizing the moisture-absorbing chemical components like the hemicelluloses, lignin and the impurities such as oils and wax from the fibre surface and increasing the fibre surface roughness, which improves the interface bonding between fibre and matrix (Hamidon et al., 2019; Muhammad et al., 2016).

The present research examined the performance of kenaf fibre-reinforced geopolymer concrete composite (KFRGC) containing different fibre lengths and volume fractions as a step toward green concrete materials. Based on the review of the relevant literature, the following research questions have been identified with respect to the KFRGC.

- What is the best treatment condition of KF that could improve the mechanical properties of the fibre and enhance the interfacial bonding with the geopolymer matrix?
- How will the different kenaf fibre length and volume fraction impact the fresh, mechanical and durability performances of KFRGC?
- How will the interaction between the fibre and matrix look like? Will increasing the length or content of the fibres improve the bond with the matrix?

1.3 Aim and Objectives

This study aims to investigate the performance of KFRGC prepared with different KF volume fractions and lengths and explore how increasing these parameters can affect the fresh, mechanical, durability and microstructural properties of KFRGC. Based on the aim of the study and the above-mentioned problem statement, the following objectives are set:

i. To study the effect of the alkaline treatment on the physical, mechanical, and microstructural properties of kenaf fibres and propose a mix design for geopolymer concrete with the required strength.

ii. To determine the fresh state, strength, deformation characteristics and microstructural behaviour of kenaf fibre reinforced geopolymer concrete composites prepared with different fibre volume fractions and lengths.

iii. To evaluate the water absorption, sorptivity and water penetration of geopolymer concrete composites containing kenaf fibre with different volume fractions and lengths.

iv. To analyse the chloride ions attacks and chemical resistance of kenaf fibre-reinforced geopolymer concrete composites and verify their microstructural behaviour.

1.4 Scope and Limitations

The research would be experimental and primarily focuses on the development of a natural fibre-reinforced geopolymer concrete incorporating alkali-treated kenaf fibres. The treatment method was limited to the alkaline treatment since the literature has proved that this method is considered the most suitable and economical technique to pre-treat kenaf fibres. Besides, the treatment parameters such as the concentration of the sodium hydroxide solution and the submersion period were fixed at 6% and 3 hours, respectively, based on the literature, and only the drying condition was tested because there is not sufficient data in the literature to verify the best drying condition. For the control geopolymer, our research focused on the effect of the alkaline activator to binder ratio (AAC/B) while the other mixed design parameters such as FA/GGBS ratio, sodium hydroxide concentration and sodium silicate to sodium hydroxide (SH) ratio (SS/SH) have been fixed.

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The kenaf fibre length and volume fraction are limited to 20 mm, 30 mm, and 40 mm and 0.75%, 1.0%, 1.25% and 1.5%, respectively, due to the difficulties in the wet state if longer and high-volume fibres are added. The study examines the physical, mechanical, durability and microstructural properties of geopolymer composites, which are believed to be within limits set by the objectives.

1.5 Thesis Layout

For this research, chapter one is designed to provide a general appraisal and an overview of the problem, and background to support the problem statement. In addition, the chapter also highlights the aim and objective, scope and limitation of the research. Chapter two presents a critical review of the related and relevant literature. Chapter three emphasizes a comprehensive description of the materials and sample synthesis methods together with the test used for characterizing the samples. The characterization of the constituent materials comprising the physical properties and chemical compositions of FA and GGBS, treatment and characteristics of kenaf fibres.

The Fourth chapter presents the results and discussion of the mix design of plain geopolymer concrete and the optimization of the alkaline activator to binder ratio in terms of workability and compressive strength. Besides, this chapter reveals the physical, mechanical, and microstructural properties of KFRGC prepared with different fibre volume fractions and lengths. These include workability in terms of slump and unit weight for the fresh mixtures. It also presents the results obtained and the discussion made on the evaluation of mechanical and deformation properties of hardened KFRGC. Tests falling in this category comprise compressive strength, tensile and flexural strengths, ultrasonic pulse velocity, and modulus of elasticity. Moreover, the related microstructure analysis by using FESEM and EDX is also present in this chapter.

Chapter five depicts the results of water absorption, sorptivity, permeability, chloride ion attack and chemical attacks resistance tests of KFRGC. While Chapter six concludes the thesis and makes some recommendations for further study in the fields of geopolymer concrete using kenaf fibres.

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