Performance of Natural Fibre-Reinforced Geopolymer Composites Exposed to Sulphuric Acid

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Abstract Increasing interest in using waste materials and by-products from different industries as renewable sources of construction materials is mainly for environmental concerns and sustainability. Geopolymer and natural fibres are examples of industrial wastes and by-products used. However, their acceptability in the industry is relatively low due to insufficient research on their durability performance. This paper presents an investigation into the durability of kenaf fibre-reinforced geopolymer composites (KFRGCs) when exposed to a 5% solution of sulphuric acid. The main parameters evaluated were the changes in the visual appearance, weight, and compressive strength. Geopolymer specimens' reinforced composites with a 1% volume fraction of different lengths of kenaf fibres (20, 30, and 40 mm) were produced to evaluate the impact of the fibre addition and fibre length on the performance of geopolymer composites. The results showed that the KFRGC in the acidic environment demonstrated a reasonable level of resistance to the attack as compared to plain geopolymer concrete (GPC). Fibres' length effects show shorter fibres which have better resistance than longer fibres. These findings display the positive impact of natural fibres on the performance of GPC in an acidic environment.

Keywords Sustainability · Geopolymer composites · Natural fibres · Kenaf fibres · Durability · Acid attack

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N. Sabtu (ed.), *Proceedings of AWAM International Conference on Civil Engineering 2022—Volume 2*, Lecture Notes in Civil Engineering 385,
https://doi.org/10.1007/978-981-99-6018-7_12

1 Introduction

The high public understanding of the environment and the protection of non-renewable petroleum-based materials have contributed to the widespread use of natural resources for industrial applications. Besides, increasing interest among consumers in sustainable and environmentally friendly materials encourages retailers and manufacturers to invest in the production of sustainable materials at reasonable costs to mitigate the effects of global warming [1, 2].

Geopolymers are inorganic polymers formed through the chemical reaction between aluminosilicate sources derived either from geological minerals like metakaolin (MK) or from industrial by-products such as the ground granulated blast furnace slag (GGBS), fly ash (FA), palm oil fuel ash (POFA) with an alkaline solution like sodium silicate and sodium hydroxide through the geopolymerization process that results in polymer chains and cross-linked networks of comparable or greater strength than that of OPC [3–5]. On the other hand, natural fibres are abundant, lightweight, and cost-effective materials [6, 7]. The composites based on natural fibres and geopolymers could produce green materials for the construction and building industry.

Previous studies have focused on the influence of the properties of natural fibres like cotton, hemp, flax, and coir on the properties of geopolymer composites [8–11]. The authors found that using the appropriate length and volume fraction of short natural fibres was an effective solution to overcome the brittleness of GPC [12]. Hence, these findings pave the way to use kenaf fibre to get more details on the properties of geopolymers reinforced by natural fibres.

Kenaf fibre (KF) is a well-known natural fibre in Malaysia that has been used in biocomposite in the local industry. Malaysian Government agencies like the National Kenaf and Tobacco Board are undertaking intensive research on the kenaf plant. Moreover, Malaysia is towards eco-friendly, with the goal of reducing the worldwide use of non-degradable materials, particularly for domestic applications. About RM25 million has been allocated under the Ninth Malaysia Plan for the kenaf crop [13]. Kenaf fibres have good mechanical properties and require low production energy with environmental benefits, which include bio-renewability, biodegradability, non-hazardous nature, and low pollutant emission [14]. The biodegradability of KF contributes significantly to a sustainable environment, while its low cost and good performance address manufacturers' economic concerns [15–17]. These fibres have great potential for replacing synthetic fibres like glass fibre [18].

The major obstacles to the usage of natural fibres as reinforcing materials 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 micro-cracking of the composites and deterioration of the mechanical properties. Surface modification by using alkaline treatment is an effective method for minimizing the water-absorbing chemical components like the hemicelluloses, lignin, and impurities such as oils and wax from the fibre surface and increasing the fibre surface roughness of KF, which improves the interface bonding between fibre and matrix [19, 20].

The use of geopolymer in concrete can enhance the durability characteristics, and the fibres demonstrate suitable behaviour in this regard [21]. The decision to utilize sulphuric acid among the other acid types was based on its practical applicability because the most commonly used concrete buildings, like the food processing industries, mining, and wastewater canals, are in touch with sulphuric acid. This paper evaluates the performance of geopolymer concrete containing 1% KF with three different lengths of 20, 30, and 40 mm under the sulphuric acid attack [21].

2 Experimental Programme

2.1 Materials

Materials used to prepare the GPC and KFRGC are the binder, alkaline solution, fine and coarse aggregates, water, and KF. The binder is mainly consisting of FA (Class F) according to ASTM C 618-12a [22] and GGBS. The alkaline solution is comprised a mixture of sodium hydroxide and sodium silicate. These are used to activate the alumina and silica in the binder. Analytical grade sodium hydroxide (98% purity collected from bt science Sdn. Bhd Malaysia) was received as pellets; the pellets were dissolved in water to prepare a sodium hydroxide solution with a 14 M concentration. The solution was prepared 24 h before mixing and left in the lab to be cool. Analytical grade sodium silicate solution was also used to prepare the alkaline activator.

2.2 Kenaf Fibres (KF)

KF is known for its high cellulose content, and thereby, it is biodegradable. Besides, the absence of silica content enables the fibres to be nonabrasive [23]. The fibres were extracted through the water retting process from the bast of the plant. The length of the fibres in the market is between 3.5 and 5 m. The fibres were treated to remove the surface impurities and increase the surface roughness, thus enhancing the interfacial bonding between the fibres and the geopolymer matrix. The alkaline treatment method was chosen to treat the fibres before their inclusion in the geopolymer composites.

The KF was treated in the laboratory by immersing the fibres in sodium hydroxide solution of 6% concentration for 3 h. The sodium hydroxide concentration and the immersion time were chosen based on previous studies [17, 24]. After 3 h, the fibres were then washed and kept in water for 24 h. After 24 h, the fibres were dried at ambient temperature. Afterwards, the specimens were ready for testing.

Table 1 Properties of KF

Diameter (μm)	76
Tensile strength (Mpa)	242
Specific gravity (kg/m^3)	1450

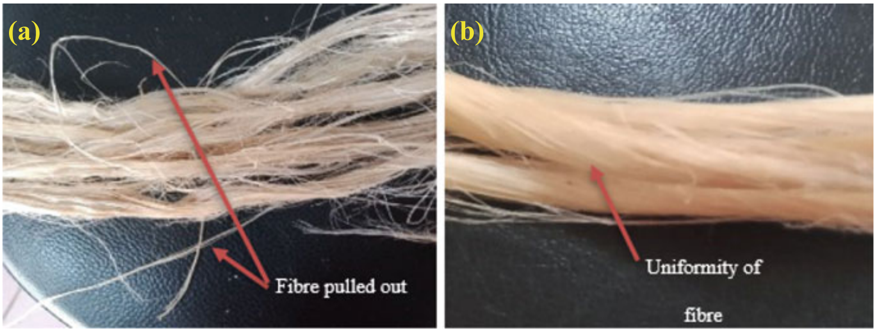

Fig. 1 Surface appearance of KF, **a** untreated KF, **b** treated KF

After the treatment, KF's diameter, tensile strength, and density were evaluated, and the results are presented in Table 1. The physical appearance of kenaf fibre before and after treatment is shown in Fig. 1. Overall observations show that the physical appearance of the fibres was greatly altered by the chemical treatment. In terms of colour, treated fibre shows a different colour after treatment. The colour of the fibre becomes more yellowish after the treatment. The fibres were cut into three different lengths of 20 mm, 30 mm, and 40 mm and added to the geopolymer mixtures.

2.3 Mix Proportions

The mix proportions are shown in Table 2. Three different KFRGC mixes besides the control mix without fibres were prepared. KF was cut to three different lengths of 20, 30, and 40 mm and used in the mixtures at a 1% dosage. The ratio of sodium silicate (SS) to sodium hydroxide (SH) was fixed at 2.5 for all mixtures.

2.4 Mixing, Casting, and Curing

To get a homogeneous mixture, the fine and coarse aggregates were dry mixed for two minutes before the binder materials were added and mixed. Afterwards, KF was added and mixed for another 3 min. Lastly, the alkaline activator was included and mixed for 3 min. Then, fresh mixtures were cast in the moulds in three levels, with

Table 2 Mix proportions (kg/m^3)

Mix	FA	GGBS	Aggregates		Solution		L^a (mm)	$V_f{}^b$ (%)
			Sand	10 mm	SS	SH		
GPC (Ref)	261	174	704.7	1065.7	170.8	68.3	0	0
KFRGC1	261	174	704.7	1065.7	170.8	68.3	20	1
KFRGC 2	261	174	704.7	1065.7	170.8	68.3	30	1
KFRGC 3	261	174	704.7	1065.7	170.8	68.3	40	1

[a] L: Fibres' length
[b] V_f: Fibres' volume fraction

each layer being consolidated for about one minute on a vibration table to permit air escape. After casting, the specimens were left for 24 h at room temperature (27 ± 1.5) °C under a relative humidity of 75%. The specimens were then removed from the mould and stored in the laboratory until the testing date.

2.5 Testing

For the entire experimental work, a total of 60 geopolymer cubes with a 100 mm size were cast. In the first stage, the air-cured compressive strength of KFRGC and GPC was determined by using 100 mm cube specimens in line with the procedures laid down by BS 12390-3 [25] at 7, 28, and 56 days.

The chemical immersion experiment was used to evaluate the performance of GPC and KFRGC against the sulphuric acid attack. After 28 days of curing, the specimens were cleaned with a cloth to remove any impurities from their surface and weighed to the nearest 0.1 g as the initial weight. The specimens were then immersed in 5% H_2SO_4 solutions for 180 and 365 days, respectively, and subsequent weights were recorded during the exposure period. To keep the pH constant throughout the study period, the acid solution was replaced every two months. After 180 and 365 days of exposure to the acid, the performance of each mixture was evaluated based on its visual appearance, weight loss, and strength loss as follows:

Visual appearance: all the samples were visually inspected to determine the degree of deterioration. The colour, texture, edges, size, and geometry of the sample were observed throughout the evaluation.

Weight loss: to determine the variation in weight, the weight loss percentage for each mix was calculated by using Eq. (1).

$$\text{Weight loss} = \frac{W_2 - W_1}{W_1} \times 100, \tag{1}$$

where

W_1 = the initial weight of specimen before immersion in the acid solution (gr), W_2 = the weight of specimen after immersion in acid the solution (gr).

Strength loss: on the other hand, the degradation of geopolymer samples was also examined by determining the strength loss factor (SLF). The SLF is expressed in percentage and calculated using Eq. 2 [26].

$$\mathrm{SLF} = \frac{F_{\mathrm{ca}} - F_{\mathrm{cs}}}{F_{\mathrm{ca}}} \times 100, \tag{2}$$

where

SLF = the percentage of loss in geopolymer's compressive strength after exposure to the acid attack, F_{ca} = the average compressive strength of air-cured geopolymer, and F_{cs} = the average compressive strength of geopolymer immersed in the chemical solution.

3 Results and Discussion

3.1 Density

The density of geopolymer composites depends largely on the unit weight of the constituent materials employed in the mix. Figure 2 shows the density of the various geopolymer mixes. KFRGC exhibited lower density compared to plain geopolymer. Besides, increasing the length of the fibre caused a higher reduction in density. This is due to the higher porosity resulting from the entrapped air within the composite during the mixing of fibres. Furthermore, because KF has a lower density than GPC, the final density of KFRGC becomes lower.

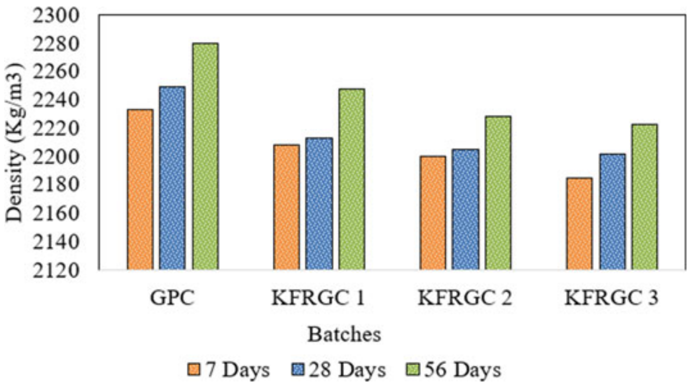

Fig. 2 Density of plain geopolymer (GPC) and KFRGC prepared with different fibre lengths

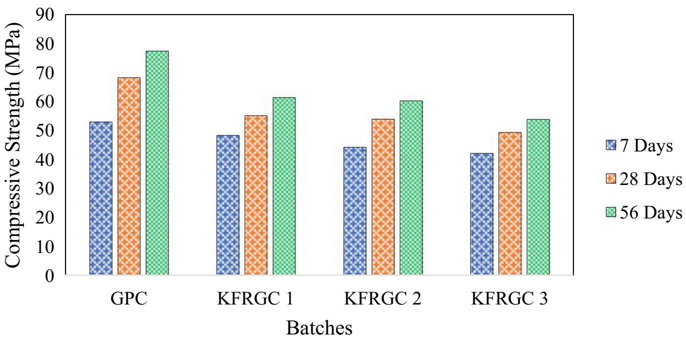

Fig. 3 Compressive strength of plain geopolymer concrete (GPC) and KFRGC prepared with different fibre lengths

3.2 Air-Cured Compressive Strength

The compressive strength values of the GPC and KFRGC are illustrated in Fig. 3. The incorporation of KF decreased the compressive strength by about 19–27% less than the plain geopolymer at 28 days. The reduction is due to the porosity of the composite, as has been shown in the density. The KFRGC containing short fibres (20 and 30 mm) shows better performance compared to longer fibres (40 mm). Longer fibres' ineffectiveness in increasing compressive strength can be ascribed to two distinct phenomena. First, if the fibres are too long, problems can arise during mixing the geopolymer. Fibre clusters develop as a result, which can result in increased porosity. Secondly, the orientation of the fibres inside the hardened KFRGC can also play a vital role in the composite's strength. The adverse impact of fibre length on the compressive strength was also reported by Xu et al. [27], Vilaplana et al. [28], and Novais et al. [29] when the authors used polyvinyl alcohol fibres, carbon fibres, and glass fibres, respectively.

3.3 Acid Attack

Visual Appearance

The appearance of geopolymer specimens after 365 days of exposure to H_2SO_4 is presented in Fig. 4. The control sample showed the highest degree of deterioration with spalling and line cracks at the edges of cubes, as in Fig. 4a. In terms of KFRGC, increasing the length of the fibres from 20 to 30 mm improved the composite's resistance and reduced the deterioration degree with smaller cracks and erosion, as in Fig. 4b, c. However, the further increase in the length of the fibres to 40 mm showed an adverse effect with more spalling, as shown in Fig. 4d.

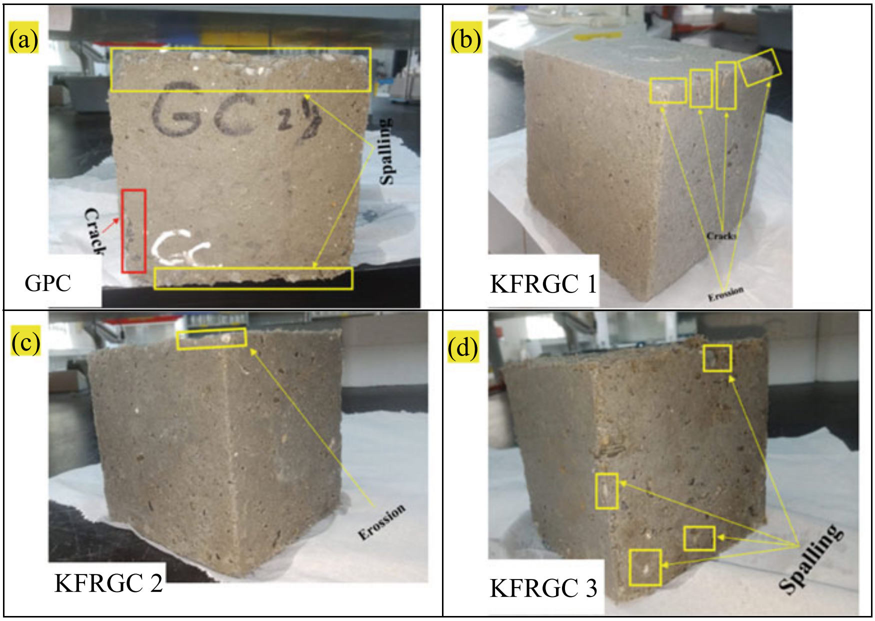

Fig. 4 Appearance of geopolymer composites exposed to an H_2SO_4 solution for 365 days: **a** plain GPC; **b** KFRGC 1; **c** KFRGC 2; **d** KFRGC 3

Weight Loss

The weight loss percentage was also evaluated after 180 and 365 days of immersion in the acid solution, as shown in Fig. 5. KFRGC samples achieved lower weight loss than the GPC sample (without fibres), showing the attributes of the fibres to improve composite materials' abrasion resistance [30]. Increasing the fibre length from 20 to 30 mm caused an insignificant increase in the weight loss rate of about 2% after 365 days of exposure. However, increasing the fibre length to 40 mm increased the weight loss of KFRGC by about 10%, showing that the porosity due to long fibres is being too large. Hence, it cannot improve the performance of the geopolymer.

Compressive Strength Loss Factor (SLF)

As the weight of the samples decreases, it is expected that the strength will follow after the immersion in solution for 180 and 365 days. The longer the immersion period, the higher the strength reduction. The deterioration was attributed to the aluminosilicate polymer depolymerization in acidic environments and the development of zeolites, which resulted in a significant loss of strength [31]. The reduction of compressive strength of plain GPC and KFRGC mixtures due to acid attack was expressed in the form of a strength loss factor (SLF), and the results are illustrated in Fig. 6. The SLF values of plain GPC were higher than that of KFRGC. However, the SLF of KFRGC highly depends on the length of the fibre. The SLF of the composites decreased with increasing the length from 20 to 30 mm; however, a further increase

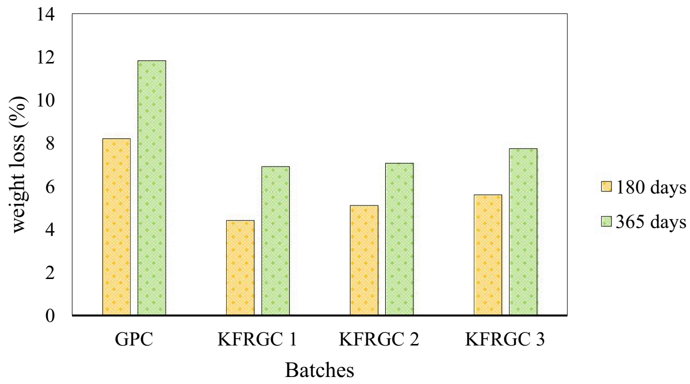

Fig. 5 Effects of KF fibres' length on the weight variation of the geopolymer after 180 and 365 days of exposure to the acid attack

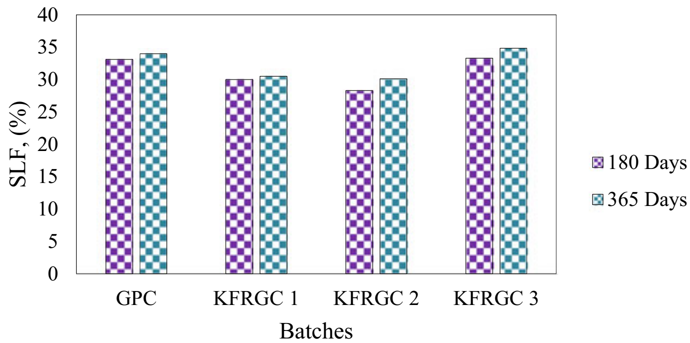

Fig. 6 Strength loss factor of plain GPC and KFRGC containing different fibre lengths exposed to the H_2SO_4 solution

in the length to 40 mm increased the SLF by 15% after 365 days. These findings reveal that the composites with longer lengths lost more strength. This could be attributed to the higher porosity and the distribution of the fibres inside the samples. This demonstrates that increasing the length of fibres to 40 mm will not be able to mitigate acid-induced damage in KFRGC.

4 Conclusion

In this study, the sulphuric acid resistance of plain geopolymer and kenaf fibre-reinforced geopolymer composites was studied. KFRGC exposed to acidic attack exhibited a reasonable level of resistance to the acid environment. The KFRGC reinforced with short lengths of fibres showed better results compared to the composites containing long fibres, and the SLF was 10 and 14.5% lower than the plain GPC for the samples with 20 mm and 30 mm, respectively. Increasing the length of the fibres from 20 to 30 mm showed insignificant change in terms of the weight and strength loss percentage. However, increasing the length of the fibres to 40 mm led to adverse results. Therefore, in terms of durability, KFRGC with 20 mm or 30 mm fibre length shows good performance and potential when exposed to an acidic environment.

Acknowledgements The authors gratefully acknowledge the financial support for this research by the Ministry of Education, Malaysia, under the Fundamental Research Grant Scheme (FRGS/1/2020/TKO/UPM/02/32) with Vot no: 5540372 for research work entitled 'An investigation of characterization and parametric effect of kenaf bast fibre in the properties of geopolymer kenaf reinforced concrete'. The authors also gratefully acknowledge the assistant engineer Mr Fairus Ismail in the materials lab of the Faculty of Engineering, University Putra Malaysia UPM.

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