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A Special Issue Devoted to
Green Technology Towards Sustainable for Composite Materials

Guest Editors
Mohd Zuhri Mohamed Yusoff, Syeed Saifulazry Osman Al-Edrus
and Ayu Rafiqah Shafi



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Pertanika Journal of Science & Technology

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Preface

The two identities of agriculture and biomaterials need to meet the growing demand for sustainability and safety. Sustainable development and environmental science and technology have experienced an unprecedented shift in the last few years. The “one health” concept, climate change, economic imperatives, and the increasing use of “green” practices across a wide range of industries (construction, agriculture, business, development management and industry) must all be considered in the context of this continual update. In terms of the environment, it is important to recognize that to halt the decline and destruction of nature (and, by extension, everything pertaining to environmental sciences), serious policies and strong and drastic measures/strategies are becoming more and more necessary. Furthermore, it is imperative that we actively engage in restoring ecosystems that have been damaged by unbridled, reckless, and utterly disrespectful human conduct toward the environment’s importance to both people and life in general.

The agricultural sector is a highly polluting industry due to the substantial quantities of solid waste and water required, resulting in considerable environmental issues that must be mitigated. By implementing this approach, it becomes feasible to manage the recycling and reduction of the waste and byproducts generated by this industry, leading to a reduced environmental footprint and the advancement of a circular economy. Multiple studies have confirmed the utilization of these byproducts or wastes as raw materials for diverse processes, ranging from food technology to material science. It has been argued that creating new materials can successfully utilize these biowastes.

Considering the previously mentioned, we have determined that a Special Issue centred around “environmental technology or industry” is highly appropriate. This issue aims to compile data and research pertaining to the topics above, specifically focusing on the following terms and fields. This Special Issue invites potential authors to explore a wide range of topics in ecology, ecosystems, pollution, environmental recovery, environmental health, “one health,” zero waste, biodegradable materials, and management of crops. Authors are encouraged to consider any relevant topic that aligns with this issue’s comprehensive and inclusive theme. This all-encompassing strategy offers a thorough structure for

comprehending and resolving environmental concerns connected to the sector. The special issue consolidates the conference themes of “Forest Plantation Management,” “Advanced Technology in Forest Plantation,” “Socio-Economic & Policy,” “Big Data/Data Science in Wood and Fiber Technology,” “Ecosystem Service and Climate Change,” and “Plantation Pest and Disease.”

Guest Editors

Mohd Zuhri Mohamed Yusoff (*Dr.*)

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Mechanical Properties of Novel Hybrid Bamboo Fibre/ Aluminium Mesh Reinforced Polymer Composite

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ABSTRACT

Bamboo fibres are one of the sustainable lignocellulosic resources explored for polymer composites in recent years. Research has shown that bamboo fibres have the potential to be used in a variety of critical applications. Nevertheless, bamboo fibres are susceptible to thermal and hygroscopic loads, and their mechanical properties are limited by the unequal interfacial strength and varying fibre dimensions. Implementing hybrid procedures or incorporating alternative materials, such as aluminium metal, is strongly advised to address

this issue. Thus, this study investigates the tensile and flexural performances of the hybrid bamboo fibre/aluminium expanded mesh-reinforced polymer composites. The composites were fabricated using epoxy resin reinforced with bamboo fibre, and an aluminium expanded mesh sheet was constructed using a vacuum infusion process utilising various stacking sequences and mesh sizes. The test findings indicated that the composite material exhibited tensile stress values ranging from 27 to 34 MPa

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and a corresponding tensile strain value between 1.1% and 1.6%. The flexural strength and strain values were measured within the range of 44 Mpa to 59 Mpa and 2.2% to 3.2%, respectively. ANOVA analysis showed that both stacking sequences and mesh size significantly affected the tensile performances of the composites, while only stacking sequences affected the flexural performance significantly. Overall, a hybrid composite of bamboo fibre and aluminium mesh is well-suited as a substitute material in industries requiring exceptional mechanical properties.

Keywords: Aluminium mesh sheet, bamboo fibre, hybrid composite, mechanical properties, natural fibre composite

INTRODUCTION

The increasing awareness of green and sustainable materials has attracted more researchers to use natural fibre-reinforced composite (NFRP) in their studies (Lopes et al., 2021; Noori et al., 2021). Natural fibre has been considered as an alternative material to synthetic fibre owing to its benefits of being lightweight, renewable, biodegradable, having high strength and elastic modulus, available at low cost, possessing low energy requirements, and abundant in nature, as reported by many researchers (Adeniyi et al., 2019; Balla et al., 2019; Cavalcanti et al., 2019; Li et al., 2020; Nascimento et al., 2021; Otto et al., 2017; Oushabi, 2019; Todkar & Patil, 2019).

Depending on its source, natural fibre can be divided into three categories: mineral, animal, and plant (Alemayehu et al., 2020; Bahja et al., 2020). Among them, plant fibres have the advantage of being abundant and having higher producibility than animal and mineral fibres. Plant fibre properties can also be classified into their origin: stems, leaves, and seeds. Often, natural fibres have drawbacks and limitations, including inhomogeneity properties, high water absorption, and water regains; nevertheless, studies on the NFRP composites keep updated and improved over the years and have been involving many types of plant fibre, such as bamboo (Wang et al., 2022; Yuan et al., 2022), kenaf (Atiqah et al., 2020; Rozyanty et al., 2020), jute (Dhiman & Sharma, 2020; Reddy et al., 2020), banana (Batu & Lemu, 2020; Girimurugan et al., 2020), and pineapple leaf (Umanath et al., 2019).

Among natural plant fibres mentioned, bamboo fibre has superior fibre properties and can be found easily, especially in China and Southeast Asia. According to the news by the International Bamboo and Rattan Organisation (INBAR), over 70 species have been reported in Malaysia alone, with 16 commercially viable species (Joest, 2017). Bamboo fibre may have higher mechanical strength, lower specific weight ratio, higher tensile strength and a higher modulus of elasticity than other natural fibres. Owing to its strength, bamboo, also known as natural glass fibre (Shireesha & Nandipati, 2019), with the tensile strength of a bamboo fibre bundle, can reach the same heights as jute fibre (Okubo et al.,

2004; Sen & Reddy, 2011). Concurrently, other studies claimed that bamboo's strength makes it an appealing alternative to steel in tensile stress applications (Rassiah & Megat Ahmad, 2013).

Nowadays, research on bamboo fibre as a compositional reinforcement has gained popularity among researchers, and most studies have investigated bamboo polymer composites. Although research on the use of natural fibre can overcome problems related to sustainability, there is no denying that natural fibre-reinforced polymer composites have their own problems. For instance, high moisture absorption and low bonding strength between natural fibre and matrix are among the problems reported by previous literature (Yadav & Singh, 2021).

Thus, research introducing an upgraded type of composite, also known as hybrid composite, has been done to overcome these problems. Hybrid composites combine more than one reinforcement material to enhance the composite's mechanical, physical and thermal properties, and one of the reinforced materials used is Aluminium (Al). Aluminium is highly used in the production of composites because of its high strength-to-weight ratio and strong fatigue tolerance (Gonzalez-Canche et al., 2017). Besides, aluminium has the best formability when hybridised with fibre-reinforced polymer composites (Kavitha et al., 2020). Table 1 tabulates the studies on aluminium and bamboo as a reinforcement of hybrid composites in recent years.

Table 1
Aluminium/bamboo reinforced composites reported in recent years

No	References	Materials	Properties
1	Sheng et al. (2023)	Aluminium plate (3- and 6-mm thickness) and bamboo scrimber (Moso bamboo)	Flexural properties
2	Harikumar and Devaraju (2020)	Aluminium oxide (nanoparticles) and bamboo fibre	Hardness, impact, tensile, and flexural properties
3	Kali et al. (2019)	Aluminium sheet (0.5 mm thickness), bamboo stripe, carbon fibre, and glass fibre	Vibration characteristics

In addition to the hybrid composite studies, fibre metal laminate (FML) is another type of hybrid composite that has been certified to improve the mechanical properties of composites. Furthermore, In their review, He et al. (2021) stated that aluminium is the most commonly used metallic material in fibre metal laminate composite due to its high impact resistance and superior ductility. Gonzalez-Canche et al. (2017) investigated the tensile properties of FML reinforced with low aluminium alloy and aramid fabric. Tensile studies demonstrated that FMLs are more ductile than their constituents, as observed by

a 230% increase in strain to failure compared to the composite laminate of aramid fabric and a 400% increase compared to aluminium sheet. Another study by Megeri and Naik (2021) focused on the impact behaviour of hybrid FML reinforced with aluminium, carbon and glass fibre, which discovered the ability of hybrid FML to withstand more impact loading than glass FML. In addition, using high-stiffness carbon fibre increased the impact resistance of hybrid FML.

While studies regarding FML demonstrated a gain in mechanical properties, these composites still have a significant disadvantage: low interface roughness between the aluminium and fibre layers. Due to this difficulty, further research has been conducted to improve the interface bonding of this reinforcement. One method for improving this bonding interface is substituting aluminium wire mesh for the aluminium sheet (Arun Prakash & Julyes Jaisingh, 2018; Megahed et al., 2021). According to a study by Megahed et al. (2021), adding aluminium wire mesh layers improved tensile strain by 54% and flexural strain by 117.5% compared to glass fibre-reinforced polymer composite. In addition, the presence of an Al mesh layer in the first and last layers, where the highest shear and normal stresses occur, allowed for ductile deformation of the Al layers, which increased the failure strain.

Using bamboo fibres and aluminium as reinforcement in the composite has been discussed, highlighting important insights and advantages. Hybridisation research can be done to examine the different qualities of both materials. However, the hybridisation of aluminium wire mesh and natural fibre, especially bamboo fibre, was less in number. There is a scarcity of durable hybrid bamboo fibre/aluminium reinforced polymer composites, essential for critical applications as a replacement for synthetic composite materials currently available. Therefore, this study was conducted to determine the feasibility and the properties of hybridising the bamboo fibre and aluminium mesh sheet in the composites, also known as bamboo fibre/aluminium mesh reinforced polymer composites (BFAMRP).

MATERIALS AND METHOD

Materials Preparation

This study employs two reinforcement materials: bamboo fibre nonwoven preform and aluminium expanded mesh. Bamboo fibres were mechanically extracted from *Schizostachyum grande* or *Buluh Semaliang* among locals. This type of bamboo was chosen for this study because it was widely distributed throughout Malaysia. Furthermore, this bamboo has been classified as one of 13 varieties of bamboo that can be commercialised in Malaysia. According to a study by Siam et al. (2019), this variety of bamboo also has some of the highest modulus of elasticity (MOE) and modulus of rupture (MOR) values, which was advantageous in the production of polymer-based composite materials. The bamboo was harvested in Sungai Petani, Kedah, Malaysia and supplied by Hangterra Bamboo Sdn

Bhd., Kedah, Malaysia. This study used two sizes of aluminium expanded mesh: a small mesh size of 3.5 mm x 2.0 mm and a large mesh size of 4.0 mm x 3.0 mm. Epoxy resin with a 2.7 g/cm³ density was used for the matrix composite. The properties of materials used in this study are depicted in Table 2.

Table 2
Materials used in the study

Material	Notes
Bamboo fibre	Type of bamboo: Schizostachyum Grande (Semaliang)
Fine mesh aluminium sheet	Mesh size: 3.5 mm x 2.0 mm, Orientation: 60°, thickness of the wire strand 0.5mm
Medium mesh aluminium sheet	Mesh size: 4.0 mm x 3.0 mm, Orientation: 60°, thickness of the wire strand 0.8mm
Epoxy resin 1006	Epoxy to hardener weight ratio 10:6

The processing of bamboo fibre nonwoven preform entails several stages. The initial stage was fibre extraction. The internode of the bamboo pole was cut into long with a width of 20 to 25 mm. Besides easing the fibre extraction process, a specific strip width size is necessary to ensure the desired physical fibre obtained is fine and homogenous. Next, the bamboo strips were retted by soaking them in normal water at room temperature for two days to allow them to soften. The retted bamboo strips were then inserted into a fibre decorticator. The fibres extracted from the fibre decorticator were collected to remove residuals and impurities before being washed. Previous reports proved that alkaline treatment would increase surface bonding between reinforcement and matrix (Manalo et al., 2015; Sánchez et al., 2020; Vigneshwaran et al., 2020). Thus, the bamboo fibres were treated by soaking them in a solution of 5% NaOH for 24 hours at room temperature, as suggested by Getu et al. (2020). Later, the fibres were washed several times under running water to neutralise them before being dried in an open space for three days. The final stage in making bamboo fibre preform was the needle punching nonwoven method. The fibres were gathered and fed into the nonwoven opening section, followed by blow room and carding, before being fed into the needle punching section. The machine parameter was set following 400 gsm of bamboo fibre nonwoven output. Each layer of bamboo fibre nonwoven mat weighed 12.5 ± 0.5 g. Figure 1 summarises the overall steps of making a nonwoven bamboo fibre preform.

Eight types of BFAMRP composites with four different stacking sequences and two mesh sizes of the aluminium mesh sheet were developed for this study. Each composite was comprised of five layers of reinforcement with a dimension of 300 mm x 300 mm. Table 3 depicts the composition of the specimen, including the code, stacking sequence, and Al mesh type. As adapted from Chokka et al. (2020), the vacuum infusion technique was used for the present study. The composites were left at room temperature for 24 hours to cure.

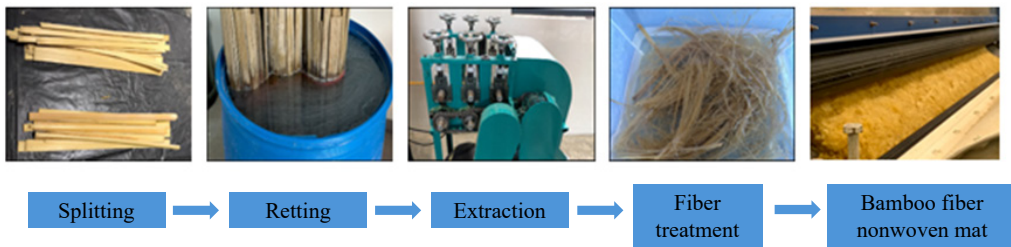


Figure 1. Steps to produce bamboo fibre nonwoven preform

Table 3
BFAMRP composites code, stacking sequence, mesh size, and average thickness

No	*Specimens code	**Stacking sequence	Mesh size	Avg. thickness, cm
1	A1S	A/B/A/B/A	S	0.51
2	A1L		L	0.54
3	S2S	B/A/A/A/B	S	0.51
4	S2L		L	0.56
5	A3S	B/A/B/A/B	S	0.60
6	A3L		L	0.63
7	S4S	A/B/B/B/A	S	0.51
8	S4L		L	0.55

* First letter indicated stacking structure; A: alternate structure, and S: sandwich structure

* Middle number indicated stacking sequence

* The last letter indicated the size of the mesh: S—small mesh, and L—large mesh

** A: Aluminium expanded mesh, B: Bamboo fibre nonwoven mat

Tensile and Flexural Tests

The tensile test of BFAMRP composites was conducted according to the ASTM D3039 standard using a Shimadzu Servopulser E-Type loading frame in the Machine Testing Laboratory at the Faculty of Mechanical and Manufacturing, UTHM. During the test, the specimens with the dimensions of 250 mm x 25 mm each were put in the machine's grips, while axial tensile loads were applied to the samples at a 2 mm/min loading rate. A repeatability test of three samples for each group of specimens was conducted to ensure the accuracy of the results. Tensile strength, tensile strain, and Young Modulus were obtained and discussed.

A flexural test was performed using the ASTM D790 three-point method. The Shimadzu Universal Testing Machine (UTM) of the Polymer Laboratory, Faculty of Mechanical and Manufacturing Engineering, UTHM, was utilised to perform the test. Specimens with 127 mm x 12.7 mm dimensions were loaded into the three-point bending fixture at 2 mm/min. The span of the supports was set to 80 mm for the flexural test, and the pin diameter of 5 mm was used throughout the test. The load was applied at the specimen's mid-span, and

the resulting load and displacement values were recorded. Five tests for each specimen set were repeated to ensure an accurate result.

Scanning Electron Microscope (SEM) Analysis

The fracture specimens of BFAMRP composites from both tensile and flexural samples were observed using a high-resolution Scanning Electron Microscopy (SEM) machine, JEOL JSM-6380LA, located at Material Science Laboratory, UTHM. Morphological assessment using SEM was performed to determine the integration between the matrix and reinforcement after applying loads. It was also used to observe the microscopic failure of composites at different stages, including elastic, plastic, local fracture of fibre layer, and delamination stages (He et al., 2021). Before observing the specimens, the specimens underwent a coating process by JEOL Auto Fine Coater.

Analysis of Variance (ANOVA)

Statistical analysis of the measured tensile and flexural performances of BFAMRP composite was performed with the general linear model (GLM) of analysis of variance (ANOVA) using MINITAB 19 Software. The analysis was conducted at a 5% significance level.

RESULTS AND DISCUSSION

Tensile Test

Table 4 shows the ANOVA table for the tensile properties of BFAMRP composites. According to Table 4, the stacking sequence significantly affected the tensile performance with a P -value of less than 0.05. However, the mesh size also significantly affected the tensile performance. The contribution percentage also showed that the stacking sequence was the main factor that affected all the tensile strength and tensile strain of the BFAMRP composite.

Figure 2 illustrates the tensile stress-strain curve of BFAMRP composites. Generally, all eight specimens displayed the same stress-strain curve trend, which was linear behaviour along the elastic region curve before the failure point. From observation, specimens with aluminium at the outer layer had better tensile stress than specimens with bamboo at the outer layer. This is a result of the ductility of the aluminium (metal mesh), which prevented the formation of fractures and, consequently, their propagation, thereby increasing the tensile stress (Choudary et al., 2020). Additionally, specimens with smaller aluminium mesh sizes have greater tensile performances compared to those with larger mesh sizes. This finding was substantiated by a study conducted by Tanawade and Modhera (2017),

wherein it was observed that the close proximity of the mesh wires resulted in elevated tensile loads on the specimens. The presence of aluminium ductile material might cause slight curvature linear trends. The curve showed a similar trend in hybrid composite material reinforced with carbon and glass fibres with wire mesh, as Hasselbruch et al. (2015) and Prakash and Jaisingh (2018) reported. The failure points for overall specimens had significant differences at different strain levels.

Table 4
ANOVA results for tensile properties

Source	DF	Adj SS	Adj MS	F-value	P-value	% Contribution
1. Tensile stress						
Stacking sequence (SS)	3	71.93	23.978	10.41	0.000	39.49
Mesh size (MS)	1	49.93	49.931	21.69	0.000	27.41
SS * MS	3	23.47	7.824	3.40	0.044	12.88
Error	16	36.84	2.302			
Total	23					
2. Tensile strain						
Stacking sequence (SS)	3	0.6461	0.21538	15.57	0.000	54.74
Mesh size (MS)	1	0.1190	0.11900	8.60	0.010	10.08
SS * MS	3	0.1938	0.06462	4.67	0.016	16.42
Error	16	0.2214	0.01384			
Total	23					

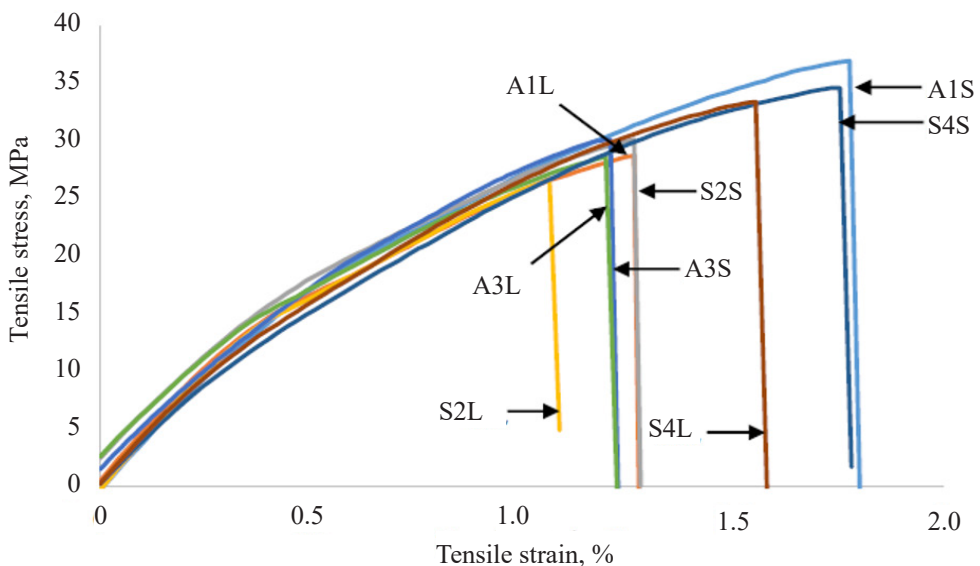


Figure 2. The tensile stress-strain curve of BFAMRP composites

Table 5 tabulates the tensile stress and tensile strain results. Among those with considerable maximum stress and strain values were specimens A1S, with a stress value of 34.22 MPa and a percentage strain value of 1.6%, respectively. This result was influenced by the aluminium mesh position on the exterior of the composite, which served to safeguard the bamboo fibre nonwoven mat layer, which was more delicate. Furthermore, the stress and strain values of this specimen were also enhanced by using a small mesh aluminium mesh sheet. These results were also supported by a study conducted by Tanawade and Modhera (2017), in which it was noted that the mesh wires being positioned closely together led to increased tensile loads on the specimens. In comparing the specimen S2L's lowest value of 27.28 MPa for tensile stress and 1.1% for tensile strain, the data showed an increase of 25.4% for tensile stress and 45.45% for tensile strain percentage.

Table 5
Tensile properties of BFAMRP composites

Specimens code	Tensile stress, MPa		Tensile strain, %	
	Mean	Std Dev	Mean	Std Dev
A1S	34.22	2.45	1.61	0.19
A1L	28.11	0.58	1.21	0.11
S2S	29.97	0.55	1.30	0.05
S2L	27.28	0.77	1.10	0.09
A3S	29.99	1.16	1.10	0.10
A3L	29.17	1.86	1.17	0.02
S4S	34.15	0.83	1.56	0.17
S4L	32.25	2.39	1.53	0.09

Regarding the failure behaviour, the specimens could be divided into two groups: (1) specimens with the aluminium mesh as the outer layer and (2) specimens with bamboo fibres as the outer layer. Group 1, which consisted of specimens A1S, A1L, S4S and S4L, indicated the same failure behaviour. The ductility of the aluminium mesh sheet at the external layer could have functioned as the shield to protect the bamboo fibre layer in the middle structure of the composites. The inclusion of the aluminium mesh with the fibre may have resulted in less void formation and an increase in the fracture strength of this composite. This improvement may result in less microcrack propagation on the composite (Singh & Rajamurugan, 2021). The aluminium mesh elongated until it broke and peeled away from the matrix due to the constant pressure on the specimens. Once the aluminium mesh was broken, the load caused the matrix and the fibre to break. Due to the fragility of the fibre and matrix, the crack spread, and the specimen broke. Moreover, the mesh geometry of the aluminium sheet was followed by the matrix crack.

Meanwhile, the specimens with bamboo in the external stacking sequence had a different trend. The specimens were obtained from composites S2S, S2L, A3S, and A3L. The load applied to the composite caused the matrix to fracture and the fibre to break, as these materials (bamboo fibre-reinforced polymer composites) are known for their brittle properties (Chin et al., 2020). Cracks soon reached the layer of aluminium mesh. The core of the composite's aluminium mesh was designed to withstand abrupt increases in pressure by stretching out until the wire broke and pulled away from the matrix, ultimately collapsing the specimen and explaining its low strength and strain.

Flexural Test

Table 6 displays the ANOVA for the flexural characteristics of BFAMRP composites. A *P*-value of less than 0.05 indicated that the stacking sequence significantly affected flexural strength and strain. In contrast to its tensile properties, the flexural properties of this composite material were not considerably impacted by the mesh size. The percentage contribution further demonstrated that the stacking sequence was the primary factor influencing the flexural strength and strain of the BFAMRP composite.

Table 6
ANOVA results for flexural properties

Source	DF	Adj SS	Adj MS	<i>F</i> -value	<i>P</i> -value	% Contribution
1. Flexural stress						
Stacking sequence (SS)	3	507.2	169.06	5.63	0.003	28.21
Mesh size (MS)	1	113.6	113.63	3.78	0.061	6.32
SS * MS	3	215.7	71.89	2.39	0.087	12.00
Error	32	961.4	30.05			
Total	39					
2. Flexural strain						
Stacking sequence (SS)	3	3.36883	1.12294	47.16	0.000	69.93
Mesh size (MS)	1	0.01170	0.01170	0.49	0.488	0.24
SS * MS	3	0.67503	0.22501	9.45	0.000	14.01
Error	32	0.76204	0.02381			
Total	39					

The flexural stress-strain curve of BFAMRP composites is shown in Figure 3. The image of the stress-strain curve illustrated that all specimens have identical curve lines since flexural stress linearly increased until it reached the maximum stress point before failure. From observation, the trend of the flexural stress-strain curve of BFAMRP composites differed from tensile properties. Specimens with bamboo fibre as the outer layer mostly had greater flexural properties, especially those with small aluminium mesh sizes. The

observed high flexural stress value was presumably attributed to the surface treatment procedure conducted on bamboo fibre, as documented by Batista and Drzal (2020). The observed phenomenon also can be attributed to the inherent structural characteristics of bamboo fibres, specifically in the configuration of a nonwoven mat. This particular form of reinforcement offers numerous advantages when incorporated into polymer composites. Notably, the bamboo fibres' commendable z-directional strength played a pivotal role in mitigating delamination issues, thereby enhancing the overall performance of the composite material (Patnaik et al., 2019). This phenomenon may indirectly impact the flexural properties of the composite. According to their stacking order, the composites' failure behaviour can be divided into two types. The first group included the specimens A1S, A1L, S4S, and S4L, composites with an exterior layer of aluminium mesh sheet.

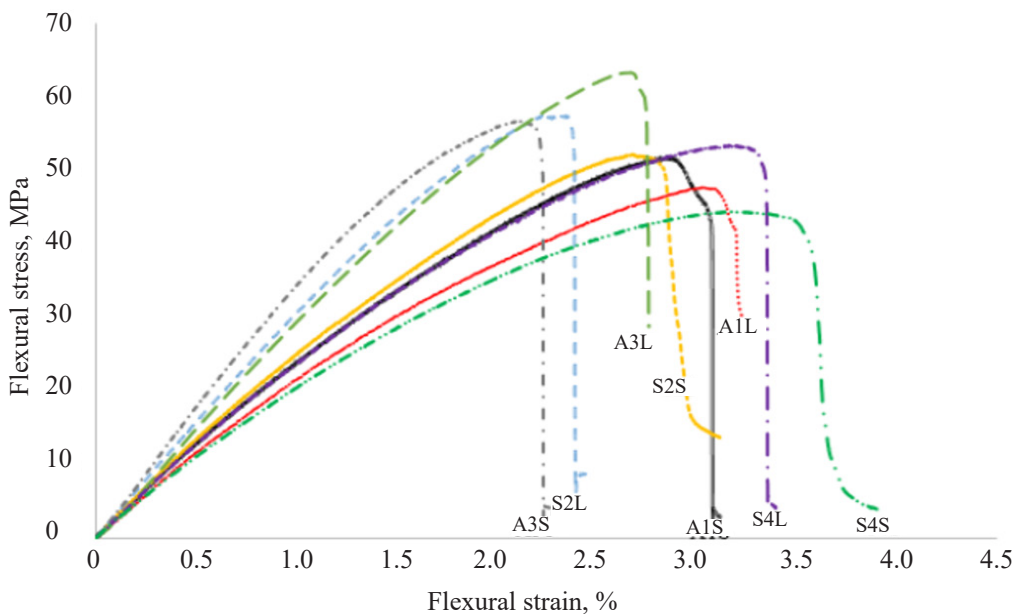


Figure 3. The flexural stress-strain curve of BFAMRP composites

The flexural stress and strain values for all specimens are shown in Table 7. Specimen A3L had the highest stress of all specimens, measuring 59.06 MPa. This specimen was expected to be made up of three layers of bamboo fibre and two layers of aluminium mesh, which is necessary since bamboo fibre had a higher tensile stress than aluminium mesh. Furthermore, using bamboo fibre nonwoven mat as the composites' outer layer improved the specimens' flexural stress since the fibre had a better bonding with the epoxy, which increased the load-sharing ability of the material, resulting in higher flexural stress of the specimens (Sadoun et al., 2021). In contrast, specimen S2L had the lowest flexural stress

of 44.63 MPa, a 24.4% decrease over A3L. Although both A3L and S2L had a layer of bamboo fibre nonwoven mat on the outside of the composite, their flexural stress values differed due to the number of bamboo fibre layers and stacking sequences. The aluminium wire mesh in the alternative lay-up of specimen A3L ensured sufficient toughness and exhibited plastic deformation that increased the flexural stress of this specimen (Hu et al., 2022). Meanwhile, specimens S4S and S4L had the largest flexural strain, with 3.16%, a 40.44% increase from the lowest percentage strain, 2.25%, for composite A3S.

Table 7
Flexural properties of BFAMRP composites

Specimens code	Flexural stress, MPa		Flexural strain, %	
	Mean	Std Dev	Mean	Std Dev
A1S	50.65	2.60	2.89	0.20
A1L	51.19	5.34	2.94	0.14
S2S	52.17	3.53	2.69	0.17
S2L	44.63	4.23	2.36	0.14
A3S	57.12	4.83	2.25	0.19
A3L	59.06	4.12	2.65	0.08
S4S	56.23	2.01	3.16	0.09
S4L	47.81	2.60	3.16	0.17

Four specimens, A1S, A1L, S4S and S4L, exhibited the same failure behaviour. At the layer of aluminium on the bottom of the composites, earlier damage initiation can be noticed. The mesh's wire elongated under constant pressure until it broke. The first ductile failure delayed the other laminate's total failure due to the aluminium's ductility, increasing the specimen's flexural strain (Sadoun et al., 2021). The specimen's bottom view revealed that the damage or crack closely resembled the mesh pattern of the aluminium mesh sheet. The matrix and the fibre laminate in the centre of the composite were still under pressure from damage propagation. The cracks grew until they reached the top layer and ultimately failed.

The specimens S2S, S2L, A3S, and A3L exhibited different types of failure. In their stacking structure of composites, all these specimens featured an exterior layer of bamboo nonwoven mat. The damage initially manifested as a matrix crack at the specimen's base. Flexural strength for this specimen was higher than that of other groups since fibre can withstand a significantly higher load than aluminium. This finding was consistent with that obtained by Xie et al. (2019). The break, however, rapidly spread until it reached the layer of aluminium mesh in the centre of the composites due to the brittle nature of the matrix and bamboo fibre.

Surface Morphology

Scanning electron micrographs of the tensile and flexural fractured surfaces were carried out to observe the interaction between the reinforcements and the matrix and the fracture behaviour. Previous researchers have stated that the bad adhesion of each component in the composites usually causes an issue such as delamination (Hasselbruch et al., 2015; Truong et al., 2019) and can cause catastrophic failure of the composites.

Figure 4 shows the fractured surfaces of the tensile test specimens. All specimens showed a similar failure mechanism: fibre breaks, aluminium strain pulled out from the matrix and matrix crack. The fractured specimen images indicate that aluminium wire was pulled out from the matrix, which might have caused the pull-out mechanism. However, the pull-out case happened in a particular area and might not have caused delamination between the aluminium mesh and the matrix. Almost all images showed the failure of the Al wire mesh in a stretched form (plastic deformation) when subjected to tensile loading. The ductile nature of the Al wire mesh caused the specimen to fail by pulling the Al out of the matrix and tearing it off in a cone fracture. Furthermore, the effect of the matrix around the Al wire mesh indicated a strong bond between the Al wire mesh and the matrix (Singh & Rajamurugan, 2021).

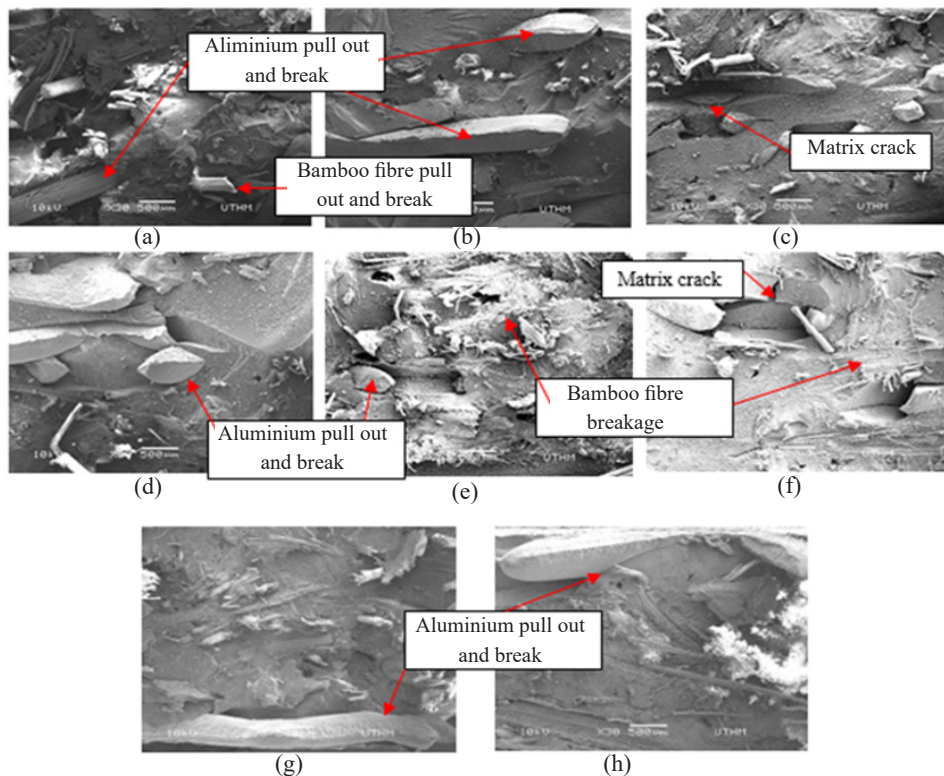


Figure 4. Cross-sectional SEM images of the tensile test specimens: (a) A1S, (b) A1L, (c) S2S, (d) S2L, (e) A3S, (f) A3L, (g) S4S, and (h) S4L

The same phenomenon was observed in the SEM image from flexural specimens in Figure 5. Most failures came from the fibre breakage, aluminium, and fibre pulled out from the matrix and matrix crack. The flexural strength of the composites was affected by the pull-out length, whether the matrix still adhered to the fibre and aluminium surface after pull-out, and whether there was fibre breakage on or near the matrix fracture surface, similar to the findings of (Cavalcanti et al., 2019).

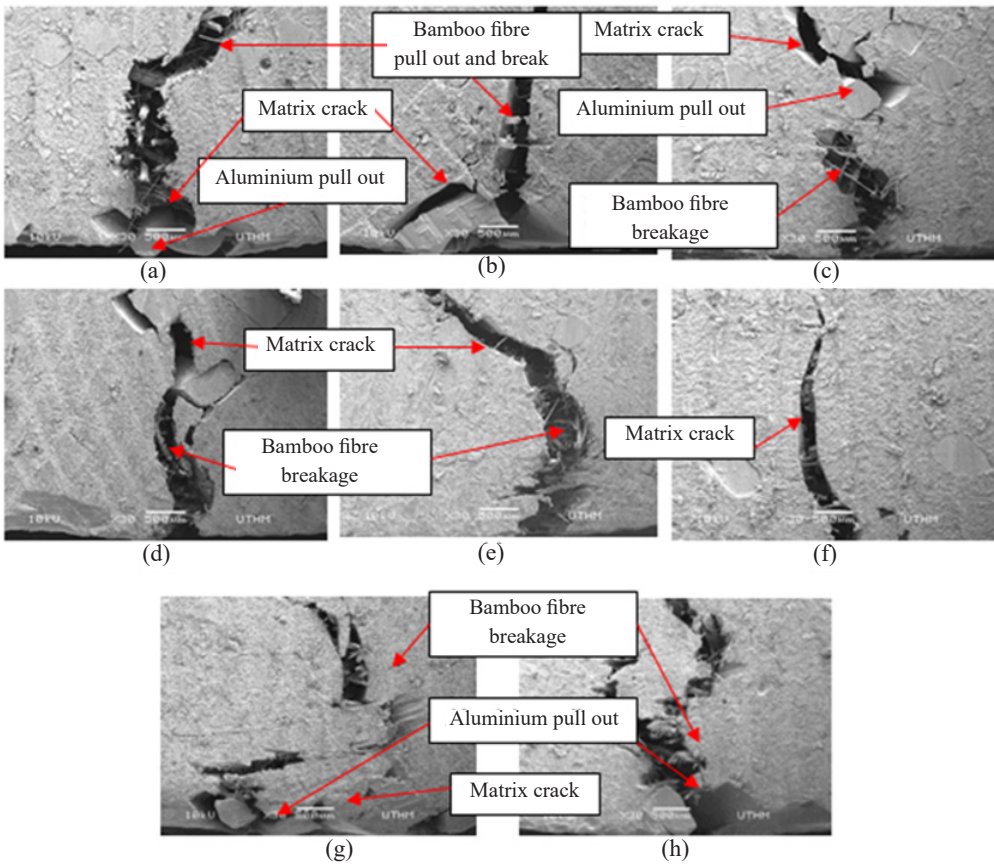


Figure 5. SEM images of the surface of the flexural test specimens: (a) A1S, (b) A1L, (c) S2S, (d) S2L, (e) A3S, (f) A3L, (g) S4S, and (h) S4L.

CONCLUSION

Eight BFAMRP composites were created and assessed for their tensile and flexural properties in this experiment. Subsequently, the specimens underwent SEM analysis. The ANOVA analysis demonstrated that the stacking sequence substantially impacted tensile and flexural performance, whereas the mesh size solely affected tensile performance. Specimens using an external layer composed of aluminium mesh demonstrated superior

tensile capabilities in comparison to specimens incorporating bamboo fibre nonwoven mats. The tensile and flexural strengths were influenced by the size of the aluminium mesh, with smaller mesh sizes resulting in greater strengths. The stacking structure also affected the flexural properties, with alternating structures exhibiting superior performance when using larger mesh sizes. In comparison, sandwich structures showed better results with smaller mesh sizes. The scanning electron microscopy (SEM) images revealed that the failure of the composite material was mainly caused by the breakage and pull-out of the fibres and aluminium, suggesting a strong adhesion between the reinforcement and the matrix.

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Characterisation of Miswak (*Salvadora persica*) Fibre-reinforced Polylactic Acid Composites Prepared by Twin Screw Extrusion

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ABSTRACT

Processing of polymer composites employing fibres from sustainable sources as reinforcement has drastically grown in recent years. This research used Miswak fibres (MF) and polylactic acid (PLA) as the main materials for composite processing. Natural fibres typically include a hydroxyl group (-OH), which makes them hydrophilic. In contrast, the hydrophobic nature of polymer matrices causes them to naturally repel water. This problem was resolved by chemically altering the surface of natural fibres using a 2 wt% sodium hydroxide (NaOH) solution. In this paper, the effect of alkaline treatment has been proven by performing chemical analysis, tensile properties, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC) to analyse the influence of treated MF content

on composite characteristics. The results revealed that biocomposites with modified miswak fibres exhibited better properties than untreated miswak fibres-reinforced polymer biocomposites. Treated MF/PLA composites showed an increase in tensile strength of 52.9% and tensile modulus of 8.16%. From the chemical composition test, lignin composition was reduced from 5.09% to 3.06% and hemicellulose from 28.12 to 10.62% after MF was treated. Meanwhile, thermal properties for both TGA and DSC

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revealed the elimination of hemicellulose and lignin characteristic peaks, improving the thermal stability of the treated MF/PLA composite. Thus, compared to a pristine sample, the resultant composites' higher mechanical strength and thermal stability demonstrated the significance of chemically treated natural fibres. The novelty of this research is the data on miswak fibre treatment, as no research has been found for this selected treated fibre.

Keywords: Alkali treated fibre, extrusion, miswak fibre, polylactic acid, thermal properties

INTRODUCTION

Biodegradable plastic development has recently garnered more attention as a potential replacement for non-biodegradable polymers in most applications. Using biodegradable plastics would reduce pollution and waste disposal concerns while being environmentally friendly. Biodegradable plastics can be created with both natural and synthetic polymers. Natural polymers are produced largely from renewable resources, while synthetic polymers are derived from non-renewable petroleum resources (Shafi et al., 2020a). Many studies have been performed to reduce the cost of production and the negative environmental effects—the prospect of employing entirely natural fibres biodegradable in conjunction with thermoplastic degradable materials. Blending natural fibre with polymer could improve the properties of composite in terms of strength and other performance. However, the limitation on incompatibility between fibre and polymer may be overcome by surface modification, chemical treatment and improved blending processing (Ezeamaku et al., 2022).

Due to the fibres' incompatibility with the polymer matrix, the composites' mechanical properties are poor due to low fibre-matrix interfacial bond strength and poor matrix resin wetting of the fibres. Therefore, surface modification using physical or chemical approaches tends to mitigate these disadvantages (Shafi et al., 2023). Miswak fibre, which belongs to the *Salvadoraceae* family, is a durable, robust, and adaptable fibre used rarely in polymer composites. Miswak is commonly used for a variety of aspects, most notably its antibacterial properties and low production costs. It is also convenient to obtain from the source. Miswak fibre, derived from *S. persica* roots and combined with PLA pellets, may be utilised as fibre reinforcement in natural fibre composites with greater sustainable benefits, such as renewable resources and a smaller carbon footprint. By testing and obtaining their behaviours and properties, there should be a good and efficient mixing quantity of blending weight percentage ratio between them. The idea is that it may be utilised in a specific application, such as a toothbrush handle. Detailed information about miswak fibre collected and obtained from previous studies is shown in Table 1.

Table 1
Characteristics of miswak fibre

Characteristics	Description	Reference
Colour	Brown/light brown/pale yellow	Moawed (2013)
pH	6.3 (1%), 4.5-4.6 (20%) (miswak storage time before preparation affects the pH)	Moawed (2013); Moawed and Abulkibash (2016)
Density (Bulk)	0.54 g/cm ³ , 0.67 g/cm ³ (dried at 250°C)	Moawed (2013); Moawed and Abulkibash (2016)
Chemical composition	Cellulose, hemicellulose, lignin	Moawed (2013); Moawed and Abulkibash (2016); Ramadan and Alshamrani, (2016)
Functional groups	Phenolic, carboxylic, alcoholic, and amine groups	Alili et al. (2014); Moawed and Abulkibash (2016); Tahir et al. (2015)
Phytochemicals	Sulphur, fluorides, chloride, vitamin C, silica, tannins, benzyl isothiocyanate, alkaloids, butanediamide, and essential oils	Abhary and Al-Hazmi (2016); Chaurasia et al. (2013); Ahmad and Rajagopal (2014); Amoian et al. (2010)

Polymer-reinforced composites have significantly improved the physical characteristics of polymeric materials. Fillers are commonly used to reinforce polymers in the creation of high-performance plastics. Manufacturers have been filling polymers with particles/fillers (polymer composites) to improve the characteristics of polymer products for many years. PLA is a biodegradable polymer manufactured from lactic acid (or lactide), which may be derived from renewable sources such as corn or sugarcane (Mukherjee & Kao, 2011; Murariu & Dubois, 2016; Nofar et al., 2019). PLA was hailed as one of the most promising biodegradable polymers, with excellent biodegradability and processability. The versatility of PLA material was expected to take the place of several non-biodegradable engineering plastics (Saini et al., 2016; Xu & Song, 2015). However, its uses were limited due to its weak heat stability and mechanical qualities (Sarasini, 2017; Satyanarayana et al., 2009; Wang et al., 2021). Many approaches, such as annealing and adding nucleating agents, have been proven to increase the thermal stability or mechanical characteristics of PLA materials (Ageyeva et al., 2021; Aliotta et al., 2017; Moser et al., 2016; Nagarajan et al., 2015), built composites with fibre or nanoparticles (Raquez et al., 2013; Yu et al., 2010; Yusoff et al., 2016; Zhang et al., 2015), extend chain (Corre et al., 2011; Correia et al., 2022), and introduce crosslinking structures.

Chemical treatment procedures often involve reagent functional groups that can interact with the fibre structures and alter their composition. As a result, the fibres' propensity to absorb moisture diminishes, facilitating better compatibility with the polymer matrix. Fibre

strands that have undergone chemical alteration exhibit changes in their surface topography, crystallographic structure, and removal of surface contaminants. On the other hand, fibres treated with alkali chemicals have rougher surfaces, which improves mechanical interlocking and increases the amount of cellulose exposed on the fibre surface. Chemical and physical treatments improve fibre-matrix compatibility and change the structure and surface of the fibres (Li et al., 2007). The mercerisation method requires soaking fibres in an alkaline solution to treat them. This procedure improves resin-fibre interfacial bonding, reduces fibre diameter, and removes oil and hemicellulose from fibres (Sherwani et al., 2021; Siakeng et al., 2020a). The surface modification of sodium hydroxide (NaOH) causes acetylation of a portion of the hydroxy surface functional groups of fibres, which inhibits moisture absorption in the fibre cell membrane, leading to high fibre wetting capacity via the matrix (Sherwani et al., 2021; Siakeng et al., 2020b). The treatment also promotes mechanical interaction between the fibres and the polymeric by changing the surface abrasion of the miswak fibres. Alkaline solutions have not been used to cure miswak fibre, yet this research attempts.

The blending of PLA and MF is performed using a twin-screw extruder. Since it delivers homogeneous, high-shear compounding, twin-screw extrusion is excellent for melt blending procedures for making thermoplastic composites. Materials mixed in a two-roll mill or a high-speed mixer have strong mechanical properties as a result (Guillaume et al., 2013; Hyvarinen et al., 2020; Kuzmin & Radaikina, 2020; Mikulyonok, 2013; Singh & Singh, 2020). When employing reinforcing agents in polymer composites, natural fillers such as clay, silica, calcium carbonate, hemp fibre, and wood fibre have been shown to produce a variety of advantageous qualities (Arbelaiz et al., 2005; Hristov & Vasileva, 2003; John & Thomas, 2008; Pluta et al., 2006; Sgriccia & Hawley, 2007; Tokoro et al., 2007). Discovery of novel natural fibres, such as miswak fibres, has become a way to address resource limitations and environmental pollution issues by examining the possible use of restricted natural fibre resources for diverse applications (Chaaben et al., 2020; Savaş, 2018). A novel plant fibre, miswak fibre (MF), was combined in a polylactic acid (PLA) matrix using a twin-screw extruder to use the agriculture byproduct fully.

This research examines the influence of MF on the thermal and mechanical characteristics of the PLA matrix. The fibre surface was chemically treated to examine the MF's blending and bonding potential on the polymer matrix. PLA was treated similarly to polypropylene; hence, it should be possible to extrude miswak fibre-reinforced composites. Tensile testing was used to examine the composites' mechanical properties; meanwhile, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were used to examine thermal characteristics.

MATERIALS AND METHOD

Materials

Miswak chewing sticks manufactured by Al-Khair Premium Natural Products, Karachi, Pakistan, were purchased from the local store Al-Imtinaan Sdn Bhd. Then, the miswak chewing stick was chopped, crushed, ground, and sieved into miswak fibre. Polylactic acid (PLA) thermoplastic polymers in pellets, grade 2003D, were obtained from Polycomposite Sdn. Bhd. in Seremban, Malaysia. They have a specific gravity of 1.24 g/cm³, a melting temperature of 145–160°C, and a glass transition temperature of 55–60°C. Evergreen Engineering and Resources, Semenyih, Malaysia, provided the sodium hydroxide (NaOH).

Alkali (NaOH) Treatment

The miswak fibre was then soaked in a 2 wt% NaOH solution at room temperature for 60 min. The fibre-to-solvent ratio was 1:10 (w/v). After pretreatment, the material was washed and rinsed several times with distilled water and dried for 24 hours at 80°C in an oven.

Composite Processing via Extrusion

PLA with treated and untreated MF were maintained at 6–8% moisture content before blending the material in twin screw extrusion. A mixture of PLA and MF was premixed first using the dry mix technique before feeding into the main feeder. The extrusion consists of 10 heating zones, and the temperature was set starting from 160–190°C. The main screw frequency was maintained at 0.44Hz with a die head pressure of 0.10 MPa. The filament will pass through the die and be dried before being cut into small pellets.

Compression Moulding

The composite pellets from the previous phase were placed in a mould with 150 mm x 150 mm x 3 mm dimensions. The compression moulding (hot-press) machine was set at 170°C and underwent preheating for 10 min, fully press heating for 10 min and cooling for 10 min. The material was then cut into shapes in accordance with characterisation testing. Five specimens for every combination were tested for each test and then formed into sheets with dimensions of 150 mm x 150 mm x 3 mm. Details on the composite process are presented in Figure 1.

CHARACTERISATION

Scanning Electron Microscopy (SEM)

The fibre surface was observed using a scanning electron microscope (Hitachi S-3400N) outfitted with an energy-dispersive X-ray at an emission current of 58 μ A and an accelerating

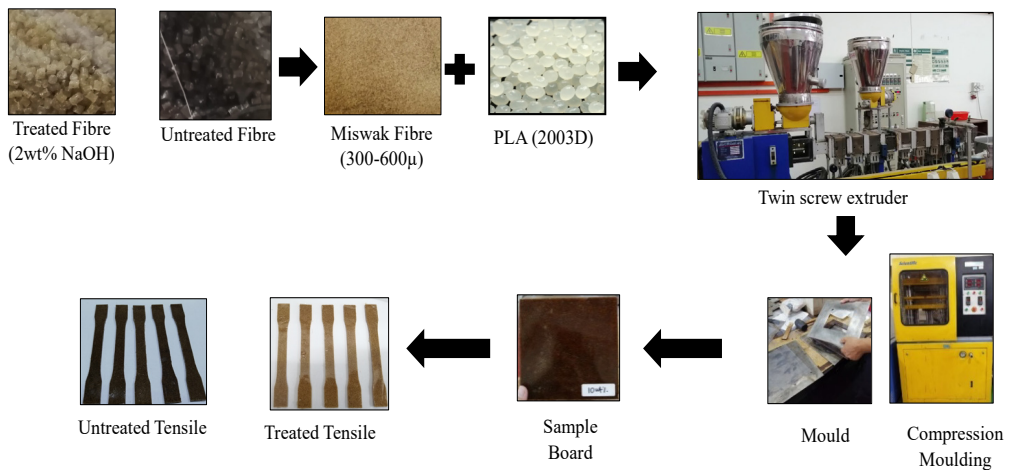


Figure 1: PLA/MF composite fabrication

voltage of 15 kV. The fracture ends of the samples were mounted on an aluminium stub and covered with a thin layer of gold to prevent electrostatic charging during testing.

Tensile Testing

A tensile test was performed on a flat dog-bone-shaped sample that was cut on the plastic mould cutter in the specified length of 150 mm, width of 23 mm and thickness of 3 mm, respectively, as per ASTM D638-14 test standards (ASTMD638-14, 2014). The 5kN Bluehill INSTRON 5566 universal testing machine was used to perform the test. The composites were grasped at a gauge length of 30 mm, and the crosshead speed was adjusted at 5.0 mm/min. A conditioning room was used to hold the five samples, and the test was run at 20.5°C and 48% relative humidity (RH). Each test was run five times for each sample to determine the outcome. The average of these five samples was then computed.

Thermogravimetric Analysis (TGA)

The samples were tested using thermogravimetric analysis (TGA) at a 10°C/min scanning rate on a Mettler Toledo TGA/DSC 1HT Stare System (Switzerland) between 30 and 600°C. TGA is a technique that monitors weight changes as a sample is heated at a consistent rate to evaluate the thermal stability of materials and their fraction of volatile components.

Differential Scanning Calorimetry (DSC)

The thermal transition properties were determined by differential scanning calorimetry using a Mettler Toledo TGA/DSC 1 HT apparatus in accordance with ASTM D3418. The weights of the treated and untreated PLA composite constructions reinforced with miswak fibre ranged from 6 to 8 mg, respectively, and the weight of the miswak fibre structure was

between 6 and 7 mg. They were heated in a nitrogen flow of 50 mL/min between 25 and 200°C at a steady rate of 10°C/min. The sample masses ranged from 5.0 to 7.0 mg in all the tests. Equation 1 is used to calculate the degree of crystallinity:

$$X_c = \frac{\Delta H_m^a}{\Delta H_m^{100}} \times 100 \quad (1)$$

where X_c is the degree of crystallinity (%); ΔH_m^a is the melting enthalpy of the sample (J/g); and ΔH_m^{100} is the melting enthalpy of 100% crystalline PLA (J/g), which was considered to be 93.7 J/g (Jia et al., 2017).

Chemical Composition Test

Chemical composition tests were conducted using the TAPPI standard procedure to determine the fibre's extractive, cellulose, hemicellulose, and lignin composition. The extractive test was initially conducted, followed by the holocellulose, cellulose and lignin tests.

The extractive test was conducted using the TAPPI T204 standard (T204). A 2g sample was put in a thimble and placed inside a condenser. Three samples containing thimbles were placed inside the condenser, each for extractive, holocellulose and lignin tests. A 160ml ethanol-toluene solution was poured into a round-bottomed flask to extract the solvent from the sample. All round-bottomed flasks and the condenser were placed on the Soxhlet extraction hotplate. The test took about 6 to 8 hours to obtain the extractive solvent. Then, a bottomed flask with the sample was placed in a Rotavapor machine to obtain the extracted solvent. The round-bottomed flask with the sample was then dried in an oven at 105°C, and the sample was periodically weighed until a constant sample weight was achieved.

A holocellulose test was conducted according to standard procedure. A 2 g sample was placed into a 250 mL beaker. A 100 mL of distilled water was poured into the beaker, followed by 1.5 g of NaClO₂ and 5 mL of 10% acetic acid. Then, 5 mL of 10% acetic acid was added in 4 repeated pours after every 30 min. Then, 4 repeated pours of 1.5 g of NaClO₂ were added every 30 min. The 250 mL beaker was boiled in a water bath at 70°C. Then, 30 min after the last addition of 1.5 g NaClO₂, the beaker was cooled in an ice bath for 1 hour. Then, the sample was rinsed with iced distilled water and acetone in a crucible before being placed in a desiccator and weighed.

The cellulose test was conducted using the TAPPI T203 standard (T203). A sample from holocellulose used for this test was then put into a beaker with 75 mL of 17.5% NaOH before adding 15 mL of the same solution and stirred for 1 min. After that, 10 mL of NaOH was poured while stirring for 45 sec before adding 10 mL of NaOH while stirring for 15 sec and let stand for 3 min. After this, 10 mL of NaOH was added 4 times, stirred every 2.5 minutes, and left to stand for 30 minutes. Then, add 100 mL of distilled water while

stirring and allow to stand for 30 min. Later, weigh the empty crucible before filtering the sample into the crucible. Rinse the sample with 25 mL of 8.3% NaOH and 650 mL of distilled water. Then, fill the crucible with 2N acetic acid and let it stand for 5 min before rinsing it with distilled water. After that, the sample was oven-dried at 105°C and weighed.

The Lignin test was conducted using the TAPPI T222 standard (T222). A sample weighing 1 g was put in a conical flask. Distilled water and 15 mL of 72% H₂SO₄ were poured into the conical flask. The solution was boiled for 4 hours with condenser reflux. Then, the sample was filtered in a crucible by rinsing it with hot water. Then, the sample was oven-dried and weighed.

RESULTS AND DISCUSSION

Scanning Electron Microscopy (SEM)

Figure 2a shows the SEM micrograph of untreated MF. The figure unequivocally demonstrates that MF's exterior comprised certain nodes, impurities, and wax. In essence, by eliminating wax and non-cellulose components from the outer layer, lignocellulosic fibre surface treatment could increase the fibre's surface wettability.

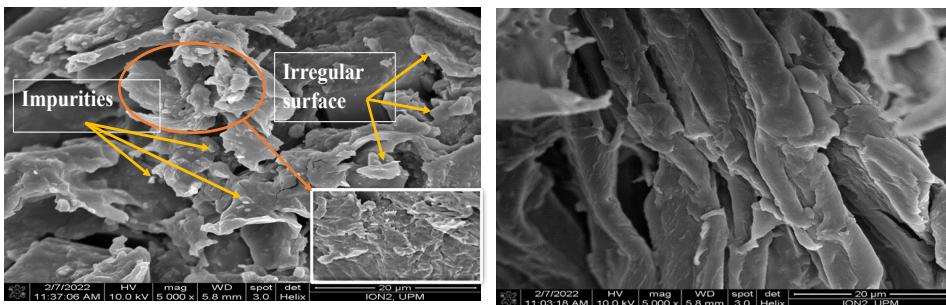


Figure 2. (a) SEM micrograph of untreated MF with 5000x magnifications and (b) SEM of 2% NaOH treated miswak fibre with 5000x magnifications

The composition of miswak fibre before and after alkali treatments is shown in Table 2. From the table, after alkali treatment, the cellulose content was increased, which led to an improvement in the properties of fibre. This treatment eliminated the lignin, wax, and oil that cover a portion of natural fibres, making the surface rougher and resulting in greater fibre interlocking with the polymer matrix. According to the micrographs, there were more residues on the surface of untreated miswak fibre (Figure 2a) than on the surface of treated miswak fibre (Figure 2b). Untreated miswak fibre is projected to have lower thermal stability than treated miswak fibre (Marques et al., 2014). The findings showed that following alkali treatment, cellulose content increased while hemicellulose and lignin content decreased. The mechanical properties may improve as a result of the observed increment in cellulose content.

Table 2
Composition of miswak fibre before and after alkali treatment

Miswak fibre	Cellulose (%)	Hemicellulose (%)	Holo-Cellulose (%)	Lignin (%)	Extractive (%)
Untreated	21.81	28.12	49.93	5.09	20.12
Alkali treated (2% w/v NaOH solution)	51.86	10.62	62.48	3.06	3.89

Tensile Properties

When defining a material, its tensile qualities are essential to be taken into consideration due to their dictate how long the material can endure controlled tension before failing. A tensile test, therefore, can be used to directly evaluate ultimate tensile strength, tensile modulus, and maximum elongation. Untreated MF/PLA composite tensile values were compared to treated MF/PLA composite. Figure 3 shows the mechanical properties of 100% PLA, 70% PLA with 30% untreated MF, and 70% PLA with 30% treated MF. Tensile strength, tensile modulus, and elongation were compared.

The tensile strength of 100% PLA was 52.11 MPa, and the tensile modulus was 2819 MPa. However, after adding 30% untreated MF, the tensile strength decreased to 38.16 MPa, and the tensile modulus decreased to 2742 MPa. It has been observed that adding natural fibre to the polymer matrix reduced the PLA/MF composite's tensile strength. This response provided a strong indication that the stress within the composite was absorbed by the matrix, indicating that the intrinsic fibres were unable to distribute stress evenly with the fibre (Shafi et al., 2020a) due to the load on the fibre mainly depends on the transfer of the matrix through the interface. The different properties of blended elements, which are PLA and MF, may cause the compound to not be fully homogenous during the blending process. The same trends were observed by Shafi et al., 2020a, who studied polybutylene succinate-reinforced empty fruit bunch fibre. However, with an addition of 30% treated MF, the results showed increments in both tensile strengths by 52.96% and tensile modulus by 8.16%. This finding proved the efficiency of the performed alkali treatment, which may influence the compatibility between fibre and polymer in composites. Other than that, the treated fibre can result in high fibre stiffness because of the higher crystallinity of hard cellulose.

Figure 4 depicts the results of elongation at the break of the composite at maximum load. The findings showed that composite with treated MF had the highest elongation rate compared with neat PLA and untreated MF composite. After adding 30% untreated MF, the elongation rate was reduced to 38.2%. On the other hand, the fibre treatment process might also contribute to the flexibility of the composite sample.

This behaviour showed increased fibre-to-PLA matrix bonding, which improved fibre-matrix interaction (Orue et al., 2016). Fibrillation was also brought on by the alkaline

treatment, which divided the composite fibre bundle into smaller fibres and shortened the fibres' diameter and length. The fibre aspect ratio rose as the surface roughness increased, resulting in better fibre-matrix interface adhesion (Das & Chakraborty, 2006; Orue et al., 2016; Yee et al., 2016). Furthermore, the alkali treatment improved fibre-matrix interaction by eliminating lignin and hemicellulose, resulting in larger inclusion. According to Rajesh and Prasad (2014), when compared to untreated jute/PLA and neat PLA, 10% alkali-treated short jute fibre/PLA showed a 7.5% increment in tensile strength because alkali surface treatment improved the compatibility of jute fibre and PLA matrix, resulting in effective transfer between fibre and matrix, as shown in the SEM study.

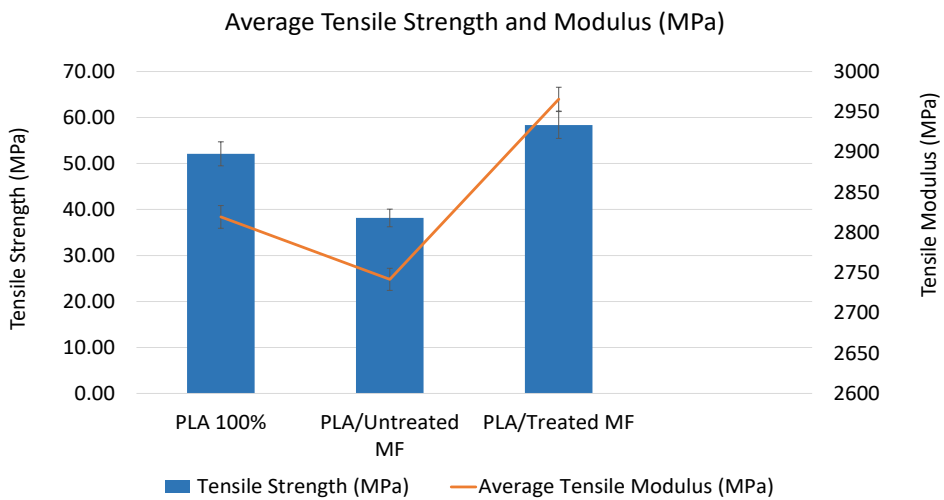


Figure 3. Average tensile strength and modulus

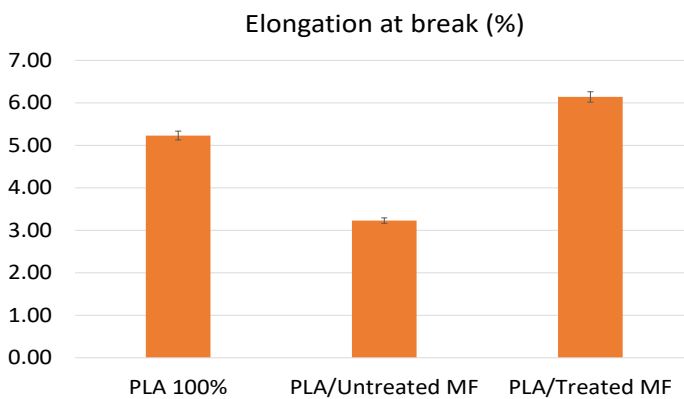


Figure 4. Average elongation at break

Thermogravimetric Analysis (TGA)

(a) Thermal stability of untreated and treated Miswak fibre

Thermal degradation is a crucial factor to consider when using high-temperature production techniques and lignocellulosic materials as reinforcements for polymers. Thermal degradation of lignocellulosic materials begins with moisture loss at temperatures below 100°C, followed by hemicellulose, cellulose, and lignin. Figures 5 and 6 demonstrate the weight loss and heat deterioration of untreated and treated miswak fibres.

The TGA curves for treated and untreated fibre are shown by the thermographs in Figure 5. At 90 to 100°C, weight loss of miswak fibre, both treated and untreated, started, suggesting that the fibre was losing moisture. The untreated fibre's next deterioration temperature, connected to hemicellulose components, was 130 to 195°C, and the third degradation temperature, related to the loss of cellulose components, was in the range of 195 to 280°C. Untreated fibre's final breakdown temperature was 280 to 600°C, denoting lignin components decomposition. The absence of a transition for hemicellulose components in treated fibre indicated that alkaline treatment successfully removed the components, and the next transition occurred in the range of 200 to 600°C for cellulose and lignin components. Untreated fibre lost 8%, 14%, 25%, and 60% of its mass at 88, 166, 261, and 334°C, respectively, whereas treated fibre lost 9% and 65% in that sequence at 94 and 353°C. Miswak fibre hemicellulose was thermally damaged at 166°C, while cellulosic content was degraded at 334°C, as demonstrated in the third stage. The cellulose content of treated miswak fibre was destroyed at 353°C, as shown by a peak in the 200 to 600°C. (Ray et al., 2002) obtained similar results, observing one peak in the weight loss curve of alkali-treated jute fibre. The initial peak did not occur in treated fibres because lignin and hemicelluloses were partially eliminated.

The DTG curves of untreated and treated miswak fibre are illustrated in Figure 6. The untreated fibre DTG curve shows 3 phases of decomposition, and the treated fibre DTG curve shows 2-phase decomposition. The untreated and treated fibre started to degrade at 88.9 and 94.6°C, respectively. This decomposition indicated moisture loss or volatile compounds in miswak fibre. The untreated fibre phase decomposition took place at 166.3°C, indicating degradation of hemicellulose components. The third phase of decomposition of untreated fibre occurred at 261.6°C, related to cellulose components degradation. At 334.2 and 353.7°C, respectively, the last phase of untreated and treated fibre degradation occurred. The results presented evidence that the alkaline treatment of miswak fibre improved the thermal stability of PLA/MF composites. The alkaline reaction removed the impurities, pectin, hemicelluloses, lignin, wax, and oil that covered the fibre surface. This treatment was predicted to improve the composites' thermal properties (Li et al., 2007).

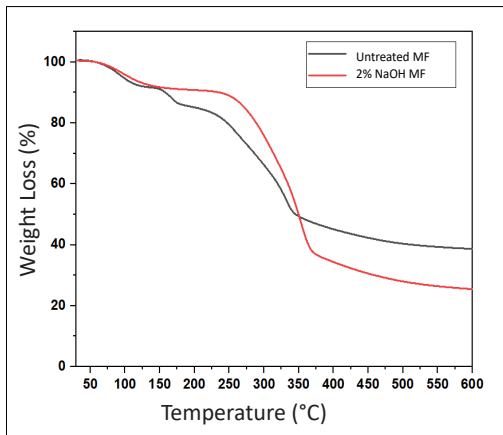


Figure 5. Thermographs of untreated miswak fibre with treated miswak fibre

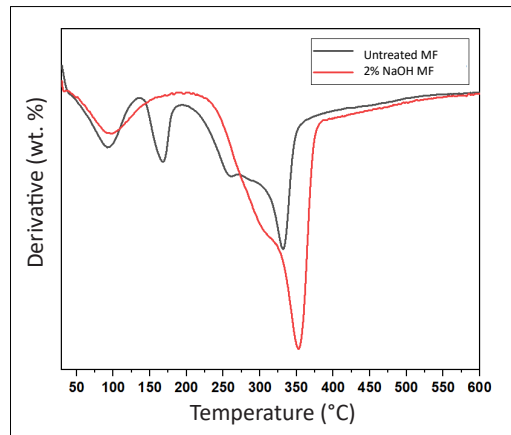


Figure 6. DTG curve of untreated miswak fibre and treated miswak fibre

(b) Thermal stability of untreated and treated MF/PLA composites

Figures 7 and 8 show the thermograms of PLA, untreated fibre/PLA, and treated fibre/PLA, with the results recorded in Table 3. Thermogravimetric curves for PLA, untreated fibre/PLA, and treated fibre/PLA composites are shown in Figure 7. At 367°C, the simple PLA weight loss curve was observed as a single step in the composite material. As demonstrated in Figure 7, the weight loss peak temperature of untreated fibre/PLA shifted to a lower temperature area. The maximum thermal degradation temperatures for untreated and treated miswak fibre were 334.25 and 353.72°C, respectively, substantially lower than the 367°C for plain PLA.

The thermal stability temperature decreased due to the low thermal degradation of the miswak fibre and the trace of moisture introduced by the miswak fibre. The pyrolytic reaction of the PLA matrix was accelerated by moisture in composite materials (Cornelissen et al., 2008). The degradation temperature tended to decrease with the addition of miswak fibre, resulting from miswak fibre's lower thermal degradation temperature. However, the treated fibre/PLA degradation temperature at 355°C was higher than the untreated fibre/PLA degradation temperature at 335°C. The mass loss for untreated fibre/PLA composite was 92% at 335°C, whereas the mass loss for treated fibre/PLA composite was 94% at 355°C in the specified sequence.

The derivative curves for PLA, untreated fibre/PLA, and treated fibre/PLA composites are shown in Figure 8. PLA had a maximum degradation temperature of 367.3°C and a residue concentration of 0.8%. The highest deterioration temperature for treated fibre/PLA was 355.2°C with a 3% residue percentage, whereas the maximum degradation temperature for untreated fibre/PLA was 335.4°C with a 3.7% residue concentration. Clearly, the presence of miswak fibre allowed for a fall in PLA thermal stability due to the miswak

fibre's reduced thermal stability. Increased treated coir fibre content caused a decrease in the thermal stability of PLA/treated coir fibre biocomposites due to coir fibre's reduced thermal stability, according to a study (Sun et al., 2017). However, alkaline treatment improved the thermal stability of treated fibre/PLA composites, with a 5.5% increase in maximum degradation temperature compared to untreated fibre/PLA composites. Moreover, the alkaline treatment resulted in fibrillation, decreased fibre diameter and length, and increased the fibre's aspect ratio. This behaviour improved the adhesion between the fibre and matrix at the interface because the higher surface roughness provided better PLA absorption. All of these factors improved the thermal behaviour of the fibre (Bakri et al., 2017).

Table 3
Thermal degradation temperature of miswak fibre/PLA composites

Samples	Tonset (°C)	Tmax (°C)	Residual (wt%)
PLA	336.3	367.3	0.8
Untreated fibre/PLA	298.5	335.4	3.7
treated fibre/PLA	320.2	355.2	3

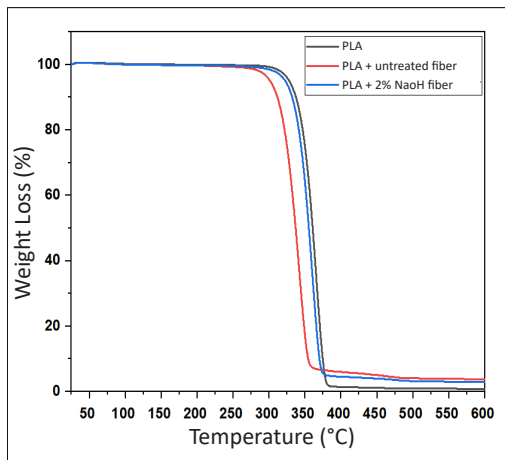


Figure 7. Thermographs of plain PLA, untreated fibre composite and treated fibre composites

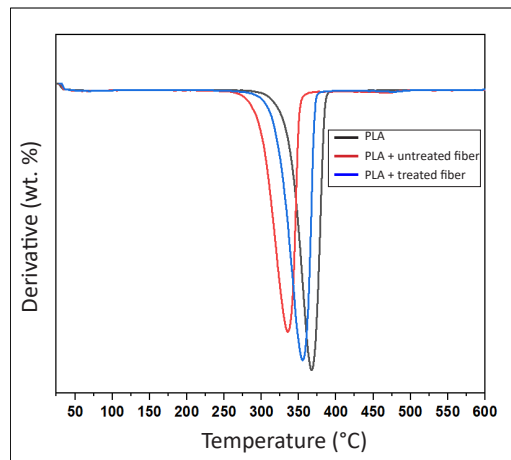


Figure 8. DTG curve of plain PLA, untreated fibre composites and treated fibre composite

Differential Scanning Calorimetry (DSC)

(a) DSC curve of untreated and treated Miswak fibre

Figure 9 shows the DSC curves of untreated and treated miswak fibre. Moisture loss caused a slight endothermic peak in the untreated fibre at temperatures below 100°C. The treated miswak fibre's DSC curve exhibited no exothermic or endothermic changes in the 25 to 200°C range, implying that the fibres were stable across the temperatures. In contrast, the

untreated miswak fibre's DSC curve revealed a strong endothermic peak at 166.70°C. This peak was caused by the breakdown of hemicellulose and cellulose, respectively. After treating bamboo fibre with alkali, Das et al. (2006) observed similar effects. It indicated that the hemicellulose and lignin fibres formed a stable network structure with the cellulose through extensive intermolecular and intramolecular hydrogen bonding, likely resulting in a stable peak between the temperature changes. In contrast, the alkali-treated bamboo exhibited two exothermic peaks, which correlated to alpha-cellulose on the DSC curves of the untreated and treated bamboo fibre samples in the range of 100 to 235°C (Das & Chakraborty, 2006).

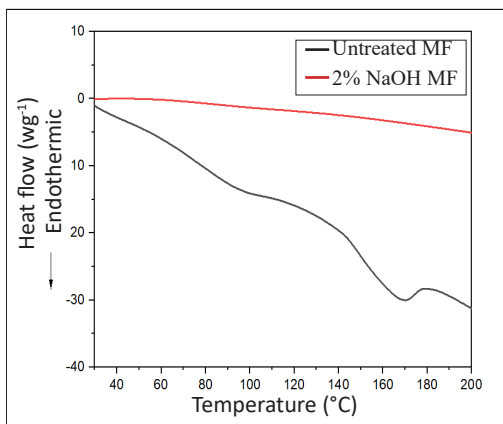


Figure 9. DSC curve of untreated and treated miswak fibre

(b) DSC curve of Miswak fibre/PLA composite

The thermograms in Figure 10 show the findings of the DSC scan listed in Table 4. The scan was performed to see how the untreated and treated miswak fibres affected the thermal performance of the composite. This thermogram was endothermic because the sensor collected the heat absorbed, and it was exothermic because the specimens released the heat. Although the curvature of the thermograms was similar, the heat released from the treated miswak fibre composites was higher compared to the untreated miswak fibre composites. For both

composite samples, the initial temperature peak occurred between 50 and 55°C. This peak was endothermic, indicating that the glass transition temperature was used as the starting point for the energy necessary to modify the molecular structure of PLA. The thermal breakdown proceeded until roughly 150°C when it was discovered that both samples were undergoing another endothermic process. This peak showed that the miswak/PLA composites were about to enter the breakdown phase.

Karsli and Aytac (2014) found that the degree of crystallinity and melting of composites made with treated short flax fibres/PLA/PC was greater in all circumstances than composites made with untreated short flax fibres/PLA/PC (Karsli & Aytac, 2014). When miswak fibres were incorporated into the PLA matrix, the crystallisation temperature (T_c) increased, indicating that miswak fibres can function as nucleating agents. Consequently, the process of matrix crystallisation was expedited (Jia et al., 2017). In comparison to plain PLA, there was a little drop in the melting temperature of composites, which might be due to the development of more defecting crystals, as seen by the minor expansion of their

endothermic peaks in the second peak DSC curve and regarding the thermal behaviour of composites, fibre composites that have undergone treatment exhibited a greater degree of crystallisation temperature in comparison to their untreated counterparts.

Table 4
Thermal properties of miswak fibre/PLA composites

Sample	Description	Tc (°C)	Tm (°C)	ΔHm (J/g)	Xc (%)
PLA	plain PLA	58.2	149.8	2	2.1
untreated fibre/PLA	PLA + untreated miswak fibre	56.9	148.3	6	6.4
treated fibre/PLA	PLA + 2% NaOH treated miswak fibre	57.3	148.6	5	5.3

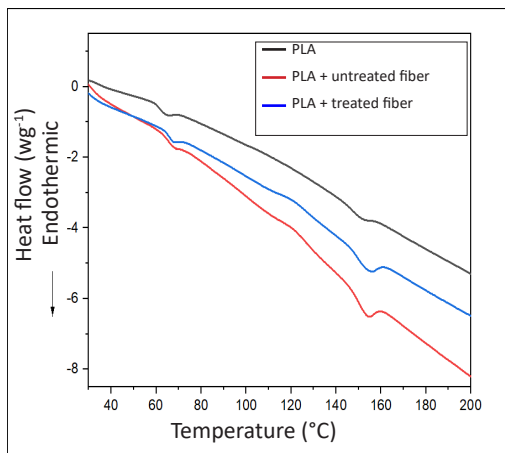


Figure 10. DSC curve of PLA, untreated fibre/PLA composite, and treated fibre/PLA composites

CONCLUSION

The current research examined the thermal stability and melting behaviour of PLA, untreated fibre/PLA, and treated fibre/PLA, in addition to their tensile strength, elongation at failure, and tensile modulus. When compared to neat PLA and untreated fibre/PLA composite, the mechanical strength of the treated fibre/PLA composites demonstrated the highest result for tensile strength, modulus, and elongation at break. The tensile strength of 30% untreated fibre decreased to 38.16 MPa, and the tensile modulus reduced to 2742MPa. However, with 30% treated MF, the tensile strength increases by 52.96% and tensile modulus by

8.16%. According to TGA and DSC data, adding miswak fibre to a composite impacted its thermal stability. Adding treated miswak fibre to this compound decreased residual weight and increased thermal stability. The melting temperature of miswak fibre was increased by alkaline treatment, which affected the melting temperature of the composite. PLA's tensile and thermal characteristics were affected by alkaline treatment. Overall, treating miswak fibre has a significant effect on the tensile characteristics and thermal stability of the PLA matrix. Future studies will examine this material to develop a miswak holder using 3D printer processing.

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Performance of Hybrid Fiber Reinforced Geopolymer Composites: Scientometric and Conventional Review

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ABSTRACT

Hybrid fibers are an interesting addition to reinforce geopolymer-based composites due to their advantages over single-fiber reinforcement. The performance of hybrid fibers is dependent on the fibers' composition, type, properties, length, and volume fraction. Therefore, this review discusses the state-of-the-art hybrid fiber-reinforced geopolymer composites (HFRGC) through two approaches: scientometric analysis and conventional review of HFRGC based on data extracted from Scopus from 2013 until 2023. The scientometric analysis was carried out by adopting VOS Viewer software and focuses on the annual publication of documents, top publication sources, co-occurrence keywords, researchers, top-cited papers, and countries. In contrast, the desk study refers to experimental data on the fresh properties and compressive, tensile, and flexural properties of HFRGC. This review output aids researchers in networking, promoting cooperative

research, exchanging ideas, and creating joint ventures among researchers of HFRGC worldwide. The performance of HFRGC obtained from the desk study showed the potential of HFRGC as an option for a greener composite that will benefit the construction industry.

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INTRODUCTION

Geopolymer is a type of inorganic substance that forms a polymeric structure made of by-product materials or waste materials like fly ash and slag that consist of aluminum (Al) and silicon (Si) as binders when reacting with an alkaline activator (Yang et al., 2022b, 2022a). Geopolymer composites (GPC) exhibit high mechanical strength and durability (Amran et al., 2021). These significant features include low porosity, high early strength (Lan et al., 2022), high performance in sulfate and acid environments (Zhao et al., 2021), high-temperature resistance, low energy utilization, and the release of less pollution during manufacture (Valente et al., 2021), making these composites a viable candidate for a variety of industrial applications. However, these composites are brittle materials with poor tensile strength. Incorporating fibers into geopolymers can enhance their durability by augmenting the toughness of the composites (Li et al., 2022).

Short and randomly dispersed fibers impact the cracking behavior, regulate the brittle fracture process, and enhance the toughness post-cracking (Abbas et al., 2022). A variety of fiber types, including synthetic and natural fibers, are widely used in geopolymer technology (Kozub et al., 2021; Silva et al., 2020). Each type of these fibers is divided into different categories, as illustrated in Figure 1. Synthetic fiber has distinguished properties such as excellent tensile strength, an extraordinary strength-to-weight ratio, electrical conductivity, and fatigue stability (Farooq et al., 2019).

Natural fibers are easily accessible, and renewable resources are widely available in several places. They possess the advantages of being biodegradable and low-cost. Many attempts have been made to increase the toughness and ductility of GPC by using different kinds of short filaments (Firas et al., 2024; Ramamoorthy et al., 2015).

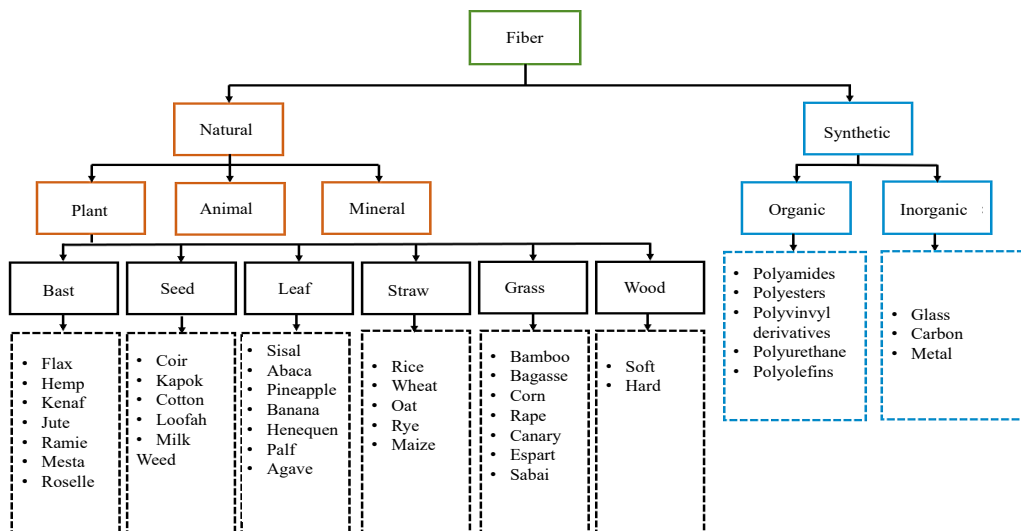


Figure 1. Classification of fibers according to their sources

Conventional fiber-reinforced concrete components only utilize a single kind of fiber. A single fiber possesses utility only within a restricted spectrum of strain and propagation of cracks. Consequently, a specific variety of fiber can enhance the strength or ductility of concrete composites. Steel, glass, polyvinyl alcohol, carbon, and asbestos fibers are examples of high-modulus and high-strength fibers that can efficiently increase the strength of geopolymer (Soe et al., 2013). Nevertheless, their inherent brittleness prevents significant enhancement in ductility. Low-strength fibers, such as polypropylene, nylon, and acrylic fibers, have greater efficacy in enhancing ductility and mitigating cracking (Pakravan et al., 2017).

As a result, mixing fibers with different chemical and mechanical properties is necessary for producing GPC with higher strength and ductility. Utilizing an appropriate combination of two or more fiber types can enhance the overall properties of concrete and synergistic performance (Feng et al., 2021). The combination of fibers is often known as hybridization (Geboes et al., 2022).

Therefore, lately, the majority of studies have been focused on utilizing different kinds of fiber in a geopolymer matrix to create hybrid composites, where the superior engineering properties of one fiber support the defects in the other type of fiber in the composite (Taghipoor & Sadeghian, 2022). Hybridization improves the mechanical characteristics of concrete to prevent cracks more effectively than single fiber-reinforced concrete. Incorporating two distinguished fibers leads to materials with distinctive properties (Alwesabi et al., 2022; Kan et al., 2020).

Bakthavatchalam and Rajendran (2021) stated that incorporating hybrid fiber steel and basalt fibers in a geopolymer matrix with the appropriate percentage could enhance the compressive, tensile, and flexural strength characteristics. Hybrid fibers improve the mechanical properties of the material by bridging cracks and distributing stress more uniformly, thereby inhibiting the propagation of cracks. Moreover, chopped basalt fiber has a great strength capability with concrete. Various hybridization systems depend on variations in several factors, like the fibers' diameter, length, and tensile strength. For instance, short fiber can prevent diffusion of the microcracks and then improve the strength of concrete, while long fiber can be effective in large-size cracks, hence improving the whole fiber's role in the concrete matrix (Chen et al., 2021a). Hence, in the hybrid composite, when one type of fiber fails, the load will transfer to another, making the composites depend on each other (Sapiai et al., 2020).

Traditional literature reviews are inadequate and subjected to prejudice in establishing an integrated and systematic correlation among multiple domains of investigation. The bibliometric analysis technique displays the current level of advancement of knowledge in different fields of science, providing data on the most active researchers and countries and providing an understanding of prior periods and future forecasts of research fields globally (Zakka et al., 2021). Numerous scientists have done bibliometric evaluations

of geopolymers across various academic disciplines. As an illustration, Matsimbe et al. (2023) conducted a bibliographic analysis of geopolymer research in the Sub-Saharan Africa region, utilizing Vos viewer software. The study results indicated that the phrases with the greatest frequency of appearances were geopolymers, inorganic polymers, and compressive strength. Moreover, the Construction and Building Materials magazine was generally recognized as a major academic publication, publishing 41 articles and boasting a substantial citation record of approximately 1488. Cameroon, Nigeria, and South Africa were the states that demonstrated the highest volume of documents.

Gu et al. (2022) carried out a scientometric analysis and research mapping to investigate the current understanding of the application of coconut fibers in concrete. The findings revealed that the leading journal is "Construction and Building Materials," with 21 articles, while India, Malaysia, and New Zealand have made contributions of 41, 28, and 20 articles. Another study was conducted by Amin et al. (2022) on an analysis of scientific publications and knowledge visualization on the topic of steel fiber-reinforced concrete. The analysis revealed that 17 sources, including journals and conferences, have published a minimum of 100 articles on SFRC until June 2022. Alkadhim et al. (2022) conducted a research investigation on a scientometric review to analyze the knowledge mapping of the published research about fiber-reinforced geopolymers. The scientometric study revealed the commonly utilized keywords in FRGC (Fiber Reinforced Geopolymer Composites) research, which include inorganic polymers, geopolymers, reinforcement, geopolymer, and compression strength. Besides, it is worth noting that 27 authors have contributed to the body of literature on FRGC by publishing more than ten papers. Moreover, as of June 2022, a noteworthy 29 articles have garnered over 100 citations, indicating their significant impact within the field.

Despite existing reviews and bibliometric analyses, there is a noticeable absence of bibliometric studies about hybrid fiber-reinforced geopolymer composites. The lack of data relates to keyword searches that are not expansive enough to produce a comprehensive dataset. Therefore, there is a demand for an unbiased and more objective visual representation of the patterns and developments in this particular domain. The current study employed two approaches: a scientometric analysis of the bibliometric data and a normal review of the HFRGC. This research summarizes the studies carried out over the last decade using maps for bibliometric data to assess research development over the last ten years. The conventional review consists of the fresh and mechanical properties of HFRGC. On the other hand, a scientometric analysis sequence for bibliometric data is used in this study to present contemporary and elaborated analysis for previous studies in the area of hybrid fiber-reinforced geopolymer composites. A scientometric analysis is developed to analyze the yearly distribution and growth tendency and number of documents based on countries, keywords, sources, and citations of the HFRGC field, carried out using VOS viewer. The methodology of this review is addressed in Figure 2.

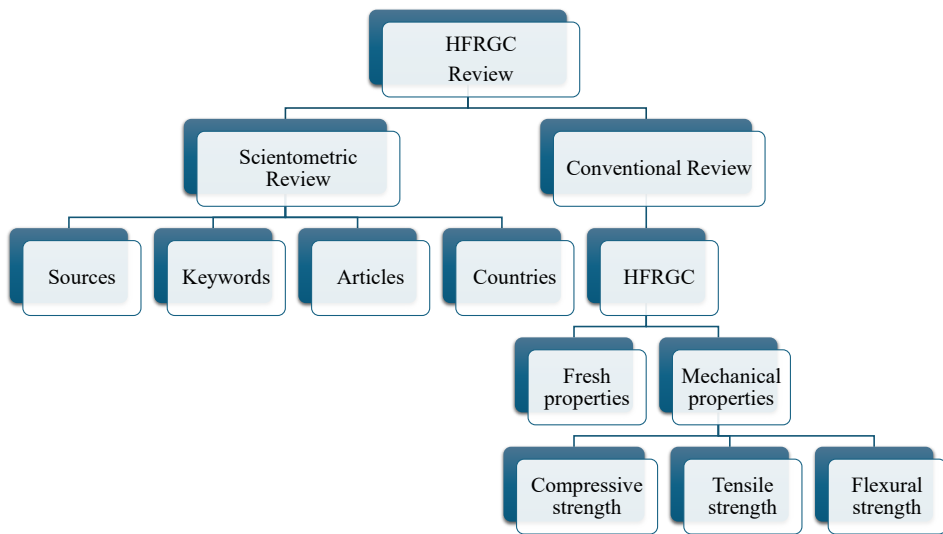


Figure 2. A schematic depicting the scientometric and conventional approaches will be covered in this review

RESEARCH STRATEGY AND DATA SOURCES OF HFRGC

The present study utilized a scientometric strategy-based review of HFRGC to evaluate scientific results and produce bibliometric geographical maps. The chosen methodology is suitable for this review since it methodically examines and evaluates the study's progress over a particular timeframe, utilizing a thorough collection of bibliographic data. (Hosseini et al., 2018). Numerous academic papers were published regarding the subject area, so selecting a dependable search tool is imperative. Scopus and WOS are renowned search platforms for their high accuracy, making them appropriate for this particular objective (Chadegani et al., 2013).

The bibliographical data related to the study on HFRGC was obtained using Scopus, a highly recommended academic resource (Meho, 2019). Nees Jan VanEck and Ludo Waltman developed VOS Viewer, a commonly employed program for data visualization. The system can build visual representations of researchers, journals, papers, and keywords by utilizing citation and co-occurrence data (van Eck & Waltman, 2021). Table 1 presents the criteria for obtaining data from Scopus between 2013 and October 2023.

The analysis focuses on the yearly publication of papers, Science visualization of top publication sources, science visualization of co-occurrence keywords, researchers, top-cited papers, and participating countries. The searched phrases in the Scopus library of "geopolymer" and "hybrid fiber" or "hybrid fiber" had successfully generated 90 from 98 articles as of October 2023 after using filtration by eliminating unnecessary documents. The limitation of the language was stated as the English language. The research focused on engineering and material science to facilitate a more comprehensive study. The information

acquired via the Scopus collection has been saved in the Comma Separated Values (CSV) form to simplify evaluation by employing a suitable computer program. The scientific map and visualization software employed in this work was VOSviewer (version 1.6.17). Figure 3 depicts a schematic diagram that represents the scientometric approach.

Table 1
A filter is used to obtain data from the Scopus database web page

Option	Filter used
Publication date	2013–2023
Language	English
Subject area	Material Science Engineering

SCIENTOMETRIC ANALYSIS RESULTS AND DISCUSSIONS

Subject Area and Annual Publication of Documents

The evaluation was conducted utilizing the Scopus database to determine the relevant research areas. Figure 4 illustrates that "Engineering" and "Materials Science"

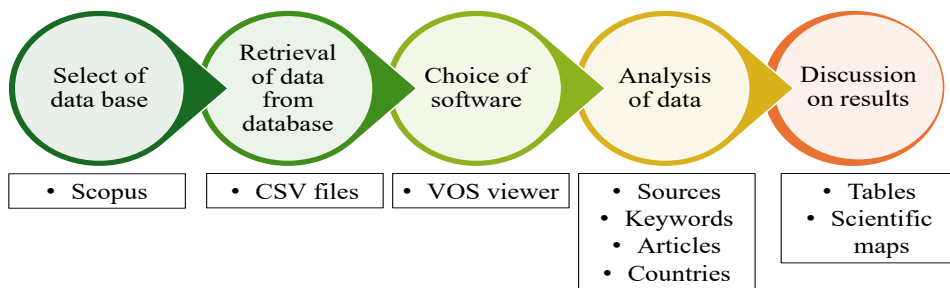


Figure 3. The sequential process of scientometric analysis employed in the present study

were the two subject areas that produced the most documents, with 42.08% and 34.97% of publications, respectively, resulting in a total of 77.05% of paperwork. Moreover, the Scopus search engine was used to search for the sources that included the paperwork in related areas of study (Figure 5). According to this analysis, articles, conference reviews, conference papers, book chapters, and reviews comprised around 58.16%, 18.37%, 15.31%, 4.08%, and 4.08% of the entire information, respectively. Figure 6 illustrates the yearly growth of the documents published in the present field of research between 2003 and October 2023, as the fresh document related to this research area was found in 2003. Based on the research done by Chen et al. (2022), the yearly number of papers can measure the trajectory of advancement within an area of interest. The data demonstrates a fluctuating pattern of studies conducted on HFRGC based on the Scopus document database between 2003 and 2023. There was a significant increase to 17 documents in 2018 from 7 in 2017. However, this trend was interrupted by a decline to 9 documents in

2019, indicating a temporary reduction. Following that, there was a slight increase to 10 documents in 2020. The year 2021 defined a notable upswing, reaching 17 documents, demonstrating a significant increase in academic output. However, this upward trajectory was not sustained, as evidenced by a reduction to 14 documents in 2022, indicating a slight downturn. Nevertheless, academic productivity rebounded in 2023, with a significant rise to 20 documents, representing a peak in research output for the analyzed period. The academic community has demonstrated a notable emphasis on the utilization of HFRGC in construction materials.

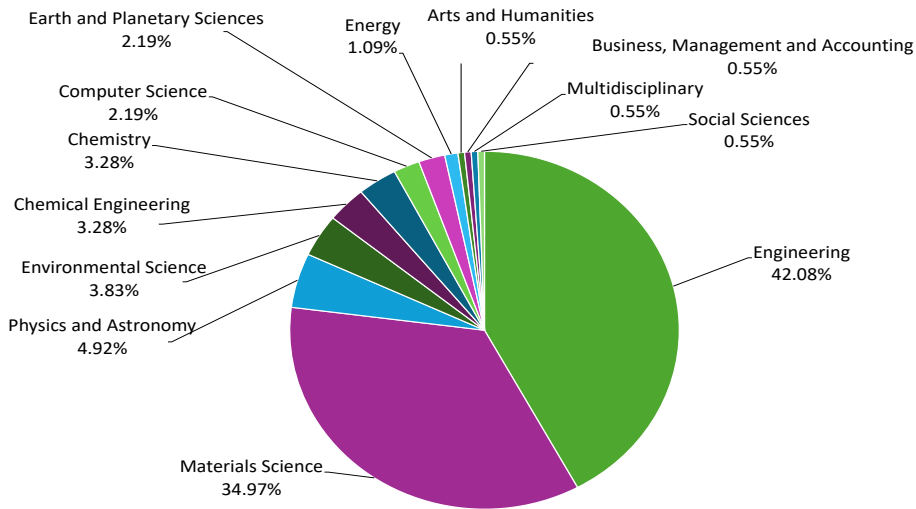


Figure 4. Subject area of documents published on HFRGC (Scopus database)

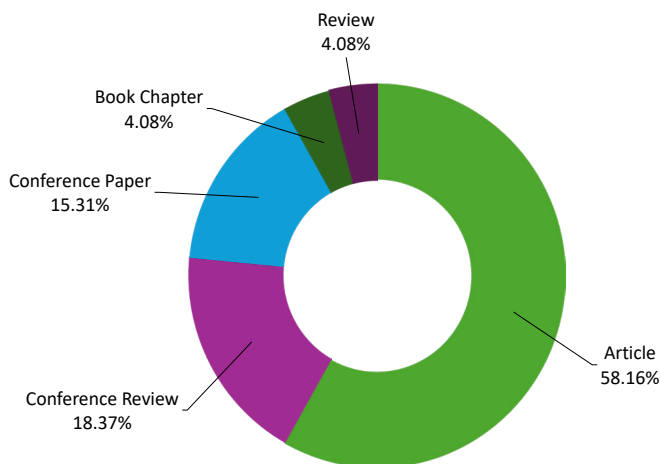


Figure 5. Types of documents released on HFRGC (Scopus database)

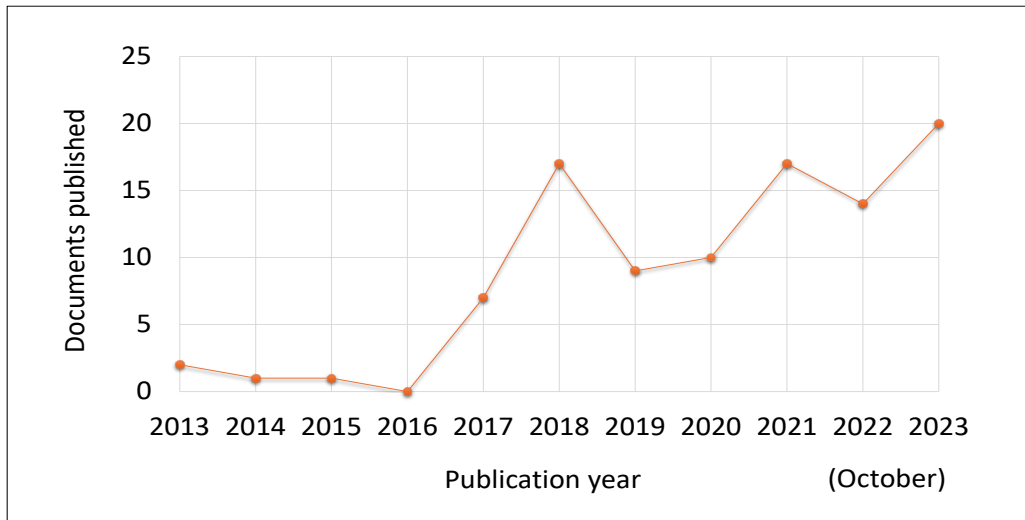


Figure 6. Annual publication of hybrid fiber reinforced geopolymer composites documents (Scopus database)

Top Publication Sources

The application of source mapping techniques promotes the analysis of growth and innovation. These sources provide the ability to access data that has some specified limitations. The research was conducted via the VOSviewer program, which employed bibliometric data from Scopus. The chosen analytical approach for this study was bibliographic coupling, whereas the selected type of analysis was sources. The minimal number of documents necessary from a source has been established to be 1, and all 42 publications fulfilled this requirement. A journal's impact can be evaluated by analyzing its overall link strength, the number of documents it has released, and its citation count, as shown in Table 2. The scientific journals "Construction and Building Materials," "Lecture Notes in Civil Engineering," and "Materials" are the most prolific three publication sources, with 15, 10, and 5 articles, respectively. The academic journals listed, specifically "Construction and Building Materials," "Lecture Notes in Civil Engineering," and "Materials," have been cited 441, 4, and 34, respectively. Furthermore, this investigation would establish a framework for future scientometric questions in HFRGC research. Moreover, prior conventional assessments could not generate visual representations of scientific maps.

The network representation in Figure 7(A) depicts the recently published journals. The journal's influence on the number of documents within the current research area is expressed by the node's dimensions; a larger node signifies a more significant impact. A noteworthy observation is that the category labeled "Construction and Building Materials" exhibited

a bigger node size compared to the other categories, revealing its heightened significance as a source within the field of research. The artwork comprised six interconnected clusters, each represented by a separate color palette: red, green, dark blue, light blue, yellow, and purple. Groups are established according to the scope of study sources or the frequency with which each source is referenced in related academic papers (Wuni et al., 2020).

The degree of connection between the research sources indicates the volume of documents within the current study domain that exhibit co-citations. Moreover, quantifying connection strength offers valuable insights into the frequency with which two academic journals are cited in the same scientific publication. As an illustration, the construction and building materials field exhibited a higher number of citations than other study areas, with a link strength of 23. The relationships among the nodes in a cluster situated close together are more powerful compared to those that are farther distant. For instance, "Construction and Building Materials" is directly linked to the "Journal of Materials in Civil Engineering" rather than "Case Studies in Construction Materials."

The colors in Figure 7(b) corresponded to the densities observed in a scholarly publication. The primary color is red, with yellow, green, and blue denoting the decreasing concentration. The bright red color of "Construction and Building Materials" represented the more significant importance of the current research.

Table 2
Top sources based on the number of documents

No.	Source	Documents	Citations	Total link strength
1	Construction and building materials	15	441	23
2	Lecture Notes in Civil Engineering	10	4	2
3	Materials	5	34	42
4	Journal of Composites Science	3	31	48
5	Polymers	3	14	37
6	Materials Today: Proceedings	3	27	4
7	Cement and Concrete Composites	2	64	20
8	Buildings	2	21	7
9	Ceramics International	2	37	5
10	Composite Structures	2	49	2

Source: Table generated by VOS viewer according to Scopus database

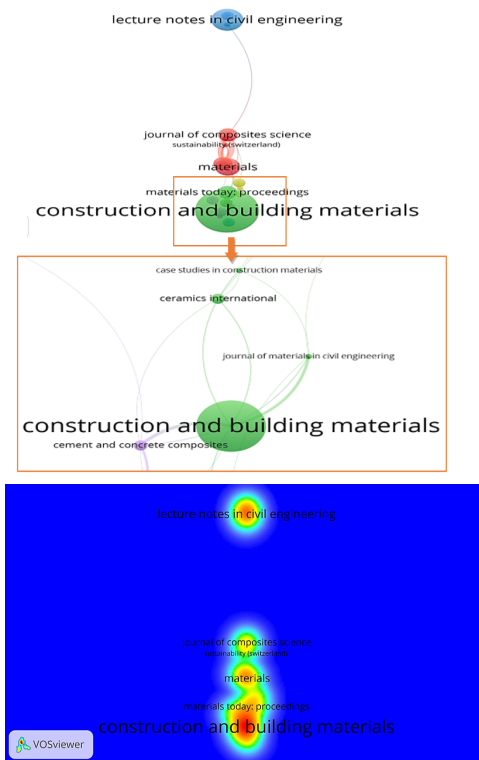


Figure 7. Science mapping of publication sources: (a) Network visualization; (b) Density visualization (Figure generated by VOS viewer according to Scopus database as a result of analysis)

Frequently Used Keywords

Keywords are essential elements of scholarly materials as they serve to recognize and represent the basic topic of the investigation. (Su & Lee, 2010). For evaluation, the method of analysis is "co-occurrence," with the unit of analysis defined as "all keywords." The minimal frequency of a keyword's occurrence was specified as 5, and only 48 of the 638 keywords met the condition. Table 3 shows the top 10 most frequently used keywords in this area. The top five most repeated in the related documents were inorganic polymers, geopolymers, steel fibers, geopolymer concrete, and hybrid fiber, with 51, 51, 35, 34, and 31 occurrences, respectively. The visual depiction of the primary keywords within the research topics is presented in Figure 8(a). The network visualization revealed the presence of four distinct groups: red, green, blue, and yellow. The larger node referred to the most important keywords and repeated more than other node

sizes in the related publication documents. The various colors in Figure 8(b) represent the dense concentration of keywords. The colors red, yellow, green, and blue were arranged in increasing order based on their respective densities, with red having the highest density and blue having the lowest density. For instance, density visualization indicates that inorganic polymers and geopolymers had a higher density. This discovery will assist aspiring authors in choosing keywords that may improve access to previously published work in a specific subject area.

Table 3
Top 10 common keywords in publication articles

No.	Keyword	Occurrences	Total link strength
1	Inorganic polymers	51	537
2	Geopolymers	51	530
3	Steel fibers	35	357

Table 3 (Continue)

No.	Keyword	Occurrences	Total link strength
4	Geopolymer concrete	34	331
5	Hybrid fiber	31	322
6	Compressive strength	31	322
7	Fly ash	28	294
8	Polypropylenes	25	285
9	Geopolymer	25	237
10	Geopolymer composites	23	216

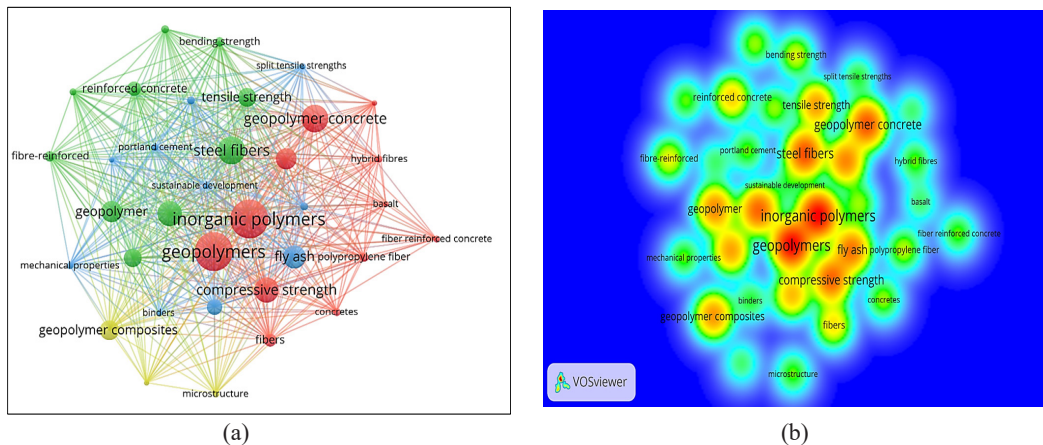


Figure 8. Science mapping of mostly used keywords: (a) Network visualization; (b) Density visualization (Figure generated by VOSviewer according to Scopus database as a result of analysis)

Science Mapping of Published Articles

The total number of citations garnered by a scholarly text provides an indicator of its importance within the field of academic investigation. In this analysis in the VOS viewer, bibliographic coupling was set as the type of analysis, and documents were set as the unit of analysis. Table 4 displays a comprehensive collection of the top 10 papers with the highest citations in the hybrid fiber-reinforced geopolymer composites domain.

Table 4 includes pertinent information, such as the authors' names and essential citation details. The article titled "Tensile behavior and microstructure of hybrid fiber ambient-cured one-part engineered geopolymer composites" by Alrefaei and Dai (2018) received 114 citations, the most cited works in the field. Sukontasukkul et al. (2018) and Guo et al. (2020) have garnered significant attention, with 111 and 92 citations among the top three most cited works. Figure 9(a) illustrates the citation-based map of relevant publications, highlighting the clustering of these works within the scope of the current research topic. The findings of the inquiry revealed that a collective sum of 41 papers were interlinked via citations. In addition, Figure 9(b) illustrates the density mapping, highlighting the concentration of publications with greater citations in red for Alrefaei and Dai (2018).

Table 4
Higher cited documents of hybrid fiber geopolymer composites

No.	Document	Title	citations	Links
1	Alrefaei and Dai (2018)	Tensile behavior and microstructure of hybrid fiber ambient cured one-part engineered geopolymer composites	114	10
2	Sukontasukkul et al. (2018)	Flexural performance and toughness of hybrid steel and polypropylene fiber-reinforced geopolymer	111	1
3	Guo et al. (2020)	Sulfate resistance of hybrid fiber-reinforced metakaolin geopolymer composites	92	0
4	Asrani et al. (2019)	A feasibility of enhancing the impact resistance of hybrid fibrous geopolymer composites: Experiments and modeling	76	4
5	Khan et al. (2019)	Mechanical properties and behavior of high-strength plain and hybrid-fiber reinforced geopolymer composites under dynamic splitting tension	63	12
6	Gao et al. (2017)	Evaluation of hybrid steel fiber reinforcement in high-performance geopolymer composites	54	0
7	Tran et al. (2020)	Effect of hybrid fibers on shear behavior of geopolymer concrete beams reinforced by basalt fiber reinforced polymer (BFRP) bars without stirrups	48	4
8	Khan et al. (2018)	Mechanical properties of ambient cured high-strength plain and hybrid fiber reinforced geopolymer composites from triaxial compressive tests	48	0
9	Su et al. (2019)	Influence of different fibers on properties of thermal insulation composites based on geopolymer blended with glazed hollow bead	43	8
10	Chithambar Ganesh and Muthukannan (2019)	Experimental Study on the Behavior of Hybrid Fiber Reinforced Geopolymer Concrete under Ambient Curing Condition	40	1

Source: Table generated by VOSviewer according to Scopus database as a result of analysis

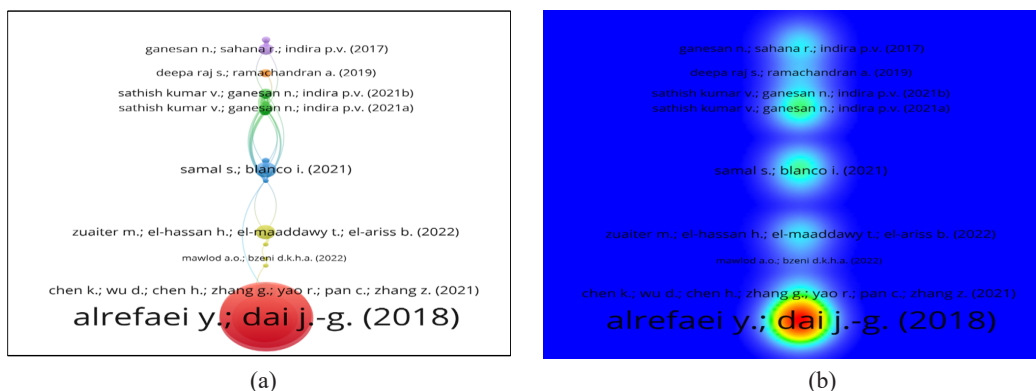


Figure 9. Science mapping for higher citation documents: (a) Network visualization; (b) Density visualization (Figure generated by VOSviewer according to Scopus database as a result of analysis)

Science Mapping of Contributing Countries

Some countries have exhibited greater participation and continued achievements in HFRGC than others. The development of the network visualization was designed to offer readers a graphical depiction of the distinct areas focused on the study domain. The analysis method employed in this study was bibliographic coupling, with the unit of analysis representing countries. The minimal threshold for the number of documents restricted to a certain country was established at 1, and 27 donations met this criterion. The nations listed in Table 5 have produced at least ten publications in the specified topic of study. The countries that contributed the greatest number of papers were India, China, and Australia, with 35, 15, and 12 documents, respectively. Furthermore, it is noteworthy that the three nations that garnered the greatest number of citations were India, China, and Australia, with citation counts of 333, 378, and 266, respectively. The quantification of publications, citations, and total link strength served as an indicator of the extent to which every country has exerted an impact on the development of the subject field.

In Figure 10(a), the visualization presented illustrates the interconnectedness of nations through the medium of citations in the field of science. The size of a node is indicative of a nation's level of contribution to the field of research. The visualization network revealed the presence of four distinct groups of countries, each distinguished by its unique color. According to the data presented in Figure 10(b), countries exhibiting greater involvement were found to have a higher population density. Employing visuals and extensive data records pertaining to all participating nations will considerably facilitate young researchers in cultivating scientific relationships, initiating joint activities, and exchanging novel methodologies and ideas. Scientists from different nations who have an appetite for improving the study of HFRGC have the chance to collaborate with established experts in this field. Through these collaborations, researchers can gain important insights and benefit from these experts' deep understanding and expertise.

Table 5
 Top 10 countries active in the related area

No.	Country	Documents	Citations	Total link strength
1	India	35	333	1118
2	China	15	378	501
3	Australia	12	266	885
4	Croatia	5	52	571
5	Pakistan	3	117	537
6	Iraq	3	20	89
7	United Arab Emirates	3	31	20
8	Saudi Arabia	2	12	619
9	Russian Federation	2	15	341
10	Poland	2	17	330

Source: Table generated by VOSviewer according to Scopus database as a result of analysis

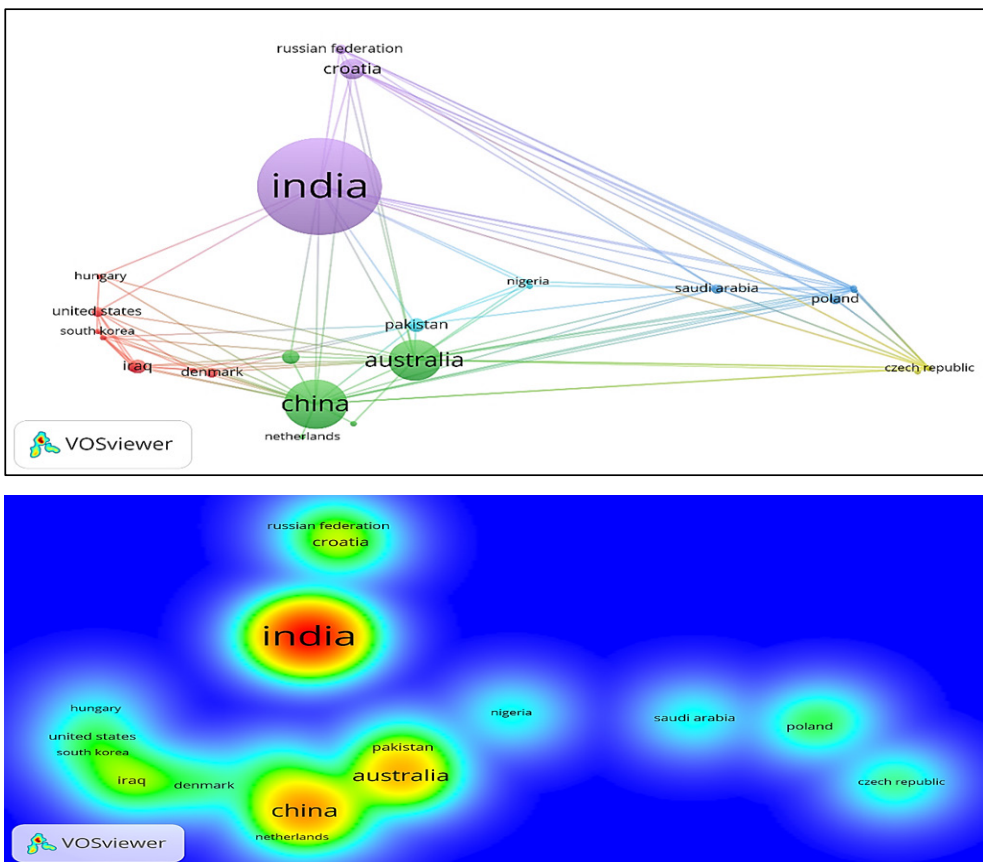


Figure 10. Science mapping of most active countries in documents and citation of related area documents: (a) Network visualization; (b) Density visualization (Figure generated by VOSviewer according to Scopus database as a result of analysis)

HYBRID FIBER REINFORCED GEOPOLYMER COMPOSITES (HFRGC)

The hybrid fiber technique involves mixing two or more sorts of fiber in a fiber-reinforced concrete mix to improve the characteristics of the concrete mix, reduce cracks, and enhance the performance of the concrete due to the superior properties of one type of fiber supporting the obstacles of the other kind (Vairagade & Dhale, 2023).

Figure 11 illustrates the function of short and long fibers in the hybridization system. Hybridization techniques rely on the length of the fiber; a short fiber can control microcracks at the fresh stages of loading and prevent their propagation, while another type is longer to offer a cross-macro crack bridging procedure. The hybridization technique depends on the interaction of the fiber elements, where one fiber is stronger than the other, which is more flexible. In addition, the behavior of fibers provides a vital role, where one type of fiber provides strength in the concrete while another type provides new mixing properties.

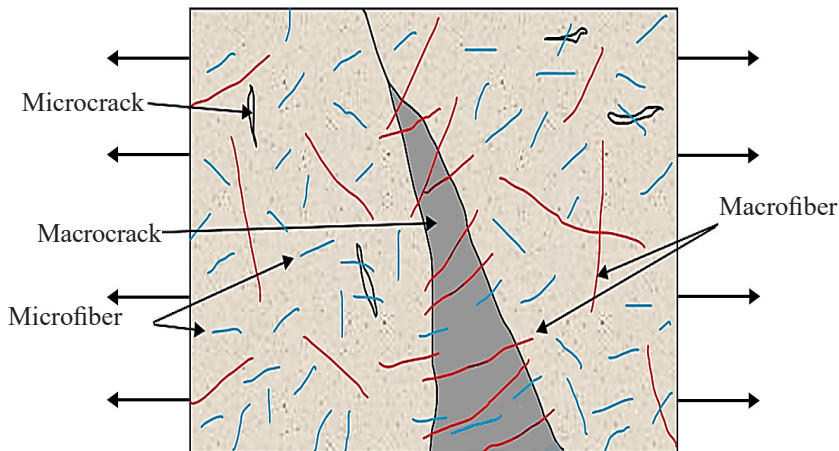


Figure 11. The distribution of short and long fibers across cracks in concrete

Fresh Properties of HFRGC

Chithambar Ganesh and Muthukannan (2019) reported that the inclusion of hybrid fiber, glass fiber (GF), and polypropylene fibers (PPF) in the GPC mixture dropped the workability of the mixture, and the fiber's type and volume fraction are factors that affect workability properties. The results showed that the mixtures prepared with a higher amount of GF had a higher compaction factor. This effect was due to the overall quantity of GF material, which was less in volume than PPF for the chosen fiber weight. Hence, the mixtures prepared with GF contain a higher amount of fiber than other mixtures. Similarly, Guler and Akbulut (2022) observed that geopolymer mixture prepared with hybrid fiber (glass and basalt fibers) showed 32% lower workability than the control GPC mixture owing to

the hydrophilic nature of basalt fiber (BF) and GF, which tend to absorb water mix causes a reduction in fluidity (Preda et al., 2021). Besides, the mixtures with a higher percentage of BF showed lower workability than other hybrid mixes because BF has a rougher and more filamentous surface GF.

Humur and Çevik (2022) also found that the workability of HFRGC with hybrid fibers polyvinyl alcohol (PVA) and polypropylene (PPF) was affected by the fiber type, and mixing with higher PVA content showed a lower relative slump because PVA fibers exhibited a greater capacity for water absorption compared to PPF fibers, causing higher water absorption in the mixture and reducing the flowability. Cheng et al. (2022) also indicated that the flowability of HFRGC with hooked-end steel fiber and PVA decreased by 29.4% with an increase in the content of hybrid fiber. Alrefaei and Dai (2018) investigated the impact of varying proportions of hybrid fiber on geopolymer composite, including steel fiber (SF) and polyethylene fiber (PE) as hybrid composites. The mix with 1.5% SF–0.5% PE showed better workability than other hybrid fiber geopolymer composite percentages. This trend can be ascribed to the augmented volume concentration of SF. The higher content of PE in the matrix increased the air trapped and then adversely impacted workability.

Zhang et al. (2021) indicated that integrating hybrid fiber with GPC had no impact on the slump of fresh concrete compared to a single fiber. In addition, steel fiber was more effective in workability than polyvinyl alcohol (PVA), polypropylene fiber (PPF), and recycled polypropylene fiber (RPF). However, the other fiber types in the mix had longer fibers than steel fiber. It is attributed to the varying density and length of fibers that substantially influence the slump test.

Another study was conducted by Gao et al. (2017) on the impact of two distinct lengths of SF of 13 mm (long fiber) and 6 mm (short fiber) in HFRGC with different percentages with a total fiber volume of 1% on the workability of HFRGC. The findings indicated that long SF influenced workability properties more than short ones, with a reduction from 259 mm to 206 mm and 231 mm for long and short steel fiber, respectively. This effect can occur due to the internal cohesive force of the mixture playing an important role in its workability properties.

Therefore, the previous study above showed that HFRGC showed a reduction in workability properties compared with the plan geopolymer mix. This reduction is affected by different factors like fiber type, length, content, and volume fraction of fiber (Table 6). Figure 12 summarizes the optimum hybrid workability percentage reduction vs. fiber volume fraction. It can be inferred that increasing the proportion of hybrid fibers leads to a greater decrease in workability.

Table 6
Summary of the workability findings of the HFRGC with the optimum mixes

Reference	Binder	Optimum hybrid fibers	Optimum total content (Vol.%)	Workability test	Control mix (mm)	Findings
Chithambar Ganesh and Muthukannan (2019)	GGBS	0.75GF-0.25PPF	1%	Compaction factor test	-	Higher by 5.3% compared to PPF and lower by 1.23% compared to GF
Guler and Akbulut (2022)	FA	24 mm GF-12mm GF	0.5%	Flowability	170	Lower by 13.3% compared to the control mix
(Humur and Çevik (2022)	FA GGBS	PPF-PVA	2%	Relative Slump	2.75	Lower by 81% compared to control
Zhang et al. (2021)	FA GGBS	SF-PVA SF-PPF SF-RPP	1%	Slump	275	lower by 37.5% compared to control
Cheng et al. (2022)	FA GGBS	0.5%SF-2%PVA	2.5%	Flowability	280	Higher performance than other percentages and lower by 24.4% compared with the control sample
Alrefaei and Dai (2018)	FA GGBS	1.5%SF-0.5%PE	2%	Slump	-	Showed better performance than other hybrid fiber percentages.
Gao et al. (2017); Yu et al. (2014)	FA GGBS	13mm SF-6mm SF 0.5%SF-1.5%SF	2%	Flowability	-	It has a better effect than other hybrid percentages and is higher by 4.2% compared with 2% long steel fiber.
Heweidak et al. (2022)	FA GGBS	12mm BF-30mm BF 0.25BF-0.65BF	1%	Flowability	750	Lower by 5.6% compared to control

*FA: Fly ash, GGBS: Ground granulated blast furnace slag, GF: Glass fiber, PPF: polypropylene fibers, PVA: polyvinyl alcohol, SF: Steel fiber, RPP: Recycled polypropylene fiber, PE: Polyethylene fiber, BF: Basalt fiber

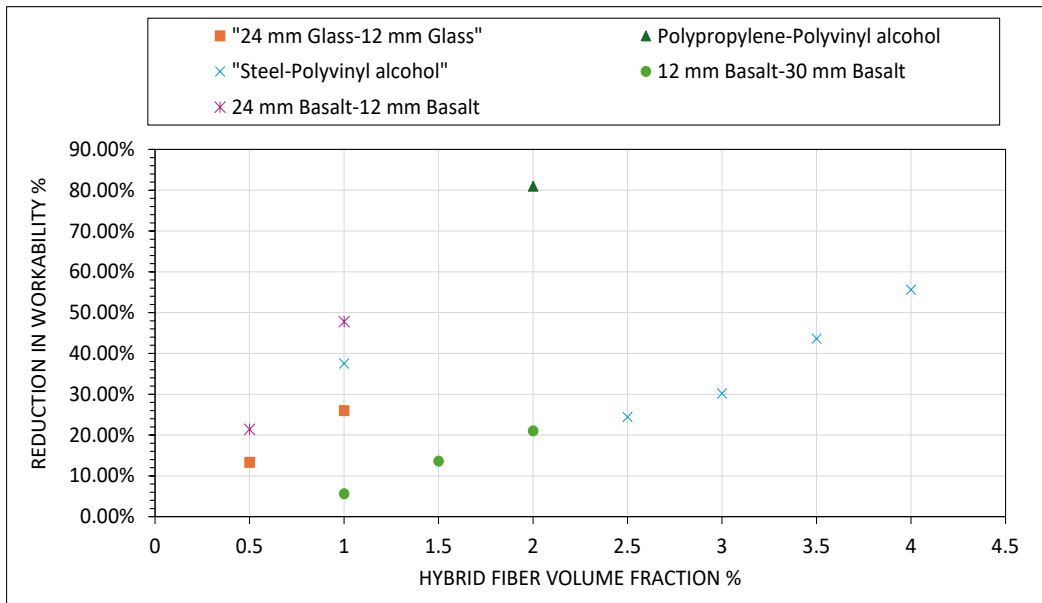


Figure 12. Summary of the reduction in workability versus. hybrid fiber volume fraction (Alrefaei & Dai, 2018; Cheng et al., 2022; Gao et al., 2017; Guler & Akbulut, 2022; Heweidak et al., 2022; Humur & Çevik, 2022; Zhang et al., 2021)

Mechanical Properties of HFRGC

Compressive Strength of HFRGC

The combination of different types of fibers in the GPC matrix has a significant impact on compressive strength. The impact of this phenomenon differs based on several criteria, such as the kind, composition, volume percentage, and length of the fiber (Sathish Kumar et al., 2021) evaluated the positive effects of HFRGC prepared with SF and PPF with different percentages. The findings indicated that the composite prepared with 1% SF–1% PPF provided a compressive strength of around 66.93 MPa, which is higher than other hybrid mix percentages and higher than the control sample by 17%. This effect may be ascribed to the hybrid fibers' capability to support each other and effectively absorb and transmit the loads from the matrix to the fiber.

Bakthavatchalam and Rajendran (2021) also found that HFRGC with 1.5% SF–0.5% BF showed a compressive strength of 62.4% higher than the strength of the control. Gao et al. (2017) researched the influence of single and hybrid long and short steel fibers (SF) in geopolymer composite on compressive strength. The SF had 6 and 13-mm lengths and were used in varying percentages ranging from 0.25 to 1% for single short and long steel fibers. A total volume of 1% was used for the hybrid fiber composite. The outcomes showed that HFRGC, with a 60/40 long/short SF ratio, attained the greatest compressive strength at around 98 MPa. The control mix had a compressive strength of 81 MPa, whereas the

composites that included single fibers were around 86–90 MPa and 90–97 MPa for short and long fibers, respectively.

Similarly, Zhang et al. (2021) noticed that the inclusion of single PVA, PPF, and recycled PPF in GPC with a total volume of 1% showed a reduction in compressive strength around 4.3%, 18.8%, and 7.1%, respectively, except SF showed improvement around 4% compared with plain geopolymer. However, the incorporation of hybrid fiber in the mixture demonstrated better compressive strength than single fiber. This effect was attributed to the better adhesion of hybrid fiber than single fiber in a geopolymer mix, and the load will transfer from one type of fiber to the other type, resulting in a matrix with higher compressive strength. Guler and Akbulut (2022) also observed that HFRGC demonstrated superior compressive strength compared to a single fiber after exposure to elevated temperatures (500–800°C).

Sukontasukkul et al. (2018) examined the effect of single and hybrid SF and PPF in geopolymer composite with different percentages of 0.5% and 1% for every single fiber and 0.8 PPF–0.2 SF, 0.6 PPF–0.4 SF, 0.4 PPF–0.6 SF, and 0.2 PPF–0.8 SF for hybridization percentages. The findings indicated that the strength of HFRGC was higher than that of the control geopolymer and the composites with a single fiber. Besides, the compressive strength of HFRGC increased with the increasing replacement percentages of SF, and the composites of 0.2 PPF–0.8 SF had the highest strength of around 56.8 MPa. This effect proved that SF prevented cracks and produced bonding forces better than PPF in the hybridization system.

Mousavinejad and Sammak (2021) made similar observations, and the authors attributed this to the tensile strength of PPF (400 MPa), which was lower than the tensile strength of SF (2000 MPa). Baziak et al. (2021) also found that augmenting the proportion of SF to carbon fiber (CF) in the HFRGC enhanced compressive strength. The authors ascribed this phenomenon to the elevated modulus elasticity of SF to CF, which provided better internal bonding in the matrix.

Bakthavatchalam and Rajendran (2021) investigated the impact of varying SF and BF concentrations mixed with geopolymer concrete in different percentages with a total volume fraction of 2%. The findings indicated that HFRGC of 1.5% SF–0.5% BF showed a compressive strength of around 49.2 MPa, 62.4% higher than the plain sample.

In another study, Chithambar Ganesh and Muthukannan (2019) examined the effect of hybrid fiber, GF, and PPF in geopolymer composite with different percentages on the compressive strength of the matrix. The outcomes unveiled that HFRGC prepared with higher percentages of GF showed higher compressive strength compared to PPF, and HFRGC prepared with 0.75% GF–0.25% PPF achieved a strength of 58.2 MPa. This effect was due to insufficient cohesive strength between the PPF and the geopolymer mixture. Zuaiteer et al. (2022) analyzed the effect of the fiber's length on the compressive strength

of HFRGC by using GF of 24 mm (short fiber) and 43 mm (long fiber) with a total volume content of 1%. The findings indicated that the combination of 0.75% long glass fibers and 0.25% short glass fibers in the HFRGC resulted in the maximum compressive strength compared to other ratios, reaching a value of 42 MPa. The result can be ascribed to the elevated proportion of long glass fiber showing better control to macrocracks than short fiber by forming bridges that cause better bonding strength in the matrix. Similarly, findings published by Heweidak et al. (2022) showed that the authors used different lengths of BF to prepare HFRGC.

In short, HFRGC exhibited compressive strength that was 19% to nearly 41% higher than the control specimen. Besides that, according to the previous studies above, the optimum hybrid percentages of reinforced geopolymer composite depend on fiber type, volume, and length. All the studies above showed that incorporating hybrid fiber in the geopolymer matrix could provide better results than using single fiber alone because loads can transfer from one type to another. Each fiber type can support the other, increasing the internal bonding strength and leading to superior properties of the geopolymer composite (Figure 13).

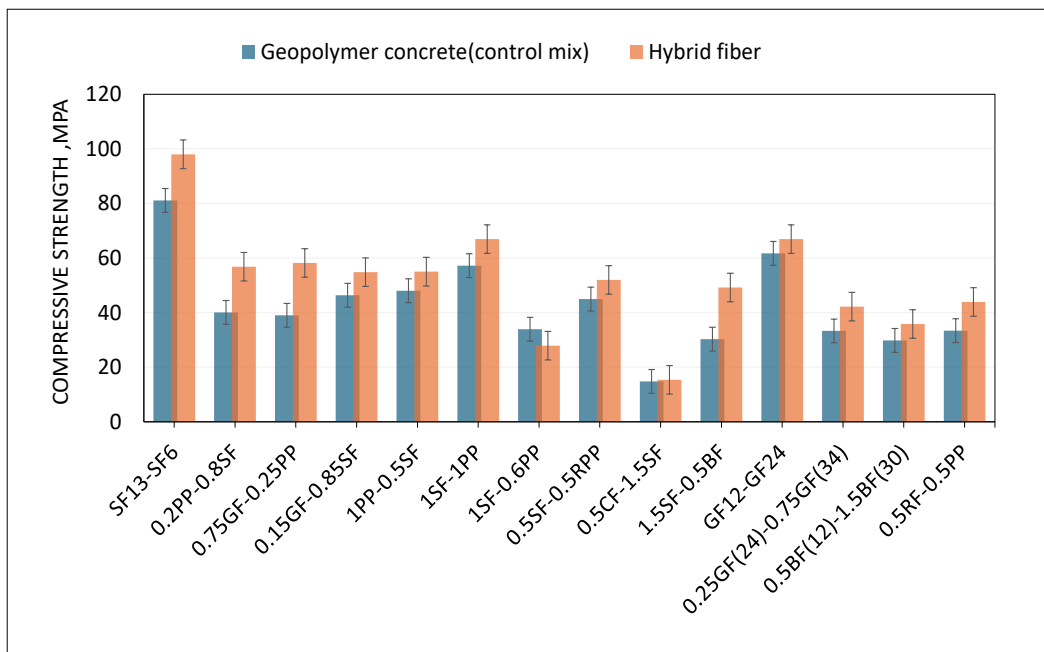


Figure 13. Compressive strength for the control and optimum mix of hybrid fiber (Aisheh et al., 2022; Arunkumar et al., 2022; Bakthavatchalam & Rajendran, 2021; Baziak et al., 2021; Chen et al., 2021b; Chithambar Ganesh & Muthukannan, 2019; Gao et al., 2017; Guler & Akbulut, 2022; Heweidak et al., 2022; Junior et al., 2021; Mousavinejad & Sammak, 2021; Sathish Kumar et al., 2021; Sukontasukkul et al., 2018; Zhang et al., 2021; Zuaiteer et al., 2022)

Splitting Tensile Strength of HFRGC

Daniel et al. (2017) examined the tensile strength of HFRGC prepared with SF and GF with a total fiber content of 1%. Different percentages of SF and GF were used (90% SF–10% GF, 80% SF–20% GF, 70% SF–30% GF and 60% SF–40% GF). The results showed that HFRGC prepared with 90% SF–10% GF attained the maximum tensile strength of around 8.9 MPa, surpassing other mixtures by 12.5–29.8%. It proves that the higher content of SF in the HFRGC provided better results owing to the higher tensile strength of SF (1100 MPa) compared to GF (600 MPa).

Chithambar Ganesh and Muthukannan (2019) analyzed the efficacy of both single and hybrid fiber geopolymers, incorporating GF and PPF, by altering the fiber percentages at a volume fraction of 1%. The findings demonstrated that the geopolymer's tensile strength was 57.1% greater when reinforced with GF compared to when reinforced with PPF. Meanwhile, the tensile strength of optimum percentages of hybrid fiber (0.75% GF–0.25% PPF) was around 5.16 MPa. This influence was due to the high modulus elasticity of GF compared to PPF, which led to a higher amount of GF being incorporated in the hybrid composite, providing better results than other mixes. Hence, hybrid fiber achieved efficiency better than using single fiber due to the single fiber's tendency to reduce the composite stiffness and integrity, leading to the earlier reaching of the ultimate stress. Sathish Kumar et al. (2021) also examined the tensile strength performance of geopolymer composites reinforced with single SF or PPF and HFRGC containing both SF and PPF with different ratios. The findings indicated that the tensile strength of the composites prepared using single fibers was 28% greater than that of the plain geopolymer. Conversely, the HFRGC, with a 1% SF and 0.25% PPF composition, exhibited a tensile strength of 39% more than the plain geopolymer.

Similarly, Zhang et al. (2021) noted that the HFRGC exhibited a significant enhancement of approximately 48.9% in its tensile strength in comparison to the control sample. Similarly, Aisheh et al. (2022) revealed that the mixes with 1% SF–0.25% PPF, 1.5% SF–0.25% PPF, and 2% SF–0.25% PPF achieved tensile strengths of about 7.4 MPa, 7.3 MPa, and 8.4 MPa, respectively. This phenomenon can be ascribed to the impact of bridging on fractures. Kumar et al. (2019) also stated that the tensile strength of HFRGC prepared with SF and GF increased with increasing the amount of the SF, and the mix of 85% SF–15% GF showed the highest strength (4.52 MPa), which was 53% higher than plain geopolymer due to the SF's higher tensile strength than GF (2670 MPa).

Bakthavatchalam and Rajendran (2021) worked on the performance of HFRGC by including SF and BF with different percentages and a total volume fraction of 2%. The findings showed that the 25% SF–75% BF tensile strength was around 6.92 MPa, which was higher than the control (4.12 MPa) by 67.96%. In another study, Heweidak et al. (2022) analyzed the impact of the fiber percentage on the tensile strength of HFRGC by

adding BF with two different lengths (30 and 12 mm) and different ratios of 1% to 2%. The findings indicated that HFRGC showed 61.96% higher tensile strength than the control, and the tensile strength of HFRGC increased with increasing fiber content. In addition, the HFRGC manufactured using a hybrid length of BF exhibited superior performance in terms of indirect tensile strength in comparison to the single-length BF with the same content.

Tensile strength increased with increasing strength, modulus of elasticity, and fiber stiffness in the geopolymer matrix. It resulted in higher interfacial bonding due to the bridge formation through which stresses were transferred from one type of fiber to another. Hence, the compound performed better than the plain sample and single fiber against the applied loads. Based on previous investigations, the tensile strength of hybrid fiber-reinforced geopolymer composite increased from 39% to almost 68% compared to plain geopolymer. Hence, hybrid fiber effectively converted geopolymer from brittle nature to ductile. Figure 14 illustrates the summary of the tensile strength of the HFRGC as compared to single fibers. “Single Fiber 1” and “Single Fiber 2” denote the different types of single fiber.

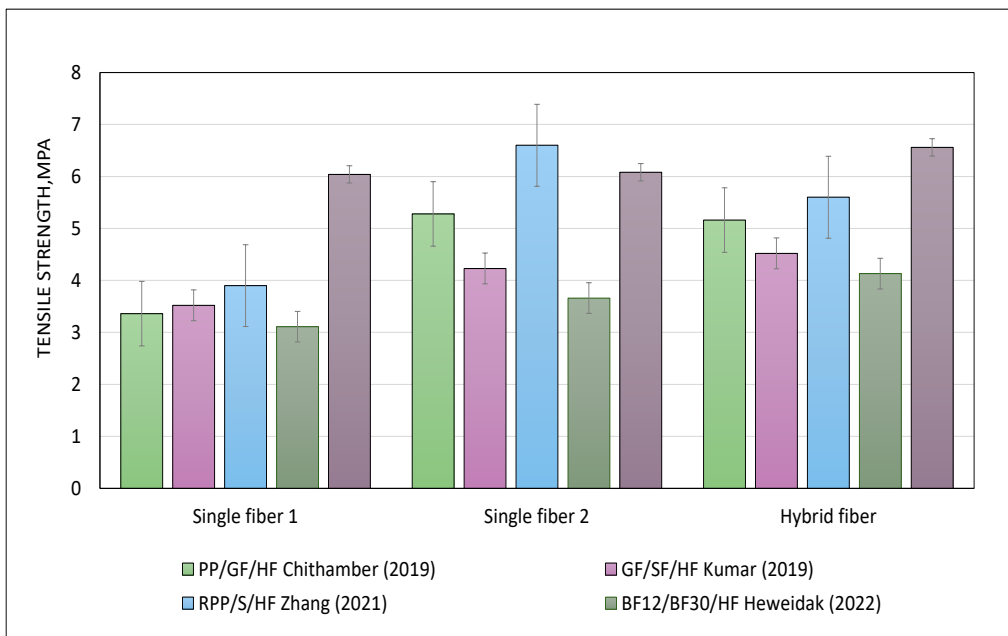


Figure 14. Summary of tensile strength of HFRGC (Bakthavatchalam & Rajendran, 2021; Chithambar Ganesh & Muthukannan, 2019; Daniel et al., 2017; Heweidak et al., 2022; Kumar et al., 2019; Sathish Kumar et al., 2021a; Zhang et al., 2021)

Flexural Strength and Toughness Index

Chithambar Ganesh and Muthukannan (2019) studied the flexural strength performance of single and HFRGC prepared with GF and PPF. The results showed that HFRGC of

0.75% GF–0.25% PPF achieved 56.7% higher flexural strength than composites prepared with single fibers. Similarly, Kumar et al. (2019) reported that HFRGC prepared with SF and GF achieved 53% higher flexural strength than the plain geopolymer. The observed result can be ascribed to the efficacy of fibers in creating bridges within the geopolymer composite, enhancing the composite's characteristics. Similar results were noted for the flexural strength, according to Zhang et al. (2021), where HFRGC showed 60% higher flexural strength compared with the control sample. Bakthavatchalam and Rajendran (2021) also observed that the flexural strength of HFRGC prepared with SF and BF of 25% SF–75% achieved 68% higher flexural strength than the control mix. This improvement was attributed to the combination of macro fiber (SF) and microfiber (BF). These hybrid fibers successfully increased flexural strength by preventing the transmission of micro and macro-cracks at various stages of loading.

Asrani et al. (2019) studied the flexural strength of two different composites of HFRGC, the first one prepared with the combination of two fibers with a total volume fraction of 1.6% (1.3% SF–0.3% GF), (1.3% SF–0.3% PF), and (0.3% PF–0.3% GF), and the second one was composed of a mix of three fibers, with a fiber content of 1.6% (1.0% SF–0.3% PF–0.3% GF). The findings indicated that the utmost increase in flexural strength was about 242.2% in HFRGC, which contained 1.3% SF–0.3% GF, compared to the control. This effect is due to the efficiency of the control cracks in the cracking area because of the presence of SF and GF, which control micro-cracking and macro-cracks in the composite. Chen et al. (2021a) discovered that the inclusion of nanoparticles has no impact on the flexural strength of a material, but it does affect its compressive performance. However, when nanomaterials are mixed with hybrid fibers, the mechanical strengths of the material can be significantly improved. The HFRGC prepared with hybrid fiber and nano-silica achieved a 200% higher flexural toughness than plain geopolymer. It could be attributed to the higher strength of steel fiber (1650 MPa) than polypropylene fiber (900 MPa), which led to better performance against cracks in the geopolymer composite, and the presence of nano-silica, which enhanced the pore enlargement due to its gel.

Junior et al. (2021a) analyzed the efficacy of the incorporation of single and hybrid fibers within geopolymer composite with different hybrid types (SF, PVA, alkali resistance glass (ARG), and PPF) and percentages. The results demonstrated that the flexural strength exhibited a notable enhancement upon the integration of hybrid fiber into the geopolymer matrix. By merging 0.5 SF with 1 PPF, the flexural strength increased by around 37.8% compared to the control sample, and toughness increased as well. Moreover, the hybridization with SF enhanced the residual strength factors for PVA and ARG single fiber by 78% and 82%, respectively. This effect was due to SF's higher modulus of elasticity than PPF, which effectively prevented macrocracking diffusion by providing better crack control and bonding strength in the composite (Figure 15).

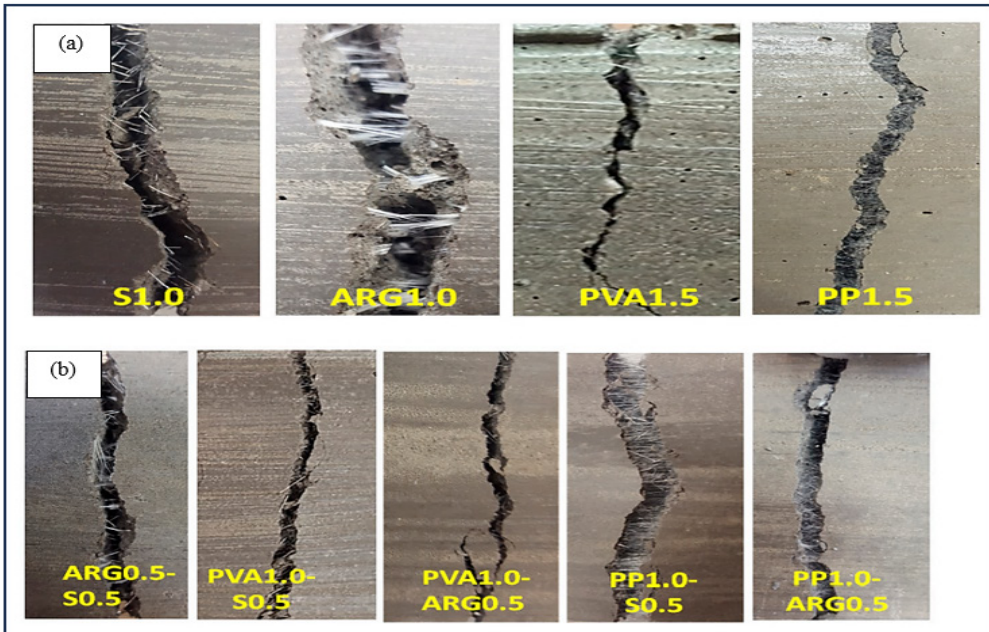


Figure 15. Failure patterns in flexural strength for (a) single fiber and (b) hybrid fiber (Junior et al., 2021a)

Sathish Kumar et al. (2021) examined single and hybrid fibers in different percentages of SF and PPF within a geopolymer composite. The flexural strength of hybrid values for the optimum hybridization percentages of 1% SF–0.25% PPF recorded a flexural strength of 40% higher than the control sample. This effect is attributed to the effectiveness of PPF in the early stages of controlling microcracks, and with increasing the load, SF acted to arrest the expansion of macrocracks, thus improving the bending strength. Similar observations were made by Mousavinejad and Sammak (2021) and Aisheh et al. (2022) when the authors used HFRGC with SF and PPF. The presence of fiber was responsible for this impact in different lengths and percentages, providing better bonding force in micro and macro cracks, which positively impacted flexural strength.

In short, the inclusion of both single and hybrid fibers had an important influence on the flexural strength of the geopolymer compound. Flexural strength was increased using the optimum hybridization value among fibers and the properties of incorporated values like modulus of elasticity, length, and volume. According to the single and HFRGC, the flexural strength increased from 20% to almost 68% compared to plain geopolymer without fiber. Hybrid fiber effectively acted as a crack arrester. Figure 16 illustrates a summary of the flexural strength of the HFRGC as compared to single fibers. “Single Fiber 1” and “Single Fiber 2” denote the different types of single fiber. Different HFRGC study summaries are listed in Table 7.

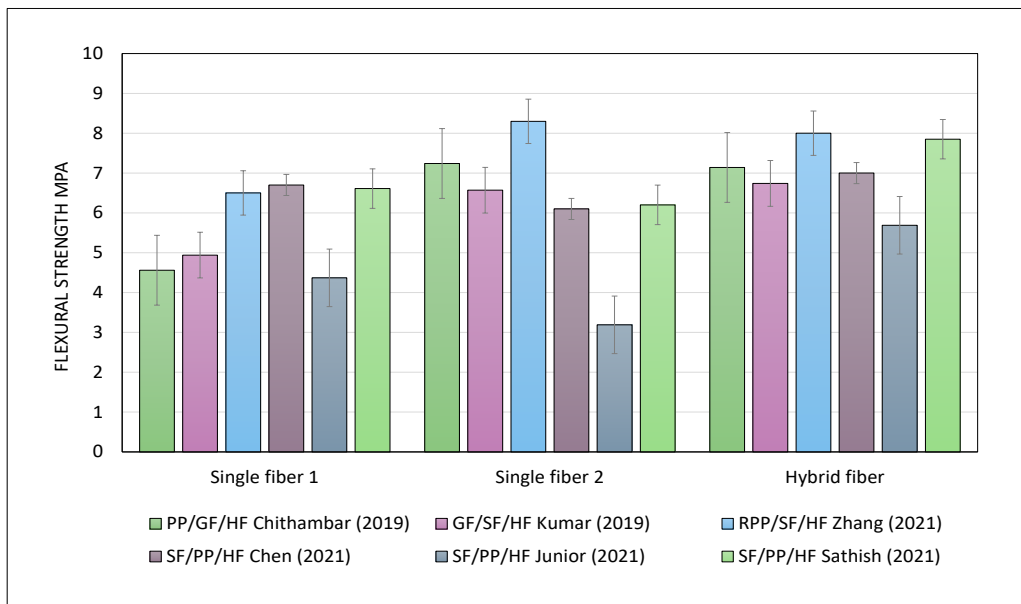


Figure 16. Summary of flexural strength of HFRGC (Chithambar Ganesh & Muthukannan, 2019; Junior et al., 2021a; Chen et al., 2021a; Kumar et al., 2019; Sathish Kumar et al., 2021; Zhang et al., 2021)

Table 7

Summary of different hybrid fibers findings of the HFRGC with the optimum mixes

Author	Source material*	Hybrid fiber % *	Optimum hybrid mix	Findings
Daniel et al. (2017)	GGBS	90 SF-10 GF 80 SF-20 GF 70 SF-30 GF 60 SF-40 GF	90 SF-10 GF	Significant enhancements were observed in FS, such as stiffness degradation, cumulative energy dissipation capacity, ductility, and ultimate load.
Sukontasukkul et al. (2018)	FA Silica fume	0.8 PP-0.2 SF 0.6 PP-0.4 SF 0.4 PP-0.6 SF 0.2 PP-0.8 SF	0.2 PP -0.8 SF	The incorporation of hybrid fibers has demonstrated favorable impacts on the characteristics of PFRG.
Alrefaei and Dai (2018)	FA GGBS	1.5 SF-0.5 PE 1 SF-1 PE 0.5 SF-1.5 PE	0.5 SF-1.5 PE	Better T.S compared to the control mix

Table 7 (Continue)

Author	Source material*	Hybrid fiber % *	Optimum hybrid mix	Findings
Chithambar Ganesh and Muthukannan (2019)	GGBS	0 GF-1 PP 0.25 GF-0.75 PP 0.5 GF-0.5 PP 0.75 GF-0.25 PP 1 GF-0 PP	0.75 GF-0.25 PP	The optimum hybrid fiber percentages increase ductility compared with other mixes.
Kumar et al. (2019)	FA	0.15 GF-0.85 SF 0.3 GF-0.7 SF 0.5 GF-0.5 SF	0.15 GF-0.85 SF	Enhancement in the attained C.S, T.S and F.S in comparison to control
Guo et al. (2020)	MK	(1-3) PP-1PVA (1-3) PP-2PVA (1-3) PVA-5WS (1-3) PVA-10WS (1-3) PVA-15WS	1 PP-1 PVA 2 PVA+15 WS	The optimum mixture exhibited superior C.S and F.S when compared to the control mixture.
Chen et al. (2021a)	FA NS	1 SF-0 PP 0 SF-6 PP 1 SF-6 PP	1 SF-6 PP	Hybrid fibers resulted in composites with superior internal properties and strong fiber/matrix interfacial bonds. FS increased significantly compared to the control mix.
Zhang et al. (2021)	FA GGBS	0.5 SF-0.5 PVA 0.5 SF-0.5 PP 0.5 SF-0.5 RPP	0.5 SF-0.5 RPP	Hybrid fibers effectively improve C.S, T.S and F.S. Enhanced the resilience of GPC and transformed the mode of failure from brittle to ductile.
Baziak et al. (2021)	FA slag	1.5 CF-0.5 SF 1 CF-1 SF 0.5 CF-1.5 SF	1.5 wt. CF-0.5 wt. SFs	The addition of fiber increased C.S and F.S.
Zhong and Zhang (2022)	FA GGBS	1.75 PVA-0.25 RTP 1.5 PVA-0.5 RTP 1.25 PVA -0.75 RTP 1 PVA-1 RTP	1.75 PVA-0.25 RTP	Hybrid fibers improved dynamic C.S and T.S properties while maintaining acceptable quasi-static mechanical characteristics.

Table 7 (Continue)

Author	Source materials*	Hybrid fiber %*	Optimum hybrid mix	Findings
Cheng et al. (2022)	FA	2 PVA-0.5 SF	2.5 PVA-1 SF	Improvement of TS, ductility, and ability to control cracks.
	GGBS	2 PVA-1 SF		
	Metakaolin	2 PVA-1.5 SF		
	Silica Fume	2.5 PVA-0.5 SF		
		2.5 PVA-1 SF		
Guler and Akbulut (2022)	FA	GL24GL12-0.5	GL24GL12-1	Hybrid fibers enhanced the workability of GPC and exhibited superior residual CS and FS characteristics in comparison to single fibers.
		GL24GL12-1		
		BA24BA12-0.5		
		BA24BA12-1		
Lin et al. (2023)	FA GGBS	0 PE-1 PP	0.5 PE-0.5 PP	The hybrid blend provides superior TS as well as great overall economic performance.
		0.25 PE-0.75 PP		
		0.5 PE-0.5 PP		
		0.75 PE-0.25 PP		
		1 PE-0 PP		

* FA: Fly ash, GGBS: Ground granulated blast furnace slag, NS: Nano silica, SF: Steel fiber, GF: Glass fiber, PP: Polypropylene fiber, PE: Polyethylene fiber, RPP: recycled polypropylene, CF: carbon fiber, PVA: polyvinyl alcohol, RTP: recycled tire polymer, WS: wollastonite fiber, MK: Metakaolin. CS: Compressive strength, TS: Tensile strength, FS: Flexural strength

CONCLUSION

This review summarizes previous studies using scientometric analysis and normal review analysis of the available literature on HFRGC to evaluate several metrics. A systematic search was performed in the Scopus database, yielding a total of 90 publications from 2013 to 2023 that were deemed relevant to the research topic. The data obtained from the search was subsequently evaluated using the VOSviewer program. According to the results, several conclusions can be derived:

- Scientometric analysis of data extracted from Scopus data by using a VOS viewer was performed. The top documents, keywords, citations, and countries were mentioned. Bibliometric analysis using scientific mapping provided a better view in detail of the researcher's search data in the same area related to hybrid fiber-reinforced geopolymer composite. Most of the citations were for "construction and building materials," with around 15 documents and 441 citations in the related field. Inorganic polymers, geopolymers, steel fibers, geopolymer concrete, and hybrid fiber were the most frequent keywords, with 51, 51, 35, 34, and 31 occurrences,

respectively. Moreover, the article titled "Tensile behavior and microstructure of hybrid fiber ambient-cured one-part engineered geopolymer composites" by Alrefaei and Dai (2018) received 114 citations. In addition, India was the most active country in the related area, with 35 documents. The utilization of scientometric analysis will assist researchers from different countries in fostering collaborative research, sharing ideas and knowledge, and establishing collaborative enterprises to augment their research efforts.

- The integration of hybrid fiber in the geopolymer matrix decreased the composite's workability properties. This reduction increased with the fiber's volume fraction due to its hydrophilic nature, which tends to absorb water and reduce workability. The reduction ranged from 20% to 50% compared with the control sample.
- In most studies, HFRGC showed higher compressive strength than single fibers, strongly affected by fiber types, volume, content, and length. Besides, HFRGC showed 19% to almost 41% higher strength than the control sample. This outcome was a result of the capability of hybrid fiber to control cracking better than single fiber because each fiber type supported the other type; hence, the loads transferred from one type to another, and this mechanical is responsible for increasing the interfacial bonding in the matrix and strengthening the overall hybrid composite.
- HFRGC showed better performance of tensile and flexural strengths than the control sample and single fiber in the geopolymer matrix. Tensile strength increased from 39% to almost 68% compared to the plain. In addition, the tensile and flexural strengths exhibited a positive correlation with an increase in the tensile strength and modulus of elasticity of the fiber used, which proved efficiency in the formation of bridges to control micro and macro-cracks and prevent diffusion with the applied loads.
- Further research could focus on investigating the durability and environmental impact of hybrid fiber-reinforced composites. Exploring advanced manufacturing methods and optimizing fiber compositions may lead to stronger and lighter materials. Additionally, exploring potential applications in sectors like aerospace, automotive, and construction holds promise for addressing engineering challenges. Hybrid fiber composites offer exciting opportunities to advance material science and engineering technologies by addressing these areas.

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An Overview of Fly-ash Geopolymer Composites in Sustainable Advance Construction Materials

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ABSTRACT

Fly-ash geopolymer composites are an exciting advancement in eco-friendly construction materials. Fly-ash has become a sustainable alternative to regular cement because the approach addresses critical concerns in construction, such as high energy use, excessive carbon emissions and the challenge of managing industrial waste. In this review, a brief discussion on how fly-ash geopolymer composites could transform construction practices and reduce their impact on the environment. The construction industry is a major contributor to climate change, whereas industrial byproducts like fly-ash can also be an environmental

challenge. Thus, the fly-ash geopolymer composites offer an innovative solution by reusing this waste to create environmentally friendly binding materials. Fly-ash can effectively replace traditional cement in construction, improving the durability and sustainability of buildings. By reducing our reliance on regular cement, these composites could revolutionise construction practices across various industries. Developing and widely adopting fly-ash geopolymer composites could bring substantial benefits. It could significantly reduce the construction

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industry's carbon footprint and contribute to global efforts to combat climate change. Additionally, ongoing research aims to enhance these composites' strength, heat resistance, and chemical durability, further promoting sustainable construction and supporting a circular economy by turning industrial waste into valuable construction materials.

Keywords: Construction material, eco-friendly, fly-ash, geopolymer composite, waste material

INTRODUCTION

Fly-ash geopolymer composites represent advanced materials formed by combining waste fly-ash generated from coal combustion with geopolymer technology. These composites, typified by the fly-ash geopolymer, act as composite binders that effectively reduce energy consumption and carbon emissions during cement production. It offers a sustainable solution with significant environmental advantages, helping mitigate climate change and health-related issues. Furthermore, they provide a robust and long-lasting substitute for typical cement-based products, reducing waste, conserving resources, and reducing carbon emissions. These characteristics make them suitable for a wide range of applications in fields like construction, infrastructure, transportation, and environmental engineering (Albidah, 2021; Amran et al., 2021; Cong & Cheng, 2021; Gollakota et al., 2019; Klima et al., 2022; X. Li et al., 2022; Z. Li et al., 2021; Moujoud et al., 2023; Nasir et al., 2022; Nayak et al., 2022; Qaidi et al., 2022; Zhang et al., 2020; Zhang et al., 2021; Zhu et al., 2021).

Figure 1 describes the schematic process in which fly-ash, a byproduct of coal power plants, is recognised as a global industrial waste (Gollakota et al., 2019; Z. Li et al., 2021; Makgabutlane et al., 2022; Moujoud et al., 2023; Zhu et al., 2021; Nasir et al., 2022). When combined with alkalis, fly-ash forms a geopolymer, a robust material noted for its strength and chemical resistance. Geopolymer composites derived from fly-ash serve as an eco-friendly alternative to traditional binders such as cement (Albidah, 2021; Z. Li et al., 2021; Nasir et al., 2023; Zhang et al., 2020; Qaidi et al., 2022; Zhu et al., 2021). Researchers enhance these composites by incorporating materials like fibres or particles, which improve their strength, thermal stability, and chemical resistance (Cong & Cheng, 2021; Grabias-Blicharz & Franus, 2023; Le Ping et al., 2022; Liu et al., 2022; Makgabutlane et al., 2022; Zhang et al., 2021). This sustainable approach reduces dependency on traditional bindings and promotes environmentally friendly construction practices. Consequently, fly-ash geopolymer composites exhibit promising applications across various industries (Cong & Cheng, 2021; Liu et al., 2022; Wu et al., 2019; Zhu et al., 2021).

Electricity generation has traditionally depended on fossil fuels such as coal, natural gas, and oil, which pose significant environmental challenges, including carbon emissions and pollution. The global shift towards cleaner energy sources—such as solar, wind, hydro, geothermal, and biomass power—reflects a growing commitment to eco-friendly

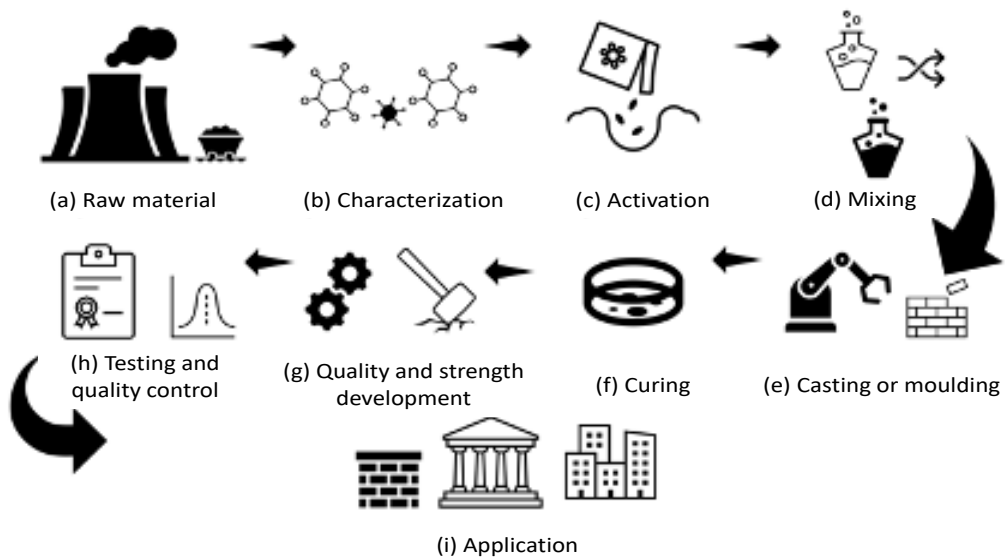


Figure 1. A schematic representation of the process involved in transforming fly-ash into a geopolymer material, offering a sustainable alternative to traditional cement-based materials: (a) Commencing the process, fly-ash is gathered as a byproduct from coal-fired power plants, (b) Subsequently, the raw fly-ash undergoes thorough analysis to determine its chemical composition and properties, (c) To initiate the geopolymerisation process, fly-ash must be activated, (d) The activated fly-ash is then mixed with water, resulting in the creation of a workable paste, (e) The geopolymer mixture is cast into moulds or shaped to achieve the desired form, (f) The geopolymer mixture undergoes a curing process, (g) Over time, the geopolymer cement or concrete gradually gains strength, (h) Samples of the geopolymer cement or concrete are subjected to various property tests, (i) Upon successful curing and testing, the geopolymer cement or concrete is considered ready for use (Grabias-Blicharz & Franus, 2023; Liu et al., 2022; Makgabutlane et al., 2022; Nasir et al., 2023)

alternatives. Solar panels harness energy from sunlight, wind turbines capture wind power, hydroelectric dams utilise flowing water, geothermal plants exploit the Earth's heat, and biomass power derives from organic waste and wood. Additionally, nuclear power offers a low-emission alternative.

The term 'global power generation capacity' encompasses the various methods employed worldwide to produce electricity. In 2008, the capacity for renewable electricity stood at 1 terawatt, compared to 3 terawatts from fossil fuels, as depicted in Figure 2. Over the past decade, renewable energy capacity has doubled, driven by decreasing costs and increasing adoption. By 2035, renewables are projected to surpass fossil fuels, generating approximately 15,000 terawatt-hours of electricity. This transition underscores the global commitment to cleaner, low-emission energy sources.

Figure 3 illustrates a decline in the production of solid byproducts from fuel combustion for electricity generation in the United States, primarily attributed to the decreased use of coal, a significant source of these byproducts. Notably, in 2020, for the first time since data collection commenced in 2008, a greater proportion of these byproducts were repurposed

rather than disposed of. The solid byproducts include fly-ash (fine particles captured from boiler flue gases), bottom ash (coarser particles collected at the bottom of boilers), and gypsum from flue gas desulphurisation systems designed to control sulphur dioxide emissions. In 2020, each tonne of coal burned for electricity generation yielded approximately 0.17 tonnes of these byproducts. These byproducts can be disposed of, utilised by power companies, sold for beneficial uses, or occasionally stored for future utilisation or sale.

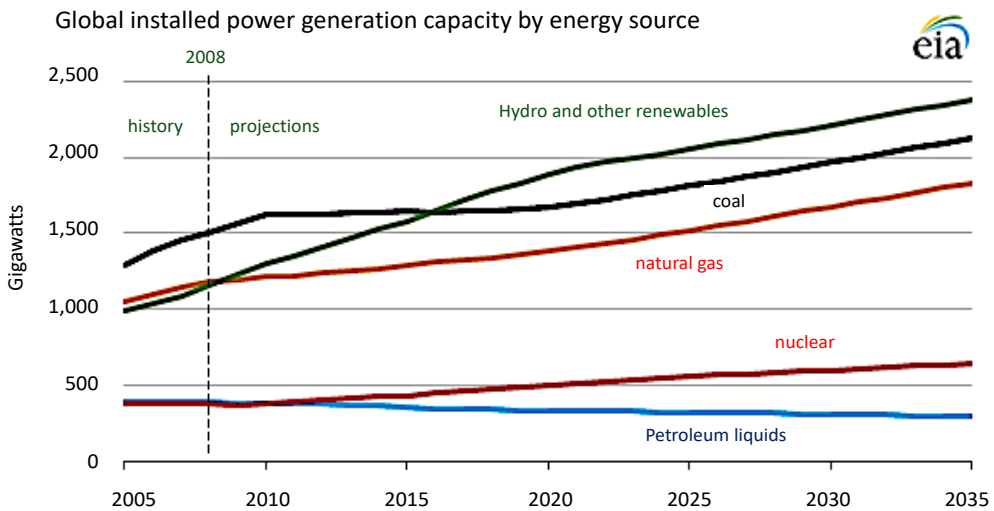


Figure 2. The source of global power generation capacity (U.S. Energy Information Administration, 2011)

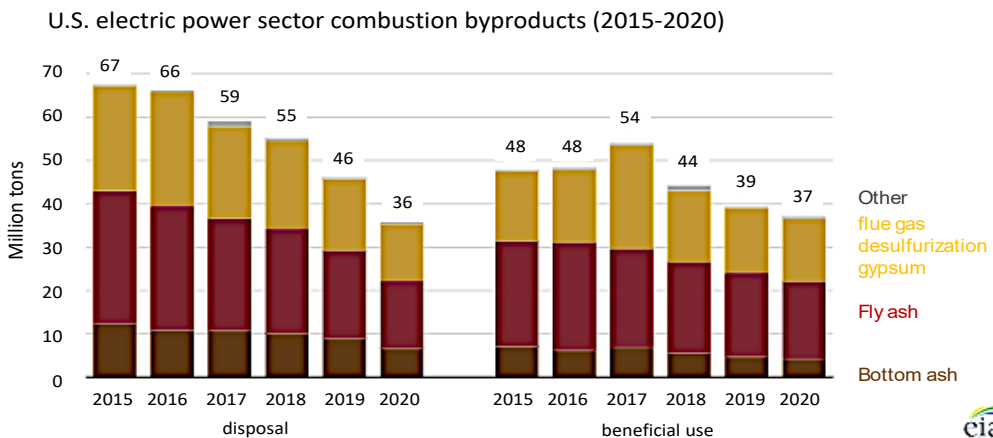


Figure 3. The U.S electric power sector combustion byproduct (year 2015–2020) (U.S. Energy Information Administration, 2022)

Between 2015 and 2020, the total production of combustion byproducts decreased by 36%, from 119 million metric tonnes to 76 million metric tonnes. This reduction is consistent with a 41% decrease in coal consumption in the electric power sector over the same period. As the overall production of combustion byproducts has declined, their utilisation has shifted from more costly disposal methods to beneficial applications. These applications include the manufacturing of products such as concrete and wallboard. Specifically, fly-ash and bottom ash are used in concrete and structural fills, while flue gas desulfurisation (FGD) wastes are employed to produce gypsum wallboard.

Furthermore, this review highlights the potential of fly-ash as an eco-friendly substitute for traditional binding agents in developing advanced composite materials within the interdisciplinary field of fly-ash geopolymer composites. Researchers aim to enhance these composites' mechanical, thermal, and chemical properties by incorporating reinforcing elements and optimising processing methods. These advancements hold significant implications for promoting sustainable construction practices and increasing the value of industrial waste through beneficial applications (Nayak et al., 2022; Zhang et al., 2021; Zhuang et al., 2016). For instance, Nayak et al. (2022) evaluated the advantages of concrete infused with fly-ash, providing practical examples from the American Coal Ash Association. Additionally, the study by Zhang et al. (2021) on the treatment techniques of fly-ash from municipal solid waste incineration has demonstrated economic benefits by characterising municipal solid waste incineration (MSWI) fly-ash for sustainable management prospects and mechanisms. Therefore, Table 1 provides a comprehensive summary of the various developments in fly-ash geopolymer composites.

Table 1
Summary of fly-ash geopolymer composite material development

Material used	Reinforcements	Processing process	Parameter's	Reference
Concrete		Mixing, Moulding, and Curing	<ul style="list-style-type: none"> Mixing (binder, aggregates, reinforcement and additive) 	Al-Majidi et al. (2016); Durak (2022); Mahmoodi et al. (2021);
Mortar	Fibre (glass steel, natural), aggregates,	Mixing, Moulding, Curing and	<ul style="list-style-type: none"> Thermal curing 	Makgabutlane et al. (2022); Marvila et al. (2021); Poloju et al. (2023); Sotelo-
Tiles	microparticles, and additives	Surface Treatment	<ul style="list-style-type: none"> Mechanical Physical Durability 	Piña et al. (2019); Wazien et al. (2016); Balakumaran et al., (2015)

Table 1 (Continue)

Material used	Reinforcements	Processing process	Parameter's	Reference
Coatings		Mixing, Application (brushing, spraying, rolling, and dipping), and Curing	<ul style="list-style-type: none"> • Mixing (binder, aggregates, reinforcement and additive) • Application (brushing, spraying, rolling, or dipping) • Thermal curing • Mechanical • Physical • Durability 	Al Bakri Abdullah et al. (2013); Biondi et al. (2019); Hamidi et al. (2022); Sotelo-Piña et al. (2019)
Foam	Gas-forming agent (Aluminium powder, hydrogen peroxide)	Mixing, Foaming, Moulding and Curing	<ul style="list-style-type: none"> • Mixing (binder, gas-binding agent and additive) • Thermal curing • Mechanical • Physical • Durability 	Al Bakri Abdullah et al. (2012); Atienza et al. (2023); Ducman and Korat (2016); Radina et al. (2023)
Bricks	Aggregates (Sand and crushed stone), fibre and nanoparticles	Mixing, Moulding, Curing and Surface finishing	<ul style="list-style-type: none"> • Mixing (binder, aggregates, reinforcement and additive) • Thermal curing • Mechanical • Physical • Durability 	Ibrahim et al. (2014); Lavanya et al. (2020); Makgabutlane et al. (2022); Wan Ibrahim et al. (2015)

PROPERTIES OF FLY-ASH GEOPOLYMER COMPOSITES

Fly-Ash Geopolymer

Fly ash is a fine particulate material generated as a byproduct during the combustion of coal in power plants. This material is collected from the flue gases using electrostatic precipitators or filter bags, which effectively separate it from the smoke. Moreover, fly-ash is known for its pozzolanic properties, meaning it can react with calcium hydroxide in the presence of

water to form compounds that possess cementitious characteristics. These unique properties make fly-ash a valuable resource in various industries, including construction, automotive, and packaging, where it is utilised to enhance the strength and durability of materials. In the construction industry, fly-ash improves the mechanical properties of concrete and other building materials, contributing to stronger and more durable structures. In the automotive sector, fly-ash creates lighter and stronger composite materials, enhancing vehicle performance and efficiency. Additionally, fly-ash is incorporated into the packaging industry to increase structural integrity and reduce environmental impact. The versatility and beneficial characteristics of fly-ash make it significant for promoting sustainability and innovation across these industries (Fediuk & Yushin, 2015; Nasir et al., 2022; Zierold & Odoh, 2020). Researchers such as Makgabutlane et al. (2022) are exploring the potential of creating sustainable and eco-friendly composite materials by combining plastic and fly-ash waste. The goal of this research is to enhance the mechanical properties of these materials for a variety of applications while also reducing their environmental impact.

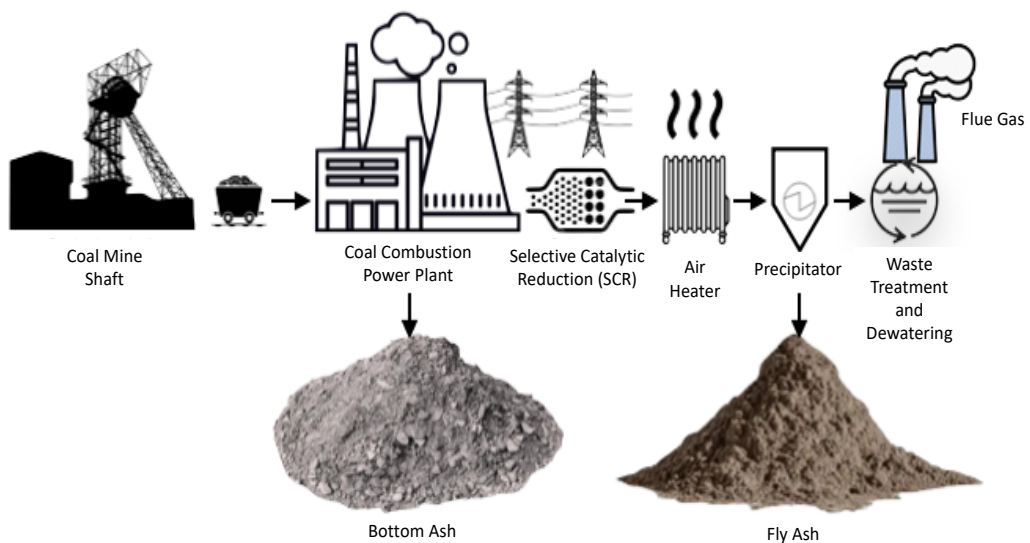


Figure 4. The production process of fly-ash and bottom ash (Baranwal et al., 2021; Sabarinath & Vittala, 2015)

Fly-ash, a byproduct of coal combustion can form a geopolymer when it interacts with water and calcium hydroxide. Geopolymers are materials with binding properties similar to those of conventional cement. This geopolymerisation process involves the polymerisation of silicon and aluminium species into a three-dimensional network, resulting in a material with excellent binding and structural properties. Researchers have been investigating the combination of fly-ash with biodegradable polymers and natural fibres such as hemp, jute, and sisal to produce eco-friendly and durable composite materials (Hamidi et al., 2022;

Khatib et al., 2009; Zhang et al., 2020). Studies such as those conducted by Zhang et al. (2020) on fly ash/slag geopolymer concrete emphasise the crucial role of geopolymer materials in promoting sustainable construction practices. These materials reduce the dependency on non-renewable resources and contribute to developing environmentally friendly composites, offering significant environmental benefits. As a result, Figure 4 illustrates a better understanding of the raw materials, characteristics, and processing methods used to obtain fly-ash, as well as its application and potential for sustainable development.

Mechanical Properties of Fly-ash Geopolymer Composites

Understanding how fly-ash particles interact with biodegradable elements is critical for creating sustainable materials. These interactions play a pivotal role in determining the overall mechanical performance of the composites. Table 2 summarises the mechanical characteristics of fly-ash biocomposites. The properties discussed include porosity (10% to 50%), indicating the presence of empty spaces within the material. Density ranges from 1.0 to 2.5 g/cm³, and hardness (measured on the Vickers scale) varies from 10 to 100. The ability of the material to withstand compression is measured by its compressive strength (8 to 11 MPa), while its resistance to bending is represented by its flexural strength (4 to 6 MPa). Impact strength (5 to 50 joules per centimetre) measures how much energy the material can absorb during impacts. This review promotes environmental sustainability and the development of eco-friendly materials by efficiently utilising waste resources to create valuable composites.

Table 2
Physical and mechanical properties of fly-ash biocomposites

Properties	Description	Reference
Density, g/m ³	1.0 – 2.5	Hager et al. (2021); Wan Mastura et al., (2013); Wazien et al. (2016)
Porosity, %	10 – 50	Alehyen et al. (2017); X. Li et al. (2022); Nayak et al. (2022)
Hardness, (Vickers) <i>HV</i>	10 – 100	Estrada-Arreola et al. (2014); Gohatre et al., (2020); Luhar and Luhar (2022)
Compressive strength, MPa	8 – 11	Qaidi et al. (2022); Wang et al. (2022); Zhuang et al. (2016)
Flexural strength, MPa	4 – 6	Korniejenko et al. (2016); Makgabutlane et al. (2022); Yu and Jia, (2022)
Impact strength, Joule/cm	5 – 50	Bajpai et al. (2020); Shi et al.(2021)

Morphological Properties

Investigations into the morphological properties of fly ash biocomposites offer various insights into the structure and constitution of composite materials derived from fly-ash and biodegradable elements. These reviews contribute to the comprehension of the physical organisation, configuration, and interactions between fly-ash particles and biodegradable components within the composite matrix, which is a core objective in this field of research (Al-Majidi et al., 2016; Nasvi et al., 2016; Poloju et al., 2023; Roviello et al., 2016). By scrutinising the inner morphology of the components and how it influences the characteristics of the composite through an analysis of the physical arrangement, dispersion, and interactions of these elements within the composite, the aim is to develop cutting-edge, sustainable materials with enhanced performance that can be applied across a wide spectrum of potential uses.

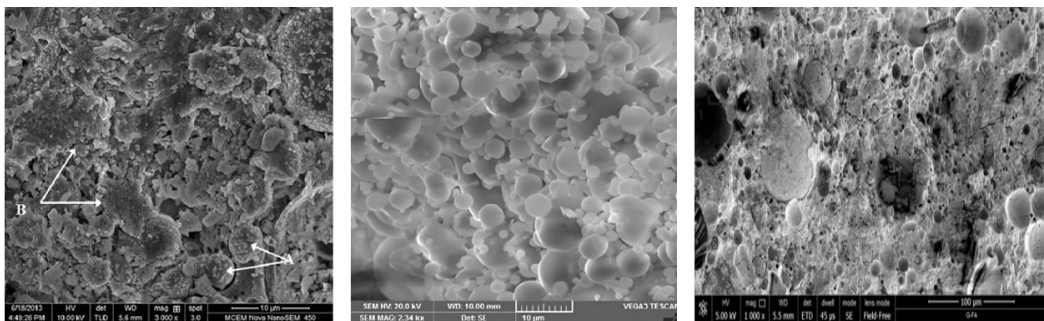


Figure 5. Morphology of fly-ash geopolymer; a) Fly-ash geopolymer cement (Nasvi et al., 2016), b) Fly-ash geopolymer based (Poloju et al., 2023), c) Fly-ash geopolymer concrete (Roviello et al., 2016)

As previously discussed, the combustion of coal results in the creation of a fine powder referred to as fly-ash. This fine powder comprises minute spherical particles with the potential to chemically interact with water and calcium hydroxide, thereby giving rise to a cementitious binding agent known as "geopolymer." These particles possess distinct pozzolanic properties, and as such, the distribution and arrangement of these particles within the composite matrix play a pivotal role in influencing the overall performance and characteristics of the material. In diverse research on morphological properties, microscopic examination techniques, such as scanning electron microscopy (SEM), are used (Figure 5). These techniques enable the exploration of the inner structure, the formation of bonds, and the dispersion of particles within the matrix, consequently affecting the composite's overall mechanical behaviour. Additionally, the morphological attributes are of paramount importance as they determine the mechanical strength and load-bearing capacity through an optimised and uniform distribution of fly-ash particles within the matrix (Luna-Galiano et al., 2016; Nasvi et al., 2016; Poloju et al., 2023; Roviello et al., 2016).

Besides investigating the morphological characteristics of fly-ash biocomposites, it becomes feasible to tailor their mechanical properties to meet specific application requirements by manipulating the arrangement and interaction of fly-ash particles with biodegradable components. By enhancing the mechanical performance of composites based on fly-ash and understanding how fly-ash particles interact with biodegradable constituents, this research endeavours to produce durable and environmentally responsible materials. Researchers employ scanning electron microscopy (SEM) or transmission electron microscopy (TEM) to explore microstructural arrangements, examining how these elements interconnect and establish bonds within the matrix. Recognising uneven distribution or clustering is crucial in preventing performance degradation and the loss of strength. An understanding of these interfacial interactions aids in load transfer and the prevention of crack formation within the composite. Researchers gain profound insights into how microstructure impacts the macroscopic behaviour of fly-ash biocomposites by examining morphological traits across various scales, thereby facilitating the design and technological optimisation of specific attributes. The investigation of morphological features ultimately contributes to the advancement of materials science by fostering the creation of innovative, environmentally friendly composites suitable for a diverse range of applications.

Thermal Properties of Fly-Ash Geopolymer Composite

Understanding the behaviour and performance of fly-ash biocomposites within the realm of geopolymer composites is heavily reliant on their thermal characteristics. The geopolymerisation process that gives rise to materials known as geopolymer composites involves a chemical reaction between fly-ash and alkali activators, such as sodium or potassium silicates, culminating in the formation of a three-dimensional, inorganic polymer network (Atienza et al., 2023; Narattha et al., 2022). Fly-ash biocomposites exhibit noteworthy attributes in various thermal aspects. Several factors influence their thermal conductivity, including the type and quantity of fly-ash, the presence of biodegradable materials, and the nature of the geopolymer adhesive. The outcome is lower thermal conductivity compared to traditional cement-based composites. In a review study conducted by Klima et al. (2022), a substantial focus was placed on exploring the thermal characteristics of fly ash-based geopolymers in high-temperature settings. The review underscored the significance of factors such as pore interconnectivity in preventing damage, the impact of mix design and curing techniques, and the role of alkali sources in bolstering heat resistance. These properties find utility in applications like fire-resistant construction materials, high-temperature insulation, and protective coatings for industrial equipment operating in exceedingly challenging thermal conditions. Moreover, Kaya and Köksal (2022) have highlighted the vital need for a comprehensive examination of mix

design and curing methods, the influence of alkali sources on thermal resistance, and the role of pore interconnectivity in averting damage, especially in the construction sector. For applications exposed to high temperatures, the careful management of thermal degradation is imperative to ensure performance under demanding conditions.

Table 3
Principal thermal properties of fly-ash biocomposites

Properties	Description	Application	Reference
Thermal conductivity, W/mK	0.1 – 4.0	<ul style="list-style-type: none"> • Insulation • Building material • Coating • Electronic enclosure • Industrial equipment • Solar panel • Cryogenic 	Atienza et al. (2023); Feng et al. (2015); Karakaş et al. (2023); Narattha et al. (2022); Novais et al. (2016); Shao et al. (2018)
Coefficient thermal expansion, $10^{-6}/^{\circ}\text{C}$	5 – 20	<ul style="list-style-type: none"> • Construction material • Infrastructure material • Electronic packaging • Automotive • Aerospace • Optical device • Industrial equipment 	Ali and Vijayalakshmi Natrajan (2021); He et al. (2020); Kaya and Köksal (2022); Ma and Dehn (2017)
Melting temperature, $^{\circ}\text{C}$	600 – 1200	<ul style="list-style-type: none"> • Furnace • Coating • Aerospace • Industrial equipment • Power plant • Energy storage system 	Durak (2022); Hager et al. (2021); He et al. (2020); Klima et al. (2022); Moujoud et al. (2023); Nasir et al. (2023)

Table 3 summarises the key thermal attributes of fly-ash biocomposites, along with their descriptions and existing applications. These characteristics encompass a thermal conductivity spanning from 0.1 to 4.0 W/mK, making them suitable for a wide range of uses such as cryogenic conditions, solar panels, construction materials, coatings, electronic enclosures, and insulation. The coefficient of thermal expansion, falling within the range of 5 to 20 x 10⁻⁶ °C, plays a pivotal role in regulating thermal stress, benefiting industrial equipment and materials in buildings and infrastructure, electronic packaging, and the automotive and aerospace sectors. The melting temperature, ranging from 600 to 1200°C, is a critical parameter for various applications, including furnaces, coatings, aerospace components, industrial equipment designed to manage thermal stress, power plants, and energy storage systems. This extensive table furnishes valuable insights into the adaptable thermal properties of fly-ash biocomposites and their diverse utility across multiple sectors.

Fly-ash biocomposites possess crucial thermal properties that render them invaluable for a multitude of applications, from thermal insulation to the mitigation of thermal expansion. They can also be formulated to serve as natural fire retardants or enhance fire resistance. These versatile materials can be customised for specific purposes, ranging from high-temperature industrial environments to the design of energy-efficient construction materials. In doing so, they offer promising solutions developing sustainable and versatile materials across numerous industries.

Physical Properties

Geopolymer composites' physical attributes significantly influence their performance and applicability. These attributes encompass a spectrum of observable traits, such as density, porosity, hardness, strength, thermal conductivity, and more. These traits play a critical role in shaping the behaviour and performance of geopolymer composites, ultimately influencing their suitability for diverse applications.

Geopolymer composites boast a multitude of noteworthy characteristics. Their density aligns closely with conventional cement-based materials, offering advantages like enhanced energy efficiency and resilience in adverse conditions, rendering them well-suited for insulation, construction, and aerospace applications. The mechanical strength they exhibit is intricately linked with their porosity; lower porosity correlates with increased wear resistance and durability. Hardness is pivotal in creating durable and reliable components, as it quantifies a material's capacity to withstand deformation and abrasion. Nonetheless, a study by Nasir et al. (2023) concerning recycled polyethylene terephthalate (rPET) and industrial waste fly-ash, in response to environmental concerns, investigated the thermal behaviour and microstructure of the composite. Excessive fly-ash was found to induce degradation, leading to voids and clustering, ultimately affecting thermal performance.

Table 4
Physical properties of fly-ash biocomposites

Parameter	Description	Reference
Particle diameter (μm)	0.01–100	Akid et al. (2023); Al-Nini et al. (2020); Alterary and Marei (2021); Ferdous et al. (2020); Grabias-Blicharz and Franus, (2023); Hager et al. (2021); Harihanandh et al. (2021); Klima et al. (2022); Korniejenko et al. (2016); Li et al. (2021); Liu et al. (2022), Maiti and Prasad (2016); Makgabutlane et al. (2022); Nasir et al. (2023); Poloju et al. (2023); Poyyamozi et al. (2023); Prusty and Patro (2015); Qaidi et al. (2022); Shao et al. (2018); Sonebi et al. (2022)

Table 4 (Continue)

Parameter	Description	Reference
Texture	Silt loam	Al-Nini et al. (2020)
Specific surface area (cm^2g^{-1})	2500–4000	Grabias-Blicharz and Franus (2023)
Specific gravity (g cm^{-3})	1.6–2.6	Hager et al. (2021)
Bulk density (g cm^{-3})	0.9–1.3	Harihanandh et al. (2021)
Water holding capacity (%)	40–60	Makgabutlane et al. (2022)
Colour	White/yellow- orange/black	Klima et al. (2022)

The fundamental physical properties of fly-ash biocomposites are outlined in Table 4. These properties include texture categorised as silt loam, particle diameters ranging from 0.01 to 100 μm , specific gravity within the range of 1.6 to 2.6 g/cm^3 , a specific surface area spanning from 2500 to 4000 cm^2/g , a water retention capacity of 40%–60%, and bulk densities ranging from 0.9 to 1.3 g/cm^3 . These biocomposites exhibit diverse colours, including white, yellow-orange, and black. This table furnishes detailed information regarding the particle size, texture, density, water-holding capacity, and colour variations, encompassing the primary physical characteristics of fly-ash biocomposites.

Additionally, a review study conducted by Qaidi et al. (2022) focusing on fly ash geopolymer concrete has unveiled a noteworthy observation. It underscores the potential of environmentally friendly materials such as fly ash to match and often surpass the mechanical properties of polymer concrete in aspects like the production process, mix design, compressive strength, and microstructure of fly ash-geopolymer concrete. Consequently, their commendable compressive strength renders them exceptionally suitable for load-bearing constructions, ensuring stability and cost-effective maintenance. Equally significant is flexural strength, which plays a vital role in structural components such as beams and panels, particularly when subjected to bending loads. In summation, it is imperative to grasp and optimise the physical characteristics of geopolymer composites to tailor the material for specific applications. Geopolymer composites, as high-performing materials, offer a more environmentally friendly and efficient solution to contemporary engineering challenges across a spectrum of high-technology industries.

ADVANCED APPLICATION OF FLY-ASH GEOPOLYMER COMPOSITES

Fly-ash geopolymer composites have garnered significant interest as sustainable advanced construction materials due to their superior mechanical properties, environmental benefits, and versatility. These composites are utilised in various applications, from infrastructure

development to specialised industrial uses, promoting sustainable construction practices. Their enhanced functionalities include improving electrical conductivity for structural health monitoring, utilising industrial byproducts for superior properties, and serving as efficient repair materials for infrastructure.

Furthermore, fly-ash geopolymer composites represent a significant advancement in sustainable construction materials, offering a wide range of applications across various sectors. Their superior mechanical properties, environmental benefits, and versatility make them crucial in developing sustainable infrastructure. As research and innovation continue to advance, the scope and potential of fly-ash geopolymer composites are expected to expand, further cementing their role in the future of construction technology. These attributes underscore the versatility and sustainability of fly-ash geopolymer composites. As research expands, the applications of these composites are expected to grow, contributing to the development of smart, durable, and eco-friendly construction solutions (Bijeljčić & Ristić, 2023; Mizerová et al., 2019; Wang et al., 2023).

Infrastructure and Building Construction

Structural Components

Fly-ash geopolymer composites are extensively used to fabricate structural components such as beams, columns, and slabs. Their high compressive strength, durability, and resistance to environmental degradation make them suitable for load-bearing applications. Studies have demonstrated that fly-ash-based geopolymer concrete can achieve mechanical properties comparable to or even exceeding those of traditional Portland cement concrete, making it a viable alternative for reinforced concrete structures.

The use of fly-ash geopolymer composites in precast concrete elements is gaining popularity due to the reduced curing times and lower energy requirements during production (Figure 6). Precast geopolymer elements, such as panels, blocks, and pipes, offer enhanced performance characteristics, including improved thermal insulation, fire resistance, and a reduced carbon footprint.

Transportation Infrastructure

Road Construction

Fly-ash geopolymer composites are being utilised in the construction of roadways and pavements. The material's superior resistance to freeze-thaw cycles, chemical attacks, and abrasion makes it ideal for such applications. Geopolymer-based road construction not only enhances the longevity and durability of road surfaces but also contributes to the reduction of greenhouse gas emissions associated with traditional asphalt and cement-based materials (Bellum et al., 2020; Y. Li et al., 2023; Parcesepe et al., 2022; Sarath Chandra & Krishnaiah, 2022; Sofri et al., 2022).



Figure 6. Various applications from fly-ash geopolymer composites: (a) Structural components, (b) Precast elements (Imtiaz et al., 2020; Selim et al., 2024; Shehata et al., 2022)

Railway Sleepers

The railway industry is exploring the use of fly-ash geopolymer composites for manufacturing railway sleepers. These sleepers exhibit excellent mechanical properties, such as high strength and durability, essential for supporting heavy rail traffic. Additionally, the adoption of geopolymer composites helps mitigate the environmental impact associated with the production and disposal of conventional concrete sleepers (Gourley, 2014; Jokubaitis et al., 2020; Salih et al., 2021; Zakeri & Sadeghi, 2007).

Figure 7 illustrates the advanced applications of fly-ash geopolymer composites in transportation infrastructure, highlighting their use in road construction and railway sleepers. For road construction, these composites offer enhanced durability through resistance to freeze-thaw cycles and chemical attacks, along with sustainability benefits such as a reduced carbon footprint and lower energy consumption. In the context of railway sleepers, fly-ash geopolymer composites provide high strength, impact resistance, and environmental benefits by utilising waste materials and reducing carbon emissions. Their low maintenance requirements and extended service life make them a superior alternative to traditional materials in both applications, contributing to developing eco-friendly and resilient transportation infrastructure systems.

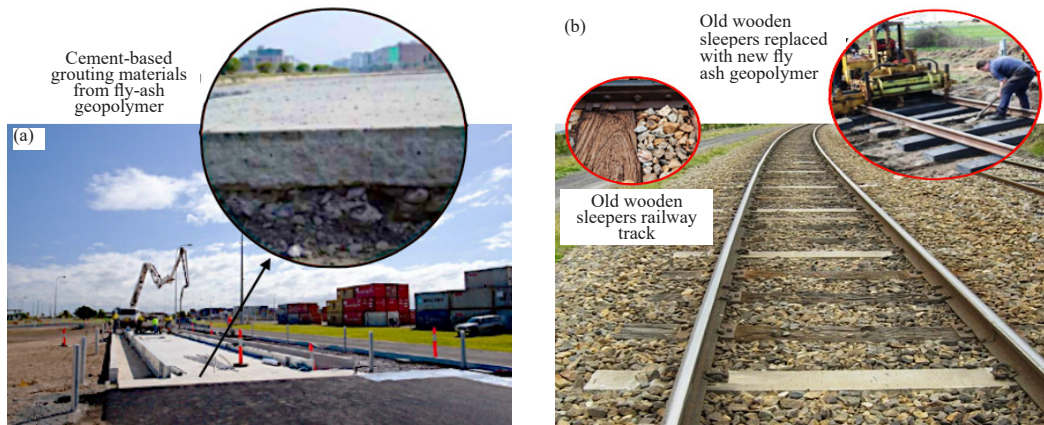


Figure 7. Advanced applications of fly-ash geopolymer composites in transportation infrastructure: (a) Geopolymer fly-ash used for concrete road pavement, (b) Railway sleeper with fly-ash geopolymer concrete (J. Li et al., 2023; Kerchof & Wu, 2012; Parcesepe et al., 2022; Plastic News, 2017)

Marine and Coastal Applications

Fly-ash geopolymer composites are highly resistant to the aggressive marine environment, making them suitable for marine and coastal construction projects. Applications include seawalls, breakwaters, and underwater pipelines. The material's inherent resistance to chloride and sulphate attacks and low permeability ensures prolonged service life and reduced maintenance costs in harsh marine conditions.

Figure 8 illustrates the advanced applications of fly-ash geopolymer composites in marine and coastal environments. These composites are ideal for constructing seawalls and breakwaters due to their excellent resistance to chloride and sulphate attacks, low permeability, and reduced carbon footprint (Latham et al., 2008; Pasupathy et al., 2021; van Gent, 2021). In underwater pipeline construction, their high compressive and flexural strength, along with erosion resistance, ensure durability and longevity. Additionally, fly-ash geopolymer composites are used in marine construction elements such as piers, docks, and offshore platforms, offering superior durability, fire resistance, and sustainability. Using recycled materials and lower energy consumption during production further contribute to their environmental benefits, making them a preferred choice for eco-friendly and resilient marine infrastructure.

Industrial Applications

Waste Containment

Fly-ash geopolymer composites are employed to contain and stabilise hazardous wastes. Their ability to immobilise heavy metals and other contaminants makes them an effective material for lining landfills, encapsulating waste materials, and constructing containment

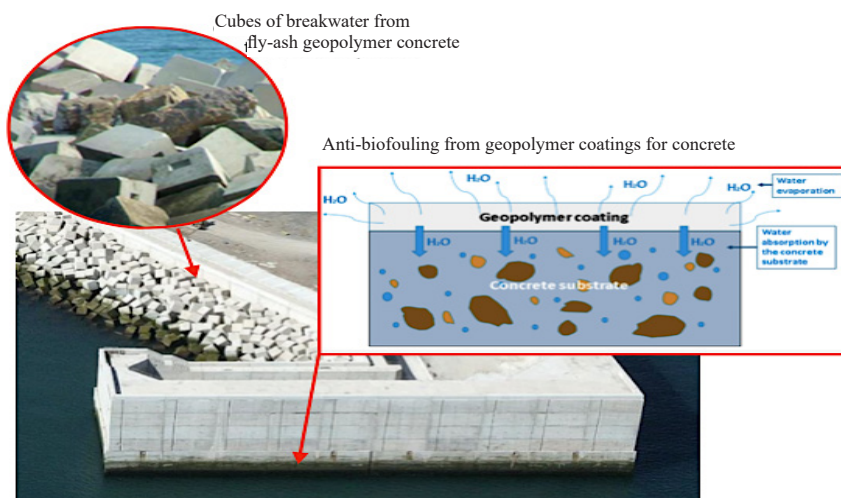


Figure 8. Advanced applications of fly-ash geopolymer composites in the marina and coastal applications (Biondi et al., 2019; Latham et al., 2008; Pasupathy et al., 2021; van Gent, 2021)

barriers. This application addresses environmental pollution and provides a sustainable solution for waste management. The advanced application of fly-ash geopolymer composites in waste containment is an exciting development in environmental science and industrial practices. Synthesis of these geopolymers from industrial byproducts like fly-ash mitigates waste and creates valuable materials for environmental remediation. Their use in adsorptive processes for removing heavy metals and dyes from wastewater exemplifies a sustainable approach to managing industrial pollutants.

Future research should focus on enhancing the performance of geopolymer-based adsorbents, expanding their application to a broader range of contaminants, and verifying their effectiveness in real-world wastewater treatment scenarios. Fly ash geopolymers' economic and environmental benefits underscore their potential to revolutionise waste containment practices and support a circular economy. By advancing these technologies, researchers and industry can work towards a more sustainable future with reduced reliance on traditional, less eco-friendly materials. Subsequently, Table 5 emphasises the materials, applications, and significant insights from a variety of studies on fly-ash-based geopolymers, thereby expounding their potential for sustainable waste management and environmental remediation.

Heat-Resistant Materials

The thermal stability and fire-resistant properties of fly-ash geopolymer composites make them suitable for applications requiring high-temperature resistance. Industries use these composites to manufacture fireproof panels, insulation materials, and refractory linings. The ability to withstand extreme temperatures without significant loss of structural integrity

is a key advantage in such applications. Figure 9 illustrates the advanced applications of fly-ash geopolymer composites in industrial settings, focusing on waste containment and heat-resistant materials. For waste containment, these composites provide excellent chemical resistance, low permeability, and the ability to stabilise and solidify hazardous materials, ensuring environmental protection and structural integrity. In high-temperature applications, fly-ash geopolymer composites are used in refractory linings, fireproofing, thermal insulation panels, and heat shields due to their superior thermal stability and fire resistance. These properties make them ideal for enhancing safety, durability, and efficiency in various industrial processes.

Table 5
Advanced applications of fly-ash geopolymer composites for industrial waste containment

Materials	Applications	Significant insight	Reference
Fly-ash-based aluminosilicate	<ul style="list-style-type: none"> • Environmental applications • Adsorbents • Catalysts 	<ul style="list-style-type: none"> • Low-cost, eco-friendly • Immobilises toxic or radioactive metals • Potential to adsorb liquid or gas contaminants • Provides significant economic benefits from resource recycling 	Adeyemi (2021)
Geopolymers	<ul style="list-style-type: none"> • Adsorption of heavy metals and dyes 	<ul style="list-style-type: none"> • High efficiency • Cost-effectiveness • Similar performance to other materials • Adsorption is spontaneous and endothermic • Future research on performance enhancement and real wastewater testing 	Siyal et al. (2018)
Fly ash-based geopolymer, Polyethersulfone (PES)	<ul style="list-style-type: none"> • Adsorption of Heavy Metal Ions 	<ul style="list-style-type: none"> • Convenient, low-cost and eco-friendly • High adsorption capacity for Pb²⁺, Cu²⁺, Cd²⁺, and Ni²⁺ • Porous structure with significant surface area 	Onutai et al. (2023)
Biomass fly-ash-based geopolymers	<ul style="list-style-type: none"> • Adsorption of methylene blue 	<ul style="list-style-type: none"> • Highly porous and lightweight • Superior adsorption capacity • Potential for use in packed beds as membranes, • Reduces the environmental footprint 	Novais et al. (2018)

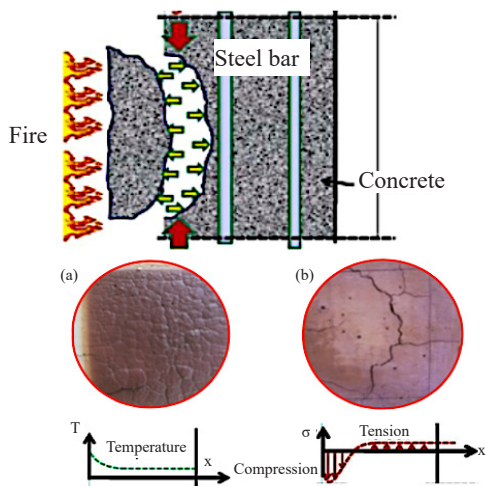


Figure 9. Fly-ash geopolymer used in the construction industry as fire resistance of geopolymer concrete deterioration due to fire damage is generally caused by two mechanisms: (a) thermal dilation and (b) Vapor pressure (Amran et al., 2022; Vickers et al., 2016)

Innovative and Future Applications

3D Printing

Advancements in 3D printing technology have opened new avenues for the use of fly-ash geopolymer composites. The material's excellent printability and rapid setting time make it ideal for additive manufacturing. 3D-printed geopolymer structures can be used in various applications, including custom architectural elements, complex geometrical forms, and rapid prototyping.

Space Construction

Research is being conducted on using fly-ash geopolymer composites for construction in extraterrestrial environments, such as the Moon and Mars. The material's ability to

be synthesised from locally available resources and its strength and durability make it a promising candidate for building habitats and infrastructure on space exploration missions.

Thus, Figure 10 describes the fly-ash geopolymer composites emerging as a transformative material for innovative and future applications in 3D printing and space construction. Their mechanical properties, sustainability, and adaptability make them ideal for creating complex, durable, and eco-friendly structures. As technological advancements continue, the potential of fly-ash geopolymer composites in these cutting-edge fields is expected to grow, contributing to revolutionary developments in construction methodologies both on Earth and beyond.

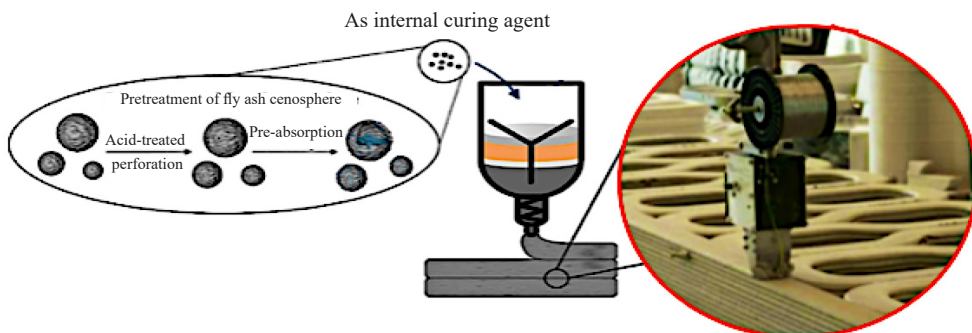


Figure 10. Pioneering the future: fly-ash geopolymer composites in 3D printing and space construction. Harnessing the power of sustainable materials for innovative applications on Earth and beyond (Tao et al., 2022)

CONCLUSION

In conclusion, the literature review underscores the transformative potential of fly-ash geopolymer composites in reshaping the construction industry while advancing environmental sustainability. A comprehensive analysis of their mechanical, morphological, thermal, and physical properties shows that these composites hold promise across diverse sectors, including the aerospace and automotive industries. Key findings suggest optimal percentages for properties crucial to their performance, laying a foundation for informed material selection and application. Moreover, ongoing research endeavours focus on enhancing the strength, heat resistance, and chemical durability of fly-ash geopolymer composites. Innovations such as incorporating reinforcing elements and refined processing techniques are driving advancements in their mechanical properties. Additionally, insights gained from morphological analyses, particularly through techniques like scanning electron microscopy (SEM), contribute to developing sustainable materials with enhanced performance characteristics.

The environmental advantages of fly-ash geopolymer composites, including reduced carbon emissions and waste generation, underscore their significance in promoting sustainable construction practices and fostering a circular economy. By repurposing industrial byproducts like fly-ash, these composites offer a viable alternative to conventional binding agents, mitigating the environmental footprint of construction activities. Overall, the literature highlights the pivotal role of fly-ash geopolymer composites in driving sustainable construction practices and supporting a circular economy model. Future research should prioritise further exploration into optimising these materials' properties and expanding their applications to address the evolving challenges of the 21st-century construction industry.

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A Comparative Analysis Review of Plant Fibres in Advanced Bio-based Material for Sustainable Drone Construction

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ABSTRACT

As researchers' awareness of ecological impact and climate change increases, several solutions were proposed to help reduce carbon emissions and promote the circularity of materials. Drones technology can help monitor the environment since it can cover a large area, collect real-time images and data, and operate in dangerous environments. Also, the drone's ecological factor could be further increased by its construction itself. Thus, many researchers are trying to develop a sustainable drone using plant fibres to reduce carbon emissions and ensure the circularity of materials. This review mainly compares the drones made from plant fibres and traditional materials such as plastics and synthetic fibres. This review also includes the introduction of material circularity, the drone's role in helping ensure material circularity and environment safety, and the advantages and disadvantages of the drone materials. The review will also compare the drone performances made from different bio-based materials with conventional ones. Plant fibres' role in drone construction significantly contributes to reducing carbon emissions and ensuring the circularity of materials. With drone construction paving the way for other critical structural applications, there is a possibility that plant fibres will soon become the most significant raw material for sustainable products.

Keywords: Drone construction, material circularity, plant fibres, sustainable drone

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INTRODUCTION

Unmanned aerial vehicles (UAVs), commonly known as drones, have a rich history dating back to the 18th century. The materials used in the construction of drones have evolved and are influenced by technological advancements and military needs. In the 18th century, the Montgolfier

brothers in France experimented with unmanned balloons, marking the early use of UAVs. The Montgolfier brothers' balloon, which made the first manned flight in 1783, was made of cotton canvas with paper glued onto both sides (Tretkoff, 2006). After that, the material was replaced with aluminium. The first use of aluminium for UAVs can be traced back to the early 20th century. In June 1919, the Junkers F.13, the first all-metal airliner, used 2017-T4 aluminium alloy as the body material, marking a significant milestone in using aluminium in aircraft construction (Haomei Aluminium, 2023). This development laid the foundation for using aluminium in the aviation industry, including manufacturing UAVs.

Therefore, it can be inferred that aluminium was first used for UAVs around the same time, during the early 20th century. During World War II, there was a surge in plastic production, which spread to UAVs (Rangel-Buitrago & Neal, 2023). After that, in the late 20th century, carbon fibre was used in high-performance applications from aeroplanes to automobiles and satellites to sporting goods (Zhang et al., 2023). The use of carbon fibre in UAVs has become widespread, primarily due to its lightweight and high-strength properties, contributing to improved aircraft performance and fuel efficiency (Parveez et al., 2022). From there, fibre-reinforced polymer composites adoption in aerospace and aviation industries gained momentum in the 1980s and 1990s (Geoff Poulton, 2017). Fibre-reinforced polymer composites are a combination of fibres, such as carbon or glass, and a polymer matrix, such as epoxy or polyester resin. These materials offer high strength-to-weight ratios, corrosion resistance, and design flexibility, making them ideal for UAV manufacturing (Rajak et al., 2019).

In recent years, synthetic fibres have been replaced with natural fibres to make UAVs due to environmental considerations and the desire for more sustainable materials. Natural fibre composites are considered more environmentally friendly than synthetic fibre materials, as they are biodegradable, derived from renewable sources, and offer advantages such as abundance, availability, and low cost (Thyavihalli Girijappa et al., 2019). The increased awareness of the environmental impact of synthetic materials has led to a growing interest in developing eco-friendly materials, including natural fibre-based composites, for various industrial sectors, including aerospace engineering. Additionally, natural fibres are introduced to make the composites lighter. While they may have some disadvantages, such as poor resistance to moisture, their overall sustainability and environmental benefits make them an attractive alternative to synthetic fibres in UAV manufacturing. Previous research stated that kenaf and PALF can be used as alternative composite materials, particularly for multifunctional applications (Rahman & Ariffin, 2022). Figure 1 summarises the evolution of the drone materials.

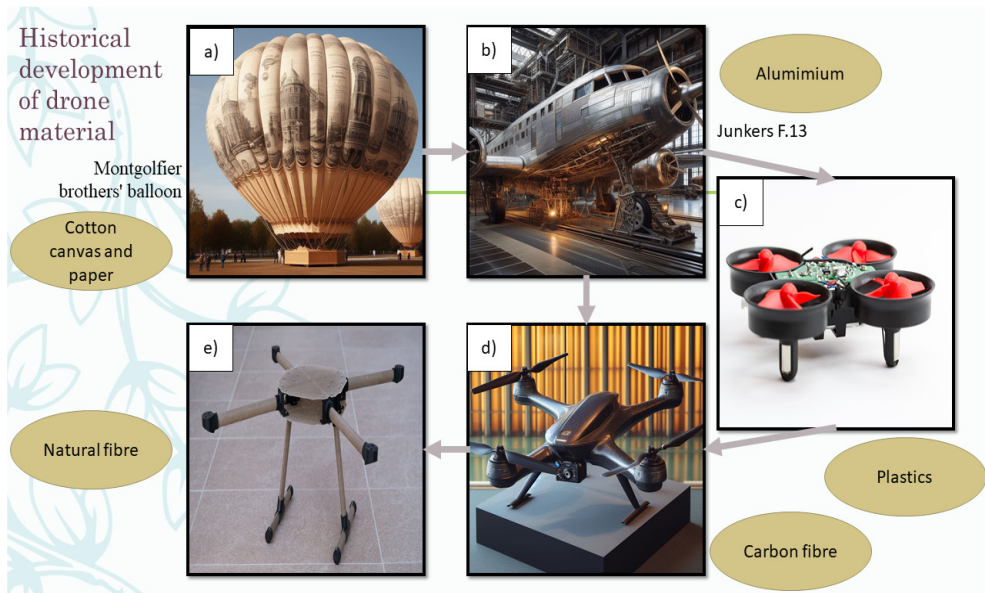


Figure 1. Historical development of drone materials

Source: Authors' work, all images are AI-generated from: a) <https://www.bing.com/images/create?FORM=GENILP>, b) <https://www.bing.com/images/create?FORM=GENILP>, c) <https://pixabay.com/photos/mini-drone-radio-control-flying-2199857/>, d) <https://www.bing.com/images/create?FORM=GENILP>, e) <https://www.thingiverse.com/thing:4800248>

The history of drone materials reflects the continuous evolution of technology and the adaptation of materials to meet the changing needs of UAVs, from their early use in warfare to their modern applications in various fields, including photography (Patil et al., 2023), filming (Patil et al., 2023), and goods delivery (Chi et al., 2023). Drones are being used to revolutionise sustainability and the circular economy. They have transformed supply chain management by optimising the distribution and logistics processes, reducing the need for traditional, fuel-dependent transportation methods (Rejeb et al., 2023). Drones are also used for waste management and recycling, and they can produce high-resolution 3D models of landfills to monitor changes over time (Sliusar et al., 2022). In addition, drones are instrumental in environmental monitoring and protection, as they can survey and assess remote and inaccessible areas, enabling rapid response to illegal logging, poaching, and environmental disasters (Bollard et al., 2022). The circular economy offers a framework to improve the sustainability of drones by designing out waste and pollution in all phases, making more durable products to extend the use phase, including drone construction (Mitchell et al., 2022).

Using natural composites in drones can positively impact sustainability and the circular economy. Composite materials play a crucial role in reducing the weight of aerospace

materials, which in turn helps to reduce fuel, energy, and emissions (Parveez et al., 2022). Replacing traditional materials with composites can make drone airframes lighter and more efficient, reducing waste and optimising resources in the circular economy (Mitchell et al., 2022). However, there are challenges related to using composites in drones, such as manufacturing processes and difficulties with recycling, as most drone airframes are not currently designed with disassembly, recovery, or reuse of materials in mind. Despite these challenges, natural composites are gaining increasing demand for various applications and can be an environmentally friendly alternative to synthetic composites, aligning with the principles of the circular economy. Figure 2 shows some drone applications developed due to the advancement of drone materials.

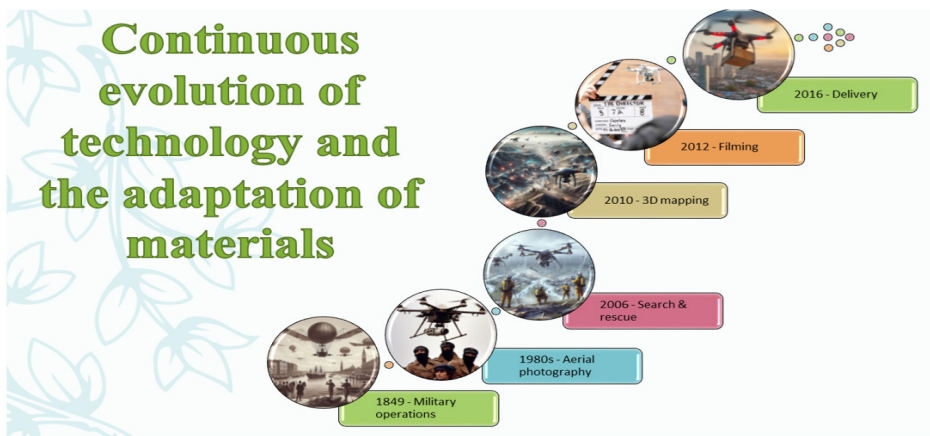


Figure 2. Innovations of drone applications due to continuous adaptation of materials

Source: Authors' work, all images are AI-generated from <https://www.bing.com/images/create?FORM=GENILP>

Various factors influence the market demand for drone natural composites. The unmanned composites market is expected to reach a value of 3.8 USD billion by 2027, with a projected CAGR of 16.8%, and is currently led by carbon fibre-reinforced polymer composites (Reports and Data, 2020). Due to their stability and durability, the increasing demand for lightweight and electric conductive composites drives the market growth. Additionally, the requirement for composite materials for weight reduction is a significant factor driving the market for unmanned composites in European countries.

PLANT FIBER'S ROLE IN THE CIRCULARITY OF MATERIALS

The circularity of materials refers to maintaining the value of products, materials, and resources for as long as possible by returning them to the product cycle, thereby reducing waste and reliance on finite virgin resources (Kirchherr et al., 2023). In a linear economy, materials are taken from nature, used to make products, and disposed of, leading to waste

and resource depletion. In contrast, a circular economy promotes the continuous use of materials to reduce waste and environmental impact. It involves using materials through reuse, repair, remanufacturing, and recycling or safely returning biodegradable materials to the earth. The circular economy aims to eliminate waste and pollution, circulate products and materials at their highest value, and regenerate nature. It involves exploring alternative materials, revamping existing infrastructure, and improving material management to capture the economic value of resources before they become waste (Jacobs et al., 2022). The circularity of materials is measured using indicators such as the Material Circularity Indicator (MCI) and the circularity rate, which represents the share of material resources used that came from recycled waste materials, thus reducing the environmental impacts of extracting primary materials (Moraga et al., 2021). By shifting from a linear to a circular economy, the focus moves from extraction to circulation, contributing to sustainability and resource optimisation.

The circularity of materials is an essential concept in drone construction as it promotes the efficient use of resources and waste reduction. The circular economy offers a framework to improve the sustainability of drones by designing out waste and pollution in all phases, making more durable products to extend the use phase (Mitchell et al., 2022). Plant fibres, such as kenaf, hemp, and flax, offer sustainable and biodegradable alternatives to traditional materials, contributing to the principles of the circular economy. Plant fibres play a crucial role in the circularity of materials due to their renewable and biodegradable nature. There are various plant fibres abundantly available in Malaysia, such as oil palm, kenaf, and screw pine.

Oil palm fibre (OPF) is an excellent raw material for biocomposites due to its high cellulose content, making it suitable for various applications (Shinoj et al., 2011). The fibre is non-hazardous, biodegradable, and abundantly available in Malaysia. The potential of oil palm fibre has been explored in various studies, highlighting its significant mechanical properties and potential as a functional food ingredient. Fibre has shown promise as an excellent antioxidant and a source of dietary fibre, with potential health benefits such as managing and lowering the risk of diabetes, colon cancer, and heart disease. The use of oil palm fibre aligns with the principles of the circular economy, promoting the efficient use of resources and waste reduction.

Kenaf fibre is a natural fibre with various advantageous properties and high potential as a reinforcement in composite materials (Kiron, 2021). It is a non-woody annual plant with a short life cycle of 100–130 days, making it a highly sustainable fibre due to its rapid growth and replenishment ability. Kenaf cultivation and processing require minimal water, fertiliser, and pesticides, contributing to its sustainability. The fibre is obtained from the bast and core of the plant, with the bast constituting 40% of the plant and the core constituting 60%. The individual fibre cells are about 2–6 mm long and slender, with a thick cell wall.

The fibre is also known for its natural absorbency and fire-retardant abilities, making it a valuable raw material in the textile industry.

Screw pine fibre is a natural fibre that is extracted from the leaves of the screw pine (*Pandanus amaryllifolius*). The fibre is in the form of long fibres, which have higher mechanical properties than short fibres like rice husk (Abral et al., 2012). Screw pine fibre has been used as a reinforcement in polymer matrix composites. Some of its unique properties include high strength, light weight, biodegradability, and environmental friendliness. Additionally, screw pine fibre has been used as reinforcement material in composites, exhibiting promising mechanical properties when combined with polyester and vinyl ester resins. The fibre can be treated with aqueous NaOH solution to reduce its hydrophilic nature, making it compatible with hydrophobic materials (Naik et al., 2021). In addition to its industrial applications, screw pine fibre is also used in traditional Malaysian weaving, a thriving artisan activity in the country (Zainol et al., 2016). The art of weaving takes years, and sometimes decades, to learn and master and is considered a piece of Malaysian cultural heritage at risk of disappearing.

Additionally, plant fibres such as hemp, flax, and pineapple leaves have a long history of use in textiles, cordage, and, more recently, in technical applications in composite materials, contributing to the sustainability of these products (Elfaleh et al., 2023). The versatility of cellulosic fibres derived from plants like cotton, flax, and timber is being further explored to mitigate the need for virgin natural resources, emphasising circularity and agricultural waste in the global fibre industry (Lawson et al., 2022). Plant fibres play a significant role in promoting circularity by offering sustainable and biodegradable alternatives to traditional materials, contributing to the principles of the circular economy.

MATERIALS FOR DRONE STRUCTURE

The process of drone production involves several stages, which start with conceptualisation and design. In this stage, the purpose and specification of the drone will be defined by considering factors such as payload capacity, flight range, endurance, and intended applications. By determining these factors at an early stage, suitable materials can be selected to optimise the performance of the flying drone. Suitable materials were chosen to develop the drone's frame, body, and components based on weight, strength, durability, and cost. Common materials include carbon fibre, aluminium alloys, composite materials, and 3D-printed plastics. Next, the drone components such as frame, arms, propellers, landing gear, and electronics housings will be fabricated by using various manufacturing techniques such as CNC machining (for precise shaping of metal and plastic parts), injection moulding (for mass production of plastic components), and 3D printing (for rapid prototyping and customisation of complex geometries). In this stage, dimensional accuracy and surface finish must also be ensured to meet the design specifications. Once the components have

been fabricated, they will be assembled and integrated with the electronic components. The drone will then be tested and calibrated to verify the operation of all systems and components. This stage ensures the optimisation and stability of the drone's performance while flying. Finally, the drone will undergo flight tests in controlled environments to evaluate the drone's flight characteristics, stability, and control responsiveness. The flight test complexity will gradually increase, and its performance under various conditions, such as different weather conditions and payload configurations, will be assessed. Table 1 shows some of the recent research conducted on drone production.

Table 1
Recent research on drone development

Aim	Method	Application	References
Designing a compact ducted drone with a co-axial propeller	Computational model and FEA analysis of UAV made from various alloys, carbon fibre-reinforced composites and glass fibre-reinforced composites	High altitude surveillance	Jayakumar et al. (2024)
Optimising drone routes	Comparative analysis of computational simulation on a case study of the Beirut Port Explosion in Lebanon using a greedy constructive heuristic (GCH) and adaptive large neighbourhood search (ALNS)	Post-disaster management	Almeida Coco et al. (2024)
Developing thermoformed composites by TiO ₂ modified aramid fibre reinforcement	Experimental study of the proposed material through physico-mechanical testing	Encapsulation of electronic parts	Pelin et al. (2024)
Design and modelling of a versatile, reconfigurable multi-rotor UAV	Computational validation of PULSAR efficacy on UAV	Forest and wildfire management	Perikleous et al. (2024)
Designing a hexacopter for a 3 kg payload	Computational simulation and analysis were used to perform a structural analysis of the UAV design using different materials	Lightweight drone	Raut et al. (2024)

Out of all the production stages, the material selection stage directly impacts the drone's performance and durability the most. The choice of materials directly impacts the weight and strength of the drone. Lightweight materials, such as carbon fibre or high-strength polymers, help reduce overall weight, which is critical for achieving longer flight times, higher payloads, and better manoeuvrability. At the same time, the materials must provide sufficient strength and durability to withstand the stresses of flight and potential impacts. The aerodynamic properties of the materials used to construct the drone's frame, wings, and other components affect its flight performance. Smooth surfaces and streamlined shapes reduce drag and improve efficiency, allowing the drone to fly faster and consume less power. Drones are also often used in demanding environments and are subjected to harsh conditions, including high winds, temperature variations, and mechanical stresses. Choosing materials with excellent durability and resistance to wear, corrosion, and fatigue ensures that the drone can withstand prolonged use without compromising performance or safety. Material selection also influences the cost of production, which is a significant consideration for manufacturers and consumers alike. Balancing performance requirements with material costs helps optimise the overall value proposition of the drone, ensuring that it delivers the desired capabilities at a competitive price point. As sustainability becomes increasingly important, choosing eco-friendly materials derived from renewable sources or recycled materials can help minimise the environmental footprint of drone production. It includes considerations such as resource depletion, energy consumption, and waste generation throughout the lifecycle of the drone.

Drone parts are made of lightweight materials such as plastic or carbon fibre to keep the drone's weight as low as possible (Anand & Mishra, 2022). Carbon fibre is one of the most commonly used materials in drone manufacturing because of its strength and lightness. Almost all drone structures are made from carbon fibre composites, in contrast to piloted aviation, where a large percentage of the structure is made from aluminium and titanium in addition to carbon fibre composites (Hexcel, 2023).

Natural fibres have been increasingly used as reinforcing materials for the construction of drones. These fibres offer an alternative to traditional synthetic materials like carbon fibre, providing potential benefits such as biodegradability and reduced environmental impact. For instance, there are examples of drones made from bio-degradable natural fibre composite materials, such as those using PLA and linen fibres as an alternative to oil-based solutions (Breznik, 2021). Using natural fibres in drone construction aligns with the growing interest in boosting drone performance and addressing environmental concerns. As drone technology advances, the selection of materials becomes more complex, requiring careful consideration of the performance of multiple components.

Conventional vs Bio-based Materials

Using conventional materials, such as carbon fibre, for drone construction offers lightweight, high-strength solutions at a relatively low cost. These materials are widely used in drone technologies due to their excellent strength-to-weight ratios and suitability for many drone components. However, as drone technology advances and the demand for more significant strength increases, light alloys like aluminium, magnesium, and titanium are also being used for their high strength and low weight, mainly in large-sized drones (Anand & Mishra, 2022; Parveez et al., 2022; Siengchin, 2023).

On the other hand, natural fibre composites are gaining attention as an alternative to conventional materials for drone construction. Research has shown that natural fibres, such as kenaf, PALF, eggshell, bagasse, and styrofoam, can be used as alternative composite materials for drone airframes, offering potential environmental benefits (Perdana et al., 2017). While conventional materials offer high performance and cost-effectiveness, using natural fibre composites aligns with the growing interest in sustainability and reducing the environmental impact of drone technology. However, there are still challenges in conducting sustainability assessments of drone materials, and more research is needed to address the end-of-life impact of composite materials, including the potential for bio-based composites to replace traditional materials.

The mechanical properties of drone materials vary depending on the material used. Some common materials used in drone manufacturing include composites, metals, and plastics. Synthetic composites like carbon fibre are known for their high strength and stiffness, making them popular for drone frames (Parveez et al., 2022). Metals like aluminium, steel, and titanium are also used in drone manufacturing, with aluminium being a common choice due to its malleability and ductility (Raj et al., 2021). Plastics like nylon are often used in cost-sensitive applications where rigidity is not as critical. However, none of these materials are environmentally friendly.

In contrast, natural fibre composites are gaining attention as an alternative to conventional materials for drone construction. The mechanical properties of these natural fibre composites, including their strength, stiffness, and durability, are essential considerations in their application for drone construction. Additionally, the mechanical design of drones involves considerations of strength, weight, aerodynamic resistance, and load-lifting ability, highlighting the significance of material properties in ensuring drones' structural integrity and performance. Table 2 compares various synthetic fibres, plastics, and natural fibres.

Most natural fibre composites' densities are comparable to plastics (Table 2). However, the water absorption of natural fibre composites is higher than that of synthetic composites and plastics. Other than that, natural composites' tensile strength and tensile modulus are comparable to those of synthetic composites and plastics, which shows that using natural

Table 2
Comparison of different material's physical, mechanical and thermal properties

Fibre	Matrix	Density (g/cm ³)	Water absorption (%)	Tensile strength (MPa)	Young's modulus (GPa)	References
Carbon	Epoxy	1.15–2.25	0.0500–3.90	0.917–3790	2.62–520.00	MatWeb (2023b)
Aramid	Epoxy	1.24	-	524.00	30.00	MatWeb (2023f)
-	ABS	0.882–3.50	0.0250–2.30	2.60–73.10	0.78–21.20	MatWeb (2023a)
-	Polycarbonate	1.01–1.51	0.0150–0.700	30.00–105.00	1.80–6.00	MatWeb (2023d)
-	Polyamide/Nylon	1.40–1.58	0.200–0.600	62.10–122.00	3.03–5.52	MatWeb (2023c)
-	Polypropylene	0.880–2.40	0.000–0.800	9.00–80.00	0.008–8.25	MatWeb (2023e)
Sugarcane bagasse	Epoxy	1.12–1.14	6.00–14.00	17.49–29.23	0.72–16.81	Abdullahi (2020); Dev et al. (2022); Prasad et al. (2020); Siddique et al. (2021)
Jute	Epoxy	1.5	0.84–40.00	393.00–773.00	0.10–0.30	Ferreira et al. (2016); Masoodi and Pillai (2012); Sujon et al. (2020)
Screwpine	Polyester	1.41	-	12.40–14.70	3.41–41.00	Abral et al. (2012); Gerald Arul Selvan et al. (2023)
Oil palm	Polyester	1.26	1.57	20.00–30.90	0.88–8.50	Rozi et al. (2021); Sahari and Maleque (2016)
PALF	Polyester	1.53	7.4–19.18	29.80–42.30	0.98–1.34	Agung et al. (2018); Siregar et al. (2014)
Kenaf	Polyester	1.13	9.46	21.44–76.56	2.42–3.18	Fajrin et al. (2022); Yahaya et al. (2016)

composites to construct drones is possible. However, the problem of high water absorption needs to be addressed before it can fully replace conventional materials. Several studies have addressed this problem by enhancing water resistivity through fibre treatment. Table 3 shows the study of treated plant fibres and the processing method used.

Table 3
Previous research conducted for water resistivity enhancement

Fibres	Chemicals	Processing method	Results	References
Sisal	Sodium bicarbonate	Alkaline treatment and barrier coating	The mechanical strength increased, and the water absorption capacity was reduced by 30%	Sahu and Gupta (2020)
Hemp	Sodium hydroxide and 3-Aminopropyl-triethoxy silane solution	Alkaline and hydrophobic treatment	Fibre treated with both alkali and silane has the lowest water absorption rate and high mechanical strength	Alao et al. (2021)
Coir	Sodium hydroxide	Alkaline treatment	Treated fibre has a lower water absorption rate compared to untreated fibre	Yew et al. (2019)
Straw	double- $[\gamma$ - (triethoxysilicon) propyl] tetrasulfide, deionised water, and lipase	Silane, hydrothermal, and lipase treatment	Compatibility with resin increased and improved the mechanical properties	Zhou et al. (2022)
Pine, eucalyptus, and sugarcane bagasse	sodium hydroxide	Alkaline treatment and corona discharge	Corona discharge treatment decreased water absorption of composites and enhanced the mechanical properties of the composites	Mesquita et al. (2017)

From Table 3, most of the fibres treated chemically have an increase in water resistivity. Chemical treatments can make fibres more hydrophobic, meaning they repel water. It is often achieved by coating the fibres with hydrophobic substances such as silicone or fluoropolymers. When water comes into contact with hydrophobic fibres, it forms droplets that roll off rather than being absorbed. Chemical treatments will also introduce cross-links between polymer chains in the fibres. Cross-linking increases the stability and strength of the fibres, making them less likely to swell or absorb water. A barrier will also be created, preventing water from penetrating the fibre structure. It can involve attaching functional groups to the fibre surface that repel water molecules.

In summary, the mechanical properties of drone materials, including strength, stiffness, durability, and weight, play a critical role in selecting materials for drone construction, whether conventional materials or natural fibre composites. These properties directly impact drones' performance, safety, and efficiency in various applications.

Advantages of Drone Materials

Drone materials are mainly made from synthetic composites or plastics, with the current addition of natural composites. These three primary materials offer several advantages, some of which are similar.

The advantages of synthetic composite materials in drones are significant and contribute to their overall performance and efficiency. Synthetic composite materials, such as carbon fibre composites, offer high strength-to-weight ratios, providing structural performance with low mass (Harussani et al., 2022). It reduces the drone's overall weight, improving flight efficiency and endurance. Synthetic composite materials exhibit good corrosion resistance, ensuring the durability and reliability of drone structures, especially in various environmental conditions (Maiti et al., 2022). Synthetic composites can be tuned to absorb specific electromagnetic frequencies and pass other frequencies, contributing to drones' efficient and reliable communication systems (Sahoo et al., 2023). These materials also allow for greater design flexibility, innovation, and the integration of components into the drone structure, leading to improved overall performance (Mishra et al., 2022). Finally, synthetic composite materials benefit from various manufacturing technologies, such as tooling, automated manufacturing, direct processing, and additive manufacturing, which can significantly reduce product development and manufacturing costs.

Plastic materials used in drones also share common benefits with synthetic composites. Drones made from plastics offer several advantages, including lightweight construction, cost-effectiveness, adaptability to complex designs, and resistance to UV rays (Habib, 2023). Engineering plastics such as PEEK and Ultem provide acceptable rigidity at lower weights, enabling the creation of more complex aerodynamic shapes with higher efficiency (Jonckers et al., 2022). Additionally, plastic materials are weatherable, neutrally buoyant,

optically clear, durable, and suitable for marine environments, making them ideal for various drone applications, including marine remotely operated vehicles (ROVs) (Curbell Plastics, 2023). Furthermore, plastic materials are integral in the fight against ocean plastic, as AI-powered drones equipped with plastic recognition algorithms are being used to combat plastic pollution in oceans (Harris, 2020). The plastic's weight reduction also improves flight efficiency and endurance. Plastics can increase the durability and reliability of drones by reducing the risk of corrosion, fatigue, or damage. It is crucial for the overall performance and longevity of drone components.

The advantages of natural composite materials in drones are not as widely discussed as those of synthetic composites. However, natural composite materials, such as those derived from plant fibres or other renewable sources, offer potential advantages for drone applications. Natural composite materials can provide lightweight structures, improving flight efficiency and endurance (Parveez et al., 2022). The combination of natural composites and 3D printing offers several advantages for drone design and development, such as weight reduction, durability, reliability, innovation, and the integration of components. Since these materials are derived from renewable sources, they offer potential environmental benefits compared to traditional composites. Some natural composite materials exhibit good corrosion resistance, which is essential for drone operation in various environmental conditions (Azman et al., 2021). Furthermore, depending on the specific properties of the natural fibres used, these composites may offer good impact resistance, contributing to the durability of drone components (Ahmed et al., 2021). Finally, natural composite materials may offer flexibility and potential advantages for aerodynamic performance, contributing to overall flight efficiency.

Disadvantages of Drone Materials

Synthetic fibre composites have several disadvantages, including low hydrophilicity, which affects their processing during wet treatments (Vigneswaran et al., 2014). Additionally, some synthetic fibres are not environmentally friendly and can damage the environment due to exposure to harsh processes. Moreover, certain synthetic fibres can be prone to electrostatic charging when rubbed against other materials and may not always be friendly to the skin (Tahir et al., 2022). Lastly, compared to other fibres, the high cost of some synthetic fibres, such as boron fibre, is also a disadvantage (Rajak et al., 2022).

As for plastics, there are several disadvantages. One of them is that plastic components may have lower durability than composite materials like carbon fibre, making them more susceptible to damage in a crash. While plastics are lightweight, they may not offer the same level of strength as other materials like carbon fibre, which can affect the overall structural integrity of the drone (Shortland, 2023). Plastics are less environmentally friendly than natural fibre composites.

Meanwhile, one of the disadvantages of natural fibre composites is that natural fibres have relatively high moisture absorption, which can lead to chemical degradation of the fibres and weak fibre-matrix interfacial adhesion (Mohammed et al., 2023). Natural fibres also have poor compatibility with many polymer matrices, which can affect the overall performance and durability of the composite material (Elfaleh et al., 2023). In addition, with the current technology, natural fibre composites can be relatively expensive compared to plastics, impacting the overall cost-effectiveness of drone construction. While natural fibre composites are lightweight and have a high strength-to-weight ratio, they may have issues with technical properties such as impact strength.

CONCLUSION

From this review, several points can be concluded. First and foremost, it was found from the review that the drone's material keeps on evolving. Not only is the material getting lighter, but it is also becoming stronger, sustainable, and cost-effective. As the material advances, the potential of drone application widens in various industries. Now, with the addition of plant fibres, the circularity of materials can also be ensured in various stages of the drone construction. Current research shows that Kenaf and PALF can be used as alternative materials for drone construction. This addition makes procuring raw materials needed for construction, repair, and maintenance more manageable.

Furthermore, using plant fibres as drone material creates a circular economy within the country. The review also found some similarities between conventional drones and bio-based drones. These similarities are their lightweight and design flexibility. Despite this, drones made from conventional and bio-based materials also show their differences in eco-friendliness. Natural fibre allows the drone to biodegrade once it is disposed of, and it does not need to use any chemicals to obtain the raw materials.

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Response Surface Methodology for Optimisation of Parameter on Water Absorption of Natural Fibre Hybrid Composite in Boat Construction

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ABSTRACT

Today, the utilisation of natural or green fibre has supplanted engineered fibre as fibreglass has become a pattern in composite boat producing and other gears because of its lightweight, excellent relative mechanical properties, and more significant elements. For example, it is eco-accommodating, has manageable materials, and has lower costs compared to fibreglass. The utilisation of fibreglass is costly and has a high effect on the natural biological system. It also gets over the area of ecological contamination, word-related well-being, and security concerns. This study used the Woven Kenaf/fibreglass as reinforcement and polyester as matrix to fabricate hybrid composite coupons. Response Surface Methodology (RSM) of Box-Behnken Design (BBD) was applied to optimise fibre contents (35 to 75 wt%) and water absorption performance for the development of Woven Kenaf/fibreglass to polyester hybrid composite material based on the parameters. The BBD revealed that 45% Woven

Kenaf/fibreglass to polyester performed the optimum fibre content and water absorption. Analyses of variance (ANOVA) showed that the model satisfactorily correlated the parameters. After immersion, 45% Woven Kenaf/fibreglass to polyester composite also gained 9.48% weight.

Keywords: Box-Behnken design, hybrid composite, kenaf fibre, response surface methodology, water absorption

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INTRODUCTION

Natural fibre polymer composites are strikingly sought after among an assortment of applications from aviation, auto, development, and sea-to-home devices because of the benefits of being eco-accommodating and manageable materials. Natural fibres can be obtained from animals and plants (Jawaid & Khalil, 2011; Kamarudin et al., 2022; Syduzzaman et al., 2020) and can furnish a material with a high-solidarity-to-weight proportion and supplant the utilisation of synthetic fibres (Kabir et al., 2007; Karimah et al., 2021). However, natural fibres have disadvantages such as low modulus strength, impact properties, moisture resistance, limited durability, and poor fire resistance. This shortcoming is overcome using the hybridisation of natural fibre with synthetic fibre, i.e., fibreglass that delivers hybrid composites with the best properties of the elements, bringing about ideal, unrivalled, however savvy composites (Karimah et al., 2021; Radzi et al., 2019). Processing characteristics for each type of natural fibre and their parameter setting are different. Ideal boundary settings for elite execution composites utilising regular fibre lattice blends are expected to deal with strategies. Different factors can impact the properties of normal fibre polymer composites. Huge fibre stacking is typically expected to accomplish great natural fibre polymer composite properties and cycle boundaries that impact composites' properties and surface highlights. Accordingly, the appropriate cycle strategies and boundaries should be painstakingly distinguished to acquire the best elements of composites (Peças et al., 2018).

ML was used to predict trends, classification, and target detection, such as speech recognition, face recognition, house price prediction, and many more. Response Surface Methodology (RSM) is one of the Design of Experiment (DOE) techniques available in ML for optimisation in conditions of several potential input variables affecting a process or product's performance or characteristic (Cai et al., 2020; Iliyasu et al., 2022). Additionally, when contrasted with other DOEs, it can evaluate the impacts of various elements and their communications on at least one reaction factor. RSM is one of the most involved trial plans for advancement. RSM permitted assessing the impacts of various elements and their communications on at least one reaction factor. It is likewise a valuable technique, supplanting traditional strategy, as does the writing or experimentation to decide for proposed quantities of exploration. RSM was carried out to foster the measurable models to assess the impact of boundaries of any cycle as well as to upgrade the circumstances for helpful reactions as utilised to build the worth of the information assortment in combustibility and thermal properties of plant fibre half-breed composite (Gholamian et al., 2017; Pugliese et al., 2021). RSM of Central Composite Design (CCD) was also used to optimise biocomposites based on poly (lactic acid) and durian peel cellulose and to obtain the highest tensile strength and impact strength performance. RSM effectively explores how parameters like density, fibre weight, and polyester weight impact the water absorption of woven kenaf/fibreglass composites (Prabhu et al., 2022).

Using sophisticated statistical models, RSM predicts how much water these composites absorb. Analysis of variance (ANOVA) helps analyse the data thoroughly (Saaidia et al., 2022). In essence, RSM helps find the best ways to reduce water absorption in these composite materials. Furthermore, RSM's predictive design can specify composite material parameters without extensive experimentation, saving valuable time and resources (Makhlouf et al., 2022). Consequently, this study is to decide the impact of info variable of boundaries (fibre thickness, fibre support weight, and polyester pitch weight) on the water retention of polyester built-up woven kenaf hybrid with fibreglass composite material, as well as the enhancement of these boundaries to deliver the ideal fibre content to create hybrid composite material utilising the RSM.

MATERIALS AND METHODS

Response Surface Methodology with Box-Behnken Design

This study used RSM based on the Box-Behnken Design (BBD) for the experimental investigation. There was a total of 15 experimental runs. The parameters were configured as shown in Table 1. Based on preliminary research, these parameter settings were considered. The tool for data statistical analysis was Design Expert software version 13 (US, Stat-Ease Inc.).

Table 1
Parameters and levels

	Parameter Unit	Minimum	Maximum	Coded Low	Coded High
A	Density (g/mm ³)	1.0000	1.03	-1 ↔ 1.000	+1 ↔ 1.03
B	Weight of fibre (%)	15	75	-1 ↔ 15	+1 ↔ 75
C	Weight of polyester (%)	15	85	-1 ↔ 15	+1 ↔ 85

Fabrication of Composite Coupon

Fabrication of Woven Kenaf/fibreglass hybrid composite materials was conducted using the hand lay-up technique. The composite coupons were prepared using different fibre content (weight percentage) compositions of Woven Kenaf/fibreglass-to-polyester, such as 15, 45, 60, and 75 wt% (Table 3). Table 2 depicts the Composite Coupon Designation of each weight percentage material. The polyester and catalyst were blended completely to ensure the combination was well-blended before filling the mould.

The composite coupons were prepared with dimensions of 230 mm (length) x 5 mm (thickness) x 160 mm (width). The mould surface was cleaned and waxed to make the composite easily removed from the mould. After completing the hand lay-up process, the top surface of the composite was rolled with a roller to remove air or bubbles. The mould with the composite was left at room temperature for 24 hours.

Table 2

Density of materials composite coupon designation of each weight percentage materials

Percentage of materials (wt%)	Designation of each weight percentage materials				
	0 wt% (Control sample)	15 wt%	45 wt%	60 wt%	75 wt%
Polyester + catalyst	90	75	45	30	15
Natural fibres (woven Kenaf)	0	15	45	60	75
Fibreglass	10	10	10	10	10

Water Absorption Test

Composite coupons were submerged in tap water in a room with 40% relative humidity at 30°C temperature. The specimens were removed from the water, cleaned, dried to eliminate the surface dampness, and afterwards weighed to obtain the weight gain because of the water ingestion. The mass of the dried samples, M_o , was measured after oven drying at 100°C for 10 minutes and left at room temperature for 24 hours. The water ingestion test was performed adhering to the ASTM D570 standard. Composite samples are considered equilibrium when their daily weight gain is less than 0.01%. Moisture content percentage, $\Delta M(t)$, was calculated using Equation 1:

$$\Delta M(t) = \frac{M_t - M_o}{M_o} \times 100 \quad (1)$$

where M_o is the weight of the coupons before immersion and M_t represents the weight of the coupon after immersion at a specific time. Assuming the absorption process was linear at the early stage of immersion, times were taken at the beginning, so the weight change is expected to vary linearly with the square root of time. The percentage of moisture absorption was plotted against the square root of time (\sqrt{t} , hours).

Materials

Treated Woven Kenaf/fibreglass was used as reinforcement, and polyester was used as the matrix to fabricate Woven Kenaf/fibreglass to polyester composite materials. The Woven Kenaf was treated by soaking the fibre in 5% Sodium Hydroxide (NaOH) for a day and then drying at 80°C for 24 hours. Woven Kenaf was provided by the Institute of Tropical Forestry and Forest Product (INTROP), UPM, while MSET Inflatable Composite Corporation Sdn supplied fibreglass (CSM 225) and unsaturated polyester resin Reversol P-9509 Sdn. Bhd.

RESULTS AND DISCUSSION

Results obtained from the experimental runs are tabulated in Table 4. The water absorption ranged from 15 to 75 wt% fibre content, while water absorption ranged between 1.39 and 10.89 g.

Table 4
Results of experimental runs

Factor 1	Factor 2	Factor 3	Response 1
A: Density g/cm ³	B: Weight of fibre (%)	C: Weight of polyester (%)	Water absorption (%)
1	45	85	1.39
1.03	45	15	3
1.015	45	50	4.43
1	75	50	6.29
1.03	45	85	6.11
1.015	45	50	6.64
1	15	50	1.78
1.015	45	50	7.65
1.015	15	15	1.78
1	45	15	8.14
1.015	75	15	10.2
1.015	15	85	2.08
1.03	75	50	10.63
1.015	75	85	10.89
1.03	15	50	2.97

Analysis of Variance (ANOVA)

Table 5 contains an analysis of variance (ANOVA) data for water absorption. The ANOVA results were applied to provide additional insights.

The ANOVA results in Table 4 show that the model was viewed as huge, with a P -value of 0.0028 (under 0.05) and an F -value of 9.49. The huge model terms were An and A2. Table 6 listed the coefficient R^2 of 0.5504 and predicted R^2 of 0.7843, which demonstrated that the model satisfactorily addressed the genuine connection between the viable factors.

Table 6
Adjusted R^2 and Predicted R^2

Std. Dev.	0.3491	R^2	0.8768
Mean	2.25	Adjusted R^2	0.7843
C.V. %	15.50	Predicted R^2	0.5504
		Adeq Precision	8.1839

Optimisation of Fibre Parameters Using RSM

The response surface condition could be utilised to compute the most extreme and ideal qualities anticipated for water ingestion from the trial. By and large, expanding the heaviness of the fibre support works on many of the mechanical properties. As per a few investigations, increasing the weight of the fibre support will, in general, work on the mechanical properties; notwithstanding, past a specific weight, one of a kind to various filaments, the physiochemical properties will often decline (Jariwala & Jain, 2019). Hence, the impacts of fibre support weight, polyester weight, and fibre thickness of the composite were researched in this review to find the ideal blend for polyester-based built-up woven Kenaf crossover with fibreglass composite material.

The impact of these three boundaries on the water assimilation can be approximated by following genuine coded condition Equation 2:

$$\text{Square root of } X \text{ (water absorption)} = 57.6159 + (-55.6075) * A + (-0.177987) * B + (-1.16926) * C + 0.201766 * AB + 1.14947 * AC + (-4.28092) * BC \quad (2)$$

Where X is the predicted response value (water absorption).

Table 6 shows the coefficient of determination (R^2) and the changed coefficient of determination (changed R^2) water ingestion, demonstrating whether a relapse model is satisfactory. The meaning of the coefficients of the quadratic polynomial conditions was resolved by utilising ANOVA. A huge F -value and a small P -value show that each term has a massive impact (Peças et al., 2018; Penjumras et al., 2015). The most extreme and ideal qualities anticipated for water retention with the collaboration of the boundaries are introduced in 3D plots in

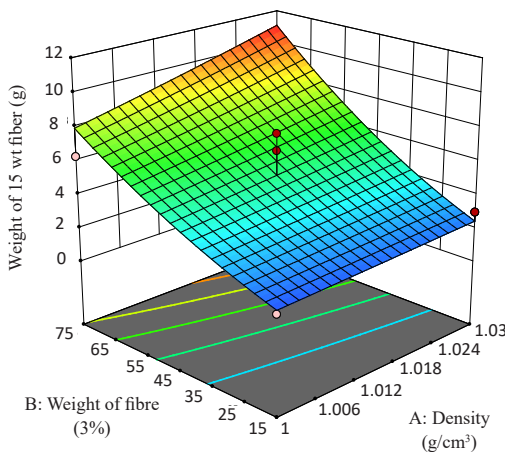


Figure 1. Response surface plots representing significant interaction on water absorption interaction with fibre reinforcement weight of 45 wt%

Confirmation Test

The confirmation test was directed to check the accuracy of forecasts and to affirm that there were no progressions and that the reaction values were near the anticipated values. The model conditions will give the greatest anticipated values to water retention and if others esteem as well as exploratory ideal qualities for amplifying the two reactions. The ideal boundary setting was at the thickness of 1.00 g/cm³, 45% of fibre support weight, and 45% of polyester acquired utilising point forecast from the mathematical streamlining accessible of

RSM. Under the ideal boundary setting conditions, the upsides of water retention from the affirmation run were acquired, as displayed in Table 7.

The weight of water absorbed increased with soaking time and stabilised after saturation, and the weight content percentage also influenced the moisture uptake. Basically, the chemical properties of natural fibres also affect water absorption (Ishak et al., 2012; Rashdi et al., 2010; Saxena et al., 2011). As Elfaleh et al. (2003) mentioned, applying natural plant fibres as reinforcement in polymer materials has limitations due to certain drawbacks, such as water absorption. Lignocellulose in plant fibres consists of hydroxyl groups, making them hydrophilic and unsuitable for use with hydrophobic thermoplastics such as polyester, resulting in poor moisture resistance. The hybridisation of natural fibres, particularly synthetic and Kenaf fibres, is an excellent method to improve the mechanical characteristics of the fabricated hybrid composite, as reported in many works (Suriani et al., 2021). Figure 2 illustrates the moisture uptake of Woven Kenaf/fibreglass-reinforced polyester composite materials.

Table 7
Results with optimal setting parameters of confirmation test

Density (g/cm ³)	Fibre weight (%)	Polyester resinweight (%)	Water absorption (%)
1.00	45	85	1.39

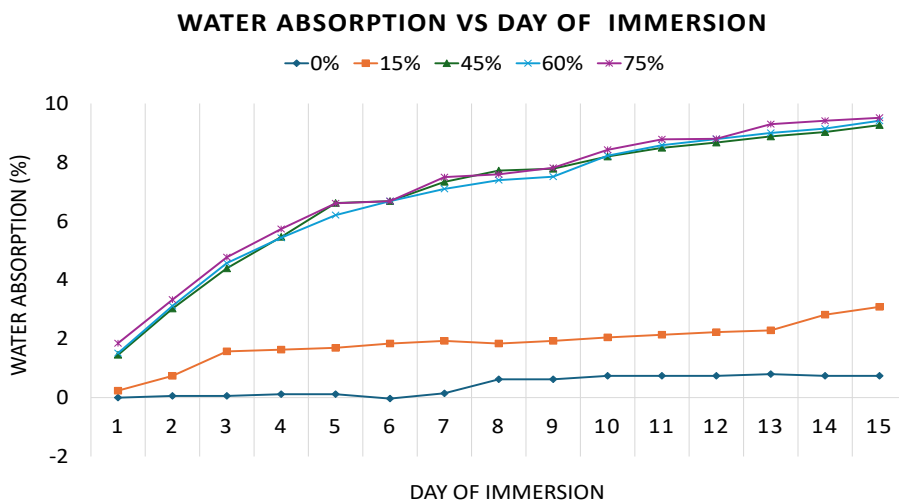


Figure 2. Water absorption results from experiments

The increase in weight content fibres in the specimen permitted more water diffusion into the interface via micro-cracks, and water absorption increased on day 15 (Hamdan et al., 2019; Verma et al., 2021). The 0% weight content sample exhibited the least moisture

absorption at 0.50% because there were no Kenaf fibres in the specimen—the most minimal moisture take-up Kenaf filaments displayed in 15 wt% of fibre content at 3.08 g. The properties of the 45% fibre content examples after 15 days of water immersion were noticed. The water retention uncovered that the examples had a direct ascent in water take-up, showing a 9.48% weight gain. The water assimilation for 60% fibre content was 9.39% on day 15, and for 75% fibre content was 9.54 g. The effect of fibre contents on water absorption of Woven Kenaf/fibreglass reinforced polyester composites was studied following immersion at room temperature for 15 days. Moisture uptake increased with fibre volume fraction increments due to increased voids and cellulose content. These results are fixed with the water absorption pattern of non-woven hemp fibre-reinforced unsaturated polyester composites reported by Dhakal et al. (2007). The water absorption pattern of these hybrid composites for 15 days at room temperature was to follow Fickian behaviour.

CONCLUSION

The impact of fibre support weight, the thickness of the composite, and the polyester gum weight boundaries on the water assimilation of woven Kenaf reinforced composite in boat development were researched through RSM. The fibre support weight-thickness of composite collaborations was found to fundamentally affect the water ingestion of the Kenaf woven crossbreed composite. 3D reaction surface plots were utilised to decide the ideal region in the assigned scope of the cycle, and 45 wt% was not set in stone as the ideal fibre content. The most noteworthy water ingestion was accomplished through the streamlined boundaries. In this review, the boundaries are essential for manufacturing Woven Kenaf/fibreglass reinforced composites, and all of these affect their water retention. Extra boundaries, as well as reactions, such as combustibility and warm properties, could be utilised for future review.

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Fabrication and Characterisation of Kenaf Fibre Reinforced Polyamide Biocomposites for Railway Sleeper Applications

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ABSTRACT

Railway passing traffic, speed, and load have significantly increased over the years, prompting industry stakeholders and researchers to seek an alternative sleeper material that can demonstrate its ability to potentially possess higher in-service bending resistance and be environmentally friendly and durable. To address these needs and due to environmental concerns, kenaf-reinforced polyamide has become of great importance. However, they could not be applied as railway track components because of the non-availability of their performance in this regard. In bridging this gap, this paper focused on fabricating and characterising six different formulations of treated kenaf fibre (TKF, 0–50% at 10% loading interval) reinforced polyamide biocomposites for railway sleeper applications. The result showed that the incorporation of TKF influenced the behaviour of the polyamide with respect to its water absorption, load-carrying capacity, and thermal stability.

The result further demonstrated that the load-bearing capacity peaked at TKF 40 wt.%, surpassing conventional wooden and concrete sleepers. However, its water absorption (64-days saturation) behaviour increased significantly between 11%–21% as TKF rose from 10–50 wt.%, as expected due to TKF hydrophilic characteristics. On the other hand, TKF thermal stability was hampered beyond approximately 220°C for

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all TKF percentages. Kenaf fibre-reinforced polyamide biocomposites have demonstrated their potential for railway sleeper applications as their load-bearing capacity exceeded the minimum recommended AREMA specifications. Despite the milestone achieved, water absorption of kenaf fibre remained high. The development of sustainable and effective materials to meet the changing needs of contemporary railway infrastructure is greatly aided by the insights gained from this study.

Keywords: Flexural strength, polyamide biocomposites, railway composite sleeper, thermogravimetric analysis, treated kenaf fibre, water absorption

INTRODUCTION

The rapid escalation in railway traffic, speed, and load over recent years has paralleled a growing demand for track components that offer enhanced durability, environmental sustainability, and improved service performance. In response to these evolving requirements and driven by increasing environmental concerns, kenaf-reinforced polyamide composites have emerged as a highly significant material. As they are meant to endure a variety of deterioration causes, distribute and reduce dynamic loads throughout the ballast layer, and maintain precise track gauges, sleepers are among the most important components of the railway track system (Askarinejad et al., 2018; Casado et al., 2016; Manalo & Aravinthan, 2012; Zeng et al., 2020; Zhang et al., 2022; Zhao et al., 2007). Wood, steel, and concrete are common traditional materials used to make railway sleepers. Their bending strengths for adequate performance are 70-110 MPa (Siahkouhi et al., 2022a), 120 MPa (Jing et al., 2022) and 110 MPa (Siahkouhi et al., 2022b) for timber, steel, and concrete, respectively. Increasing train speed, weight, and passing traffic have resulted in several mechanical and durability difficulties documented in the literature (Jokubaitis et al., 2020; Lee et al., 2016; Yu et al., 2021).

Railway industry stakeholders' and researchers' quest for composite material has increased due to the existence and rise of these problems. The quest for improved structural properties, reduced cost of maintenance, and increased lifetime of the sleeper have become a subject of concern to researchers and railway industry stakeholders (Axion, 2022; Integrated Recycling Pty Ltd, 2022; KLP Main Track Sleeper-Sustainable Plastic Railway Solutions, 2014; Sicut Enterprises Ltd., 2022; TieTek, 2022; TVEMA, 2022). Because of the environmental benefits, natural fibre composite materials should be preferred when developing and manufacturing railway composite sleepers.

Hitherto, the application of natural fibres as one of the constituents' materials in biocomposites and as a substitute for synthetic materials have been considered by global industries because of their advantages of being renewable and possession of marketing appeal in the composite manufacturing industries as well as their commensurable

physical and mechanical properties compared to synthetic fibres. Most importantly, the unique properties of kenaf fibre-reinforced polymer composites, such as low density, low cost, recyclability, biodegradability, and sustainability in terms of resources as well as being abundantly available, make kenaf fibre a choice material. Additionally, the simple preparation process requires little and simple equipment. Moreover, harmful gases that pollute the environment are not generated during preparation. Thus, the use of fibres obtained from agricultural crop residues as the main source of the composite's product can mitigate open burning that can lead to air pollution, thereby help in protecting the environment (Bujjibabu et al., 2018; Mochane et al., 2019; Nurazzi et al., 2021; Shireesha et al., 2019). Synthetic (glass) fibre-based composites are now considered for replacement by natural fibre-based composites development, especially for structural applications by some relevant major industries such as civil construction, aerospace, and automobile industries. These composite-based natural fibre materials can produce automobile body panels, interiors, storage devices, buildings, and industrial panels (Karthi et al., 2020; Syduzzaman et al., 2020).

The main hindrance in natural fibre composite applications is its deterioration tendencies in environments with high moisture and lack adhesion between the fibre-matrix interface, especially when proper fibre surface modification is lacking. Moisture intrusion into composite materials has a detrimental effect on the fibre-matrix interfacial adhesion, hydrolyses, and sporadically introduced microcracks in the matrix (Nurazzi et al., 2021). Water can be discovered in polymers in a variety of forms, including bound water, which has strong contact with the matrix molecule and free water existing in vessels and micro gaps within the polymer.

The application of composites in civil engineering has surged due to their advantageous properties, such as high strength-to-weight ratio and resistance to corrosion and wear. Historically, synthetic fibres have dominated this field (Hwang et al., 2019; Kaewunruen, 2015; Koller, 2015; Takai et al., 2006). However, shifting towards natural fibres is essential for sustainable and efficient composites. This study focuses on evaluating the feasibility of kenaf fibre-reinforced polyamide biocomposites for railway sleeper applications. It aims to experimentally determine the optimal fibre loading to achieve the required load-bearing capacity and to assess the effects of water absorption and thermal stability on the composite.

MATERIALS AND METHODS

Materials

The raw materials used in this study contain only two components: a matrix component made from Polyamide six (PA6) and a grade A plant-based fibre long reinforcement from kenaf fibre. The quality of the raw materials is highly dependent on their production source and processing. PA6 was supplied by the Shanghai King Chemicals Co. Ltd., having a

density of 1.13 g cm^{-3} as the matrix, and kenaf fibre was purchased from Lembaga Kenaf dan Tembakau Negara (LKTN), Kelantan, Malaysia. These companies follow standard procedures in material production. The grade A long kenaf fibre was processed using a mesh size of 2 mm at the Fibre and Biocomposite Centre (FIDEC), Malaysian Timber Industry Board (MTIB). The surface of the long kenaf fibre was treated and then dried in the industrial oven for 24 h at 60°C prior to processing at FIDEC. An optimum alkaline solution (6%) of sodium hydroxide (NaOH) (Abdullah et al., 2022) was used for the kenaf fibre surface modification. Hence, a treated kenaf fibre (TKF), 2 mm in length, was used throughout this study.

Composites Preparation

Mixture Design

In the production of recycled high-density polyethylene (RHDPE) and filler composites, research consistently reports the use of filler proportions by weight ranging from 10% to 50% (Almeida et al., 2021; Atikler et al., 2006; Bartczak et al., 1999; Esmaeili et al., 2023; Ratanawilai & Taneerat, 2018; K. Yang et al., 2007). This weight range is widely adopted to optimise the material properties and performance of RHDPE/filler composites. A notable bonding behaviour was observed at 40 wt% in producing wood flour-reinforced hydrophilic polymer composites (Lu & Forcada, 2006). Similarly, Agnantopoulou et al. (2023) reported positive results with hydrophilic thermoplastic starch reinforced with 50 wt.% wood flour. These studies, among others, have demonstrated the effectiveness of using wood flour and fillers in composite production. However, this research focuses on using short-treated kenaf fibres (2 mm) as reinforcement in biocomposites. It aims to investigate the performance and behaviour of the composite when these fibres are introduced. This study investigated six PA6/TKF biocomposites with varying TKF loadings (10, 20, 30, 40 and 50 wt.%) alongside a control comprising neat PA6 without any fibre, as shown in Table 1.

Table 1
Formulation of TKF/Polyamide six biocomposite

Sample ref.	PA6 (wt. %)	TKF (wt. %)
PA6	100	0
90PA6/10TKF	90	10
80PA6/20TKF	80	20
70PA6/30TKF	70	30
60PA6/40TKF	60	40
50PA6/50TKF	50	50

Fabrication of Kenaf Fibre Reinforced Polyamide 6 Biocomposites

Treated kenaf fibre and PA6 particles were oven-dried for 24 h at 110°C. The TKF/PA6 biocomposites were prepared in two stages: internal mixing with a brabender machine and compression moulding based on the earlier formulation in Table 1. Prior to processing, the components were manually dry-mixed for 10 minutes before being fed into a Brabender feeder. The mixture was then processed using a counter-rotating twin screw extruder at a speed of 50 rpm, with the temperature profile in zones 1–3 set to 230°C. The constituents were mixed for 10 minutes, after which the blend was manually extracted. The mixture instantly solidified at room temperature and cooled within 3 to 5 minutes. The resulting biocomposite pellets were then compression-moulded into boards (150 mm × 150 mm × 3.2 mm) using a hydraulic hot thermosetting press set to 220°C and 400 kN pressure for 5 minutes. The fabrication and characterisation sequence are shown in Figure 1.

Test specimens of specific sizes were cut from the biocomposite boards (Table 2). Figure 1 (a) and (b) show a view of the typical specimen. Sixty test specimens were prepared based on the fibre loadings.

Table 2
Number of specimens and standards used for characterization

Test name/Type	Test standard	No. of specimen	Dimension (mm)		
			Thickness	Length	Width
Water absorption	ISO 62:2008	30	3.2	50	50
Flexural strength	ASTM D790-10	30	3.2	120	12.7
TGA	ASTM E1131-20	36 mg	6 mg x 6 nr.		

CHARACTERISATION

Water Absorption

The water absorption test was conducted according to ISO 62 (2008). Five replicates of samples with dimensions of 50 x 50 x 3.2 mm were dried in an oven maintained at 50 ± 2°C for at least 24 h, placed in a desiccator, and allowed to cool to room temperature before weighing them to the nearest 0.1 mg. The process was repeated until the specimen mass became constant (mass, m_1). The specimens were then placed in a container filled with distilled water. After immersion in the water for 24 h, the specimen was removed from the water one after another, their surface water was dried off, and the dried specimen was weighed within one minute of removing from the water (mass, m_2). The water content at saturation was measured by re-immersing the test specimens and reweighing them at time intervals 24, 48, 96, 192, 384, 768, and 1536 h. At each time interval, the sample was removed, its surface water cleaned off and reweighed within one minute of removal from the water (mass, $m_2/24_h$).

$$C = (m_2 - m_1)/m_1 \times 100\% \tag{1}$$

Percentage by mass of water absorbed,

Where m_1 is the mass of the test specimen in milligrams (mg), and m_2 is the mass of the test specimen in milligrams (mg) after immersion.

Thermal Gravimetric Analysis (TGA)

The samples were tested using thermogravimetric analysis (TGA) at a scanning rate of 10°C/min on a Mettler Toledo TGA/DSC 1HT Stare System (Switzerland) between 30 and 600°C in accordance with ASTM E1131-20. TGA is a technique that monitors weight changes as a sample is heated at a consistent rate to evaluate the thermal stability of materials and their fraction of volatile components.

Flexural Strength Testing of TKF Reinforced Polyamide Biocomposites

The flexural test was conducted based on ASTM D790-10 standards using a universal tensile testing machine INSTRON model with a maximum load of 50 kN in a typical laboratory environment with 50% relative humidity and 23°C. Five replicate specimens accurately sized and cut out using a band saw were examined for flexural strength at a crosshead loading speed and the span-to-depth ratio of 1.36 mm/min and 16:1, respectively (Figure 1) (ASTM D790, 2010). The findings show the average results of tests run on all the biocomposite samples.

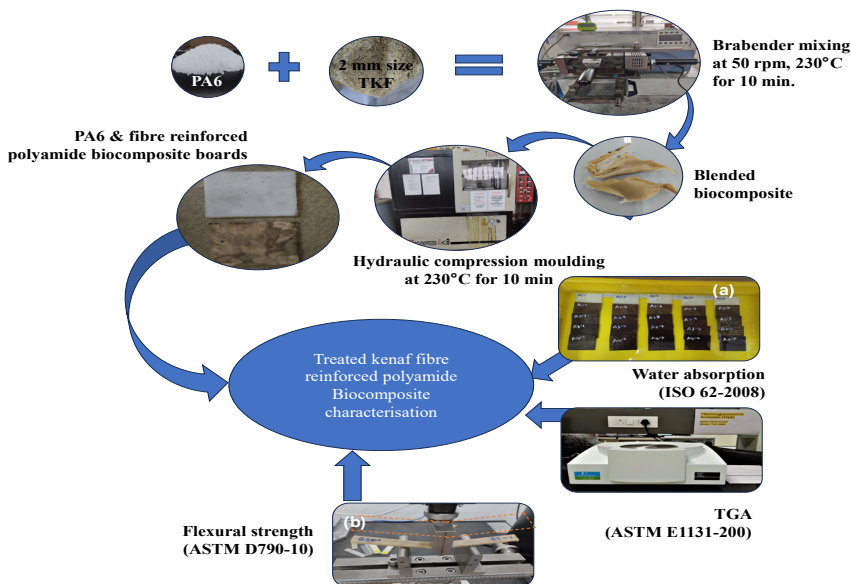


Figure 1. The sequence of treated kenaf fibre-reinforced polyamide biocomposites fabrication and characterisation: (a) water absorption and (b) flexural strength tests

RESULTS AND DISCUSSION

Water Absorption

Figure 2 illustrates the water absorption behaviour of treated kenaf fibre-reinforced polyamide biocomposites compared to neat polyamide six (PA6) as a control specimen. Over a submersion period of up to 64 days, the control specimen reached saturation at less than 10% water uptake within 32 days. Higher fibre loading (10–50%) in the composites resulted in increased water absorption, aligning with the hydrophilic nature of treated kenaf fibre (TKF) due to its hydroxyl (-OH) groups because of the presence of cellulose content, as indicated by Fourier Transform Infrared (FTIR) spectroscopy (Figure 3), (Maslinda et al., 2017; Rozali et al., 2017; Son et al., 2001; H.-S. Yang et al., 2016). The hydroxyl groups are the primary contributors to moisture ingress, enhancing the composites' susceptibility to water (Maslinda et al., 2017).

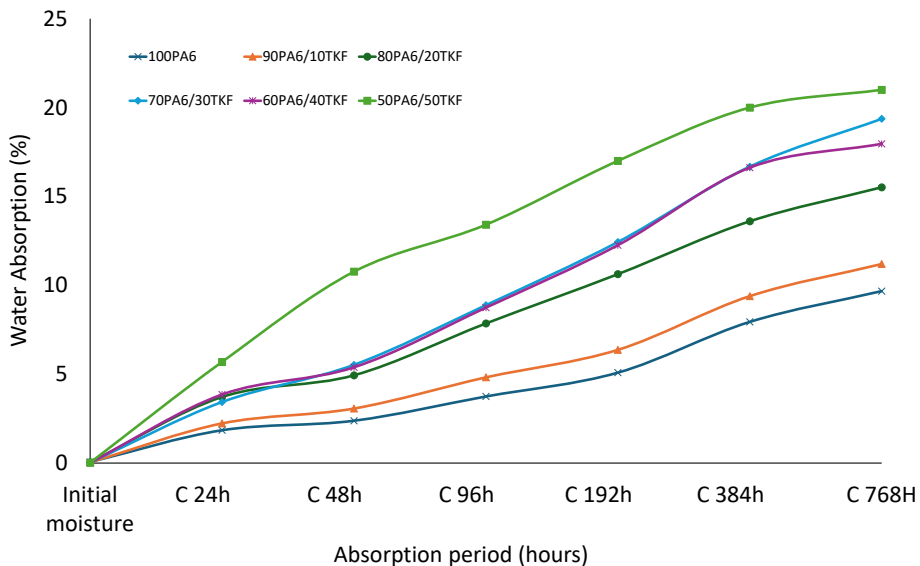


Figure 2. Rate of water absorption of PA6/TKF 0–50% (at 10% intervals) reinforced polyamide biocomposites at 64 days

Absorption levels ranged from 10% to 21%, with the highest reading observed in composites with 50 wt.% treated kenaf fibre (TKF). This elevated water uptake is likely due to the matrix's inadequate performance shielding the fibres from water exposure because of pores, as shown in Figure 4. The inability to effectively coat the fibre surfaces may result in the formation of microcracks (Figure 5), facilitating active and dominant moisture ingress in these regions. As a result, biocomposite panels with higher fibre loadings of up to 50 wt.% exhibited greater water absorption compared to those with lower fibre loadings of 10 to 40 wt.%.

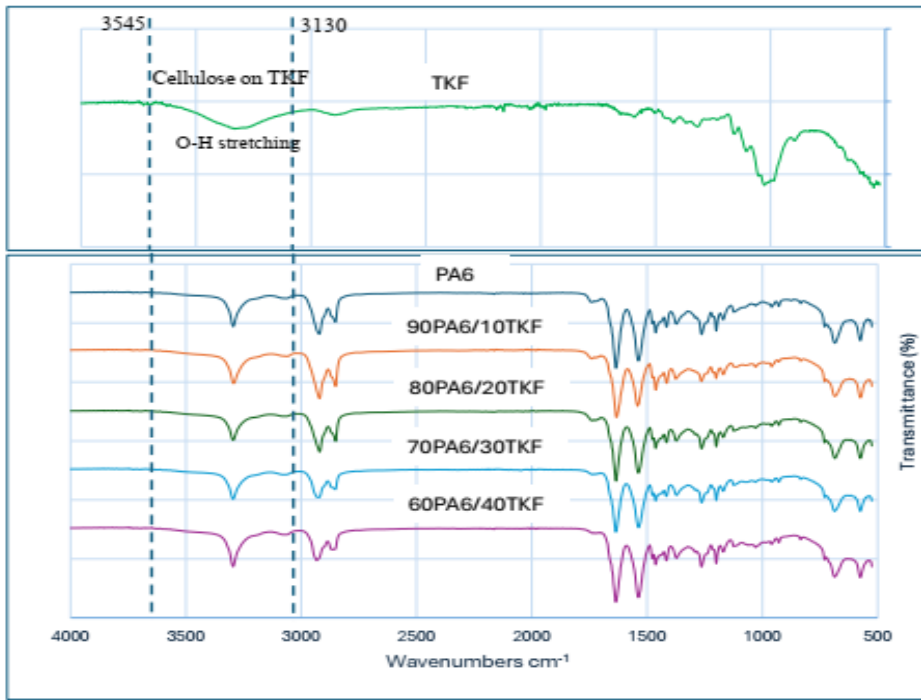


Figure 3. FTIR analysis showing the presence of cellulose on TKF

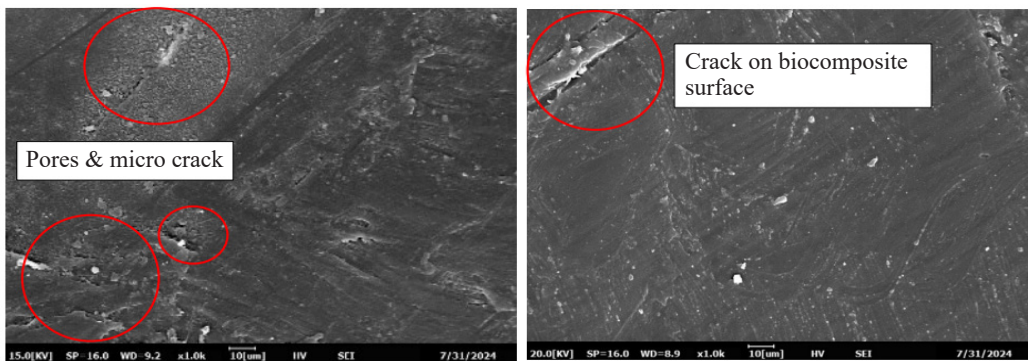


Figure 4. SEM images of PA6/TKF reinforced polyamide biocomposites

Thermogravimetric Analysis (TGA)

The thermal behaviour of the composites is an important parameter for composites determined by the thermogravimetric analysis technique. The effect of fibre loading on TGA and DTG (derivative thermogravimetric analysis) curves for thermosetting plastic/kenaf fibre biocomposites are presented in Figure 5. In contrast, the TGA data of the biocomposite samples is depicted in Table 3. Three zones were identified by the TGA curves of the PA6 biocomposites reinforced with TKF. The first zone's weight loss, less

than 250°C and not more than 3% may have been caused by small molecular components and residual moisture evaporating during sample processing and storage (Hirçin et al., 2021; Xu et al., 2019). In the second zone, weight loss ranging between 50–90 wt.% was exhibited up to 500°C, while up to 600°C in the third zone recorded loss up to 12 wt.%. It showed a varied degradation temperature between neat PA6 and TKF/PA6 biocomposites. The thermal stability of the biocomposites was reduced as the fibre loading was increased, resulting in the shifting of the main DTG peak to a lower temperature (Figure 4). The higher onset temperature associated with PA6 led to the conclusion that TKF had lower but quicker thermal degradation temperature at onset than PA6 composites. This finding is similar to Kiziltas et al. (2016) and Abdullah et al. (2022).

The different fibre loadings used revealed that it caused a decrease in the thermal performance of the matrix biocomposite. Furthermore, the increase in weight loss upon increase in fibre loading matched other researchers' findings (Bijwe et al., 2002; Harsha & Tewari, 2003). The PA6's thermal constancy was notably higher than that of TKF, as presented in Figure 5.

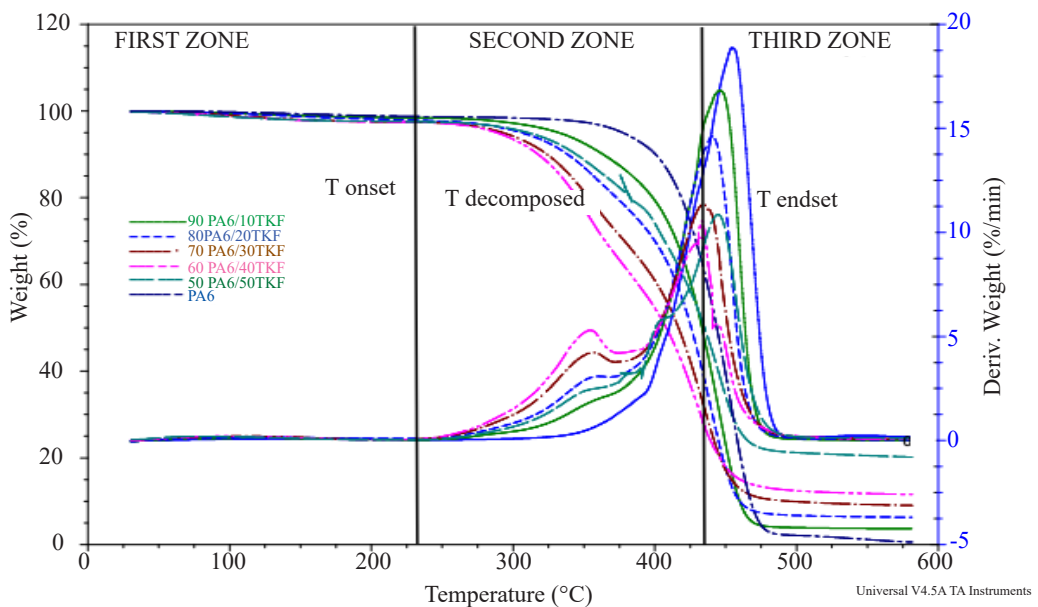


Figure 5. TGA curves and DTG peaks for PA6/TKF reinforced polyamide biocomposites

Flexural Strength

The load-bearing capacity of the kenaf fibre-reinforced polyamide biocomposite was evaluated by its ultimate flexural strength. The ultimate flexural strength was plotted against fibre loading, as presented in Figure 6. The initial decline in flexural strength with the addition of kenaf fibres (at 10, 20, and 30 wt.%) can be attributed to several factors.

Table 3
TGA data for neat PA6 and PA6/TKF biocomposite

Sample	Temperature (°C)			Weight loss (%)	Residue at 600°C (%)
	T onset	Tendset	Tdecomposed		
PA6	250	475	418.13	98.15	0.543
90PA6/10TKF	225	470	405.03	94.77	3.662
80PA6/20TKF	230	470	414.87	75.24	6.288
70PA6/30TKF	213	460	412.30	63.47	9.003
60PA6/40TKF	220	463	410.28	53.90	11.560
50PA6/50TKF	225	465	369.27	77.47	20.170

Firstly, the dispersion and interfacial bonding between the fibres and the polyamide matrix play a crucial role. Inadequate bonding can lead to stress concentration points, reducing mechanical properties. Furthermore, fibres might introduce microstructural flaws, such as voids or microcracks, which can act as stress concentrators and weaken the composite. However, at 40 wt.% fibre loading, an increase in flexural strength to 92.4 MPa is observed, suggesting that the fibre-matrix interaction and stress transfer are optimised at this particular loading. It implies a more efficient load-bearing mechanism, where the fibres contribute positively to the overall strength of the composite due to good stress transmission by the fibre through the matrix with less stress concentration in the composite. This value was greater by more than 50% of similar results reported by Arema (2013), Esmaeili et al. (2023), Khalil (2018), and Khalil et al. (2019). The minimum plastic sleeper bending requirements should be more than 28 MPa as ISO 12856-1 (2014) recommended, and this study surpassed the recommended minimum by more than 200%.

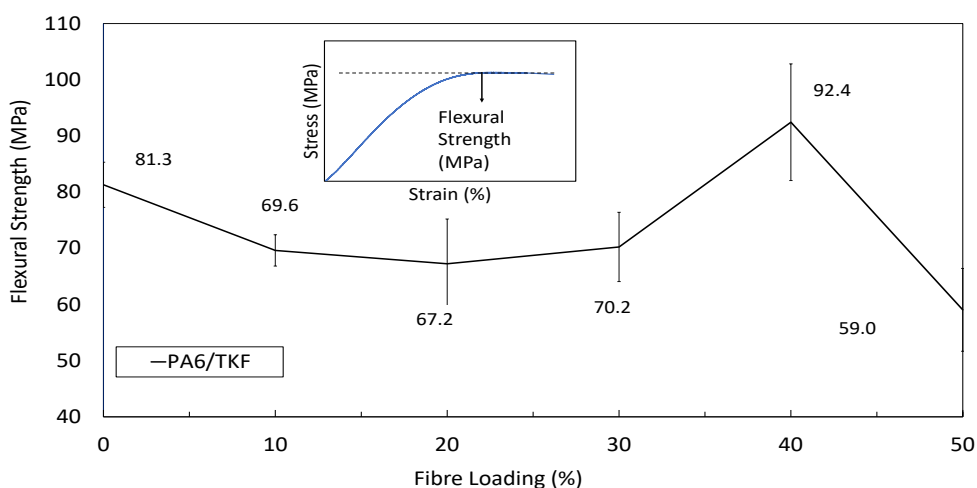


Figure 6. Flexural strength of PA6/TKF reinforced polyamide biocomposites

However, the flexural strength significantly drops to 59.0 MPa at 50 wt.% fibre loading. This substantial decline is likely due to fibre agglomeration, which can occur at higher loadings. Fibre agglomeration reduces the effective stress transfer from the matrix to the fibres, increasing the likelihood of voids and defects within the composite structure. Additionally, the high volume of fibres can disrupt the continuity of the polyamide matrix, leading to compromised structural integrity and reduced mechanical performance.

The TKF-reinforced polyamide biocomposites have demonstrated their potential for railway sleeper applications. Their modulus of elasticity exceeded some available plastic sleepers in the industry by more than 29%. The initial tangent modulus of the biocomposite specimens tended to increase when fibre loading was increased. The increase in fibre loading up to 40 wt.% resulted in a large increase in modulus, thereby indicating lesser damage of the biocomposite under loading. Consequently, it would result in a longer sleeper fatigue life (Esmaeili et al., 2023).

In summary, the variation in flexural strength with different fibre loadings results from the complex interplay between fibre dispersion, interfacial bonding, and the intrinsic properties of the fibres and the matrix. Optimal fibre loading is critical to achieving the desired mechanical properties in fibre-reinforced composites.

CONCLUSION

In conclusion, kenaf fibre-reinforced polyamide biocomposites show significant promise for railway sleeper applications, exceeding the minimum load-bearing capacity recommended by AREMA specifications. Thermogravimetric analysis (TGA) has confirmed the thermal stability of kenaf fibre below 220°C, indicating its suitability as reinforcement at moulding temperatures below this threshold. Fibre loading should be limited to 40 wt.% of TKF for optimal mechanical performance. This study provides critical insights for developing sustainable and efficient materials to meet the demands of modern railway infrastructure, achieving an optimal flexural strength of 92.4 MPa, three times higher than the AREMA standard and existing plastic sleepers. A modulus of elasticity of 4.1 GPa is 29.3% higher than current industry standards.

Overall, natural fibre polymer composites, including kenaf fibre polymeric biocomposites, have demonstrated their potential in measuring up to or even better than synthetic (glass) fibre-reinforced composites, especially in mechanical properties, which is significant in sleeper construction. Notably, the weight reduction ability of kenaf fibre as a substitute to synthetic fibre composites and its low environmental impact coupled with its competitive, stable price makes kenaf fibre a choice material in railway infrastructure material. Hence, natural fibre composites, including kenaf polymeric biocomposites, are rapidly growing, and their application in the railway transport infrastructure seems to have a brighter prospect in the near future.

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Potential Utilisation of Solar-Assisted Kiln Dryer in Bamboo Drying

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ABSTRACT

Bamboo is increasingly used as an alternative material for producing renewable and environmentally friendly products. Bamboo should be dried before use to increase its stability and improve its resistance against biodeterioration agents. The most common drying method for bamboo is through air-drying. Alternatively, artificial drying, such as solar drying, can produce optimum drying results regarding the drying rate and quality of bamboo throughput. This study investigated the potential utilisation of solar drying methods for processing local bamboo. The drying characteristics and physical and mechanical properties of solar-dried *Gigantochloa levis* bamboo culms' bottom, middle, and top sections were determined. The drying time of *G. levis* culm has been reduced to about 40 days compared to the conventional air drying of 70 days using the solar-assisted kiln dryer. Solar-dried culms have a lower final moisture content of 20% than air-dried ones. The average circumference and diameter shrinkage values of solar-dried *G. levis* culms from green to approximately 12% moisture content were 3.22% and 4.29%, respectively, and the wall thickness shrinkage was 8.12%. The mean values of modulus of rupture and modulus of elasticity of solar-dried *G. levis* culm were 63.75 and 12567.99 N mm⁻², respectively,

while its mean values of compression and shear parallel to fibre were 45.87 and 10.01 N mm⁻², respectively. The quality of solar-dried *G. levis* culms produced in this study showed the viability of using a solar-assisted kiln-dryer as a potential alternative processing method for drying local bamboo species in Malaysia.

Keywords: Bamboo, density, drying, mechanical, moisture content, shrinkage, solar

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INTRODUCTION

Bamboo is classified as a monocotyledon plant in the larger grass family of Poaceae and subfamily Bambusoideae (Singhal et al., 2018). About 36.8 million hectares of bamboo plantation area with about 100 genera and over 1500 bamboo species were reported worldwide (Xu et al., 2020). In the Asia country region, the bamboo plantation area was dominated by China, followed by Japan, India, Indonesia, Philippines, Myanmar, and Vietnam (Li & He, 2019). Malaysia has about 0.6 million hectares of bamboo plantation, whereby 31% are in Peninsular Malaysia, 45% in Sarawak, and 24% in Sabah (Kamaruzaman et al., 2016). About 70 bamboo species are reported in Malaysia, and the most commonly exploited bamboo species are *Gigantochloa scortechinii*, *Gigantochloa levis* and *Dendrocalamus asper* (Azmi & Appanah, 1995).

Bamboo is known as the fastest-growing woody plant in the world and can reach maturity as early as 3 to 4 years old. Bamboo has become one of the most important lignocellulosic materials in the timber-based industry. Bamboo is regarded as a sustainable, highly renewable, and environmentally friendly resource material that has the potential to replace wood in the future. Due to its fast-growing characteristics, satisfactory strength properties, and flexibility for diverse products, bamboo has been accepted as an alternative resource that can offer great potential to contribute to Malaysia's economic growth.

Bamboo is traditionally used as a household utensil for chopsticks, toothpicks, joss sticks, skewers, poultry cages, and handicrafts. With the advancement of technology and innovation in bamboo processing techniques, bamboo is increasingly used for structural materials in housing and building construction, such as scaffolding, bridges, poles, purlins, and fencing (Azreena et al., 2016). Furthermore, developing engineered products from bamboo, such as laminated bamboo, plybamboo, and reconstituted densified bamboo board, has led to the increasing demand to produce value-added products such as laminated wall panels, beams, and high-end furniture.

Bamboo's physical and mechanical properties are reported to be comparable to selected tropical hardwood species. According to Abdullah Siam et al. (2019), the density of selected commercial bamboo species ranged from 355–751 kg/m³, approximately equivalent to light hardwood timber species (400 to 720 kg/m³). Meanwhile, the Modulus of Rupture (MOR) and Modulus of Elasticity (MOE) of 4-year-old *Schizostachyum zollingeri* bamboo are 143 Nmm⁻² and 9717 Nmm⁻², respectively which is comparable to the MOR and MOE of selected plantation timber species such as *Acacia mangium* and *Hopea odorata* (Mohamad Omar & Mohd Jamil, 2011; Mohamad Omar & Khairul, 2020).

Drying removes water molecules from the material through evaporation and then moves them to the ambient air. Drying is integral in the timber-based processing sector as it determines the production capacity and revenue generated by the mills. Drying could enhance the quality of bamboo by increasing its dimensional stability and improving its

resistance against biological degradation. Well-dried bamboo is stronger and has better glueing and finishing quality than wet bamboo (Liese & Tang, 2015). In general, the rate of drying is mainly influenced by its structural features, density, culm wall thickness, and the presence of internodes (Liese & Tang, 2015; Tang et al., 2013). The drying rate of young culms is generally faster than mature ones, and bamboo splits dry faster than the culms (Vetter et al., 2015).

Generally, air drying is the most common drying method for bamboo practised worldwide. According to Vetter et al. (2015), air drying of round bamboo takes about 6–12 weeks, and the drying duration depends on the initial moisture content of the bamboo, the environmental conditions, and the wall thickness. However, air drying involves a long drying time and depends on weather conditions. Furthermore, there is a high risk of fungal and insect attack, which causes decay and discolouration of the bamboo.

The use of environmentally friendly and energy-saving resources such as solar energy has been increasingly discussed as solar energy has a low impact on environmental pollution and involves a low operation cost. Solar drying kilns are widely used in the agricultural and food industries, where low drying temperatures and short drying cycles are required (Teussingka et al., 2023). Solar drying may become a potential technology for major energy saving because of its simplicity, ease of operation, and low cost (Sattar, 1992), especially in bamboo drying. Furthermore, solar drying is conducted in a controlled environment chamber, which offers a faster drying time with enhanced-quality bamboo throughput compared to conventional air-drying methods.

Research on the solar drying of bamboo, an important alternative to timber in the construction and furniture manufacturing industries, is still scarce, especially in Malaysia. Thus, a study was conducted to assess the potential utilisation of solar-assisted kiln dryers to improve the processing technique and treatment of bamboo in Malaysia. This study evaluated the drying characteristics and quality of solar-dried *Gigantochloa levis* bamboo culms regarding their physical and mechanical properties. The findings obtained may serve as a guide in optimising the bamboo processing technique through solar drying methods, which could contribute to the development of bamboo applications in Malaysia.

MATERIALS AND METHODS

Sampling

The material used for this study was 4-year-old *Gigantochloa levis* (Beting) bamboo culm obtained from Sungai Lui, Hulu Langat, Selangor, Malaysia. The bamboo culms were cut into nine meters long and then cut equally into three sections of three meters in length, i.e., top, middle and bottom. The dimensions of each bamboo culm were measured for its diameter, wall thickness, and internode length.

Solar-assisted Kiln Dryer

The solar-assisted kiln dryer established at Forest Research Institute Malaysia (FRIM) Kepong, Selangor, Malaysia, has a dimension of 6 x 3 x 4 meter (length x height x width) with a capacity of up to 24 tons or 34 m³ (Figure 1). The greenhouse-concept drying kiln was made of a stainless-steel frame structure and covered with two layers of transparent ultraviolet (UV) plastic anchored on the concrete foundation. A black-coloured fabric-like material is fitted under the transparent plastic as a heat absorber from solar radiation. It will help to increase the temperature inside the kiln. The kiln was equipped with three industrial-sized fans, each operating at 1 horsepower respectively. The fans were mounted on a plywood wall to circulate heated air uniformly throughout the chamber. The kiln was equipped with an exhaust fan to effectively remove excess moisture from the chamber and transfer it to the surrounding air. The kiln's monitoring system was equipped with a proportional integral derivative (PID) kiln control instrumentation to efficiently monitor temperature and relative humidity during drying.

Solar Drying of Bamboo

Approximately 5 m³ of freshly *G. levis* bamboo culms, each 3 meters long, were dried in a solar-assisted kiln dryer. Before the commencement of the drying trial, ten per cent sampling methods were carried out based on the total kiln charge, whereby the bamboo culms were randomly selected from the stacks as sample boards. The dimension of each sample board was measured for its diameter, wall thickness, and circumference. The sample boards' initial moisture content (MC) was determined according to the Indian Standards Institution (Anonymous, 1976), whereby a 20 x 20 mm strip was cut from the edge of each sample board. Each MC strip was weighed before and after drying in an oven of temperature 103 ± 2°C for 24 hours. The initial MC of each sample board was calculated based on the formula (1):

$$\text{Moisture content, MC (\%)} = \left(\frac{W_g - W_o}{W_o} \right) \times 100 \quad (1)$$

Where W_g is the weight (g) of the specimen in green condition, and W_o is the specimen's oven-dry weight (g).

The sample boards were placed within the bamboo stacks. The drying process was monitored by weighing the sample boards daily until they reached a constant weight. The oven-dry weight (W_o) and current moisture content (MC_c) of the sample board were estimated based on the formulas (2) and (3):

$$\text{Oven dry weight, } W_o \text{ (kg)} = \frac{W_g \times 100}{MC + 100} \quad (2)$$

$$\text{Current moisture content, } MC_c (\%) = \left(\frac{W_c - W_o}{W_o} \right) \times 100 \quad (3)$$

Where W_g is the initial weight (kg) of the sample board, MC is the moisture content (%) of the MC specimen, and W_c is the current weight (kg) of the sample board.

The air velocity in the solar kiln was measured using a digital anemometer. The kiln's temperature and relative humidity readings were recorded regularly using a psychrometer consisting of dry bulb and wet bulb thermometers. The relative humidity was calculated based on wet bulb depression (subtracting the temperature of the wet-bulb thermometer from the temperature on the dry-bulb thermometer) and dry bulb temperature. Air drying of *G. levis* culms was also conducted by stacking the bamboo on an open site under the shed.

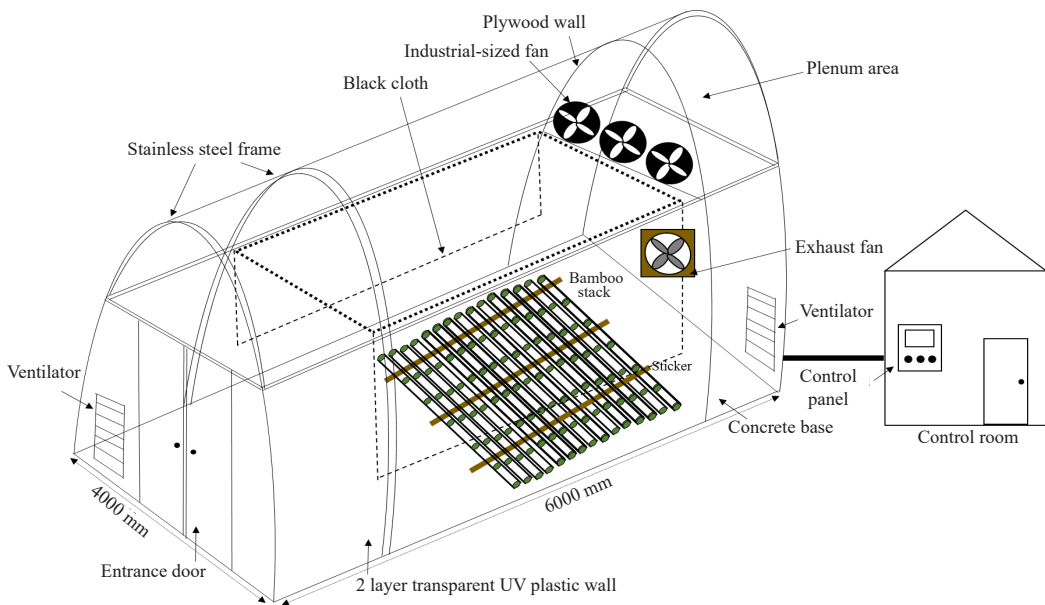


Figure 1. Schematic diagram of solar-assisted kiln dryer

Assessment of Dried Sample

After the completion of the drying trial, the final MC and dimension of the sample boards were determined, and the estimated current moisture content of the sample boards was normalised accordingly. Specimens measuring 20 mm x 20 mm x culm wall thickness (mm) were obtained from the dried bamboo culms' bottom, middle, and top sections to determine final moisture content and density according to the Indian Standards Institution (Anonymous, 1976). A total of 30 specimens were used in the test. The density (ρ) of each specimen was calculated using formula (4):

$$\text{Density, } \rho \text{ (kgm}^{-3}\text{)} = \frac{W_g}{V_g} \quad (4)$$

Where W_g is the weight (kg), and V_g is the volume (m^3) of the specimens.

The shrinkages (%) of the dried culms were calculated from the initial and final dimensions of the sample board using formula (5):

$$\text{Shrinkage, } \beta \text{ (\%)} = \frac{D_g - D_0}{D_g} \times 100 \quad (5)$$

Where D_g is the corresponding circumference, diameter, and thickness dimensions (mm) of the green culms and D_0 is the corresponding circumference, diameter, and thickness dimensions (mm) of the dried culms.

Mechanical Properties Test

The bottom, middle, and top sections of the solar-dried *G. levis* bamboo culms were cut based on specific dimensions for mechanical testing following ISO 22157:2019(E). A total of 30 bamboo culms samples comprising the bottom, middle, and top sections were used in each test. The static bending test included Modulus of Rupture (MOR), a measure of maximum strength that a bamboo material can withstand, and Modulus of Elasticity (MOE), a measure of the stiffness of the bamboo culms. A 3-point static bending test was conducted by subjecting the specimen of 3 meters in length to load heads that move at a constant speed of 8 mm min^{-1} and span of 2.3 m (Figure 2). Compression parallel to fibres and shear parallel to fibres tests for both node and internode specimens of 70 mm in length were performed by applying load at a constant rate movement of 0.6 mm min^{-1} and 0.4 mm min^{-1} , respectively.



Figure 2. Arrangement for 3-point static bending test of 3-meter-long bamboo culms

Statistical Analysis

Statistical analysis was conducted using one-way analysis of variance (ANOVA) to determine the significant variation of physical and mechanical properties of dried bamboo culm at different height positions.

RESULTS AND DISCUSSION

Green Moisture Content (MC)

The average green MC of *G. levis* culm in the present study was 104.13% (50.25–147.33%). The green MC at the bottom

section ranged from 116.79 to 147.33%, averaging 130%. The average green MC of the middle and top culms was 99.15% and 83.24%, with range values of 67–116.55% and 50.25–118.55%, respectively. Based on all samples tested, the green MC decreased from the bottom towards the top parts of the culm, and the variation was significantly different at $p > 0.5$.

The green MC range of *G. levis* culms was substantially comparable to India's green MC bamboo *Dendrocalamus strictus* (Wakchaure & Kute, 2012). The freshly cut bamboo culms were reported to have MC from 70 to 150%, with an average value of 103%. The findings in the recent study align with Vetter et al. (2015). He found that the *Bambusa vulgaris* MC from Brazil decreased vertically from bottom to top. Furthermore, Wakchaure and Kute (2012) reported that the MC of 3-year-old *Dendrocalamus strictus* from India varies along the culm height, with the top portions having consistently lower moisture content than the middle or bottom. The green MC of Malaysian bamboo is also highest near the bottom part (Azmi & Appanah, 1995). Age, anatomical structure, species, and harvesting season influence the difference in MC from the bottom to the top. Furthermore, bamboo is a heterogeneous material with different cellular structures and chemical compositions, which may affect its moisture sorption behaviour.

Drying Characteristic

Gigantochloa levis culms were dried in a solar kiln at 30 to 50°C and relative humidity of 50.7 to 86.3%, respectively (Figure 3). The solar kiln exhibited a higher temperature than the ambient temperature (Figure 4) because the solar kiln is equipped with appropriate drying equipment, such as a fan and ventilator, to effectively remove excess moisture to the surroundings and facilitate the circulation of heated air throughout the kiln. The relative humidity of the solar kiln decreases with an increase in the temperature due to the increment of the water-holding capacity of the air (Simo-Tagne et al., 2018). The increase in temperature will subsequently decrease the equilibrium moisture content and cause the moisture to be removed from the bamboo and transferred to the surroundings (Sasongko et al., 2020).

Gigantochloa levis culms could dry from an average initial MC of 95.63% to approximately 12.23% in 43 days, with a moisture loss rate of 1.9% per day (Figure 5). Based on the drying curve, the drying rate below the fibre saturation point (FSP) from 18 to 10% MC was 0.02%/h and varies according to the height position. In comparison, the drying rate above FSP is faster by more than 80% with a drying rate of 0.14%/h. *G. levis* was found to be able to dry uniformly along the culm, and the variation of final MC between height positions was within the permissible range of 3.5% (Table 1). Therefore, the drying of *G. levis* culms can be performed in one kiln charge without pre-sorting according to a specific height before drying.

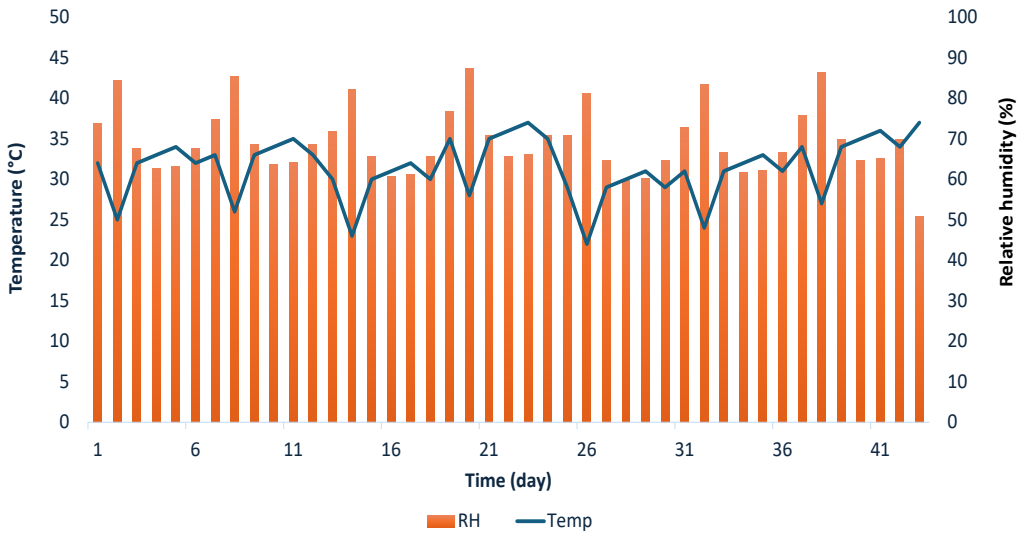


Figure 3. Temperature and relative humidity of the solar kiln during drying trial

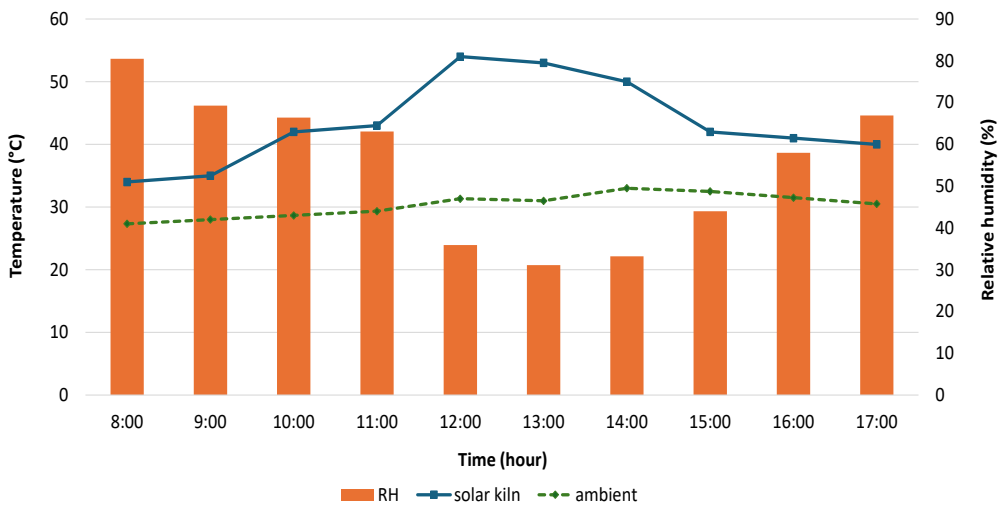


Figure 4. Ambient and solar kiln temperature

The solar drying method reduces the drying time of *G. levis* culms by approximately 40% compared to the air-drying method (Figure 6). In addition, solar-dried culms have a lower final moisture content of 20% compared to air-dried culms, which are suitable for indoor applications (Table 2). Significant drying defects were not observed in solar-dried bamboo culms. However, slight end checks and bowing were observed in 11% of the culms. No other defects, such as cupping, surface checking, and twisting, were observed in all

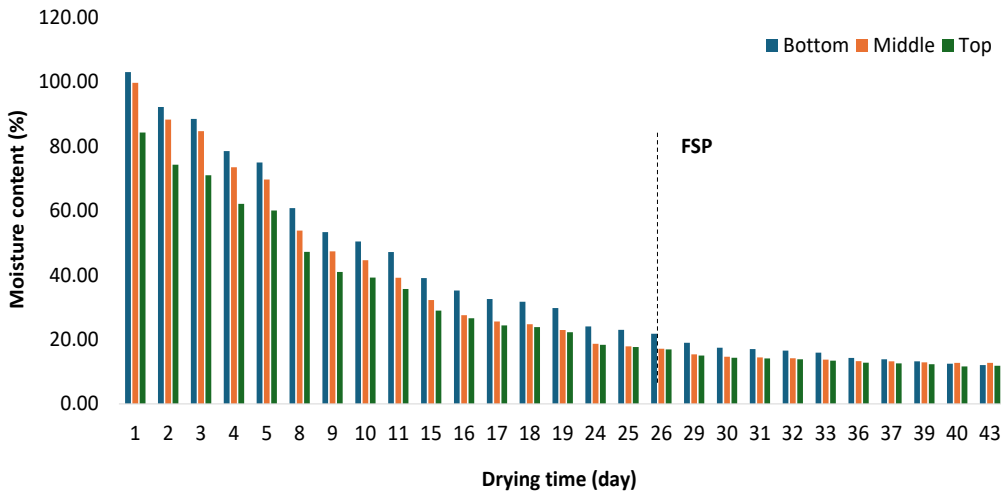


Figure 5. Drying time and moisture content of *Gigantochloa levis* at different height positions during the solar drying process

Table 1
Final moisture content and density of solar-dried *G. levis* culms

Height position	Final moisture content (%)			Overall
	Bottom	Middle	Top	
Mean	13.37 (1.01)	12.92 (0.33)	12.35 (1.02)	12.88 (0.79)
Range	11.67–14.99	12.16–13.38	11.34–14.06	11.34–14.99
Density (kg m ⁻³)				
Mean	657 (53) ^a	643 (87) ^b	754 ± (105) ^c	685 (82)
Range	579–731	512–797	573–884	512–884

Figures in parentheses are standard deviations. The values with different superscript letters in each row are significantly different ($p > 0.05$)

culms. Bowing and end checks could be attributed to the excessive and non-uniform water movement from the inner to the outer part of the culms. In addition, no sign of fungal and insect attacks was observed in all solar-dried bamboo culm. Air-dried *G. levis* culms show some observable fungal attacks, especially for the sample located at the bottom stacks, due to the prolonged exposure to humid conditions where low ventilation and uneven air flow through the stacks have slowed down the process of moisture loss from the bamboo, which subsequently encourages the growth of fungi.

The bamboo culms could achieve a lower equilibrium moisture content (EMC) than the air-drying method using a solar drying kiln. The solar drying method could dry the bamboo up to 10–12% MC, which is the MC that the air-drying process could not achieve. Solar-dried bamboo has a lower MC than air-dried bamboo, which is suitable for fabricating

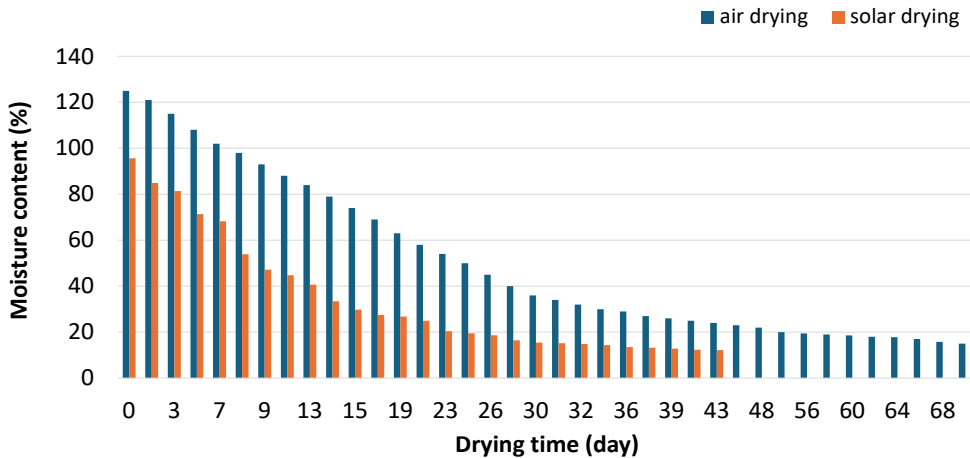


Figure 6. The drying curve of *Gigantochloa levis* dried using solar drying and air-drying methods

Table 2
Drying characteristics of *G. levis* culms

Condition	Initial MC (%)	Final MC (%)	Drying time (days)
Solar-dried	95.63	12.23	43
Air-dried	97.64	15.64	70

bamboo products intended for indoor applications such as furniture components and souvenirs, as well as value-added products such as laminated ply bamboo. Solar drying is more efficient than air drying in terms of drying time. Since the solar kiln was equipped with proper ventilation and heat-trapping system for efficient heat generation and optimum air circulation throughout the drying process, the drying time of the bamboo culms could be reduced effectively. Furthermore, the solar drying method could enhance the quality of bamboo culms by protecting against biological deterioration attacks such as fungi and mold. Moreover, the drying defects, such as splits and checks in bamboo, could be reduced with the solar drying method due to the reduction of temperature at night, which would exhibit a mild reconditioning treatment, which encourages bamboo to release stress accumulated during the day (Haifaa et al., 2004).

Density

The density of solar-dried *G. levis* culms ranged from 512 to 884 kg m⁻³ with a mean value of 685 kg m⁻³ (Table 1). The density decreased slightly from the bottom to the middle and increased towards the top of the culms, but the value was not significantly different at $p < 0.05$. Generally, the density of bamboo tends to increase with height (Santhoshkumar &

Bhat, 2014; Zakikhani et al., 2017). This trend may be associated with the increase in the proportion of vascular bundles, silica content, and sclerenchyma fibres with culm height (Correal & Arbelaez, 2010; Wang et al., 2016). Furthermore, increased density could also be attributed to increased fibres to parenchyma ratio from the bottom upwards (Vetter et al., 2015).

Shrinkage

The shrinkage value of solar-dried and air-dried *G. levis* culms is presented in Table 3. The average circumference and diameter shrinkage values of solar-dried *G. levis* culms from green to approximately 12% moisture content were 3.22% and 4.29%, respectively. Meanwhile, the solar-dried culm has a wall thickness shrinkage of 8.12%. In this study, the bottom, middle, and top sections of solar-dried bamboo culms have a significantly ($p > 0.05$) lower shrinkage value of approximately 10 to 40% compared to air-dried culms. Solar kilns could maintain a higher humidity during the early drying stage by evading moisture from the bamboo material (Sattar, 1994). The high humidity could retard the drying process and provide a conditioning treatment to the bamboo, which could minimise the occurrence of shrinkage during the drying process.

Table 3

Shrinkage values of air-dried and solar-dried G. levis bamboo culm

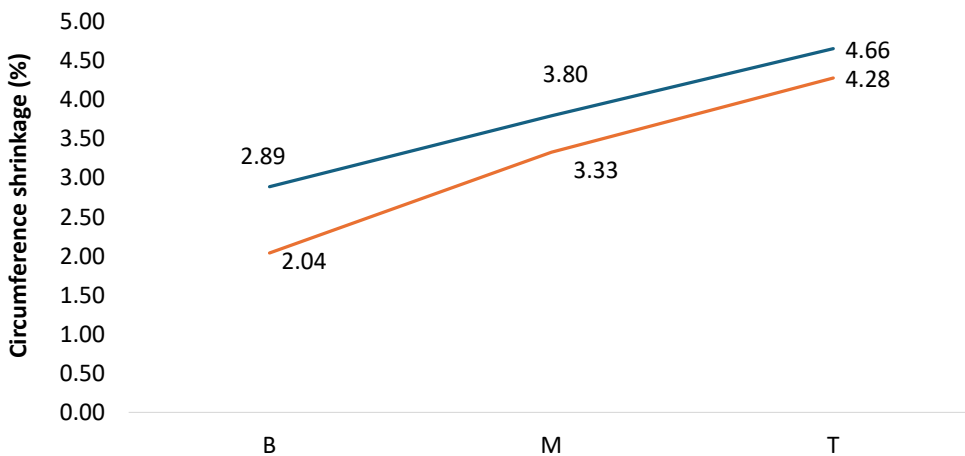
Condition	Height position		Shrinkage (%)		
			Wall thickness	Circumference	Diameter
Air-dried	Bottom	Mean	9.25 (5.88)	2.89 (1.00)	8.26 (5.18)
		Range	2.13–18.15	1.41–4.69	3.56–20.16
		Middle	Mean	11.41 (8.92)	3.80 (4.31)
		Range	2.33–29.06	0.00–13.64	0.00–17.07
	Top	Mean	13.21 (8.74)	4.66 (4.86)	5.83 (3.96)
		Range	4.23–28.76	0.00–13.37	1.34–12.38
		Overall	Mean	10.89 (7.90)	3.60 (3.57)
		Range	2.13–29.06	0.00–13.64	0.00–20.16
	Solar-dried	Bottom	Mean	4.99 (2.08)	2.04 (1.63)
Range			2.38–7.46	0.00–(3.99)	1.34–16.10

Table 3 (Continue)

Condition	Height position	Shrinkage (%)			
			Wall thickness	Circumference	Diameter
Middle	Mean		8.18	3.33	4.14
			(1.33)	(1.69)	(2.85)
Top	Range		6.30–9.17	1.89–5.69	1.20–8.00
		Mean	11.18	4.28	2.34
Overall	Range		(5.32)	(0.44)	(1.86)
			4.40–(17.39)	3.92–4.90	0.00–(4.55)
Overall	Mean		8.12	3.22	4.29
			(2.91)	(1.25)	(3.86)
	Range		2.38–17.39	0.00–5.69	0.00–16.10

Values in parentheses are standard deviations

The culm wall thickness and circumferences shrinkage tend to increase from the bottom to the top part of the culms for both drying methods. Meanwhile, the diameter shrinkage shows an opposite trend. The bottom part reported the highest diameter shrinkage value compared to other parts. Meanwhile, the top part showed the highest wall thickness and circumference shrinkage value, followed by the middle and bottom parts (Figure 7). Based on the statistical analysis, shrinkage variation within the culm height was not significantly different ($p > 0.05$).



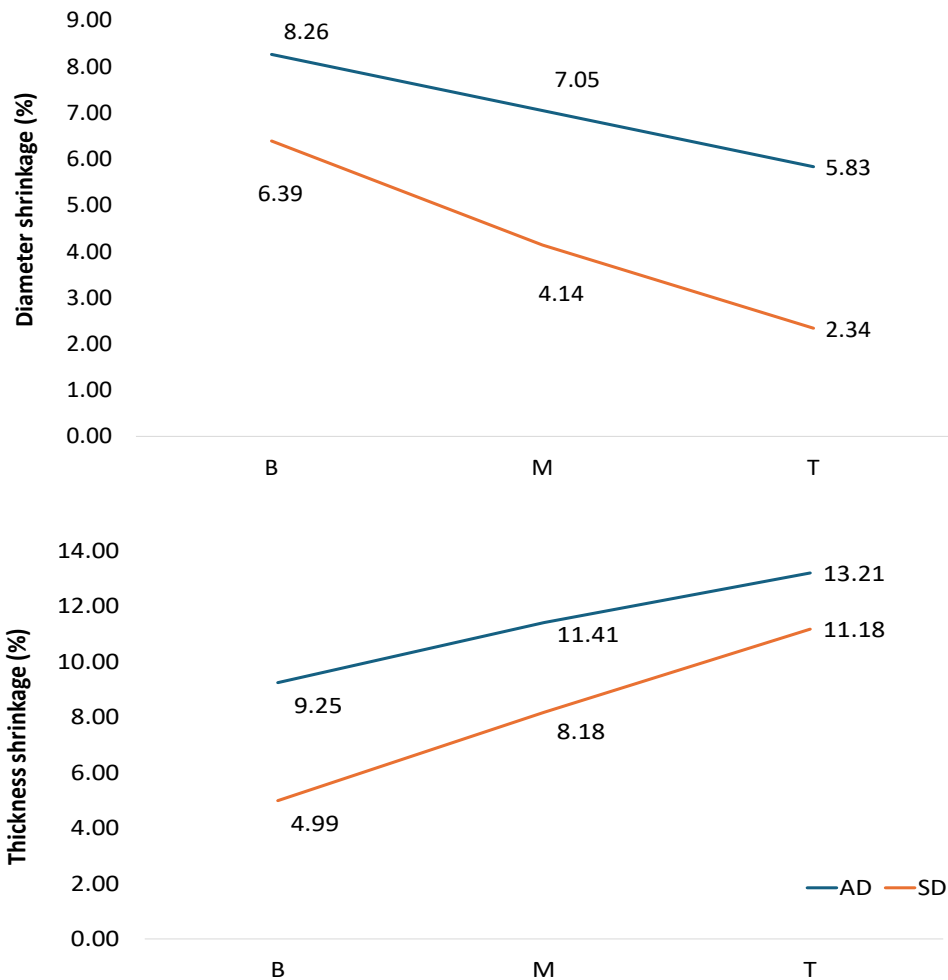


Figure 7. Variation of shrinkage of air-dried (AD) and solar-dried (SD) *Gigantochloa levis* culms at different height positions (B = bottom, M = middle, T = top)

Like wood, bamboo starts to shrink and change in dimension when it reaches its fibre saturation point (FSP). The shrinkage in diameter is higher at the bottom parts, probably due to higher initial moisture content (Rehman & Ishaq, 1947). Thus, proper drying practices must be implemented to avoid excessive shrinkage of bamboo culm, which may lead to severe warping, such as bowing and splits. The top part experiences a higher wall thickness and circumference shrinkage due to the higher number of vascular bundles in the top section compared to the bottom and middle sections. The condition is further compounded by the distribution of fibre that increases from the inner layer towards the outer layer of the bamboo culm wall (Azedah & Ghavami, 2018). Due to the different volume fractions of fibres at the internal and external sides of bamboo at the transversal

cross-section, the trend of non-uniform shrinkage of the wall thickness and circumference was observed along the culm's height.

Mechanical Properties

Table 4 shows the mechanical properties of solar-dried *G. levis* culms. Three-point static bending test results showed the mean values of modulus of rupture (MOR) and modulus of elasticity (MOE) were 63.75 and 12567.99 N mm⁻², respectively. The mean values for compression and shear parallel to fibre were 45.87 and 10.01 N mm⁻² under the required test condition.

Table 4
Mechanical properties of solar-dried G. levis bamboo culms

Mechanical properties	Bottom	Middle	Top	Overall	<i>p</i> -value
MOR (N/mm ²)	66.89 (18.82)	51.62 (22.41)	72.75 (28.94)	63.75 (23.39)	0.082 ns
MOE (N/mm ²)	12896.08 (2298.03)	10248.24 (3707.01)	14559.66 (5570.77)	12567.99 (3858.60)	0.037 s
Compression (without nodes) (N/mm ²)	49.69 (9.04)	41.04 (13.50)	45.89 (8.54)	45.54 (10.36)	0.079 ns
Compression (with nodes) (N/mm ²)	50.52 (9.23)	40.87 (11.98)	47.20 (9.97)	46.20 (10.39)	0.095 ns
Shear (without nodes) (N/mm ²)	10.35 (2.44)	8.23 (2.08)	9.22 (2.15)	9.27 (2.22)	0.077 ns
Shear (with nodes) (N/mm ²)	11.48 (3.72)	9.83 (2.71)	10.94 (1.84)	10.75 (2.76)	0.341 ns

Figures in parentheses are standard deviations. MOR = modulus of rupture; MOE: modulus of elasticity; ns = not significant; s = significant at $p < 0.05$

The analysis showed the MOR and MOE value of solar-dried *G. levis* was highest at the top, followed by the bottom and middle sections of the culms. Meanwhile, the compression and shear values were highest at the bottom, followed by the top and middle sections due to the variation of density, physical dimension, and size and the number of vascular bundles along the culm height and across the culm wall (Liese & Köhl, 2015). Awalluddin et al. (2017) and Daud et al. (2018) reported that the compression and shear values of round betung bamboo (*Dendrocalamus asper*), minyak bamboo (*Bambusa vulgaris*), semantan bamboo (*Gigantochloa scortechinii*) and semeliang bamboo (*Schizostachyum grande*) was highest at the top section followed by middle and bottom parts of the culms. The MOR, compression, and shear values did not vary significantly ($p > 0.05$) between the bottom, middle, and top sections except for MOE. The sample tested with nodes showed a

higher compression and shear value than those without nodes. However, the result was not significantly different. The presence of nodes can influence the tensile strength of bamboo and engineered bamboo lumber by preventing the propagation of splits and buckling during services (Liu et al., 2021).

The mechanical properties of solar-dried *G. levis* bamboo culms were compared with previously published data on air-dried bamboo culms. The mean values of MOR, compression, and shear parallel to the fibres of solar-dried samples obtained in this study were slightly higher compared to other Malaysian bamboo species in air-dried conditions (Awalluddin et al., 2020; Awalluddin et al., 2017; Daud et al., 2018). Furthermore, the mean shear value of solar-dried culms obtained in this study is higher by approximately 20% compared to air-dried *G. levis* culms reported by Mohd Tamizi (2010). The finding showed that solar-dried *G. levis* had better mechanical properties than air-dried bamboo. This improvement could be due to the densification and chemical modification of the bamboo during the solar drying process. Studies on wood dried under controlled environments such as kiln drying and solar drying also revealed better mechanical properties than wood dried using air drying (Jacket et al., 2014; Samson et al., 2021; Uetimane, 2020). Overall, the solar-dried bamboo culms reported in this study have good mechanical properties and are suitable to be fabricated into furniture components and laminated bamboo products.

CONCLUSION

The solar-assisted kiln dryer used in this study reduced the drying time of *G. levis* culms by approximately 40% compared to the air-drying method. The drying time of *G. levis* culms has been reduced to 40 days compared to the conventional air-drying time of about 70 days. Solar-dried culms have a lower final moisture content of 20% compared to air-dried culms, which are suitable for indoor applications. Overall, the solar drying method enhances the quality of *G. levis* culms. The incidence of significant drying defects, such as cupping, surface checking, and twisting, as well as a sign of fungal and insect attacks, were not observed in all culms. Solar-dried bamboo has a significantly lower shrinkage value of approximately 10 to 40% compared to air-dried culms. The solar-dried *G. levis* culms have mechanical properties comparable to those of the air-dried sample in terms of static bending, compression, and shear parallel to the fibre. The quality of solar-dried bamboo culms produced in this study indicates the viability of using solar-assisted kiln dryers to produce quality bamboo materials for further fabrication into value-added products.

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