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Microstructure and mechanical performance of bamboo fiber reinforced mill-scale—Fly-ash based geopolymer mortars

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A R T I C L E I N F O

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A B S T R A C T

Natural fiber reinforcement in cementitious matrices is being explored to provide an environment-friendly solution for lowering the overall carbon footprint of construction materials while giving the matrix much-needed tensile strength. Short bamboo fibers extracted from *Bambusa blumeana* or Kawayan tinik using 5% sodium hydroxide solution and treated with 10% aluminum sulfate solution are used to reinforce zero-cement geopolymer mortars. Bamboo fibers with varying lengths of 10 mm, 20 mm, and 30 mm are mixed with mill-scale – fly ash-based geopolymer in varying 0%, 0.5%, 1%, 1.5%, and 2% fiber loading per weight of specimen sample. Compressive strength and split tensile strength tests are administered to small cylinder samples, 50 mm in diameter by 100 mm in height, in accordance with ASTM C780. An optimum fiber length of 20 mm and fiber loading of 1.4% by weight is determined using Response Surface Methodology (RSM). The addition of bamboo fibers increased the unconfined compressive strength up to 292.41% compared to specimens without bamboo fibers. The split tensile strength also improved by up to a 355.82% increase compared to control samples. The corresponding high-strength and low-strength samples are also subjected to Fourier-transform Infrared Spectroscopy – Attenuated Total Reflectance (FTIR-ATR) to investigate and compare the stretching of bands between the raw materials and tested specimens. Scanning Electron Microscopy – Energy Dispersive X-Ray analysis (SEM-EDX) is used to show microscopic images and the elements present in the selected samples. The implications of the results on the material development of bamboo fiber-reinforced geopolymer mortar for construction are discussed.

1. Introduction

The contribution of Ordinary Portland Cement (OPC) to the overall carbon footprint in construction, as estimated to be around 8% of the global carbon dioxide (CO_2) emissions by Andrew (2019) magnifies the part of OPC in climate change. Alternative materials and replacements to cement such as fly ash and slags for geopolymer-making are gaining interest as they offer lower carbon footprints (Bernal et al., 2016). Davidovits (1979) first defined the term geopolymer as the type of inorganic polymer made from activated aluminosilicate-rich precursors. Geopolymer materials have shown promising performance over the years and are seen to have the potential to replace OPC in concrete making (Singh, 2018). A typical design mix of geopolymer consists of precursors and activators. Strong alkali activator promotes the rapid dissolution of silicate and aluminum compounds in the raw materials while providing additional soluble silicate ions, thus caustic alkali and sodium waterglass are usually used (Yu and Jia, 2022).

Coal Fly Ash (CFA), which is usually used as a precursor for geopolymer-making, has many uses including different purposes such as a catalyst for organic contaminant reduction found in wastewater that is toxic to humans (Kuźniarska-Biernacka et al., 2022). The latest research studies regarding geopolymer concrete that uses CFA includes Life Cycle Assessments (LCA) with variable mixes and self-healing geopolymer (Garces et al., 2022). The use of geopolymer concrete has been seen to contribute to lowering the overall carbon footprint of construction, up to 37% better in terms of global warming potential compared to OPC-based concrete (Garces et al., 2021).

The current study explores the use of CFA and other alternative materials found in steel-making wastes in the form of mill-scale powder as precursors in geopolymer-making, with the addition of short bamboo fibers extracted from Kawayan tinik to enhance the mechanical properties of the resulting geopolymer matrix.

A study by Quiatchon et al. (2021) optimized a design mix consisting of a low calcium Class F fly ash (FA). It was observed that a lower water-solid ratio of 0.2 and synthesized using a mix of sodium

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Nomenclature

Symbols

ln	natural logarithm
°C	temperature, degrees Celsius
%wt	percent by weight

Terms

g	grams
min	minutes
h	hours
C/FA	coal/fly ash
BOF	blast oxygen furnace
GGBS	ground granulated blast furnace slag
MS	mill scale
MK	metakaolin
RHA	rice husk ash
NaOH	sodium hydroxide
WG	waterglass (sodium silicate)
RSM	response surface methodology
OPC	ordinary Portland cement
CO_2	carbon dioxide
Fe-O-Fe	iron and oxygen compounds
Si-O-T	Silica-aluminate network, T is Si/Al
FTIR-ATR	Fourier transform infrared spectroscopy –attenuated total reflectance
SEM-EDS	Scanning electron microscopy – energy dispersive X-ray spectroscopy
UCS	unconfined compressive strength
UTM	universal testing machine

hydroxide (NaOH) and sodium silicate or waterglass (WG), with 1:2.5 NaOH-to-WG ratio, yields higher compressive strength compared to samples that used 1:1 NaOH-to-WG ratio and higher water-solid ratio. Developed geopolymer material can be studied further to conform with different applications. Various geopolymer applications are discussed by Abulencia et al. (2021) leading to architectural products and some structural-grade materials that can be used as strengthening materials for restoring old structures which do not require higher compressive strength compared to the existing materials of the structure. The benefits of geopolymers, however, are still limited in scope and observed to have some indicators of carbon footprint in production and supply (Habert et al., 2011). Partial and full replacement of fly ash with other potential precursors in geopolymer-making are being explored to lower carbon footprint even more. Studies by Rafeet et al. (2017) suggest a design mix for geopolymer concrete with a combination of coal fly ash (CFA) and ground granulated blast furnace slag (GGBS). Elevated temperature, which is a common technique for curing geopolymers, can also be eliminated with the partial replacement of CFA with GGBS. It has also been observed that the presence of GGBS is a factor that reduces the activator requirements for geopolymer-making (Serag Faried et al., 2020).

To further minimize the negative environmental impact of geopolymer-making, this study made use of mill-scale powder that can be used as a partial replacement for the main precursor. Additional materials that can be used to enhance mechanical properties such as short natural fibers were explored.

1.1. Mill scale

Scrap materials such as Blast Oxygen Furnace (BOF) and mill scale (MS) can also be used as partial and full replacements for FA in geopolymer-making (Kumar et al., 2021). It is observed that using 30% MS to replace class F - FA in geopolymer-making yields higher compressive strength. The alkali present in the geopolymer leads to the formation of ferro-silicate gel which is responsible for the additional strength.

A study by Novack et al. (2022) shows the infrared spectrum of the MS sampled by the authors, and it is observed to have the existence of vibration bands in the 477, 450, and 413 cm^{-1} range that can be attributed to the stretching vibration of iron oxides, Fe–O–Fe. This can influence the formation of ferro-silicate gel when exposed to an alkali environment and mixed with some silica-rich materials. R.G.D. Libre et al. (2022a) used mill-scale as a partial replacement for low calcium class F fly ash. It is observed in the study that the significant factor affecting compressive strength is the NaOH-to-WG ratio with the optimum ratio of 1:2.5 by weight, considering that the water-solid ratio is to be kept constant at 0.3 and the activator-to-precursor ratio is to be kept at 0.38. The study suggests an optimum MS-to-FA replacement of 20%.

1.2. Short fiber reinforcement

Enhancement of the mechanical properties of cementitious materials can be achieved through the addition of short fibers. A study by Abdullah et al. (2017) investigated the effect of steel fibers as introduced in plain geopolymer concrete. The study shows that short steel fiber loading of 7% in weight of geopolymer concrete yields a maximum value of mechanical strength up to a 42.36% increase in compressive strength and 121.52% in flexural strength.

Some authors use more environment-friendly reinforcement such as basalt and natural fibers. Heweidak et al. (2022) investigated the mechanical performance of basalt fiber-reinforced self-compacted ambient-cured geopolymer mix. It is reported that the incorporation of a weight loading of 2% basalt fiber with 12 mm and 30 mm hybrid lengths, yields a 61.96% increase in indirect (split) tensile strength compared to an unreinforced geopolymer mix. A study by Lazorenko et al. (2020) investigated the use of flax fibers in geopolymer. The flax influenced the decline in mechanical properties mainly because of added air voids, poor fiber-matrix bond adhesion, and low strength resistance of the fibers. Saccani et al. (2021) evaluated the performance of hemp, kenaf, and bamboo mixed with metakaolin-based (MK) geopolymer. With the incorporation of 3% fibers by specimen weight, the flexural strength of the samples with kenaf and bamboo increased to 80% and 20%, respectively.

1.3. Bamboo fiber

Various performances of bamboo fibers are investigated for different purposes. Sá Ribeiro et al. (2021) used Amazonian bamboo species *Guadua angustifolia* as short fibers with 12.5 mm to 40 mm length as reinforcement to MK-based geopolymer and immersed in 5% by weight sulfuric or hydrochloric acid solution for 7 up to 112 days. It is observed that bamboo fiber geopolymer composite can perform in applications exposed to sulfuric environments as the fibers were not dissolved and are still capable of keeping the geopolymer matrix intact. A study by Zhang et al. (2022) used bamboo shavings or the residuals from a bamboo workshop as additive to MK-based geopolymer and it is observed to increase the resulting compressive strength up to 7.76% after 28 days of curing.

One of the most abundant bamboo species in the Philippines is the *Bambusa bluemeana* or locally known as Kawayan Tinik which is a potential source of raw materials for construction and bioenergy production (Mendoza et al., 2019). Villanueva et al. (2022) discussed the rejection rates of Kawayan Tinik for structural grading in the Philippines. It ranges between 2% to 10% from visual grading at the harvest site and up to 2.6% rejection rate after seasoning or after treating, grading, and preservation which is equivalent to 26 poles per 1000 poles. These poles can be used for other purposes such as shavings and fiber extraction. A study by R.G. Libre et al. (2022b) used the rejected and scrapped Kawayan Tinik bamboo poles for fiber extraction. It is observed that the bamboo fibers extracted using a 5% sodium hydroxide solution yield higher tenacity compared to fibers extracted using a higher concentration of sodium hydroxide. A rougher surface was observed for the fibers treated with 10% aluminum sulfate solution. The rough surface enhancement is important for better bond performance between the fibers and cementitious matrices. This development can be used for reinforcing geopolymer matrices.

1.4. Response surface methodology

Due to its complex proportioning and multiple factors to consider, researchers use different ways to develop their own design mix method for geopolymer production and it is yet to establish a standard for the design (Matsimbe et al., 2022). Various approaches that can be used are the trial-and-error approach, the Taguchi approach, the particle packing fraction method, and the response surface methodology (RSM) in optimizing design mix proportions. Taguchi's approach has the ability to identify a significant level of a specific factor that affects the response parameter while RSM has the ability to show clearly what parameter has the highest effect on the resulting response. Most of the researchers use RSM as their tool in optimizing geopolymer mix, utilizing computer-aided design such as I-optimal that can create a design of experiment with minimum integrated variance. Zahid et al. (2018) emphasized the need to use robust tools for the design of experiments and RSM is usually used for a systematic and statistically accepted way of optimization. In RSM, a regression model is being developed to predict the optimized points in the resulting response surface of a given data set. Buyondo et al. (2020) used RSM to optimize geopolymer mix with Rice husk ash (RHA) and metakaolin (MK) in geopolymer-making, with compressive strength as a response parameter. In the study, the optimal composition was observed to have 11.67% RHA and 12.22% MK by weight of the whole geopolymer specimen.

In this current study, RSM is used to optimize a design mix for short bamboo fiber-reinforced MS-FA geopolymer mortar. Short bamboo fibers from Kawayan Tinik extracted through 5% NaOH solution and treated with 10% aluminum sulfate are used as reinforcement to geopolymer mixes with a 20% MS-to-FA ratio. From these, the effect of the variability in fiber length and the% fiber loading by weight of specimen were investigated.

2. Methodology

The mechanical performance of short bamboo fiber reinforced geopolymer mortars was investigated through unconfined compressive tests and split tensile tests in accordance with the standard test method for preconstruction and construction evaluation of mortars by the American Society for Testing and Materials (ASTM C780). Fourier transform infrared spectroscopy – attenuated total reflectance (FTIR-ATR) and Scanning electron microscopy – energy dispersive X-ray spectroscopy (SEM-EDS) are used to characterize selected raw materials and geopolymer mixes.

2.1. Materials

In this study, the mill scale powder and the low calcium Class F fly ash precursors discussed in the study of Libre et al. (R.G.D. 2022) for developing MS-FA-based geopolymer paste are used to develop fiber-reinforced MS-FA geopolymer mortar with sand (3.5% moisture content) as the fine aggregates and short bamboo fibers extracted from Kawayan Tinik using 5% NaOH solution and treated with 10% aluminum sulfate solution are used as the fiber reinforcement to the mortar. Sodium hydroxide flakes with 98% purity obtained from Taiwan and sodium silicate in the form of waterglass are used to form the alkali activator.

2.2. Parameters

The study follows the parameters of the optimum MS-FA geopolymer paste derived by Libre et al. (R.G. 2022): MS-to-FA ratio is to be kept at 1:5, NaOH-to-WG ratio at 1:2.5, water-solid ratio at 0.3, activator-to-precursor ratio at 0.38 and binder-to-aggregate ratio at 1:1. A randomized I-optimal design using Design Expert v11 (Design Expert® software, version 11) is used with two (2) factors: (a) bamboo fiber loading (%wt) by weight of specimen, and (b) bamboo fiber length in mm, for the response surface study. Table 1 shows the parameters of each factor used.

2.3. Experimental procedure

The experiment followed the procedures described by the standard test method of ASTM C780 for the preparation, casting, and testing of small cylinders, 50 mm in diameter and 100 mm in height. To start with, mass precursors of 1:5 MS-to-FA ratio were estimated in order to fill ten (10) small cylinders for each run. This is to provide five samples of each run for compressive tests and five samples for split tensile tests. In this study, 1600 g of FA, 400 g of MS, and 2000 g of sand are combined with varying% loading of short bamboo fibers; 0%, 0.5%, 1%, 1.5%, and 2% and varying fiber length of 10 mm, 20 mm, and 30 mm. The addition of the short fibers to the dry mix followed an interval of thirds to minimize the clumping of fibers. Using the constant activator-to-precursor ratio of 0.38 by mass, the mass of NaOH and waterglass for the activator solution was determined. The amount of water to be used was determined by the constant water-solid ratio of 0.3 by mass. The NaOH flakes were dissolved in the water and cooled down before mixing the waterglass and then continuously stirred for 5 min to produce the activator solution. The solution was mixed with the MS-FA dry mix for 8 min using an automatic mortar mixer. The resulting mortar was poured into small cylinders, with 10 replicates for each run. The cylinder molds were tamped and compacted to remove excess air. The cylinder samples were allowed to rest for at least 24 h before demolding. Cling wrap is used as the curing technique and the wrapped cylinder samples were left in an undisturbed area for ambient curing with temperature ranges from 34–38 °C, and relative humidity of 40±5%, for 28 days before testing.

2.4. Compressive strength and split tensile strength tests

After 28 days of curing, the geopolymer mortars were subjected to unconfined compressive tests and split tensile tests. The compressive test was administered using Shimadzu Universal Testing Machine (UTM) model AG-100kNXplus, displacement controlled at 5 mm/min rate. The split tensile test was administered using the same equipment at a 1 mm/min rate.

2.5. Response surface analysis and confirmatory tests

Two response parameters: (1) Unconfined compressive strength, and (2) split tensile strength, were recorded along with the two factors in a randomized design of experiment generated using Design Expert v11. The factors and responses are shown in Table 2.

3. Results and discussion

The results obtained from the response surface study and the characterizations described in the previous section are described in detail in the subsequent sections.

3.1. Factors affecting compressive strength

The resulting compressive strength of the mortar samples with respect to the factors; (a) bamboo fiber loading in%wt and (b) bamboo fiber length in mm, shows improvement after the addition of short bamboo fibers. Considering that the water-solid ratio was kept at 0.3 by

Table 1
Parameters of each factor.

Factors	Low-level	Mid-level	High-level
Factor A: Bamboo fiber loading (%wt)	0%	0.5%, 1%, 1.5%	2%
Factor B: Bamboo fiber length (mm)	10 mm	20 mm	30 mm

Table 2
Design data and results.

Mixture Sample	Bamboo Fiber Loading %wt	Bamboo Fiber Length mm	Ave. Compressive Strength MPa (std.dev.)	Aver. Split Tensile Strength MPa (std.dev.)
1	0%	N/A	0.856(3.151)	0.163(1.689)
2	0.5%	30 mm	2.967(9.432)	0.466(4.486)
3	1%	20 mm	3.348(13.205)	0.743(5.475)
4	1.5%	10 mm	2.423(18.179)	0.368(8.061)
5	1.5%	20 mm	2.658(11.942)	0.561(7.815)
6	1.5%	30 mm	3.333(8.979)	0.573(14.206)
7	2%	10 mm	0.542(12.185)	0.128(22.672)
8	2%	20 mm	0.638(2.903)	0.136(5.518)

Average value (Standard Deviation).

mass, and the NaOH-to-WG ratio at 1:2.5, which are the main factors affecting compressive strength for a low calcium fly ash-based geopolymer (Quiatchon et al., 2021), the response surface show that the factor that influences compressive strength is mainly the bamboo fiber length. Mixture sample S3 yields the highest compressive strength with 20 mm bamboo fiber length and 1% bamboo fiber loading with a 292.41% increase of UCS compared to samples without short bamboo fibers.

The typical failure mode observed for the tested cylinders was a columnar crack pattern for samples with short bamboo fibers and conical failure for the samples without fibers. The results of mechanical properties as well as the chemical tests may vary based on the consistency of the workmanship. Thus, practice runs were done to improve workmanship and minimize the clumping of the fibers that may lead to voids and weaker results.

3.2. Factors affecting split tensile strength

The response surface for the correlation between the resulting split tensile strength of the mortar samples versus the two factors are shown in Fig. 2 (Right). Following the results of split tensile tests, the governing factor that influences the increase on split tensile strength is still the fiber length. Mixture sample S3 still yields the highest strength with around 355.82% increase in strength compared to mortar without bamboo fiber and those samples with 10 mm bamboo fiber lengths. The bamboo fibers helped on holding the geopolymer matrix together as shown in Fig. 1.

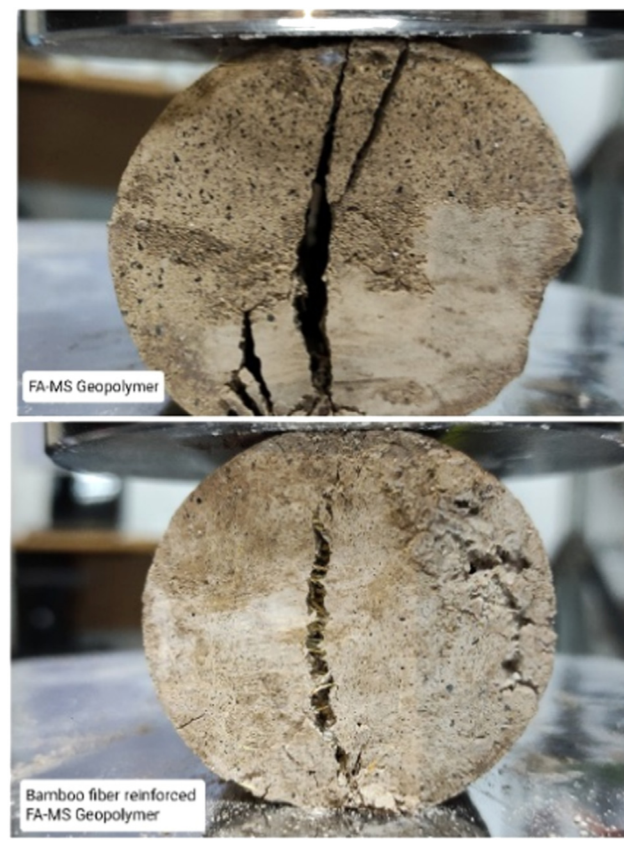

Fig. 1. Split tensile tests with and without bamboo fibers.

3.3. FTIR spectra

The FTIR spectra of the raw materials and selected geopolymer mortars are done with Bruker Tensor 27 FTIR with ATR using a transmittance mode range of 400–4000 cm^{-1}. The spectra shown in Fig 2.

The spectral peak in the range of 578 cm-1 that appears to all inspected materials is the bending vibration mode of Si-O-T bonds, where T is either Si or Al (Tigue et al., 2018).

It is notable that new stretching vibration peaks at 1398 and 1652 cm^{-1} appeared in curves of S1 and S3 compared to the curves of the raw materials. This can be attributed to the stretching of Si-O-C bond (Lu et al., 2022). The introduction of Si-O-C bond in the silica-aluminate network structure complicates the network and influences the increase in compressive strength.

The broad stretching between 3000 to 3400 cm^{-1} is related to the vibration of O-H groups and can be related to the presence of moisture in the samples (Zerzouri et al., 2021) while the shifting vibration at the range 900–990 cm^{-1} suggests the formation of calcium alumina silicate hydrate (Lekshmi and Sudhakumar 2022). The shift from 1024 cm^{-1} of fly ash sample to 970 cm^{-1} is an indication of geopolymer gel formation, thus affirming the occurrence of geopolymerization in the matrices (Samadhi et al., 2017).

3.4. SEM – EDX

The scanning electron microscopy with Energy Dispersive X-Ray analysis (SEM-EDX) is done with Thermo Scientific Phenom XL SEM.

The microstructure of the MS-FA geopolymers was observed as shown in Fig 3. The SEM of fibers treated with additional 10% aluminum sulfate shows rougher surface compared to fibers without treatment. This is important for the bondage between the fibers when mixed

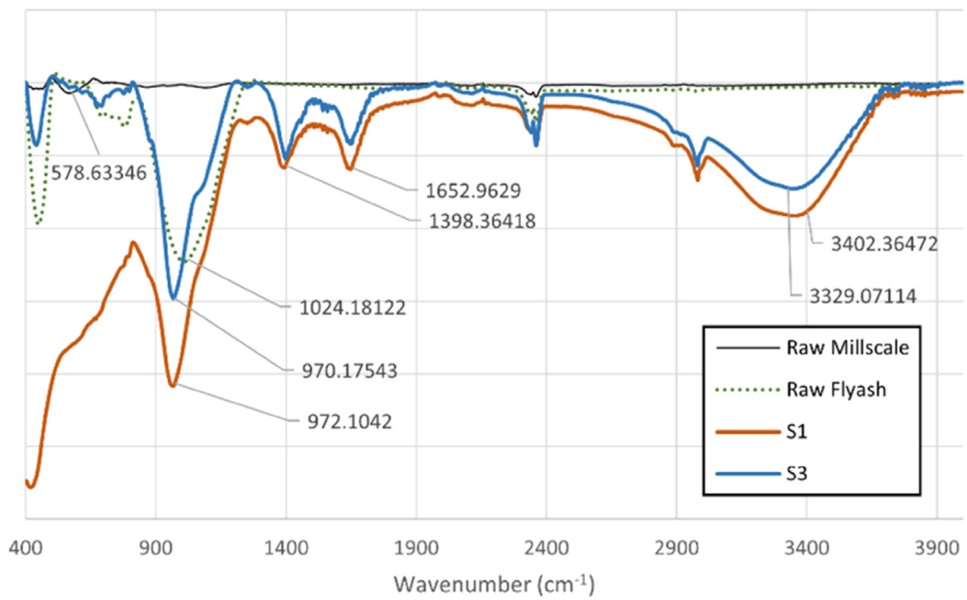

Fig. 2. FT-IR spectra of raw mill scale, raw fly ash, lower strength mortar (S1) and higher strength mortar (S3).

Table 3
EDX results of geopolymer mortars.

Element Symbol	Element Name	SampleS1	SampleS3
O	Oxygen	21.23	7.9
Si	Silicon	3.08	1.38
Na	Sodium	2.4	0.99
Al	Aluminium	1.21	0.48
Ca	Calcium	0.59	0.53
C	Carbon	-	2.29

Table 4
Summary of results for the two regression models.

Equation	Std. Dev.	R^2	F-Value	P-Value	Lack of fit
Eq 1	0.440	0.7945	6.19	0.0123	0.62
Eq 2	0.378	0.7946	6.19	0.0123	0.32

to the matrix. The corresponding EDX results of lower and UCS samples are shown in Table 3.

4. Confirmatory tests

To confirm the results gathered, regression models generated from the response surfaces are used to determine optimum bamboo fiber loading (%wt) and optimum bamboo fiber length in mm. The response surface for the correlation between the resulting compressive strength of the mortar samples versus the factors; (a) bamboo fiber loading in%wt and (b) bamboo fiber length in mm, are shown in Fig 4.

The regression model for compressive strength and split tensile strength is in the form of natural logarithmic after the model transformation suggested by the software. The summary of results is shown in Table 4 while the regression models are as shown:

$$\ln\text{(compressive strength)} = 0\ .8737 - 0.1516A + 0.1473B - 0.0955AB - 1.46A^2 + 0.3097B^2 \quad (1)$$

$$\ln\text{(split tensile)} = -0.6873 - 0.1602A + 0.0728B - 0.0443AB - 1.29A^2 + 0.1353B^2 \quad (2)$$

where A is the bamboo fiber loading in%wt and B is the bamboo fiber length in mm.

The models appear to have statistically acceptable F-values. P values that are greater than 0.1 indicate that the model terms are not significant. In our models, resulting P values are smaller than 0.05 which suggests that the model is significant thus the variables in the model do help to predict the specific response. Moreover, lack of fit for both models are not significant. Non-significant lack of fit is good as the model would likely to fit the actual data.

An optimized design mix was obtained by setting a target compressive strength equal to 3 MPa. The target should be in the range of the actual data set. This strength is comparable to the minimum requirement for mortar described by ASTM C270 or the standard specification for mortar, type O (2.4 MPa), which is used usually on plastering. Setting the factors bamboo fiber loading (%wt) and bamboo fiber length (mm) in the range of the original data set, while maximizing the possible resulting split tensile strength, the resulting optimum bamboo fiber loading is 1.4% and optimum bamboo fiber length is 20 mm. Confirmatory tests are done using the optimum values, resulting in average compressive strength of 3.08 MPa with a 5.7% coefficient of variance for the 5 replicates tested. This confirms the optimum values and the regression models used.

5. Conclusions

In this paper, the exploration of the use of low calcium fly-ash with mill-scale powder waste in geopolymer-making is presented. The performance of short bamboo fiber in reinforcing mill-scale – fly ash-based geopolymer mortars was investigated. The addition of the iron-rich waste and the natural fibers as reinforcement to the matrix maximizes the potential of the resulting geopolymer mortar in lowering of carbon footprint of construction. Response surface methodology (RSM) is used to optimize bamboo fiber loading (%wt) and bamboo fiber length (mm) with respect to its resulting compressive and split tensile strengths.

The optimum bamboo fiber loading of 1.4% and optimum bamboo fiber length of 20 mm are set to produce 3 MPa compressive strength, given the set parameters in this study. The addition of bamboo fibers increased the unconfined compressive strength up to 292.41% compared to specimens without bamboo fibers. The split tensile strength also improved by up to a 355.82% increase compared to control samples.

FT-IR was used to show the microstructure of the resulting lower and higher-strength samples geopolymer mix samples. FT-IR results showed the spectral peak in the range of 578 cm^{-1} as seen in all the inspected samples which is attributed to the bending vibration mode of Si-O-Si/Al

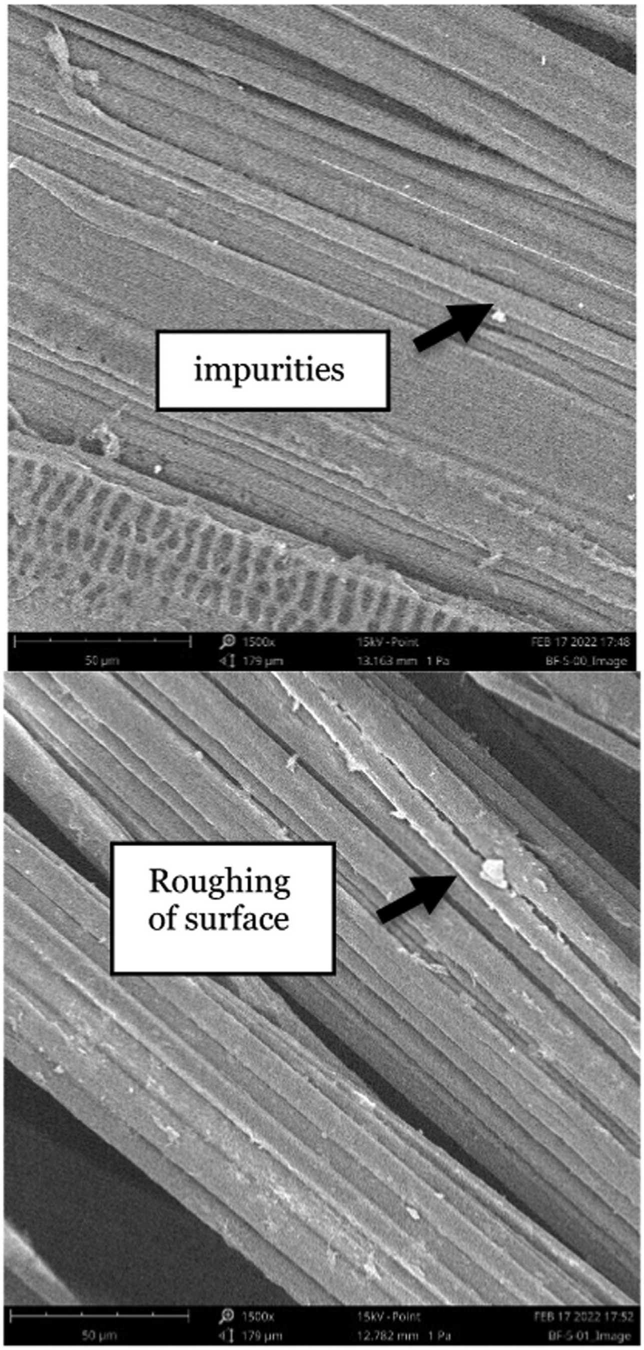

Fig. 3. SEM images bamboo fibers: (top) without aluminum sulfate treatment, and (bottom) with aluminum sulfate treatment.

bonds while shifting of 1024 cm^{-1} from the FA sample to 970 cm^{-1} of the geopolymers is a clear indication of the occurrence of geopolymerization. Notable new stretching vibration peaks at 1398 and 1652 cm^{-1} were observed in the resulting geopolymer mortars that can be attributed to the stretching of the Si-O-C bond, influencing the enhancement in compressive strength.

This study provided optimum values for developing low-calcium Class F Fly ash with mill-scale powder geopolymer mortar, reinforced by short bamboo fibers. The resulting short bamboo fiber-reinforced geopolymer mortar can be used for plastering applications in construction or strengthening schemes in retrofitting buildings, with comparable strength of at least 3 MPa, compared with the standard specification for mortars applied for plastering, type O (2.4 MPa) based on ASTM C270.

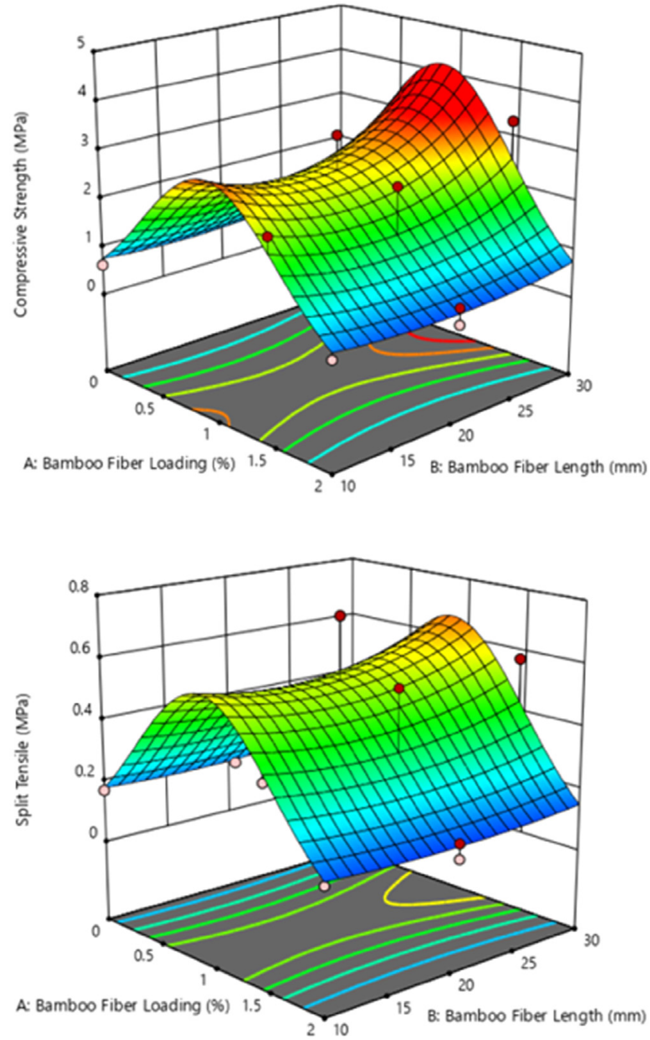

Fig. 4. (Top) UCS vs. bamboo fiber loading and bamboo fiber length; and (Bottom) Split tensile strength vs bamboo fiber loading and fiber length.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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