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Development of a sustainable flax fiber- geopolymer composite matrix incorporated with copper slag as a filler

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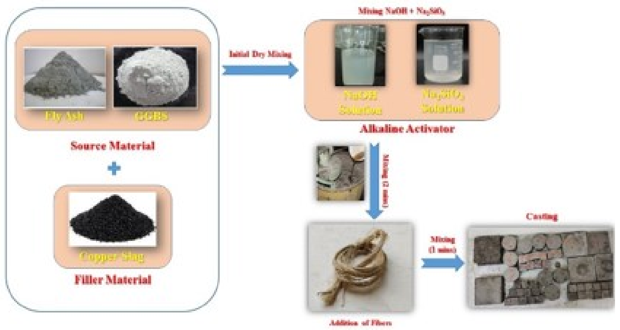
Graphical abstract



Abstract

Efforts have been put forth in the past to develop sustainable construction material by reducing the use of cement and river sand which can directly provide a positive impact on our ecosystem. In this work, an attempt is made to develop sustainable geopolymer composites with the use of fly ash (FA) and Ground Granulated Blast Furnace Slag (GGBS) as precursor binder and copper slag as a complete replacement for conventional fine aggregate. In addition, discrete natural fiber such as flax is added to the composite matrix to provide sufficient tensile strength and better bonding capabilities. The fiber volume fractions were fixed as 0.25%, 0.50%, and 0.75% to the total volume of the composite matrix. All the cast specimens undergo an ambient curing process to complete the polymerization mechanism for strength gain. Results reveal that the geopolymer composite mix with 0.25% of flax fiber was found to outperform the other mixes based on the enhancement in mechanical strengths. Moreover, the addition of fibers of more than 0.5% results in the marginal reduction of compression and flexural tests which could be due to improper fiber dispersion or fiber bundling at specific locations leading to the absence of required fibers at the fracture plane. In addition to the mechanical tests, a detailed micro-structural analysis was performed using scanning electron microscopy (SEM) to understand the interface between the fiber and the matrix.

Keywords: Activator, Copper Slag, Micro-structural Analysis, Flax Fiber, Geopolymer.

1. Introduction

Cement production is a major source of greenhouse gas emissions, consumes lots of energy and also contributes to water pollution and air pollution (Oyebisi *et al.* 2019). In developing countries like India, infrastructure is essential for economic growth. As the population increases, the demand for housing and other infrastructure projects also increases (Lepech and Li 2008). This in turn creates a large demand for basic construction raw materials, such as cement, sand, and coarse aggregates. These materials are also used to enhance the residing structure, making it more comfortable and durable (Singh *et al.* 2023a,b; Lăzărescu *et al.* 2017). Concrete, being the predominant construction material used worldwide, contributes significantly to environmental issues due to the substantial emission of greenhouse gases during its production and manufacturing processes (Kugler *et al.* 2022; M. Naveen Saviour 2012). Apart from the aforementioned concern, the utilization of aggregates as filler material in concrete, which involves mining activities, leads to a severe disruption of the ecological balance in the surrounding environment (Sudarvizhi and Ilangovan 2011; Waqas *et al.* 2021; Maheswaran *et al.* 2022; Hari *et al.* 2022; Rahul *et al.* 2022). To address these problems and mitigate greenhouse gas emissions, it is crucial to identify an alternative for cement-based concrete which has low environmental impact and offers superior suitability (Arunachelam *et al.* 2022).

Slags from various industries are one of the major issues for them to handle and dispose of safely. Disposal of this generated slag causes major environmental concerns in terms of land, air and groundwater pollution and also changes the land use pattern and ecology of the disposal site. The slags from the industries are majorly classified as ferrous and non-ferrous slags. Ferrous generation of one slag includes blast furnace slag, Basic Oxygen Furnace (BOF) Slag and Electric arc furnace (EAF) slag, whereas Non-Ferrous metal includes copper slag, Lead slag, Zinc slag and

Nickel Slag are generated from their ores during the smelting process. India stands in the second position in steel manufacturing in the world and it generates 19 million tons of steel slag as a solid waste. During the process of iron ore containing around 60 to 65% iron in a blast furnace for tons of pig or crude iron, about 300 to 540 kg of slag is generated. It was reported that around 20 to 30% of steel slag is generated by the mass of the crude steel output in the Indian country every year. The steel industry in India is producing about 24 million tons of blast furnace slag and 12 million tons of steel slag annually. It is expected that the BF slag generation may reach around 45 to 50 million tonnes and BOF slag around 15– 20 million tonnes per year by 2030. Copper slag an industrial by-product generated from the smelting process during copper manufacturing been used as fine aggregate in the conventional concrete (Arunachelam, Maheswaran, Chellapandian, and Ozbakkaloglu 2022; Brindha and Nagan 2011; Khanzadi and Behnood 2009; Rajasekar, Arunachalam, and Kottaisamy 2019; Resende, Cachim, and Bastos 2008) and in GPC also copper slag was used as fine aggregate (Arunachelam *et al.* 2022; Mahendran and Arunachelam 2016; Mithun and Narasimhan 2016; Singh and Singh 2019; Yan *et al.* 2021). The properties of copper slag resemble similar to that of the conventional river. Hence, it can be used as a fine aggregate for the production of concrete (Brindha and Nagan 2010). Moreover, copper slag being a fine material possesses potential pozzolonic properties and hence contains the potential to aid in the polymerization process when used in the production of GPC.

Joseph Davidovits initially introduced “geopolymer technology” as an environmentally friendly approach that harnesses industrial wastes and by-products, combined with an alkaline solution, to provide a binding property (Sajan *et al.* 2021). This geopolymerization process differs from Ordinary Portland Cement chemistry(Bell *et al.* 2008; Davidovits and Resins 1980). Various source materials rich in silica and alumina such as fly ash, Metakaolin, Ground Granulated Blast Furnace Slag, Red Mud, and Metakaolin materials are used in the production of Geopolymer concrete (GPC) (Abbass, Singh, and Singh 2021; Part, Ramli, and Cheah 2015). In certain instances, a small portion of calcium-rich materials may be substituted for the source materials in order to enhance the geo-polymerization process (Kim *et al.* 2022). This substitution leads to the generation of hydration products that are not classified as polymers and are commonly referred to as C-A-S-H (calcium-alumina-silicate-hydrate)(Jamil *et al.* 2020). The main advantages of using geopolymer are early high strength, and higher resistance to acid or extreme chemical exposures (Jaarsveld and Deventer 1999; Van Jaarsveld, Van Deventer, and Lorenzen 1998). The curing has been a crucial part in the geopolymerization process and is the only factor limiting its widespread practical applications. Most of the research reported that GPC cured at 40ºC to 80ºC gained optimum compressive strength within 6 hours due to the polymerization process(Kanagaraj *et al.* 2023). The heat curing imparts additional strength but has a negative impact on sustainability issues such as heating consuming a huge amount of energy which leads to additional costs resulting in limited applications towards the sustainable concept (Al-Majidi *et al.* 2017).

Apart from these issues, few researchers reported that GPC has a shrinkage deformation due to the loss of free water which was been added for the workability of the geopolymer matrix, which may be dissipated during the hot air curing (Deb *et al.* 2015). Upon loading these shrinkage deformations makes them brittle and develop cracks in the matrix and this can be mitigated by introducing fibers in the matrix which also reduces the brittleness of the composites. The choice of fiber depends mainly on various factors such as compatibility, fiber matrix interaction and effective post-cracking behaviour. In focus on the sustainability of composite manufacturing, natural fibers are preferred in this study, as artificial fibers incur a certain amount of energy during the manufacturing process resulting in CO_2 emission. Incorporating fibers into concrete results in several key enhancements in its engineering characteristics, including increased strain hardening beyond the peak load, improved fracture toughness, and enhanced resistance to both fatigue and thermal shock (Soutsos *et al.* 2012). Keeping in view of these issues, in this study, the influence of various levels of flax fiber on the geopolymer composites in terms of strength through compression, flexure, and impact resistance was studied and reported. In addition to these tests water absorption tests and exposure to elevated temperature were also performed to study its durability performance. To improvise the strength and to accelerate the Strength gain in ambient conditions, GGBS was replaced in the geopolymer concrete by 30% in place of the fly ash. Recent studies indicate that blast furnace slag, a by-product of the steel industry, is frequently utilized in combination with fly ash as a binder during geopolymer preparation to make the concrete gain strength in ambient curing conditions (Giasuddin *et al.* 2013; Huseien *et al.* 2016).

2. Research significance

Large scale use of cement-based composites requires a high amount of energy and emits a larger amount of carbon dioxide during its manufacturing stage, which contributes to the greenhouse effect. To reduce the ill effects of cement-based composites, geopolymer composites can be used as a potential alternative. The objective of the present study is to understand the mechanical and micro-structural characteristics of sustainable natural fiber reinforced geopolymer composites through the ambient curing process.

3. Materials and methods

The materials used in the study are Fly ash, GGBS, Copper Slag, Sodium Hydroxide (NaOH), Sodium Silicate (Na_2SiO_3) and Flax Fiber. The material properties such as specific gravity, fineness modulus, chemical composition, and morphology were studied for the preparation of the composites.

3.1. Materials used

The Fly ash used in the study was collected from Tuticorin Thermal Power Plant (TTPS) and commercially available GGBS and natural fiber Flax were procured from the local vendor with fiber tensile properties of 1100 MPa and its average diameter of 10µm and length of 20mm to enhance the flexural properties [Kumar *et al.* 2022; Chellapandian *et al.* 2023].

3.1.1. Alkaline activator

The laboratory grade NaOH and Na_2SiO_3 was used in the manufacturing of Geopolymer composites with a molarity of 10M NaOH. It was prepared from the deionized water. Na_2SiO_3 with a modulus ratio of 2.5 (SiO_2/Na_2O, SiO_2 = 30% and Na_2O = 12%).

3.1.2. Fly ash and GGBS

The microstructure of the fly ash and the GGBS was identified through the SEM micrographs and it was presented in Figure 1. The particle shape of the fly ash was found to be spherical of different shapes, whereas the GGBS looks angular and its average size was found to be 10µm. along with SEM analysis, XRF was also performed the elemental results are presented in Table 1. The traces found in the fly ash were silica and alumina and in GGBS, silica, calcium and alumina are the major traces in the analysis. In addition to EDAX analysis, the fly ash and GGBS were studied for their mineralogical characteristic through XRD analysis and its spectrum is presented in Figure 1b). At the degree 26º (2-theta) the sharp crystalline peaks show the quartz and mullite mineral and other amorphous peaks like hematite and Magnetite were found between the range 14º and 30º in fly ash (Arunachelam *et al.* 2022). The GGBS used in the study has a fineness of 360 m^2/kg with a specific gravity of 2.9. The oxide present in the GGBS was Calcium which has a percentage of 42.24 and the next to is silica with 35.20 % [Maheswaran *et al.* 2022; Chellapandian *et al.* 2023; Maheswaran *et al.* 2023].

3.1.3. Copper slag

The black glossy granular generated as a by-product from the copper industry during copper manufacturing was used as an alternative for sand in composite manufacturing. The microstructure of the structure shows the slag was angular in structure also the surface of the slag looks rough which was clearly seen from the micro images of the copper slag presented in Figure 2a. Also, it was worth mentioning that the copper slag has cementation characteristics when it was exposed to the alkaline solution (Mithun and Narasimhan 2016). XRD analysis of copper slag shown in Figure 2b revealed its amorphous nature and highlighted the absence of distinct major peaks in the material.

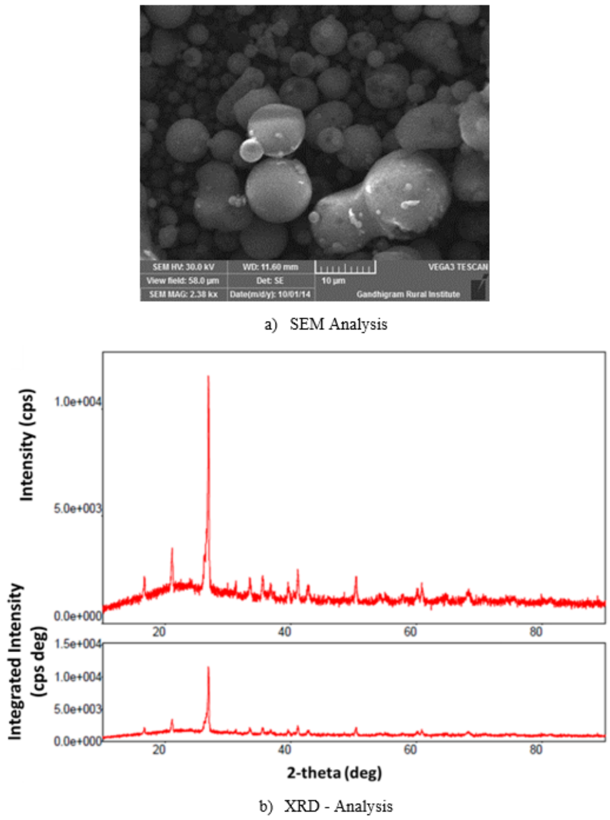


Figure 1. XRD analysis and SEM Micro Image of fly ash

Table 1. Oxide Composition of Fly Ash and GGBS using XRF analysis

Details of Oxide	Fly Ash	GGBS
Silica	69.6	35.20
Alumina	14.8	10.70
Iron oxide	3.30	0.33
Calcium oxide	4.50	42.24
Sodium oxide	4.70	0.15
Magnesium oxide	1.60	8.4
Sulphur trioxide	0.50	2.85

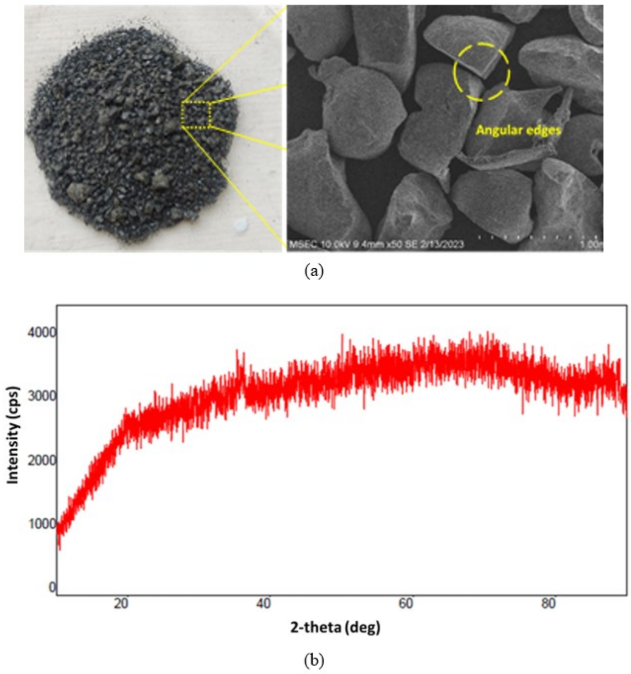


Figure 2. SEM and XRD analysis of Copper Slag

The properties of the slag such as specific gravity, water absorption, bulk density and fineness modulus were found to be 3.89, 0.4%, 2.20 g/cc and 2.64 respectively. The distribution of various sizes of particles was analysed using sieve analysis as per the IS 383 confirming the zone II and the gradation curve was presented in Figure 3.

3.1.4. Flax fiber

Flax fiber is a natural fiber, extracted from the inner bark of the linseed plant. They are available around regions of temperature not exceeding 25˚ during its growth. the flax fiber for the research was procured from Fiber Region, Chennai, India and all its physical properties were tested as per ASTM D3822M- 14 and reported in the companion paper of authors (Chellapandian *et al.* 2024; Chellapandian, Maheswaran, and Arunachelam 2024) The fiber had a density of 1.5 g/cc and it's maximum elongation was about 1.2%. The tensile strength and the Young's modulus of the fiber was 959.6 MPa and 107.6 GPa, respectively. The SEM micro images of flax fibers are presented in Figure 4. The microstructural analysis reveals the presence of bio-degradable contents such as cellulose, hemicellulose, and lignin thereby making them less efficient when used in structural applications [Kumar *et al.* 2022; Chellapandian *et al.* 2023]. The flax fibers were added in the percentage of 0.25%, 0.5% and 0.75% to the volume fractions added in the composite and compared with the control composite specimens to study its characteristics. The fiber dosages were fixed based on the preliminary investigation in which any dosage beyond 0.75% resulted in fiber-concrete balling effect. (Arshad *et al.* 2020)

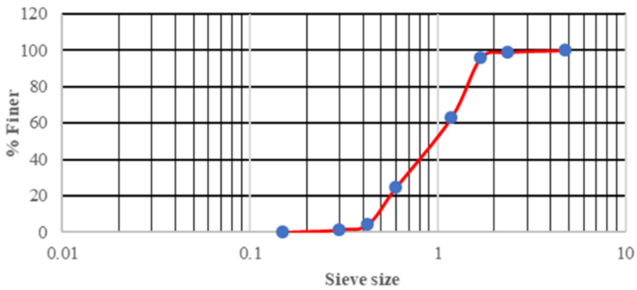
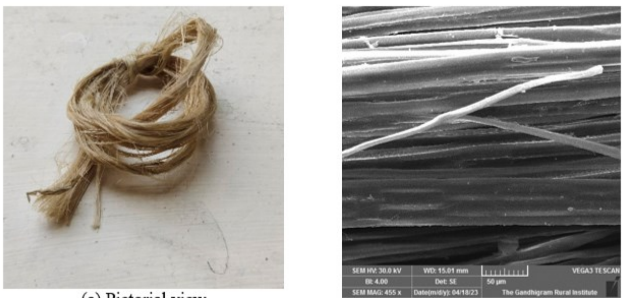


Figure 3. Particle size distribution graph of Copper Slag



(a) Pictorial view (b) SEM image

Figure 4. Representation of flax fiber used

4. Experimental program

4.1. Specimen preparation

The cube specimens of size 70.6 x 70.6 x 70.6 mm which confirms to IS 4031 (Part 6) -1988 were cast to study the compressive strength, exposed to elevated temperature and water absorption. The tile specimen of size 250 x 250 x 10 mm confirms to the IS 1237-2012 was cast to study the flexure strength. The disc specimen of cylindrical size 150 mm x 63 mm was used to determine the impact resistance of the geopolymer composites in accordance with standard ACI 544-2R.

4.2. Mixing, casting and curing.

The specimens were cast with the ratios of fly ash to copper slag as 1:2 to the required volume for the specimen. A 10M NaOH solution was used during the mixing process, and the required quantities of NaOH flakes were dissolved in the distilled water and left for 24 hours to cool down(Nath, Sarker, and Rangan 2015). After the cooling period, 1 part of NaOH solution was mixed with 2.5 parts of Na_2SiO_3 and stirred for two minutes to get the homogenous alkaline liquid. The schematic diagram for the preparation of the geopolymer composite matrix is presented in Figure 5. The mortar mixture was used to mix the solids and liquids to get the composites, initially the fly ash and GGBS was dry mixed thoroughly, followed with the introduction of copper slag and the activator solution was slowly added into the mix and the mixing continued until the uniform mix is obtained. After the addition of liquids, the fibers were scattered uniformly (Ranjbar *et al.* 2016). The prepared mix is then transferred into the designated moulds. The cast specimens were then transferred and kept for curing under ambient curing conditions.

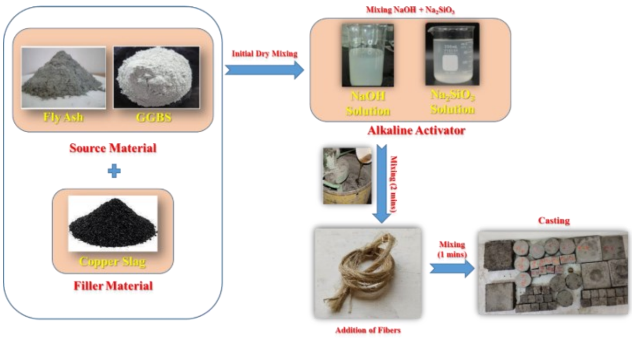


Figure 5. Procedure for the development of geopolymer composites

5. Results and discussion

5.1. Compressive strength test.

The experimental work mainly evaluates the influence of flax fiber content in the geopolymer composites, the flax fibers were added in composites with a percentage of 0.25%, 0.5%, and 0.75% and the result values are compared with the control specimens. The compressive strength results for this various percentages of fibers content are presented in Figure 6. For the test results it was observed, that the mix with 0.25% flax fiber had a maximum compressive strength of 60.04 MPa on the 28th day which was 21% higher when compared to the control specimens. Followed with 0.5% fiber content attains a compressive strength of 50.27 MPa. It was also observed that when the fiber content starts to increase the strength starts to reduce, this may be due to the reduced binder volume which was insufficient to hold the fibers with one another resulting in reduced load-bearing values of composites. Also, the influence of copper slag was there in the strength enhancement, by reacting with the alkaline solution the outer layer of the slag gets polymerized and forms a strong bond between the materials resulting in the higher compressive strength.

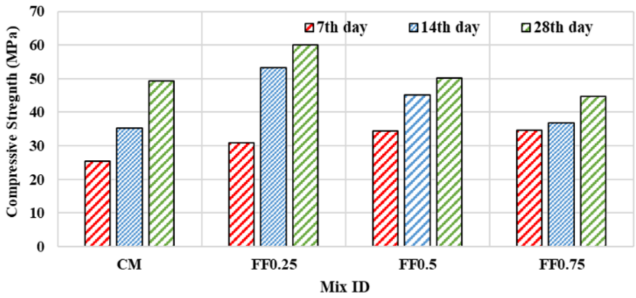


Figure 6. Comparison of compressive strength test result

It was observed during the testing control specimen that micro cracks developed readily around the weak spots like pores initially and propagated in larger areas whereas in the fibrous concrete initiation of cracks and propagation was greatly reduced. When compared to the control specimen, FF0.25 exhibits an increased compressive strength of 21.88% which was due to the confining effects and fibers bridging thereby require the higher amount load to rupture the matrix (Hedjazi and Castillo 2020). It was also noted that failure of the specimen was ductile instead of brittle except the control specimens and this attributed to the addition of fibers which help to carry the tensile stress in the specimens resulting in the ductile behaviour. When the percentage of fibers increases beyond 0.25% there is a reduction in the compressive strength caused due to the poor void regions and micro-pores resulting in the possible fracture plane for the matrix (Smarzewski 2018). Also it was reported that increased addition of fibers reduces the ability of the matrix to infuse in between the fibers resulting in weaker interface for possible region of crack developments (Bayraktar *et al.* 2023).

5.2. Flexural strength test

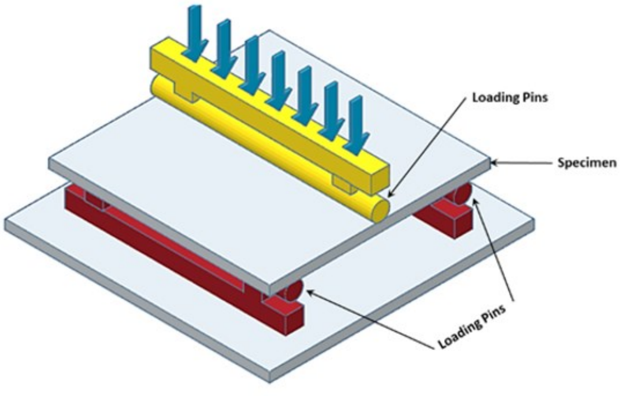
Flexural strength is the major character for the composites as it helps to study the anti-cracking behavior of the composites under bending load (Moujoud *et al.* 2023). For understanding the flexural strength of geopolymer composite matrix, the specimens in the form of tiles were prepared of size 250 mm (width) x 250 mm (length) x 10 mm (thickness). The uniform face of the tile is used for the load application to ensure proper load transfer whereas the cast face was used for placing over the supports. The test protocol was performed following the Indian Standards. The test setup used in this study is presented in the schematic form in the Figure 7a. The actual image of the testing along with the test specimen is depicted in Figure 7b. The test was continued till the complete failure of the tile specimens.

Figure 8 represents the flexure test results for the specimen having different percentages of fibers. It was observed that the control specimen achieved a flexural strength of 2.45 MPa, whereas the composites having 0.5% flax fiber achieved a max value of 3.56 MPa which was 45% higher compared to the control specimens. It was also observed that specimens with 0.25% and 0.75% fibers have a marginal variation when compared with 0.5% fiber specimens. On compared to the compressive strength results, the flexural test follows the same behavior. The increase in the flexure strength was mainly due to the fiber bridging in the matrix which increases the tensile strength of the Geoploymer matrix. The enhancement in the tensile strength is mainly due to the formation of C-A-S-H and N-A-S-H gel in the composite's matrix which helps to hold the fiber in a stronger position (Kanagaraj *et al.* 2023). The inclusion of GGBS holds the responsivity for the formation of C-S-H, C-A-H/C-A-S-H gel, as it has Ca in the source material which reacts with catalysts and produces it (Mishra *et al.* 2022).

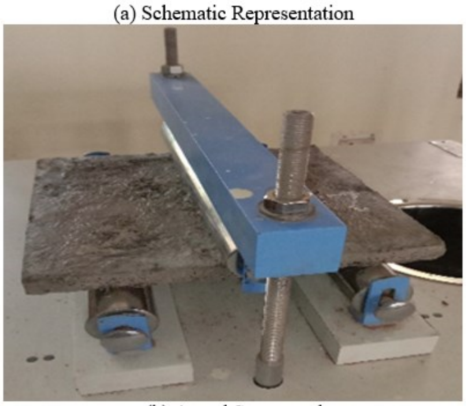
The surface of fiber plays a major role in improving the bonding, as the binder matrix holds the fiber in position makes the composites ductile along with the addition gel formation from the Ca present in the GGBS enhancing the alkaline reaction (Nath, Sarker, and Rangan 2015). Compared to the other natural fibers, Flax fiber has higher stiffness value which could prevent the propagation of the cracks along the failure plane by releasing the stress and enhance the flexural strength of the composites (Rehman *et al.* 2020). It was also noted from the test results, even though decrease in strength due to addition of fibers by 0.75% the increment in the strength when compared to the Control mix, was 40%. The reduction in strength was due to uneven distribution of the fibers in the composite matrix as the volume of fiber content increased when the fiber content of 0.5% in the composite matrix (Bayraktar *et al.* 2023). The increase in the flexural capacity due to the fiber content and orientation of the fiber helps to increase the load-transferring capability and makes the concrete ductile. The higher tensile strength of the flax fiber helps to absorb the larger load compared to the control specimens.

5.3. Water absorption.

Figure 9 depicts the water absorption test result for the composites. The water absorption percentage for all the specimens was less than 5%. The absorption value for the control mix was found to be lowest as 1.15% and the highest value was found on the mix having 0.75% fiber was around 3%. Compare to Ka-based geopolymers, Na has a better resistance against water resistance which might be the reason for the reduced water absorption in the composites (Rehman *et al.* 2020). This increase in percentage of water absorption was due to the presence of natural fibers and increased pores in the specimens.



(a) Schematic Representation



(b) Actual Setup used

Figure 7. Flexural Strength Test on Composite Tiles

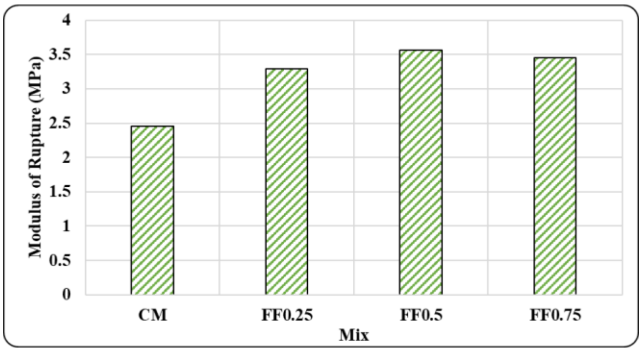


Figure 8. Comparison of Flexure Test Results for Geopolymer Composite Tiles

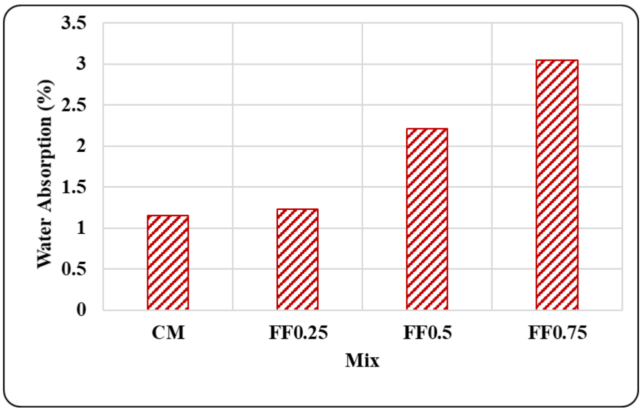


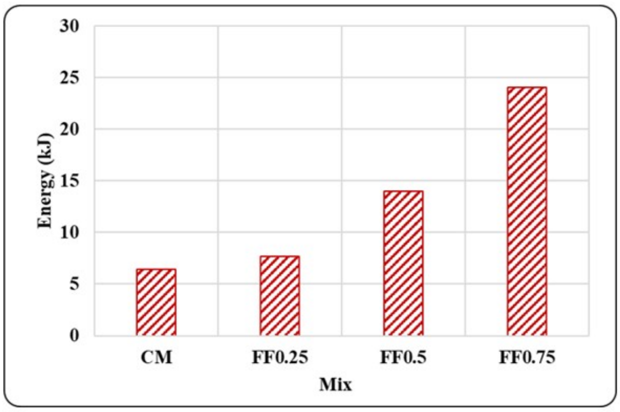
Figure 9. Results of water absorption test for Geopolymer Composite Samples

Flax fiber this induces the increased water absorption and it directly affects the strength properties of the prepared composites. The mechanism happening behind the increased water absorption characteristics was due to the hydrophilic nature of the flax fiber, which causes the fiber to absorb more water. The higher cellulose content in the fiber makes the concrete absorbs extra water (Deepa Raj and Ramachandran 2019; Dev, Chellapandian, and Prakash 2020). Even though the water absorption was higher for FF0.75 mix when compared to the Control mix the limit of water absorption percentage was within the limit prescribed by the standards considered. Another possible reason for the increased water absorption was due to shrinkage behavior of the geopolymer composites which may create a weak contact between the matrix and the fiber surface by enlarging the gap making the water to contact in a larger manner to absorb more (Ranjbar *et al.* 2016).

5.4. Impact test

One of the main aims of the fiber addition is to provide excellent resistance against shock or vibration loading. Hence, the impact test was performed to ascertain the number of blows required to cause failure on the natural fiber reinforced geopolymer composite specimens. The impact tests were done on the standard disc specimen of diameter 150mm and 63mm thick cylindrical specimens. The standard mass was dropped on the specimens and the number of blows where the specimen gets fractured was noted and the energy was calculated. The impact test results are presented in the Figure 10. And it was observed that the specimens with max fiber content has the maximum energy values of 24.05 kJ which the specimen failed at 1210 blows, whereas the control specimens fail at 280 blows with an energy of 6.4 kJ.

(a) Failure mode



(b) Comparison of energy absorption

Figure 10. Result of the Impact Test and failure mode comparison

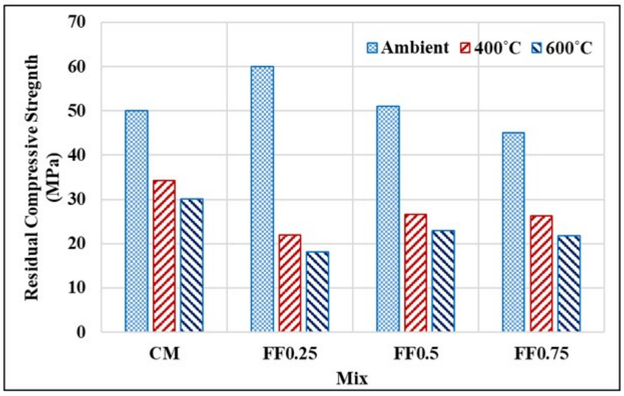
It was noted that the increase in fiber content increases the specimen's impact energy. This increase in energy was due to the interlocking behaviour of the fiber in the matrix. Geopolymer matrix, these prevent the disintegration of the fibers and separation from the matrix which strongly holds the fibers. Despite having higher energy absorption, composites with stronger binders exhibit a lower deformation capacity. However, the introduction of fibers mitigates this decrease in deformation capacity without impacting dynamic compressive strength (Vilaplana *et al.* 2016). The high energy absorption was due to the characteristics of the fiber mechanism such as fiber pull-out, breakage and delamination between the matrix and fiber (Moujoud *et al.* 2023). These characteristics of the fiber also reduce the crack formation by bridging it with the matrix and preventing the propagation of the cracks on the load increment (Alomayri, Shaikh, and Low 2014).

5.5. Elevated temperature testing

The geopolymer cube specimens were exposed to 400˚C and 600˚C in the furnace to understand the reduction in compressive strength after thermal exposure. The specimens exposed to elevated temperature are compared with the control specimen tested at ambient temperature and presented in Figure 11.

The control GP matrix with no fibers showed a reduction in compressive strength of 30% i.e. 50 N/mm^2 to 35 N/mm^2 (30%) at 400°C exposure. Similarly, at a temperature exposure of 600°C for two hours, a strength reduction of 40% was witnessed when compared to the control specimen. (from 50 N/mm^2 to 30 N/mm^2) when subjected to 400°C and 600°C respectively. Compared to the control specimens, the GP composite matrix reinforced with natural flax fibers showed a higher reduction in strength. This observation could be due to the degradation of the fibers at higher temperatures. This disintegration of fibers makes reduction in the volume at the fiber place and makes the plane weaker initiating the failure of the concrete. The FF0.25 specimen strength has reduced from 60.04 MPa to 41.92 N/mm^2 at 400˚C and 35.1 N/mm^2 at 600˚C respectively. The specimens exposed above the 400˚C, the mass loss was due to the evaporation of free water and bonded water present inside the specimen. Similarly, with the addition of 0.75% flax fiber, a reduction in compressive strength from 45 N/mm^2 to 27.0 N/mm^2 (40%) and 22.5 N/mm^2 (50%) were observed when subjected to 400°C and 600°C respectively.

(a) Actual Test Setup

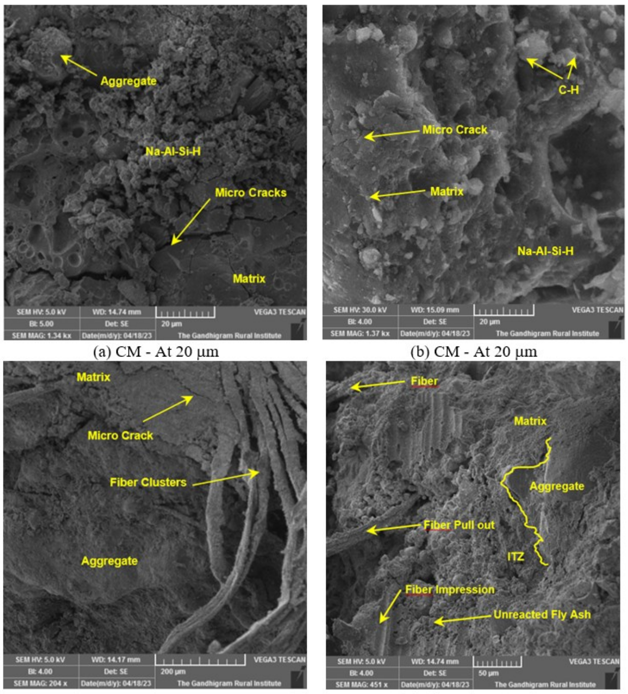


(b) Comparison of different mixes

Figure 11. Elevated temperature testing of geopolymer composite samples

5.6. Micro structural characterization

The microstructural analysis performed through scanning electron microscope (SEM) of the geopolymer composite samples with different combinations of fibers is presented in Figure 12. From the SEM images, several distinct characteristics such as dense Sodium-Aluminium-Silicate-Hydrate (Na-Al-Si-H) gel, anhydrous calcium hydroxide (C-H), unreacted fly-ash particles, etc. can be traced out. From Figure 12d, the reaction between the activator and the source material was evident through the formation of a dense homogenous matrix leading to a better bond mechanism. Another interesting observation from the micro-structural study is the formation of an interfacial transition zone (ITZ). The thickness of the ITZ which acts as a layer of separation between the aggregate (copper slag) and the matrix acts as an important factor to judge the bonding between the two different surfaces.



(a) CM - At 20 μm (b) CM - At 20 μm

(c) Flax GP Matrix -At 200 μm (d) Flax GP Matrix At 50 μm

Figure 12. SEM Micrographs of geopolymer composites with and without fibers

As seen in Figure 12(c), the thickness of the ITZ between copper slag aggregate and binder matrix is dense enough to justify the excellent bonding and the same was reflected from the results of mechanical characterization tests. Another important observation from Figure 12(a) is the presence of un-reacted fly ash particles which could be spotted from their characteristic spherical shape (Arunachelam, Maheswaran, Chellapandian, Murali, *et al.* 2022; Maheswaran *et al.* 2023; Vignesh, Mahendran, and Arunachelam 2020). The unreacted fly ash present even after 28 days of curing denotes the reduced level of polymerization reaction as a result of the ambient-curing process. However, these unreacted fly ash particles may not be present when visualized after allowing a longer curing time of more than 56 or 90 days.

To understand the efficiency of natural fibers in improving the performance of the geopolymer composite matrix, it can be seen from Figure 12(b) that the fibers were pulled out during the loading of the specimens and also few slippages in the fibers were evident through the fiber impression. The presence of fiber in the matrix contact, bridging and pull which characterize the energy absorption behavior of the matrix. The high energy absorption creates branching of cracks, deflection and debonding of the fibers from its position. Hence, it can be concluded that the fiber-matrix interface was strong enough confirmed by the presence of matrix over the surface of the fibers without showing any delamination. Figure 12(c) represents the presence of dense Sodium-Aluminium-Silicate-Hydrate (Na-Al-Si-H) gel formed as a result of the polymerization reaction by the ambient curing method. In addition to the Na-Al-Si-H gel, several white crystals of anhydrous calcium hydroxide ($Ca(OH)_2$) can be traced which could be due to the presence of calcium from the GGBS binder. These white crystals are unstable and may react further to form Calcium-Alumino-silicate (C-A-S-H) gel. From Figure 12(d), several micropores can be spotted between the reacted alumino-silicates. However, the diameter of the pores was too small even in the micro-level scales. Another characteristic feature spotted from the SEM image is the agglomeration of the reacted fly ash is evident from the SEM analysis. In addition, a major micro-crack can be spotted which corresponds to an average width of 2.01 μm. This micro-cracking could be due to the stressing of the specimen under compression loading which creates a plane of fracture.

5.7. SPM analysis for 3D surface representation

The SEM images of the geopolymer composite matrix reinforced with 0.25%, 0.5% and 075% natural flax were further processed using the scanning probe microscopy (SPM) analysis to visualize the three-dimensional (3-D) surface and measure the roughness. Understanding the 3-D surface texture of the geopolymer composite matrix is highly essential to quantify the change in surface roughness due to the addition of different types of fibers. The scanning probe microscopy (SPM) analysis uses an indirect image profilometry method for identifying the coefficients of surface roughness (Pavlović, Risović, and Novaković 2012). Table 2 depicts the details of the surface parameters such as roughness and skewness obtained for different fiber reinforced geopolymer composite matrix.

Table 2: Comparison of 3-D profile parameters for Fibre-reinforced GP Composites

Specimen Details	R_q (μm)	R_a (μm)	R_q/R_a	R_{sk}	Max peak height (μm)	Max height R_z (μm)
0.25% Flax	5.06	3.88	1.304	0.345	29.0	57.6
0.50% Flax	4.24	3.30	1.284	0.665	32.2	46.5
0.75% Flax	4.18	3.03	1.379	1.069	29.3	47.8

From the table, it can be observed that there are four important parameters such as the R_q, R_a, R_{sk} and R_q/R_a. The parameter R_q characterizes the root mean square roughness i.e., the standard deviation of the peak profile from the average roughness value. The second parameter R_a helps in correlating the average roughness value. The third parameter R_{sk} represents the skewness of the surface profile above the mean plane. From the previous study, it was reported that the negative value of R_{sk} indicates the occurrence of valley depth over the surface heights. Otherwise, the positive value of R_{sk} indicates the presence of surface heights. The last important parameter is the R_q/R_a ratio which corresponds to the statistical distribution of peak heights along the sectional profile. For achieving the Gaussian distribution of surface peak heights, the value of R_q/R_a ratio must be less than 1.5 (Shen *et al.* 2021).

Figure 13 depicts the three-dimensional (3-D) surface pattern of geopolymer composite matrix reinforced with 0.25%, 0.5% and 075% natural flax fibers. From the results of 3-D profilometry, it is significant that the use of a high fiber volume fraction of 0.75% results in the reduction of the roughness coefficient (R_q). In specific, the R_q values of 0.25%, 0.5% and 075% natural flax fiber reinforced geopolymer matrix were 5.06, 4.24 and 4.18 respectively. corrosion of steel specimens resulted in the reduction of root mean square roughness and average roughness values (R_a). The reduction of average roughness values from 3.88 to 3.30 and 3.88 to 3.03 for the 0.5% and 075% natural flax fiber reinforced specimens respectively when compared to the flax fiber reinforced one. The R_q/R_a ratio is positive for all the specimens analyzed which indicates the presence of surface heights over the valley depth. This also confirms the minimum porous nature of the matrix and most of the surface is above the surface plane.

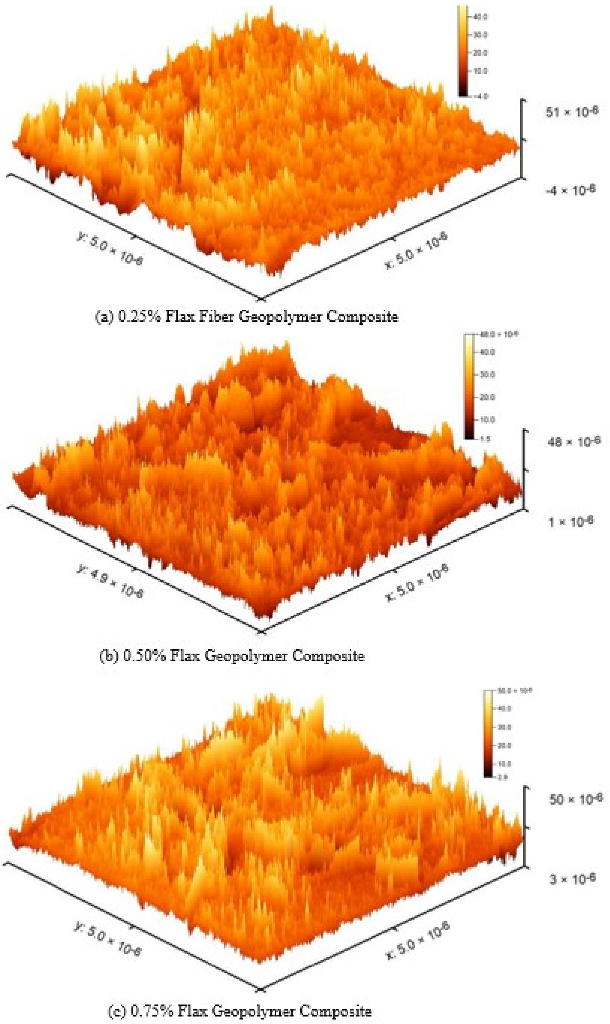


Figure 13. 3-D profile view of geopolymer matrix with different types of fibers

6. Summary and conclusions

The paper presents an experimental camping on the use of natural flax fibers in different volume fractions for the production of a geopolymer composite matrix. The novel portion of the work lies in the complete replacement of natural river sand with copper slag as the fine aggregate. The composite specimens were tested for their mechanical as well as micro-structural characteristics. From the results obtained, the following conclusions can be derived:

The compressive strength of the geopolymer composite matrix showed an increase of 21% when the flax fiber volume fraction was about 0.25%. With the further increase in the fiber volume fraction, a marginal increase in the value of compressive strength can be witnessed.

Similar to the compressive strength, the value of flexural strength increased up to a value of 45% for the flax fiber volume fraction of 0.5%. However, with the higher fiber dosage, the flexural strength is reduced marginally due to improper fiber distribution or the presence of low fiber dosage in the fracture plane.

Due to the addition of natural fibers, the percentage of water absorption increased from 1% to 3% when compared to the control GP specimen. This increase can be attributed by the hydrophilic nature of the natural fibers due to the presence of cellulose. However, this increase in water absorption was well within the limits prescribed by Indian Standards.

Addition of a higher dosage of flax fiber led to a significant enhancement in the energy absorption capacity under impact loads. The maximum energy absorption was found to be 24.05 kJ corresponding to the fiber dosage of 0.75%. This high energy absorption capacity was due the characteristics of the fiber mechanism such as fiber pull-out, crack-bridging and effective bonding between the fiber-matrix interface.

Under elevated temperatures, the addition of a higher dosage of fibers was found to reduce the total reduction in mass and compressive strength. The control GP matrix with no fibers showed a reduction in compressive strength from 50 N/mm^2 to 35 N/mm^2 (30%) and 30 N/mm^2 (40%) when subjected to 400^oC and 600^oC respectively. However, with the addition of 0.75 flax fiber, a reduction in compressive strength from 45 N/mm^2 to 27.0 N/mm^2 (40%) and 22.5 N/mm^2 (50%) were observed when subjected to 400^oC and 600^oC respectively.

Considering the limitation of the present work, the use of natural flax fiber in untreated form could have imparted the mechanical and thermal behaviour of geopolymer composite matrix. As the scope for further work, the effect of different pre-treatment methods for natural fibers such as mercerization, cementitious treatment, etc. will be explored and the treated natural fibers will be used for the development of GP composite matrix. This will be the scope for further work.

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