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Effect of Coir Fiber Reinforcement on Properties of Metakaolin-Based Geopolymer Composite

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Abstract: This study explored the use of coir fibers extracted from coconut husks, an agro-waste material that constitutes sanitation and environmental pollution problems, as a reinforcing element in the production of metakaolin-based geopolymer composites with improved properties. A series of sample formulations were produced with varying coir fiber content (0.5, 1.0, 1.5, and 2.0 percent weight of metakaolin powder). The investigation was conducted using a 10 M NaOH alkaline solution with a 0.24 NaOH:Na_2SiO_3 mass ratio. Samples were cured for 28 days and tested for bulk density, ultrasonic pulse velocity (UPV), and compressive and flexural strength. Microstructural examinations such as X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) were also performed on samples. Compressive strength values up to 21.25 N/mm^2 at 0.5% fiber content and flexural strength values up to 10.39 N/mm^2 at 1% fiber content were achieved in this study. The results obtained showed a decreasing bulk density of geopolymer samples (2113 kg/m^3 to 2045 kg/m^3) with increasing coir fiber content. The geopolymer samples had UPV values varying from 2315 m/s to 2717 m/s. Coir fiber with 0.5–1.0% fiber content can be incorporated into metakaolin-based geopolymers to produce eco-friendly composite materials with improved mechanical properties for sustainable development.

Keywords: coir fiber; metakaolin; geopolymer composite; compressive strength; flexural strength; ultrasonic pulse velocity

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1. Introduction

Advances in research and technology require improved building materials that perform better in service. This requirement often necessitates the use of fibers to improve composite properties [1]. Carbon, metallic, polymer, Kevlar, and E-glass fibers have been used as reinforcement materials in the production of fiber-reinforced composites for building construction applications around the globe [2–4]. Research findings have revealed that although these fibers significantly improve the mechanical properties of the composites, the high cost of the fibers limits their use in the production of composites [5]. Current materials and engineering research are increasingly showing concerns about developing energy-efficient and eco-friendly materials that will sustain the building industry [6–9]. Thus, researchers in the building industry are exploring the use of natural fibers such as

coir, sisal, palm, hemp, kenaf, wool, banana, bamboo, cotton, bagasse, jute, and flax. These fibers are cheaper substitutes for man-made fibers as reinforcement materials in composite production [10,11]. The factors that guide the selection of these fibers for composite production include the type of matrix, fiber compatibility to the matrix, fiber dispersion, and the composite manufacturing process. Other factors include fiber type, extraction and treatment method, fiber aspect ratio, fiber content, fiber/matrix interface strength, hydrophobicity, and hydrophilicity of the fibers. One of such composite material matrices currently being explored in research is geopolymer [12,13].

Geopolymers are eco-friendly and emerging sustainable alternative materials to ordinary Portland cement (OPC) in order to improve the environment's carbon footprint [14]. Geopolymers are a group of materials formed when aluminosilicate source materials are mixed with an alkaline reactant solution [15]. In addition to the eco-friendliness of this material, geopolymers are excellent fireproofing materials, resistive to corrosive environments, and have good thermal properties and compressive strength [16]. Interestingly, they also have low water permeability properties [17]. With these advantages, the manufacturing of geopolymers requires special handling due to the different chemicals used in the production process. The technology is more suitable in precast applications where the control of sensitive materials can be ensured. Moreover, like many other cement-based materials, geopolymers have low tensile strength and exhibit quasi-brittle behavior under low mechanical loading, limiting their widespread use in many applications [18]. Thus, to improve geopolymer toughness, fibers are added to the geopolymer matrix [19,20]. The addition of natural fibers changes the brittle fracture nature of the geopolymer to a more ductile fracture [16].

The use of natural fibers in the production of geopolymers offers a myriad of advantages as these fibers are lightweight, resistant to a high alkaline environment, biodegradable, readily available, renewable, and non-hazardous [21,22]. For instance, using cotton fibers up to 0.5 wt % improved the flexural strength and fracture toughness of fly ash geopolymer composite [23]. Further improvement in the performance (fiber and matrix adhesion) of these fibers is achieved through different forms of fiber treatment. These treatment forms include mercerization, hornification, and silane treatment [24,25].

Extensive work has been carried out to investigate the performance of natural fibers such as coconut, sisal, flax, cotton, raffia, sweet sorghum, wood, bamboo, and wool in the production of geopolymer composites [23,26–31]. The results of these studies prove promising. Hitherto, the utilization of coir fiber in the fabrication of eco-friendly geopolymer composites with improved properties is still limited. Coir fibers extracted from coconut husks (an agro-waste) are most times incinerated or used in landfills, constituting environmental pollution and sanitation problems [32]. Moreover, only a small fraction of coir fiber is used in composite material applications from the more than 2 million tons of yields of fibers from the annual 42 million tons of coconuts produced worldwide [32,33]. The use of coir fibers in geopolymer composite production may be a way to recycle and reuse this waste material more sustainably. Hence, there is a need to explore coir fiber as a potential reinforcement material in the production of geopolymer composites for sustainable building applications.

Coir fiber is an abundant fiber obtained from the coconut palm (*Cocos nucifera* Linn) with plantation and cultivation mainly in the tropics. This fiber is processed by de-husking coconut and separating it from its fibrous mesocarp. The average length of the fiber is in the range of 0.15–0.28 m, and the diameter varies from 0.1 to 0.5 mm [34]. Coir fibers are used to manufacture rubberized coir pads, ropes, mats, brushes, carpets, thermal insulation, and road construction materials. Coir fiber is non-toxic, chemically reliable, cheap, and abundant. Like any other plant fiber, coir fiber is biodegradable with a slow decomposition rate of 20 years [35]. Furthermore, it retains a high percentage of its original tensile strength in composites [34] and has recently been used as a reinforcing material in plastics, clay cement bricks, and geopolymers.

However, an in-depth literature review has revealed a lack of published work on coir fiber as reinforcement in geopolymers. Although works were reported on reinforcing coir fiber in fly ash matrices [1,19,36], polymer matrices [37–44], and other cement-fiber composites [45,46], nothing is reported on the effect of coir fibers on the properties and microstructure of metakaolin-based geopolymer matrix. The study by Korniejenko et al. [1] shows that 1 weight percent 3 mm length, 0.5 mm diameter coir fiber reinforcement in fly ash geopolymer had good mechanical properties. Zulfiati et al. [36] also studied coir fiber as a reinforcing element in fly-ash-based geopolymer mortar composite. The weight percent of fibers used in the study was 0, 0.25, and 0.5 wt % of the geopolymer. For each fiber content, the length of coconut fibers was varied in the composite (10, 20, and 30 mm). The use of coconut fibers with up to 0.5% fiber content in the study improved the mechanical properties of fly-ash-based geopolymer mortar. Wongsa et al. [19] also investigated the properties of high-calcium fly ash geopolymer mortar containing varying proportions of coir and sisal fibers at 0%, 0.5%, 0.75%, and 1% volume fractions. The study proved coir fiber to be a good reinforcement for improving the geopolymer's tensile and flexural strength performances, although the ultrasonic pulse velocity (UPV), dry density, workability, and compressive strength values tended to decrease with increasing fiber content.

In the work of Kochova et al. [45], pre-treated coir fibers were used to reinforce cement–fiber composites as insulating material. Coir–cement composites demonstrated mechanical properties close to convectional wood–wood cement boards (WWCBs). The bending strength of 1.7 MPa in an acceptable density range of 500–600 Kg/m^3 was recorded. Pre-treated coir fibers also showed better cement/fiber interaction than untreated ones, with less slip softening and more slip hardening during fiber pull-out tests. In addition, the resultant composite material proved to be a good alternative where good thermal properties are desirous as the thermal conductivity is in the range of 0.08–0.11 W/mK.

The mechanical and morphological properties of chemically treated coir-filled polypropylene (PP) composites were also investigated by Islam et al. [38]. Treated coir fibers were mixed with PP granules at 10/90, 15/85, 20/80, and 25/75 weight percent mixing ratios. The mechanical properties of the composites fabricated from treated coirs were better than those of untreated ones. The study's findings showed a decrease in tensile strength of coir fiber–PP composites with increasing fiber content. The flexural and impact strength of the fabricated composites increased with increasing fiber loading. The highest flexural, tensile, and impact strength values recorded in this study were 57 MPa (25/75 wt %), 31.3 MPa (10/90 wt %), and 73 J/m (25/75 wt %), respectively. Moreover, the microstructure of the composite revealed fewer fiber agglomerations, microvoids, and stronger fiber–matrix interfacial adhesion.

In another study, Da Luz et al. [37] produced two natural-fiber-reinforced polymer composites to assess their interfacial adhesion characteristics and critical length. On one end, the researchers incorporated untreated coir fiber (30 vol%) in the matrices of epoxy resin, and on the other end, pineapple leaf fiber (PALF) (30 vol%). The findings showed that the critical length of coir fibers was 70% higher than those of PALF. However, the interfacial strength of coir fibers in the epoxy resin matrix compared to PALF was 3.5 times smaller. In terms of tensile strength, the Epoxy/30 vol% coir fiber composite gave a strength value of 28.7 $\pm$ 11.0 MPa, while the Epoxy/30 vol% PALF composite gave 86.4 $\pm$ 16.9 MPa.

The study conducted by Ayeni et al. [14] highlighted the need to integrate natural fibers in the matrices of metakaolin-based geopolymers for improved mechanical properties. The current study on coir-fiber-reinforced geopolymer composite will provide information on the performance of coir fiber as a reinforcing agent in the matrices of metakaolin-based geopolymer for sustainable building applications. Furthermore, since the trend in our today's world is the promotion of sustainable materials, the environmental pollution and sanitation problems resulting from the disposal of coconut husks will be alleviated. Moreover, the use of coir fibers in metakaolin-based geopolymers for building applications may serve as a benchmark for material scientists and engineers to develop

other coir–fiber geopolymer composite products with improved properties for use in other sustainable applications.

Research Significance

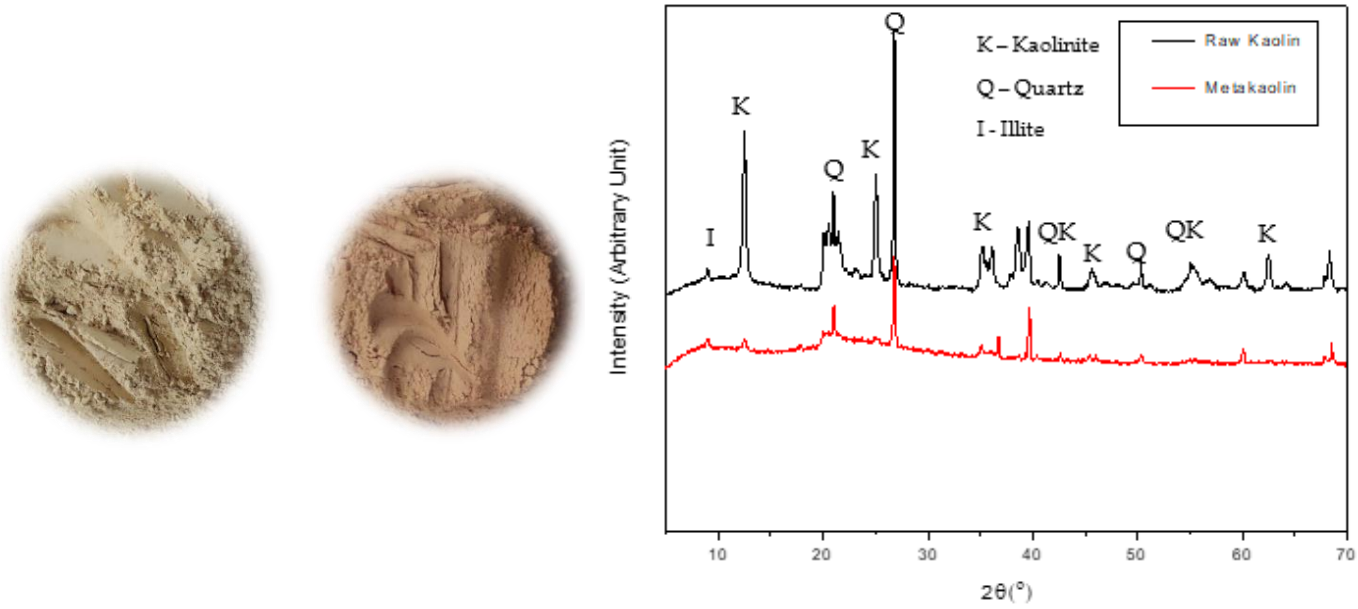
Presently, sustainable and affordable housing is still a topical issue in the building construction industry of most developing nations. The mass production of robust prefabricated bricks made of geopolymers and natural fibers has the potential to meet increasing housing demands. The findings of this study will benefit the construction industry if the compressive and flexural strength of geopolymer composite meets minimum ASTM requirements adequate for construction applications such as low-tech building material (clay bricks). In addition, it will present sustainability benefits when this geopolymer material is used as an alternative to ordinary Portland cement. This research will also help improve the understanding of using coir fiber as a reinforcing material in geopolymer composite production. The use of coir fibers as a reinforcement element in the production of geopolymer composites has not been fully reported in previous studies. Incorporating coir fibers in metakaolin geopolymer to improve the mechanical properties may be another waste disposal solution for sustainable development.

Specifically, this study evaluated the (i) modification in the physical property (bulk density), and ultrasonic pulse velocity (UPV) values of metakaolin-based geopolymer at a varying weight percent of coir fibers; (ii) changes in strength (compressive and flexural) of metakaolin-based geopolymer due to the inclusion of varying weight percent of coir fibers; and (iii) the effect of coir fibers on the phase composition, bonding mechanism, and microstructure of metakaolin-based geopolymer. This investigation will play a role in sustainability efforts and the preservation of the ecosystem.

2. Materials and Methods

2.1. Geopolymer Precursor Material

Metakaolin (MK) is the primary precursor material used in synthesizing the geopolymer matrix in this study. The MK was made by thermal treatment of kaolin clay mined from Kankara, Katsina State, Nigeria. Previous research by Ayeni et al. [14] confirmed its suitability for use as a matrix in the geopolymer composite. Before heat treatment, pulverization and wet beneficiation of the raw kaolin clay were carried out following the procedures reported by Ahmed et al. [47] and Salahudeen et al. [48]. The raw kaolin was calcined into metakaolin in a Nabertherm electric furnace chamber at 700 °C for 2 h. The previous work of Ayeni et al. [14] has shown these treatment conditions to be suitable for processing MK. The chemical composition of the kaolin and MK powder was investigated by X-ray florescence analysis (Olympus VANTA XRF Analyzer). The result (Table 1) showed silica (60.50%) and alumina (34.30%) to be the major constituents of the metakaolin. The MK powder's Si/Al ratio and loss on ignition (LOI) were 1.76 and 2.15%, respectively. The metakaolin's bulk density and specific gravity were computed and found to be 650 kg/m^3 and 2.86, respectively. Metakaolin was sieved with a 150 µm sieve before use, so the metakaolin particle size was <150 µm. The raw Kankara kaolin clay was cream-like before heat treatment and turned pink after the calcination process (Figure 1a,b). Figure 1c shows the XRD diffractogram of the kaolin and metakaolin as obtained using a D-5000 PSC-8 X-ray diffractometer. The major minerals in the XRD diffractograms are kaolinite, quartz, and illite [14]. The main characteristic peaks of kaolinite in the raw kaolin were exhibited at 12.35° and 24.88° two-theta (2θ). The intense peak observed at 26.63° 2θ corresponded to quartz. Comparing the diffractogram of raw kaolin and metakaolin, changes in the intensity of the peaks of kaolinite and quartz were observed. The color change and changes in chemical composition and diffractogram of kaolin indicated the transformation from kaolin to metakaolin material.



(**a**) (**b**) (**c**)

Figure 1. (**a**) Raw kankara kaolin clay. (**b**) Calcined raw kankara kaolin clay (metakaolin). (**c**) XRD diffractogram of raw kaolin and metakaolin.

Table 1. Chemical composition of kaolin and metakaolin (MK).

Mass Ratio (%)	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	K_2O	MnO	ZrO_2	CaO	BaO	LOI
Kaolin	60.09	26.15	1.46	0.27	0.70	0.20	0.01	0.06	0.21	10.85
MK	60.50	34.30	2.41	0.15	0.20	0.15	0.09	0.04	0.01	2.15

LOI = loss on ignition.

2.2. Alkaline Solution

The alkaline solution used for the dissolution of the aluminosilicate precursor material (MK) is a mixture of sodium hydroxide (NaOH) pellets and sodium silicate (Na_2SiO_3, water glass) solution. The NaOH pellets and Na_2SiO_3 solution (water glass) were locally purchased from a chemical supplier (Cardinal Scientific Supplies) in Zaria, Kaduna State, Nigeria. All were technical grade, with NaOH having a purity level of 99%. Before mixing, a 10 M solution of NaOH was prepared. The 10 M NaOH concentration was used throughout the experiment. Due to the exothermic nature of the reaction, the resulting NaOH solution was allowed to cool down to room temperature (26 °C ± 1 °C) before use. The mixing of NaOH solution with Na_2SiO_3 solution at a mass ratio of 0.24 followed. The resulting alkaline solution was kept for 24 h before sample preparation. The mass ratio of Na_2SiO_3 to NaOH (0.24) was fixed throughout the investigation. The choice of the 10 M and 0.24 mass ratio was influenced by the previous study carried out by Ayeni et al. [14] on Kankara metakaolin-based geopolymers. The prepared alkaline solution was then used to prepare the geopolymer matrix in this study.

2.3. Coir Fiber

Coir fiber was selected for the present study as it is considered by the Food and Agriculture Organization (FAO) of the United Nations as one of the future fibers. Coir fiber is available in large quantities with a global annual production of about 650,000 tonnes. Its cross-section is multicellular, with an average of 30–300 or more cells. Each cell has a diameter of 12–14 µm and a length-to-diameter ratio of the order 35 [49]. The tensile strength, Young's modulus, and total elongation of bulk coir fibers vary from 95 to 230 MPa, 2.2 to 6 GPa, and 15 to 51.4%, respectively [1]. This fiber is covered with a waxy-like

material "cuticle". Studies have shown that pure coir fiber has cellulose (32.86–43.44%), lignin (40.52–45.84%), hemicellulose (0.15–0.28%), pectin, and water-soluble as its main chemical constituents [49–51]. These relative percentages vary with the maturity age of the nuts from which the fiber has been extracted [50].

The coir fibers used in this study were extracted from the outer shell of coconut husks purchased locally and supplied by a coconut vendor in a central market in Kaduna State, Nigeria. Figure 2a–c shows the coir fiber's processing flow from de-husking to air-drying. De-husking of the coconut was the first step taken to process the coir fibers from the coconut husks. The coir fibers were observed to be brown, indicating that the fibers were extracted from matured coconuts. The de-husked coconut fibers were then soaked in distilled water for about 30 min to make the coir fibers soft and facilitate ease of combing, stretching, and separating fibers into individual single filaments. The fibers were then pre-treated in 5% NaOH solution (mercerization) for 24 h to remove the wax, oil, and different impurities present in the coir fibers [32,52]. According to Hassan et al. [32], 5% NaOH concentration is the optimum alkaline concentration to treat coir fibers using the traditional mercerization technique. A higher NaOH concentration could degrade the coir fibers' cellulosic structure. The treatment of the coir fibers improves the fibers' durability in service [53–55].



(**a**) (**b**) (**c**)

Figure 2. (**a**) De-husked coir fibers. (**b**) Pretreatment of fibers in 5% NaOH. (**c**) Air drying of PCFs.

After the pre-treatment process, the coir fibers were washed in distilled water until a pH value of ≈8.0 was achieved. Air drying of the pre-treated coir fibers (PCFs) for 24 h to remove free water followed. The PCFs were observed to change color from light brown to dark brown. The air-dried PCFs were then combed, stretched, cut into lengths of ≈30 mm, and subsequently stored in an airtight plastic bag. Table 2 shows the index properties of the pre-treated coir fiber used in this study. Table 3 presents the chemical composition of the untreated and pre-treated coir fiber. The result shows an increase in the cellulose content of the pre-treated coir fibers against their untreated counterpart. The increase in cellulose content is attributed to the removal of some amount of lignin and hemicellulose from the fiber [49].

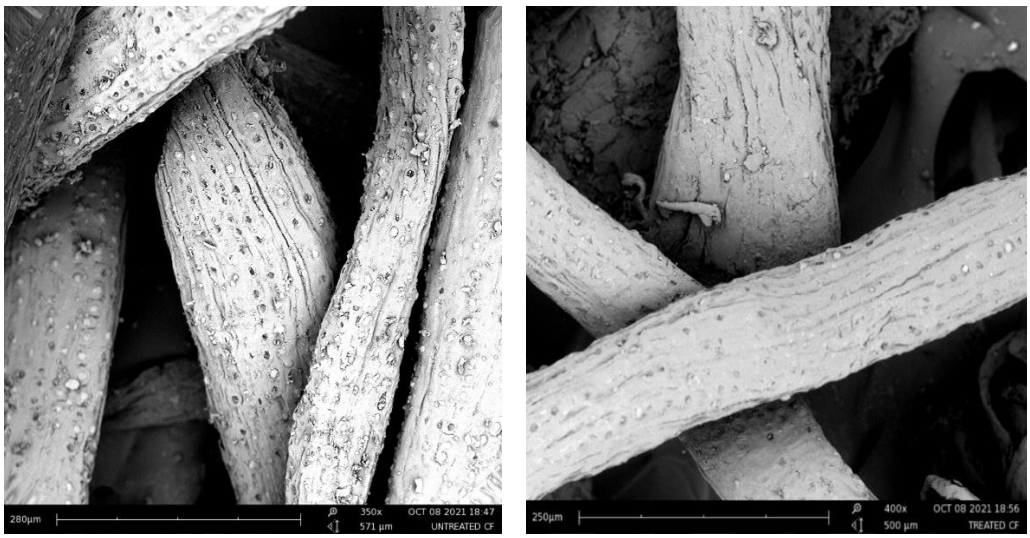
The longitudinal morphology of the untreated and pre-treated coir fibers from scanning electron microscopy (SEM) is shown in Figure 3a,b. The micrograph of the coir fibers revealed that the fibers are solid though irregular in size. The pre-treatment also modified the surface texture of the coir fibers.

Table 2. Index properties of pre-treated coir fiber.

Property	Value
Diameter (mm)	0.35
Tensile strength (MPa)	138
Young's modulus (GPa)	6.2
Elongation at break (%)	27
Density (g/cm^3)	1.25

Table 3. Chemical composition of untreated and pre-treated coir fiber.

Chemical Composition	Untreated Coir Fiber (%)	Pre-Treated Coir Fiber (%)
Cellulose	27.0 ± 1.70	57.0 ± 1.10
Lignin	32.80 ± 3.81	17.20 ± 1.40
Hemicellulose	22.50 ± 4.30	8.20 ± 1.31



(**a**) (**b**)

Figure 3. SEM micrograph of the longitudinal view of (**a**) untreated coir fibers and (**b**) pre-treated coir fibers.

Figure 4 shows the diffraction pattern of the untreated and pre-treated coir fibers as analyzed from X-ray diffraction (XRD). The coir fiber diffractograms showed characteristic peaks indicative of cellulose with slight traces of quartz [56]. The main crystalline peak at 22.50° 2θ corresponded to cellulose (002). The lower-intensity peak observed at 17° and 35° 2θ, which corresponded to cellulose (101) and (004), respectively, indicated a higher amount of amorphous material such as lignin and hemicellulose in the coir. The improvement in the crystallinity of the pre-treated coir fiber at 22.50° 2θ confirmed the increase in cellulose content of the fiber. Furthermore, quartz traces in the untreated coir fibers were not visible after the pre-treatment process.

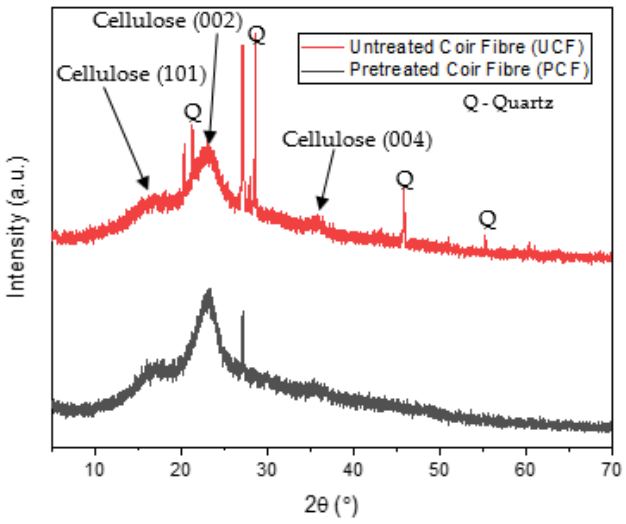


Figure 4. XRD pattern of untreated and pre-treated coir fiber.

This study investigated the effects of coir fibers in varying percentages (0.5–2 wt % of MK) in metakaolin-based geopolymer. Literature and preliminary studies (trial tests) conducted before this research guided the selection of the coir fiber content. The dosage of coir fiber was restricted to 2%, as previous studies have shown that higher fiber dosages are not beneficial to the mechanical strength development of composites [57,58]. Moreover, the selected coir fiber content allowed for comparison with previous studies on other fiber-reinforced geopolymers where similar fiber contents were used.

2.4. Preparation of Samples

Unreinforced (MGP control) and reinforced metakaolin-based geopolymer (MGP—0.5% PCF, to MGP—2% PCF) were prepared to study the effects of coir fiber reinforcement on the properties of the geopolymer. Table 4 summarizes the mix proportion used in the preparation of the samples. A total of five different sample formulations based on fiber composition that varied from 0% to 2% were investigated for compressive and flexural strength. For each sample formulation, three number samples were prepared at each fiber composition for the various mechanical strength tests performed.

Table 4. Mix proportions of samples.

Sample Formulation	MK (g)	Pre-Treated COIR Fiber (PCF) (g)	Na_2SiO_3/NaOH (by wt.)	Solid/Liquid (S/L) Ratio
MGP control	1200	0	0.24	0.97
MGP—0.5% PCF	1194	6	0.24	0.97
MGP—1% PCF	1188	12	0.24	0.97
MGP—1.5% PCF	1182	18	0.24	0.97
MGP—2% PCF	1176	24	0.24	0.97

In the preparation of the specimen, for each formulation, an appropriate amount of metakaolin (MK), chopped pre-treated coir fibers (PCF), and alkaline solution (Na_2SiO_3/NaOH) were measured separately as per Table 4. The measured MK and PCF were poured into a mortar mixing machine and then dry-mixed for about 60 s to ensure the homogeneous distribution of the coir fibers within the MK powder. The addition of PCF varied from 0.5% to 2% of the mass of MK in the MGP control formulation. This step was followed by the gradual addition of alkaline solution into the mixer until a homogeneous paste was obtained. The MK powder to alkaline liquid that represented the solid to liquid (S/L) mass ratio used was fixed at 0.97 for all the formulations. The mixing time for each formulation was kept at 3 min. Mixing of the specimen was preceded by specimen filling in different molds specific for the test to be carried out. Different cubic (50 × 50 × 50 mm) and prismatic (40 × 40 × 160 mm) molds were filled with the geopolymer paste in two layers. Each was subjected to vibrations on a vibrating table for about 10 s to relieve the geopolymer mixture of air bubbles trapped within. The top of the molds were leveled with a smooth trowel, and various identification marks were inscribed on cast samples for ease of sampling.

Furthermore, polythene sheets were used to cover the top surface of the molds to prevent efflorescence and excessive moisture loss. The sealed samples were ambient cured in the laboratory (26 °C ± 1 °C, 60% ± 5% humidity) for 24 h. Samples were de-molded (Figure 5a,b) and kept in sealed polythene bags at room temperature until mechanical tests were performed after 28 days. In our previous study [14], the early stage (7 days) and 28-day strength of this geopolymer formulation (without the inclusion of fibers) was reported. Since this geopolymer's early stage (7 days) and 28-day performance (without the inclusion of fibers) was already known, we explored only the effect of coir fiber addition at 28 days.



(**a**) (**b**)

Figure 5. (**a**) Sample cubes for compressive strength testing. (**b**) Sample prisms for flexural strength testing.

2.5. Physical and Mechanical Property Measurement of Samples

In this study, geopolymer samples' bulk density (ρ_b) was the only physical property examined. On the other hand, two mechanical properties (compressive and flexural strength) of samples were investigated. For the flexural strength of samples, the 3-point flexure test arrangement was used. All mechanical testing was performed on cured samples of unreinforced metakaolin-based geopolymer and coir-fiber-reinforced metakaolin-based geopolymer (CFRMG) composites using a STYE 2000 machine (Okhard Machine Tools Ltd., Lagos, Nigeria) with a 3000 KN capacity at a loading rate of 2.4 kN/s. The tests were conducted on three samples for each formulation after the curing age of 28 days in line with ASTM C109 [59] for compressive strength and ASTM C293 [60] for flexural strength.

Inferential statistics were used to test the set hypothesis in the study after the different property tests. One-way ANOVA was used to determine if there are any significant differences between the mean values of the examined properties of unreinforced metakaolin-based geopolymers and CFRMG composites. A probability value (p-value) of ($p \leq 0.05$) and confidence level of 95% was used for all analysis. If $p \leq 0.05$, the null hypothesis was rejected (no statistically significant differences between the mean values of the physical or mechanical property under consideration).

2.6. Ultrasonic Pulse Velocity (UPV) Measurements

Ultrasonic pulse velocity (UPV) is a non-destructive technique used to estimate the depth of cracks or void presence in samples, uniformity in samples, or changes in properties of samples with time. UPV measurements were performed after the curing age of 28 days to understand the homogeneity of pore distribution. This was conducted using a UPV PL-200 machine (Proceq Pundit Lab, Screening Eagle Technologies, Zurich, Switzerland) per ASTM C597-02 [61].

2.7. Microstructural Characterization

Different techniques were employed to characterize samples, identify crystalline phases, describe crystallinity changes, and examine the bonding mechanism. X-ray diffraction (XRD) was performed to understand the crystal structures that were present in the MK-based geopolymer and those containing coir fibers (CFRMG composites). Before XRD analysis, samples were ground to a fine homogeneous powder and poured on a sample holder for analysis. The XRD patterns of samples were obtained using a D-5000 PSC-8 X-ray diffractometer equipped with a 40 KV power source and an X-ray source of 1.54 Å CuKα radiation, which was operated within a 2θ range of 5°–70° at a scanning rate of 1°/min and step size of 0.0396°. Fourier transform infrared (FTIR) spectroscopy (Agilent Cary 630

FTIR analyzer, Agilent Technologies, Victoria, Australia) examined the bonds present in the CFRMG composites and geopolymer samples without coir fibers in the wavelength 650–4000 cm^{-1}. To characterize the structure of MK-based geopolymers and CFRMG composites at different fiber content, scanning electron microscopy (SEM) was performed using SEM (JEOL 7000F, JEOL Inc., Peabody, MA, USA). Before SEM imaging, samples were mounted on a double-sided copper tape with one side affixed to an aluminum stud. Coating of samples with a 5 nm gold-palladium layer was performed to reduce sample charging during imaging.

3. Results and Discussion

3.1. Effect of Coir Fiber Addition on the Compressive Strength of Geopolymer Specimen

The compressive strength is a key property for measuring the mechanical properties of building materials. Figure 6 shows the compressive strength results of the metakaolin-based geopolymer reinforced with coir fibers at different fiber contents (0.5–2%). Upon the introduction of coir fibers at 0.5% fiber content, the compressive strength value increased from 16.9 1 N/mm^2 (MGP Control) to 21.25 N/mm^2. The addition of 0.5% PCF fiber may have enhanced the formation of a denser composite than the control, with less porosity and cracks, thus increasing the compressive strength. The 0.5% fiber content was evenly distributed in the matrix. Crack arrest at the metakaolin/fiber interface and the high aspect ratio of the fiber improved the mechanical strength. This is consistent with the result of Wongsa et al. [19], who reported that 0.5% coconut fiber content improved the compressive strength of high calcium fly ash geopolymer. This result suggests that pedestrian paving bricks can be fabricated using coir fiber-reinforced metakaolin geopolymers since it satisfies the minimum compressive strength requirement standard of the American Society for Testing and Materials (ASTM C216) [62]. Surprisingly, 1% coir fiber addition decreased the compressive strength, suggesting that at 1% fiber, the dispersion of the coir fiber in the geopolymer matrix was not good. Fiber clustering may have promoted nucleation cracks within the geopolymer matrix. A similar trend was also observed in the study of Wongsa et al. [19], in which the compressive strength of composites decreased with increasing fiber content from 0.5% to 1%. However, this result is not consistent with the findings of Korniejenko et al. [1], who recorded improved compressive strength at 1% coir fiber addition in fly ash geopolymer composite. At higher coir fiber content (1.5%), the geopolymer composite strength corresponded to 16.94 N/mm^2. It is similar to the control strength of the Mk-based geopolymer without fibers. At this fiber content, the presence of coir fibers had no significant effect in contributing to the compressive strength of the synthesized coir-fiber-reinforced geopolymer composite. Worthy of note also is the decrease in strength at 2% coir fiber addition, suggesting a less dense composite structure. At higher fiber content, the density of samples tends to reduce, packing of geopolymer matrix becomes difficult, and porosities of specimen increase, which in turn reduces compressive strength [19]. Figure 7 illustrates the mode of failure of the tested cubes. The failure mode of the cubes is non-explosive [63]. The cubes almost maintained their original shape. The energy absorption capacity of the coir fibers might have helped control the cracks. Coir-fiber-reinforced metakaolin-based geopolymer composite fabricated with 0.5% coir fiber content can therefore be used to produce eco-friendly building materials as it satisfies the ASTM C62 standard [64] for building. Bricks having a compressive strength of 17.20 N/mm^2 is the minimum requirement. Our results suggest that 0.5% coir fiber content in the metakaolin geopolymer matrix composite is the optimum for improved compressive strength.

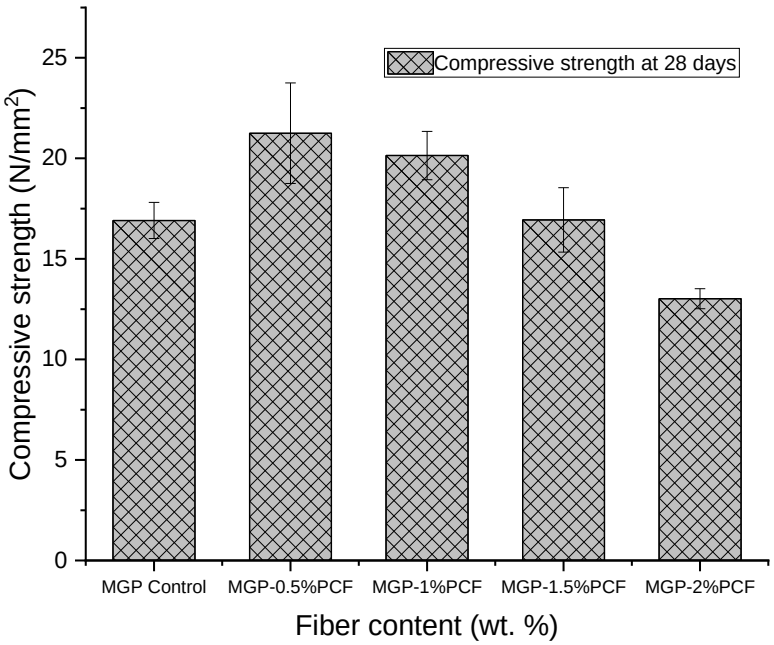


Figure 6. Compressive strength of specimens at different coir fiber contents tested after 28 days.



Figure 7. Mode of failure of cubes after compressive strength test.

3.2. Effect of Coir Fiber Addition on the Flexural Strength of Geopolymer Specimen

Fibers are added to matrices to form composites with improved strength and toughness. Crack arrest at the fiber–matrix interface and increasing crack path via fibers with high aspect ratios are among the mechanisms for improved toughness. The effect of various fractions of coir fibers on the flexural strength of metakaolin-based geopolymer composites synthesized is presented in Figure 8. From Figure 8, it is clear that incorporating pre-treated coir fibers at different percentages (0.5% to 2%) enhanced the bridging effect and generally increased the flexural strengths of metakaolin-based geopolymer composites. This may also be attributed to the high tensile strength and elastic modulus of the coir fibers, as well as the possibility of stress transfer from the specimen to the coir fiber through the interface of the geopolymer matrix [19]. The basic mode of failure of the coir-fiber-reinforced geopolymer specimen formulated is mode I [15] (opening mode: tensile stress is orthogonal to the local plane of the crack surface) (Figure 9). When a fiber-reinforced composite is subjected to bending load(s), the bending moment causes tensile stresses. At the fiber–matrix interface, the tensile stress transforms into shear stress, which builds resistance at the fiber/matrix interfaces. As fiber contents are increased, the formation of microcracks within the composite structure is anticipated instead of a few large macrocracks. For the MGP-0.5% geopolymer composite formulation, a flexural strength of 9.74 N/mm^2 was recorded. At higher coir

fiber content (1%), a significant increase (28%) higher than the control sample without coir fibers was observed. Wongsa et al. [19] reported a similar increase in flexural strength of high-class fly ash geopolymer mortar as fiber content increased from 0% to 1% volume fraction, although the strength values in this current study were higher.

Compared with the work of Korniejenko et al. [1], a reduced value in the flexural strength was recorded when 1% coir fiber content was used as a reinforcing element in fly-ash-based geopolymer. The flexural strength observed for 1% coir fiber addition was the highest (10.39 N/mm^2) in this study. Flexural strength tends to decrease with increasing coir fiber content from 1% to 2%. The flexural strength of the 1.5% and 2% fiber contents were 9.13 N/mm^2 and 8.58 N/mm^2, respectively. Compared with the control sample (MGP Control) that gave 7.60 N/mm^2, these values are still higher. At higher fiber content (1.5% and 2%), a weak composite interface might have been created due to increased fiber-to-fiber interactions. The coir fibers in the composite may not have perfectly aligned with the geopolymer matrix. Hence, at higher flexure stress, weak interface cracks could form easily leading to failure.

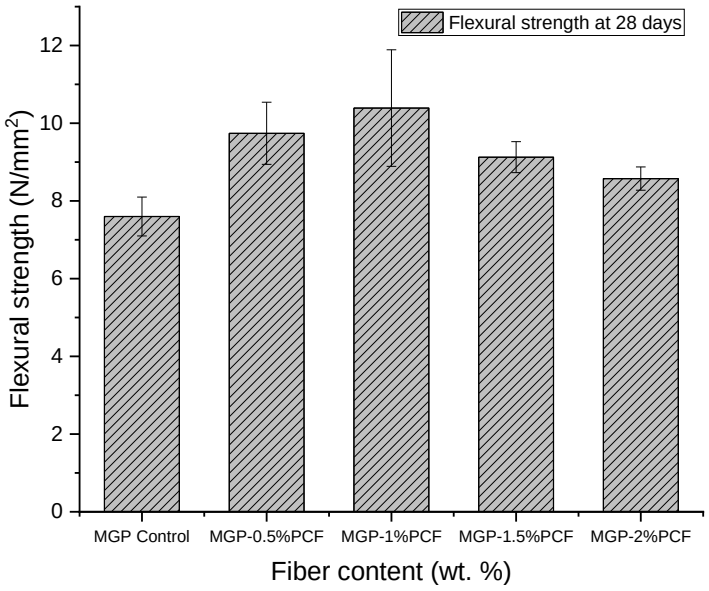


Figure 8. Flexural strength of specimens at different coir fiber contents tested after 28 days.

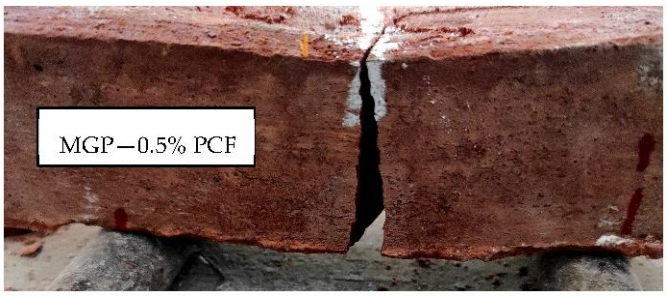


Figure 9. Mode of failure of the tested prism.

3.3. Effect of Coir Fiber Addition on the Bulk Density of Geopolymer Specimen

Figure 10 shows metakaolin-based geopolymer composite bulk density at varying coir fiber contents. It was seen that the bulk density of the geopolymer composites decreased as the weight percent of coir fiber increased. There was a strong correlation between the amount of coir fibers (wt %) present in the geopolymer composite and the bulk density of the CFRMG composite. The geopolymer control sample (MGP Control) had the highest bulk density of 2113 kg/m^3, whereas the geopolymer composite reinforced with 2.0 wt % of coir

fiber (MGP—2% PCF) had the lowest (2045 kg/m^3). The bulk densities of the geopolymer composites were observed to decrease with increasing coir fiber content. Comparing these results with other investigators on composites, an agreement in the trend of decreasing values can be observed. From the result of Abdullah et al. [65], for instance, coconut fiber as a reinforcing element in cement composite decreased the bulk density with increasing fiber content. In addition, the study by Alomayri et al. [23] on the use of cotton fibers as reinforcing elements in fly ash-based geopolymer composite showed decreased geopolymer composite density with increasing cotton fiber content.

Similarly, the study on wood-fiber-reinforced geopolymer composites, as reported by Korniejenko et al. [29] and Berzins et al. [66], showed that density decreased in value with increasing fiber content. The reduced bulk densities in these studies could be attributed to the specific gravity of reinforcing fibers being lower than that of the geopolymer matrix. Moreover, the density of geopolymer-based composites tends to decrease with an increase in fiber content because fibers clump together during the mixing process, entrapping water-filled spaces that, in turn, become porosities or air voids [10]. Furthermore, as lighter materials result in easier handling in the construction sector, the decreased bulk density of the composites as coir fiber content increases will promote the ease of handling the geopolymer composites in service.

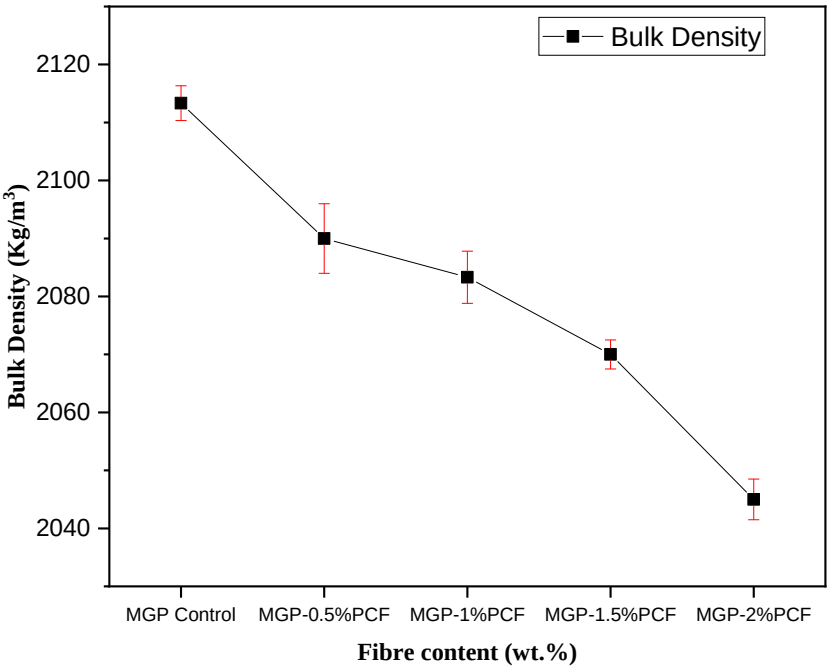


Figure 10. Bulk density of geopolymer specimens at different coir fiber content cured at age 28.

3.4. Effect of Coir Fiber Addition on the Ultrasonic Pulse Velocity (UPV) of Geopolymer Specimens

Table 5 shows the results of the ultrasonic pulse velocity (UPV) measurements performed on the metakaolin geopolymer samples and those with varying coir fiber content cured for 28 days. The addition of coir fibers into the matrices of the metakaolin-based geopolymer could influence the UPV values of samples as ultrasonic waves travel at a higher speed in fibers than in the geopolymer mortar itself [19]. The UPV result revealed the geopolymer samples' quality and homogeneity of pore distribution per ASTM C597-02 standard [61]. The geopolymer samples had UPV values varying from 2315 m/s to 2717 m/s. The control sample (MGP Control) had the least value of UPV (2315 m/s). The addition of 0.5 wt % caused a ≈15% increase in the UPV value (2717 m/s). This 0.5 wt % gave the highest UPV value. Conversely, the 0.5 wt % formulations also provided relatively high compressive and flexural strength, which indicated that a more homogeneous pore distribution within the composite for the 0.5% addition improved the compressive and

flexural strengths. At coir fiber weight percentages of 1.0, 1.5, and 2.0%, the recorded UPV values were 2688 m/s, 2577 m/s, and 2475 m/s, respectively. This study showed higher values of UPV values for reinforced samples than the unreinforced sample. The increased specimens' UPV with fiber content can be partially attributed to the bridging effect of the coir fiber, which leads to the reduction of micro-cracks in the geopolymer matrix [67]. This study showed higher values of UPV when compared with the results of the study conducted by Maras et al. [68] that reinforced fly ash geopolymer with polypropylene (PP) fibers at 0.5 and 1.0% total volume of mixture, in which UPV values varied from 1800 m/s to 2000 m/s.

Furthermore, compared to traditional concrete materials, the UPV values here are considerably low. In this regard, the lower UPV values of the geopolymer composites compared to traditional concretes are supported by the study of Wongsa et al. [69], which noted that geopolymers often have less UPV values than conventional binder materials. Furthermore, the UPV values recorded in this study may have been strongly affected by the composite density, which is related to air voids and the porosity of the structure [19]. In addition, as reported by Gosh et al. [70] and Shankar et al. [71], UPV values tend to increase with the increase in compressive strength values and was the trend in this study.

Table 5. Ultrasonic pulse velocity (UPV) of samples.

S/N	Sample Formulation	Transmission Time (μs)	UPV (m/s)
1	MGP control	21.6	2315
2	MGP—0.5% PCF	18.4	2717
3	MGP—1% PCF	18.6	2688
4	MGP—1.5% PCF	19.4	2577
5	MGP—2% PCF	20.2	2475

3.5. Effect of Coir Fiber Addition on the Crystal Structure of Geopolymer Specimen

Figure 11 reveals the XRD patterns obtained for MK-based geopolymer specimen without coir fibers (MGP Control) and a geopolymer formulation with pre-treated coir fibers (MGP—1% PCF). The diffuse hump/halo peak present at the two-theta (2θ) range 18°–38° confirmed the transformation of the MK precursor material and the formation of geopolymer paste [14]. It is unclear as to whether crystalline zeolitic phases formed with the amorphous binder phase. The main crystalline phases identified in the two samples were hydroxysodalite, herschelite, cristobalite, sanidine, quartz (SiO_2), and mullite ($3Al_2O_3 \cdot SiO_2$), with hematite (Fe_2O_3) appearing in a small amount [14,31]. Quartz was the most widely found phase in the formulations, having 2θ at 21.5°, 27.5°, and 38°. In addition to the quartz phases, mullite phases were found at approximately 16°, 32°, 42°, and 58° 2θ. The crystalline phases in the MGP Control were not altered due to the presence of coir fibers that act as reinforcing elements in the geopolymer matrices. It shows that fiber and matrix phases do not react during the geopolymerization process but act as inactive fillers in the geopolymer network. Furthermore, geopolymers with coir fiber addition were more crystalline than the pure MK-based geopolymer, suggesting that the formation of amorphous phases via geopolymerization was less when fibers were added. However, comparing the intensities of the crystalline peaks of the two formulations, a decrease in the intensities of crystalline peaks at 16°, 19°, 27.5°, 32°, and 51° 2θ were observed as coir fibers were added into the matrices of the formulation (MGP—1% PCF). On the other hand, an increase in peak intensities was seen in 28° and 33° 2θ. In addition, the coir-fiber-reinforced metakaolin geopolymer composites showed new crystalline peaks at 57° and 59° 2θ. These differences may have contributed to improvement in the mechanical properties of the geopolymer composite.

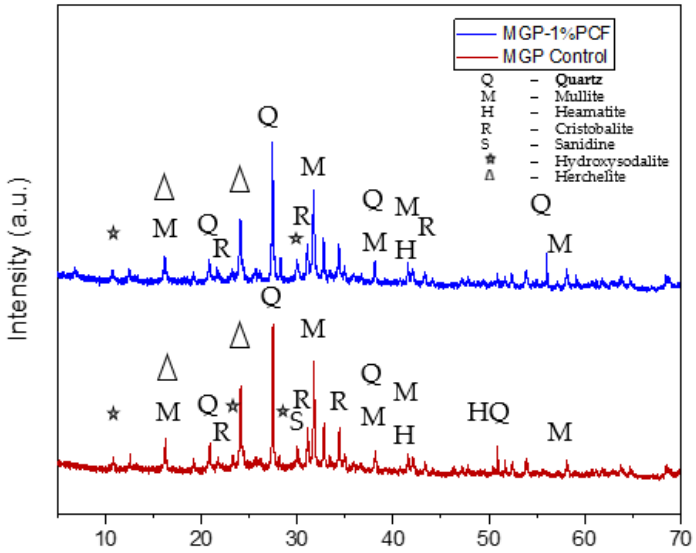


Figure 11. XRD pattern of MK-based geopolymer and CFRMG composite specimen at 28 days curing age.

3.6. Effect of Coir Fiber Addition on Absorption Bands of Geopolymer Specimen

The FTIR spectra of metakaolin-based geopolymer (MGP Control) and coir fiber-reinforced geopolymer composite (MGP—1% PCF) formulations at 28 days as observed in the wavelength 650–4000 cm^{-1} are shown in Figure 12. The observed FTIR absorption bands were used to understand some of the changes that took place in the MK-based geopolymer and that incorporated with coir fibers. It can be observed that the spectra of the MK-based geopolymer specimen and geopolymer specimen having coir fibers as reinforcing elements were relatively similar. The MGP control formulation had main characteristic absorption bands at approximately 3340 cm^{-1}, 2102 cm^{-1}, 1651 cm^{-1}, 1398 cm^{-1}, 947 cm^{-1}, and 745 cm^{-1} [72]. The wide absorption band at 3340 cm^{-1} can be attributed to O-H stretching and intramolecular vibrations of H-O-H, Si-OH, and Al-OH bond groups [73,74]. The band observed at 1651 cm^{-1} was due to H-O-H bending vibration of absorbed water molecules in the silica matrix. The absorption band at 1398 cm^{-1} was assigned to the C-O stretching vibration of carbonate. The MGP Control specimen also exhibited absorption corresponding to Si-O-Al vibrations and Al-O stretching vibrations at 947 cm^{-1} and 745 cm^{-1}, respectively [75,76]. Worthy of note is the high absorption (low transmittance) at the 947 cm^{-1} bands, indicating more stretching vibration of the Si-O-Al bonds in the system. The intensity of the IR spectrum at this band may also be attributed to the high polarity of the bond. However, certain shifts in some absorption bands of the spectrum of MGP Control were seen as coir fibers were introduced into the matrices of the MK-based geopolymer (MGP—1% PCF). While the MGP—1% PCF specimen exhibited similar absorption bands to MGP Control at 3340 cm^{-1} (O-H stretching) and 947 cm^{-1} (Si-O-Al vibrations), the MGP—1% PCF spectrum showed a slight shift towards lower wavenumbers (1651 cm^{-1} to 1640 cm^{-1} and 1398 cm^{-1} to 1387 cm^{-1}). However, an exception was observed in wavenumber 2102 cm^{-1}, which shifted to 2110 cm^{-1} of the MGP Control specimen. These slight changes as observed may be attributed to the interactions of the coir fibers with the matrix of the MK-based geopolymer and may have contributed to improved mechanical properties of the CFRMG composites.

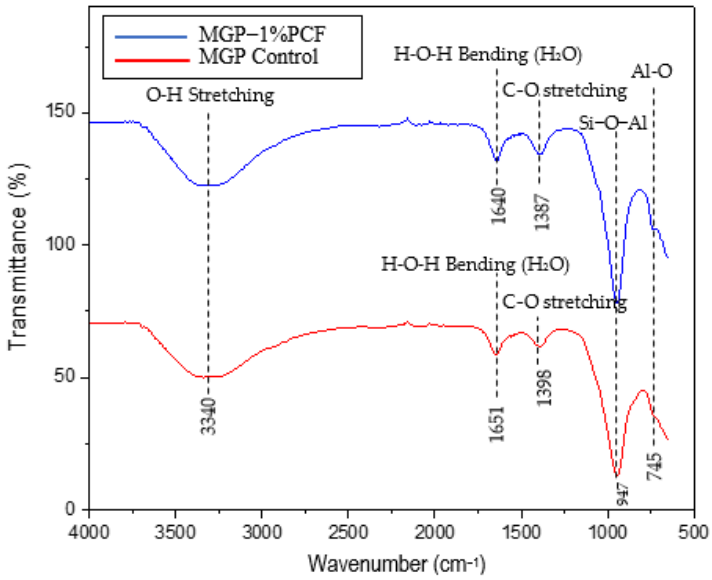


Figure 12. FTIR spectra of MK-based geopolymer and CFRMG composite specimen at 28 days curing age.

3.7. Effect of Coir Fiber Addition on the Microstructure of Geopolymer Specimen

The micrographs in Figure 13a–e show the microstructure of the metakaolin-based geopolymer for different coir fiber weight percentages added. The SEM observation was performed on fractured geopolymer samples after the flexural strength tests. Observation of the MGP Control sample showed a porous geopolymer matrix with many surface cracks. However, the addition of coir fibers in varying weight percentages in the matrices of the metakaolin-based geopolymer resulted in a more compact structure with fibers bridging the propagation of cracks in the composite. This may explain the increased flexural strength values obtained in Figure 8. At 0.5 wt % coir fiber content in the geopolymer matrix (MGP—0.5% PCF), some form of fiber–matrix adhesion was noticeable, which was the onset of a homogeneous structure. At this fiber content, some pores within the structure were still noticeable with visible surface microcracks, which can be attributed to the crack bridging effect of the coir fibers [77]. At a higher coir fiber content, for instance, at 1.0 wt % of coir fibers, a more compact structure of the geopolymer composite was seen, with some form of a nonlinear crack pattern (crack deflection). Also noticeable was coir fiber–matrix debonding. Furthermore, at 1.5 wt % coir fiber content, other forms of crack bridging in the metakaolin-based geopolymer composite were observed. The micrograph of the MGP—1.5% PCF formulation showed some crack deflection patterns and microcracks, as also observed in the MGP—1.0% PCF formulation.

3.8. Implications of the Current Study

On the basis of the results obtained in this study and the abundance and availability of agro-waste material (coconut) and kaolin clay in considerable quantities, these materials can be processed and engineered into valuable and sustainable construction materials. The results of CFRMG composites are promising and valuable for regions of the world where affordable housing is still a problem due to the high energy requirement for producing convectional materials. It proves beneficial to regions where kaolin clay materials are readily available and agro-waste materials such as coconut constitute a nuisance in the environment. The production of CFRMG composites will further result in the preservation of the global ecosystem.

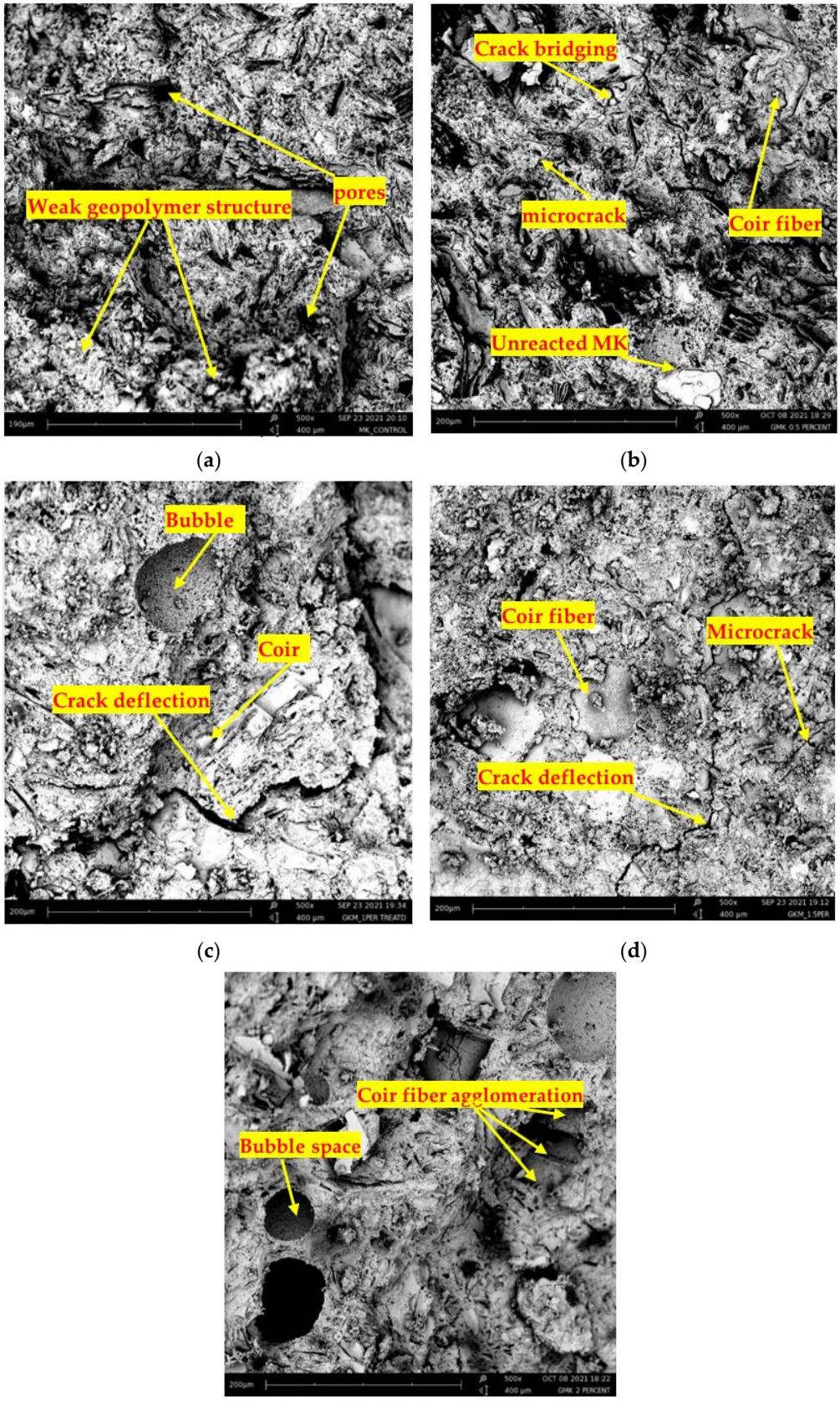


Figure 13. Morphology of geopolymer specimen at different coir fiber content cured at age 28: (**a**) MGP Control, (**b**) MGP—0.5% PCF, (**c**) MGP—1.0% PCF, (**d**) MGP—1.5% PCF, (**e**) MGP—2.0% PCF.

4. Conclusions

In this article, the experimental investigation of the effects of different weight percentages of coir fibers (0.5%, 1.0%, 1.5%, and 2.0%) as a reinforcement in the fabrication of sustainable metakaolin-based geopolymer composites for use in the construction industry was presented. The investigation provided a more scientific understanding of the effects of coir fibers on the micro- and macrostructural behavior of coir fiber-reinforced metakaolin-based geopolymer (CFRMG) composites. The main findings of the study are as follows:

i. Increasing the weight percent of coir fibers in MK-based geopolymers from 0% to 2.0% led to a corresponding decrease in the bulk density of CFRMG composites from 2113 kg/m^3 to 2045 kg/m^3. The decreased bulk density will promote the ease of handling the CFRMG composites.
ii. The geopolymer samples had UPV values varying from 2315 m/s to 2717 m/s, with the control sample giving the lowest value of UPV while the 0.5 wt % gave the highest value. The UPV result indicated a more homogeneous pore distribution within the 0.5 wt % CFRMG composite.
iii. The addition of 0.5 wt % coir fibers in metakaolin-based geopolymers was most beneficial to the compressive strength development of CFRMG composites as this fiber content gave the highest compressive strength value of 21.25 N/mm^2, which met the minimum compressive strength values of ASTM C216 and ASTM C62 for producing paving bricks and building bricks, respectively.
iv. The optimum coir fiber weight percent that favored flexural strength values of CFRMG composites was 1.0% (10.39 N/mm^2). At higher fiber content (1.5% and 2%), a weak composite interface was created due to increased fiber-to-fiber interaction, causing flexural strength values to decrease.
v. Incorporating coir fibers in the matrices of metakaolin-based geopolymers did not change their crystal structures or result in new chemical bonds in the CFRMG composite structure.

5. Limitation and Future Research

As with every other study, our study had a limitation. The coir fibers used in this study were sourced from one point. There was a possibility that the index properties and chemical constituents of the coir fibers determined may not be representative of the coir fibers from other locations within the country. The index properties and chemical constituents of the coir fibers may influence the properties of the geopolymer composite produced. Due to this limitation, further research needs to be conducted with coir fibers from other locations across the country to bring in newer insights.

This research is just the first step in studying the effect of coir fiber reinforcement in metakaolin-based geopolymers. There is still a need to investigate the durability performance of this composite in aggressive and corrosive environments such as acids, chlorides, and sulfates. Furthermore, the impact of high-temperature regimes on the fire resistance properties of the CFRMG composites should be explored.

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