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Flexural Strength and Porosity of NaOH-Treated Maize Stalk Cellulose-Fibers-Reinforced Geopolymer Composites

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Abstract

This study characterizes the flexural strength and porosity of NaOH-treated maize stalk cellulose-fibers-reinforced geopolymer composites. Flexural strength tests are conducted, and the fracture surfaces of the composite and geopolymer powder are observed using a scanning electron microscope (SEM). Moreover, porosity analysis is also performed using Image J software from SEM images. The formation of geopolymer is confirmed using X-ray diffraction (XRD) and Fourier transform infrared (FTIR) analysis. The addition of 1.5 wt% of NaOH-treated maize stalk cellulose fibers improves flexural strength by 2.4 times. The results show that the main failure mechanisms, namely fiber breakage, fiber pullout, and debonding of the fiber and matrix, can increase flexural strength and reduce failures during service life. During the analysis for fiber and particle pullout, SEM images under 25^2 pixels of pore areas are not considered, and an average porosity of 36.7% is achieved.

Keywords: maize, cellulose, geopolymer, composite, characterization, calcined kaolin

1. Introduction

The popularity of green materials has grown; moreover, due to increased awareness of the limited resources and the availability of local resources for economic development, attention is given to the development of new materials from locally available kaolin clay and maize stalk fiber. The raw material used for the fabrication of geopolymers commonly consists of industrial wastes, such as fly ash and metakaolin, which are both rich in silica and alumina. Thus, geopolymers are an economically achievable alternative for minimizing the environmental impact of carbon sequestration.

The addition of reinforcing agents can improve the mechanical properties of geopolymer. On the other hand, the fibers enhance various properties such as tensile strength and toughness, which can control the cracking mechanism and the ductility of the geopolymer composites. Among the fibers used as geopolymer reinforcement, emphasis should be placed on metallic, inorganic, organic, and natural fibers. Each of these reinforcement materials has a positive impact on the mechanical properties of the geopolymer composites. Thus, an extensive range of applications should be advocated, specifically in civil construction [1]. Geopolymers have been formed using metakaolin produced from kaolin, which can be obtained in several regional soils. Kaolin is transformed into metakaolin by calcination temperatures ranging from 650°C to 800°C [2].

Plant fibers are less expensive and have low density, and they exhibit good mechanical properties compared to industrial fibers [3-5]. Many researchers have used natural fibers such as basalt, corn husk, wool, jute, rice stem, and fique for reinforcing metakaolin-based geopolymer composites. Adding ten weight percent of 13 μm by 6.35 mm long basalt fibers to potassium-based geopolymer composites yields 19.5 MPa of three-point flexure strength [6]. Sodium-based geopolymer reinforced with

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five-weight percent wool fiber bundles yields 8.1-9.1 MPa three-point flexure strength [6]. By increasing the chopped basalt fiber length to 12.7 mm, it yields 27.07 MPa of three-point flexure strength [7]. Potassium-based geopolymer reinforced with 6.4 wt% of rice stem yields 18.45 MPa three-point flexure strength [8]. The fracture surfaces of the composites are also analyzed using scanning electron microscopy (SEM) [9].

Mechanical bonding in ceramic matrix composites is also a big consideration for achieving toughness, and fiber surface roughness can play an important role in mechanical bonding [10-12]. A higher level of porosity in the matrix or weak matrix encourages toughness [13-14]. The porosity in the matrix provides a means to effectively stop crack propagation by relieving the stresses at the crack tip as it encounters these voids in the matrix [15-17]. The presence of fiber pullout is often consistent with a weak interfacial bond, and it is a significant fracture energy-absorbing mechanism in composites [18].

The interfacial bonding between the fiber and the matrix affects the mechanical properties of the composite material [19-20]. Mechanical locking at the interface also plays a key role in determining interfacial strength [21]. Budiman et al. [22] reported that mechanical locking could contribute to interfacial strength up to 50% under shearing load, which is very significant in influencing overall composite performance.

According to Baranitharan and Mahesh’s research [23], the primary components of the maize stalk are cellulose, hemicelluloses, and lignin. Free sugars starch, protein, extractives, and inorganic are present in trace levels. The cellulose percentage in raw maize fibers was 62.42%, whereas Lignin content was 27.59%. Raw maize fibers were treated with an alkali solution which was 15% diluted. Following the observation, the cellulose and lignin contents of the alkali-treated maize fiber were 69.19% and 13.22%, respectively. In comparison with raw fibers, lignin content is lower and other impurities are eliminated. This study aims to improve the brittle behavior of clay mineral geopolymer by reinforcing it with maize stalk cellulose fibers. The NaOH treatment on the surface morphology of maize stalk cellulose fibers and its loading on calcined kaolin-based geopolymer is to determine the flexural strength and porosity of the composites.

2. Materials and Methods

2.1. *Maize stalk cellulose fibers*

Cellulose fibers extracted from maize stalk through the retting process were used as reinforcement of calcined kaolin-based geopolymer, as shown in Fig. 1. The fiber surface morphology was modified using sodium hydroxide. The measured ultimate tensile stress for maize stalk cellulose fibers treated with 98% purity NaOH for 30 minutes ranges from 625 MPa to 1478 MPa (average 1184 MPa), and the young modulus ranges from 6.73 GPa to 24.11 GPa (average 16.27 GPa) [24].

Fig. 1 Maize stalk cellulose fibers

2.2. *Kaolin clay*

The present study particularly emphasizes Ethiopian kaolinitic clay minerals and their ability to form a geopolymer after thermal and alkaline activation. The chemical or oxide compositions of Burayu sampled soils were tested at the Geological Survey Laboratory of Ethiopia. First, preparation was made to sort the sample soils with a size of 200 meshes. Then, the soils were characterized for oxide composition using the following analytical methods: $LiBO_2$ fusion, high frequency (HF) attack, gravimetric, colorimetric, and atomic absorption spectroscopy (AAS). Metakaolin (calcined kaolin clay) was prepared from kaolin clay using the calcination process. The calcined kaolin can serve as a precursor material in the preparation of the geopolymer matrix. Moreover, sodium hydroxide flake and sodium silicate solution were used to prepare the activating solution to form a geopolymer [24].

2.3. *Sample preparation*

Firstly, the geopolymer was prepared at the construction material laboratory. Calcined kaolinite clay that passed through a 75 μm sieve was mixed with 1.5 wt% dried maize stalk cellulose fibers in a high shear mixer. Secondly, sodium water glass was slowly added to the precursor and mixed until complete integration occurred. Thirdly, the slurry was poured onto a high-strength plastic mold and attached to a vibration table for a more uniform distribution of maize stalk cellulose fibers and fewer voids formation. The filled mold was wrapped in plastic to prevent water loss during setting and curing for 3 hours in a 50°C oven. Then, after three days, the geopolymer composite plate was remolded and set to dry at room temperature for 28 days. Finally, according to ASTM C348 standard, specimens of the maize stalk cellulose-fibers-reinforced calcined kaolin-based geopolymer composites (40 × 40 × 160 mm) were prepared from each mix design for the third-point loading flexural strength test, as shown in Fig. 2.

Fig. 2 Specimens of maize stalk cellulose-fibers-reinforced calcined kaolin-based geopolymer composites (40 × 40 × 160 mm)

2.4. *Characterization*

In addition to SEM for observing the fiber morphology after sodium hydroxide treatment, the formation of geopolymer was also characterized using X-ray diffraction (XRD), Fourier transform infrared (FTIR), and SEM. The analyses of the X-ray diffractometer, Rigaku Miniflex 600, were conducted using X-ray beams generated from a Cu Kα radiation source with a wavelength of 1.54 Å to scan samples from 5° to 60° (2θ) angles. The resolution for this analysis was set at 0.020, with a scan speed maintained at 0.12° per second.

The FTIR equipment used Perkin Elmer spectrum 65 for testing. The samples for this analysis were prepared by the KBr tablet method, which involved mixing the solid sample with a transparent alkali halide (KBr) in a mold that had been subjected to a clamping force. Subsequently, a clear pill was obtained and allowed its intrusion into the analysis equipment. The spectral range that was used to characterize this type of material was between 500 and 4000 cm^{-1}.

To study the fiber morphological properties, the cellulose fibers extracted from the maize stalks using the retting process were treated with 98% purity NaOH for 30 minutes and observed under SEM at Mepco Schlenk Engineering College's Nano-Technology Lab in India. Alkalization is a common pre-processing technique used on base natural fiber to remove hemicelluloses, fats, and waxes that may reduce the interfacial strength when processed into composite forms. Therefore, the extracted cellulose fibers were treated with 98% purity NaOH for 30 minutes.

To investigate the geopolymer morphological properties, the geopolymer was ground to pass 150 μm and observed under an SEM. SEM image of the geopolymer powder was taken under several magnifications to observe the surface. The SEM micrographs of the samples show information about the morphology of a geopolymer.

Characterization of maize stalk cellulose-fibers-reinforced calcined kaolinite-based geopolymer composites was carried out experimentally based on ASTM standards. Controls universal compression/flexural tester was used to measure the flexural strength of the samples, and the flexure strength test was performed following the ASTM C348 standard. Additionally, SEM was utilized to investigate their morphology, microstructure, and failure mechanisms. Finally, porosity analysis was conducted using Image J software based on scanned SEM images.

Flexural strength, which also refers to bending strength, is the mechanical property that measures the stress in the material right before it yields or deforms in a bend test. It is the highest stress loaded to the material at the point of failure. Tests were carried out by a controlled testing machine after 28 curing days as shown in Fig. 3. The test spanning length is 100 mm, while the test crosshead speed is 0.47 mm/s. The three-point flexural strength values of maize stalk cellulose-fibers-reinforced calcined kaolin-based geopolymer composites were tested on 40 × 40 × 160 mm specimens after 28 days of curing time.

Fig. 3 Flexural strength test setup

The morphology of maize stalk cellulose-fibers-reinforced geopolymer composites and the fractured surface observed under an SEM were investigated at Mepco Schlenk Engineering College’s Nano-Technology Lab in India. Despite overcoming the limitations associated with the use of widely accepted experimental procedures, the picture resolution and the segmentation algorithm used to partition the image into the void and grain space have a significant influence on the accuracy of the porosity value generated from 2D images. Characterization allows for examining all fundamental porosity characteristics, including pore volume fraction and pore size.

3. Results and Discussion

3.1. XRD results and discussion

Due to the inherent strong thermal resistance of geopolymers, research has been completed to ascertain the mechanical and micro-logical impacts of exposing different geopolymer cement and mortar to extremely high temperatures. The diffractograms display the intensity of the detected diffraction function of the angle of incidence, where the intensity is characteristic of each crystalline constituent of the sample. After calcination, the Kaolinite disappeared, while the peaks representing quartz remained.

XRD patterns in Fig. 4 displayed the familiar amorphous structure of the metakaolin geopolymer exposed to 800°C with a broad diffraction hump in the range of 25° to 30° (2θ) at ambient curing conditions. The amorphous structure of Metakaolin converted from one structure to another due to geopolymerization process. The result outlooks the XRD intensity of geopolymer exposed to 800°C [25].

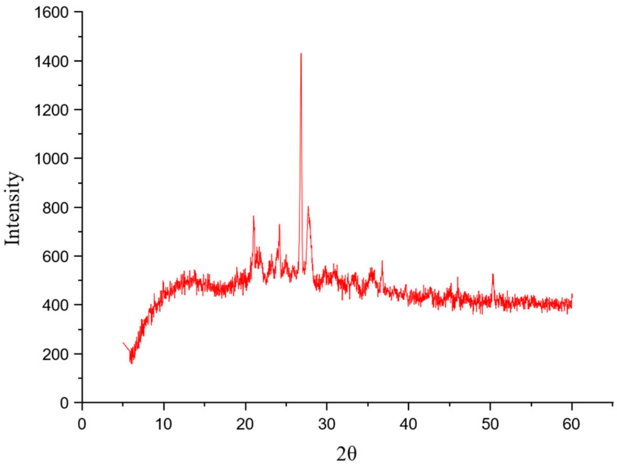


Fig. 4 XRD calcined kaolin-based geopolymer exposed to 800°C

3.2. FTIR spectroscopy

Fig. 5 shows the FTIR transmittance spectra of maize stalk cellulose-fibers-reinforced calcined kaolin-based geopolymer composites. Due to the presence of water in the fibers and geopolymers, the wavenumber positions for molecular vibrations of different bonds are usually present in geopolymers. The strong characteristic peaks between 3000-3500 cm^{-1} and 1650-1655 cm^{-1} are the stretching and deformation of vibration of OH and H-O-H groups within water molecules. However, 900-1300 cm^{-1} corresponds to the Si-OT linkage, and bands around 1400 cm^{-1} are assigned to the Si-O-Si stretching. The bands around 700 cm^{-1} and 660 cm^{-1} show the characteristic of an amorphous polymer formation, which is the Si-O-Si and Si-O-Al symmetric stretching. The peak 537 cm^{-1} originates from Si-O-Al bonds, with Al present in the octahedral coordinate. The presence of maize stalk cellulose fibers in the composites can be recognized by the peak at 1418 cm^{-1}, which is attributed to the CH_3 bending vibration of cellulose. According to Chen et al. [26], this FTIR transmittance spectrum of alkali-activated metakaolin-based geopolymer is also at the peak of 1418 cm-1.

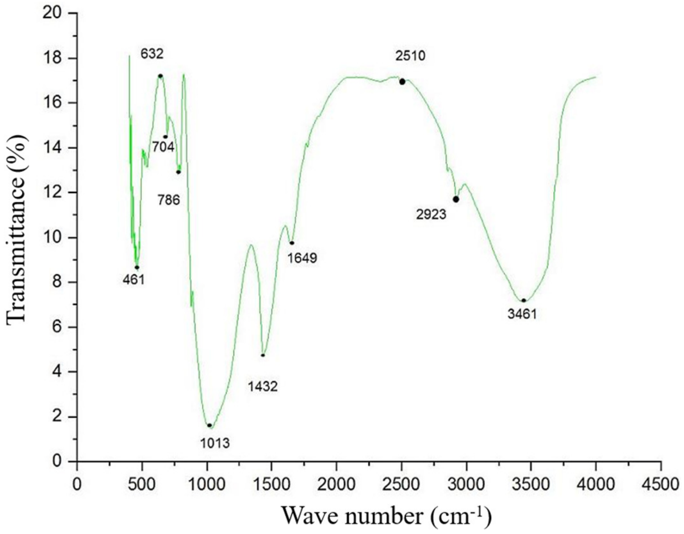
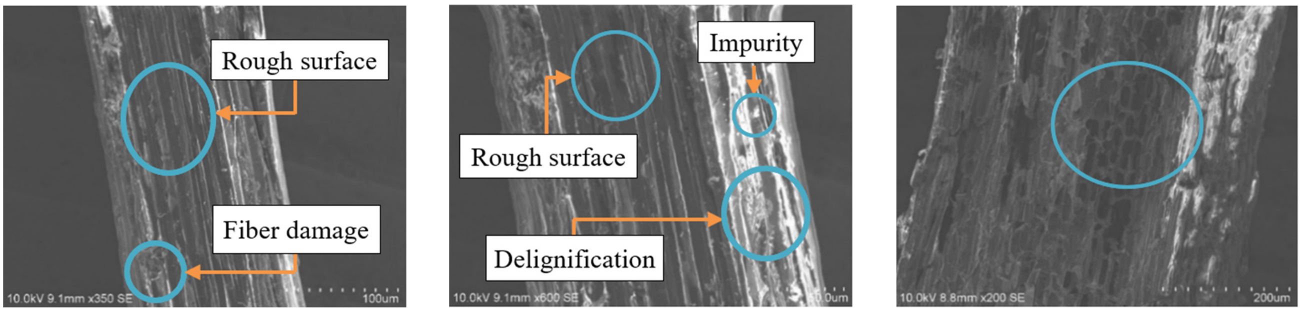


Fig. 5 FTIR transmittance of the composite

3.3. Surface morphology of maize stalk cellulose fibers

The expected changes in the chemical treatment were the diameter of the reduced natural fibers and the hemicellulose. Lignin constituents were partly removed resulting in good surface area and better adhesion between fibers and the matrix. Fiber modification by alkali treatment improves the surface properties considerably in maize stalk cellulose fibers. The rough surface observed in Figs. 6(a)-(b) gives better mechanical interlocking with the geopolymer matrix at the interface, as compared to fiber without treatment as indicated in Fig. 6(c).

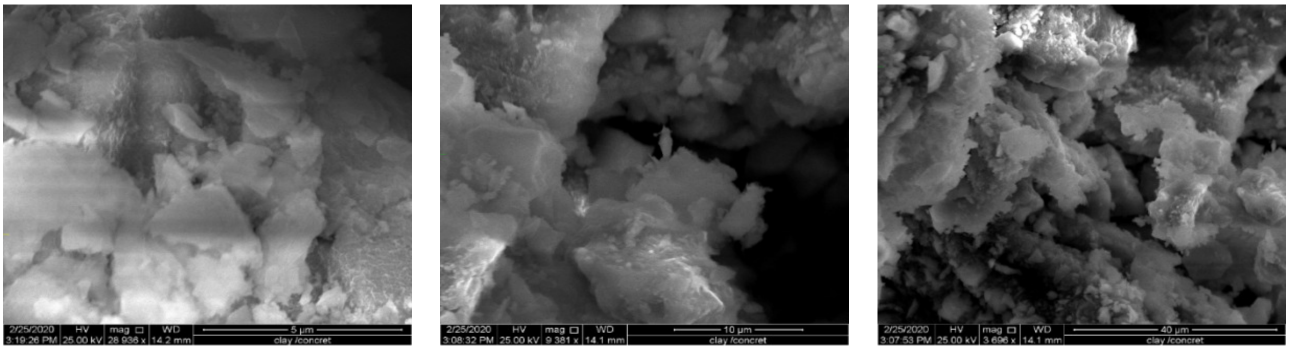


(a) Observed rough surface (100 μm) (b) Observed rough surface (50 μm) (C) Without treatment

Fig. 6 SEM images of maize stalk cellulose fibers

3.4. *Morphology of calcined kaolin-clay-based geopolymer powder*

Representative surfaces of the geopolymer were analyzed using the SEM as shown in Fig. 7. Different morphologies of heterogeneous geopolymers, such as textured spheres, porous, and crystalline-like structures, were observed. Kamarudin et al. [27] researched the SEM micrography approaches and the SEM micrography of kaolin-based geopolymer with various NaOH concentrations. Pure kaolin crystals have a plate-like shape that cannot be seen on a geopolymer constructed from calcined kaolinite. Instead, a gel that resembled a sponge formed indicates that the structure underwent a process of "growth". This demonstrates that the geopolymerization reaction has occurred.



(a) SEM micrograph of geopolymer (5 μm) (b) SEM micrograph of geopolymer (10 μm) (C) SEM micrograph of geopolymer (40 μm)

Fig. 7 SEM micrograph of calcined kaolinite-based geopolymer

3.5. *Third point flexural strength test results and discussion*

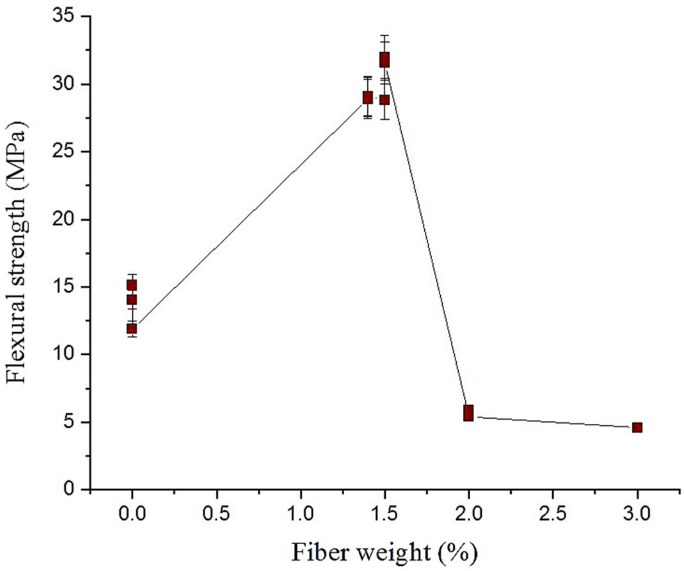
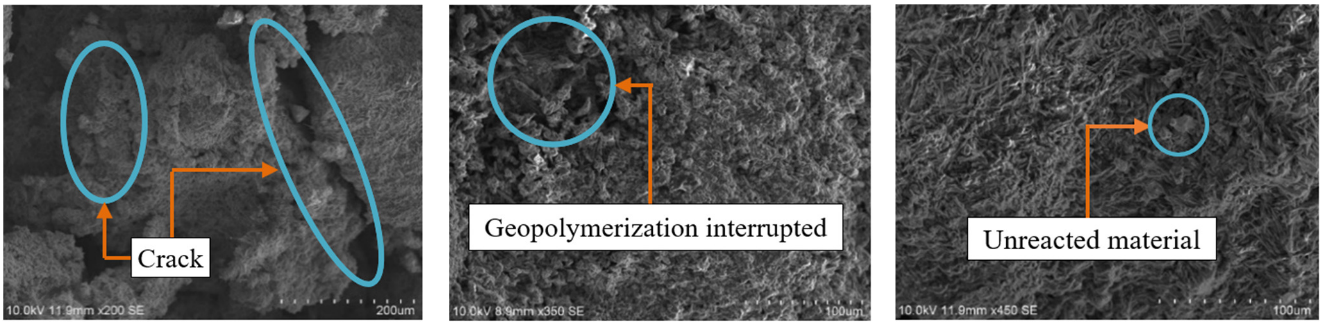


Fig. 8 Flexural strength versus fiber weight (%)

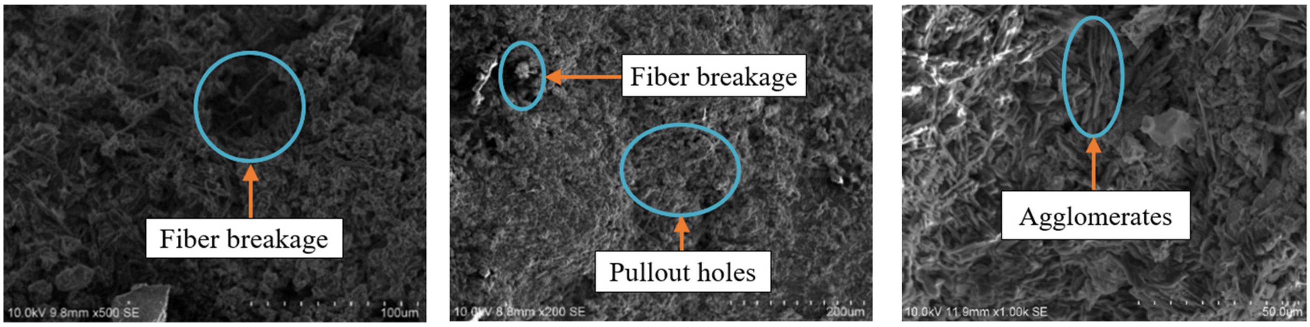
Increasing the strength of the specimen up to a certain level, and then the strength starts declining. For pure geopolymer cured for 28 days, registering the average three-point flexural strength was 13.298 MPa. Nevertheless, the 1.5 wt% treated maize stalk cellulose-fibers-reinforced calcined kaolin-based geopolymer composites cured in an oven at 50°C for three hours have registered average three-point flexural strength of 31.8 MPa, where the optimum percentage of maize stalk cellulose fibers stood at 1.5 wt%. As shown in Fig. 8, composites made from 1.5 wt% NaOH-treated maize stalk cellulose fibers were 2.4 times higher in flexural strength, compared to pure geopolymer. The improvement of flexural strength resulted from the addition of chopped maize stalk cellulose fibers into metakaolin precursors to provide crack control and enhance the bending strength of the composites. The results are comparable with chopped basalt fibers reinforced with potassium-based geopolymer, and it yielded 27.07 MPa of three-point flexure strength [7].

3.6. Morphology of fractured surface

The image was taken under several magnifications to observe the surface. The SEM micrographs of the samples indicate the information about the interface morphology of calcined kaolin clay-based geopolymer and maize stalk cellulose fiber. It is commonly agreed that a rough surface raises the number of anchorage points; hence, better mechanical interlocking with the matrix is achieved. Therefore, when a good bonding is formed at the fiber-matrix interface, the stresses can be adequately transferred to the reinforcement and provide a true reinforcing function.



(a) Observed crack (b) Geopolymerization interrupted (c) Unreacted material under SEM micrograph



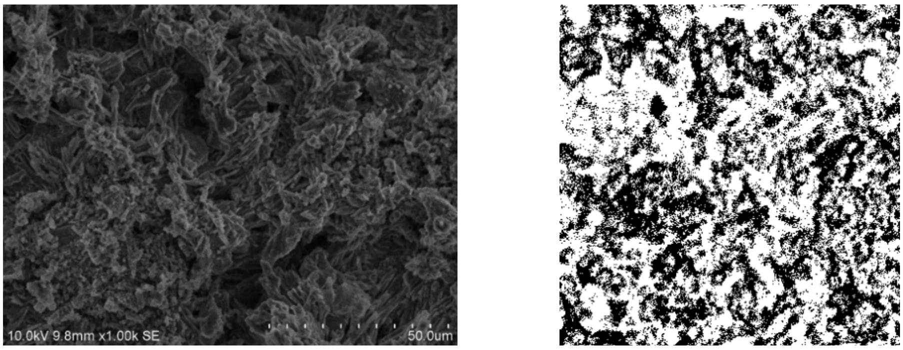
(d) Fiber breakage under SEM micrograph (e) Pullout holes under SEM micrograph (f) Agglomeration under SEM micrograph

Fig. 9 Morphology of the fractured surface of geopolymer composite after the flexural strength test

Due to the applied load, cracks were observed in the pure geopolymer matrix as shown in Fig. 9(a). Geopolymerization interruption and some unreacted precursors were observed in Figs. 9(b) and (c), which retard the strength. The debonding of the fiber and the matrix, fiber breakage, and fiber pullout were observed on the scanned images as shown in Figs. 9(d) and (e), respectively. These failure mechanisms absorb energy during the loading process, resulting in the improvement of the composites' flexural strength and prevention of catastrophic failures during service life. In this study, the rough surface of the maize stalk cellulose fiber enhances its mechanical interlock with the geopolymer matrix. When the fiber volume ratio increases, fiber agglomerates as shown in Fig. 9(f); moreover, the porosity of the composite increases. As a result, when the stress can not transfer adequately from the matrix to the fiber, the flexural strength will decline.

3.7. *Porosity analysis using Image J software*

The porosity of the sample maize stalk cellulose-fibers-reinforced calcined kaolin-based geopolymer composites was analyzed using Image J software. The pore area was observed by SEM. A micrograph of the fractured surface after the flexural strength test is presented in Fig. 10, which is 1280 × 960 pixels in size.



(a) SEM image

(b) SEM image porosity analyzed using Image J software

Fig. 10 The indication of porosity from SEM micrograph

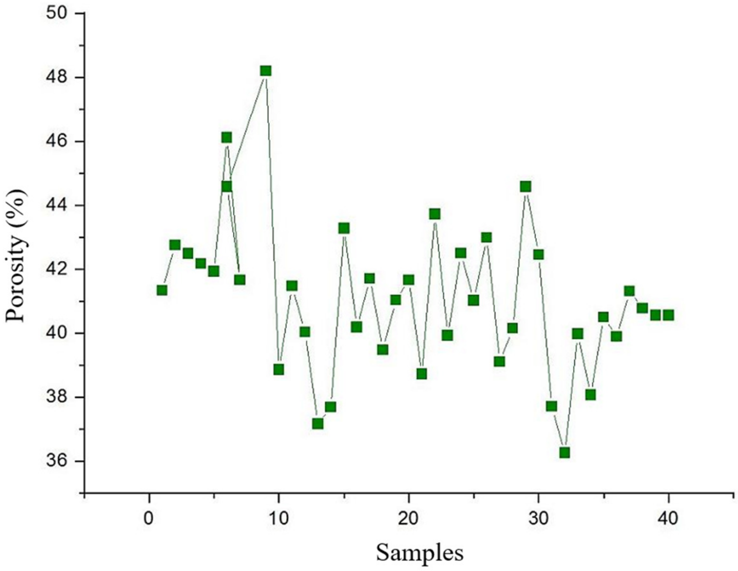


Fig. 11 Porosity size from 0-infinity

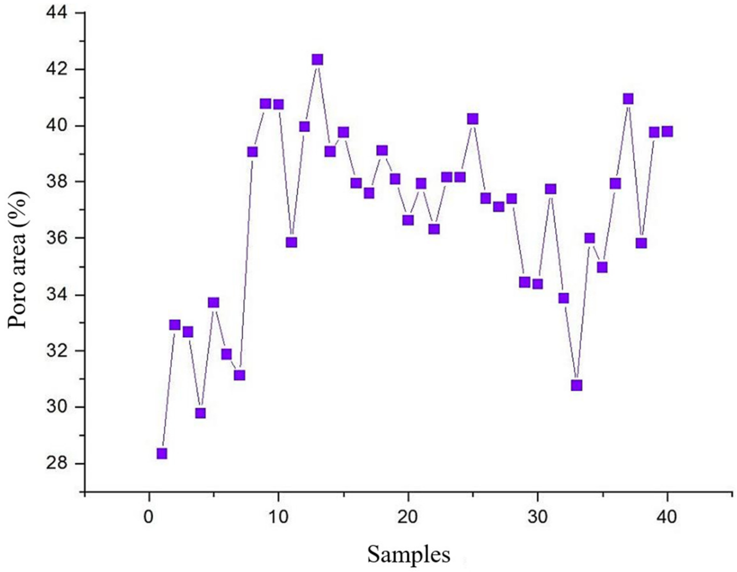


Fig. 12 Porosity from 25 pixel^2-infinity

The pore area size, ranging from zero to infinity, has been analyzed. A graph plotted based on the results is demonstrated in Fig. 11. A pullout of fibers and large particles was considered as porosity, ranging between 36-48%, with an average of 41%. For fiber and particle pullout, a pore area sized 25^2 was set as a parameter based on the fiber diameter. The pore areas with less than 25^2 pixels were excluded from the analysis for fiber and particle pullout. The porosity results, which are shown in Fig. 12, ranged between 28-42%, with an average of 36.7%.

4. Conclusion

The flexural strength of the maize stalk cellulose-fibers-reinforced calcined kaolin-based geopolymer composites was determined based on ASTM standards. The addition of 1.5 wt% maize stalk cellulose fibers in the geopolymer matrix can increase the three-point flexural strength from 13.298 to 31.8 MPa (2.4 times higher). Due to mechanical interlocking, the rough maize stalk cellulose fibers formed interfacial adhesion with calcined kaolin clay-based geopolymer matrix as observed on the SEM micrograph. Debonding of the fiber and the matrix, the fiber breakage, and pullout during the loading process absorbs energy, which improved the composites' flexural strength and prevented catastrophic failures during service life. The pore areas where less than 25^2 pixels were not considered during analysis for fiber and particle pullout, and the result of porosity ranged between 28-42%, with an average of 36.7%.

Conflicts of Interest

The authors declare no conflict of interest.

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