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Mechanical and Water Resistance Properties of Nano TiO_2 Coated Sisal (*Agave sisalana*) Glass Fiber Hybrid Reinforced Composites

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ABSTRACT

Natural fibers are increasingly used as reinforcements in composite materials owing to their sustainability and low cost. However, their poor interfacial bonding with polymer matrices, particularly thermoset resins, often limits their mechanical performance. Surface treatment of natural fibers is a widely adopted strategy for enhancing fiber-matrix adhesion. This study investigated the effect of varying glass and sisal fiber contents on the mechanical and water resistance properties of hybrid composites. The objective of this study was to evaluate the contribution of fiber composition and surface treatment to improved composite performance. Sisal fibers were treated with 1% titanium dioxide (TiO_2) and then combined with glass fibers to reinforce the composite. The volume percentages of the fiber utilized were 30%, 40%, and 50%. A manual lay-up pressing technique was used to fabricate the composites. The thicknesses, swelling, densities, and water absorptions of the composites were also assessed. Furthermore, the American Society for Testing and Materials (ASTM) D 3039 and D 790 tensile and flexural tests were performed on the composite materials. The combination of sisal fibers coated with glass fibers tended to have less water absorption than composites with alternating sisal and glass fibers. The sisal-glass fiber-reinforced composites demonstrated low water absorption (0%–2%) and thickness swelling (0%–1.7%), indicating good moisture resistance. Mechanical testing revealed that a composite with 50% fiber volume fraction in a glass-sisal (G-S-G) configuration achieved a maximum tensile strength of 140.9 MPa. The highest flexural strength (303.5 MPa) and flexural modulus (9.2 GPa) were observed for the composite with 50% fiber volume fraction arranged in a glass-sisal (G-S-G-S-G) configuration. TiO_2-coated glass-sisal fiber composites showed improved strength and water resistance, with potential for semi-structural use. However, challenges, such as cost, environmental impact, and durability, require further study.

Keywords: hybrid composite, sisal fiber, fraction volume, treatment

1. INTRODUCTION

Composite materials have a wide range of applications and have become widely used in recent years (Mouritz and Gibson, 2006), In particular, polymer composites have been used in both academic and industrial

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fields (Lee and Yoo, 2016). Polymer composites have the advantages of low manufacturing cost, low moisture absorption, high strength, no curing requirements, reprocessing flexibility, and resistance to high temperatures (Maras *et al.*, 2024; Oladele *et al.*, 2020; Ozdemir *et al.*, 2023). Polymer composites comprise synthetic fiber reinforcements such as glass, carbon, aramid, and Kevlar, which contribute to environmental pollution (Begum and Islam, 2013). The production of synthetic fibers involves the use of non-renewable fossil fuels and other materials that contribute to global warming by releasing carbon dioxide (CO_2) into the environment. The use of natural fibers as substitute reinforcement materials in polymer-based composite constructions has significantly increased to reduce the detrimental effects of synthetic fibers. With 32 million tons produced worldwide in 2018, the use of natural fibers has expanded owing to the growing awareness of environmental sustainability (Townsend and Sette, 2016). In addition to being widely used in the automobile industry and as reinforcements in composite materials (Sreenivas *et al.*, 2020), natural fibers are widely used in food packaging (Marlina *et al.*, 2021), footwear (da Costa *et al.*, 2013), nautical (Bel Haj Frej *et al.*, 2021), energy (Cortés *et al.*, 2017), and construction (Aida *et al.*, 2022).

Natural fibers provide several benefits, including the reinforcement of composite materials, low density, high tensile strength, cost-effectiveness, and wide availability (Karimah *et al.*, 2021). However, the advantages of natural-fiber-reinforced composites are frequently undermined by the inherent mismatch between the hydrophilic nature of natural fibers and the hydrophobic polymer matrix, and the presence of lignin in natural fibers lowers their tensile strength. Lignin is generally unsuitable for polymer composites because of its complex, irregular structure, poor compatibility with polymers, and brittle nature, which weakens the interfacial bonding and mechanical performance (Collins *et al.*, 2019). One of the main obstacles in using natural fibers as reinforcement is the weak interfacial bonding between the fibers and the matrix, which eventually results in reduced mechanical properties of the composite materials. One drawback of natural fiber–reinforced composites, despite their many benefits, is the poor contact between the fiber and matrix surfaces, which results inweak interfacial strength, low water resistance, and challenging manufacturing.

Natural fiber alteration is one way to address this issue. Fiber modification has long been used to create composite materials with better properties. The fiber orientation, fiber strength, physical characteristics, and adherence of fibers to the matrix affect the performance of natural fiber composites (Mohammed *et al.*, 2015). The mechanical and physical characteristics of natural fibers differ depending on their nature. Therefore, to enhance the performance of natural fiber composites, modifications that improve the fiber-matrix adhesion and optimize the fiber orientation are critical. These adjustments help increase the load-bearing capacity of the composite. Such modifications typically include alterations in the fiber surface and structural changes in the composite components within the polymer matrix. The inherent characteristics of natural fibers often weaken the interfacial bonding with the matrix, negatively affecting the composite's mechanical strength, water resistance, and dimensional stability (Sakthi Vadivel and Govindasamy, 2021).

To enhance the interaction between the fiber and the matrix, several fiber treatments have been investigated, including surface coating (Vančo *et al.*, 2016), evaporation (Liu and Tisserat, 2018), and alkaline treatment (Sakthi Vadivel and Govindasamy, 2021). These procedures improve the connection between the fiber and matrix by eliminating lignin, hemicellulose, and other contaminants from the fiber surface (Jamasri and Yudhanto, 2022). Among these methods, sodium hydroxide (NaOH) treatment is particularly effective in reducing the hemicellulose content of the fibers. Hydroxyl groups (−OH) react with water molecules (H-OH) during alkaline treatment, making it easier to remove them from the fiber structure and lower the hemicellulose, which has an amorphous branching structure.

This article presents the research results by coating with titanium dioxide (TiO_2) on sisal fibers. Previous studies have reported that hybrid composites reinforced with TiO_2 and glass fibers offer significant advantages over conventional glass fiber-reinforced composites. The incorporation of TiO_2 the mechanical performance of the composite, including notable increases in the tensile strength, flexural strength, and modulus, owing to improved load transfer and stronger fiber–matrix interfacial bonding (Arora *et al.*, 2024; Kumar *et al.*, 2023; Meshref *et al.*, 2020). Compared to conventional composites that lack these multifunctional properties, TiO_2–glass fiber hybrid composites represent a significant advancement in the design of high-performance multifunctional materials. The mechanical, morphological, and physical characteristics of reinforced sisal fibers coated with TiO_2 and mixed with glass fibers were studied to determine optimum composite qualities. Mechanical characterization was performed in the form of tensile testing and bending testing of the sisal fiber-reinforced composite.

2. MATERIALS and METHODS

2.1. Materials

Sisal fiber (*Agave sisalana*) was sourced from Sukabumi, West Java, Indonesia. The matrix was an unsaturated polyester (Yukalac 157) hardened using methyl ethyl ketone peroxide (MEKPO) by PT Justus Kimia Raya, Indonesia. Technical-grade NaOH was used for chemical treatment to modify the fiber. A mixture of ethanol (CAS No. 64-17-5, Merck, Darmstadt, Germany), vinyltrimethoxysilane (CAS No. 2768-02-7, TCI Chemical, Tokyo, Japan), and titanium (IV) oxide (CAS No. 13463-67-7, Sigma-Aldrich, St. Louis, MO, USA) was used to improve the quality of the composites.

2.2. Methods

The fibers used were first cleaned and dried, and then treated with 5% NaOH for 2 h. Then, the fibers soaked in NaOH were rinsed with clean water and placed in an oven at 60℃ for three days. The moisture content of the fibers was less than 10%. Subsequently, the alkali-treated fibers were coated with TiO_2 following a silane coupling agent and an ethanol mixture (1:10) and stirred for 15 min. TiO_2 was added at concentrations of 1% and stirred for 30 minutes. The fibers were submerged in the solution for half and then rinsed with distilled water. Additionally, the material underwent an eight-hour drying procedure at 100℃. The press-hand lay-up process, which entails combining the resin and hardener (4% of the resin weight), putting the matrix into the mold, covering the fiber layer by layer, and then cold-pressing it for three hours, was used to create the composite. A longitudinal orientation characterizes the sisal fibers. There were several combinations of chopped mats, glass, and sisal fibers. After that, the sample is taken out of the mold and baked for 24 h at 60℃. The sample was conditioned for one to three days at room temperature (27℃) in an open area. The manufacturing process of the hybrid reinforced composites used in this study is shown in Fig. 1.

A Shimadzu 10KN Universal Testing Machine (UTM) was used for tensile and flexural testing under the American Society for Testing and Materials (ASTM) D 3039 and ASTM D 790 requirements. The ASTM E23 standard with a V-notch was used for impact testing. The composite cross-sectional morphology was observed using a KEYENCE VHX 6000 digital microscope and a Prisma scanning electron microscope (SEM). Table 1 shows the arrangement of the sisal and glass fiber layers in the composite composition. IBM SPSS Statistics version 23 (IBM, Armonk, NY, USA) was used to analyze the data. The data were analyzed by the research was conducted using a single-factor, fully randomized

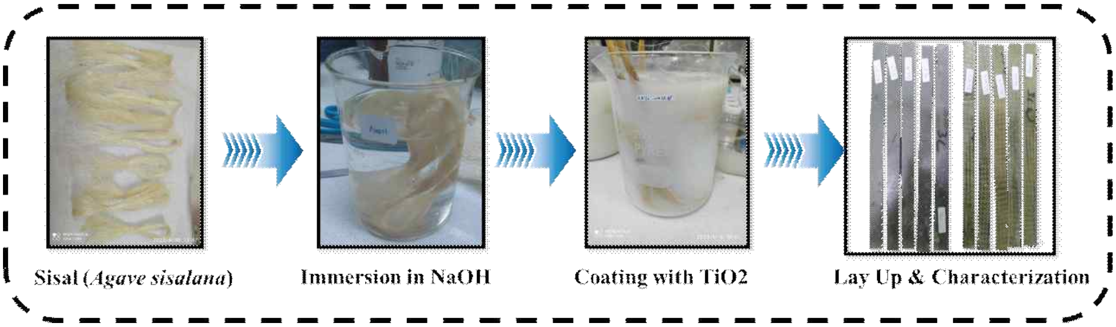

Fig. 1. Manufacturing of the hybrid reinforced composite in this study.

Table 1. Composite composition

Type of hybrid reinforced composite	Layering sequence	Sisal fiber volume fraction (%)	Glass fiber volume fraction (%)
G1	G-S-G	15	15
G2	G-S-G	20	20
G3	G-S-G	25	25
GS1	G-S-G-S-G	15	15
GS2	G-S-G-S-G	20	20
GS3	G-S-G-S-G	25	25

G: glass fiber, S: sisal fiber.

design (type of hybrid reinforced composite). The hybrid reinforced composite properties were evaluated using analysis of variance (ANOVA), and Duncan's multiple range test, with *p*-values $\leq$ 0.05 indicating statistical significance.

3. RESULTS and DISCUSSION

3.1. Mechanical properties

3.1.1. Tensile test

The tensile strength of the sisal-glass-fiber hybrid-reinforced composite was influenced by the proportion of glass fibers and the fiber volume fraction, as shown in Table 2. With an increase in the fiber volume fraction, the tensile strength of the sisal glass fiber hybrid-reinforced composites increased. The tensile strength of the sisal-glass fiber-reinforced composites reached the optimum value in the combination type of glass fiber sisal with a fiber fraction value of 50%, which reached 140.9 MPa. The fiber content of 50% had the highest value for the composition of sisal fiber and glass fiber type G (glass fiber as a composite cover) and type GS (sisal fiber and glass fiber were arranged alternately). This tensile strength value was higher than that reported in a previous study (108 MPa), which used a three-layer fiber composition with glass fiber as the outer layer and the bamboo fiber in the middle (Vaghasia and Rachchh, 2018). Adding sisal fiber and glass fiber by 20% in type G composites can increase tensile strength by up to 46% (96.5 MPa to 140.9 MPa), while in type GS composites, it is 60% (85.8 MPa to 136.7 MPa). A previous study

Table 2. Tensile strength and elastic modulus of hybrid reinforced composites

Type of hybrid reinforced composite	Tensile strength (MPa)	Elastic modulus (GPa)	Strain (%)
G1	96.53 (7.40)[ab]	1.78 (0.63)[a]	7.54 (1.36)[ab]
G2	107.16 (21.09)[bc]	2.04 (0.27)[a]	7.17 (0.73)[ab]
G3	140.93 (17.76)[d]	1.77 (0.20)[a]	8.36 (1.09)[b]
GS1	85.83 (10.92)[a]	1.42 (0.22)[a]	7.23 (0.75)[ab]
GS2	124.52 (13.22)[cd]	2.23 (0.16)[a]	6.77 (0.76)[a]
GS3	136.75 (17.03)[d]	2.02 (1.35)[a]	7.38 (0.37)[ab]

Values in parentheses are SDs.
[a–d] Values followed by the same letter are not statistically different ($p < 0.05$) according to Duncan's multiple-range test.

reported that an improvement in tensile strength was obtained by varying the bamboo fiber composition from 3% (72 MPa) to 9% (108 MPa) and then decreasing successively with an increase in the bamboo fiber percentage from 12% (88 MPa) to 15% (42 MPa; Vaghasia and Rachchh, 2018). The elastic modulus value tends to fluctuate for various variations of glass fiber and sisal fiber, ranging from 1.7–2.2 GPa. The composite strain value tended to be stable at 7%, except for the strain value in the sisal-glass fiber-reinforced composite with a combination of glass fiber-sisal fiber-glass fiber with a volume fraction of 50% (v/v) of 8.3%.

3.1.2. Flexural test

The flexural test results are listed in Table 3. The fiber type and volume fraction directly affect the flexural strength of the sisal-glass fiber hybrid-reinforced composites. The flexural strength value can be increased by up to 41%, or from 217.2 MPa to 303.5 MPa, by adding fibers up to 50% of the volume fraction, as shown in Table 3. However, the flexural strength of the sisal-glass fiber-reinforced composites increased by 15% with a 10% increase in the fiber volume percentage. The flexural strength above was almost the same as the research results of Shifa *et al.* (2024), who developed a hybrid composite combination of woven glass and cotton fibers,

Table 3. Flexural strength and flexural modulus of hybrid reinforced composites

Type of hybrid reinforced composite	Flexural strength (MPa)	Flexural modulus (GPa)	Strain (%)
G1	217.29 (12.66)[a]	6.39 (1.36)[a]	4.34 (0.64)[a]
G2	250.07 (7.34)[b]	7.13 (0.38)[ab]	4.06 (0.29)[a]
G3	246.19 (22.83)[b]	7.07 (1.23)[ab]	4.01 (0.34)[a]
GS1	214.71 (14.84)[a]	6.30 (0.98)[a]	4.21 (0.86)[a]
GS2	259.89 (25.72)[b]	7.98 (0.96)[bc]	4.02 (0.43)[a]
GS3	303.54 (14.62)[c]	9.20 (1.13)[d]	3.68 (0.31)[a]

Values in parentheses are SDs.
[a–d] Values followed by the same letter are not statistically different ($p < 0.05$) according to Duncan's multiple-range test.

which reached 65–265 MPa. Using a combination of sisal and glass fibers alternately in a GS-type composite impacts increasing flexural strength by 24% at a fiber composition of 50%. Table 3 lists the flexural moduli of the sisal-glass-fiber hybrid reinforced composites. The flexural modulus at 30% fiber content was 6.3 GPa, and at 50% fiber fraction volume, it increased to 9.2 GPa. Both the fiber volume percentage and the fiber-glass fiber combination increased the flexural modulus.

3.1.3. Impact test

Table 4 shows composites' impact strength and energy graphs reinforced with a combination of sisal and glass fiber. Adding sisal and glass fiber to the G-type composite by 20% increases the composite impact value by 4% and is optimal at a fiber volume fraction of 40%, 0.28 J/mm^2, an increase of 14.6%. Meanwhile, adding a fiber volume fraction of 20% in the GS-type composite can increase the impact strength value by 17.6%, with a maximum value at a volume fraction of 50% of 0.27 J/mm^2. Overall, the largest impact energy was obtained in the composition of sisal fiber and glass fiber, with a volume fraction of 50% in the GS-type composite, which was 7.67 J. The impact strength value above was greater than the impact strength value of the research results of Nayak *et al.* (2022), which used a combination of jute-bamboo fiber and glass fiber, around 0.06–0.1 J/mm^2. Through hybridization, glass fiber was incorporated into natural fiber-reinforced polymer composites to boost their impact strength. Under hydrothermal circumstances, the absorbed water increase flexibility which in turn improved composite materials' impact strength (Nayak *et al.*, 2022). The results of Costa *et al.* (2020) who employed mallow fiber and epoxy matrix, werefollowed by the rise in impact energy and the addition of fiber volume percent.

3.2. Water resistance properties

Table 5 presentsthe findings of the physical and water resistance characterization. The densities of sisal-glass fiber hybrid-reinforced composites varied between 1.32 and 1.35 g/cm^3, with the densities of reinforced composites composed of five layers of sisal and glass fiber having a higher value. The densities of the hybrid-reinforced composites made of sisal and glass fibers ranges from 1.32 to 1.35 g/cm^3. Generally, the density of the composite is only slightly affected by variations in the sisal and glass fiber combinations. As more sisal fiber volume fractions are added, the water absorption value of the sisal-glass fiber-reinforced composite increased from 0% to 2%. The performance of the composites with a combination of glass and sisal fibers showed that glass fibers have a smaller water absorption value than

Table 4. Impact energy and impact strength of hybrid reinforced composites

Type of hybrid reinforced composite	Energy (J)	Impact Str (J/mm^2)
G1	5.50 (0.35)^{ab}	0.25 (0.02)^{ab}
G2	6.33 (0.41)^{b}	0.28 (0.02)^{b}
G3	5.67 (0.41)^{ab}	0.24 (0.02)^{ab}
GS1	4.50 (0.61)^{a}	0.23 (0.03)^{a}
GS2	4.67 (0.41)^{a}	0.22 (0.02)^{a}
GS3	7.67 (1.95)^{c}	0.27 (0.06)^{ab}

Values in parentheses are SDs.

$^{a-c}$ Values followed by the same letter are not statistically different ($p < 0.05$) according to Duncan's multiple-range test.

Table 5. Physical properties of hybrid reinforced composites

Type of hybrid reinforced composite	Density (g/cm^3)	Water absorption (%)	Thickness swelling (%)
G1	1.32 (0.06)^a	0.00 (0.00)^a	0.00 (0.00)^a
G2	1.33 (0.08)^a	0.86 (0.06)^c	0.33 (0.02)^b
G3	1.39 (0.03)^a	1.21 (0.01)^d	1.77 (0.01)^d
GS1	1.31 (0.11)^a	0.41 (0.13)^b	0.00 (0.00)^a
GS2	1.33 (0.09)^a	0.84 (0.01)^c	0.32 (0.03)^b
GS3	1.35 (0.06)^a	2.07 (0.03)^e	0.48 (0.02)^c

Values in parentheses are SDs.
$^{a–e}$ Values followed by the same letter are not statistically different ($p < 0.05$) according to Duncan's multiple-range test.

composites with a combination of glass and sisal fiber alternating hoses. The swelling thickness of the sisal-glass fiber-reinforced composite has a thickness swelling –of 0%–1.77%. Composites with alternating glass and sisal fibers exhibited better dimensional stability than those reinforced with sisal and glass fibers, with a maximum value of 1.2%. The high water absorption level in natural fiber-reinforced polymer matrix composites was caused by the abundance of polar hydroxyl groups in the natural fibers. This enabled water absorption to approach 2% at a volume percentage of 50%.

3.3. Morphological characterization

The fracture cross-section of the tensile test specimen of the sisal fiber-reinforced composite is shown in Figs. 2 and 3. The unsaturated polyester matrix appeared to be predominant at low fiber volume percentages. The composite fractures appeared more brittle and elastomeric at high fiber volume fractions, whereas they appeared less elastic at low fiber volume fractions. SEM analysis of the hybrid glass and sisal composites revealed distinct fracture behaviors in the tensile test specimens. Micrographs of the 3-layer and 5-layer composites showed that the sisal fibers underwent pull-out and entanglement with random orientation after fracture, indicating non-brittle failure and cracks or interfacial voids caused by fiber extraction during tension. In contrast, the glass fibers exhibited brittle failure characterized by smooth, flat fracture surfaces with the matrix material remaining on the fibers, demonstrating a strong fiber-matrix bond. For hybrid composites, the analysis highlighted brittle failure in glass fibers and pull-out behaviors in sisal fibers, reflecting the contrasting failure mechanisms of the two materials as glass fibers have a more uniform size than sisal fibers (Das *et al.*, 2021; Sekar *et al.*, 2020). Figs. 2 and 3 show that composites with high volume fractions (50%) tend to be denser, and some fiber components are not fully soaked in the polyester.

The TiO_2 coating chemically enhanced interfacial adhesion by forming hydrogen bonds or covalent linkages with the polymer matrix, while physically increasing surface roughness to promote mechanical interlocking. These effects improved stress transfer and reduce fiber pullout, leading to improved tensile and flexural properties. Additionally, TiO_2 acted as a barrier to moisture by covering hydrophilic sites on the fiber surface, thereby reducing water uptake and preserving the fiber-matrix interface (Ma *et al.*, 2018; Xu *et al.*, 2019).

The performance gains observed in the composite are attributable to the combined effects of both the hybrid fiber structure and the TiO_2 surface treatment. The

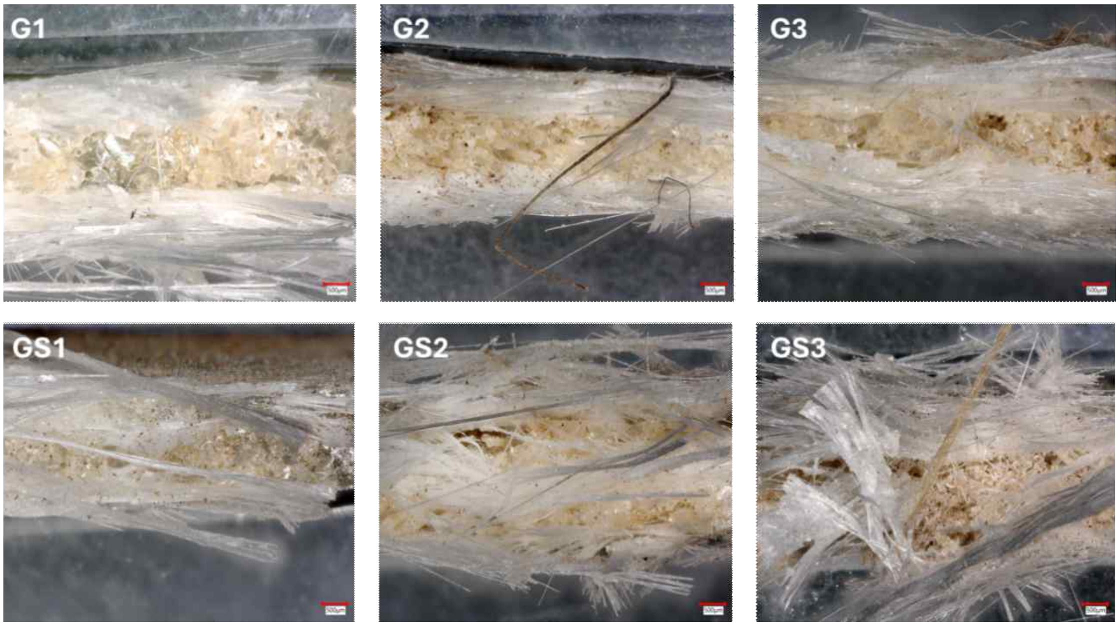

Fig. 2. Morphology of hybrid reinforced composites with the optical microscope.

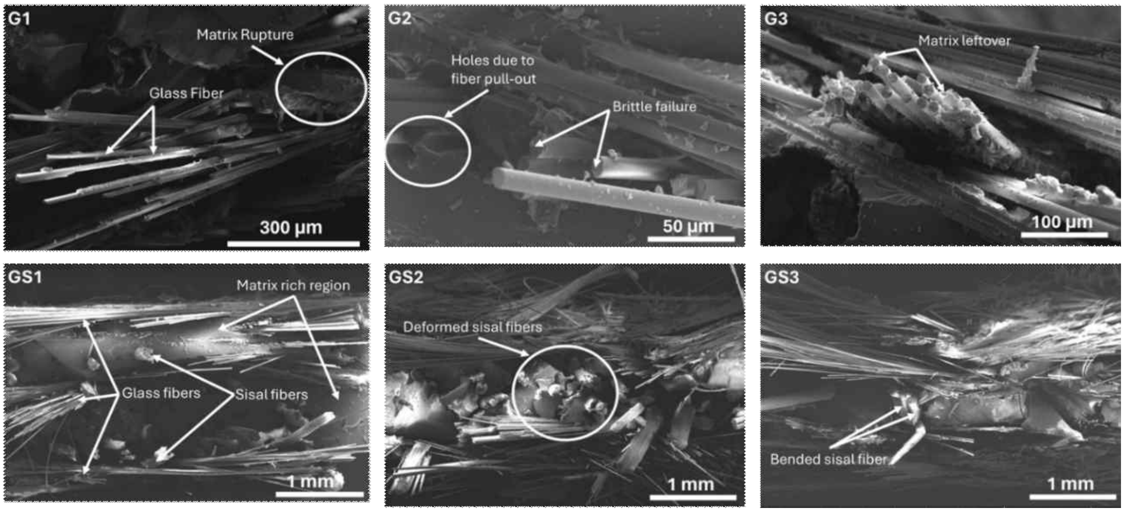

Fig. 3. SEM of hybrid reinforced composites. SEM: scanning electron microscope.

hybridization of sisal and glass fibers enhanced the mechanical performance by leveraging the high strength of glass and toughness of sisal, resulting in improved stress distribution and load-bearing capacity. Meanwhile, the TiO_2 coating enhanced fiber-matrix adhesion through chemical bonding and surface roughening, while also improving water resistance by reducing the availability of hydrophilic sites on the fiber surface. These two strategies acted synergistically, providing greater improvements than either approach alone (Arora *et al.*, 2024; Kumar *et al.*, 2023; Meshref *et al.*, 2020).

4. CONCLUSIONS

The density of the sisal-glass fiber-reinforced composite tended to be stable with an increased fiber volume fraction and variation in the sisal-glass fiber combination. The combination of sisal fibers coated with glass fibers exhibited lower water absorption than composites with alternating sisal and glass fibers. The composite density ranged from 1.32–1.35 g/cm^3, water absorption from 0%–2%, and thickness swelling from 0%–1.7%. The composites with 50% fiber volume fraction and a glass-sisal-glass fiber combination had a maximum tensile strength of 140.9 MPa. With the same fiber volume fraction and a sisal-glass fiber combination, the maximum flexural strength was 303.5 MPa, and the flexural modulus was 9.2 GPa. The fiber pull-out phenomenon increased with an increase in the fiber volume fraction because the sisal-glass fiber-reinforced composite exhibited brittle cracks.

The enhanced mechanical strength and water resistance of the TiO_2-coated sisal-glass fiber hybrid composites suggest strong potential for use in semi-structural applications such as automotive panels, building materials, and marine components where lightweight, durable, and moisture-resistant materials are required. However, certain limitations must be considered for real-world implementation. These include the scalability and cost of the TiO_2 surface treatment process, potential environmental concerns related to nanoparticle use, and long-term performance under varying environmental conditions such as UV exposure and temperature cycling. Future studies should focus on life-cycle assessment, environmental durability, and industrial processing to fully validate the practical utility of this composite.

CONFLICT of INTEREST

No potential conflict of interest relevant to this article was reported.

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