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Production and Characterization of Silane-treated Sisal and Flax Fibers with Mechanical Properties Enhanced through TiO_2 Reinforcement

S. Mohammad Malik Mubeen[1*], M. Shanmuga Priya[2] and M. Vijayaraj[3]

[1]Department of Electrical and Electronics Engineering, M.A.M. School of Engineering, Tiruchirappalli, TN, India

[2]Department of Computer Science and Engineering, M.A.M. College of Engineering, Tiruchirappalli, TN, India

[3]Department of Electronics and Communication Engineering, Government College of Engineering, Tirunelveli, TN, India

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*malikmubeen87@gmail.com

ABSTRACT

This study examines the mechanical characteristics of a novel hybrid composite of sisal/flax/glass fiber and various percentages of titanium dioxide particles. Sisal fiber is renowned for its exceptional tensile rigidity and durability, while flax fiber is highly prized for its abundant availability and biodegradability. The purpose of including titanium dioxide particles is to enhance the mechanical characteristics of the composite. Silane treatment is done for both sisal and flax fibers. Mechanical tests, such as tensile and bending, microhardness, and fracture toughness, were conducted to evaluate the composite's behavior. The results were encouraging, indicating an average tensile strength of 77.16 MPa, flexural strength of 94.3 MPa, microhardness of 62, and fracture toughness of 1.82 MPa $m^{0.5}$. These findings suggested that the material can be used in technical applications that demand exceptional strength, rigidity, and resistance to impact. Incorporating glass fibers and titanium dioxide particles results in a harmonious amalgamation of functionality and ecological soundness.

Keywords: Mechanical properties; Chemical treatment; Silane; Nanocomposites; Nanoparticles.

1. INTRODUCTION

Over the last 10 years, researchers and engineers have shown considerable interest in natural fibers because of their distinctive characteristics, which make them suitable substitutes for synthetic fibers in composite materials (Thanikodi *et al.* 2024; Arunachalam *et al.* 2024). Natural fibers have several advantages that include cost-effectiveness, favorable mechanical qualities, high strength-to-weight ratio, environmental friendliness, capacity to decompose naturally, decreased health risks, fewer energy requirements, and non-abrasive nature (Solairaju *et al*. 2024; Valin Rivera *et al.* 2023). The increasing interest in green composites has emerged due to their status as ecologically benign materials, which are projected to be the materials of the future (Melo *et al.* 2020). These applications include secondary and tertiary structures, sheets, automobile interior spaces, athletic apparel, recreation items, wrapping, and development (Maurya *et al*. 2021; Gudayu *et al.* 2022; Arunachalam *et al.* 2024). Renewable resources offer the possibility of substituting synthetic reinforcements like glass and carbon fibers with natural fibers (Chowdary *et al.* 2022). Flax, coir, sisal, hemp, kenaf, and jute are often utilized as natural fibers in polymer composites (Dhanasekar *et al.* 2023).

(Oksman *et al.* 2002) revealed that horseshoe-shaped sisal fiber bundles were not uniform throughout the matrix. In contrast to numerous hardwood composite materials, the polymer binder did not occupy the lumens. The actual modulus of technical sisal fibers in the laminates was greater, with a measured value of 40 GPa, compared to the 24 GPa obtained in the technical fiber test. Nevertheless, the actual strength of the technical fibers in the composites was around 400 MPa, which is lower than the recorded tensile strength of 550 MPa technically. An analysis was conducted to investigate the fundamental causes behind these observations. (Sundeep *et al.* 2022) enhanced the mechanical and thermal characteristics of sisal and flax fibers. Natural fibers have increased significant recognition as alternatives to synthetic materials because of their exceptional ecological advantages. The findings suggested that the composites exhibited improved thermal and mechanical capabilities, with distinct attributes based on the flax fiber concentration. As a result, these advanced composites can be customized depending on the individual requirements of the user. (Jeffrey *et al.* 2021) investigated the influence of combining flax fiber with sisal fiber-polypropylene polymers on their physical qualities. The interaction between the fibers and polypropylene was improved. The investigators discovered that the addition of flax fibers to the polypropylene-silica composite materials resulted in enhanced tensile strength, mobility, and impact strength. Moreover, the incorporation of flax fibers significantly improved the water resilience of the materials. (Samouh *et al.* 2021) used a technique that allows for the evaluation of the mechanical characteristics of the fiber. Furthermore, a comprehensive assessment and analysis were conducted to determine the physical and mechanical qualities of the produced sisal yarns. The results demonstrated substantial promise for utilizing Moroccan sisal fiber in the advancement of bio-sourced hybrid materials. Sisal and flax fibers were positioned to have a substantial impact on the manufacturing of different car parts through the utilization of epoxy resin (Jothi *et al.* 2024; Rachoti *et al.* 2022). This study investigated the mechanical properties (tensile, flexural, compression), and fire-retardant characteristics of composites containing various percentages of weight of sisal and flax fibers modified by NaOH. The materials were produced with the compression molding method.

The findings suggested that composites containing 40 wt. % fiber content provides improved strengths in comparison to other materials. (Kumar *et al.* 2020) assessed the mechanical characteristics, resistance to wear, and force of friction of materials with a unidirectional hybrid fiber alignment. The fibers were aligned by hand according to the dimensions and depth of the manufactured plates. The results suggest that the unidirectional hybrid made from sisal, carbon, and flax fibers demonstrated exceptional mechanical and wear properties. Oval and Erhan, (2022) examined the impacts of several surface treatments on the mechanical characteristics of LDPE/jute composites. The materials were tested with and without the use of maleic anhydride additive. The findings indicated that subjecting sisal fibers to alkali and silane procedures led to a significant enhancement of almost thirty percent in the mechanical characteristics of the materials in comparison to untreated LDPE. Incorporating maleic anhydride into the polymer in ideal ratios with unprocessed jute fiber resulted in a significant enhancement of the mechanical characteristics of the materials. (Sajin *et al.* 2021) investigated the impact of fiber length on the mechanical characteristics of polyester materials enhanced with jute plant fibers; tests were conducted under ASTM standards to assess the mechanical characteristics of the manufactured materials. The findings indicated that the optimal characteristics were obtained with a fiber length of 5 mm.

2. EXPERIMENTAL WORK

2.1 Materials

The weaved mats made of sisal, flax, and glass fiber were purchased from Hayel Pvt. Ltd., Chennai, India. Epoxy resin and hardener were obtained from Jevanthee Enterprise Pvt. Ltd., Chennai, India. Titanium dioxide (TiO_2) nanoparticles with a size of 15 nm were acquired from Adnano Technologies Pvt. Ltd., Bangalore, India.

2.2 Silane Treatment for Sisal and Flax Fibers

The sial and flax fiber mat were extensively washed with refined water to eliminate any dust. The fibers were dried in a heated air oven at 100 °C for 4 hours following the washing process. The desiccated fibers were subsequently subjected to a silane treatment to eliminate the wax, oil, hemicellulose, and lignin content. The efficacy of silane treatment is contingent upon the temperature, duration, and concentration of the solution. After immersing the sisal and flax fiber mats in 5% (5 g of silane in 100 g of water) and 10% silane solutions for 4 hours, with intermittent stirring, the fibers were extensively washed with deionized water and dried again in a hot air oven at 100 °C for 4 hours following the treatment. This silane treatment improves the interaction between the fiber and matrix by increasing the crystallinity of the fiber. The aspect ratio is enhanced by the development of an uneven surface, which results in improved strength.

Fig. 1: Tensile Test Machine

2.3 Composite Preparation

Layers of sisal, flax, and glass fiber mat, each measuring 300 mm x 300 mm, were reinforced with epoxy, and mixed with a hardener in a ratio of 10:1. Both silane-treated and nanoparticles were used to manufacture the composite lamina. Composites with 0, 0.5, 1, 1.5, and 2% nanoparticle fibers were produced, and conclusions were drawn based on tensile and flexural microhardness, and fracture toughness test outcomes. The fibers were placed between two wooden plates for easy removal of laminate after fabrication. Even pressure was given to all specimens, which were then left for 24 hours, followed by curing at 80 °C for 4 hours and 100 °C for 2 hours (Jothi *et al.* 2024). Matrix modification was done using TiO_2 nanoparticles of size 15 nm to improve mechanical properties. The nano-TiO_2 was mixed into the epoxy resin using mechanical stirring at

1500 rpm for 60 minutes, followed by mixing with the hardener. Hand layup approaches were used to manufacture the hybrid composite. The tensile test machine is shown in Fig. 1.

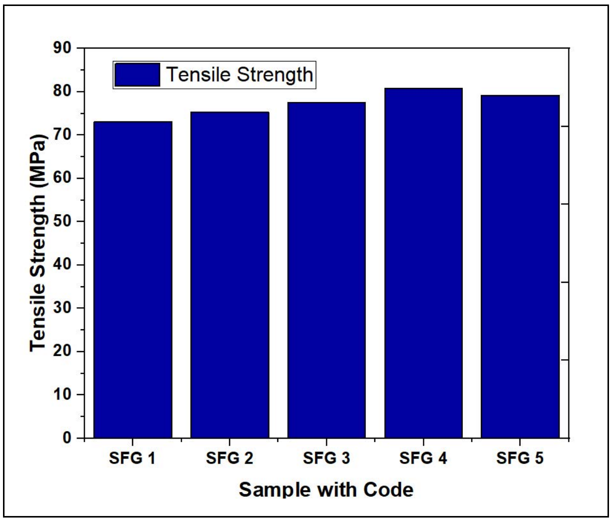

Fig. 2: Tensile Strength of the specimens

Table 1: Results of the Tensile Test

Specimen	Fiber %	Titanium dioxide %	Epoxy %	Tensile Strength (MPa)	Young' s Modulus (GPa)	Elongation at break %
SFG1	55	0	45	73.1	4.5	3.0
SFG2	55	0.5	44.5	75.3	4.9	3.3
SFG 3	55	1	44	77.5	5.1	3.5
SFG 4	55	1.5	43.5	80.7	5.5	3.9
SFG5	55	2	43	79.2	5.2	3.6
		Average		77.16	5.04	3.46
		Standard Deviation		2.71	0.33	0.30

(S-sisal Fiber, F-flax fiber, and G-glass fiber)

3. RESULTS AND DISCUSSION

A. Tensile Strength

Based on the tensile test data analysis, the entire laminated material had an average tensile strength of 77.16 MPa, with individualized material values varying from 73.1 to 80.7 MPa, as depicted in Fig. 2. This composite material exhibited a median Young's modulus of 5.04 GPa, which suggested that it is highly resistant to displacement under tensile stress. An additional characteristic of the composite material is its average elongation at the break of 3.4%, which suggests that it can endure deformation before failure. The findings of the tensile experiments performed on the hybrid laminated materials, which consist of sisal, flax, and glass fibers, along with TiO_2 nanoparticles, are presented in Table 1.

The standard deviation values for elongation at break, Young's modulus, and tensile strength are 0.30%, 0.33 GPa, and 2.71 MPa, respectively. These parameters demonstrate that the material properties of the specimens are uniform, as they reveal an assortment of results within the sample set. Hybrid fiber and TiO_2 nanoparticles serve as a synergistic reinforcement system and are responsible for the composite laminated material's tensile strength. The composite material's ultimate strength is significantly influenced by the inherent rigidity and tensile strength of hybrid fibers. Furthermore, the mechanical characteristics of the material are further improved by the TiO_2 nanoparticles, which serve as reinforcing agents. The Young's moduli and elongation at the break of the specimens are depicted in Fig. 3.

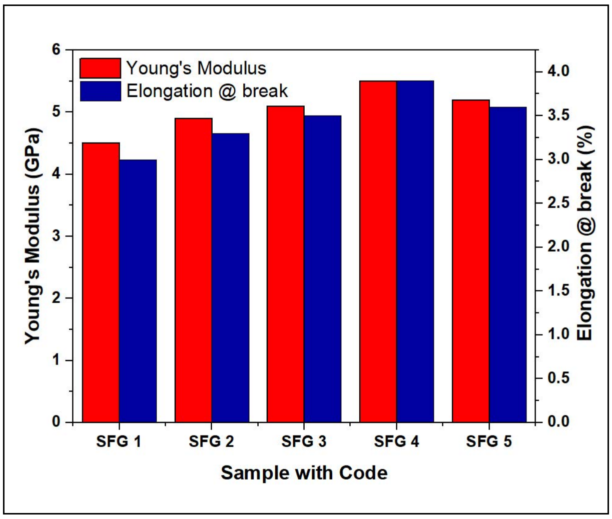

Fig. 3: Young's Modulus and Elongation at the break of the specimens

Additionally, the tensile strength of the layered material is improved by the transmission of stress and the prevention of layer separation, which is contingent upon the firm bonding between the fibers and the matrix. Fig. 3 demonstrates the amount of elongation that the specimens experienced before fracturing. Finally, the hybrid composite's capacity for applications necessitating both remarkable rigidity and strength is illustrated by the tensile test results. Its ductile qualities are indicated by its capacity to endure deformation till its failure. Agglomeration, which generates weak points in the matrix, is the cause of the decrease in tensile strength observed with nanoparticles containing more than 1.5 wt.%. Poor bonding can result from the disruption of stress transfer caused by an overabundance of nanoparticles. Furthermore, the overall strength may be impacted by interfacial stress concentration or compatibility issues with the matrix at higher concentrations.

B. Flexural Strength (FS)

As depicted in Fig. 4, the hybrid laminate material's average flexural strength is 94.3 MPa, with each specimen varying from 90.7 MPa to 98.6 MPa, as indicated by the flexural strength test. Furthermore, Fig. 5 illustrates that the mean flexural modulus, which quantifies the material's rigidity in response to bending, is 5.2 GPa. The findings of the flexural test for the composite lamination comprising sisal fiber, flax fiber, glass fiber, and TiO_2 nanoparticles are summarized in Table 2.

Table 2: Results of the Flexural test

Sample	Fiber (%)	TiO_2 (%)	Epoxy (%)	Flexural Strength (MPa)	Flexural Modulus (GPa)
SFG 1	55	0	45	90.7	4.8
SFG 2	55	0.5	44.5	92.3	5.0
SFG 3	55	1	44	94.7	5.3
SFG 4	55	1.5	43.5	98.6	5.7
SFG 5	55	2	43	95.4	5.4
			Average	94.3	5.2
			SD	2.71	0.31

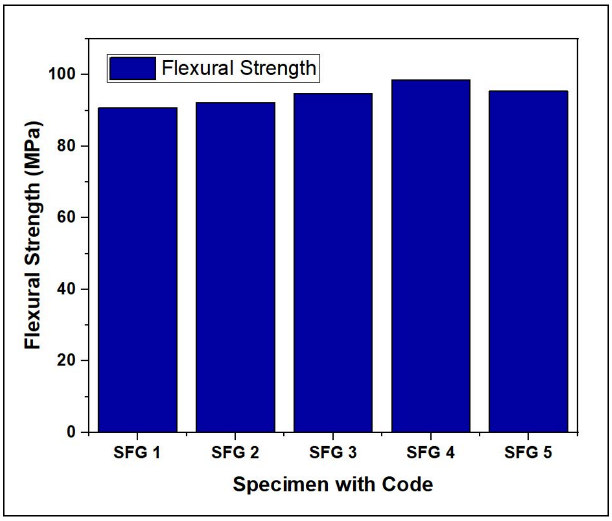

Fig. 4: Flexural strength of the specimens

The standard deviation for flexural strength and modulus at breakage are 2.71 MPa and 0.31 GPa, respectively. The uniformity of material characteristics among specimens is underscored by the variations noticed across distinct specimens, as illustrated by these figures. The combined reinforcing effects of hybrid fibers, silane treatment, and TiO_2 nanoparticles are likely responsible for the flexural properties of the composite material. The material's capacity to endure bending stresses is substantially enhanced by the additional reinforcement offered by TiO_2 nanoparticles, in addition to the inherent strength and rigidity of this fiber. Numerous nanoparticles may disrupt the uniform distribution of stresses and impair the bond between the matrix and fiber. The material's capacity to resist deformation forces may also be compromised by potential compatibility issues and increased interfacial stress. Furthermore, the composite flexural strength is improved by the effective stress transfer and layer separation that is prevented by the firm adherence between the fiber and the matrix. In conclusion, the hybrid composite material's efficacy for structural applications necessitating substantial resistance to bending stresses is emphasized by the FS test results. The mechanical properties of the composite can be significantly improved by further refining the chemical treatment and nanoparticle inclusion techniques.

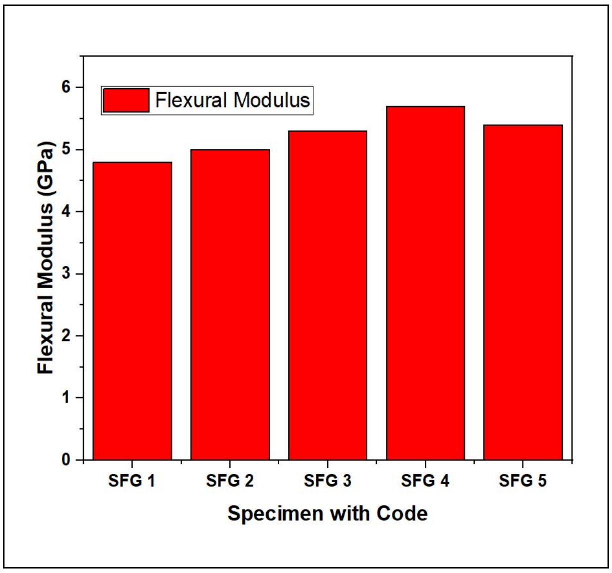

Fig. 5: Flexural modulus of the specimens

Table 3: Results of the Microhardness test

Sample	Fiber (%)	TiO_2 (%)	Epoxy (%)	Microhardness (HV)
SFG 1	55	0	45	55
SFG 2	55	0.5	44.5	57
SFG 3	55	1	44	59
SFG 4	55	1.5	43.5	62
SFG5	55	2	43	60
			Average	58.6
			SD	2.70

C. Microhardness (HV)

The microhardness of the specimens is shown in Fig. 6. The hybrid laminate material's average microhardness of 58.6 HV, with individual specimens ranging from 55 to 62 HV, as determined by the Microhardness test. The results of the Microhardness tests for the composite laminate, which incorporates sisal fiber, flax fiber, glass fiber, and TiO_2 nanoparticles, are summarized in Table 3.

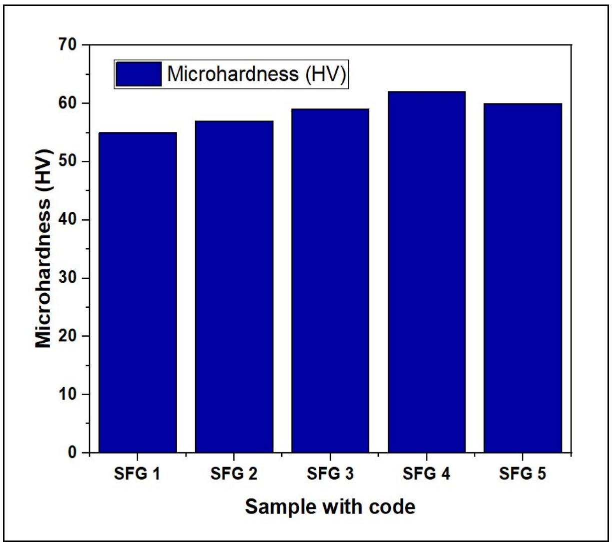

Fig. 6: Microhardness of the specimens

The standard deviation for microhardness is 2.70, indicating a high level of uniformity in the material properties among the specimens. The variations reflect the combined reinforcing effects of hybrid fiber, silane treatment, and TiO_2 nanoparticles on the composite's hardness characteristics. The increase in microhardness in composites can be significantly attributed to the effective adhesion of nanoparticles to the matrix. When nanoparticles, such as TiO_2, are well-dispersed and adhere strongly to the matrix, they serve as reinforcement, enhancing the composite's resistance to deformation. This strong adhesion allows for efficient stress transfer between the matrix and nanoparticles, which helps prevent localized failure and contributes to an overall increase in microhardness.

Additionally, the strong adhesion between the fiber and matrix promotes effective stress transfer and prevents layer separation, further contributing to the improvement in microhardness. These mechanisms have efficient stress transfer, uniform stress distribution, and restricted dislocation movement resulting in significantly enhanced microhardness in the composite material. Refining the chemical treatment process and optimizing nanoparticle inclusion could further enhance the composite's mechanical properties.

D. Fracture Toughness

The hybrid laminate material demonstrated an average fracture toughness of 1.66 MPa·m^0.5, with values for individual specimens varying between 1.51 and 1.82 MPa·m^0.5, as measured in the fracture toughness test. The results of the fracture toughness tests for the composite laminate, which incorporates sisal fiber, flax fiber, glass fiber, and TiO_2 nanoparticles, are summarized in Table 4.

Table 4: Results of the Fracture Toughness test

Sample	Fiber (%)	TiO_2 (%)	Epoxy (%)	Fracture toughness (MPa $m^{0.5}$)
SFG 1	55	0	45	1.51
SFG 2	55	0.5	44.5	1.58
SFG 3	55	1	44	1.67
SFG 4	55	1.5	43.5	1.82
SFG5	55	2	43	1.75
			Average	1.66
			SD	0.125

The standard deviation for the fracture toughness is 0.125, indicating a high level of uniformity in the material properties among the specimens. These variations reflect the combined reinforcing effects of hybrid fiber, silane treatment, and TiO_2 nanoparticles on the composite's fracture toughness properties.

4. CONCLUSION

The development and assessment of the hybrid composite material, incorporating sisal, flax, and glass fibers, along with titanium dioxide nanoparticles, with silane treatment, yielded promising results across various mechanical tests, including tensile, and flexural evaluations. The material demonstrated a mean tensile strength of 77.16 MPa, indicating its capacity to withstand tensile stresses. The mean value of flexural strength was found to be 93.4 MPa, confirming its resistance to bending forces. Microhardness testing showed a mean value of 2.70 HV, confirming its resistance to microhardness. The mean fracture toughness of 1.66 MPa $m^{0.5}$ with an SD of 0.125 MPa $m^{0.5}$ confirmed its improvement. The tensile, flexural strength, microhardness, and fracture toughness increased by 8, 9, 12, and 20% respectively on sample SFG 4, compared to the composite without nanoparticles. These findings underscore the potential of hybrid composite materials for diverse technical applications that demand high levels of stiffness, and toughness. The reinforcement provided by TiO_2 nanoparticles, and hybrid fiber enhances the material's mechanical properties, offering a balanced combination of sustainability and efficiency. Future advancements in composite processing and formulation methods are expected to further improve these mechanical properties to meet specific engineering needs.

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CONFLICT OF INTEREST

The authors declared no conflict of interest in this manuscript regarding publication.

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