

Study on the Effect of Surface Modification on the Mechanical and Thermal Behaviour of Flax, Sisal and Glass Fiber-Reinforced Epoxy Hybrid Composites

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Abstract: Natural fiber-reinforced hybrid composites can be a better replacement for plastic composites since these plastic composites pose a serious threat to the environment. The aim of this study is to analyze the effect of surface modification of the natural fibers on the mechanical, thermal, hygrothermal, and water absorption behaviors of flax, sisal, and glass fiber-reinforced epoxy hybrid composites. The mechanical properties of alkaline treated sisal and flax fibers were found to increase considerably. Tensile, flexural and impact strength of glass/flax-fiber-reinforced hybrid samples improved by 58%, 36%, and 51%, respectively, after surface alkaline treatment. In addition, the hygrothermal analysis and water absorption capacity are studied and also the Interfacial bonding properties were analyzed using Scanning Electron Microscopic images. The thermal analysis using thermogravimetric analyzer reveals that the decomposition temperature for hybrid fiber reinforced composites are between 306 and 312°C. In conclusion, surface treatment improves the performance of natural fiber in hybrid fiber-reinforced composites, particularly flax fiber.

Keywords: Hybrid composite; surface treatments; mechanical testing; thermal analysis; hygrothermal testing; water absorption; SEM analysis

Nomenclature

GFG-T/UT-Glass/Flax/Glass Fiber-reinforced Composites-Treated Flax/Untreated Flax.

GSG-T/UT-Glass/Sisal/Glass Fiber-reinforced Composites-Treated Sisal/Untreated Sisal.

SGF-T/UT-Sisal/Glass /Flax Fiber Reinforce Composites-Treated/Untreated Sisal and Flax Fiber.

1 Introduction

Recently researchers are showing increased interest in the use of biodegradable materials in the structural applications [1,2]. Natural fiber-reinforced composites are analyzed for its applicability in structural and engineering field. Asia is one of the largest resources of cellulose fibers such as bamboo, hemp, flax, sisal, and banana. The researchers are showing interest in reinforcing cellulose fiber in plastics. The advantages of these reinforcing-fibers are their low density, an important factor of consideration in the automobile and aerospace industry. [3] Proved that the banana/sisal hybrid fiber-reinforced polyester composites result in a positive effect on tensile and flexural properties. [4] Found that flax is a suitable structural replacement for E-glass for using in small wind turbine blade applications.[5] Identified the plant fiber as a good replacement for synthetic fiber in terms of cost, density, renewability, and CO₂ emission. [6] Found that the banana and jute hybrid composites are showing better mechanical properties compared to monocomposites. [7] Showed that the sisal/Glass fiber composite has better tensile strength. [8] Found that the fiber surface treatment will improve the roughness and increase the adhesion between fiber and matrix.

According to [9] mercerization using 10% NaOH will improve the fiber-matrix interfacial adhesion. Research in [10] showed increases in the mechanical properties of composites like tensile, flexural, and impact strength as a result of surface modification of coir fiber. [11] Found out decrease in the percentage of hemicelluloses, lignin, and increase in cellulose content, major portion of natural fiber which provides strength and influences the mechanical properties after alkali treatment. [12] showed that the Alkalization process breakdowns the fiber bundles into smaller fibers, which in turn increased the effective surface area available for wetting by the matrix. [13] Found that jute/glass fiber composite is having the good tensile strength and jute/sisal/glass fiber is having good flexural strength. [14] Discussed the less impact on the environment on disposal after end life of the natural fiber-reinforced composites. Apart from material characterization, there are researches going on regarding the manufacturability of composites. [15] Studied the drilling of CFRP composites using Fuzzy logic [16] has proved the possibility of natural fibers in gears.

In this study, six types of hybrid composite laminates are fabricated using epoxy resin with flax, sisal, and glass fibers in various combinations. In the six laminates, three were with surface-treated natural fibers and the other three with untreated natural fibers as reinforcement. The treated and untreated fiber-reinforced laminates are compared for their tensile, flexural, and impact strength and interfacial adhesion with the matrix using Scanning Electron Microscopy (SEM). Thermal and Hygrothermal properties are also studied.

2 Experimentation

2.1 Materials

Epoxy resin (LY 556) with the hardener (HY-951) in the ratio of 10:1 is used for matrix preparation. The natural fibers used are biaxial form of flax and sisal fibers brought from Weavers Association, Chennai. The glass fiber used is 600 GSM biaxial mats from Sakthi fibers, Chennai. The properties of the fibers are given in Tab. 1.

Table 1: Properties of fibers (As per supplier given information and same verified in [17])

S.No	Material	Density g/cm ³	Tensile Strength MPa	Elastic Modulus GPa
1.	Glass fiber	2.54	3450	72
2.	Flax fiber	1.45	800 to 1500	60 to 80
3.	Sisal fiber	1.45	468-700	22

2.2 Alkali Treatment

The mechanical strength of the composites depends on the interfacial bonding between the fiber and the matrix. The poor wetting of the fiber in the resin will result in lower strength of the composites [18]. According to [9], to improve the bonding between the fiber and the matrix, the surface of the fiber should undergo treatments such as mercerization, esterification, and ionized air treatment. In this work, mercerization, a chemical process to enhance the roughness of the fiber surface, is carried out on the flax and the sisal fiber to improve the adhesion. Actually, all the natural fibers are lignin-cellulose based fibers; the presence of hemicelluloses material on the outer surface prevents the proper bonding between matrix and the fiber [19]. It is identified that mercerization will remove the wax, pectin, lignin, and hemicelluloses on the fiber's surface and will enhance proper bonding [20]. The fiber surface is treated as follows [21]:

1. Flax and sisal fiber mat were washed with distilled water and dried.
2. Then the dried fiber mats were treated with 10% NaOH solution for 1 hr at room temperature in a separate tray.
3. The treated fibers were washed with distilled water again to remove excess NaOH adhered to the fiber mat
4. The washed mats were sun-dried for 8 hr and were then oven dried at 50°C for 2hr (Fig. 1).



Figure 1: NaOH treatment of flax and sisal fiber

2.3 Fabrication of Composites

The laminates are prepared by layering-up fibers in a thin cleaned mold coated with mold-releasing agents; the size of the mold is 300×300 mm. Initially, the glass, sisal, and flax fiber mats are cut into 300×300 mm size. Epoxy and hardener are mixed in the ratio of 10:1. Different types of laminates are prepared with the combinations of glass-flax-glass (GFG), glass-sisal-glass (GSG), and sisal-glass-flax (SGF). In each combination, four laminates are prepared, two with alkali-treated natural fiber mats and two with untreated fiber mats. The process has been done carefully to produce a uniform and homogeneous composite laminates of $300 \times 300 \times 3$ mm, and air entrapment is avoided by allowing the resin to flow over the fiber mat completely. The 3 mm thickness is achieved by layering up alternate layers of resin and fiber mat in the above-mentioned order. The fabricated specimens are shown in Fig. 2, and the Tab. 2 shows the types of laminates and their configuration.

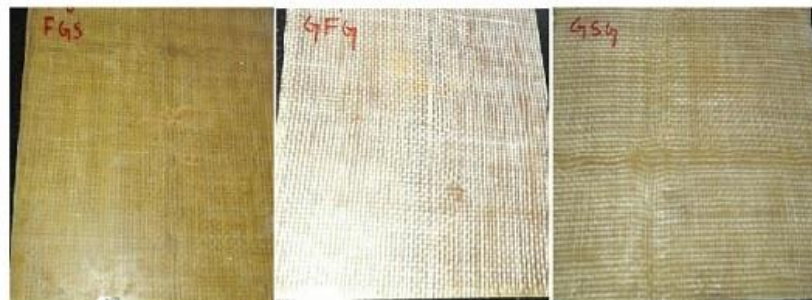


Figure 2: Sample Laminates of different type of Composite

Table 2: Types of Composites and their Configuration

S.No	Composite Name		Composite Type	Layer 1	Layer 2	Layer 3
	Treated	Untreated				
1	GFG-T	GFG-UT	Glass and Flax Fiber-reinforced Epoxy	Glass Fiber Mat	Flax Fiber Mat	Glass Fiber Mat
2.	GSG-T	GSG-UT	Glass and Sisal Fiber-reinforced Epoxy	Glass Fiber Mat	Sisal Fiber Mat	Glass Fiber Mat
3.	SGF-T	SGF-UT	Sisal- Glass -Flax Fiber-reinforced Epoxy	Sisal Fiber Mat	Glass Fiber Mat	Flax Fiber Mat

2.4 Characterization

2.4.1 Mechanical Testing

The tensile test is carried out as per ASTM D-638 standards on a Universal testing machine (UTM) UTN-60 with minimum graduation 1KN and maximum capacity of 600 KN at room temperature. The machine setup and the samples before and after the test are presented in Figs. 3 and 4, respectively. The flexural test is carried out in the UTM on the samples prepared as per ASTM standards D-790. The testing procedure is as per the three-point bending test method; the machine setup and the samples before and after the test are represented in Figs. 5 and 6, respectively. Impact energy is the energy that the specimen absorbs once a sudden load is applied. Charpy impact testing is one of the ASTM standard methods of determining the impact resistance. Material specimens are prepared as per ASTM D-4812 standard. Samples before and after the test are shown in Fig. 7. The test results presented here are average of five specimens.

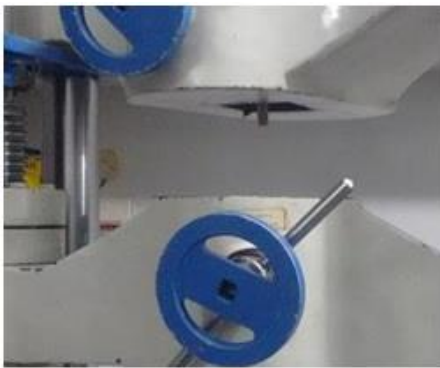


Figure 3: Tensile Test setup



Figure 4: Tensile Test specimens



Figure 5: Flexural Test setup



Figure 6: Flexural Test specimens



Figure 7: Impact test specimens

2.4.2 Thermal Analysis: Thermogravimetric Analysis

The thermal analysis is mainly carried out to study the effect of temperature on the composite samples. Thermogravimetric analysis is one of the important tests to understand the thermal stability [22] of composite materials, and it is conducted here to identify the effect of surface modification on thermal behavior of natural fiber polymeric composites. Thermogravimetric analysis is carried out on Inkart TG DTA analyzer at the heating rate of $10^{\circ}\text{C min}^{-1}$ from 30°C to 800°C under nitrogen environment. 10 mg sample of each type is placed on the pan to identify the decomposition temperature and the residual mass and plotted on a graph. The thermogravimetric analyzer is shown in Fig. 8.



Figure 8: Thermogravimetric analyzer

2.4.3 Hygrothermal Analysis

According to [23], many properties such as physicochemical, thermal, and mechanical properties could be affected by Hygrothermal aging. This phenomenon has to be studied carefully before recommending PMC to such environmental application, and to understand the changes in material property long-term study is recommended; here the same is done using short-term experimental investigation with accelerated experimental test conditions.

Hygrothermal testing is carried out on a tensile test samples; the temperature is maintained at 45°C , average high temperature in India, and a humidity of 65% is maintained, and the test is carried out for 72 hrs; the TDS value of the water used is 1500. The test specimens of all the type of laminates are weighed carefully before placing in the chamber as shown in Fig. 9. After exposing the samples to hygrothermal environment for 72 hrs, the samples are taken and wiped out for removal of excess water and sundried.

Then the dried samples are weighed and subjected to tensile testing on the UTM to calculate average tensile strength value for all the types.



Figure 9: Tensile Test specimens in Hygrothermal setup

2.4.4 Water Absorption Test

According to Z.N. Azwa et al [24], the water absorption of biocomposites is typically 0.7% to 2 % after 24 hrs and 1% to 15% after a week, which will lead to failure in the wet environment. Hemicellulose is the fiber component responsible for moisture absorption in natural fiber. The water absorption test is carried out on both treated and untreated sample to study the effect of surface treatment on the hemicelluloses and to absorb the reduction in water absorption due to surface treatment. The water absorption test is one of the important tests concerning natural fiber-reinforced composite as the natural fiber tends to absorb the moisture, which has to be carefully noted when it is going to be used in structures which will be exposed to moisture. The sample was cut into a flat shape (30 × 30 × 3) mm. They were oven dried and specimens were weighted accurately using the weighing balance of ± 0.1 mg accuracy.

Then the specimens are immersed in distilled water, and weights measured after 24 hours, 48 hours, and 72 hours by taking out the specimen and drying it. Using the two readings, the water absorption percentage was calculated as per the formula:

$$\frac{W_1 - W_0}{W_0} * 100 \text{ [25,26,27]}$$

where, w_1 is the weight of the sample after taking it out of distilled water, and w_0 is the initial weight of the sample.

2.4.5 SEM Analysis

The images of the tensile-fractured surfaces of the samples are captured using Scanning electron microscope (SEM) Vega 3 TESCAN. The intended pieces of surfaces from the samples are being coated with a conducting material; the SEM images are obtained.

3 Results and Discussion

3.1 Mechanical Properties

3.1.1 Tensile Test

The tensile test is performed in the UTM. The tensile strengths of all the composite samples are found, and also the yield, ultimate, and the breaking stresses are also obtained. The sample load versus displacement graphs obtained from the machine, and the average value of Tensile strength of each type of composites are given in Tab. 3. The result shows that the tensile strength of all the three combinations (SGF, GSG, and GFG) with alkali-treated natural fibers are greater than that of the untreated fiber-reinforced laminates, and the comparison is shown in Fig. 10. From the graph, it is understood that in all the three

combinations, treated fiber-composites outperformed the untreated fiber composites. Among the six types, the alkali-treated GFG combination's tensile strength improved by 58%.

The theoretical value of tensile strength of all the composites are calculated using the formula, $\sigma_t = P/bh$, [28]

where, P is the ultimate load of the specimen, the b is the initial width of the specimen, and h is the initial thickness of the specimen.

The values obtained for different composites are $\sigma = 50 \text{ N/mm}^2$ for GFG untreated, 60 N/mm^2 for GSG untreated, 94 N/mm^2 for GFG treated, 71 N/mm^2 for GSG treated, 33 N/mm^2 for SGF untreated, and 45 N/mm^2 for SGF treated respectively. The values are found matching with the experimental values.

Table 3: Overall mechanical performance of different composite laminate

S.No	Composite type	Tensile strength (N/mm ²)	Flexural strength (N/mm ²)	Impact Energy (J/m)
1	GSG-untreated	62.4	188.95	17
2	GFG-untreated	52.978	155.334	13.4
3	SGF -untreated	30.274	95.884	13
4	GSG-treated	69.141	187.99	18
5	GFG-treated	95.9062	224.48	22
6	SGF -treated	46.3046	198.48	20

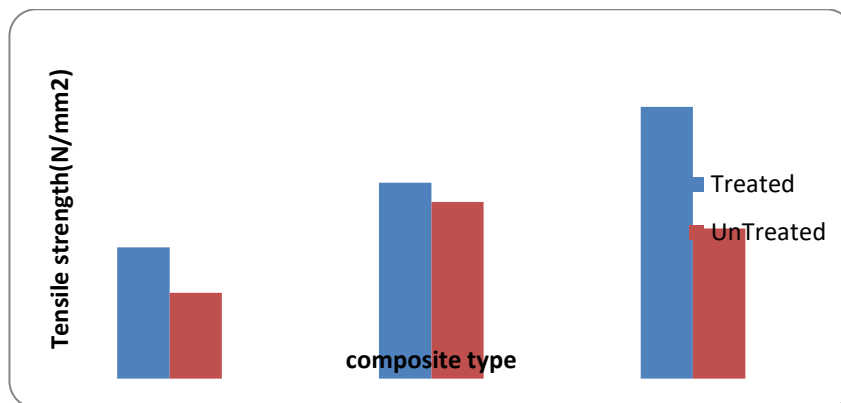


Figure 10: Comparison of tensile strength

3.1.2 Flexural Test

Fig. 11 shows the flexural test results of all the treated and untreated composites and their corresponding values are given in Tab. 3. From the graph, we could understand that the flexural strengths of all the alkali-treated samples are greater than those of the untreated natural fiber samples. From these values, it is understood that the flexural strengths of all the natural fiber composites are very high, and the alkali treatment increases the flexural strength of the composites. Especially in the case of flax fiber, there is a tremendous increase by 36%, and it is also noted that the flexural strengths of both the treated and untreated sisal composites are in the same range of 187-188 N/mm².

The theoretical values of flexural strengths of all the composites are obtained using the formula:

$$\sigma_f = 3PL/2bh^2 [28,29]$$

where P is the ultimate load of the specimen, L is the span length of the specimen, b is the width of the specimen, and h is the thickness of specimen.

The value obtained for different composites are $\sigma_f = 155 \text{ N/mm}^2$ for GFG untreated, 189 N/mm^2 for GSG untreated, 222 N/mm^2 for GFG treated, 186 N/mm^2 for GSG treated, 94 N/mm^2 for SGF untreated, and 199 N/mm^2 for SGF treated respectively. The experimental and theoretical values match with each other.

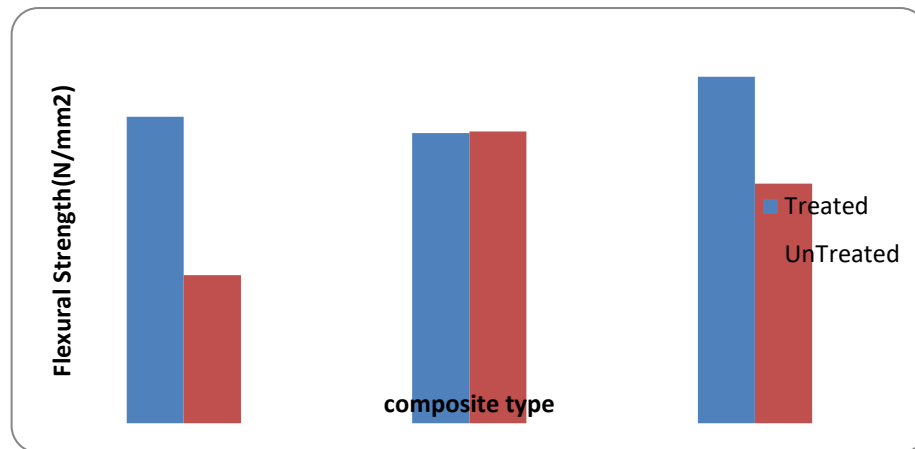


Figure 11: Comparison of flexural strength

3.1.3 Impact Test

The impact test is carried out to find the impact strengths of the composite specimens prepared. The patterns of failure observed in impact testing are fiber breakage and delamination. The impact resistances of all the treated natural fiber-reinforced composites are greater than those of the untreated natural fiber composites as shown in Fig. 12. The impact resistance values of all the treated fiber composites are in the range of 18- 22 J/m and those of untreated samples are in the range of 13 J/m to 17 J/m, respectively. Also, the impact resistances of the treated flax fiber composites outperform all the other types with an improvement of 51%. Average values of the impact strengths for all the types of composites are given in Tab. 3.

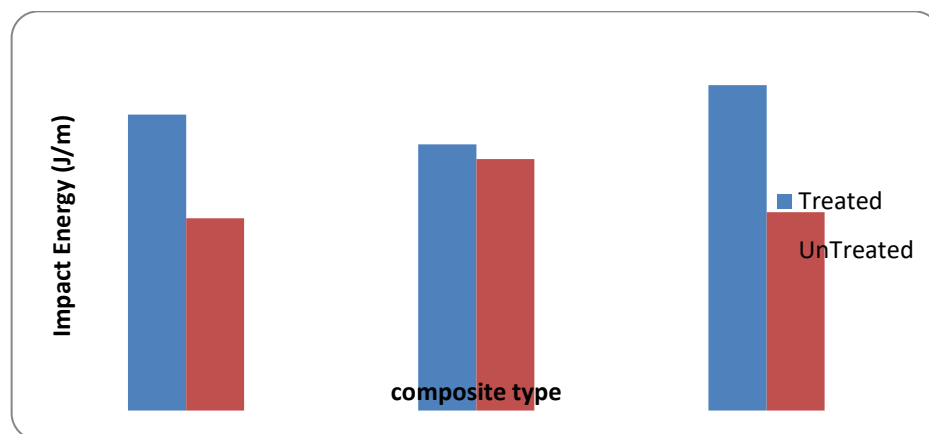


Figure 12: Comparison of impact strength

3.2 Thermal Properties

3.2.1 Thermogravimetric Analysis

The sample TG curves of the treated and untreated flax/glass fiber composites are shown in Fig. 13. The composite shows a weight loss with the application of temperature, due to the thermal degradation of the cellulose and resin, and the curve shows that the decomposition starts after 300°C. The same pattern is followed in all the types of samples tested which points out that the thermal stability of hybrid composite with natural fiber is good till 300°C and, irrespective of surface treatment the samples, decomposes in the range of 306-315°C. The decomposition temperature and the residual mass percentage for each type of sample are given in Tab. 4, where the amounts of UV rays absorbed are also listed.

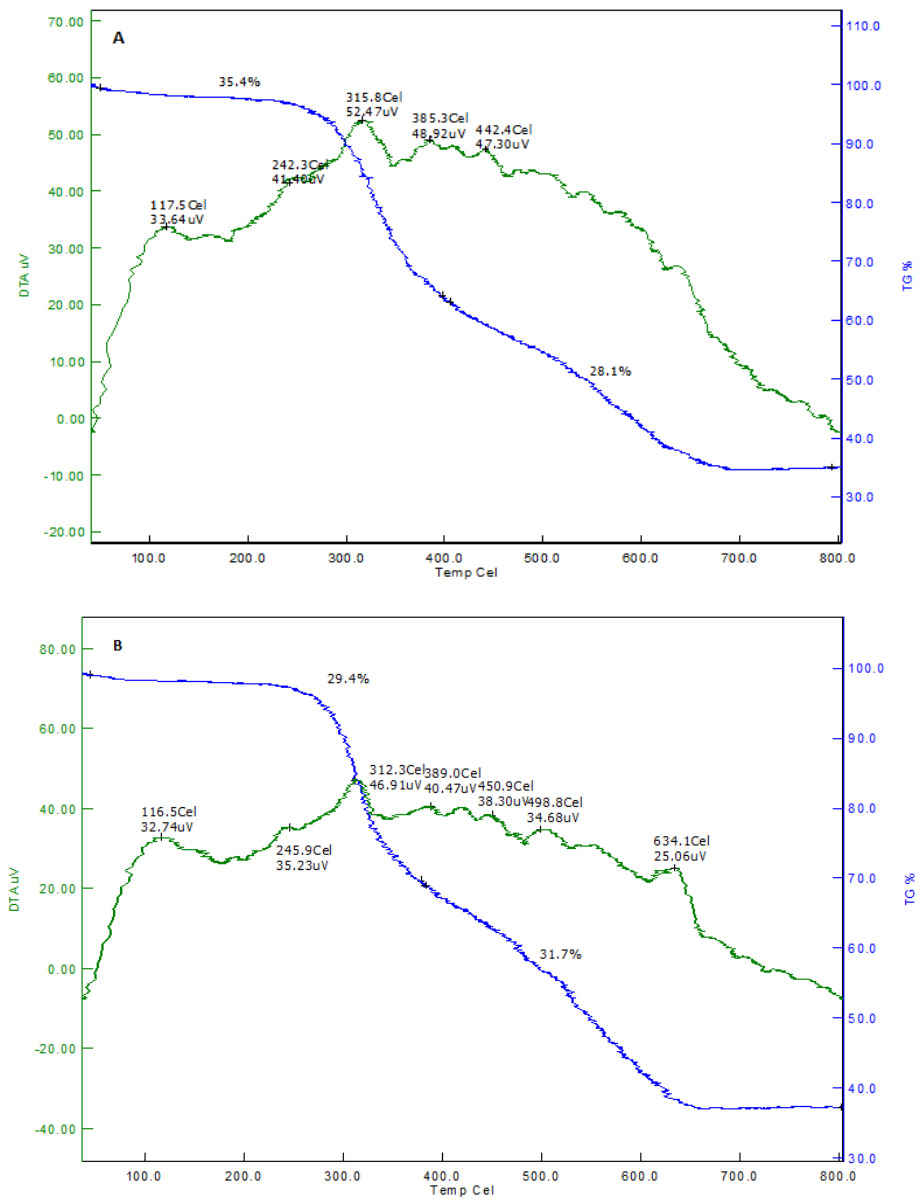


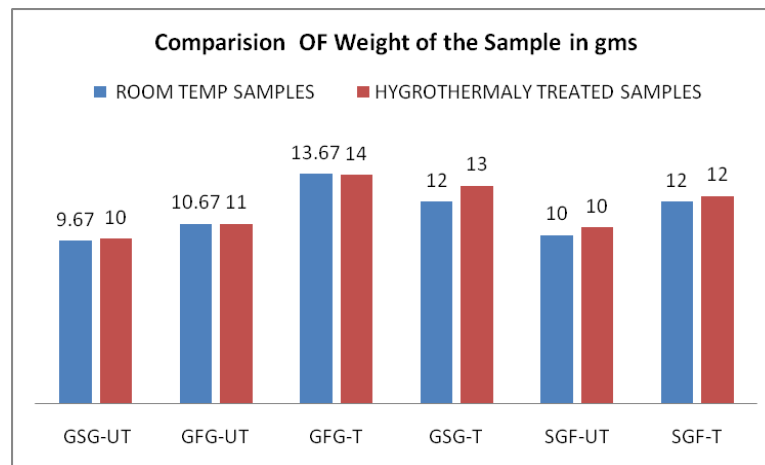
Figure 13: TG graph of flax (A) GFG-UT and (B) GFG-T samples

Table 4: Decomposition temperatures of different types of samples

S.No.	Composite Type	Decomposition Temp ^o C	Percentage of Mass Left Out	Dta uV
1	GSG-UT	312.6	37	62
2	GFG-UT	315.8	35	52
3	GFG-T	312.3	38	47
4	GSG-T	316.8	24	51
5	SGF-UT	312.8	64	58
6	SGF-T	306.7	28	58

3.2.2 Hygrothermal Analysis

During this analysis, the temperature is kept above the actual average service temperature in order to accelerate the moisture diffusion and degradation process. The hygrothermal aging by water absorption in PMCs will damage the bonding between the fiber and the matrix and results in debonding. Fig. 14 shows the comparison of the weights of the sample before and after exposing it to the hygrothermal environment, and the maximum weight increase is 1gm in sisal/glass-treated sample. The samples of SGF treated and untreated have not shown any variation in weight, and the samples of GFG treated and untreated show an increase in weight of less than 0.5 gm. The weight increase is due to the water absorption. Fig. 15 shows the comparison of tensile strengths before and after hygrothermal exposure irrespective of surface treatment. All the types of the sample show deterioration in tensile strength, and the percentage of deterioration falls in the range of 1-12 N/mm².

**Figure 14:** Comparison of Weight of Room Temperature and Hygrothermal sample

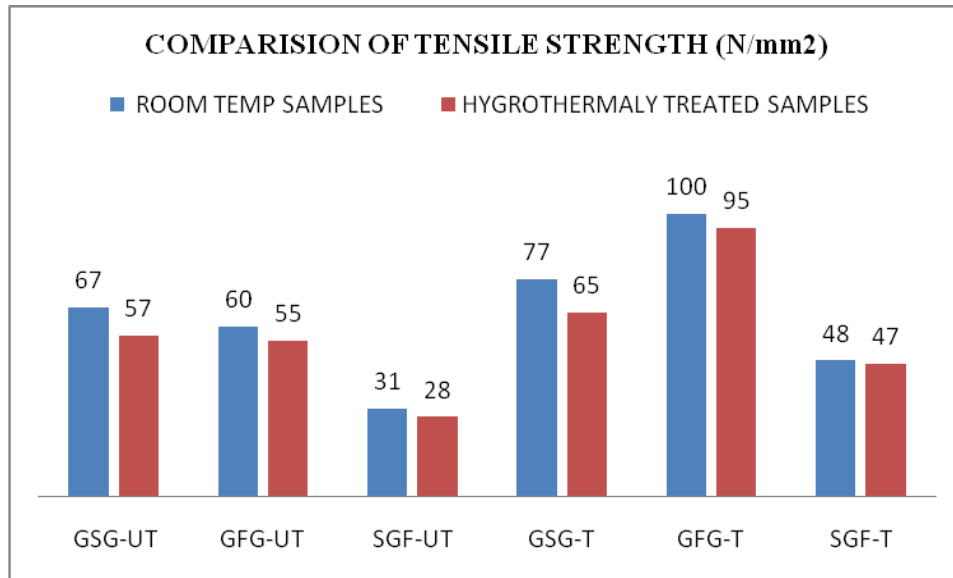


Figure 15: Comparison of tensile strength

3.3 Water Absorption Behavior

The results for the water absorption test for the different hybrid composites when immersed in distilled water at room temperature are shown in Fig. 16, which show the absorption curves for the combinations, the moisture uptake is increasing linearly. Tab. 5 shows the values of water absorptions in percentage for different samples which show that the water absorption percentage of treated samples is less than that of the untreated natural fiber-reinforced samples. Also, surface treatment has very much improved the water retardation behavior of flax fiber, and the percentage of absorption considerably reduced in these samples than in other samples after treatment. Moreover, sisal is having less water-absorbing nature with and without treatment. From the result, we can understand that when the proportion of natural fiber increased, the water absorbing-capacity also increases irrespective of surface treatment as both SGF treated and untreated have more values compared to other types.

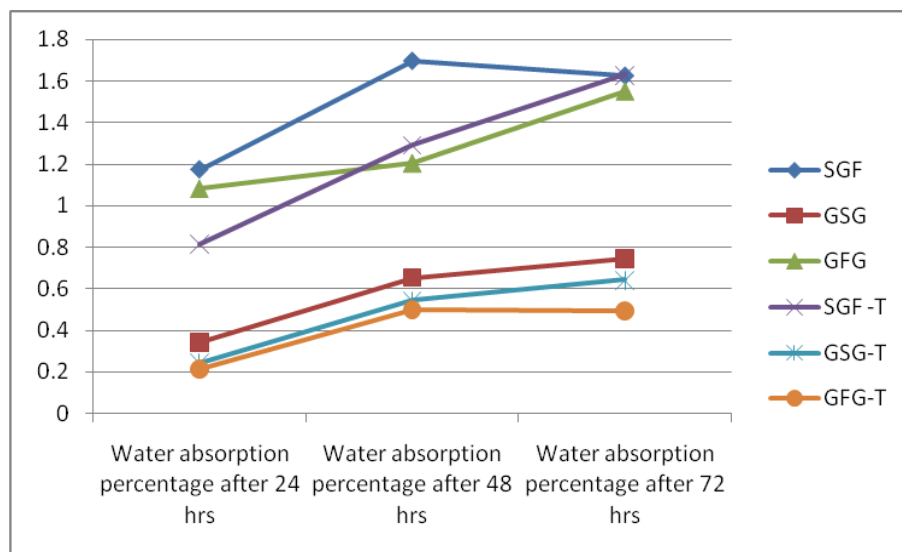


Figure 16: Comparison of water absorption behaviors

Table 5: Water absorption percentage of different types of sample

S.No.	Composite type	Water absorption percentage after 24 hrs	Water absorption percentage after 48 hrs	Water absorption percentage after 72 hrs
1	SGF-UT	1.1724	1.6946	1.6232
2	GSG-UT	0.342	0.6532	0.7434
3	GFG-UT	1.0834	1.2058	1.5526
4	SGF -T	0.8134	1.2918	1.6296
5	GSG-T	0.242	0.5432	0.6424
6	GFG-T	0.2124	0.501	0.4934

3.4 Morphological Analysis

The images of the tensile fractured surfaces of the samples are taken using Scanning electron microscope (SEM). The intended pieces of surfaces from the samples are being coated with a conducting material; the SEM images obtained are presented in Fig. 17 to Fig. 22. SEM images can be used to identify the interfacial bonding adhesion [30], with which the impact of alkali treatment on the fiber-matrix bonding is studied in this section.

Fig. 17 and Fig. 18 show the fractured surfaces of the untreated and treated GFG tensile-tested samples, respectively; a fiber pullout is observed on the fractured surface of the untreated sample whereas the treated sample is not showing such behavior. Fig. 19 and Fig. 20 show the fractured surfaces of the untreated and treated GSG tensile tested samples respectively. Fig. 19 shows voids and the incomplete distribution of resin leading to poor interfacial bonding between the resin and untreated fibers, whereas Fig. 20 shows a good interfacial bonding between the treated fiber and the resin.

Fig. 21 and Fig. 22 show the fractured surfaces of the untreated and treated SGF tensile-tested samples, respectively. From Fig. 22 it is observed that there is a uniformity on the fractured surface, whereas in Fig. 21 there is observed an improper bonding between the fiber and the resin. From all these images, it is found that alkali treatment has improved the bonding between resin and fiber, avoiding problems like voids, fiber pullout, and improper filling of resin which will weaken the strength of the composite. Therefore, it is seen that the surface treatment like mercerization (Alkali Treatment) will improve the interfacial bonding strength.

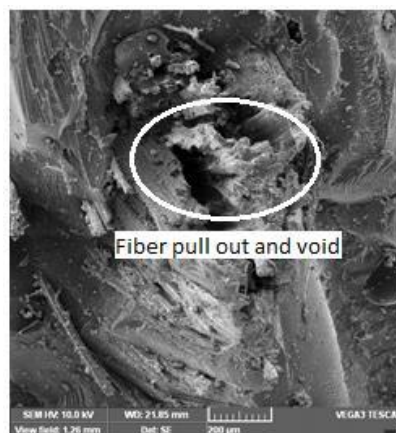


Figure 17: SEM image of the Tensile Fracture surface of GFG-untreated

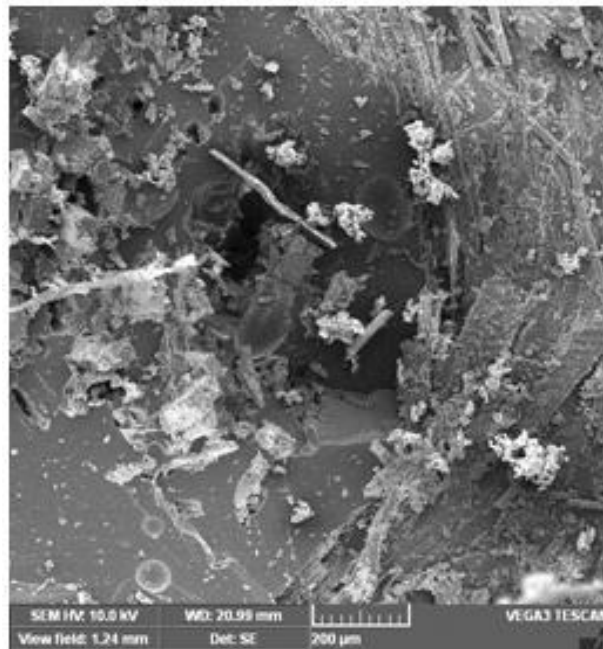


Figure 18: SEM image of Tensile Fracture surface of GFG-treated

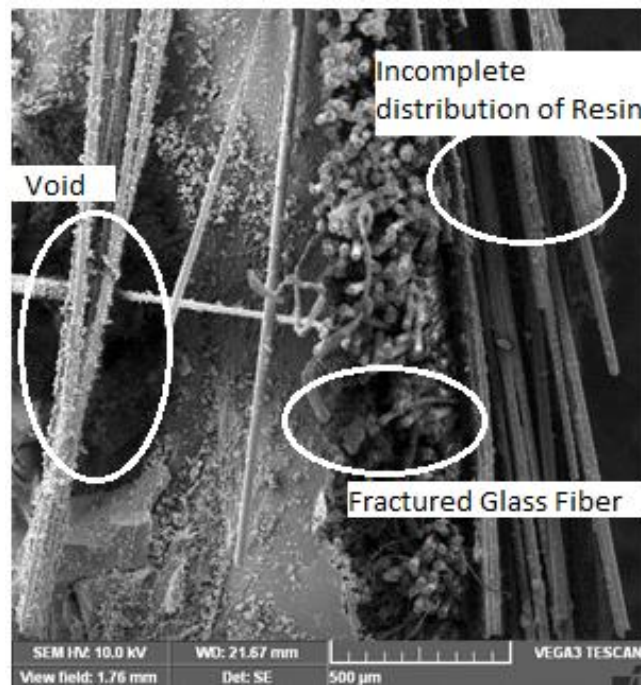


Figure 19: SEM image of the Tensile Fracture surface of GSG-Untreated

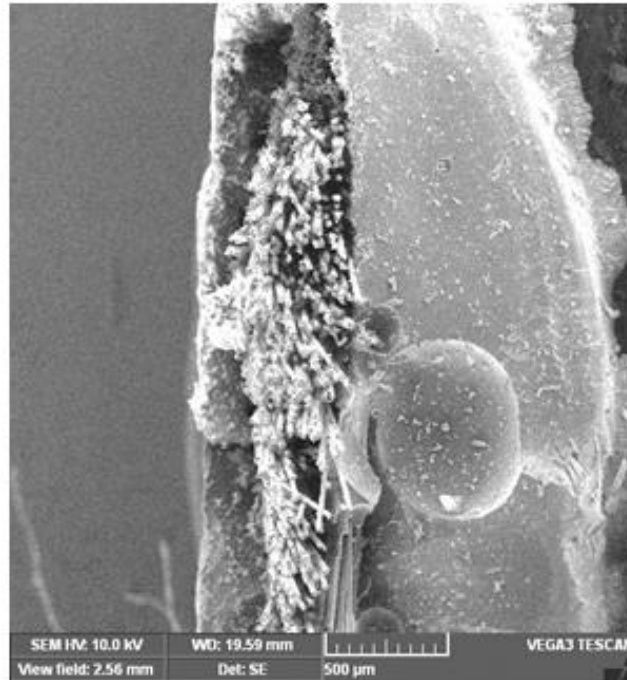


Figure 20: SEM image of the Tensile Fracture surface of GSG-Treated

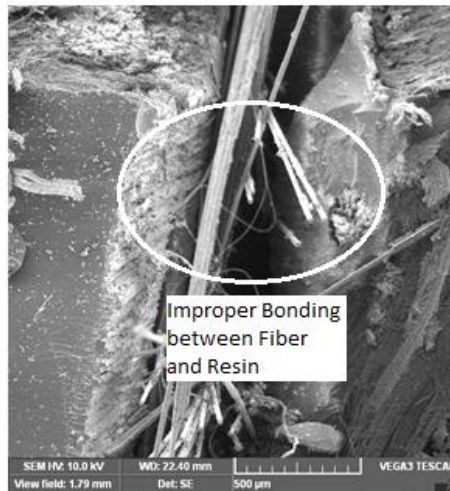


Figure 21: SEM image of the Tensile Fracture surface of SGF-Untreated

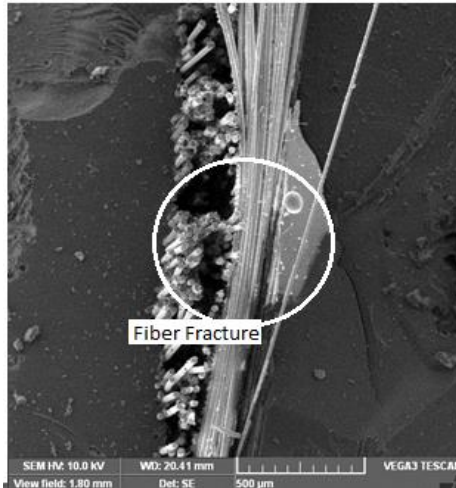


Figure22: SEM image of Tensile Fracture surface of SGF-Treated

4 Conclusion

This research work was carried out to compare the mechanical, thermal, hygrothermal, and water-absorbing behavior, and the bonding strengths of the treated and untreated glass and natural fiber-reinforced hybrid epoxy composites and the following conclusions are made:

- Flax fiber is showing a very good improvement of 30-60% in mechanical strength after alkali treatment.
- The tensile and impact strengths of the composite laminates are improved greatly by means of surface-treated natural fibers.
- The thermogravimetric analysis shows that the decomposition temperatures of hybrid composite laminates fall in the range of 306-315°C irrespective of its surface treatment.
- The hygrothermal analysis comparison of the weights of samples before and after test shows that there is not much variation in weights, and the deterioration in tensile strength is little greater in the sisal hybrid fiber composite by 12 N/mm², whereas in the other samples, it is less in the range of 1-5 N/mm².
- The water absorption phenomenon of hybrid composites shows that treated flax hybrid composite is absorbing less percentage of water compared to the other types.
- The SEM images show that surface treatment like mercerization/alkaline treatment improves the interfacial bonding strength between the fiber and the resin.

Overall, the surface treatment improves the performance of natural fiber in the hybrid fiber-reinforced composites. Particularly, flax fiber shows a very positive improvement.

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