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Mechanical and thermal properties of a novel Spinifex Littoreus fiber reinforced polymer composites as an alternate for synthetic glass fiber composites

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Abstract

The usage of natural fiber reinforced composite is large in engineering applications due to the presence of high extensive properties and economy. This paper aims at presenting a new fiber, its characteristics, and composite in a polymeric matrix empowering the generation of less weight composites for load conveying applications. Spinifex Littoreus Fiber (SLF) is a genus of perennial coastal plants in the grass family. The comprehensive characterization that includes physical analysis, chemical analysis, thermal analysis, mechanical and microstructural characteristics have been carried out on the fiber. The effect of fiber weight percentage was studied from the point of view of the mechanical properties of the compression moulded Spinifex Littoreus Fiber Composites (SLFC). Mechanical properties attained at 40 wt% of fiber content were better. In addition to more fiber, it tends to cause inadequate bonding among the matrix and fiber, resulting in a decrease in mechanical performance. The specific properties of SLFC polymer composites are comparable to those of the glass fiber composite. This makes SLFC composite as an alternate lightweight material. SEM was performed for a study of the interfacial mechanism.

1. Introduction

Minimal cost and better mechanical properties facilitate extensive usage of glass fiber reinforced polyester composites. The utilization of green fiber reinforced polymer composites is gradually increasing in various countries during the last few years due to environmental issues. This will make a positive attitude among industries for the approach of green fiber composites due to their biodegradable features and economy [1]. An investigation reports that cancer is caused by the use of man-made vitreous fibers [2]. However, the features of man-made glass fibers are the same as those of asbestos fibers and cause lung cancer, in which the glass fiber affects the respiratory system [3]. Increasing concern for the environment increases the need for biodegradable materials with polymeric compounds filled with natural organic fillers, from renewable and biodegradable sources instead of using synthetic fibers. Researchers are finding better alternatives to the man-made vitreous fibers, primarily, the glass fiber [4, 5].

In recent years, the increasing numbers of reviews among the publications on green fiber composites consider the importance of growing new bio composites [6]. Due to the use of renewable plant resources for products, this century is being called as cellulosic century [7]. Innovative improvements associated with expectations and consumer demands keep on expanding the interest for worldwide resources, prompting serious issues of material accessibility and environmental sustainability [8]. Instead of using synthetic fibers, the researchers are finding out better alternatives to the man-made vitreous fibers especially the glass fiber. The
production, use and evaluation of conventional composite structures over manmade fibers such as carbon and glass are known for minimal cost, reduced health hazards, adequate specific properties, simplicity of division, low density and improved recovery of vitality, CO2 sequestration and biodegradability [9–11]. Without compromising on mechanical properties and being cost effective, some plants are yielding better natural fibers [12]. The fiber extracted from plants, for example, banana, kenaf, barley, paper-mulberry, oats, jute, pennywort, coir, grass reeds, rice husks, wheat, rye, ramie, sisal and papyrus, etc are used as reliable reinforcement's stronghold in composites [13, 14]. In engineering industries, natural fibers as reinforcements for composite find use due to eco-friendly mechanical properties. In the beginning of the 20th century, modern applications included sheets, aero plane seats, fuel-tanks, tubes, and pipes which were manufactured utilizing of natural fibers as reinforcement. The demand was not extremely high mechanical resistance but, low maintenance and purchasing cost [15, 16]. However, identifying new plant fibers and creating composite and using them are inevitable in the field of research, particularly for environmentally friendly commodities. The newly identified SLF is extracted from the Spinifex Littoreus grass using simple hand-operated and biodegradable methods [17]. These grass species are commonly found on Arid Middle East, Africa, and Australia and also in India in the Kanyakumari district, Tamilnadu. This paper reveals the Physico-chemical and mechanical properties of SLFC for the first time. Mechanical properties were investigated in composites with various fiber percentages 10, 20, 30, 40 and 50, and these optimized. The morphological structure of the failure mode in the fiber composite has been examined using an SEM. The Thermal examination has been performed at maximum temperatures, for the determination of the thermal stability of natural SLF and SLFC.

2. Composite preparation and characterization

2.1. Fiber extraction

Spinifex Littoreus plant stems were collected from the Kanyakumari district in Tamilnadu state of India. Using the stamping process, the lacerated stems were crushed and the flush of the fiber was removed with a hammering force of 50–80 N. They were cleaned by immersing in water for about 24 h. They were then washed until the removal of the dirt particles. The moisture content of fiber was removed by placing it in a heating coil oven at 100 °C as recommended in the literature [18]. The moisture removed fibers were then cut to size according to standard dimensions. The extracted fibers were shown in figure 1. The Physico-chemical analysis and thermal analysis were done to determine the fiber’s ability to get adhesion. SLF density and wax content was determined using liquid immersion test using toluene and standard protocol respectively [19]. Using standard test strategies, the major bio - compound elements of SLF such as cellulose, hemicelluloses, and lignin content were

Figure 1. (a) Spinifex Littoreus Plant (b) SLF Stem (c) Dried SLF.
Table 1. SLF Properties with other natural and synthetic fibers [11, 24–27].

| Fiber     | Cellulose (wt%) | Hemi cellulose (wt%) | Lignin (wt%) | Wax (wt%) | Moisture (wt%) | Diameter (μm) | Density (g·cm⁻³) | Length (mm) | Tensile Strength (MPa) | Young's modulus (GPa) | Elongation (%) |
|-----------|-----------------|----------------------|--------------|-----------|----------------|---------------|------------------|-------------|------------------------|----------------------|-----------------|
| Spinifex  | 76.20           | 10.9                 | 18.5         | 1.28      | 10.22          | 425.6         | 1.033            | 90–150      | 240–380                | 3–7                  | 3.25–9          |
| Cissus    | 77.17           | 11.02                | 10.45        | 0.14      | 7.3            | 440           | 1.51             | 30–160      | 1857–5330              | 68–203               | 3.57–8.37       |
| Areca     | 57–58           | 13–15                | 23–24        | 0.12      | 7.31           | 395–475       | 0.75–0.8         | 12–60       | 146–323                | 1.23–3.156           | 10.24–13.16     |
| Pine apple| 81              | —                    | 12.7         | —         | 13             | 20–80         | 0.8–1.6          | 3–9         | 400–627                | 1.44                 | 14.5            |
| Jute      | 64.4            | 12                   | 11.8         | 0.7       | 1.1            | 17–20         | 1.3              | —          | 393–773                | 26                   | 1.5–1.8         |
| Coconut   | 32.5–43.5       | 0.18–22              | 42–46        | —         | 7.8            | 102–450       | 1.12–1.40        | 22–140      | 97–228                 | 2.7–6.5              | 15.2–51         |
| Palm      | 59–66           | —                    | 20–30        | —         | —              | 152–510       | 0.6–1.3          | —          | 82–245                 | 0.6–3               | 18–24           |
| Sisal     | 65              | 12                   | 9.9          | 2         | 11             | 7–47          | 1.5              | 0.8–8       | 511–635                | 9.4–22               | 2–2.5           |
| Bagasse   | 55              | 16.6                 | 25.4         | —         | 8.6            | 12–36         | 1.3              | 0.9–3       | 280                    | 19                   | 0.85            |
| Hemp      | 65              | 16                   | 12           | 0.85      | 9.5            | 24–36         | 1.5              | 5.5–56      | 695                    | 72                   | 1.65            |
| Abaca     | 57–64           | 22–26                | 7.2–8        | 3.2       | 15.5           | —             | 1.6              | —          | 420                    | 12.5                 | 3.5–11          |
| Cotton    | 82.7            | 5.7                  | —            | —         | —              | 10–20         | 1.5–1.6          | 15–56       | 287–800                | 5.5–12.6             | 7–8             |
| S-glass   | —               | —                    | —            | —         | —              | —             | 2.5              | —          | 4570                   | 86                   | 2.8             |
| E-glass   | —               | —                    | —            | —         | —              | 8.3–15.5      | 2.8              | —          | 1900–3600              | 75                   | 2.7             |
| Carbon    | —               | —                    | —            | —         | —              | 5.5–110       | 1.9              | —          | 2450–4100              | 235–390              | 1.45–1.9        |
determined [20]. ASTM E 1755–61 the standard was used to evaluate the ash content of the fiber. The water content present in the sample could be eliminated through heating at 104 °C for 4 h in a boiler [21]. Tensile testing machine INSTRON (5500R) was used in the evaluation of the mechanical properties of SLF in accordance with ASTM D382207. Twenty tests were carried out under suitable environmental conditions by maintaining the cross - head speed of 5 mm for every min in four trials with various lengths of 10 mm to 50 mm.

2.2. Fabrication of composite
Isophthalic Unsaturated Polyester was used as resin; Cobalt Naphthalene was used for accelerator and Methyl Ethyl Ketone Peroxide as a catalyst for manufacturing the composite. These were brought from New Emperor Pvt. Ltd, Nagercoil, Tamil Nadu, and India. The composite was fabricated using a compression moulding method. The mould was prepared to the required size of 300 mm × 125 mm × 3 mm. Wax was applied over the mould surface, fiber was spread over and resin mix was poured over it. Then it is compressed using a compression moulding machine by applying 15 MPa pressure. Composite was taken from mould and kept for curing for about 24 h at room temperature. After curing, the test samples were cut to the required size as per ASTM standards.

2.3. Mechanical testing
Tensile tests were performed on the composite samples of dimensions 200 mm × 20 mm × 3 mm as per ASTM D3039 and the flexural properties on the specimen of dimension 127 mm × 12.7 mm × 3 mm by three point bending test using ASTM D790. The Charpy test was done with composite sample of dimensions 65 mm × 13 mm × 3 mm according to ASTM D256. Hardness was noted using the ASTM D785–98 standard computerized Rockwell hardness testing machines. The mean value of five specimens was calculated and reported for each test. All the tensile and flexural samples were tested in Instron universal testing machine. Before testing, the machine is set to the load control mode through cross head speed as 10 mm min⁻¹. All the tensile samples were held under a wedge grip with which there is no slip. In flexural and impact testing there is no error due to slip.

2.4. Morphology and thermal testing
The samples were coated with a thin layer of gold for making them conductive and for maintaining a strategic distance from electron charge gathering. Scanning Electron Microscope (SEM) JEOL M-6390 was used for examine the tensile fractured specimen. The specimens were then given gold coating and kept in an ionizer. Thermal Jupiter Simultaneous analyzer (Model STA 449 F3, NETZSCH, Germany) was used for assessment of the thermal behavior of the SLF and SLFC independently by the TGA. TGA was executed in the nitrogen atmosphere at a flow rate of 20 ml min⁻¹ for neglecting oxidation. To avoid temperature variations, SLF and SLFC were crumbled and kept in an alumina crucible at a heating rate of 10 °C min⁻¹ for avoiding temperature variations. For thermal analysis the samples were tested according to ASTM E1131 standard is followed [22].

3. Results and discussion

3.1. Investigations of SLF
The mechanical, Physico-chemical properties of SLF were correlated with the other natural fibers. Details are listed in table 1. SLF was examined using a polarized microscope in which variations the diameter from the range

![Figure 2. Tensile strength versus fiber weight.](image-url)
396 μm to 476 μm were found. The density of SLF noted was 0.78 ± 0.03 g cm⁻³. The chemical property of SLF was greatly dependent on the properties such as resilience, mechanical, biodegradability and fire resistance which will reflect on its application as reinforcement. The high cellulose content of SLF polymer composites 76.20 wt.% strengthens the mechanical properties of the composite [23]. The existence of Lignin enhances surface morphology, cell wall texture and fiber property for holding the composite matrix durable. The wax in SLF had a wt.% of only 1.28, which is an advantage for improve interfacial bonding among the fiber and matrix. It is low in examination with the other fibers such as sisal, Abaca, etc.

### 4. Tensile properties

Figure 2 shows the tensile strength of SLFC with varying fiber content compared with man-made glass fiber composites. The mechanical strength of the SLFC primarily depends on the fiber direction and the amount of reinforcement. However, the stress transfer among the fiber and matrix can take up different magnitudes with changes in fiber wt. %. The strength of unsaturated polyester (UPE) composite attained at 16 MPa without reinforcement. In the composite material, the expansion of fiber weight percentage shows the increment of tensile strength as per the expectation. The SLFC is found to have good mechanical strength at a fiber content of 40 wt. % as compared with other natural fibers for examples palmyra, empty fruit bunch and sisal [26, 27]. The strength attained in the SLFC fiber content is 22 MPa with 40 wt. % which is lower compared to the Glass Fiber Composites. While increasing the fiber content beyond this level, the tensile property is decreasing due to the retention of fiber straightness, decreased the elasticity and less interaction between the matrix and fiber [23]. Like

**Figure 3. Specific tensile strength versus fiber weight.**

**Table 2.** Average values of mechanical properties of SLFC compared with other composites.

| Fiber/Matrix     | Weight percent (%) | Tensile Strength (MPa) | Flexural Strength (MPa) | Impact strength (J m⁻¹) | Hardness (HRRW) | References |
|------------------|--------------------|------------------------|-------------------------|-------------------------|----------------|------------|
| SLF/Polyester    | 40                 | 22                     | 55                      | 28                      | 68             | Present Work |
| CQRF/Polyester   | 40                 | 27.34                  | 63.7                    | 39.91                   | 75             | [28]       |
| Palmyra/Polyester| 30                 | 18                     | 48                      | 42                      | —              | [29]       |
| Borassus/HDPE    | 15                 | 24.6                   | 20.1                    | 105                     | 70             | [30]       |
| S.cylindrica/Polyester | 40            | 75                     | 83.85                   | —                       | —              | [26]       |
| AFHF/Polyester   | 40                 | 68.20                  | 73.91                   | —                       | 76             | [31]       |
| GF/Polyester     | 40                 | 72.06                  | 53.16                   | —                       | 88             | [15]       |
| Banana/Epoxy     | 16                 | 16.39                  | 57.53                   | —                       | —              | [16]       |
| Empty fruit      | —                  | 15.89                  | —                       | —                       | —              |            |
| bunch/PET        | —                  | —                      | —                       | —                       | —              |            |
| Hemp/UPE         | —                  | 58                     | 110                     | —                       | —              | [15]       |
| Coir/Polypropylene | —              | 34                     | 49.3                    | —                       | 87             | [33]       |
| Abaca/Polypropylene | —             | 72                     | —                       | —                       | —              |            |
| Sisal/Epoxy      | —                  | 21.2                   | 62                      | —                       | —              |            |
| Hemp/Polypropylene | —              | 53                     | 54                      | —                       | —              |            |
the above mentioned reference, in this research work also at 40 wt. % SLFC the tensile strength becomes decreases due to the poor bonding of fiber. It leads to fiber evacuation and deception of matrix. On increasing the SLF at 50 wt%, the tensile strength becomes 17.5 MPa. Therefore for the fabrication of SLFC, 40 wt% fiber is recorded as the optimum processing parameter with tensile strength of 22 MPa. The mechanical properties of SLFC in comparison with other fiber reinforced composites are listed in table 2.

5. Specific tensile strength

Figure 3 shows a comparison between the specific tensile strength of SLFC and GFC with varying fiber weight percentage. It is defined as the ratio between the strength of the materials to their density. Specific strength is the most important factor while considering the loads of the engineering applications for reducing the dead load in critical conditions. As per the expectation, the increasing fiber weight percentage results in the increase specific tensile strength in composite material. Compared to GFC at 40 wt. %, SLFC comparable specific strength due to lower SLF fiber density (∼0.65 g cm⁻³), which is 28.42 kN m kg⁻¹ through specific tensile strength of GFC is higher. For lightweight applications without considering stiffness, the usage of SLFC is more due to high specific strength.

6. Flexural strength

Flexural property is considered as one of the most important parameters in structural applications. Figure 4 shows the comparison between the flexural strength of SLFC and GFC with varying fiber weight percentage. A gradual increase in flexural strength of composite up to 40 wt. % is seen. The orientation of fiber caused similarity in the flexural properties of SLFC to those of the tensile properties. Gradual increment of fiber content causes the increase in flexural strength of SLFC composite to a value of 52 MPa at 40 wt. %. In comparison with
UPE, the flexural strength of SLFC is 45% more. The trend of GFC shows identity among SLFC, but it shows better results at low wt. % of fiber. For the fiber content above 40 wt. %, the flexural strength of the composite faces a gradual reduction due to insufficient bonding of fiber and matrix.

7. Impact strength

Figure 5 shows the comparison between the impact strength of SLFC and GFC with varying fiber weight percentages. The resistance and toughness of the composite material were evaluated using impact tests. There are two main factors in deciding the impact strength of polymer composites. First, due to poor interfacial bonding crack is easily initiated because of micro-space created between the matrix and fiber and the second is the absorption energy in fiber that promotes the propagation of crack [28]. The addition of filler with the SLFC caused an increase in the hardness of the material. A gradual increase in impact strength has been noted from 0 to 50 wt. % of fiber content. Compared with UPE, the impact strength of SLFC is 25% more. The fiber and matrix have less interfacial bonding, which leads to a failure by fiber pulling out. At 40 wt. % fiber contented, the impact energy of SLFC increases which was less than GFC. The impact energy of SLFC was usually greater than used natural fiber composites.

8. Hardness

Figure 6 shows the hardness of SLFC fiber content compared with synthetic glass fiber composites. Hardness signifies surface strength against external loads. The composite hardness mainly depends upon the interfacial bonding between the filler and the matrix. This phenomenon is reflected on both SLFC and GFC during the testing of the composite. SLFC attains higher hardness at 40 wt. %. During the increase in SLF content, more than 40 wt. %, it leads to a decrement in mechanical performance as a result of lesser fiber-matrix bonding. The increasing weight percent causes a sudden drop in hardness.
Figure 7. SEM Images of fractured samples (a), (b) 10 fiber wt% (c), (d) 20 fiber wt% (e), (f) 30 fiber wt% (g), (h) 40 fiber wt% (i), (j) 50 fiber wt%.

Table 5. ANOVA test for impact strength.

| Source of Variation | SS     | df  | MS       | F       | P-value | F crit  |
|---------------------|--------|-----|----------|---------|---------|---------|
| Between Groups      | 871.657| 4   | 217.9142 | 4151.67 | 1.24E-22| 3.05568 |
| Within Groups       | 0.787325| 15  | 0.052488 |         |         |         |
| Total               | 872.4443| 19  |          |         |         |         |
A statistical one way ANOVA is performed for tensile, flexural and impact strength and it's shown in tables 3, 4 and 5 respectively. The entire table shows that, there is a significance difference for mechanical properties under various fiber weight percentages of polymer composite materials.

A one-way ANOVA was conducted for all the mechanical properties in order to find the statistical significance difference between the groups. One-way ANOVA is one of the most widely used approaches for analyzing the statistical difference between the groups.

H0. The null hypothesis is that all the groups have the same mean

H1. At least one of the means is different from the others.

In this analysis, the group refers to the various wt% of composites. Each wt% of composites is tested with five samples. Five specimens considered under five various wt% for analysis. Total 25 samples were used for testing. All related parameters like, for example Sum of Squares (SS), the degrees of freedom (DF), the Mean Squares (MS), the ratio of MS among-groups to MS within-groups (F) and the P-value (p) for all the mechanical properties are listed out in tables 3, 4 and 5 respectively. The F critical value is found as 3.05 on the basis of the value of the degrees of freedom (4, 15) derived from the analysis. From this table, if \( F_{\text{actual}} > F_{\text{critical}} \), then it can be inferred that the values of the groups are likely to be statistically unequal. From all the ANOVA table, the \( F_{\text{actual}} \) is greater than the \( F_{\text{critical}} \) value. Its indicates that reject the H0 statement and the mechanical properties are statistically significance different from each groups. The \( F_{\text{actual}} \) value for tensile strength of composites is 405.3 > 3.05 which is greater than the \( F_{\text{critical}} \).

9. Micro-structural analysis

The SEM micrographs of tensile fractured samples of SLFC reinforced composites are presented in figures 7(a)--(j). Figures 7(a) and (b) indicate the porous fiber surface that enhances bonding with matrix fractured tensile sample at 10 wt. %. The lower fiber content present in the matrix leads to failure in conveying the load and initiates a crack in the fiber matrix bonding. Figures 7(c) and (d) shows the micrograph of the broken tensile sample with 20 wt. %. Inadequate bonding between fiber and matrix interphase is seen in leading to failure of SLFC. Meanwhile, a limited amount of delamination was seen at the fiber-matrix interface. At 30 and 40 wt. % (figures 7(e) and 7(f)) also shows better bonding between fiber and matrix. An increase in the wt. % of fiber content, shows a possible increase in the bonding among interfaces. The presence of honeycomb structure observed at 40 wt.% fiber content composite sample (figures 7(g) and (h)) shows better bonding among the fiber and matrix. However, the minimum fiber pullout was observed. This increases the tensile strength of the composite in figures 7(i) and (j) at 50 wt.%. It shows an increase in the fiber to fiber contact due to lack of matrix content in the composite, leading to the formation of voids causing a decrease in mechanical properties. Accordingly, there is a decrease in the capability of load transfer capacity between the reinforcement and the matrix.
9.1. Thermal analysis of SLF and SLFC
Natural fibers may be affected by the application of heat, due to thermal degradation; the mechanical properties may be inferior. Results of the Thermal Analysis of SLF and SLFC are shown in figures 8 (a), (b) presents the TG curve of SLF and SLFC. Commonly the natural fiber based polymer composites are initially degraded at a temperature of 42 °C–58 °C due to evaporations of water molecules [34]. The thermal stability of SLF and SLF made with polyester a minimum weight reduction was observed up to 50 °C. The second degradations of thermal stability of SLF and SLFC made with polyester matrix was noted up to 300 °C. The main reason for second stage of mass loss is removal or degradations of lingo cellulosic fibers. SLF’s thermal stability is comparable to that of other natural fibers like wild, veldt grape, date palm. The third stage of degradation observed for SLF is around 340 °C and SLFC has over 400 °C. The thermal stability of SLFC is more compare to SLF. Fiber was seen burning out completely at the temperature of 340 °C–58 °C due to evaporations of water molecules [34]. The thermal stability of SLF and SLFC made with polyester matrix was noted up to 300 °C. The main reason for second stage of mass loss is removal or degradations of lingo cellulosic fibers. SLF’s thermal stability is comparable to that of other natural fibers like wild, veldt grape, date palm.

10. Conclusions
A study of the results of mechanical and thermal degradation shows SLF as an excellent alternative to man-made hazardous fibers and can be used for making green composites. The low density of SLF (0.65 g cm⁻³) with better mechanical properties in the polymer matrix provides a high specific tensile strength (28.42 kNm kg⁻¹) to SLFC. Hence the mechanical properties of SLFC compare well with the properties of the GFC. Higher mechanical strength and excellent toughness are found to be possessed for a combination of 40 wt.% fiber loading composite. SEM morphological analysis in tensile tested samples, shows the fiber pull out, voids, cracks and interfacial gap during the fiber loading condition from 10 wt.% to 30 wt.% of the composite. At 40 wt.% the composite has a minimum fiber pull out which results in the transfer of high loads. The TGA clearly shows the stability of SLFC up to 400 °C and the capacity to withstand the polymerization process temperature. This suggests opening up of another opportunity to replace unsafe GFC in many applications with an environmentally friendly natural fiber SLFC. The newly fabricated polymer composites materials are applicable to Automobile interior components, Engine guard for two-wheelers, Door panels, table fan blade, etc.

Data availability statement
The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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