Characteristic Study on Sisal Fibre Hybrid Composites Filled With Nano SiO$_2$ and Marble Dust Particles

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Abstract. The synthetic fibres are having high stiffness and strength still high manufacturing cost with environmental issues limited the use of synthetic fibres. This encourages towards thinking of intense research of natural fibres having similar property as synthetic fibres. To obtain the better mechanical and thermal property, hybridization of fibres is mostly preferred and that leads to minimize the use of synthetic fibres. As natural fibres are strong and having more ductile property, though moisture absorption is the primary limitation. So fibres is subjected to various treatments such as physical, thermal, chemical and laser treatments etc., to improve the surface characteristics with better interfacial bonding. Nano particle addition also helps in improving the performance of composites and develops cross linking in bonding of matrix and fibre. This research work mostly focuses on hybridization of short mercerized sisal and glass fibre filled with nano silica and marble dust particles, by injection moulding process with polypropylene matrix. It is noticed that among the addition of different fillers, marble dust added composite gave the better result than SiO$_2$ added composite. Incorporation of SiO$_2$ particles as filler material improves the flexural strength and thermal property of the composite.

Keywords: Fibre Hybrid Composites, sisal fibre (SF), glass fibre (GF) and SiO$_2$

1 Introduction

The environmental friendly nature of natural fibres are most attracted to minimise the use of unnatural fibres because of their light weight, high specific strength, low energy combustion, renewable, biodegradable characteristics and reduced manufacturing cost. Flax, jute, hemp, sisal, bamboo, banana are the frequently used fibres in most of the automobile, aerospace structures, marine, sports equipment etc. due to its light weight and which ultimately leads to less power consumption. Nowadays natural fibres are also having wide application in most of the civil structural components. But it has some limitations like poor moisture resistance, low modulus which is enhanced by hybridization of composite. Mainly natural fibre are produced from three sources i.e. Vegetable, Animal, Mineral.

Many investigations have already been done, to upgrade the adhesive behaviour among the interface of fibre and matrix by adaptive selection of reinforcement, following the application of few chemical and physical treatments before fabrication. The physical approach such as heat treatment, application of plasma is employed to modify the fibre/matrix [1-4]. Thermal treatment is much more important as it gives the accurate shape of the fibre and reduces shrinkage after the composite preparation. The harmful effect of alkali treatment towards the fractured toughness of sisal fibre is eminently studied but at the same time, for coconut fibre composite the fracture toughness increased with the alkaline treatment [6]. Alkali treated fibre and heat treated fibre enhanced the tensile property of the composites than the untreated fibre composite [7].

To enhance the mechanical strength as well as moisture protection of the natural and thermoplastic composite, man-made fibres are introduced for hybridization of the polymer composites. Srisuwana et al. [8] reported that, by adding glass fibre to sisal–
Polypropylene composites, thermal properties and water absorption resistance of the composites materials are significantly enhanced. Cicala et al. [9] profoundly obtained the importance of glass fibre reinforcement with natural fibres like Flax, Hemp, and Kenaf in the piping industry. After they studied different properties like tensile and flexural, the result shows that E-glass gave better properties than the natural fibre. Micro and nano organic or inorganic filler addition up to a certain limit, mostly improves the mechanical, physical and thermal behaviour of composite material. Vieira et al. [10] reported that the latency of nano silica particles added significantly to improve the mechanical characteristics of the sisal reinforced composites, exhibiting superior stiffness and flexural strength. Using Portland cement as a filler material in sisal glass polymer composite, increases toughness in the laminate [11].

In addition to the above and few more literatures survey, it has been found out that none of the work on sisal fibre hybrid composites filled with nano SiO2 and marble dust particles has carried out so far as per the knowledge of authors. Present investigation is an effort to get a high strength and toughness hybrid composite which was tested in various mechanical and thermal properties.

### 2 Experimental Details

#### 2.1 Materials Preparation

Polypropylene was used as a matrix, short sisal-glass fibres were the reinforcement and nano marble dust (MD), SiO2 were considered as filler materials. Polypropylene (PP), sisal fibre (SF), glass fibre (GF) and SiO2 were purchased from Go-green products Pvt ltd, Chennai. The glass fibre was short in length (length approximately 6 mm). The density of polypropylene, sisal fibre and glass fibre were 0.91 g/cm³, 1.58g/cm³ and 2.53 g/cm³ respectively. The marble dust was prepared by cutting the marble piece by cutter and obtained in powder form by the help of a nano strainer As shown in below Table 1, Table 2 and Table 3.

To eliminate the impurities and wax the sisal fibres were applied to treatment of 1% NaOH solution. The solution is prepared in 25 litres of water adding 250gm NaOH and after the solution is prepared, immersed all the sisal fibre into the solution for 24 hrs following the neutralisation of fibre with 3% acetic acid solution. Then fibres are cleansed with distilled water and subjected to dry for 24 hrs in room temperature then 3 hrs in the oven at 60º C. After that the sisal fibres were cut into a length of approximately 6 mm

### Table 1. Sample composition and its reinforcement wt%.

| Sample | Classification | Polypropylene (wt %) | Sisal fibre (wt %) | Glass fibre (wt %) | SiO2 (wt %) | Marble Dust (wt %) |
|--------|----------------|---------------------|-------------------|-------------------|-------------|-------------------|
| A      | PP-SF-GF       | 70                  | 15                | 15                | ___         | ___               |
| B      | PP-SF-GF-SiO2  | 70                  | 12.5              | 12.5              | 5           | ___               |
| C      | PP-SF-GF-MD    | 70                  | 12.5              | 12.5              | ___         | 5                 |

A batch mixer Rheomix 600 having chamber volume 60-90 cm³ was used to melt and mix the PP with short sisal fibre, glass fibre, and additive material by the help of two opposite rotating rotors. One batch mixing contains 50 gm of material and during the process the temperature maintained was 190ºC and time of mixing is 10 min. After the formation of a proper mixture in the chamber, the material was taken out from it and cut into small pieces. Then small pieces were fed into the injection moulding machine to prepare the specimen, maintaining the fibre content 30% and polypropylene was fixed 70% in the entire sample. The test specimen was fabricated by the help of injection moulding and specimens were prepared for tensile using standard ASTM D 638, ASTM D 790 for flexural and ASTM D 256 impact respectively. Composition of sample and its reinforcement wt% of the polymers and reinforcements in composites are shown in table 1. The prepared tensile, flexural and impact specimens were shown in figure 1.
2.2 Mechanical Test

Tensile test were carried out according to ASTM D638 standards using INSTRON Universal Testing Machine (UTM) Model 8801 with crosshead speed 50 mm/min. For each sample at least three specimens were tested. The transverse beam test or Flexural test is conducted to evaluate the behaviour of material subjected to simple beam loading. Maximum stress-strain was calculated and results were plotted in a stress-strain diagram. Flexural test conducted by two types one is 3-point flexural and other one is 4-point flexural. 3-point flexural test was commonly used for polymer according to ASTM D790 standards using INSTRON 4486. The rate of energy absorption by a material during fracture is determined by impact test. The Izod test is a dynamic test in which a v-notched sample clamped vertically is broken by a freely swinging pendulum and the specimen was prepared according to the standard of ASTM D 256 with a head speed of 10 mm/min.

2.3 Thermal Test and Physical Tests

Thermo gravimetric analysis or Thermal gravimetric analysis (TGA) is most preferably used for analysis of thermal properties, where the mass of a sample is measured over time at different temperatures. N₂ and O₂ gases are used to conduct thermal stability by the TA (USA) machine. Moisture absorption behaviour of the composites was operated by ASTM D 570. Two specimens of each composition were exposed to water at room temperature for 24 hour. The following equation represents the Water absorption.

\[ WA = \frac{W_2 - W_1}{W_1} \times 100 \]  

(W1 represents the specimen weight before water immersion and W2 represents the weight of the specimen after water immersion.)

2.4 Thickness swelling and Density measurement

Thickness Swelling is same as water absorption. The difference is here to measure the thickness of the specimen before and after water immersion. Thickness swelling is calculated by following equation

\[ Thickness\ Swelling(\%)= \frac{T_1-T_0}{T_0} \times 100 \]  

Here \( T_0 \) = Thickness of the specimen before water soaking and \( T_1 \) = Thickness after soaking.

ASTM D 1895 standard is appreciably used for the density measurement of the composites. The following equation is used to calculate the density of the samples.

\[ \text{Density (g/cm}^3) = \frac{M}{V} \]  

Where, \( M \) = mass of the composite and \( V \) = volume of the composite.

3 Results Analysis

3.1 Mechanical characteristics

3.1.1 Tensile result

The tensile result of samples is represented in Table 2 and it is remarkably noticed that addition of marble dust in composite gave better tensile stress as compared to nano SiO₂ powder in composite. But the glass and sisal fiber reinforced composites without any filler provides better tensile stress i.e 33.208 MPa among all. The nano SiO₂ composite gave 29.975 MPa tensile strength and which is increased by 2.66%, with the incorporation of 5% marble dust filler. In comparison to PP-SF-GF composite the addition of marble dust and nano SiO₂ to it, decreased the tensile strength by 7.2% and 9.7% respectively. The tensile result and tested specimens of sample A, B and C are plotted in the graph which is shown in Figure 2. The modulus value increases by adding nano SiO₂ than marble dust filler. As, SiO₂ is a ceramic crystalline or...
amorphous material and behaves like brittle material, ultimately that reduces the tensile properties but carries the maximum load due to good interfacial bonding and lower specific surface area.

Table 2. Tensile results of samples.

| Sample | Tensile stress at Maximum Load (MPa) | Tensile stress at Break (Standard) (MPa) | Modulus (MPa) |
|--------|-------------------------------------|-----------------------------------------|---------------|
| A      | 33.208                              | 33.164                                  | 2107.44314    |
| B      | 29.975                              | 29.897                                  | 1828.06937    |
| C      | 30.796                              | 30.624                                  | 1738.02601    |

3.1.2 Flexural result

Table 3 represents the flexural test results of the specimens. PP-SF-GF composite filled with SiO$_2$ gave better flexural modulus result among the all above three compositions that was 2612.733 MPa but without filler PP-SF-GF and PP-SF-GF filled with marble dust gave flexural modulus of 2685.295 MPa and 2365.021 MPa respectively. Figure 3 shows the flexural modulus and flexural tested specimens of sample A, B, C. Here composite is a ductile material so it gives better bending stress. The composite filled with nano silica (SiO$_2$) provides better bending stress due to good adhesion in interfacial zone of fiber and matrix along with enhanced structural property.

Table 3. Flexural results of samples.

| Sample | Maximum Load (N) | Maximum Stress (MPa) | Flexural Modulus (MPa) |
|--------|------------------|----------------------|------------------------|
| A      | 79.722           | 46.616               | 2685.296               |
| B      | 81.790           | 47.825               | 2612.733               |
| C      | 78.408           | 45.847               | 2365.021               |

3.1.3 Impact results

Impact strength was maximum i.e. 2.5691 KJ/m$^2$ in case of sample A which contained PP-SF-GF that does not contain any additive but after adding marble dust and nano SiO$_2$ the impact strength decreased by 7% and 19% respectively. By comparing Marble dust and SiO$_2$, marble dust composite increases 13% impact strength than the SiO$_2$ composite because of the ductile behaviour and maximum energy absorption capacity of marble dust. The Impact strength of all the three compositions has shown in Table 4. Impact strength of sample A, B and C with tested specimens are plotted in Figure 4. The matrix polypropylene and the manufacturing process,
injection moulding itself is the key factor of improving the impact strength in sample A, as shown in below Table 4 and Table 5, Table 6,7.

**Table 4. Impact results of samples.**

| Sample | Break (Joule) | Impact Strength (KJ/m²) |
|--------|---------------|-------------------------|
| A      | 0.0835        | 2.5691                  |
| B      | 0.0795        | 2.0718                  |
| C      | 0.09175       | 2.3904                  |

**Table 5. TGA results of samples.**

| Sample | Peak point temperature (°C) | Derive weight change (%/°C) | Weight Change (%) |
|--------|----------------------------|-----------------------------|-------------------|
| A      | 414.79                     | 1.498                       | 85.58%            |
| B      | 415.51                     | 1.400                       | 80.93%            |
| C      | 414.07                     | 1.372                       | 84.57%            |

**Fig. 4.** Impact strength of sample A, B and C with tested specimens

**3.2 Thermal properties**

**3.2.1 TGA Analysis**

In the thermal stability analysis of sample A the rate of change of mass occurred at the temperature of 414.79°C and in 800°C the mass decreased 85.58% of total mass. In case of sample B the mass of change occurs at the temperature of 415.51°C and the mass decreased 80.93% of total mass at 800°C, similarly in sample C has decreased 84.57 % of total mass at 800°C. The TGA results of sample A, B and C were shown in Table 5. Figure 5 shows the weight change of samples, where there was a significant weight change in case of sample A.

**Fig. 5.** Weight change % of sample A, B and C.

**3.3 Physical Characterization**

**3.3.1 Water Absorption Analysis**

The percentage of moisture retention in composite samples was estimated and ultimately it is found that water absorption capacity of all the three samples of WAA, WAB, and WAC were 0.44%, 0.3760%, 0.388% respectively. Sample A gave higher water absorption capacity because it contains higher percentage of
natural fibre than other two compositions. But in case of sample B and C, moisture absorption is comparatively less due to the nano fillers that show better compatibility in fibre and matrix by minimizing the voids in between them.

\[
W_{A} = \frac{2.83975 - 2.8273}{2.8273} \times 100 = 0.44\%
\]

\[
W_{A} = \frac{3.31005 - 3.29765}{3.29765} \times 100 = 0.3760\%
\]

\[
W_{A} = \frac{2.8717 - 2.8606}{2.8606} \times 100 = 0.388\%
\]

| Table 6. W1&W2 |
|----------------|
| Sample | Specimen 1 weight (gm) | Specimen 2 weight (gm) | Mean (gm) | Sample | Specimen 1 weight (gm) | Specimen 2 weight (gm) | Mean (gm) |
| A | 3.077 | 7 | 2.576 | 9 | 2.82 | 73 | A | 3.092 | 3 | 2.587 | 2 | 2.83 | 975 |
| B | 3.469 | 8 | 3.126 | 1 | 3.29 | 765 | B | 3.482 | 5 | 3.137 | 6 | 3.31 | 005 |
| C | 2.966 | 6 | 2.754 | 6 | 2.86 | 06 | C | 2.976 | 6 | 2.766 | 8 | 2.87 | 17 |

Where \( W_{A} \), \( W_{B} \) and \( W_{C} \) represented the water absorption result of sample A, B and C respectively.

### 3.3.2 Thickness Swelling Analysis

Thicknesses before and after water soaking was recorded in Table 7. The percentage of thickness swelling was calculated and it is found that, the thickness change of all the three samples of \( T_{S_{A}}, T_{S_{B}}, T_{S_{C}} \) were 10.322%, 7.09% & 7.7419% respectively. Sample A shows extreme water absorption capacity because it contains higher % of natural fibre than other two compositions and also in other two samples the fillers were having better bond strength due to least specific surface area.

\[
T_{S_{A}} = \frac{3.42 - 3.10}{3.10} \times 100 = 10.322\%
\]

\[
T_{S_{B}} = \frac{3.32 - 3.10}{3.10} \times 100 = 7.09\%
\]

\[
T_{S_{C}} = \frac{3.34 - 3.10}{3.10} \times 100 = 7.7419\%
\]

| Table 7. T0 and T1 values |
|--------------------------|
| Sample | T0 values in mm | T1 values in mm |
| A | 3.10 | 3.42 |
| B | 3.10 | 3.32 |
| C | 3.10 | 3.34 |

### 3.3.3 Density of the composite

Mass and volume of specimens were recorded in Table 8. The composites which contain marble dust as a filler material gave higher density as compared to the other two compositions because marble dust has higher density among them as shown in below Table8.

The density of sample A, B and C can be calculated as

\[
\rho_{A} = \frac{m}{v} = \frac{2.830}{2.5074} = 1.12865 \text{ g/cm}^3
\]

\[
\rho_{B} = \frac{m}{v} = \frac{2.850}{2.4777} = 1.15026 \text{ g/cm}^3
\]

| Table 8. Mass and volume of the Samples |
|---------------------------------------|
| Sample | Length (cm) | Width (cm) | Thickness (cm) | Volume (cm³) | Mass (gm) |
| A | 6.440 | 1.256 | 0.31 | 2.5074 | 2.830 |
| B | 6.324 | 1.264 | 0.31 | 2.4777 | 2.850 |
| C | 6.364 | 1.250 | 0.31 | 2.4660 | 2.930 |

### 4 Conclusions

The above experimental investigation mostly predicts some important characteristics of sisal reinforced polypropylene composites filled with SiO₂ and marble dust nano-particles.
The fabrication of PP-SF-GF composite filled with SiO$_2$ and marble dust particles was done by an injection moulding process which is more superior than hand layup process in case of short fibers in improvement of the characteristics of the composites. PP-SF-GF (sample A) composite gave the better tensile strength as compared to other composite samples. It is noticed that among the addition of filler, marble dust composite gave the better tensile strength than SiO$_2$ composite. As SiO$_2$ is a ceramic material of crystalline or amorphous nature that reduces the tensile strength due to its brittle behaviour. Incorporation of SiO$_2$ particles as filler material improves the flexural strength of the composite as compared to marble dust filler due to better interfacial adhesion that enhance the structural strength. Impact strength is maximum in case of sample A which contains PP-SF-GF (does not contain any filler material) because it carries a maximum percentage of sisal fibre than other samples. Marble dust additive composite increased 13% impact strength than SiO$_2$ additive composite.

As SiO$_2$ is ceramic in nature, its incorporation as a filler results significant improvement in the thermal property. Sample B gave better results in TGA analysis which shows the result of total mass decreases up to 80.93% where sample A, sample C decreases mass up to 85.58% and 84.57% respectively. In the field of physical properties it shows that sample A absorbed a high amount of water as compared to the other two samples. The addition of filler SiO$_2$ and marble dust water absorption decreases due to better interfacial bonding and low specific surface area of nano particles. By using nano particles like SiO$_2$ it gave better flexural and water resistance capacity because due to good agglomeration, homogeneous dispersion and high aspect ratio developed good interfacial bonding that finally enhance the overall strength of the composites. The composites which contain marble dust as a filler material have higher density as compared to the other two compositions because marble dust has higher density among them.

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