Steel and Composite Structures, *Vol. 33, No. 2* (*2019*) *299-306*
DOI: https://doi.org/10.12989/scs.2019.33.2.299

Experimental and microstructural evaluation on mechanical properties of sisal fibre reinforced bio-composites

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(*Received May 18, 2019, Revised October 1, 2019, Accepted October 6, 2019*)

Abstract. The natural fibre composites are termed as bio-composites. They have shown a promising replacement to the current carbon/glass fibre reinforced composites as environmental friendly materials in specific applications. Natural fibre reinforced composites are potential materials for various engineering applications in automobile, railways, building and Aerospace industry. The natural fibre selected to fabricate the composite material is plant-based fibre e.g., sisal fibre. Sisal fibre is a suitable reinforcement for use in composites on account of its low density, high specific strength, and high hardness. Epoxy is a thermosetting polymer which is used as a resin in natural fibre reinforced composites. Hand lay-up technique was used to fabricate the composites by reinforcing sisal fibres into the epoxy matrix. Composites were prepared with the unidirectional alignment of sisal fibres. Test specimens with different fibre orientations were prepared. The fabricated composites were tested for mechanical properties. Impact test, tensile test, flexural test, hardness test, compression test, and thermal test of composites had been conducted to assess its suitability in industrial applications. Scanning electron microscopy (SEM) test revealed the microstructural information of the fractured surface of composites.

Keywords: sisal fibre; bio-composites; epoxy; flexural; tensile; matrix; SEM

1. Introduction

The demand for a new generation of advanced materials in modern industrial applications has led to the development of composite materials. A composite material is a physical combination of two or more different materials that result in a component with superior properties compared to any individual component (Jones 1999). When compared to metallic alloys, each material retains its own physical, chemical, and mechanical properties. The main advantages of composite materials are their high specific strength and stiffness when compared with other materials allowing for a reduction in weight of finished parts. The constituents of a composite are categorized as the matrix and reinforcement. The matrix is the continuous phase while their reinforcement is the discontinuous phase (Barbero 2017). The reinforcement is usually embedded in the matrix and the reinforcement is usually in the form of sheets, fibres or particles whereas the matrix is either a polymer, ceramic or metal. The reinforcement provides strength and stiffness to the composite material (Jones 1999).

A re-emerging area of fibre reinforced composites is that of natural fibre reinforced composites. Natural fibres are increasingly being considered as an environmental friendly substitute for synthetic fibres in the reinforcement of polymer-based composites. Although many different types of natural fibres are available, sisal fibre is particularly preferred for its low cost and rapid growth over a wide range of climatic conditions (Daniel *et al*. 2006). Although the use of natural fibres partly satisfies the requirements of regulations that enforce the use of environmentally friendly and sustainable materials, the matrix system must also be considered in this regard. Completely biodegradable matrices such as polylactic acid are preferable, but the current high cost of these matrices is a disadvantage in comparison to epoxy resin. Sisal/epoxy composites are therefore of considerable commercial interest.

Chemical treatments of natural fibres are done in order to achieve the necessary compatibility of surface energies between the fibres and the matrix (Srisuwan *et al*. 2014). Chemical treatments of natural fibres are done in order to achieve the necessary compatibility of surface energies between the fibres and the matrix (Srisuwan *et al*. 2014). Chemical treatments such as alkali treatment, silane treatment, acetylation, benzoylation, use of maleate coupling agents, peroxide treatment, permanganate treatment, and isocyanate treatment are done to improve the bond between fibre and matrix. Amongst the various methods presented, alkali treatments and silane treatments have been widely used for its low cost (Sood and Dwivedi 2018). One advantage of natural fibers is their lowdensity, which results in higher tensile strength and stiffness than glass fibers, besides its lower manufacturing costs (Maurya *et al*. 2015). As such, bio-composites could be an excellent ecological alternative to glass, carbon and man-made fiber composites. Natural fibers have a hollow structure, which provides insulation against heat and noise. It can be easily processed, and thus, they are suited to a wide range of applications, such as building, aerospace, electronics,

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ISSN: 1229-9367 (Print), 1598-6233 (Online)

military applications, consumer products and medical industry (prosthetic, total hip replacement, bone plate, and composite screws and pins). In literature, various researchers have reported studies on the mechanical and dynamic behavior of bio/natural fibre composites. (Srisuwan *et al*. 2014) studied the effects of alkalized and silanized woven sisal fibers on mechanical properties of natural rubber modified epoxy resin. (Rajesh *et al*. 2016) have studied the influence of surface pre treatment with sodium hydroxide and hybridization effect of natural fiber are investigated on the flexural test and free vibration behavior. In this work sisal and banana natural fibers are used in short and random orientations to prepare the polymer composites using compression moulding method. From the experimentation, it is found that chemical treatment improves the mechanical and free vibration properties of polymer composites due to the enhancement of interfacial bond between fiber and matrix as the result of chemical treatment. (Prasad *et al*. 2017) did a comparative study between treated and untreated sisal polyester composites are tested to study its impact strength characteristics. ASTM D256 norms are followed to conduct the impact test on the specimens of 2 mm, 3 mm, 4 mm, 5 mm and 6 mm thicknesses for fiber volume of 10%, 15%, 20%, 25%, and 30%. (Gupta and Srivastava 2014) presented the tensile and flexural properties of sisal fibre reinforced epoxy composite and carried out a comparison between unidirectional and mat form of fibres. de Souza Castoldi *et al*. (2019) compared the mechanical behavior of concretes reinforced with polypropylene and sisal fibers. Both fibers were 51 mm long and were incorporated in fractions of 3, 6 and 10 kg/m^3 into the matrices. The composites were tested under three point monotonic and cyclic flexural loads. Pullout tests were performed to study the fiber matrix interaction. It was observed that the sisal fiber could provide the same level of residual strength as the polypropylene fiber, as long as the equivalence of the dosages of the fibers is taken into account. Gupta and Srivastava (2015) studied the effect of sisal fibre loading on dynamic mechanical analysis and water absorption behaviour of jute fibre epoxy composite. The dynamic mechanical properties (storage modulus, loss modulus, and damping parameter) and water absorption characteristic parameters (sorption, diffusion and permeability coefficient) of hybrid sisal and jute fibre reinforced epoxy composite were studied. Morphological analysis was carried out to observe uniform dispersion of fibres in the matrix using a scanning electron microscope. The results showed that the hybrid composite with 30 wt.% jute and sisal fibre content shows the maximum value of storage and loss modulus whereas a lower value of damping parameter and percentage water absorption. Singh *et al*. (2015) investigated the effect of reinforcing sisal and hemp fibers, either alone or simultaneous, into a polymer matrix, containing 50:50 mixture of fresh and recycled HDPE, on the tensile behavior. Prior to the manufacturing of composite, the fibers were chemically treated with NaOH and Maleic Anhydride (coupling agent). The tensile strength of the sisal/hemp fibers/HDPE hybrid composite is observed to be superior to single fiber (hemp/sisal) reinforced HDPE composites. Elanchezhian *et al*. (2018) reported a paper on review of mechanical properties of NFRC, they tested the fibers namely abaca, jute, sisal, banana, coir, etc for their mechanical properties. Ding *et al*. (2019) studied the mechanical behaviors of concrete filled rectangular steel under torsion load. They have shown that if it is reinforced with FRP its strength improves significantly. Relevant studies have been reported by several researchers in the literature (Han *et al*. 2017, Tuhta 2018, Mustafa 2018, Kumar 2018, Sood and Dwivedi 2018, Ebrahimi and Barati 2018, Ebrahimi and Haghi 2018, Sreehari *et al*. 2017).

2. Material and experimental methods

2.1 Material

Although many different types of natural fibres are available, sisal fibre is a particularly attractive option due to its low cost and rapid growth over a wide range of climatic conditions. Although the use of natural fibres partly satisfies the requirements of regulations that enforce the use of environmentally friendly and sustainable materials, the matrix system must also be considered in this regard. Completely biodegradable matrices such as polylactic acid are preferable, but the current high cost of these matrices is a disadvantage in comparison to epoxy resin. Sisal/epoxy composites are therefore of considerable commercial interest.

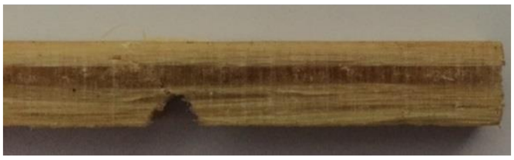
Reinforcement- Sisal Fibre

Matrix- Epoxy LY556

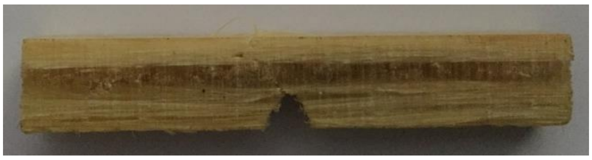
Hardener- HY 951(Araldite hardener**)**

Fig. 1 Sisal fibres

Fig. 2 Composite plate after fabrication



(a)



(b)

Fig. 3 (a) Sample for Izod Test; (b) Sample for Charpy Test

2.2 Fabrication of composite

The most common methods used in the manufacture of composite materials are compression moulding, hand layup, resin transfer moulding (RTM), injection moulding, and filament moulding among which hand layup technique is the most economical method so hand layup technique is used for fabrication of Sisal/epoxy composite.

Fibre Combing is done to separate the entanglement and the inter-twinning of the fibres to have long separate strands of fibre for better bonding of fiber with the matrix.

Matrix Epoxy LY556 and Hardener HY951 are mixed in the ratio of 10:1 and the mixture is coated over each layer of fibre placed inside a square mould box of inner dimensions 30 cm × 30 cm × 1 cm. Three layers of fibres are placed in the 90-0-90-degree orientation inside the mould box placed over a flat plate and are impregnated with resin. Finally curing was carried out at normal atmospheric condition under a load of 50 kg and curing time was 36 hours. Dimension of the composite plate is 30 cm × 30 cm × 1 cm. Fiber volume fraction used is 0.4.

2.3 Samples preparation

Samples were prepared for different testing as per ASTM standards.

2.3.1 Impact test

For impact test, a V-notch of 45° ± 1°, root radius of 0.25 ± 0.05 mm, and a notch width of 2 mm was made. The specimens were, thereafter, sanded off using emery cloth of grade 220 in order to ensure that no out-of-plane notches were introduced onto the specimens. The impact test is a standardized high-strain rate test which is used to determine the amount of energy absorbed by a material during fracture. Sample dimensions for Izod and Charpy tests are 7.5 cm × 1 cm × 1 cm and 5 cm × 1 cm × 1 cm respectively. Figs. 3(a) and (b) show the samples for impact testings.

2.3.2 Tensile test

The tensile test specimen was prepared according to ASTM D3039; the most common specimen for ASTM D3039 has a constant rectangular cross section, 50 mm wide and 200 mm long. The specimen was mounted in the grips of the Instron universal tester with 10 mm gauge length.

2.3.3 Flexural test

The flexural test was using the 3-point bending method according to ASTM D790. The flexural test was conducted to study the behavior and ability of material under bending load. The load was applied to the specimen until it is totally broken.

Fig. 4 Tensile testing specimen

Fig. 5 Sample for flexural test

Fig. 6 Compression specimen

Fig. 7 Sample for thermal test

2.3.4 Compression test

The compression specimen is prepared as per the ASTM D6641 standard. A compression test involves mounting the specimen in a machine and subjecting it to the compression. The compression process involves placing the test specimen in the testing machine and applying the compress to it until it fractures.

2.3.5 TGA and DSC analysis

TGA (thermo-gravimetric analysis) and DSC studies were done on SFRC to study its thermal stability and the variations of the mass of composite with the change of temperature. Sample dimensions of the specimen for this test 15 cm × 5 cm × 1 cm.

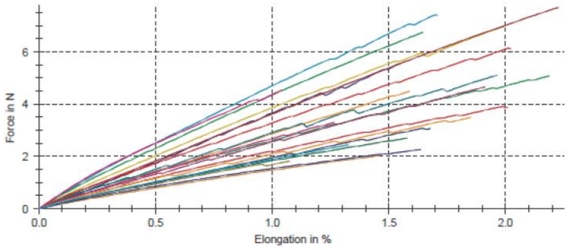


Fig. 8 Tensile strengths of individual fibres

3. Results and discussion

3.1 Individual fibre tensile testing

Individual fibres were tested for tensile strength. 30 fibres of uniform length and diameter were selected for the study and tensile testing was carried out as per ASTM D3822 and test speed was 10 mm/min. Grip to grip separation at the start position was 100 mm.

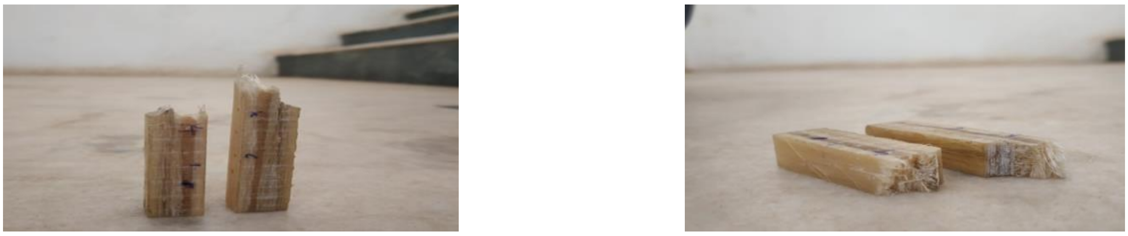
From Fig. 8 it is clear that the average maximum tensile modulus of individual fibre is 1.92 GPa and maximum elongation is observed to be 1.7%.

3.2 Impact testing

Impact testing was done for sisal fibre reinforced composites to assess its suitability for use in industrial applications. Charpy and Izod impact testing are conducted on the samples of bio-composites. Maximum stored energy observed to be 2 joules and 3 joules for Izod and Charpy test specimens respectively.

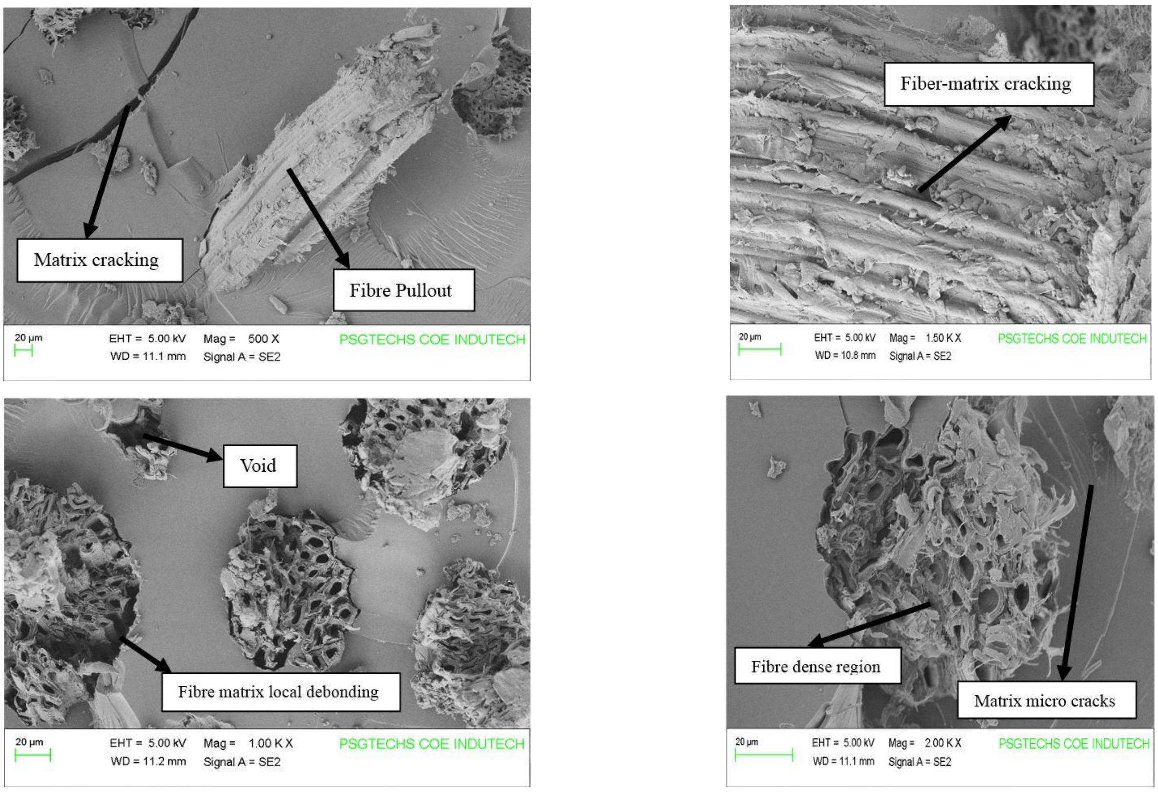
Microstructural evaluation of the fractured specimens is carried out using filed emission scanning electron microscopy (FE-SEM) technique. Results are presented in Figs. 10(a) and (b).

Results show that matrix undergoes micro-cracking on



(a) (b)

Fig. 9 (a) Fractured specimen of Izod Test; (b) Fractured specimen for Charpy Test



(a) (b)

Fig. 10 (a) SEM Images for Izod Test; (b) SEM Images for Charpy test

Table 1 Hardness test results for SFRC

S. No	**1**	**2**	**3**	**4**
Hardness	53	61	83	78

Table 2
(a) Compression test results for SFRC

σ_{low} **MPa**	σ_{high} **MPa**	L_{ocH} **mm**	E_c **GPa**	E_{Sec} **GPa**	σ_1 **MPa**
0.015	0.14	10.0	0.0185	0.0191	-

(b) Compression test results for SFRC

σ_2 **MPa**	σ_M **MPa**	€$_M$ **%**	€$_{cM}$ **%**
2.21	35.3	10	10

the surface while there is no visible sign of fibre matrix interface debonding. In Izod test fractured specimen suffers fibers pull out but there is no sign of fibre breakage. In fibre dense region there is the very minute presence of void observed.

3.3 Rockwell hardness testing of SFRC

Rockwell B scale (for composites) is set and 1/16 "diameter of steel ball intender under a load of 100 kgf is used to determine the Rockwell hardness of sisal fibre reinforced composites. All the specimens are maintained at a uniform orientation of 0°. Good bonding with the reinforcement and the epoxy which transmit the entire load to the strong and rigid fibers. Results are tabulated in Table 1.

Average hardness (HRB) is found to be 68.75.

3.4 Compression test

Compression test of the Sisal fibre reinforced composites is carried out as per ASTM D6641 using 100 kN universal tension machine. Tables 2(a) and (b) show the compression test results.

The specimen was subjected to a maximum stress of 0.14 MPa and it was observed that maximum compressive strain developed is 10%. Specimen maximum compressive strength is found to be 32 MPa.

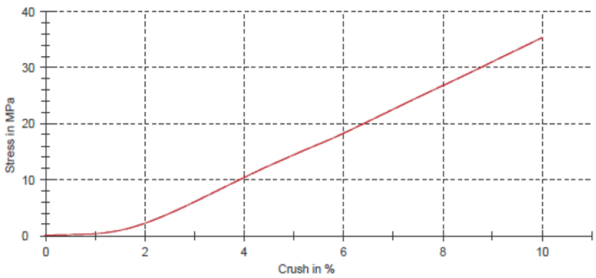


Fig. 11 Stress and crush variation for SFRC under compression

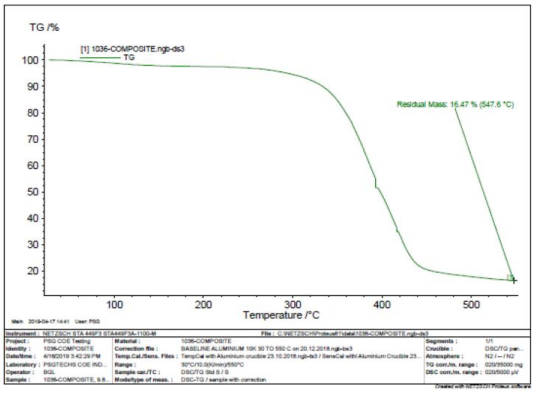


Fig. 12 TGA analysis of SFRC

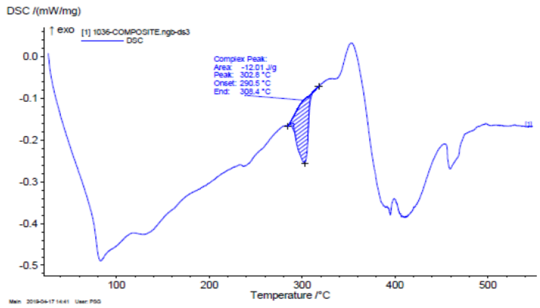


Fig. 13 DSC analysis of SFRC

From Fig. 11 it is clear that maximum compressive stress at failure is 35 Mpa and crush % is 10. In most of the regions stress and % crush remain linear and specimen undergoes elastic deformation.

Fig. 12 shows the TGA analysis of SFRC and it shows the mass lost with the temperature. Composite has shown the thermal stability up to the temperature of 600-degree C. SFRC undergoes degradation beginning at 330-degree C moisture is burnt. The maximum loss of mass occurs at or near 410-degree C and residual mass is found to be 16.4 % at 547.6-degree C.

Fig. 13 shows the differential scanning calorimetry (DSC) of sisal fibre reinforced composite. It is the technique that measures the difference in the heat flow to a sample and to a reference sample as a direct function of time or temperature under heating. Maximum thermal capacity is observed at or near 302 degrees C. the shaded area shows the complex peak.

3.6 Tensile testing of sisal fibre reinforced Composite

The tensile test is conducted on sisal fibre reinforced composite as per ASTM D3039 Standard using 10 kN Universal tensile testing machine. Results are tabulated in Table 3.

Table 3 Tensile test results for SFRC

E MPa	**Load N**	**Stress MPa**	**ε at failure %**	**A mm^2**
2070	11200	20.4	1.6	550.00

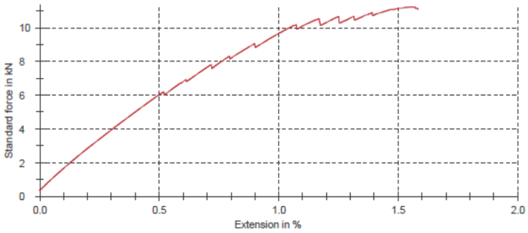


Fig. 14 Applied force vs strain curve of SFRC

From Fig. 14 it is clear that strain varies linearly with stress i.e., composite undergoes elastic deformation maximum stress is found to be 20.4 MPa and young modulus for the SFRC is 2070 MPa.

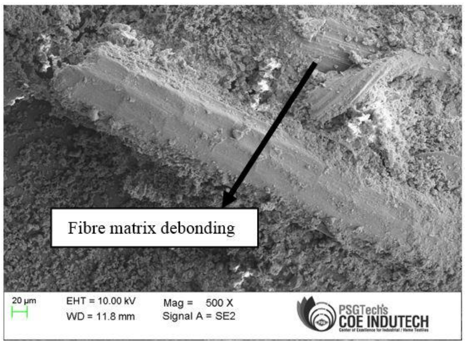
Micro-structural evaluation of fractured surface is carried to study the presence of void, interlayer failure and fibre interface debonding using FE- SEM technique. Figs. 15(a) and (b) presents the SEM images for the fractured surface of specimen for the tensile stress. When the composite specimen undergoes in tension in some regions fiber-matrix de-bonding is observed but there are no voids present in the surface.

Figs. 16(a) and (b) shows the edge view of fractured sisal fibre reinforced composite specimen. When specimen fails under tensile loads there is a small indication of fibre pullout on some local regions while in the rest of the place there is no visible sign of fiber-matrix debonding or failure.

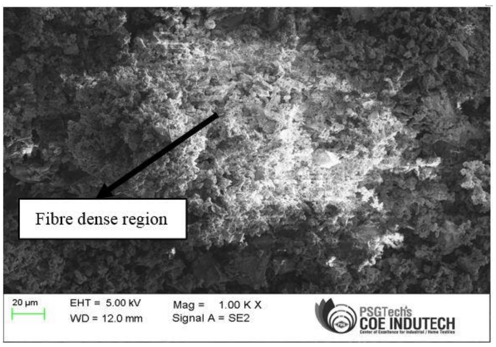
3.7 Flexural testing of SFRC

The flexural test was conducted on SFRC as per ASTM D790 Standard using 10 kN universal tensile testing machine. The tests are used to determine the flexural strength of a material. The testing material is laid horizontally onto the two points of contact and then a force is applied on the top of the material through either one or two points of contact until the specimen fails.

From Fig. 17 it is clear that strain varies linearly with stress and yield point stress is observed to be 82 MPa. Ultimate tensile stress is 90 Mpa and beyond this stress, failure occurs in the specimen. Strain at failure is observed to be 1.6%.

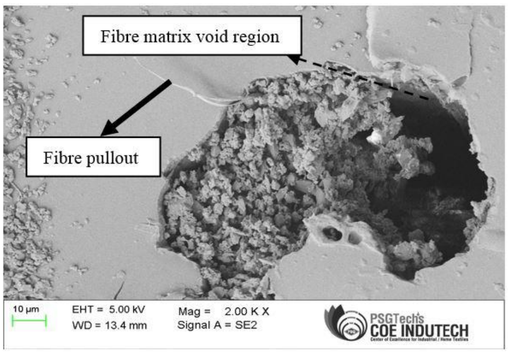


(a)

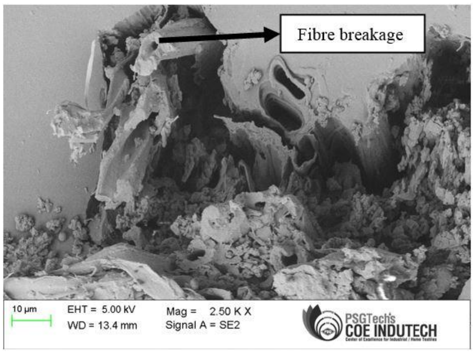


(b)

Fig. 15 (a) SEM image of the fractured surface; (b) SEM image of sectional view



(a)



(b)

Fig. 16 (a) SEM image of the fractured surface; (b) SEM image of an edge view

Table 4 Flexural test results for SFRC

S. No	E_H MPa	E_{Sec} Mpa	$S_{0.1}$ MPa	S_1 MPa	S_2 MPa	r_M MPa	$r_{M(Corr.)}$ Mpa	S_M MPa	r_{max} %	L Mm	d mm	b mm
1	2910	3870	81.8	37.6	76.6	2.5	2.5	90.1	3.3	51.2	10	50

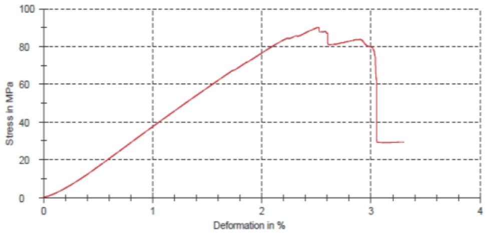


Fig. 17 Stress-strain curve of SFRC

4. Conclusions

In this study, the sisal fibre reinforced Bio-composite was tested to assess its suitability for possible applications in automobile and aerospace industries. The composite was subjected to mechanical testing such as Individual fibre testing followed by thermal analysis, tensile, hardness, flexural, and impact testing. Based on the results, the following conclusions are drawn:

- The impact strength is obtained for the sisal fiber composite and has the value of 3 joules.
- The Rockwell hardness test the strength is 68.75 HRB.
- The microstructure of the fiber is obtained in the breaking point of tensile and impact test of the specimen. To obtain the interfacial properties, internal cracks and internal structure of the fractured surfaces of the composite materials.
- Composite has shown the thermal stability up to the temperature of 600 degree C. SFRC undergoes degradation beginning at 330 degree C moisture is burnt.
- The maximum loss of mass occurs at or near 410 degree C and residual mass is found to be 16.4 % at 547.6 degree C.
- Maximum compressive stress at failure is 35 Mpa and crush % is 10.

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