

Mechanical, Thermal and Morphological Properties of Sisal Fibres

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Abstract. Natural fibres are gaining attention day by day in industrial applications. The aim of current investigation is to enhance the performance of sisal fibre by its surface modifications. The surface modifications include alkali treatment with varying concentrations (2, 5, 10, 15 & 20 %) and coating of PLA with varying concentrations (2, 4, 6, 8 & 10 %). Treated and untreated sisal fibres were subjected to tensile test and thermogravimetric analysis (TGA). In addition to this, sisal fibres were characterized by X-ray diffraction (XRD), and its structural morphology was studied by scanning electron microscope (SEM). Moreover, FTIR spectroscopy analysis was also carried out to see the removal of hemicelluloses and lignin from sisal fibre by alkali treatment. It was observed that surface modifications had much influenced the performance of sisal fibre. On other hand, PLA coated fibre had a better performance than alkali treated fibre.

1. Introduction

Natural fibres belong to environment-friendly materials which can be degraded completely in the soil. In addition, increased environmental consciousness and awareness has developed the increasing attention in natural fibres and its application in various fields [1]. The advantages of natural fibres are recyclability, high strength to weight ratio, non-carcinogenic, easy separation, abundantly available, renewability, biodegradability, and have no health hazard [2-10]. They have been used for industrial applications such as textiles, paper products, absorbents, packaging and building materials [11-21]. Among natural fibres, sisal fibres have been frequently used owing to its high strength and modulus. This fibre is mostly used for ropes, strings, bag fabrics, carpets, mats and fishnets [22]. It is hydrophilic in nature due to extraction from lignocelluloses as it contains strongly polarized hydroxyl groups. The major problem of sisal fibre is impurities as natural oils and wax. Poor bonding between polar hydrophilic fibre and non-polar hydrophobic matrix is critical limitation of sisal fibre to be used as reinforcement in polymer composite [22].

PLA is one of the strongest candidates among biopolymers due to various advantages like high strength and modulus, thermal plasticity, biocompatibility and complete biodegradability [23]. PLA is mainly synthesised by two process; (i) ring-opening polymerization of lactide and (ii) direct



poly condensation of lactic acid [24]. Though PLA has various advantages but it is brittle in nature which restricts its application in many areas.

For better applications of sisal fibres, its properties were tried to improve by alkali treatment and using a new technique as PLA coating in present work. Treated and untreated sisal fibres were subjected to tensile test and characterizations include TGA, SEM, XRD and FTIR.

2. Materials and method

2.1. Materials

Sisal fibres were obtained from Uttarakhand Bamboo and Fibre Development Board, Dehradun, Uttarakhand, India. Epoxy and PLA were purchased from the local resources.

2.2 Surface modification

2.2.1. Alkalization

Sisal fibres were treated with NaOH aqueous solution having different concentrations i.e. 2%, 5%, 10%, 15% and 20% for 30 min at room temperature.

Nomenclature

SF Untreated sisal fibre

SF (T1) NaOH treated sisal fibre

SF (T2) 2% NaOH treated sisal fibre coated with PLA

2.2.2. PLA coating on sisal fibre

Initially, PLA solution was prepared by pouring PLA pellets into chloroform and then mechanical stirrer was used for dissolving homogeneously. Sisal fibres were immersed in prepared solutions having various concentrations i.e. 2%, 4%, 6% and 8% for 5 min, thereafter, dried in open air at room temperature. Finally, PLA coated sisal fibres were dried in hot air oven for 3 hrs at 50°C.

Table1. Properties and chemical composition of sisal fibre [22]

Properties	Sisal fibre
Density (g/m ³)	1.45
Tensile strength (MPa)	126-860
Tensile modulus (GPa)	7-13
Elongation at break (%)	1.54-3.85
Cellulose (%)	65-78
Hemicellulose (%)	10-14
Wax (%)	2
Lignin (%)	9.9
Pectin (%)	10
Moisture content (%)	10
Ash (%)	0.6-1

2.3. Fourier transforms infrared spectroscopy (FTIR)

FTIR was carried out to study chemical structure of fibres (in powder form) using instrument, FTIR Spectrum 2, Perkin Elmer (Figure 1, b). It is the most extensively used characterization technique to find out the effect of surface modification on sisal fibre. IR spectra were recorded in the spectral range of $400\text{--}4,000\text{ cm}^{-1}$ with a resolution of 4 cm^{-1} .

2.4. Tensile test

The tensile test of sisal fibre was carried out as per ASTM D3039. Ten fibres of each untreated, treated and alkali treated sisal fibres coated with PLA, with gauge lengths of 50 mm and average diameter of $150\text{--}190\text{ }\mu\text{m}$ were tested using Tinius Olsen H 10 K-L machine (Figure 1, a) with a cross head speed of 1 mm/min .

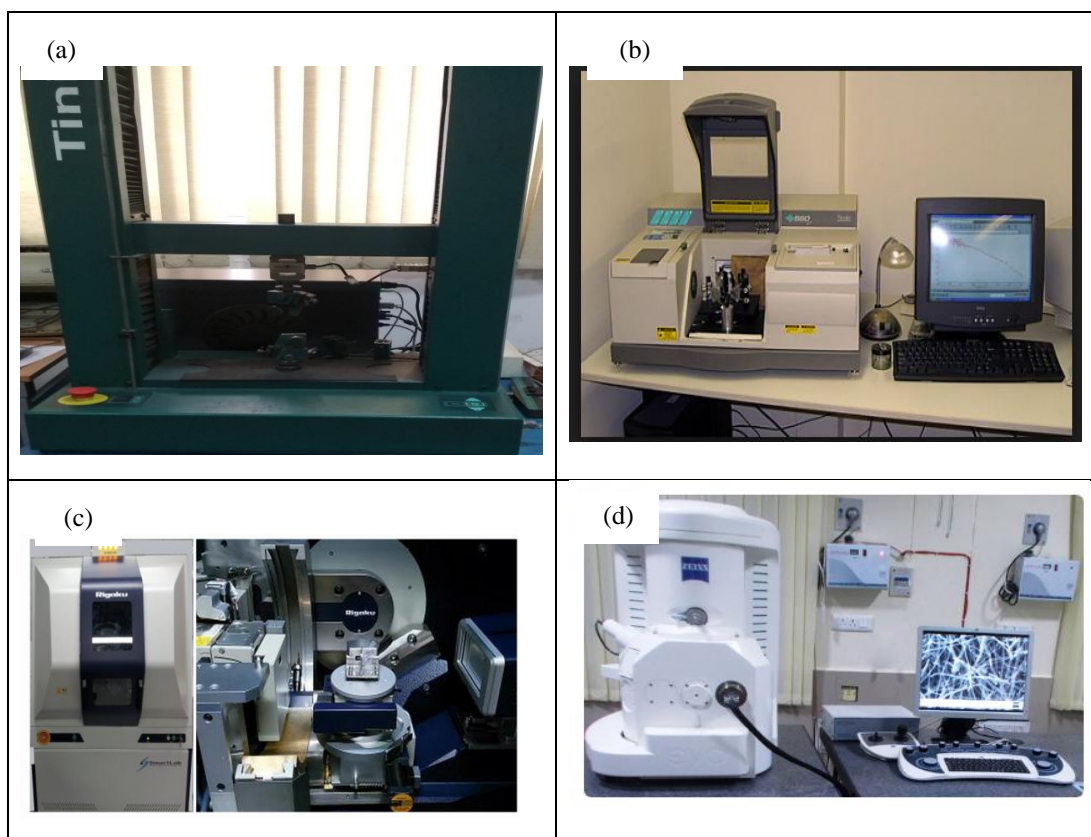


Figure 1. Pictures of instruments: (a) Tinius Olsen H 10 K-L machine, (b) FTIR Spectrum 2 (Perkin Elmer), (c) XRD Smart lab 3KW model, (d) SEM Carl Zeiss EVO 50.

2.5 Thermogravimetric analysis (TGA)

Thermogravimetric analysis was performed using the instrument, Perkin Elmeris, STA 6000. Fibres having weight of 25 mg were heated from 25°C to 600°C at heating of 10°C/min .

2.6 X-Ray Diffraction (XRD)

X-ray diffraction was done to decide crystallinity of sisal fibre using a Smart lab 3KW model, employing $\text{CuK}\alpha$ ($\lambda = 1.54$) radiation. The diffraction intensity was in the range of $5^{\circ}\text{--}60^{\circ}$ of 2θ with scanning speed of $0.02^{\circ}/\text{s}$.

The Crystallinity indexes (C.I) and Percentage Crystallinity (%Cr) of fibres were calculated by following equations[25].

$$\%Cr = \frac{I_{22}}{I_{22}+I_{am}} \quad (I)$$

$$C.I = \frac{I_{22}-I_{am}}{I_{22}} \quad (II)$$

Where ' I_{22} ' shows maximum intensity of diffraction at 2θ angle (22.38° - 22.62°) and ' I_{am} ' indicates intensity of diffraction of the amorphous material at 2θ angle (15.98° - 16.62°).

2.7. Scanning electron microscopy (SEM)

Morphological analysis of untreated, alkali treated and alkali treated sisal fibre coated with PLA was observed by instrument, SEM carlzeiss EVO 50 (Figure 1, d), after gold coating on them.

3. Results and discussion

3.1. Mechanical properties

Tensile properties of untreated and alkali treated sisal fibres are given in Table 2. Tensile strength and modulus of sisal fibre were found to be maximum at 2% alkali and beyond it decreasing trend was found. Increase in the tensile strength and modulus of 2% alkali treated sisal fibre is mainly because of removal of hemicellulose which causes close packing of cellulose fibrils by forming strong hydrogen bond [26]. On increasing the concentrations of NaOH above 2%, tensile properties were found to decrease due to delignification and degradation of cellulose chains [27]. After getting maximum tensile properties at 2% alkali treatment, PLA coating was done on treated fibres with various concentrations. Tensile properties in terms of tensile strength, tensile modulus and percentage elongation at break of 2 % alkali treated sisal fibre with PLA coating at various concentrations are provided in Table 3. It was observed that sisal fibre with 2% PLA coating has better tensile properties than those of other fibres. Tensile properties of sisal fibre were increased by 2% PLA coating due to increase in stiffness of fibres. On other hand, it was found that tensile properties decrease with increase in concentration of PLA due to increase in thickness of coating thereby increase in brittleness of fibres.

Table 2.Effect of alkali treatment on tensile properties of sisal fibres

S.No.	Concentration of NaOH (%)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at break (%)
1	0	303.00	3.578	2.78
2	2	427.32	3.789	8.60
3	5	313.00	2.957	12.57
4	10	270.26	2.296	9.78
5	15	217.80	1.725	10.23
6	20	196.84	1.626	11.66

Table 3.Effect of PLA coating on tensile properties of 2% alkali treated sisal fibres

S. No.	Concentration of PLA (%)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at break (%)
1	0	303.00	3.578	2.78
2	2	447.84	4.531	9.28
3	4	300.70	3.399	8.29
4	6	269.21	2.989	7.79
5	8	258.25	2.732	7.87

3.2. FTIR spectroscopy

The FTIR spectrum of the untreated, treated, and treated and coated sisal fibres are shown in Figure 2. Comparison in results of FTIR spectral data of SF, SF(T1) and SF(T2) are tabulated in Table 4. The broad band at 2918.57 cm^{-1} was assigned to C-H symmetrical stretching. The peak at 3304.13 cm^{-1} represents axial stretching of hydroxyl group ($-\text{OH}$) leads to hydrocarbon constituents and correspond to H bonded O-H stretching, shifted to 3310.08 for alkali treated showing participation of free hydroxyl group in reactions. The peak at 1233.51 cm^{-1} is attributed to C-O stretch of the acetyl group of lignin. A peak at 1736 cm^{-1} is corresponding to the C=O bonds stretching of carboxyl & acetyl group of hemicellulose shifted to 1749.47 for SF (T2). Difference in intensity is observed may be due to removal of acetyl group from hemicellulose. The peak at 1610.50 cm^{-1} signifies to the C=C bonds in hemicelluloses, the drop in intensity is largely due to partial removal of hemicelluloses from treated and coated fibre surface. The peak 1423.27 shows presence of CH_3 deformation in lignin and 1024.90 cm^{-1} is assigned to aromatic C-H in plane deformation and C-O deformation for primary alcohol in lignin.

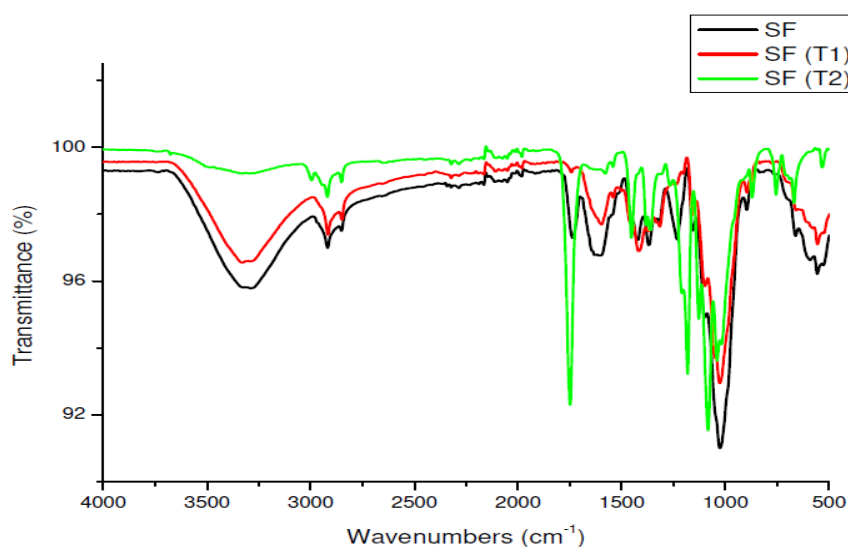
**Figure 2.**FTIR spectra of untreated, treated, and treated and coated sisal fibres

Table 4. FTIR Spectral data of untreated, treated, and treated and coated sisal fibres

Wave no (cm ⁻¹)			Possible assignments
SF	SF (T1)	SF (T2)	
3304.01	3310.08	-	O-H stretching
2918.57	2916.75	2919.91	C-H stretching
1736.56	-	1749.47	Carboxylic acid C=O Stretching.
1610.15	1600.30	-	Alkene C=C
1423.27	1416.01	1452.85	CH ₃ deformation
1024.90	1028.11	1039.67	C-H ,C-O deformation

3.3. Thermogravimetric analysis (TGA)

Percentage weight loss of untreated, treated, and treated and coated sisal fibres was found from TGA is presented in Figure 3. Thermal stability of natural fibres is mainly influenced by chemical constituents i.e. cellulose, hemicellulose and lignin presented in the fibres. The TGA curves of sisal fibres show three steps degradation in the ranges of around 100°C, between 200 - 400°C and 400-550°C due to decomposition of lignin, hemicellulose, and cellulose respectively. Lignin decomposed at a slower rate compared to the other constituents of sisal fibre [28]. Treated fibre is more thermally stable than untreated fibre, as shown in Table 5. The first step degradation up to 100°C may be related to moisture removal from fibre surface. [29]. A weight loss of 4.35-6.54% occurs due to loss of moisture existing in the fibres. It is observed that alkali treated sisal fibres reveals smaller weight loss as compare to SF and SF (T2). Sisal fibre, which is highly hydrophilic in nature, is treated with alkali leads to partial removal of hemicellulose and this hemicellulose is responsible for water absorption in sisal fibres.

At temperature of 219-249°C, second step of degradation starts because of breakdown of hemicellulose. The thermal decomposition of treated & PLA coated sisal fibre is completed at 473°C & 460°C respectively. It might be owing to PLA coating as protective barrier which protect the fibres from the degradation. However, it is shifted to 523°C for SF (T2), 560°C for alkali treated fibre. Over 400°C, degradation occurs as result of breakup of the protolignin bonds. It is found that some of the cementing material is removed by alkali treatment of sisal fibre followed by the removal of moisture, results more stability of fibre than untreated fibre. At final step of degradation, it was noted that SF (T1) show lowest weight loss with respect to SF and SF (T2) as shown in Table 5 which indicate its greater thermal stability.

Table 5. Weight loss of untreated, treated, and treated and coated sisal fibres with respect to temperature

Temperature (°C)	SF	SF (T1)	SF (T2)
100	6.23	4.35	5.75
200	7.05	8.05	9.14
300	24.26	35.91	37.37
400	72.48	63.94	66.16
500	97.05	70.31	80.67

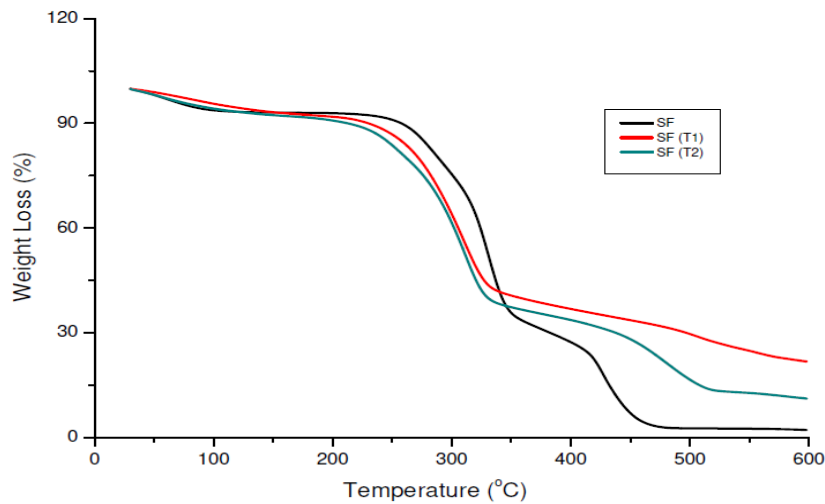


Figure 3. TGA graph of untreated, treated, and treated and coated sisal fibres

3.4. X-Ray Diffraction (XRD)

Crystallographic structure of sisal fibres was analysed by X-ray diffraction using $\text{CuK}\alpha$ ($\lambda = 1.54$) radiation and a graphite monochromator having diffraction intensity in the range of 5 to 60°. The X-ray graph of untreated, treated, and treated and coated sisal fibres are present in Fig 4. Fibres mainly consist of cellulose, hemicellulose, and lignin. Cellulose mainly comprises of amorphous and crystalline structures, however lignin and hemicellulose are amorphous. XRD data reveals that the major crystalline peak obtained at around 2θ value varies from 22.38°-22.62°, whereas the amorphous peak found at 2θ value between 15.98°-16.62° as shown in Table 6. Formation of new hydrogen bonds between cellulose is improved the crystallinity index and crystalline percentage of sisal fibre by alkali treatment [30]. The lignin part is amorphous in nature and the cellulose is crystalline. The removal of noncellulosic parts makes the fibre more crystalline and it is revealed from XRD pattern.

Table 6. Variation of crystalline index and crystalline percentage of untreated, treated, and treated and coated sisal fibres

Fibre treatment	Maximum Intensity I_{002}	Angle (2θ) at I_{002}	Maximum Intensity I_{am}	Angle (2θ) at I_{am}	Crystallinity Index (CI)	Percentage of crystallinity
SF	4085	22.42	2246	15.98	0.45	64.52
SF (T1)	5814	22.38	2688	16.22	0.53	68.30
SF (T2)	3187	22.42	1887	16.62	0.40	62.88

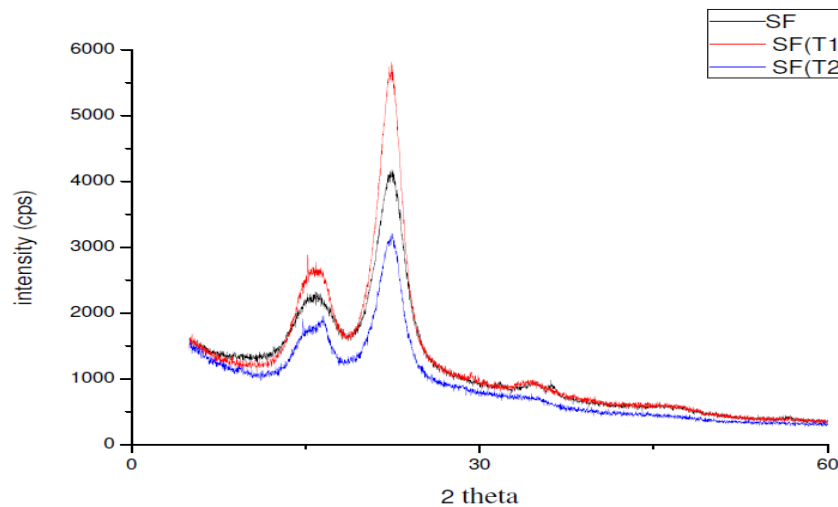


Figure 4. XRD analysis of untreated, treated, and treated and coated sisal fibres

3.5. SEM

The SEM images indicate the differences in the surface morphologies of SF, SF (T1) and SF (T2) as shown in Figure 5 (a-c). The surface of untreated fibre appeared rough due to the presence of non-cellulosic components like lignin, wax, oil, and surface impurities (Figure 5a), which are partially removed by alkali treatment (Figure 5b) and further removed by PLA coating (Figure 5c). Removal of wax and oil from the surface of treated fibre has been observed which shows enhanced compatibility of fibre and matrices. SEM micrographs show elimination of non-cellulosic material from the fibre surface due to mercerization and therefore an improvement in the wet ability between fibre and matrix occurs.

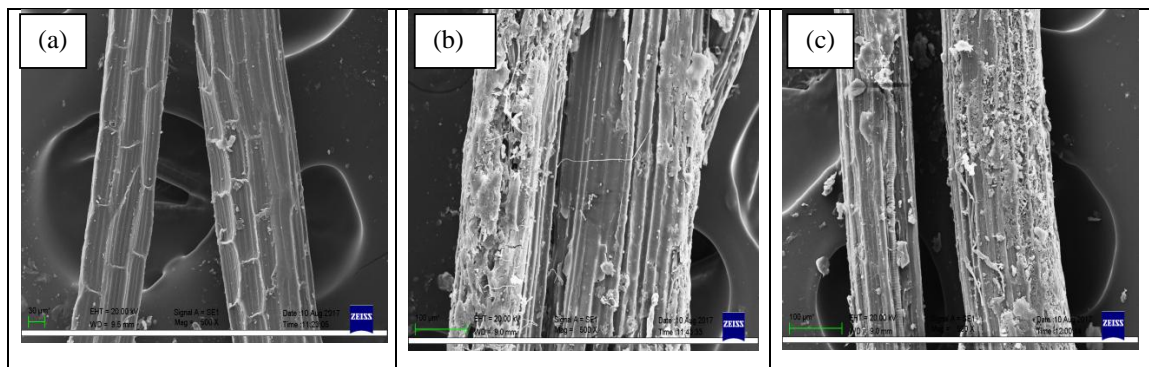


Figure 5. SEM micrographs of untreated, treated, and treated and coated sisal fibres

4. Conclusions

- Sisal fibre was effectively coated with PLA bio polymer.
- There was an increase in tensile properties of sisal fibre by alkali treatment up to 2%, and then decrease.
- TGA revealed that alkali treated sisal fibres were thermally more stable than untreated fibre.
- Treated and coated fibres offered overall better performance.

- It can be proposed that PLA coating on alkali treated sisal fibre could be an effective way to improve the performance of sisal fibre.

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