

## EFFECT OF ALKALI AND SILANE TREATMENT ON WATER ABSORPTION AND MECHANICAL PROPERTIES OF SISAL FIBER REINFORCED POLYESTER COMPOSITES

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### Abstract

The present work deals with the effect of water absorption on the mechanical properties of untreated, 10% alkali-treated, and 10% alkali plus 1% silane treated sisal fibers (5%, 10%, and 15%) reinforced polyester composites. Hand lay-up was used to create the composite. The samples were prepared in accordance with ASTM standards, and tests for tensile strength, flexural strength, impact strength, and water absorption were performed. An increase in the tensile, flexural and impact strength was observed with an increase in fibre loading for untreated, alkali-treated and alkali plus silane treated sisal fibre reinforced polyester composites without water absorption, the increase being maximum for 10% alkali plus 1% silane treated fibre composite. Water absorption reduces tensile strength while increasing flexural and impact strength in untreated sisal fiber reinforced composites. There is an increase in tensile, flexural, and impact strength with higher fiber loading for 10% alkali-treated and 10% alkali-treated plus 1% silane treated sisal fiber reinforced polyester composites with and without water absorption. The tensile, flexural, and impact strength of alkali plus silane treated fiber is maximum at any given fiber loading, indicating that the alkali plus silane treatment is effective in improving the fiber matrix interface. Water absorption increases with fiber loading in untreated, 10% alkali-treated, and 10% alkali plus 1% silane treated sisal fiber reinforced polyester composites, with the rate being lowest in alkali plus silane treated fiber reinforced composites.

**Keywords:** SISAL fiber; polyester; silane; alkali treatment; water absorption; reinforced composites.

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## Nomenclature

DS	Dry Sample
WS	Wet Sample
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscope

## Introduction

Natural fiber composites are a viable option for addressing global environmental issues [1]. Natural fiber composites have several advantages, including low cost, environmental friendliness, low density, and high strength [2,3]. Automobile manufacturers are turning to natural fiber composites to improve fuel efficiency and reduce weight [4,5]. Natural fiber composites range from 9% to 59% in the automobile and construction industries [6]. The interfacial adhesion between fiber and matrix is critical to the performance of natural fiber composites [7]. The addition of hyacinth fiber to an epoxy matrix improved mechanical properties [8]. The tensile and flexural strength of a graphite/pineapple leaf fiber epoxy composite improved [9].

The main challenge to overcome in order to improve the performance of natural fiber composites is poor compatibility and high water absorption. The effect of different chemical treatments on the performance of natural fiber composites has been reported [10].

Alkali treatment of the surface roughness improves interaction with the matrix [11]. Silane coupling agents improve the interfacial adhesion between the fiber and the matrix, resulting in improved mechanical properties [12,13]. On wheat straw reinforced polystyrene composites, the effect of various chemical treatments such as alkali, silane, maleic anhydride, and alkali plus silane was reported, with the alkali plus silane treatment being found to be the most suitable [14].

Water absorption in natural fiber composites degrades the mechanical properties of the composites [15,16]. Water absorption on the fiber surface, due to its hydrophilic nature, deteriorates the fiber matrix interface [17]. When compared to silane treatment, alkali treatment results in greater water absorption [18].

There is an increase in the mechanical properties of sisal fibre reinforced polymer composite [19, 20]. Alkali treatment of sisal fiber improved the mechanical properties of sisal fiber reinforced polyester composite [21]. The hydrophilicity of sisal fiber is reduced by chemical treatment [22]. Tensile strength and tensile modulus have been reported to decrease for sisal fiber reinforced polypropylene composites with water absorption [23].

## Material and Methods

### *Materials*

UVAS solutions private Ltd provided the unsaturated polyester (matrix). As a reinforcing fiber, sisal fiber is used. Arihanta Metallica provided the cobalt octate used as an activator. Veekay Chemicals supplied the MEKP (methyl ethyl ketone peroxide) used as a catalyst. Evonik Industries supplied the silane used as a coupling agent.

### *Treatment of Fiber*

Two different types of treatment were carried out. In one treatment fibers were treated with 10% NaOH and in another treatment, fibers were treated with 10% NaOH plus 1% silane.

#### *Alkali Treatment (10 % NaOH)*

Sisal fibers were cut into 4 to 6 mm lengths. Distilled water was used to make a 10% NaOH solution. For 24 hours, the fibers were immersed in the solution. The fibers were washed and dried for two days using distilled water. By incorporating these completely dry fibers, sisal fiber reinforced polyester composites were created.

#### *Silane Treatment (10% NaOH plus 1% Silane)*

Sisal fibers were cut into 4 to 6 mm lengths. Distilled water was used to make a 10% NaOH solution. For 24 hours, the fibers were immersed in the solution. The fibers were washed and dried for two days using distilled water. Acetone was used to make a 1% silane solution with a pH of 4. For 24 hours, completely dried sisal fibers were immersed in the solution. After 24 hours, the fibers were removed from the solution and dried. By incorporating these completely dry fibers, sisal fiber reinforced polyester composites were created.

### **Processing (Experimental)**

The Hand Lay Up method was used to create sisal fiber reinforced polyester composites. To prevent polymer from sticking to the mold surface, a release gel is applied. The unsaturated polyester (UPR) is thoroughly mixed with the catalyst (4% methyl ethyl ketone peroxide) and activator (6% cobalt octate). Chopped fiber (approximately 4-6 mm length) was placed on the mould surface, and the mixed resin was poured on top. The polymer is applied evenly with the brush and roller. The composite was made with different percentages of fiber (5%, 10%, and 15%). A roller is used on the fiber-polymer layer to remove any trapped air as well as any excess polymer. The procedure is repeated for each polymer and fiber loading. After curing for 30 minutes at room temperature, the composite parts were removed. Figure 1 depicts the test specimens prepared in accordance with ASTM standards for tensile, flexural, and water absorption. Three samples were taken on average. The samples were made wet by immersing them in water for 24 hours.



Fig. 1. Matrix and Sisal fiber reinforced polyester composites samples.

## Results and Discussion

### Tensile Strength

Fig. 2 depicts the change in tensile strength of sisal fiber reinforced polyester composites with and without water absorption for untreated fiber (Ref. Table 1), 10% alkali-treated fiber (Ref. Table 2), and 10% alkali and 1% silane treated fiber (Ref. Table 3). Tensile strength increased with fiber loading for untreated, 10% alkali-treated, and 10% alkali and 1% silane treated sisal fiber reinforced composites with no water absorption. When compared to untreated fiber reinforced composites, the rise is greater with alkali treatment. The removal of the waxy layer and improvement in surface roughness, resulting in greater fiber-matrix adhesion, can be attributed to an increase in the tensile strength of alkali-treated sisal fiber reinforced polyester composites [24,25]. The increase in tensile strength is greatest in alkali and silane treated fiber reinforced composites with no water absorption. Alkali treatment improves fiber matrix compatibility by increasing the efficiency of the silane coupling agent, resulting in improved matrix/fiber interaction [26]. Tensile strength declines with increasing fiber loading for untreated sisal fiber reinforced polyester composites with water absorption; the decline is greater at 15% fiber loading. Water absorption increases due to an increase in fiber loading due to the hydrophilic nature of the fiber. Water absorption causes swelling, plasticization [16,27], and weakening of the fiber/matrix interface [28,29], which reduces mechanical properties [30].

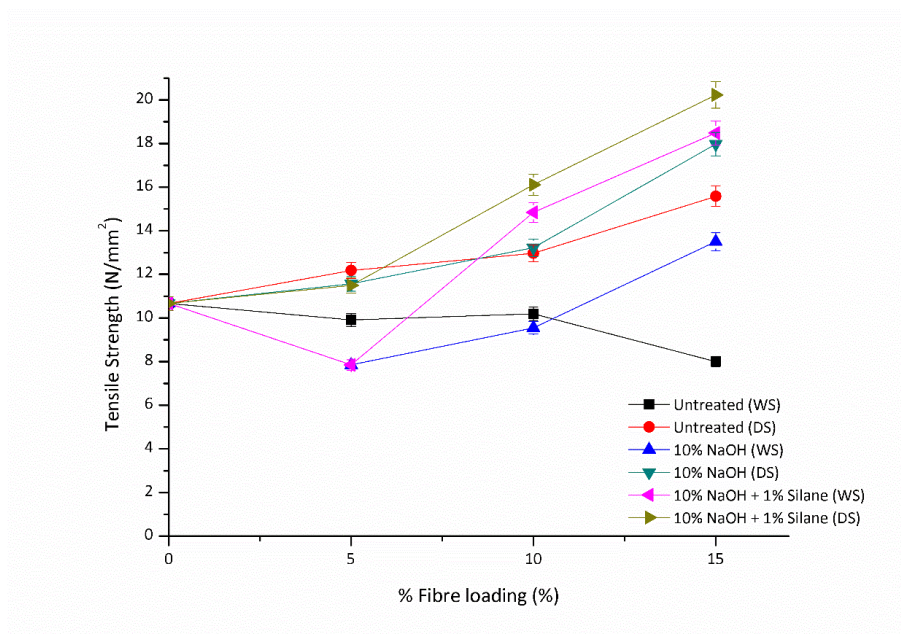


Fig. 2. Tensile strength of sisal fiber reinforced polyester composites with percentage variation in fiber loading for untreated, 10% alkali treated and 10% alkali plus 1% silane treated fiber reinforced composites with and without water absorption.

Tensile strength decreases at 5% fiber loading in an alkali-treated sisal fiber reinforced polyester composite with water absorption, followed by a rise in tensile strength as fiber loading increases. The initial decrease in tensile strength for alkali-treated and alkali plus silane treated sisal fiber reinforced polyester composites at 5% fiber loading can be attributed to the predominating effect of water absorption in reducing mechanical properties versus reinforcing effects of fibers in enhancing mechanical properties. Tensile strength increased with increased fiber loading, with the increase being greater in the case of alkali plus silane treated fiber, indicating a dominating effect of fiber reinforcement in overcoming the decrease in tensile strength caused by water absorption on the mechanical properties.

### Flexural Strength

Fig. 3 depicts the change in flexural strength of sisal fiber reinforced polyester composites with and without water absorption as a function of the percentage change in fiber loading for untreated (Ref. Table 1), 10% alkali-treated fiber (Ref. Table 2), and 10% alkali plus 1% silane treated fiber (Ref. Table 3). Flexural strength increased with fiber loading for untreated, alkali-treated, and alkali plus silane treated fiber reinforced composites without water absorption; however, the increase is greatest for alkali plus silane treated fiber reinforced composites. Again, the rate of increase is greatest for sisal reinforced polyester composites treated with alkali plus silane.

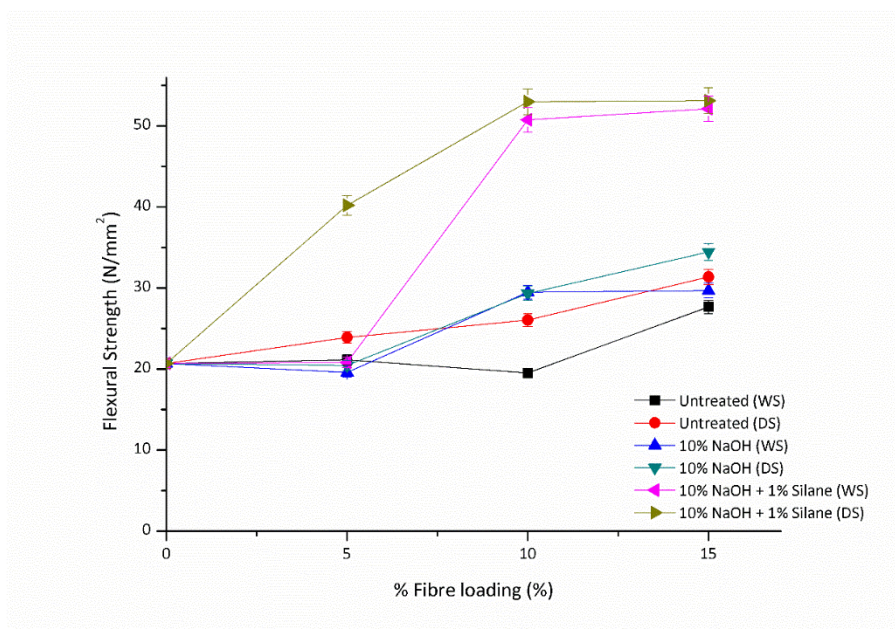


Fig. 3. Flexural strength of sisal fiber reinforced polyester composites with percentage variation in fiber loading for untreated, 10% alkali treated and 10% alkali plus 1% silane treated fiber reinforced composites with and without water absorption.

Chemical treatment of natural fibers improves interfacial adhesion and porosity of composites, preventing debonding, detachment, or fiber pull out [31]. The increase in flexural strength for alkali-treated fiber reinforced composites can be attributed to the fact that alkali-treated fiber becomes rougher and more fibrillated than untreated fiber, resulting in a better fiber matrix interface [32]. The increase in flexural strength is greatest for alkali plus silane treated fiber because silane and alkali provide a higher aspect ratio, resulting in a better fiber matrix interface [33]. There is a slight reduction in flexural strength up to 10% for untreated sisal fiber reinforced polyester composites with water absorption, followed by an increase in it at 15% fibre loading. The initial decrease in flexural strength following water absorption can be attributed to a weak fiber matrix interface as a result of water absorption [34]. Flexural strength for 10% alkali-treated sisal fiber reinforced with water absorption initially decreases up to 5% fiber loading, then increases up to 10%, remaining stagnant at higher loading up to 15%. Flexural strength remains nearly constant up to 5% fiber loading, then increases with 10% and 15% fiber loading for alkali plus silane treated sisal fiber reinforced composites. The initial stagnation in flexural strength can be attributed to water absorption weakening the fiber matrix interface and counterbalancing the reinforcing effect of fiber. The combination of alkali and silane treatment improves fiber matrix compatibility.

### Impact Strength

The variation in impact strength of untreated (Ref. Table 1), alkali treated (Ref. Table 2), and alkali plus silane treated (Ref. Table 3) sisal fiber reinforced polyester composites with and without water absorption is shown in Fig. 4. Impact strength increases with increasing fiber loading for untreated sisal fiber reinforced polyester composites without water absorption up to 10% fiber loading, remaining stagnant. The impact strength of alkali-treated sisal fiber reinforced polyester composite without water absorption increases with increasing fiber loading; the rate of increase is higher at higher fiber loading. The impact strength of untreated and alkali-treated sisal fiber reinforced polyester composites with water absorption increases with fiber loading. The plasticization of the sisal fiber and polyester matrix interface can be attributed to the higher impact strength of untreated sisal fiber reinforced polyester composite with water absorption compared to without water absorption [23].

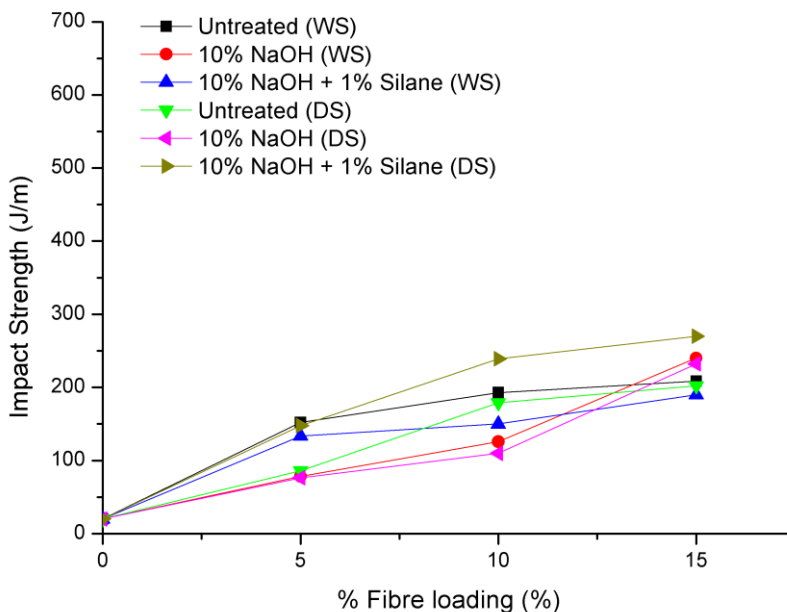


Fig. 4. Impact strength of sisal fiber reinforced polyester composites with % variation in fiber loading for untreated, 10% alkali treated and 10% alkali plus 1% silane treated fiber reinforced composites with and without water absorption.



The impact strength of alkali plus silane treated sisal fiber reinforced polyester composite with and without water absorption increases with increasing fiber loading, with the rate of increase being faster at higher fiber loading. At any given fiber loading, the increase in impact strength is greater for alkali plus silane treated sisal fiber reinforced polyester composites, indicating its effectiveness in improving the mechanical properties of the sisal fiber reinforced polyester composites.

*Water absorption*

The change in water absorption for untreated (Ref. Table 4), 10% alkali-treated (Ref. Table 5) and 10% alkali plus silane-treated (Ref. Table 6) sisal fiber reinforced polyester composites is shown in Fig. 5. Water absorption increased as the percentage of fiber loading increased; however, water absorption is lowest for alkali plus silane treatment and highest for alkali treatment. Higher diffusion coefficients of samples with higher fiber loading due to higher cellulose content [35] and an increase in diffusion transport of water due to fiber swelling and microcracks [36] can be attributed to an increase in water absorption.

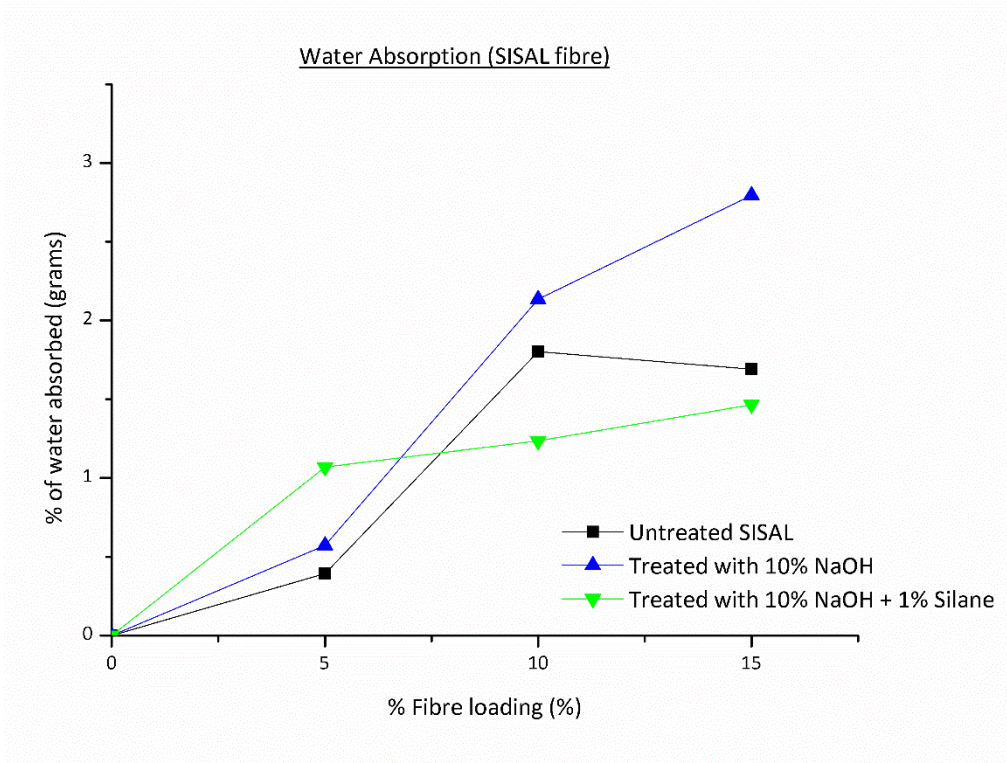


Fig. 5. Water absorption for untreated, 10% alkali treated and 10% alkali plus silane treated sisal fiber reinforced polyester composites.



### SEM Micrographs

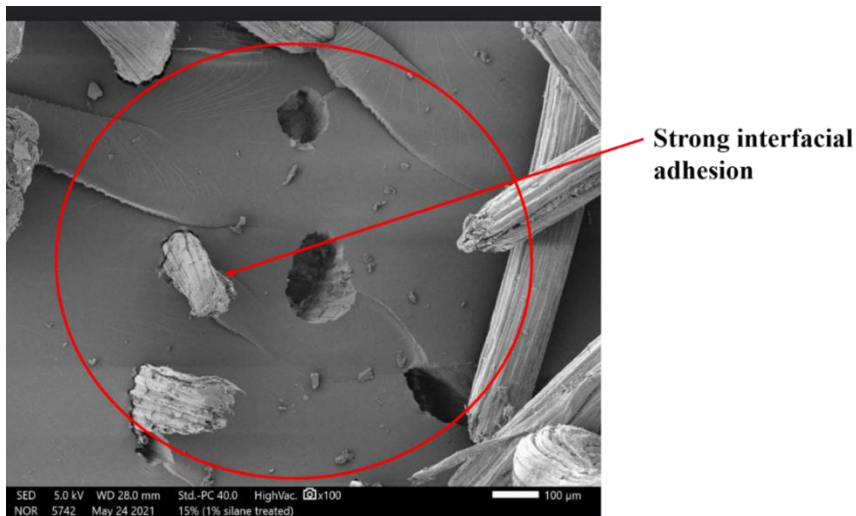


Fig. 6. SEM for alkali plus silane treated sisal fiber (15%) reinforced polyester composites.

SEM for alkali plus silane treated sisal fiber reinforced polyester composites with 15% fiber loading is shown in Fig. 6. Figure 5 shows that there is strong interfacial adhesion between the sisal fiber matrix because the fiber is clearly attached to the matrix after loading and there are no voids or debonding on the surface of the composite, resulting in higher mechanical strength. It is possible to conclude that alkali plus silane treatment improves fiber matrix bonding.

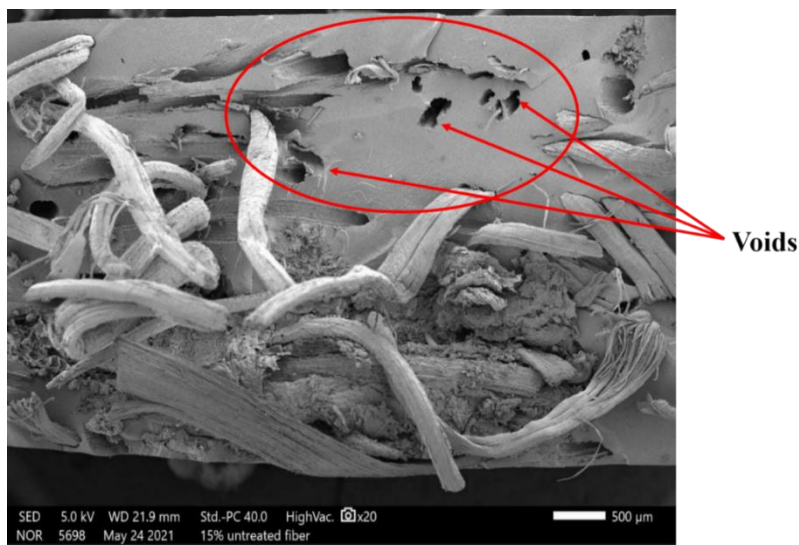


Fig. 7. SEM for untreated sisal fiber (15%) reinforced polyester composites.

SEM for untreated sisal fiber reinforced polyester composites with 15% fiber loading is shown in Fig. 7. Poor interfacial adhesion is observed, indicating that the fiber is separated from the matrix, leaving voids on the surface and resulting in lower mechanical strength when compared to treated sisal fiber reinforced composites.

#### FTIR Spectrum

The treated and untreated samples were subjected to FTIR analysis (Ref. Table 7). Figures 8-10 depict the absorption bands. The untreated sample's composite spectrum contains hemicellulose, cellulose, lignin, and an inorganic component. As shown in Table 1A, the broad absorption band at  $3739.14\text{ cm}^{-1}$  is caused by O-H stretching of -OH groups of hemicellulose, cellulose, and lignin in sisal fiber (refer supplementary information). The C=O stretching of -COOH/ ester groups is assigned to the bands at  $1696.57$  and  $1740.84\text{ cm}^{-1}$ , respectively. The polyester resin contains both carbonyl and ester. The absorption band at  $1520.43\text{ cm}^{-1}$  represents skeletal vibration (C=C) of the benzene ring of UPR, whereas the band at  $780.34\text{ cm}^{-1}$  is due to hemicellulose -CH<sub>2</sub> & -CH bending.

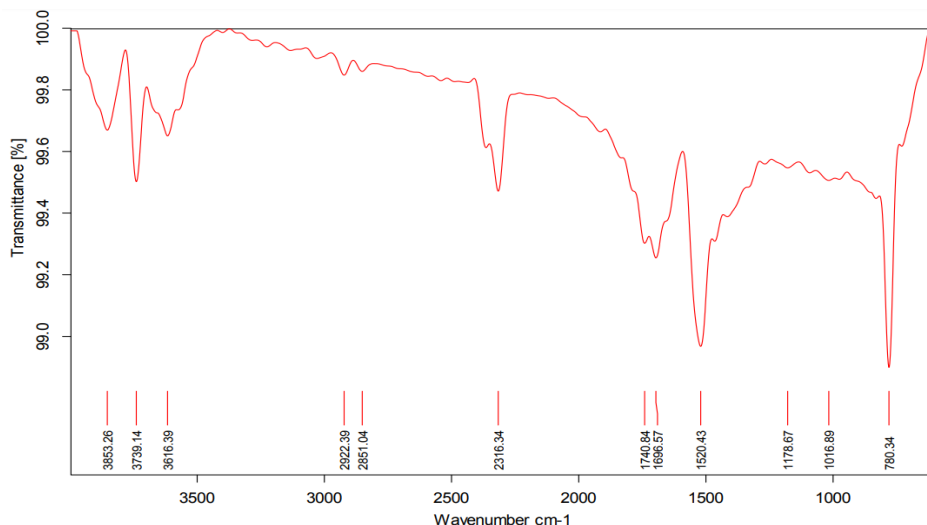


Fig. 8. FTIR Untreated SISAL fiber reinforced polyester composite (15% fiber).



Fig. 9. FTIR 10 % alkali treated reinforced sisal fiber reinforced polyester composite (15% fiber).

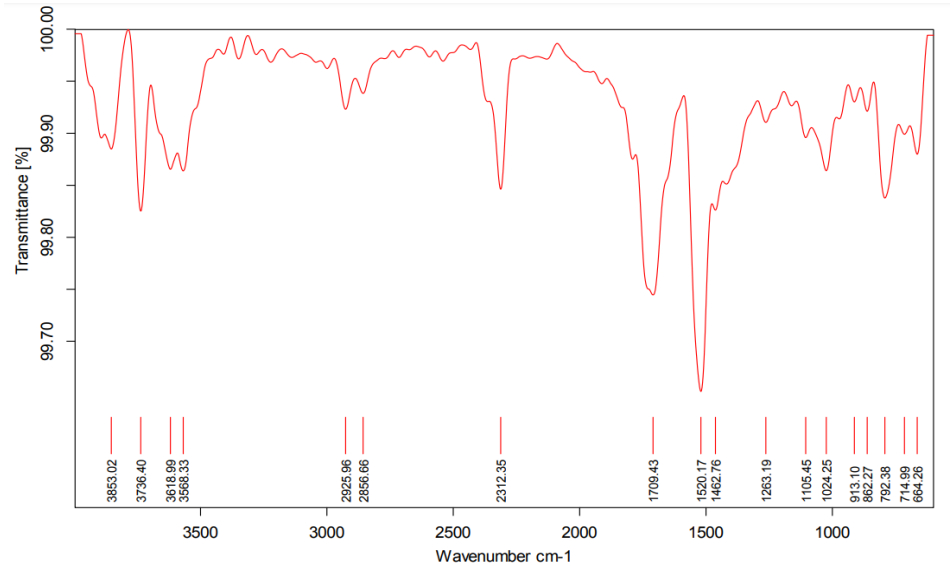


Fig. 10. 10% alkali plus 1% silane treated sisal fiber reinforced polyester composites (15% fiber).

The C-H stretching vibration shifts from  $2922.39\text{ cm}^{-1}$  to  $3003.69\text{ cm}^{-1}$ , indicating that alkali treatment removed some hemicellulose and lignin from sisal. Furthermore, a decrease in -OH group concentration results in a decrease in the corresponding peak height after alkali treatment. The FTIR spectra after silane treatment revealed the addition of an absorption band, as shown in Fig 10. The stretching vibration of Si-O-Si and Si-O-C and Si-C groups, which are products of silane treatment, is represented by the absorption band that emerged at  $1106$ ,  $1024$ , and  $862\text{ cm}^{-1}$ . FTIR confirms that silane grafting occurred at the surface of silane-treated fiber, resulting in decreased hydrophilicity of silane composite and increased interfacial adhesion.

*Table 1. Experimental readings of Untreated SISAL fiber.*

% Fiber loading	Tensile Strength (N/mm <sup>2</sup> )				Flexural Strength (N/mm <sup>2</sup> )			
	Wet Sample (WS)	Error bar 3%	Dry Sample (DS)	Error bar 3%	Wet Sample (WS)	Error bar 3%	Dry Sample (DS)	Error bar 3%
0	10.670	0.3201	10.670	0.3201	20.680	0.6204	20.680	0.6204
5	9.910	0.2973	12.180	0.3654	21.120	0.6336	23.900	0.717
10	10.190	0.3057	12.970	0.3891	19.500	0.585	26.040	0.7812
15	8.000	0.24	15.580	0.4674	27.660	0.8298	31.360	0.9408

*Table 2. Experimental readings of Treated SISAL fiber with 10% NaOH.*

% Fiber loading	Tensile Strength (N/mm <sup>2</sup> )				Flexural Strength (N/mm <sup>2</sup> )			
	Wet Sample (WS)	Error bar 3%	Dry Sample (DS)	Error bar 3%	Wet Sample (WS)	Error bar 3%	Dry Sample (DS)	Error bar 3%
0	10.670	0.3201	10.670	0.3201	20.680	0.6204	20.680	0.6204
5	7.850	0.2355	11.570	0.3471	19.580	0.5874	20.450	0.6135
10	9.550	0.2865	13.220	0.3966	29.490	0.8847	29.340	0.8802
15	13.500	0.405	17.970	0.5391	29.690	0.8907	34.450	1.0335

*Table 3. Experimental readings of Treated SISAL fiber with 10% NaOH + 1% Silane.*

% Fiber loading	Tensile Strength (N/mm <sup>2</sup> )				Flexural Strength (N/mm <sup>2</sup> )			
	Wet Sample (WS)	Error bar 3%	Dry Sample (DS)	Error bar 3%	Wet Sample (WS)	Error bar 3%	Dry Sample (DS)	Error bar 3%
0	10.670	0.3201	10.670	0.3201	20.680	0.6204	20.680	0.6204
5	7.850	0.2355	11.490	0.3447	20.830	0.6249	40.210	1.2063
10	14.840	0.4452	16.110	0.4833	50.780	1.5234	52.980	1.5894
15	18.480	0.5544	20.230	0.6069	52.120	1.5636	53.120	1.5936

*Table 4. Experimental readings of % water absorption for untreated SISAL fiber.*

% Fiber loading	Initial Weight (gm)	Final Weight (gm)	% Water absorbed
0	5.570	5.570	0
5	7.122	7.150	0.393
10	10.814	11.012	1.802
15	11.958	12.164	1.691

*Table 5. Experimental readings of water absorption for treated SISAL fiber with 10% NaOH.*

% Fiber loading	Initial Weight (gm)	Final Weight (gm)	% Water absorbed
0	5.570	5.570	0
5	10.106	10.164	0.572
10	12.409	12.680	2.134
15	13.836	14.234	2.795

*Table 6. Experimental readings of water absorption for treated SISAL fiber with 10% NaOH + 1% Silane.*

% Fiber loading	Initial Weight (gm)	Final Weight (gm)	% Water absorbed
0	5.570	5.570	0
5	10.255	10.366	1.070
10	11.432	11.575	1.235
15	15.123	15.348	1.466

*Table 7. FTIR characteristics absorption bands [42].*

Wavenumber (cm <sup>-1</sup> )	Assignment
3800-3200	-OH, Stretching Vibration
3029, 2921, 2924	CH-Stretching in cellulose
1718-1740	C=O Associated with carbonyl compounds
1480-1450	CH <sub>2</sub> Symmetric bending
1380-1386	In plane C-H bending
1205	-COH in plane Bending
1118	S-O-C Stretching vibration
1069	S-O Stretching Vibration
1037 (1020-1055)	Si-O-Si trenching vibration
744	Si-O-Si Organic Siloxane

## Conclusion

- Improvement in the tensile strength were observed with an increase in fiber loading for untreated, alkali-treated and alkali plus silane treated sisal fiber reinforced polyester composites without water absorption, the rise is highest for 10% alkali plus 1% silane treated fiber.
- There is a reduction in tensile strength with an increase in fiber loading for untreated sisal fiber reinforced polyester composites with water absorption with an increase in fiber loading for untreated fiber. For alkali-treated and alkali plus silane treated fiber with water absorption, initially, there is a decline in the tensile strength up to 5% fiber loading followed by an enhancement in it with a further increase in fiber loading. Rise is higher for alkali plus silane treated sisal fiber reinforced composites.
- Improvement in flexural strength were observed with an increase in fiber loading for untreated, alkali-treated and alkali plus silane treated sisal fiber reinforced polyester composites without water absorption, the improvement is highest for 10% alkali plus 1% silane treated fiber.
- Initially the flexural strength remains same up to 5% fiber loading for alkali-treated and alkali plus silane treated sisal fiber reinforced polyester composites with water absorption, followed by an increase in it with further increase in fiber loading. There is a slight decrease in the flexural strength up to 5% fiber loading followed by an increase in it up to 10% fiber loading and thereafter it remains stagnant for untreated sisal fiber reinforced polyester composite with water absorption. The increase is maximum for alkali plus silane treated sisal fiber polyester reinforced composite, as alkali plus silane treatment is effective in overcoming weakening of fiber matrix interface due to water absorption.
- There is an increase in the impact strength with increase in fiber loading for untreated and treated SISAL fiber reinforced polyester composites.
- There is an increase in water absorption with an increase in fiber loading, however, water absorption is minimum for alkali plus silane treated sisal fiber reinforced composites.

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## Competing Interest

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