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Tensile behaviour of (3-Aminopropyl) trimethoxysilane and (3-Glycidyloxypropyl) trimethoxysilane treated coir for composites applications

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Abstract

The tensile properties of as-received coir treated using 0.25% (3-Glycidyloxypropyl) trimethoxysilane and those treated with 0.25% of (3-Aminopropyl) trimethoxysilane have been investigated and compared. The tensile strength, Young's modulus and elongation at break of (3-Glycidyloxypropyl) trimethoxysilane treated coir were; 133.29 MPa, 1.94GPa, and 32.52% respectively; corresponding to 5%, 8% and 15% respectively higher than those of (3-Aminopropyl) trimethoxysilane treated coir fibres. The Weibull modulus of the fibres treated with (3-Glycidyloxypropyl) trimethoxysilane and (3-Aminopropyl) were 4.30 and 3.08 respectively, this means 28% higher than the Weibull modulus of the 3-(Aminopropyl) trimethoxysilane treated coir fibres. The Weibull shape parameters of the treated fibres show lower probability of failure than the untreated fibres from literature. The failure traces show an initial linear region followed by a non-linear region before fracture occurred, typical of natural fibres. The microstructure of the silane treated coir fibre exhibited rough surfaces which might bring about enhanced interlocking of the fibres/matrix and improved interfacial bond strength between the fibre and the matrix during composites manufacture and subsequently, improved mechanical properties of the composites.

Keywords: Coir fibre, tensile strength, (3-Aminopropyl) trimethoxysilane, (3-Glycidyloxypropyl) trimethoxysilane, Weibull modulus

1. Introduction

Several research works are continuously carried out on natural fibres in order to enhance their properties for composite applications. Natural fibres are environmentally friendly and can be sustained. They are much less heavy when compared to synthetic fibres such as glass and carbon. Coir is a lignocellulosic fibre with porosity ranging between 22.9-33.5% (Tran *et al.*, 2015; Luz *et al.*, 2017) contributing to coir fibres possessing densities as low as 1345 kg/m^3 (Madueke *et al.*, 2022) hence savings in fuel and energy in comparison with synthetic fibres. Coir is impervious to weather hence used in geotextiles. Coir is used as pigment for coating (Freitas *et al.*, 2022), as thermal insulator(Mahmud *et al.*, 2023), in automobile Clush (Sundarapandian, G; Arunachalam, 2020), even in wipers (Yashwanth, 2021) and Packing material (Khalid and Pedro, 2021).

Coir fibre is abundant in tropical regions including Nigeria where it is discarded and considered as waste or as firewood, therefore many more applications of coir is necessary. However, a major hindrance to its application is as a result of its hydrophilic nature (Siy *et al.*, 2020; Jamadi *et al.*, 2021) making them susceptible to moisture ingress (Salem *et al.*, 2020) and hampering their mechanical properties. The hydrophilicity of natural fibres has been attributed to their chemical constituents such as cellulose and lignin (see Table 1). If they are used as reinforcement in composites without prior treatment can lead to poor interfacial bond strength between the fibre and the matrix (Michelena *et al.*, 2022; Seisa and Ude, 2022). Surface modifications are therefore carried out on natural fibres for improvement on their properties as single fibre and to improve their performance in composites (Salem *et al.*, 2020; Khalid and Pedro, 2021). These modifications include; alkali treatment (Amroune *et al.*, 2018; Okafor et *al*., 2022; Ahmed *et al.*, 2022; Ghori *et al.*, 2023), acetylation (Khalid & Pedro, 2021; Seisa & Ude, 2022), silane treatment (Asim *et al.*, 2016; Mustakim & Ghaztar, 2022), isocyanate (Khalid & Pedro, 2021), and Plasma treatment (Seisa & Ude, 2022).

Silane treatment has been used to address the inherent moisture absorption of natural fibres which limit their mechanical properties as single fibres and in composites where it results in poor adhesion and hence weak interfacial bond strength(Siy *et al.*, 2020; Khalid and Pedro, 2021). Silane treatment is usually in form of soaking the fibre in different concentrations of silane for a given duration and temperature. One of the common silanes that has been used is the organo-silane with the structure (Wang *et al.*, 2020) as shown in equation 1.

$$X - S_I - (OR)_3 \qquad (1)$$

The functional group is represented by R (R= aminipropyl, vinyl or methapropyne). R reacts with the non-polar resin matrix while the X functional group (X= methoxy, chloro and ethoxy) can hydrolyse to form a silanol group in an aqueous solution reacting with hydroxyl (OH) group of the cellulosic fibre. Silane treated fibres possess lower density and diameter than untreated (Rait, 2014), which can lead to improved strength (Madueke, 2021).

This study hence focuses on treatment of coir using (3-Aminopropyl) trimethoxysilane and (3-Glycidyloxypropyl) trimethoxysilane in preparation for composites. From literature, coir has rarely been treated using these silanes and their effects on their tensile properties rarely compared at equal concentrations of the silane

Table 1 Chemical and Physical Properties of coir

Properties	Content	References
Cellulose (%)	26.8±0.05	(Hassan *et al.*, 2023)
Hemicellulose (%)	8.2 ± 0.3	(Mathura and Cree, 2016)
Lignin (%)	33.5	(Arsyad, 2017)
Ash (%)	3.7	(Hassan *et al.*, 2023)
Pectin (%)	4	(Latif, Wakeel and Khan, 2018)
Density (Kg/m^3)	1400	(Freitas *et al.*, 2022)

2.0 Materials and Method

2.1 Materials.

Matured as-received brown coir fibres were cleaned to remove debris and dust particles. The fibres were washed with warm water of about 45 ^oC. They were washed three times, rinsed with distilled water and left to dry at room temperature for two days. (3-Aminopropyl) trimethoxysilane and (3-Glycidyloxypropyl) trimethoxysilane used in the fibre treatment were supplied by Sigma-Aldrich Company, Ltd UK. Each of the silanes was prepared at specified concentration of 0.25%. From literature, treatment of natural fibres with more than 1% silane concentration and at longer immersion time have resulted in fibre damage and reduction in their mechanical properties (Orue *et al.*, 2015; Jamadi *et al.*, 2021; Mustakim and Ghaztar, 2022).

2.2 Treatment of coir fibre using silane solutions

The silane concentration was prepared as follows:
Ethanol: Deionised (DI) water
4: 1

Silane 0.25% = 0.25g

Ethanol + DI water = 100-0.25=99.75

Ethanol = 4/5 x 99.75=79.8g

DI water =1/5x99.75 =19.95g

The required quantity of deionised water was measured out using 1000ml glass beaker. A corresponding quantity of ethanol was weighed out on a balance. Ethanol and deionised water were combined at a weight ratio of 4:1 respectively. Ethanol and water are fully miscible azeotropic mixture forming a homogenous solution. The required amount of (3-Aminopropyl) trimethoxysilane (APTS) was added to the mixture of ethanol and deionized water. 2.5g of selected fibres were submerged in this solution at room temperature and for a duration of 30 minutes. The fibers were washed with deionized water to remove excess silane and dried in the air oven at 50 °C for 12 hours. Tensile tests were carried out on the treated samples. Similar approach used for treatment of coir using APTS was adopted for the treatment of coir using (3-Glycidyloxypropyl) trimethoxysilane (GPTS) at the same concentration of 0.25%.

2.3 Tensile tests of the treated samples

30 fibres treated using APTS and another 30 fibres treated using GPTS were selected. The fibre diameters were taken at three different points along the gauge length. The samples were each mounted on the Intron 5566 universal testing machine with the fibre axis in line with the cross head axis to simulate a uniform stress condition over the fibre cross-section. The full scale load was 100N, the cross head displacement rate was 1mm/minute and the gauge length was 20mm. Not disturbing the setup, both sides of the paper tab were carefully slit at the mid gauge. The sample was then tensioned with the software recording the test load and the elongation until the specimen fractured.

2.4 Weibull distribution

2-Parameter Weibull was used to determine the strength distribution of the treated coir fibres as has been done previously (Trujillo *et al.*, 2014; Madueke *et al.*, 2022).

The general Weibull equation is as shown in equation (2);

$$F_a = 1 - e\,\frac{-(a-a_u)^m}{a_0} \tag{2}$$

From equation (2), then;

$$F_{(\sigma)} = 1 - e^{-(\frac{\sigma}{\sigma o})^m}$$

(3) (Two-parameter Weibull equation).

F_σ = Strength distribution

σ_0 = Scale parameter, m = Weibull shape parameter. The higher the shape parameter, the smaller the variation in the data and n = Sample size assessed.

2.5 Scanning Electron Microscopy

The coir samples were sectioned and mounted on sample holders, they were sputter-coated with gold to render them conductive and then taken to Table top SEM for microstructure determination.

3.0 Results and Discussions

3.1 Tensile properties and Weibull distribution of treated fibres

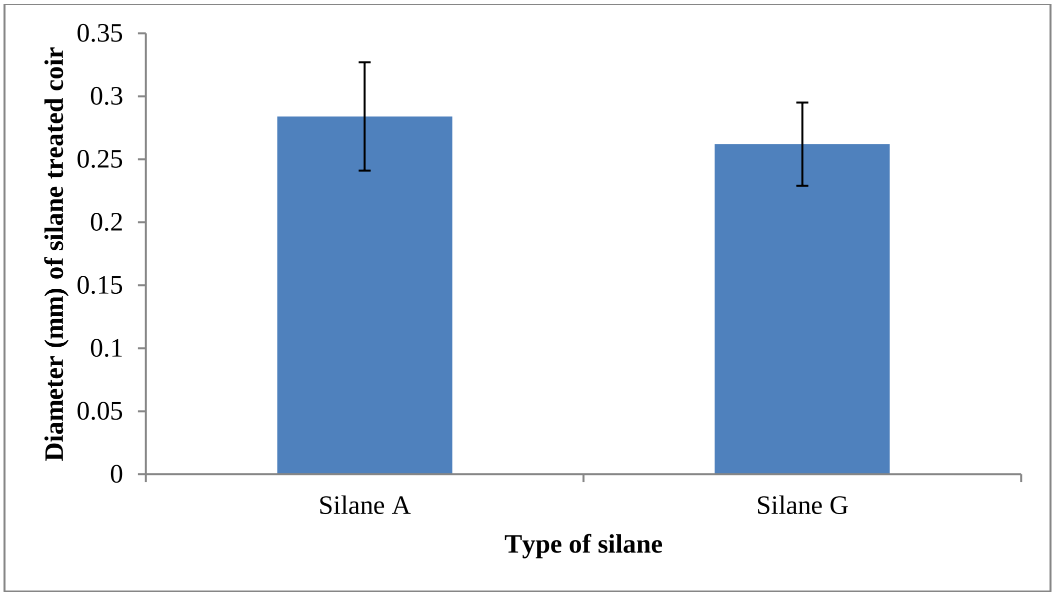
Table 2 shows the summary of the average values of the tensile properties and Weibull modulus of the silane treated coir fibres and untreated fibres from the present work and from literature for comparisons. High standard deviations can be observed especially from the tensile strength and Young’s modulus, these are in line with natural fibres and have been attributed to the defects or imperfections in the fibre (Mathura and Cree, 2016). The tensile strength, stiffness and elongation at break of GPTS treated coir were found to be 5%, 8% and 15% respectively higher than those of APTS treated coir. The tensile strength and elongation at break obtained are comparable with those obtained by (Rait, 2014) (Table 1). However, the Young's modulus differs appreciably, this can be attributed to the difference in the silane concentrations and the treatment duration. The tensile strengths obtained from the present work are higher than those of untreated coir from literature as shown in Table 1 with over 30% for(Brintz, 2014; Rait, 2014) . The differences in the tensile properties of the treated and untreated may not only be attributed to the treatment but also to the fibre origin, diameter, gauge lengths and processing conditions (Mathura and Cree, 2016; Madueke, 2021) The diameters of the APTS and GPTS as shown in Figure 1 are; 0.284 and 0.262 mm respectively, these are within the range obtained from literature. Hemp fibres were treated with 3-[(2-aminoethylamino) propyl] trimethoxysilane at room temperature and for a duration of 45 minutes, a 4% increase in the tensile strength was observed (Sawpan *et al.*, 2011). A 6% drop in strength was recorded on the treatment of sisal fibre using 2% 3-(2-aminoethylamino) propyl trimethoxy silane under reflux (Orue *et al.*, 2015). An increase in the tensile strength of silane-treated natural fibre composites have been reported as a result of improved adhesion of fibre on the polymer matrix due to a reduction in the cellulose hydroxyl group at the fibre/matrix interface (Siy *et al.*, 2020; Wang *et al.*, 2020; Jamadi *et al.*, 2021).

The scatter in the values of the fracture strength can be attributed to the flaws present in the fibre and their distributions as mentioned earlier. The Weibull parameter, m, used to check the degree of flaws shows that the fibres treated with GPTS has Weibull parameter, m, 28% higher than those of the APTS treated coir as shown in Figure 5 signifying lower degree of flaws in the GPTS treated fibres. The increase in the Weibull parameter m with decrease in diameter (GPTS) shows that the flaw distribution is more evenly spread in fibres of lower diameters. The Weibull distribution shows that the treated fibres have a high degree of linearity; R^2= 0.9829 and 0.9880 confirming that Weibull can be used to analyse tensile strength of coir. The strength distribution of the treated fibres has lower scatter than the untreated (Table 2) and some natural fibres (Ernestina *et al.*, 2013; Guo *et al.*, 2014). This result confirms that the quality of the silane treated fibres produced is high. The scale parameter (141.06MPa) corresponding to failure probability of 63.2% is higher than the average strength of the silane treated fibres.

Table 2 Comparison of the tensile properties of silane-treated and untreated coir fibres

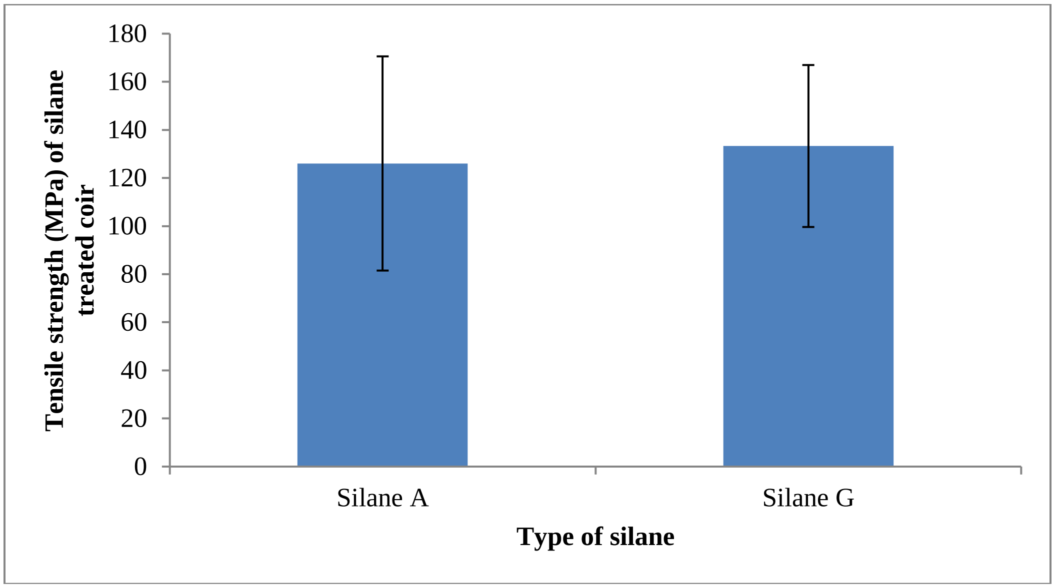
Silane Type	Conc. (%)	Dura (ms)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at break (%)	Weibull modulus	References
APTS	0.25	30	126.03± 44.53	1.79±0.50	27.70 ± 8.76	3.08	Present work
GPTS	0.25	30	133.29± 33.67	1.94±0.46	32.52 ±7.65	4.30	Present work
APTS	0.15	1440	120.28 ± 47	4.40± 2.62	30.20±10.31	1.83	(Rait, 2014)
Untreated	-	-	81.40 ± 40.85	2.69 ±1.23	28.97±13.24	1.83	(Rait, 2014)
Untreated	-	-	120 ± 18.76	2.31± 0.49	-	-	(Mathura and Cree, 2016)
Untreated	-	-	95-118	8	-	-	(Sundarapandian, G; Arunachalam, 2020)
Untreated	-	-	85.64±8.71	2.85± 0.23	26.0 ±2.23	-	(Brintz, 2014)

Dura(ms)= duration (minutes)



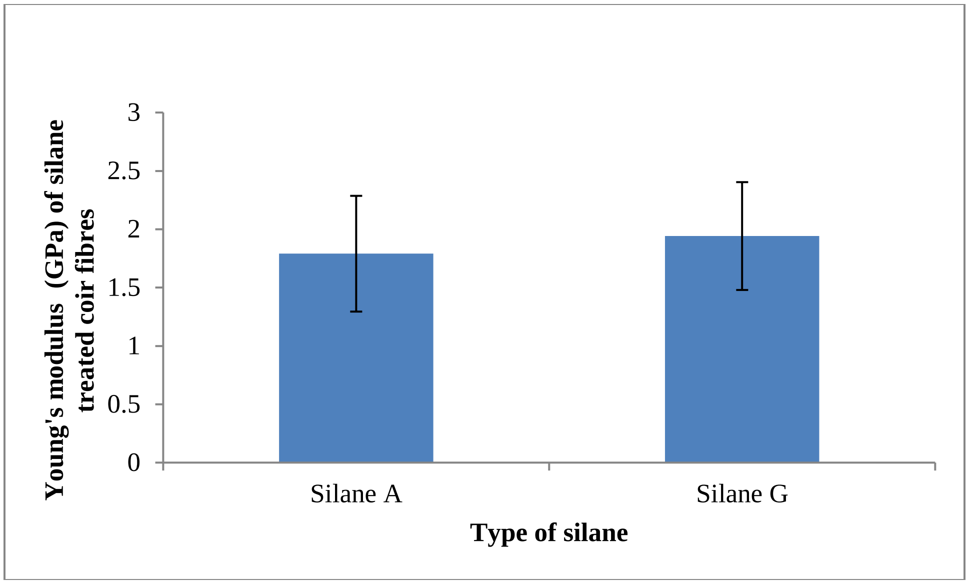
Silane A= (3-Aminopropyl) trimethoxysilane; silane G=(3-Glycidyloxypropyl) trimethoxysilane)

Figure 1 Comparison of the diameters of coir fibres treated using 0.25% (3-Aminopropyl) trimethoxy and 0.25% (3-Glycidyloxypropyl) trimethoxysilane



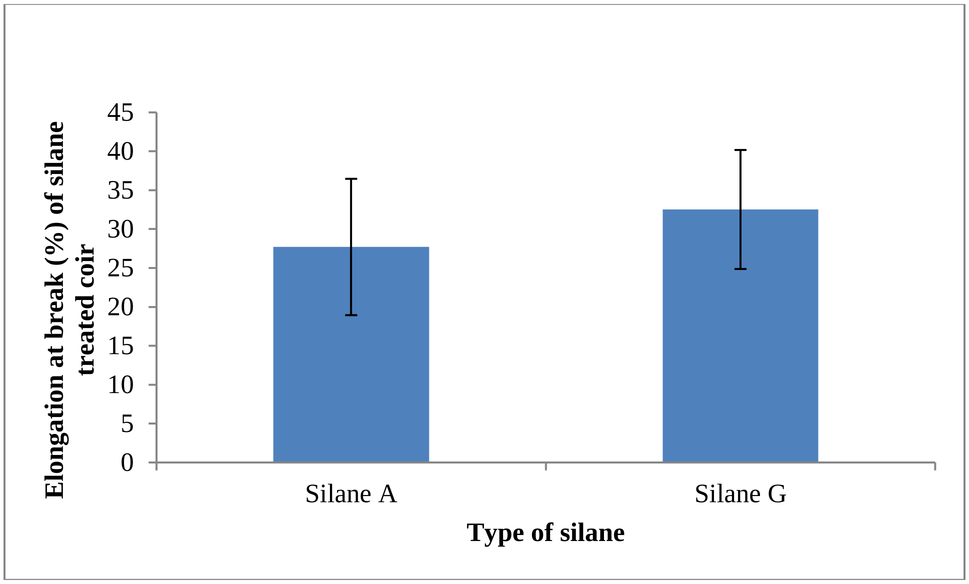
Silane A= (3-Aminopropyl) trimethoxysilane; silane G=(3-Glycidyloxypropyl) trimethoxysilane)

Figure 2 Comparison of the tensile strength of the 0.25% (3-Aminopropyl) trimethoxy and 0.25% (3-Glycidyloxypropyl) trimethoxysilane treated coir fibres



Silane A= (3-Aminopropyl) trimethoxysilane; silane G=(3-Glycidyloxypropyl) trimethoxysilane)

Figure 3 Comparison of the Young's modulus of the 0.25% (3-Aminopropyl) trimethoxy and 0.25% (3-Glycidyloxypropyl) trimethoxysilane treated coir fibres



Silane A= (3-Aminopropyl) trimethoxysilane; silane G=(3-Glycidyloxypropyl) trimethoxysilane)

Figure 4 Comparison of the elongation at break of the 0.25% (3-Aminopropyl) trimethoxy and 0.25% (3-Glycidyloxypropyl) trimethoxysilane treated coir fibres

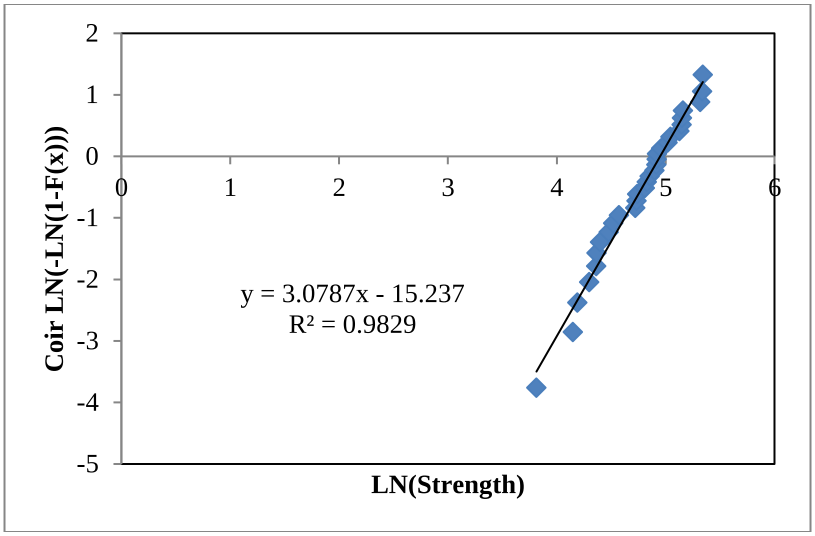


Figure 5 Weibull distribution of coir fibres treated using 0.25% (3-Aminopropyl) trimethoxysilane

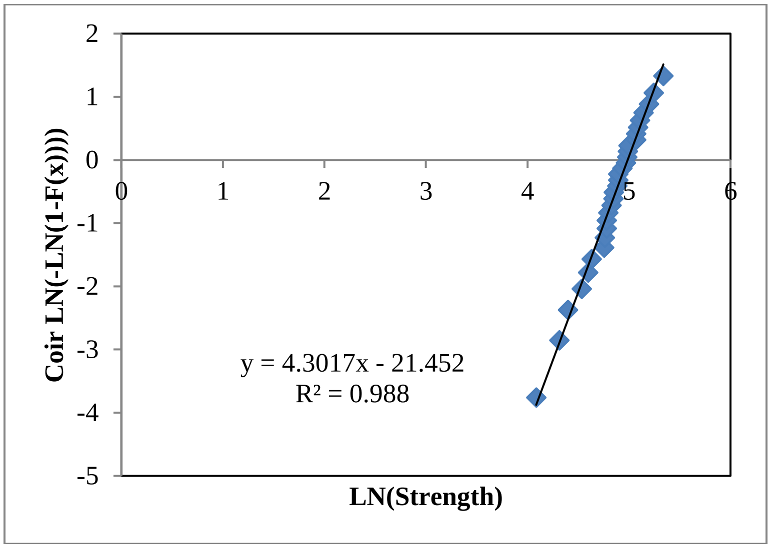


Figure 6 Weibull distribution of coir fibres treated using (3-Glycidyloxypropyl) trimethoxysilane

3.2 Failure curves of Amino and epoxy silane treated coir fibres

Failure curves of the of (3-Aminopropyl) trimethoxysilane and (3-Glycidyloxypropyl) trimethoxysilane treated coir fibres were characterised with smooth traces without load drops as shown in Figures 7 and 8. The shapes of the tensile curves are in line with natural fibres. An initial linear region was followed by a non-linear region before fracture occurred. The nonlinear regions can be attributed to delamination of the fibre cells as they are being pulled in tension. No significant difference existed between the maximum failure stresses of the fibres treated with (3-Aminopropyl) trimethoxysilane and the fibres treated with (3-Glycidyloxypropyl) trimethoxysilane. The same can be said of their mid and low failure stresses.

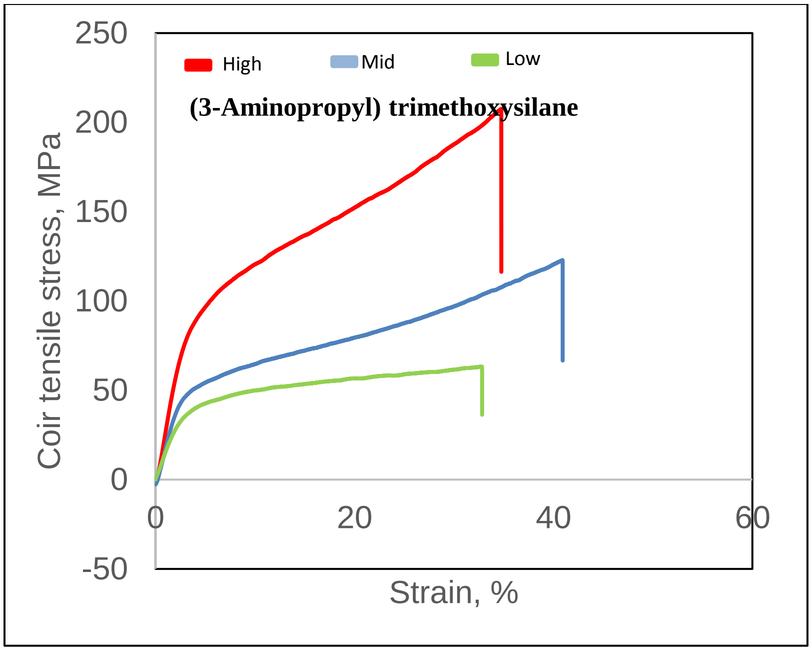


Figure 7 Failure curve of (3-Aminopropyl) trimethoxysilane treated coir fibre

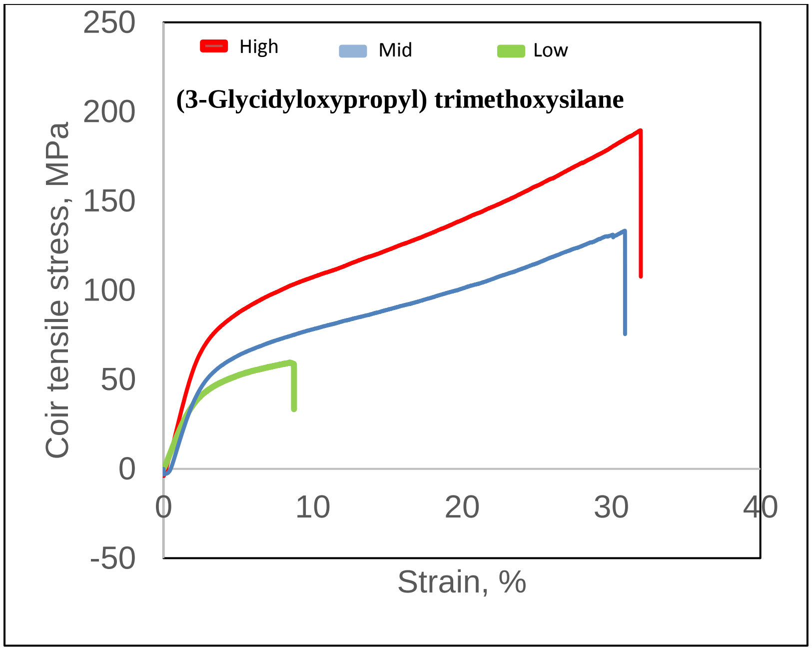


Figure 8 Failure curve of (3-Glycidyloxypropyl) trimethoxysilane treated coir

3.4 Scanning Electron Microscopy coir fibres

The cross-section and surface of as-received coir fibre as shown in Figure 9 show the presence of pores (lumens). The surface of the as-received coir fibre shows globular protrusions called tyloses and the presence of debris. The cross-section and surface changed after treatment with silane as shown in Figure 10. The fractured surfaces of the treated fibres show cell fracture and delamination within the cells as the fibre is pulled in tension. This delamination has been attributed to the weakening of the lignin and cellulose connection in the course of tensile testing (Guo *et al.*, 2014). The non-linear portion of the stress-strain curve depicted in Figures 7 and 8 can be attributed to delamination. The silane-treated coir fibres show rougher surfaces and striations than the untreated. This might enhance better interlocking between the fibre and the matrix in composite manufacture.

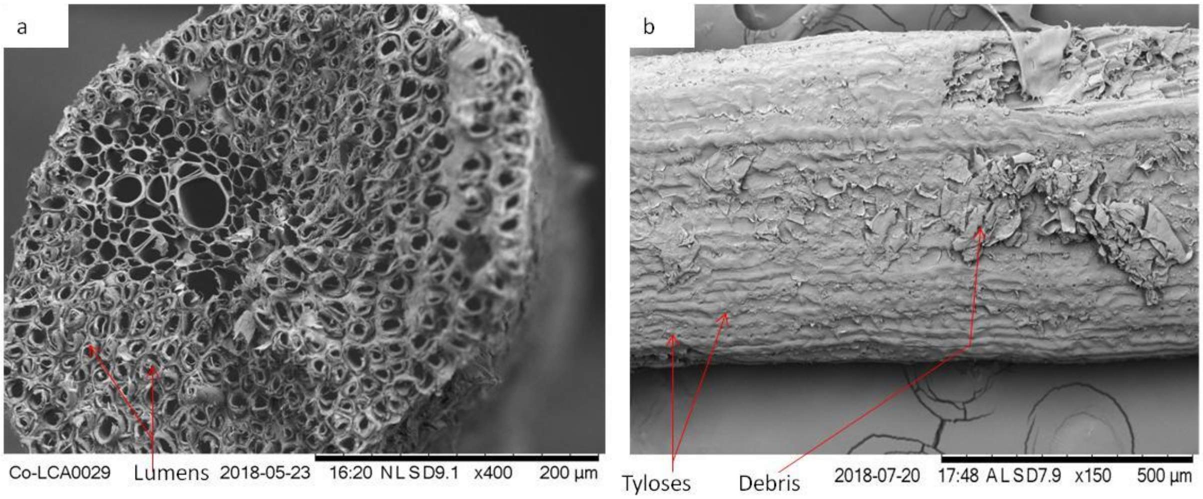


Figure 9 Scanning electron micrograph of as received coir fibre (a) Cross-section (b) unwashed surface

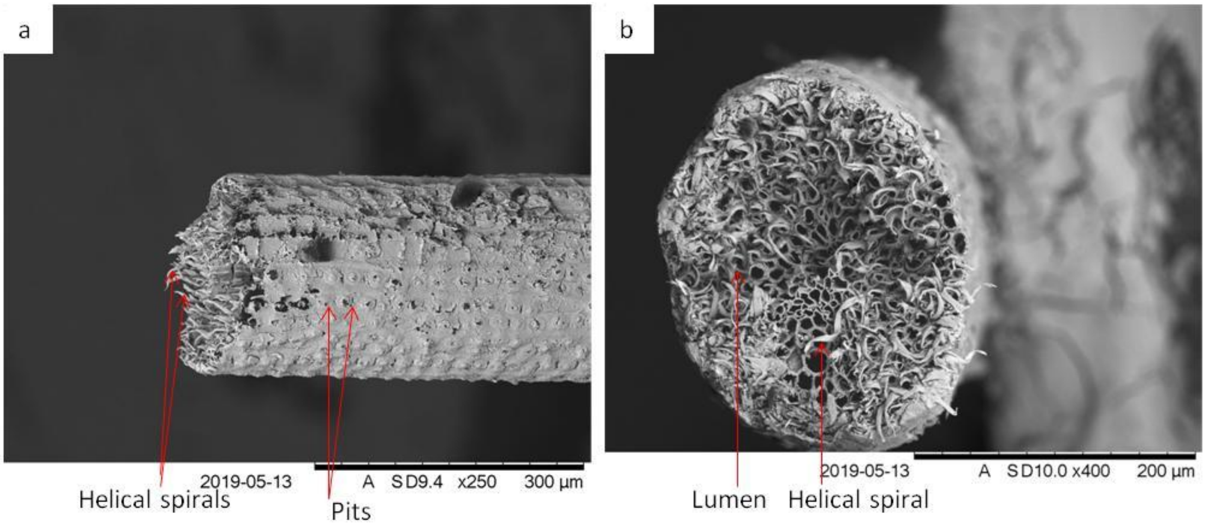


Figure 10 Micrographs of (3-Aminopropyl) trimethoxysilane treated coir fibre at 0.25% concentration.

Coir has been treated with (3-Aminopropyl) trimethoxysilane and (3-Glycidyloxypropyl) trimethoxysilane at 0.25% concentration in preparation for composite manufacture. The silane treated fibres showed improved tensile properties when compared with the untreated from the literature. The Weibull shape parameters of the silane treated fibres show lower probability of failure than untreated fibres from literature.

4.0. Conclusion

The tensile properties of coir treated with 0.25% (3-Glycidyloxypropyl) trimethoxysilane and those treated with 0.25% of (3-Aminopropyl) trimethoxysilane have been investigated and compared. The tensile strength, stiffness and elongation at break of (3-Glycidyloxypropyl) trimethoxysilane treated coir fibres were found to be 5%, 8% and 15% respectively higher than those of (3-Aminopropyl) trimethoxysilane treated coir fibres. The Weibull shape parameter of the fibres treated with (3-Glycidyloxypropyl) trimethoxysilane was 28% higher than those of the 3-(Aminopropyl) trimethoxysilane treated coir fibres. The Weibull shape parameters of the silane treated fibres show lower probability of failure than untreated fibres from literature. The microstructure of the silane treated coir fibre exhibited rough surfaces which might bring about enhanced interlocking of the fibres/matrix and improved interfacial bond strength between the fibre and the matrix during composites manufacture and subsequently, improved mechanical properties.

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