



## Analysis of the effects of heat treatment on the tensile properties of coir fibres using Minitab-18 statistical software

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### Abstract

Vacuum oven heat treatments were done to enhance the properties of coir fibre for improved performance in composites. Their effects on the mechanical properties of coir fibres were investigated. As received (AR) coir fibres were subjected to heat treatment temperatures of 80 °C and 160 °C for 6 hours at each temperature in a vacuum oven. The effects of the heat treatment temperature on AR coir fibres were statistically analyzed using ANOVA, *F*-tests, Fisher Pairwise grouping and Fisher Individual Tests for Difference of Means (FITDM) with Minitab-18 Software. This was done to determine the significance of these heat treatments on the tensile properties of AR coir fibres. Fisher Pairwise grouping and FITDM show that strength and elongation of AR coir fibres differed significantly from coir fibres heat-treated at 80 °C. The tensile strength of AR coir fibres was found to be, respectively, 49% and 24% lower than coir fibres treated at 80 °C and 160 °C. The stiffness of coir fibres (AR) was found to significantly increase after heat-treating at 160 °C, while the elongation at break of AR was 44% higher. The strength distributions obtained from the tensile test data were subjected to two-parameter Weibull statistics. AR coir fibres displayed Weibull moduli that were 16% and 56% higher than coir fibres treated at 80 °C and 160 °C, respectively. SEM was conducted on the samples to delineate the morphological changes affecting the properties of coir fibres.

**Keywords:** Coir fibre, Heat treatment, Vacuum oven, Tensile properties, Statistical analysis, Weibull distribution

### 1. Introduction

The quest for a greener environment and lightweight materials has led to many of investigations of natural fibres for use as reinforcements in composite materials. Enhanced awareness in the use of natural fibres could be attributed to their many advantages such as lower material costs, reduced densities and production cost savings compared to synthetic fibres.

Coir is a natural lignocellulosic fibre obtained from coconuts. It consists of 36-43% cellulose, 41-45% lignin and 10-20% hemicellulose [1]. An estimated average of 55 billion coconuts are harvested yearly with only about 8.25 billion (15%) of the husks used for coir fibre production [2]. Therefore, wider applications of coir fibres are imperative. Coir possesses one of the lowest densities, 1150 to 1160 kg/m<sup>3</sup> [3, 4] among natural fibres. Their affinity for moisture and the presence of moisture in natural fibre is a major concern when plant fibres are considered for reinforcement of a polymer. Water may reduce the strength, initiate swelling, create defects such as micro-cracking in the formed composites, hence heat treatments are necessary.

Drying at 120 °C and above alters the layer structure and brings about changes in the mechanical properties of fibres [5]. An earlier research group [6] subjected coir fibres to air oven treatment temperatures of 150 and 200 °C for durations of 10, 20 and 30 minutes. The highest increase in strength and stiffness was observed at 150 °C for 20 minutes, with values of 72.4 MPa and 2.0 GPa, respectively. However, a reduction of 11.32% in elongation at break was reported in comparison with untreated fibres. Other researchers [7] investigated the tensile strength of bamboo after heating it in air. They discovered a gradual decrease in strength of fibres treated at 160 °C for 30 minutes, as well as a rapid change in strength with treatments at 180 °C for 30 minutes and 200 °C for 120 minutes. However, at 140 °C for 15 minutes, the strength was 516 MPa, the same as the untreated material. Kenaf fibres were heat-treated in a vacuum oven at temperatures of 130, 140 and 160 °C. The highest increase in the tensile strength of the heat treated fibres was observed at 140 °C [8]. Unfortunately, the diameter, Young's modulus and fracture strain of the treated fibres were not reported. Research needs to be carried out on the heat treatment of single coir fibres using a vacuum oven as well. This paper, therefore, examines the tensile properties of vacuum heat-treated as-received coir fibres subjected to treatment temperatures of 80 °C and 140 °C for the same duration, 6 hours. *F*-tests and Fisher Pairwise grouping were used to ascertain the degree of significance in tensile property changes of the conditioned fibres. A Weibull approach was adopted to characterize the strength distribution of each category. The failure pattern and the changes in the properties of these fibres were further characterized by conducting scanning electron microscopy on the samples.

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## 2. Materials and methodology

### 2.1 Materials

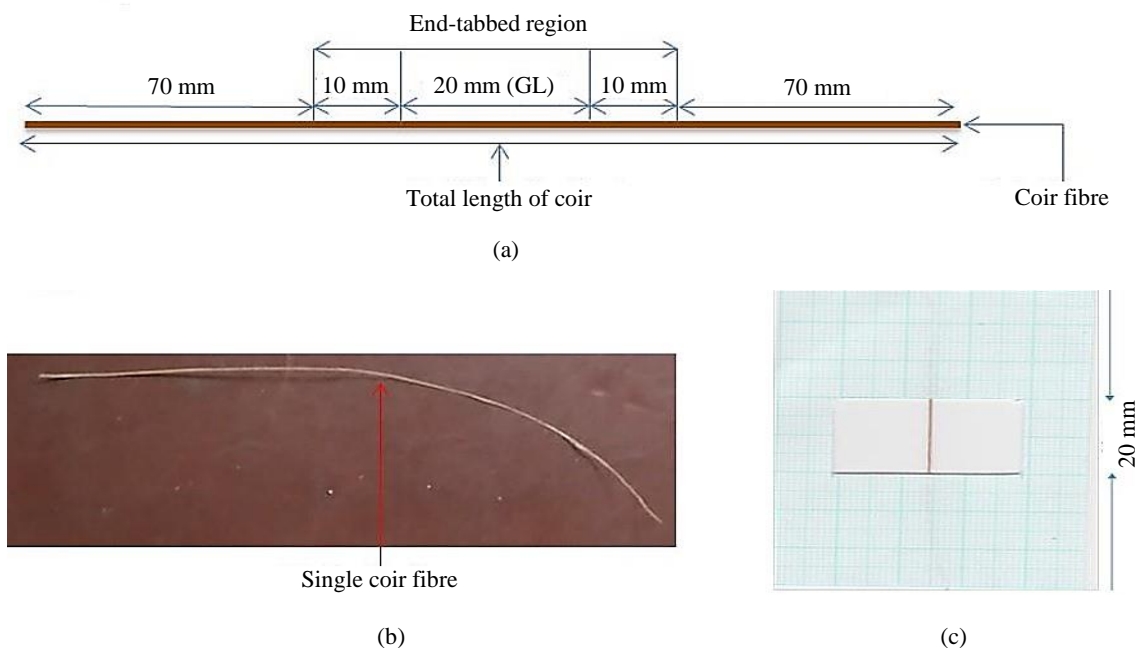
As-received coir fibres were washed five times in tap water and finally with distilled water. They were left to dry at room temperature for two days. Coir fibres were selected that had a minimum length of 60 mm, but varying diameters ranging between 0.153 and 0.433 mm.

### 2.2 Heat treatment of coir fibres

Heat treatment was carried out by placing AR coir fibres in a Gallenkamp vacuum oven (Gallenkamp; Cambridge, UK) at 80 °C for 6 hours. This experiment was repeated for another set of AR coir fibres, but at a temperature of 160 °C. Cooling to room temperature was achieved over silica and the cooled samples were kept in a desiccator before tensile testing. These temperatures were selected as a result of the material properties as a function of temperature, behaviour and applicability. Most biopolymers that are used in the manufacture of biocomposites require a processing temperature that is above 150 °C to bring about degradation of plant fibre [9]. Therefore, temperatures of 160 and 80 °C were chosen for comparison above and below 150 °C. The influence of vacuum oven heat treatment on the tensile properties of these fibres was evaluated. Minitab 18 software was used to determine their statistical significance.

### 2.3 Tensile tests on single coir fibres

A schematic illustration of a coir fibre is shown in Figure 1(a), while Figure 1(b) is a photographic image of coir from which the sample gauge length was obtained. Coir fibres were individually bonded onto a paper frame (Figure 1(c)) to keep the fibres straight and aligned as well as to allow for good gripping. The two opposite sides of the paper frame were aligned with the 20 mm gauge window that was slit after mounting on an Instron universal testing machine (Model 5566; Instron; Norwood, MA USA) according to ASTM D3822-07. The two thinner sides of the gauge window paper frame, shown in Figure 1(c), were cut open using a pair of scissors. Tests were carried out using a 100 N load cell. The crosshead speed was 2 mm/minute with a gauge length of 20 mm. A minimum of 30 samples per condition were tested to obtain a statistically significant result. The test was carried out at 27 °C and 37% relative humidity.



**Figure 1** (a) Schematic illustration of a 180 mm long coir fibre, (b) Coir fibre from which a gauge length of 20 mm was sectioned (c) Fibre aligned straight in the centre of a window.

Optical microscopy was used in measuring the diameter of coir fibres [4, 10] with a Zeiss Axioskop 2 MAT optical microscope and Axio Vision analysis software (Carl Zeiss Ltd; UK). Coir fibres were assumed to have circular cross-sections. ImageJ software (US NIH; Washington, DC USA) was used to determine the circularity (roundness) of thirty specimens sectioned from the middle of the fibres. An average circularity value of  $0.900 \pm 0.03$  was obtained on a scale of 0-1, where 1 corresponds to a perfect circle. The fibre specimens from which the diameters were measured were taken from the middle sections of the fibres, where they are more uniform [11] and circular. These samples were taken at six different points along the length of the fibre and the average value was used for tensile calculations.

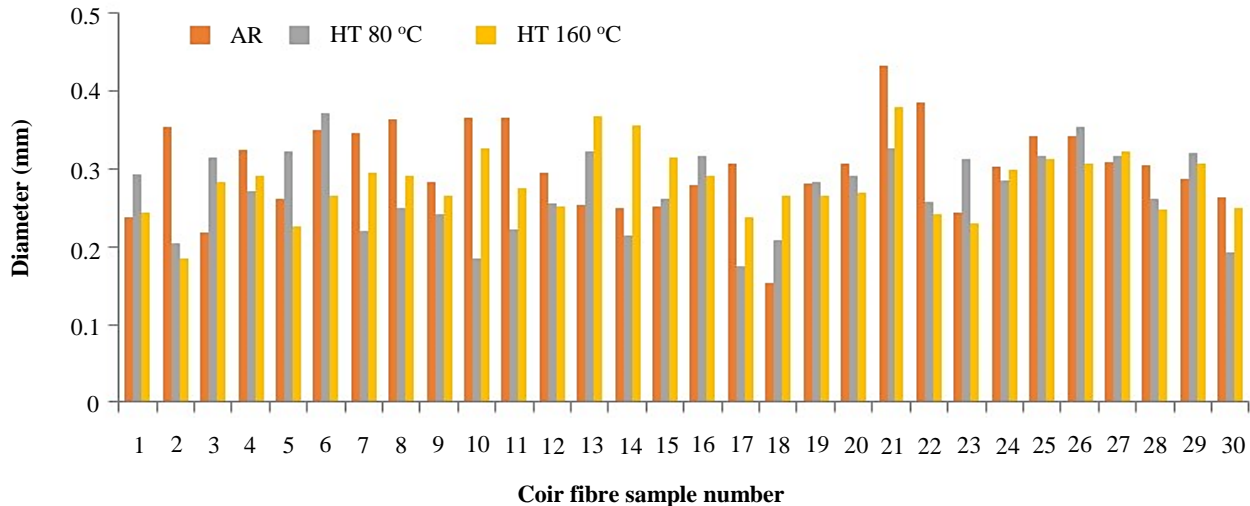
### 2.4 Scanning Electron Microscopy (SEM)

As-received and heat-treated coir fibres were sputter-coated with gold (Emscope SC 500; Quorum Technologies, East Sussex, UK) to provide conductivity for scanning electron microscopy. Then, fractured samples were characterized with a tabletop SEM model TM 3030 (Hitachi; Tokyo, Japan).

### 3. Results and discussion

#### 3.1 Effects of heat treatment temperature on fibre diameter, tensile strength, Young's modulus and elongation at break of coir fibres

Variability in the fibre diameter can be observed in Figure 2. The disparity in the diameters of the fibres is attributed to several factors, such as the different treatment methods used on the fibres, presence of debris or contaminants or possibly that the fibres could have been mixed and from different sources.



**Figure 2** Fibre diameter distribution graph for AR and heat-treated coir fibres

Heat treatment of natural fibres above 120 °C leads to softening of lignin and depolymerization with subsequent loss of tensile strength [12, 13]. Therefore, the tensile strength of the fibres heat-treated at 80 °C was 33% higher than those heat-treated at 160 °C. At increased temperature, there is a decrease in the flow of the polysaccharide matrix and thus a realignment of the microfibrils leading to a decreased microfibril to fibre axis angle and consequently, increased stiffness [5, 9]. As the temperature is increased, the lignin softens and the fibres become more brittle. Hence, there is a reduction in the elongation at break of the heat-treated fibres [14]. A weight loss was observed after heat treatment as a result of thermal degradation of the fibres [12]. Thermal degradation of coir fibres has been observed to occur in three stages by monitoring the weight loss of the material as a function of temperature. Weight loss at temperatures of 0-200 °C arises from evaporation of water. Degradation of hemicellulose occurs at 200-360 °C and thermal degradation of cellulose is seen at temperatures above 360 °C [15, 16]. The surfaces of the heat-treated fibres appear rough, which has been attributed to the partial removal of impurities and waxy substances [17]. This leads to improved bonding between the fibre and the matrix as the composite is formed. Earlier researchers reported [17, 18] an increase in the crystallinity index and higher tensile strength as a result of improved bonding between the kenaf fibres and a polyester matrix.

The average tensile properties, *i.e.*, strength, Young's modulus and elongation at break of as-received and heat-treated (HT) fibres are given in Table 1. From this table, average tensile strength, stiffness and elongation at break values of AR coir fibres are  $70.03 \pm 22.42$  MPa,  $2.44 \pm 1.49$  GPa and  $23.79 \pm 7.58\%$  respectively, which are comparable to previously reported values [6]. The tensile strength, stiffness and elongation values are 68.4 MPa, 1.6 GPa and 21.2% respectively. From Table 1, an increase in the tensile strength was observed for heat-treated fibres. An earlier study [8] reported observations of a similar increase in tensile strength, however a decreased strength was observed above 140 °C. Stiffness of the fibres with increased heat treatment temperature was reported for bamboo fibres [19].

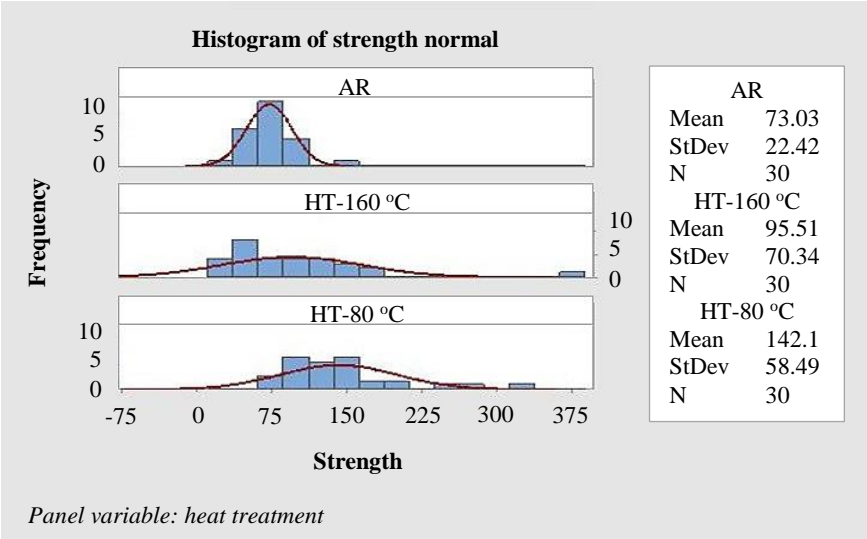
**Table 1** Summary of the average values of tensile properties of AR and heat-treated coir fibres

Fibre condition	Sample size	Av. Diameter (mm)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
As-received	30	$0.302 \pm 0.06$	$73.030 \pm 22.42$	$2.44 \pm 1.49$	$23.79 \pm 7.58$
HT 80 °C	30	$0.27 \pm 0.05$	$142.12 \pm 58.49$	$2.81 \pm 1.36$	$33.19 \pm 11.30$
HT 160 °C	30	$0.28 \pm 0.04$	$95.51 \pm 70.34$	$3.07 \pm 1.21$	$13.22 \pm 6.64$

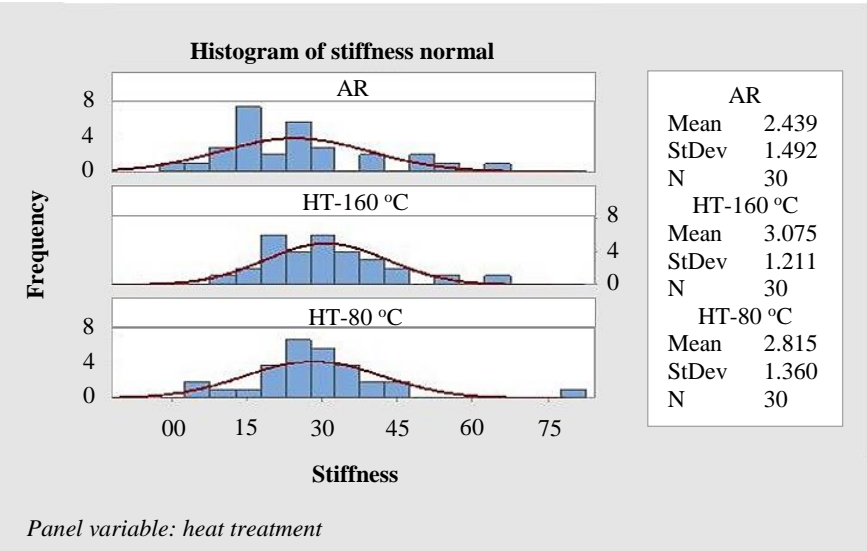
Av. = average

The histogram values of tensile properties in Figures 3(a-c) indicate a wide distribution of the data for each set of conditions with associated standard deviations for a sample size of 30. In these figures, the fitted normal distributions for the tensile properties are not perfect. This can be attributed to small sample size. A larger sample size would result in a better fit for a normal distribution. The highest standard deviation for strength, stiffness and elongation for the materials in the current study are for HT 160 °C, AR and HT 80 °C, respectively.

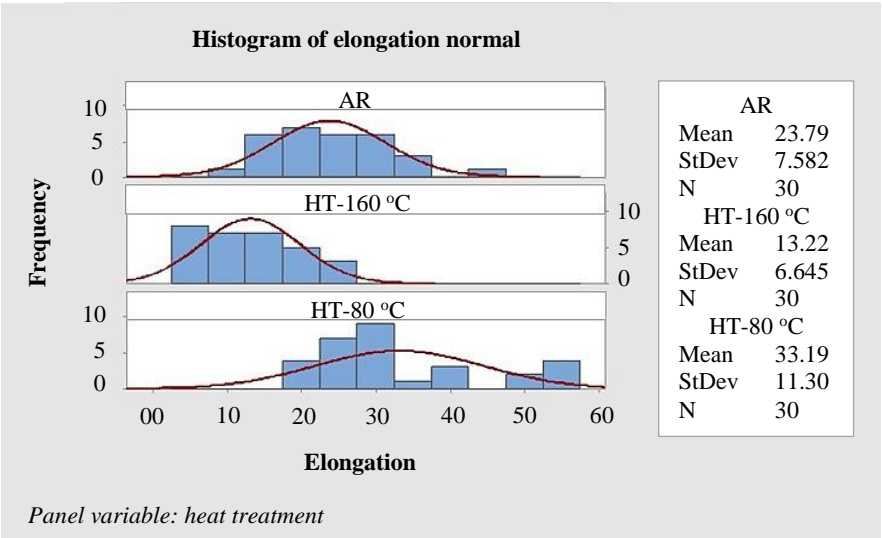
ANOVA and *F*-tests (Tables 2, 3 and 4) show that tensile strengths and elongation at break are influenced by heat treatment with a *P*-values that were less than 0.05. However, the stiffness of the fibre was not significantly influenced by heat treatment as evidenced by its *P*-value ( $> 0.05$ ).



(a)



(b)



(c)

**Figure 3** Histogram showing variations in the (a) tensile strength (b) stiffness and (c) elongation at break data using Minitab 18 software.

**Table 2** ANOVA Result at 0.05 significant level for tensile strength of AR and heat-treated coir

HT	SS	df	MS	F	P-value
Error	74517	2	37259	12.6	< 0.01
Within-group	257276	87	2957		
Total	331793	89			

HT = Heat treatment; SS = sum of squares, df = degree of freedom, MS = mean square

**Table 3** ANOVA Result at 0.05 significant level for stiffness of AR and heat-treated coir fibres

HT	SS	df	MS	F	P-value
Error	6.13	2	3.07	1.66	0.20
Within-group	160.72	87	1.85		
Total	166.85	89			

HT = Heat treatment; SS = sum of squares, df = degree of freedom, MS = mean square

**Table 4** ANOVA Result at 0.05 significant level for elongation of AR and heat-treated fibres

HT	SS	df	MS	F	P-value
Error	5988.438	2	2994.219	39.17767	7.37727E-13
Within-group	6649.122	87	76.42669		
Total	12637.56	89			

HT = Heat treatment; SS = sum of squares, df = degree of freedom, MS = mean square

Further statistical analysis *via* Fisher Pairwise grouping (Table 5) shows that strength and elongation at break of AR fibres (B, B), are significantly different from strength and elongation at break of heat-treated samples at 80 °C (A, A), as they do not share the same grouping in this table.

**Table 5** Grouping information (fisher pairwise comparisons: means that do not share the same grouping are significantly different) using fisher LSD method at a 95% confidence level

Factor	n	Tensile Strength		Young's modulus		Elongation at break	
		Mean	Group	Mean	Group	Mean	Group
As-received	30	73.03	B	2.439	A	23.79	B
HT 80°C	30	142.1	A	2.815	A	33.19	A
HT 160 °C	30	95.5	B	3.075	A	13.22	C

n = sample size; Mean = average value

The results of Fisher Individual Tests for Difference of Means (FITDM) given in Tables 6, 7 and 8 and displayed in Figures 4(a-c) show that the tensile strength of AR fibres is significantly different from that of HT 80 °C, but not significantly different from that of HT 160 °C, as indicated by their P-Values of <0.001 and 0.113, respectively. Furthermore, the tensile strength of the fibres treated at 160 °C is significantly different from those treated at 80 °C, but not significantly different from the AR coir fibres. However, significant differences were not observed in Young's modulus in any of the samples, as shown in Table 7. Significant differences were observed for elongation at break among all the fibre conditions shown in Table 8 (P-Value=<0.001).

**Table 6** Fisher individual tests for differences of means: strength

Difference of levels	Difference of means	SE of difference at 95%CI	Adjusted t-value	P-Value
80 °C-AR	69.1	14.0 (41.2, 97.0)	4.92	<0.001
160 °C-AR	22.5	14.0 (-5.4, 50.4)	1.60	0.113
160 °C-80 °C	-46.6	14.0 (-74.5, -18.7)	-3.32	0.001

SE = Standard error; CI = Confidence Interval

**Table 7** Fisher individual tests for differences of means: stiffness

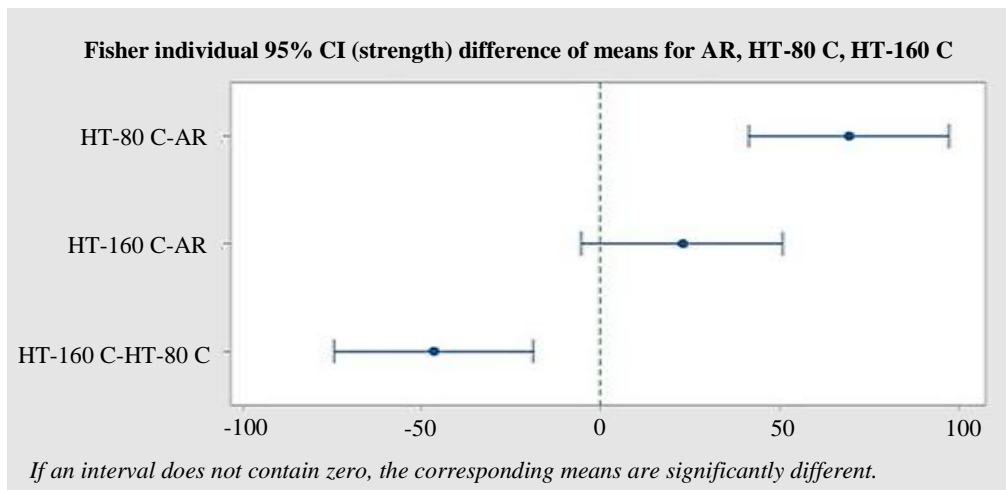
Difference of levels	Difference of means	SE of difference at 95%CI	Adjusted t-value	P-Value
80 °C-AR	0.376	0.351(-0.322, 1.073)	1.07	0.287
160 °C-AR	0.636	0.351(-0.062, 1.333)	1.81	0.073
160 °C-80 °C	0.260	0.351 (-0.437, 0.958)	0.74	0.460

SE = Standard error; CI = Confidence Interval

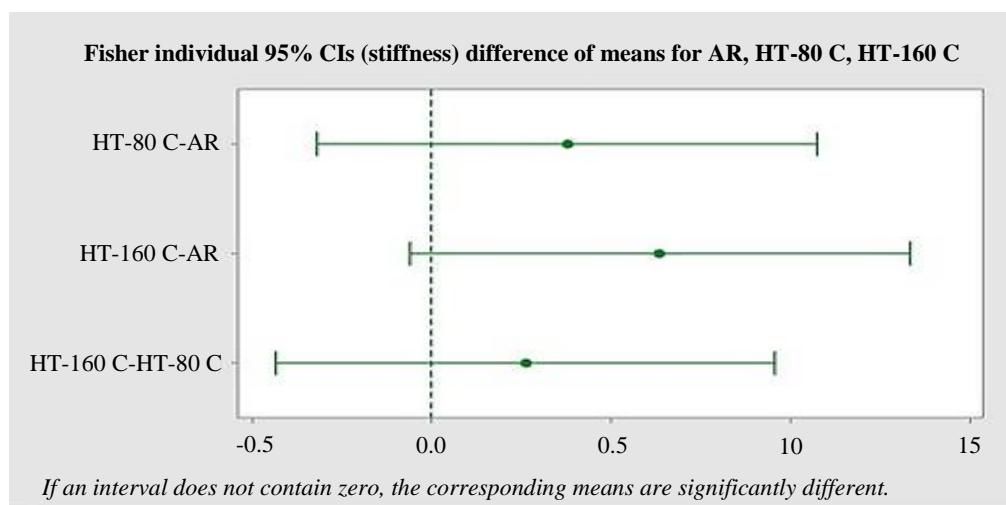
**Table 8** Fisher individual tests for differences of means: elongation at break

Difference of levels	Difference of means	SE of difference at 95%CI	Adjusted t-value	P-Value
80 °C-AR	9.40	2.26(4.91, 13.88)	4.16	<0.001
160 °C-AR	-10.57	2.26(-15.06, -6.09)	-4.68	<0.001
160 °C-80 °C	-19.97	2.26 (-24.46, -15.48)	-8.85	<0.001

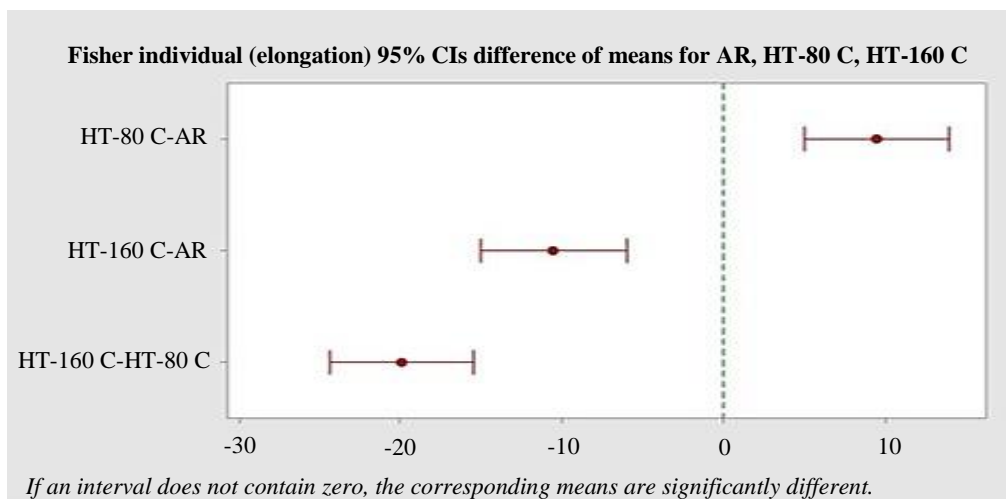
SE = Standard error; CI = Confidence Interval



(a)



(b)



(c)

**Figure 4** Fisher individual tests for differences of means for (a) strength (b) stiffness and (c) elongation at break.

### 3.2 Weibull distribution: median rank

The strength distribution of natural fibres was assessed using a Weibull continuous probability distribution [20]. The Weibull modulus is often associated with the degree of scatter in the data, which depends on the degree of flaws present in the fibres. The Weibull equation is given as:

$$F_{\sigma} = 1 - e^{-\left(\frac{\sigma}{\sigma_0}\right)^m} \quad (1)$$

where  $F_{\sigma}$  is strength distribution,  $\sigma_0$  is a scale parameter and  $m$  is the Weibull modulus. A higher Weibull modulus indicates fewer flaws and more consistent fibre strength.

Strength distribution  $F_{\sigma}$  is calculated using the median rank estimator shown in Equation (2):

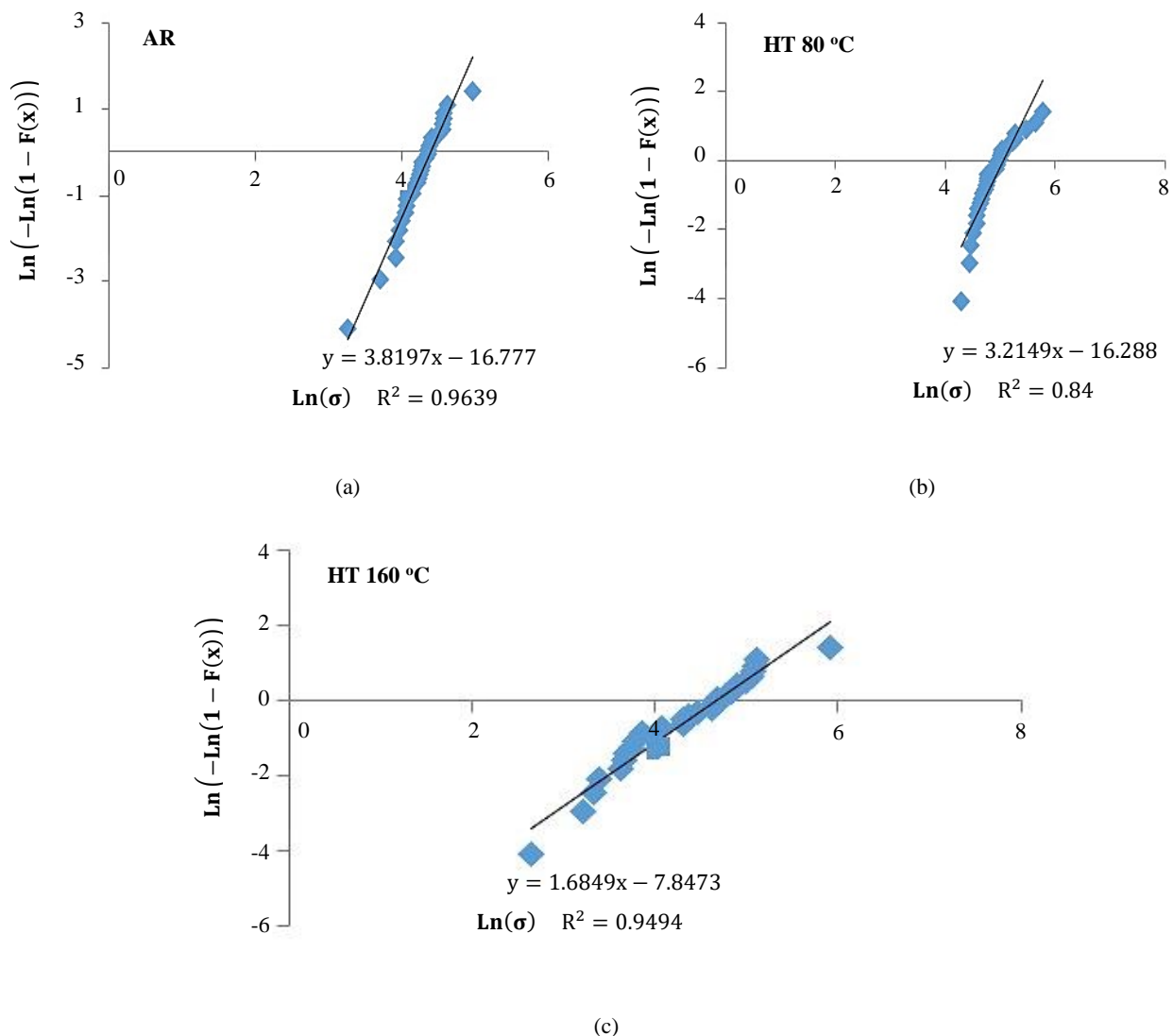
$$F_{\sigma_i} = \frac{i - 0.3}{n + 0.4} \quad (2)$$

where  $n$  is the number of samples assessed, in this case, 30, and  $i$  is the rank position. A summary of the Weibull parameters obtained from the coir fibre samples is given in Table 9 and graphically displayed in Figures 5(a-c). AR coir fibre has the highest shape parameter, 3.82, in comparison to HT 80 °C and HT 160 °C, while HT 160 °C shows the highest level of scatter with the lowest Weibull modulus, 1.68.

Figure 6 displays the survival probability of AR coir fibres. The survival probability of natural fibres, such as coir fibre, is equal to 1 minus the failure probability, or  $1 - F(t)$ . Survival probability is the fraction of the samples that remain intact during loading to a tensile stress,  $\sigma$ . The value of this function at  $\sigma = \sigma_0$ , where  $\sigma_0$  is the tensile stress at which 36.8% of the samples survive. It was found that  $\sigma_0$  for HT 80 °C > HT 160 °C > AR. This shows that fibres heat-treated at 80 °C have the highest tensile stress at the point where 36.8% of the fibres remain intact.

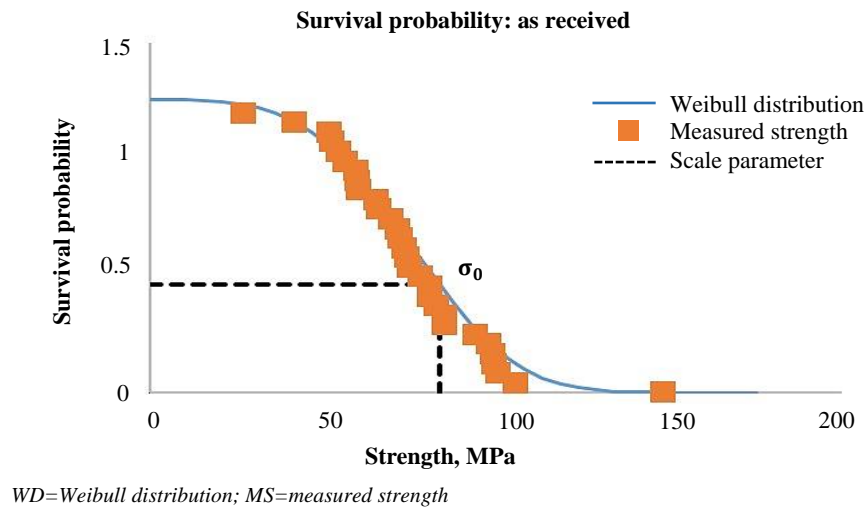
**Table 9** Weibull parameters of the coir fibre conditions

Fibre condition	Weibull shape parameter, m	Scale parameter (MPa)	R <sup>2</sup>
AR	3.82	80.82	0.96
HT 80 °C	3.21	158.60	0.84
HT 160 °C	1.68	105.36	0.95



**Figure 5** Weibull plots of (a) AR (b) HT 80 °C and (c) HT 160 °C

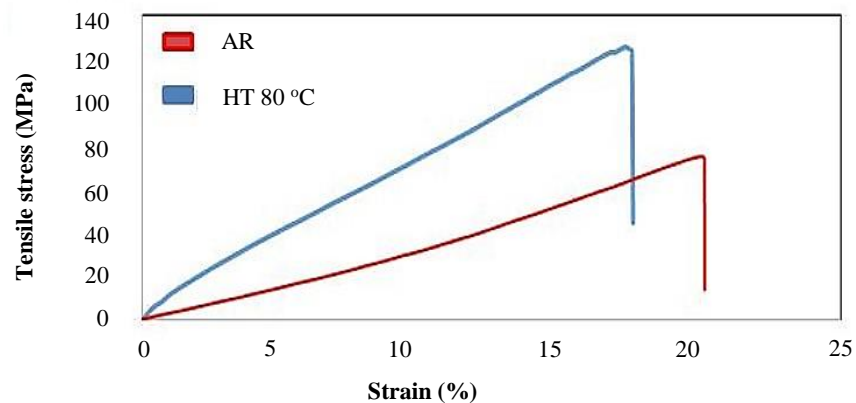




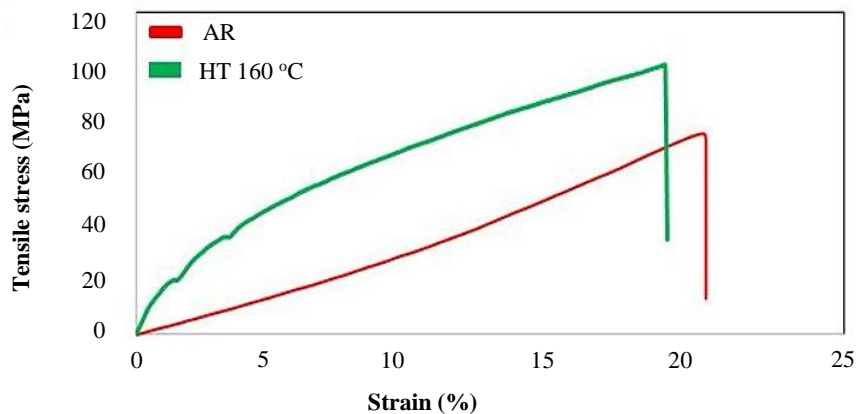
**Figure 6** The survival probability of AR fibres

### 3.3 Failure analysis/stress-strain curves of the tensile tests

The stress-strain curves of the untreated and the heat-treated coir fibres are shown in Figures 7(a, b), where variation in the shapes of the curves is seen. As-received coir fibres showed more linearity than the heat-treated material. Brittle failure was observed in all the samples. At 160 °C, the curve appeared more concave, which could be attributed to reorientation of the microfibrils [5]. At 160 °C, load drops are observed at low strains and then an increased elastic modulus was observed before fracture occurred. These significant load drops could have possibly resulted in some local stresses and hence a significant reduction in the tensile strength occurred with a heat treatment of 160 °C compared to that at 80 °C.



(a)



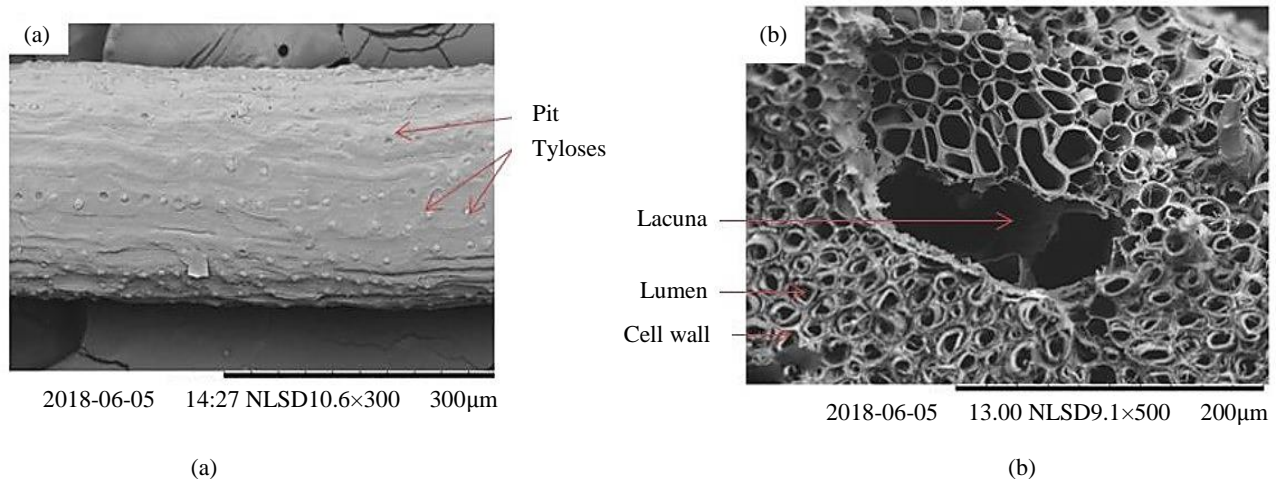
(b)

**Figure 7** Stress/strain curves of AR coir fibres compared with (a) HT 80 °C (b) HT 160 °C.

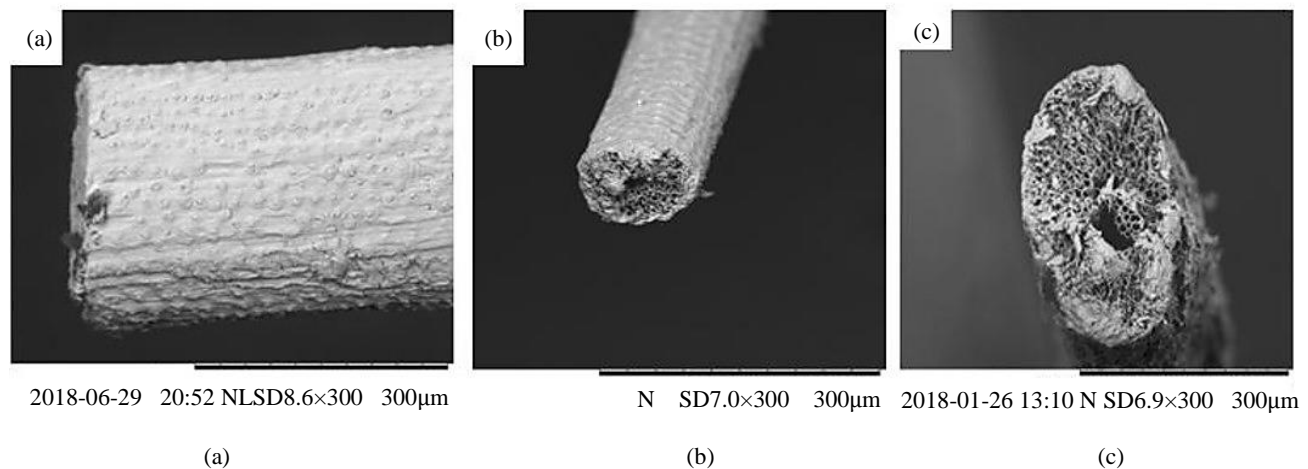


### 3.4 Scanning electron and optical microscopy analysis

SEM Images of as-received fibre samples are shown in Figures 8(a, b). The cross-section of coir fibres has a central pore (lacuna) and numerous lumens, making the fibres porous. Its surfaces consist of many tyloses and pits, giving it a rough appearance. Fracture plane images are shown in Figures 9(a-c). The fractured surfaces and the diameters of the fibres seem to vary. These variations could have led to the differences in the tensile strengths. High tensile strength has been attributed to low diameter [4, 15]. The failure pattern is a possible result of varying tensile properties. Some of the fibres were observed to be more brittle. Other fibres, exhibited premature failure at lower tensile strength values. Premature failure has been attributed to an increased number of defects [21]. The fracture surfaces of the HT 80 °C and HT 160 °C samples show evidence of microfibril splitting. This splitting is more pronounced in the HT 160 °C sample, which has been attributed to the softening of cellulose and other lower molecular weight components [6, 21]. Observation under higher magnification is necessary to examine detailed microstructural features of the heat-treated fibres.



**Figure 8** SEM Images of as-received coir fibre (a) surface and (b) cross-sectional images



**Figure 9** Fractured coir surfaces (a) AR; (b) HT 80 °C (c) HT 160 °C. The scale bar is 300 µm long.

## 4. Conclusions

The average tensile strengths of as-received coir fibres and coir fibres heat-treated at 80 and 160 °C are 73.03, 142.12 and 95.51 MPa, respectively, under the given test parameters. *F*-tests revealed that heat treatment influences the strength and elongation of as-received coir fibres. However, *F*-tests confirmed that heat treatment does not influence their stiffness. The strength distribution exhibited by the fibres under each condition was assessed using Weibull parameters. They show that AR coir fibres have the highest shape parameter, 3.82, of the three samples with a survival probability of 0.368, for a sample gauge length of 20 mm and a cross-head speed of 2 mm/minute. The observations also show that heat treatment introduced structural changes within the fibres.

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