Variation in the Inter-Node Length During Plant Growth Influences the Tensile Strength of Maize Stem Fibres.

Hareenanden Ramasawmy (haree@uom.ac.mu)
University of Mauritius

Jaykumar Chummun
University of Mauritius

Piyushaa Devi Emrith
University of Mauritius

Research Article

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Abstract

This study describes an investigation of the evolution of the mechanical and chemical properties of maize stem fibres with the growth stages of the plant, and how the tensile strength is influenced by the presence of nodes along the fibre length. Furthermore the variation of the tensile strength and chemical functional groups among four common maize varieties were determined. In this context, the fibres were characterised by performing tensile test, density & linear density tests, Fourier Transform infra-red spectroscopy (FTIR), X-ray diffraction (XRD), Thermo-gravimetric analysis (TGA) and surface morphology (SEM image analysis). The fibres were all extracted manually, and in some cases preceded by a water retting process for ten days. The thermal analysis, FTIR and x-ray results showed that in general the fibres from the different maize varieties and from the different growth stages are semi-crystalline in nature. The SEM micrographs revealed the presence of equi-spaced nodes along the fibre length, which are believed to be due to the growth stresses induced in the plant stem. The inter-node distance varied in relation to the growth stage of the plant, and yielded a good correlation (coefficient of 0.91) with the tensile strength of the fibres. Finally a better fibre yield was obtained from the stem at the senescence stage of the maize plant.

Statement Of Novelty

This study attempts to explain the evolution of the tensile strength of fibres extracted from various growth stages of maize stem through mechanical, chemical analysis, thermal analysis, x-ray diffraction and surface morphology (SEM). The information derived from the study can help to harvest fibres at the appropriate time in order to obtain better mechanical strength. The results have shown that though all the fibres exhibited a semi-crystalline structure, the tensile strength is related to the presence of nodes along the fibre length. These nodes are not typical fibre defects/damage but rather deformation during the growth of the plant. The higher the frequency of occurrence of the nodes along the fibre length, the higher was the resulting tensile strength.

Introduction

Maize is the second highly grown agricultural crop in the world, with about 50 maize varieties available on the world market [1]. On the African continent and other SIDS-African countries such as Mauritius, there are hybrid maize varieties which have good tolerance to maize diseases as well as good resistance to droughts and heat. Four maize varieties chosen for this study, notably, local, SC602, SC608 and SC719, were those available locally in Mauritius. SC602 and SC608 are both medium maturing maize strains producing yellow maize grains while SC719 is a late maturing maize strain which produces white maize grains [2]. Both SC608 and SC719 are among the leading maize varieties on the African continent.

Following maize cob harvest, the remaining maize stover has very little secondary uses such as livestock feed and green manure or is simply considered as a waste. However, this can be a good source of cellulosic fibres. Furthermore, the few experimental-based research studies [3-9] on maize fibres done till
date have all shown that maize fibre has proved to be interesting by reason of its appreciable mechanical and chemical properties. From literature, it has been noted that extraction of maize fibres has been done by using either chemicals accompanied by boiling or milling equipment [3-5, 7, 9]. Thus, the economical aspect of such fibre extraction has not been considered – which would seriously affect the scale up of such process. However, there are no explicit characterization of maize fibre in terms of its mechanical and chemical properties. Hence, studies on the maize fibre, itself, are relatively scanty.

There is significant studies in the literature about the effect of fibre defects and fibre deformation particular for wood pulp fibres on the mechanical strength. However, to the best of the knowledge of the present authors, there are no known study about the evolution of fibre deformation during the growth stages of the maize stem, and the impact on the tensile strength (TS) of the fibres. Moreover, the evolution of the mechanical properties of maize fibres during the growth of the maize plant has not been investigated. This would be useful given that baby corn cultivation is very profitable by virtue of its high nutritional value, good taste, short harvest time and its ability to give good returns in short periods of time [10]. Furthermore, the influence of maize variety on the fibre is not available in literature.

This study aims at presenting the findings for the room temperature maize stem fibre extraction process, and the evolution of the fibre yield, tensile strength and chemical properties of maize fibres extracted from different maize plant species at specific stages of their development. Furthermore the effect of fibre deformation in terms of nodes/crimps have been evaluated over the four stages of the stem growth and related to the fibre tensile strength.

**Material & Methods**

**Supply of plant material**

The Food and Agricultural Research and Extension Institute (FAREI), Mauritius, supplied the necessary maize plant materials at the four different growth stages of the maize plant. The first stage, called the ‘Young Stage’, occurs at 1 month after sowing. Then, at 2 months, baby corns start to be produced and thus, this is named as ‘Baby Corn Stage’. At 3 months, the baby corns grow into mature corns, denoted as ‘Mature Stage’. Ultimately, at 4 months whereby all the corns have been harvested, the maize plant becomes dry: this stage is termed as ‘Dry Stage’.

The maize plants were planted in a split plot design over an area of 474 m². 612 maize plants for each of the four varieties were cultivated.

**Fibre extraction, Yield, Weight, Fibre dimensions**

**Fibre Extraction and Measurement of Yield**
The maize stems were cut at their nodes. The chopped inter-node maize stems were weighed and then water-retted for 10 days, except for the dry stage fibres. After 10 days, fibres were manually extracted. They were subsequently oven dried at 60°C for 24 hours. The dry mass of the fibres was then measured, and the fibre yield was calculated.

Measurement of Density & Fibre Linear Density

The fibre density for each maize variety (at the Dry stage) was measured by using a Micromeritics Accupyc II 1340 fully automated pycnometer. The instrument used has a 10 cm³ sample cup with 3.5 cm³ insert. Before the tests were conducted, volume calibration was done using the 3.5 cm³ insert. After the calibration the sample was packed into the sample cup with the 3.5cm³ insert so that it was at least 2/3 full and the weight was recorded. The sample was put in the instrument sample chamber and the volume of the sample was measured. Helium was used as gas. The density reported for each sample is an average of five measurements. All the tests were done at the Institute of Applied Materials, University of Pretoria, South Africa.

A Crimp Tester (Model: Eureka; Type: EY07) was used to measure the linear density of the fibres. The length of each fibre for a set of 20 fibres for each sample was first measured. The mass of the bunch of 20 fibres was then measured. The linear density was then computed as follows:

\[
\text{Linear Density (Tex)} = \frac{\text{Mass of 20 fibres}}{\text{Sum length of 20 fibres}} \times 1000
\]

Tensile Testing

Tensile tests of the fibres were carried out using a universal machine Testometric M500-50AT equipped with a 10 kgf load cell and having a gauge length of 25.4 mm, as per the international standard ASTM C1557-03. A minimum of 50 individual successful tensile tests were performed for fibres from each stage of each maize species.

To determine the cross-sectional area of the tested fibres, 2 - 3 imprints at five different locations along each fibre were made on plasticine at room temperature. The images of the imprints were captured using a microscope (model: DigiMicro Profi at 5 Mega-Pixels) at a magnification of ×300. The areas of the imprints were computed using the ImageJ software. The mean area for each tested fibre was then computed based on the areas of the imprints obtained.

It is to be noted that for the two species most commonly grown locally (Local and SC608), maize fibres from the young stage were also tested in order to find any trend in the strength at the specific stages of growth of the plant.
FTIR, TGA, SEM & XRD Testing

The chemical functional groups of the extracted fibres were analysed using a Bruker FTIR Spectrometer in the range of wavenumbers from 400 to 4000 cm^{-1}. The FTIR graphs obtained were normalized before performing any interpretation.

The SEM micrographs were acquired using the ThermoFischer Apreo Volumescope FESEM at 2 kV accelerating voltage and 0.20 nA probe current using T1 and T2 trinity detectors with OptiPlan use-case. SEM micrographs were captured in TIF format at a resolution of 3072 x 2304 pixels. Image processing was carried out using the ImageJ software. Measurements were conducted after calibrating the software to each individual scale bar of the SEM image to ensure accuracy. All the tests were performed at the Central Analytical Facilities, University of Stellenbosch, South Africa.

The crystallinity data were obtained from a Bruker D2 Phaser X-ray diffractometer, with a copper X-ray source. The tests were done at the Department of Materials, University of Loughborough, UK.

Results & Discussion

Fibre Characterization

Yield & Weight Ratio

Based on the results of Figure 1, in general a higher mean % fibre yield is obtained at the Dry stage, with a lower extent of variation. Thus it is economically more viable for users to allow the stem to dry one month after the harvest of the maize cob before extracting the fibres. Even if the farmers need to remove the mature plant from the field after harvest, the mature stems should be kept for a period of one month until they are dried before proceeding to the fibre extraction process. It is to be noted that the fibre yield for only the local variety at the dry stage is low because spongy materials were still present at this stage, thereby decreasing the fibre yield. It is also interesting to note that a relatively high % fibre yield was obtained for the baby corn stage, with only a difference of 4% when compared with the Dry stage for the SC608 variety and a difference of 14% for the local variety.

Fibre Linear Density & Density

In this study, the fibre length is, in fact, the length from one node to another in one maize stem. Thus, the longer the internodes, the longer are the fibres. It is observed that SC608 Dry fibres are generally longer than fibres from other maize varieties with a lower variation as compared to SC719, and the fibres tend to be finer with a linear density of (12.43 \pm 2.19) tex. These results are comparable to those obtained by Yilmaz [9], who treated corn husk fibres by alkali method and obtained a minimum linear density of 16.1 tex. In fact, fibre fineness is important as it helps to decide whether the fibre is fit for use in apparels. The
average fibre fineness for the fibres in this study is 16.43 ± 4.26 tex (equivalent to 164.3 dtex). The fibre fineness for yarns used in the textile industry ranges from 1.6 dtex (for cotton) to 3.5 dtex (for wool) [11]. Thus, it can be inferred that maize fibres require treatment to increase their fineness.

The length of maize fibres compare well with fibres of other common natural fibres such as flax, 1.5-20.5 cm [12], coir 6-25 cm [13], bamboo, 22-27 cm [14].

Based on the pycnometer method, the SC 608 maize variety exhibits the highest density with the lowest coefficient of variation of 0.048 %. Despite having the highest density among the four maize varieties, SC 608 fibres are more appealing from an industrial point of view since the fibres tend to be longer and finer.

**Tensile Strength Results**

In general there is no a major difference in the tensile strength (TS) at the Dry stage of the four maize varieties; with the TS varying between 91 and 111 MPa (Figure 4). However, there is a marked difference in the tensile strength of fibres during the different growth stages, particularly for the local, SC 608 and SC 602 varieties. The TS for the baby corn fibres for the local and SC 608 varieties is very low, 74% and 77% % lower than the TS at the Dry stage for the local and SC 608 variety respectively. On the other hand for SC 602, the TS of baby corn is 60% higher than the TS at dry stage. The difference for the TS of baby corn and dry stage fibres is rather marginal, about 7 % for the SC719. The TS for the Young stage for SC 608 fibres is the highest of all the sample, but also the one with the largest standard deviation.

The analysis and interpretation of the TS results through FTIR, XRD, TGA and SEM are discussed in the next sections.

**Maize Fibres from the four growth stages of SC608**

**FTIR Results**

Figure 5 represents the FTIR spectra for fibres from SC608 variety from 800 to 1800 cm\(^{-1}\). The peak at 1730 cm\(^{-1}\), which corresponds to the C=O acetyl group in hemicellulose is hardly noticeable at the young stage (absorbance height: 0.00937). The peak at this wavenumber increases in prominence for the two later stages, Baby corn (absorbance height: 0.0176) and Mature (absorbance height: 0.0269) stages. Then it decreases for the dry stage (absorbance height: 0.0142),

The same trend is also observed for lignin which is characterised at peaks of 1510 cm\(^{-1}\) and at 1220 cm\(^{-1}\) [15,16]. The lignin content is lowest at the young stage (absorbance height at 1220 cm\(^{-1}\): 0.0111) as compared to the other stages (absorbance height at 1220 cm\(^{-1}\) for baby corn stage: 0.0240; mature stage: 0.0352; dry stage: 0.0346). A relatively comparable trend in the lignin content was observed by Longaresi *et al* [17] for fibres from different growth stages of cornstalk. Furthermore, the peak at 1420 cm\(^{-1}\), which
对应于无定形纤维素结构，最不显著的是幼年阶段（吸光度高度为0.0264）并且在其他三个阶段更为显著（吸光度高度：玉米 = 0.032；成熟 = 0.035；干燥 = 0.038）。

吸光度高度为897 cm⁻¹的峰通常归因于C-O、C-C和C-H伸缩振动存在于纤维素I型结构[18]。从图5，幼年阶段的峰（吸光度高度：0.023）与玉米幼嫩阶段的峰（吸光度高度：0.022）接近，这表明在这些两个生长阶段纤维素I的水平几乎相同。此外，纤维素I的含量在成熟和干燥阶段略有增加，因为其峰值略高（成熟阶段的吸光度高度：0.028；干燥阶段的吸光度高度：0.026）。

因此，观察到在玉米纤维的四个生长阶段中，无定形纤维素的相对含量较高，而结晶纤维素含量相对较低，这可能解释了四种生长阶段SC 608玉米的拉伸强度的主要差异。

**XRD Results**

XRD分析表明，在四个生长阶段中，SC 608玉米纤维的百分比结晶度没有显著变化（图6）。这些数值表明，纤维素在所有四个生长阶段均表现出半结晶结构。

一个清晰的峰在2θ = 22°附近获得，对应于纤维素I型的衍射面（002）而一个较宽的峰在2θ = 16°附近获得，对应于纤维素的晶格面（101）。

相对较低的%结晶度对于玉米纤维的四个阶段可以一般解释为相对于其他天然纤维如亚麻（71-81%）、大麻（68%）和黄麻（61-71%）[19]的拉伸强度的低值。然而，没有直接的正相关性可以解释在图4中四个生长阶段SC 608（图4）的拉伸强度和图6的%结晶度之间的关系。

**TGA Results**

从DTG曲线图7，第一个峰出现在100 °C以下，代表纤维素从纤维中去除的水分，这在幼年阶段高于其他三个阶段。观察到第二个峰发生在282.1 °C, 290.9 °C, 329.9 °C and 301 °C for the Young, Baby Corn, Mature and Dry stages respectively。乔治等[20]提到，半纤维素降解发生在200–265 °C的温度范围内，而迭泽等[21]报道半纤维素降解发生在200–300 °C（作为纤维素降解峰的变形）范围内。乔治等[20]报告了纤维素的降解

Page 7/21
took place over the range of 265–400 °C, and Diez et al. [21] also reported a rather similar range of 250–380 °C.

Based on the above literature, and the DTG curves shown in Figures 7a-d, it is observed that the second peak occurred at a relatively high rate varying between 7.6 and 9.9 %/min for fibres of the four growth stages. This tends to show that there was a rapid degradation of the hemicellulose and cellulose in those fibres. Furthermore, the second peak for the Mature stage fibres occurred at a higher temperature as compared to the Young stage fibres, which could have indicated a higher crystallinity of the cellulose in the fibres at the Mature stage but this is not supported by the XRD results of Figure 6. The second peak for the fibres at the Young stage occurred at the lowest temperature of 282.1 °C as compared to the fibres of the other three growth stages. Yet the fibres at the Young stage exhibited the highest tensile strength and a higher % crystallinity as compared to fibres of the Mature and Dry stages. Moreover, a small peak at 372.2 °C is observed only for fibres from the Baby Corn stage. According to Fiore et al. [16], degradation of α-cellulose occurred at 370 °C, and this could explain the third peak in Figure 7(b). Given that there are no peak at around 370 °C for the fibres from the Young, Mature and Dry stages, this can thus also explain and substantiate the highest % crystallinity of the fibres of the Baby Corn stage from the XRD results of Figure 6.

In most cases, a last peak is observed at around 430-455 °C which correspond to the degradation of lignin, and this degradation occurred at a much lower rate as compared to hemicellulose and cellulose.

According to Sacui et al. [22] molecular packing of cellulose extracted from wood samples are rather similar to the form I-α, but with more disordered and less uniform packing environments. The XRD results of Figure 6 and the relatively lower degradation temperature of cellulose as exhibited in Figures 7a-d tend to show that the SC608 maize fibres of the four growth stages have a lower proportion of highly crystalline α-cellulose, and a rather more disordered and less structurally packed cellulose molecules. However, it would seem that there is a variation in the % of the different native crystalline cellulose of the four growth stages of the maize fibres as well as a difference in the amount of amorphous cellulose (as shown by the FTIR results).

In summary, the FTIR, XRD and TGA analysis of maize fibres do not adequately explain the change in the tensile strength (TS) of these fibres over the growth stages.

**SEM image analysis of the effect of inter-node distance on the fibre tensile strength.**

The SEM images of SC608 fibres (Fig 8) revealed a highly prominent rib-like structure in all the four growth stages of the maize fibres. These rib marks tend to be equi-spaced, and more frequent, that is, with the smallest mean inter-rib spacing for fibres from the Young stage of growth (71.58 ± 18.08 µm) as compared to the largest inter-rib distance for the Baby corn stage (123.89 ± 27.34 µm). Figure 9 shows the results of the average inter-node distance for the four growth stages.
Page et al. [23] reported different types of fibre deformation and defects from wood pulp fibres such as dislocations, and nodes. Dislocations or slip planes are regions where the alignment of micro-fibrils have been modified, whereas nodes (or crimps/kinks) are defects of a larger scale and are regions where failure in the fibre has occurred due to bending and compression. In the latter case, there is delamination of the fibre wall due to very high compressive strain.

The arrows on the SEM image of Figure 10 clearly shows that the small swelling or protrusion of the fibre wall are like kinks on the wall surface. These deformations of the fibre wall are on the micron level (as shown in Figure 9), and could be categorised as node/crimps rather than dislocations. The crimped zone is rather similar to the stage IV, a triangular swelling (Figure 11), as reported by Wathén [24], who cited Page and Seth (1980).

The absence of any cracks at these node/crimps locations would suggest that the localised stress level has not exceeded the ultimate tensile strength of the fibre but sufficient enough to cause a plastic deformation leading to delamination and a swelling at these node locations.

According to Thumm and Dickson [25] the occurrence of such types of fibre deformation are principally due to the method(s) of extracting and processing the fibres, and are more present in mechanical processed fibres as compared to chemically processed ones. These authors further suggested that fibre deformation has an effect on the fibre strength. Joutsimo et al. [26] reported that the fibre deformation could also occur during the plant development due to growth stresses, and that there is a change in the fibre wall as well as breaking of the hydrogen bonds at these localised defect zones.

Bourmaud et al. [28] have reported that there is a decrease in the tensile strength of fibres which contain defects such as kink-band, knees, and dislocations. These authors mentioned that these defects occurred during the processing stages of fibres through the development of bending and compression stresses. Bourmaud et al. [28] also reported that defects observed on the surface of non-scutched flax fibres have probably developed during the growth of the plant.

In the literature some authors, Thumm and Dickson [25], Joutsimo et al. [26], and Kouko et al. [27] have used either the term ‘fibre defects’ or ‘fibre deformation’ or both when reporting on nodes/crimps.

In the present study, no mechanical method was used for the fibre extraction; only water retting and manual separation of the fibres, except for the dry stage fibres where no retting was required (since they were easily detached from the epidermal layer). Thus, it would seem that the presence of the fibre nodes/crimps could be principally due to the growth stresses generated along the maize stem. So it is important to differentiate fibre defects from fibre deformation, and in the present study, these nodes/crimps are defined as fibre deformation rather than fibre damage.

Based on the experimental data obtained in this present study and shown in Figure 9, there seems to be a relationship between the inter-nodes distance and the resulting tensile strength of the single fibre for the four growth stages. From Figure 12, it can be observed that at a smaller inter-node distance the fibre
tensile strength is a maximum (Young stage fibres). The correlation coefficient of the exponential equation \( y = 139.54 \) is 0.91. This tends to show that the presence of the nodes/crimps developed during the growth stages of the maize stem improves the tensile strength. Bourmaud et al [28] have mentioned that plants do response to external constraints such as wind or rain during their growth stage as well as due to the varieties in the gene pool. This response is known as thigmomorphogenesis and does have an effect on the mechanical properties of the fibres as well as affecting the quantity and stiffness of strengthening tissues.

Thus it is believed that the nodes/crimps observed on all the fibres are due to the plant’s response to environmental stimuli and/or due to the stresses induced due to specific developmental stages of the plant. One example of such a response is at the young stage where the plant tissues are more fragile and susceptible to stresses [29], and thus must respond strongly to survive difficult environmental stimulations such as windy conditions in the fields. This can lead to a strengthening of the tissues of the fibre wall, leading to the visible swelling at relatively shorter interval. This can thus explain the smaller inter-node distance on the fibres from the Young stage (Figure 9). On the other hand older tissues have a weaker thigmomorphogenetic response than young ones [29]. Hence the inter-node distance could be larger in length.

According to Kouko et al [27] the presence of microcompressions and dislocations along a fibre can cause a higher elongation before a break of the individual fibers. These authors have cited Hornatowska [30], who suggested that areas with disorders of the fiber structure such as dislocations or microcompressions behaved more elastically.

In the present study, the results of the individual fibre elongation showed that there was no significant difference in the % elongation of fibres from the four growth stages, with the mean values lying within 2.8-3.3 %.

**Maize Fibres from all four varieties at the Dry Growth Stage**

**FTIR Results**

From Figure 13 it is noted that peaks at 1730 cm\(^{-1}\), which corresponds to Hemicellulose, are of the same low level for all the maize varieties at the dry stage. Thus, this shows that the amount of hemicellulose at this stage, is almost negligible. Furthermore, at around 1510 cm\(^{-1}\), the peaks corresponding to lignin for Dry SC719 and Dry SC602 are most prominent and they are almost of the same height (0.04).

As for wavenumber 1220 cm\(^{-1}\), the peaks for all dry species almost overlap (absorbance height for SC608: 0.031; Local: 0.024; SC602: 0.033; SC719: 0.035). Thus, the extent of lignin is almost similar for the four varieties which, in turn, explains their relatively similar fibre yields (ranging from 4 to 7%).
The absorbance of the peak at 897 cm$^{-1}$ is almost the same for the four varieties, ranging between 0.02108 and 0.02667. It would imply that there is no significant difference the relative percentage of cellulose I for the four varieties of maize fibres. However, the peaks at 1420 cm$^{-1}$ corresponding to amorphous cellulose are most prominent for SC719 and SC602 varieties (same absorbance height of 0.060) and least prominent for SC608 (absorbance height of 0.036) and local (absorbance height of 0.043) varieties. This shows that the relative extent of amorphous cellulose is greatest for the two maize varieties (SC719 and SC602) and least for SC608 variety.

Given the narrow range of the TS for the four varieties of dry maize fibres, and the range of standard deviation, it is deemed that there is not a major difference in the TS of these varieties at the dry stage. However, it seems that the relatively lower % of amorphous cellulose does lead to a slightly higher TS for SC 608 fibres.

**Conclusion**

In this study, it has been observed that the maize SC 608 stem fibres exhibit a semi-crystalline structure throughout its growth stages until the senescence/dry stage. The XRD, FTIR and TGA analysis tend to confirm this finding, and this is reflected in a relatively low TS as compared to other common fibres such as jute and flax. The study of the fibre inter-node length has revealed a strong correlation with the TS, and the presence of the nodes can be attributed to the growth stresses induced by conditions such as wind regime, water uptake, type of soil, etc. The study also reveals no major difference in the TS of stem fibres from four different maize varieties, most of which are commonly used on the African continent. Finally based on the findings, it is recommended to extract fibres from the dry/ senescence stage of the maize stem since generally a better fibre yield as well as a better tensile strength were obtained.

**Declarations**

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**Figures**

![Figure 1](image_url)

**Figure 1**

Fibre Yield for Different Maize Varieties at Different Stages of Growth
Figure 2

Variation of Fibre Length (Black line with circular markers) and of Fibre Linear Density (Grey line with square markers) of maize fibres of Different Maize Plant Varieties from Young Stage to Dry Stage.

Figure 3

Density of maize fibres (g/cm³) for the Different Maize Plant Varieties at the Dry Stage.
**Figure 4**

Average Tensile Strength of fibres from each Maize Variety at the four growth stages.

**Figure 5**

FTIR spectra for fibres for the different growth stages of SC608 variety from 800 to 1800 cm⁻¹.
Figure 6

XRD percentage crystallinity for the four growth stages of SC 608 variety.

(a) Young  
(b) Baby Corn  
(c) Mature  
(d) Dry

Figure 7

TG and DTG curves for the four growth stages of SC 608 maize variety.
Figure 8

(a-d): SEM image of the four growth stages of SC608 fibre (×100).

Figure 9

Variation of Inter-node Length with fibres from different growth stages of the SC 608 maize plant.
Figure 10

SEM of a mature SC 608 fibre at 1000X showing the nodes.

Figure 11

The four states of fibres and the corresponding stress-strain curves (Page and Seth, 1980) cited by Wathén [24].
Figure 12

Correlation between Inter-node Length from SEM images and Tensile Strength of fibres from different growth stages of SC608 variety.

Figure 13

FTIR spectra for fibres from the different varieties at the Dry Growth Stage from 800 to 1800 cm\(^{-1}\).

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