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(REVIEW ARTICLE)

Techniques for measuring cross-sectional area of plant fibers: A mini-review

Komlavi Mawunyo GOGOLI, Kokouvi Happy N'TSOUAGLO *, Soviwadan DROVOU and Ayaréma AFIO

Department of Mechanical Engineering, Polytechnic School of Lomé, University of Lomé, Togo.

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Abstract

As more and more scientific studies look for sustainable industrial materials, plant fibres are increasingly recognized for their potential as environmentally friendly alternatives to synthetic fibres. However, one of the most important factors limiting their widespread use is the variability of their mechanical properties. One of the main causes of this variability is the difficulty of accurately measuring the cross-sectional area (CSA) of these fibres, since certain mechanical properties such as stress at break depend directly on it. Indeed, the determination of the CSA of plant fibres presents significant challenges due to the complexity of their morphology. This mini-review critically examines a range of methodologies employed to measure the CSA of plant fibres, from traditional optical microscopy to sophisticated digital reconstruction and automated laser scanning techniques. Each method influences the mechanical properties of fibres differently, affecting the reliability of results and complicating comparisons between different studies. As well as listing the different methods used, the study shows how the choice of measurement method can have a significant impact on the assessment of fibre mechanical properties. Finally, this review argues in favor of international harmonization of cross-sectional measurement methods for plant fibres, which could ultimately make the use of plant fibres more reliable in the industrial world.

Keywords: Plant Fibre; Cross-Section Area (CSA); Fibre Morphology; Measurement Techniques; Mechanical Properties; Method Standardization

1 Introduction

As part of the quest for industrial solutions in line with sustainable development, plant-based fibres are emerging as encouraging substitutes for petrochemical-derived materials. Their appeal lies mainly in their greater environmental suitability and competitive advantages in terms of specific properties. However, due to their micrometric dimensions, measuring the cross-section of these fibres is a complex task. Yet determining CSA is essential for assessing mechanical properties such as stress and stiffness.

However, there is no standardized technique for measuring cross-sectional dimensions or studying the morphology of plant fibres. Various techniques or methods are used by authors: optical microscopy [1–3] scanning or transmission electron microscopy [1,4], automated laser scanning [5,6], Fraunhofer diffraction[7,8], X-ray tomography [9], or mathematical modeling combined with experimental data [10]. Not only, the method used to measure cross-sectional area (CSA) influences mechanical properties [5,11,12], but the use of these various techniques makes comparison of results from different studies complex, as each technique entails specific biases [6].

The aim of this review is to present a comparative overview of the various methods used to assess the CSA of plant fibres. In doing so, it aims to facilitate the optimal selection of a technique in relation to available resources. In the first part, we examine the importance of determining the cross-sectional dimensions of plant fibres. In the second part, we

* Corresponding author: Kokouvi Happy N'TSOUAGLO

will describe the main techniques used for this purpose. It should be emphasized that the methods described are applicable to both elementary fibres and fibre bundles.

2 The importance of determining the CSA of plant fibres

The methods used to determine the cross-sectional dimensions (diameter and CSA) of plant fibres have a direct impact on the estimation of their mechanical properties. For example, tensile strength (Equation 1) is calculated from raw force/displacement data. This physical quantity, intrinsically linked to plant fibre geometry, depends directly on the calculation of CSA.

$$\sigma = \frac{F}{CSA} \tag{1}$$

Here, σ $[MPa]$ represents the tensile stress, F $[N]$ is the applied force, and CSA $[mm^2]$ denotes the cross-sectional area of the sample.

In the measurement of the CSA of plant fibres, three main questions arise. Firstly, it is necessary to determine the shape (circular, elliptical, polygonal, or other) to be considered in order to approximate the cross-sectional shape (Figure 1a & 1b).

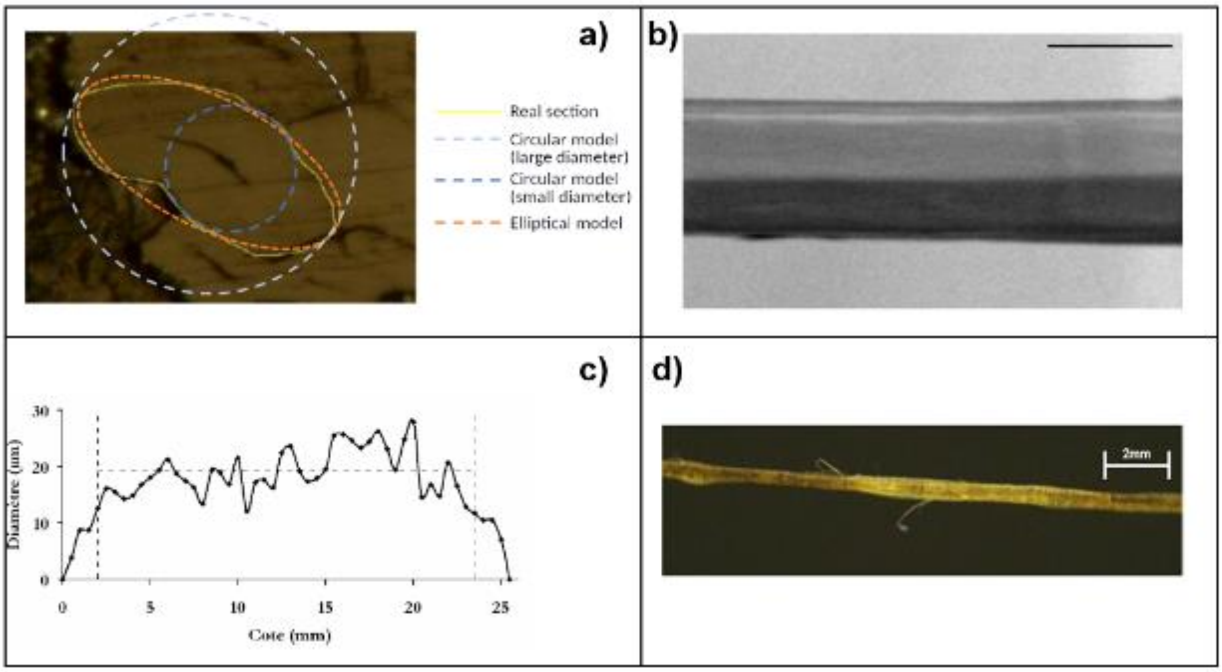


Figure 1 a) Considered models to represent the cross-section of a natural fibre [12]; b) Electron micrograph of a flax fibre highlighting the polygonal cross-section [1]; c) Diameter profile of a flax fibre observed under an optical microscope, adapted from [1]; d) Flax fibre bundles section seen in profile [13].

Traditionally, the assumption of a circular cross-section is often adopted, but studies have shown that the shape of the cross-section can vary according to the type of fibre, being more elliptical or at any rate more complex than circular. Thus, the simplifying assumption of a circular geometric model may not correspond to the morphological reality of plant fibres. Often taking into account the apparent dimensions (apparent diameter) of the fibre, enable us to derive an apparent CSA, as shown in Figure 1a.

Once this assumption about the geometric shape of the cross-section has been established, the second step is to choose the technique or technology used to determine this CSA. Also, as in the case of plant fibres, the CSA varies longitudinally along the sample (Figure 1c & 1d), it is necessary, in the simplest cases, to measure the area at several points and then calculate an average. This average is considered representative of the sample CSA ~~area~~ and can be used, for example, in Equation 1 to calculate mechanical properties. The third question, then, is this: ~~D~~depending on the length of the sample, how many measuring points are sufficient to obtain a fairly representative average area? In practice, a compromise often has to be found between the speed or the complexity of the technique used and the number of samples to be characterized. These three major issues ultimately influence the determination of the CSA of plant fibres, and consequently the mechanical properties derived from them.

Several scientific studies have investigated the influence of the geometric model or technique used on the cross-section area of plant fibres. For example, for the same batch of samples, the failure stress of the flax fibre bundle varies from (470 ± 147) MPa (cross-section measured by SEM) to (1465 ± 638) MPa (cross-section measured by high-resolution scanner), a total variation of 300% [6]. Garat *et al.* also demonstrated that using a circular geometric model to measure the CSA of flax fibres bundle could lead to an underestimation of tensile strength by a factor of 1.5 [5].

Moreover, morphological features such as the presence of a lumen are rarely taken into account during the calculation of plant fibres CSA. This situation highlights the urgent need for standards and standardization of the method for measuring the cross-sectional dimensions of plant fibres, both at the scale of the elementary ~~individual~~ fibre and at that of the fibre bundle. This will enable a more reliable comparison of the various studies on the subject and, by extension, facilitate more accurate dimensioning of biosourced structures.

3 Review of different methods for measuring cross-sectional dimensions of plant fibres

Historically, most research into the morphology of plant fibres has taken a simplified approach, considering the fibre as a circular, uniformly shaped object, although in reality its morphology is more complex. However, to better represent this morphological reality, a growing number of studies have explored hypotheses on cross-sectional shape, ranging from simple (circular) to complex (elliptical, polygonal, etc.) [2,5,6,12]. To simplify understanding of our work and in line with the classification adopted by Garat [14], we will begin by presenting studies that have adopted a circular shape hypothesis. Next, we'll look at studies that have taken into account the complexity of the geometric cross-section by considering more complex geometric model assumptions.

3.1 Studies using a circular model to approximate the geometric cross-sectional shape of plant fibres

Given its simplicity, the assumption that the cross-section of plant fibre is circular and longitudinaly homogeneous is the most widely used in the literature. According to Garat [14], it is based in particular on standard NF T25-501-3, established in 2015, which defines the methods for determining tensile mechanical properties such as tensile strength, strain at break and stiffness ~~modulus of elasticity~~ of elementary fibres and bundles of flax fibres.

3.1.1 Gravimetric Method

The gravimetric method assumes that the plant fibre is a cylinder with a constant circular cross-section. Knowing the density ρ of the plant fibre and the length L of the sample, it is possible to calculate the CSA by dividing the mass m of the sample by its average density:

$$CSA = \frac{m}{\rho L} \tag{2}$$

This technique is rarely used in the literature. It was used by Defoirdt *et al.* to determine the CSA of individual coir, bamboo and jute fibres [15].

3.1.2 Optical methods

In this article, we use the term "optical method" to encompass all techniques that make use of optical means, whether more or less advanced.

Measuring apparent diameter with a microscop

In the literature, the predominant technique for measuring the cross-section of plant fibres is optical microscopy (OM) [16–21]. This method involves measuring the apparent diameter of the fibre at several points (three points minimum) distributed along its length, then calculating an average apparent diameter d_m. With the plant fibre considered as a cylinder, the apparent CSA is then generally estimated using Equation 3.

$$CSA = \pi \times \frac{{d_m}^2}{4} \tag{3}$$

Increasing the number of measuring points leads to greater accuracy of the mean apparent cross-section. For example, in a study conducted by Placet *et al*. on hemp elementary fibres [22], the authors measured 10 apparent diameters distributed along the profile using polarized light optical microscopy (Figure 2).

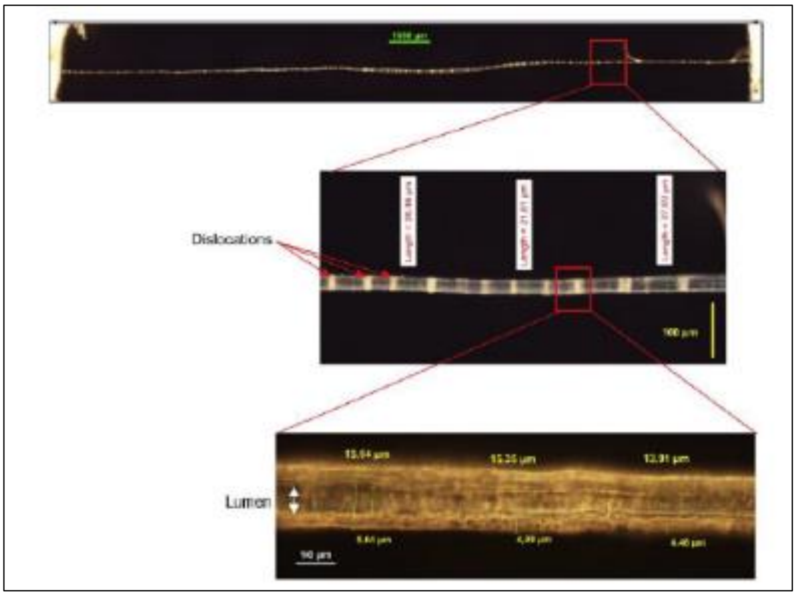


Figure 2 Polarized optical microscope image of a elementary hemp fibre [22].

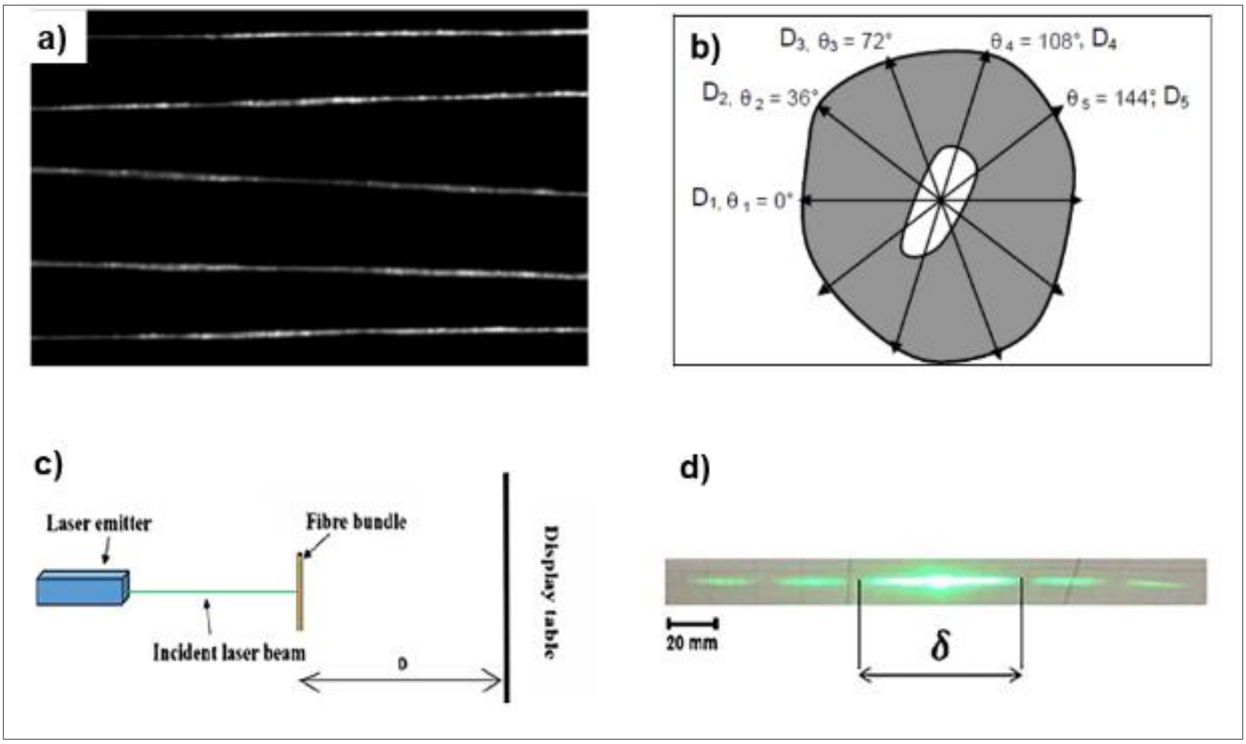


Figure 3 a) Microscopic images of an elementary hemp fibre for five different angular orientations; b) Cross-sectional profile of an elementary fibre and diameter measurements for five different orientations; c) Schematic diagram of the Fraunhofer diffraction principle and d) Diffraction pattern recorded on the display table [13].

However, measuring the apparent diameter of a plant fibre at a single angle is insufficient, as in reality the cross-sectional contour is not perfectly circular and presents irregularities (Figure 3a & 3b). To remedy this shortcoming, some authors have opted to measure the apparent diameter at various angles or by rotating the fibre in different orientations. By increasing the number of measurement orientations, the set of apparent diameters obtained provides a better approximation of the actual cross-section of the plant fibre. With this in mind, Ilczyszyn in his study of hemp elementary fibres, developed an innovative approach by proposing a method for measuring transverse dimensions for different angular orientations: 0°, 36°, 72°, 108° and 144° [2]. The method involves capturing images of the fibre along its length for each of these orientations using an optical imaging system, followed by a digital reconstruction of the profile. Finally, an average apparent diameter, calculated from the collected values, is used to estimate the CSA from Equation 3.

High-resolution scanner [6,23]

In this method, plant fibres are laid flat and scanned in transmission mode to obtain a digitized image of the transverse profile at different resolutions. Image processing software is then used to measure the apparent dimensions using the coordinates describing the profile, the number of pixels making it up, and taking into account the size of a pixel in meters. As with other experimental methods, the CSA is estimated using Equation 2, assuming a circular shape and calculating an average apparent diameter.

Fraunhoffer diffraction

It involves focusing a laser beam of wavelength λ on the cross-section the user wishes to characterize. Then, a diffraction pattern composed of several spots is collected on a screen whose plane is perpendicular to the direction of the laser beam and placed at a given distance D from the sample (Figure 3c &3d). The apparent diameter of the cross-section is inversely proportional to the measured width of the diffraction pattern's central spot.

$$d = \frac{2D\lambda}{\delta} \qquad (4)$$

With:

λ: laser beam wavelength,
D: distance between sample and display screen,
δ: width of the central spot of the diffraction pattern.

The light diffraction method may be poorly suited to plant fibres, as the diffraction pattern may be slightly altered by the presence of defects on the sample surface. For example, in their study of hemp elementary fibres, Romão *et al.* compared this method with observation by optical microscopy, assuming in both cases a circular cross-section [8]. They concluded that the light diffraction method may be less suitable for plant fibres, as the diffracted image may also be influenced by the presence of surface defects or by the disentanglement of fibre bundles.

3.2 Studies based on a non-circular hypothesis

In practice, several studies have revealed that the cross-section of plant fibres is not circular and shows significant irregularity along the samples. In order to perform more realistic morphological analyses, it is now common practice to consider assumptions or geometric models to represent the cross-section, which are not circular but more complex, such as elliptical models or even digital reconstructions of the exact contours [2,5,6,12].

3.2.1 Optical methods

Digital reconstruction after optical microscopic observations [1]

Fibres are prepared by treating them with paraformaldehyde and ethanol, then embedding them in epoxy resin. Samples are cut transversely with a microtome and observed under a light microscope. Images of the sections are analyzed using a computer program developed in the laboratory. Each cell is reconstructed from its contours, and surface area and perimeter are calculated using geodesic expansion. The difference between the total surface area and the lumen surface area is used to determine the cell wall surface area (Figure 4).

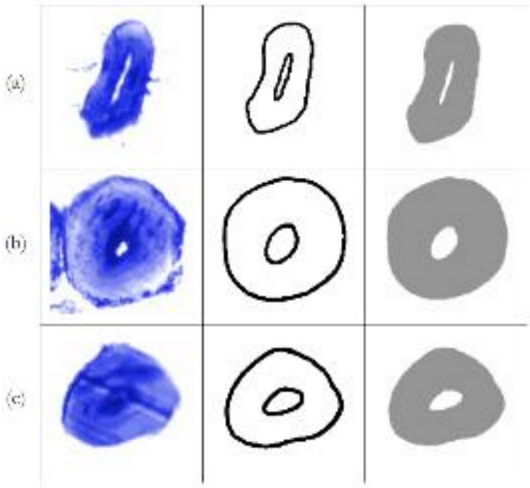


Figure 4 First column: optical micrographs; second column: decaled and digitized fibre contours; third column: reconstructed fibres

Microscopic observation after surface polishing [24–27]

After polishing the cross-section, scanning electron microscopy (SEM) was used to examine the morphology of the flax and sisal fibre bundles [26]. This method involves coating the plant fibre sample vertically in resin, then polishing the cross-section for microscopic observation. The results of this method were compared with those obtained by calculating a circular cross-section from the apparent diameters measured on photographs of the samples under the microscope. The results indicate that the use of a circular model leads to an overestimation of CSA and an underestimation of mechanical properties, such as stress at break and modulus, by a factor of two or more. In addition, this study revealed great variability in the cross-section of flax and sisal fibre bundles [26].

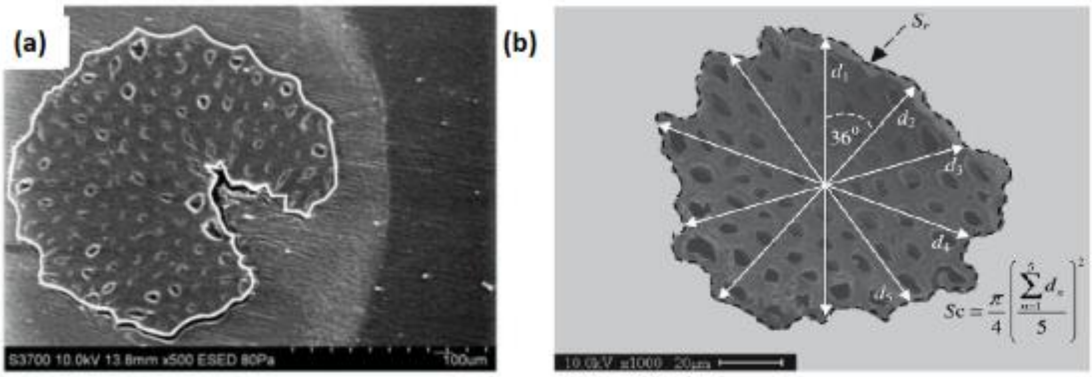


Figure 5 (a) SEM image of a section of a sisal fibre bundle [26] and (b) correlation graph between real area and area estimated from a circular shape assumption [28].

The polishing method was also used by Virk [27]. Scanning electron microscopy (SEM) was used to determine the actual CSA of jute fibre bundles. In this method, section contours were drawn manually instead, then image processing software was used to extract maximum, minimum and mean diameter values to model different shapes: circular, elliptical and convex (on Matlab®). The study examined 106 cross-sections. The results show that the calculation of the average area for an elliptical or convex model shows less variation and dispersion than that for a circular model. However, these values are still overestimated in relation to the actual mean value, with respective overestimations of 27.3 ± 15.6% for the elliptical model, 12 ± 5.2% for the convex model and 76.6 ± 47.1% for the circular model.

The polishing method was also used by Garat *et al*. to observe the cross-section of plant fibres such as nettle or sisal [5]. As illustrated in Figures 6b and 6c, the contour of the cross-section of the fibre bundle was manually cropped to compute its area. With 12 samples of fibre bundles and 10 consecutives sanding and polishing procedures, this results in roughly 120 measurements of CSA for each of the five plant species (sisal, palm, flax, nettle, hemp).

Direct Microscopic observation: Comparison between MEB and optical microscopic observations

In their study of sisal, ramie and kenaf fibre bundles, Munawar *et al.* compared two experimental techniques: the scanning electron microscope (SEM) and the conventional method using the optical microscope [28]. Initially, the mean CSA was estimated by optical microscopy based on five apparent diameters measured at 36° angular intervals, identified under the notation "S_c" in Figure 5b. Next, the real CSA, designated as "S_r" in Figure 5b, was deduced from direct SEM observations, by manually tracing the fibre outline from contrasting images using image processing software. The results obtained demonstrated that a coefficient, defined as the ratio between the CSA "S_c" obtained by optical microscopy and the actual CSA "S_r" deduced, could be applied. An underestimation of the CSA, varying from 4% to 8% depending on the botanical origin of the beam tested, was observed, with coefficients of between 0.92 and 0.96 respectively.

Laser scanning

The automated, non-destructive laser scanning measurement method is being increasingly adopted. It involves rotating and translating the sample in front of a laser beam, followed by monitoring the evolution of the apparent diameter for several sections along the fibre axis. This method offers several advantages. In addition to being able to apply the circular or elliptical cross-sectional shape assumption by measuring the maximum and minimum apparent diameters, it also enables several thousand diameters to be measured along the fibre profile, unlike conventional methods which are limited to a small number of measurement points. The smaller the rotation step and the smaller the translation step, the more this method can reconstruct the fibre's three-dimensional shape. While this method is effective in capturing the fibre geometry, it can induce acquisition errors due to the presence of surface defects or disentanglement, especially in the case of fibre bundles.

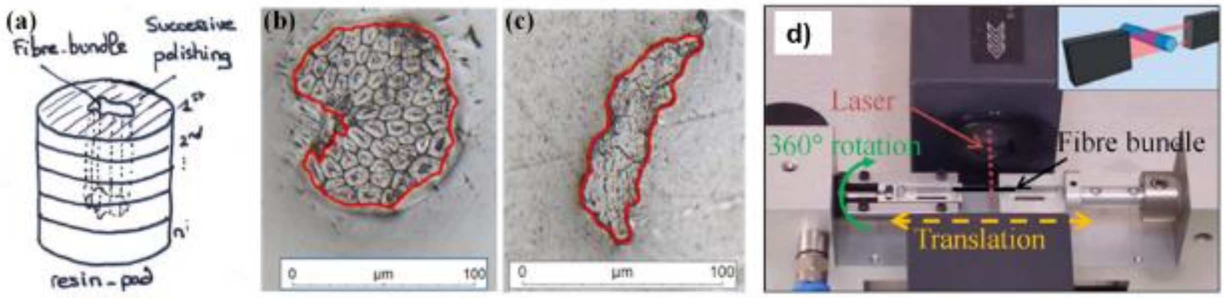


Figure 6 (a) Scheme of an embedded fibre bundle in an epoxy resin-pad; Manual outline of the fibre bundle perimeter with the image processing software (Archimede®, Microvision Instruments, France) for (b) sisal an (c) nettle; (d) FDAS device principle [5].

The automated laser scanning method is versatile, as it can be used for both circular and non-circular hypotheses. However, because of its ability to measure several diameters per unit of time, it is often preferred in the context of non-circular hypotheses. Moreover, it is frequently used to compare results obtained with the circular hypothesis.

Bourmaud *et al.* applied this approach to evaluate the apparent transverse dimensions of fibre bundles from date palms [29]. They made five rotational observations for each sample, enabling both a maximum (D_{max}) and minimum (D_{min}) apparent diameter to be determined for each section. These measurements were then integrated into Equation 5 to estimate the CSA of the section under consideration, using an elliptical model. The maximum apparent diameter is taken as the major axis of the ellipse, and the minimum apparent diameter as the minor axis.

$$CSA = \frac{\pi}{4} \times (D_{max} \times D_{min}) \quad (5)$$

This study also compared the results of the elliptical model with those of the circular model. The latter was based on five apparent diameters measured along the fibre bundle using an optical microscope. The results showed that the elliptical model provided a better representation of the actual shape of date palm fibre bundles. According to their study, using the elliptical model, based on five sections observed in rotation, would lead to an estimate of CSA around 14 ± 7% higher than that obtained with a circular model.

Using the automated laser scanning technique known as Fibre Dimensional Analysis System (FDAS) and optical microscope measurements, Garat *et al.* examined the morphology of flax fibre bundles [5] (Figure 7). The FDAS device moves and rotates the sample in front of a laser beam. By analyzing the cross-section, it is possible to determine an average apparent diameter from the data collected at different rotations. By adjusting the appropriate displacement, an average apparent diameter or cross-section can then be calculated for the sample. The study using the automated laser collected 45,000 apparent diameter values on beams 3 mm long. The median apparent diameter found was 91 µm (min = 49 and max = 140). In addition, characterization of 120 cross-sections, with 10 sections per fibre bundle observed under an optical microscope, yielded a median cross-section of 6148 μm^2 (min = 1929 and max = 11030). The work also revealed that for flax, hemp and nettle fibre bundles, using an elliptical model to calculate the cross-section leads to more accurate results than the circular model, which tends to overestimate the CSA.

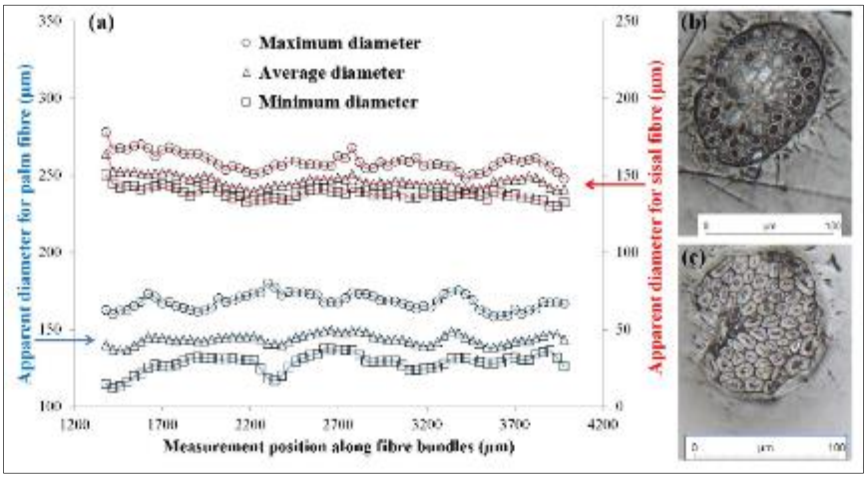


Figure 7 a) Lengthwise morphometric variations for palm and sisal fibre bundles from the FDAS, and typical cross-section of b) palm and c) sisal fibre bundles embedded in epoxy resin pad as observed by optical microscopy

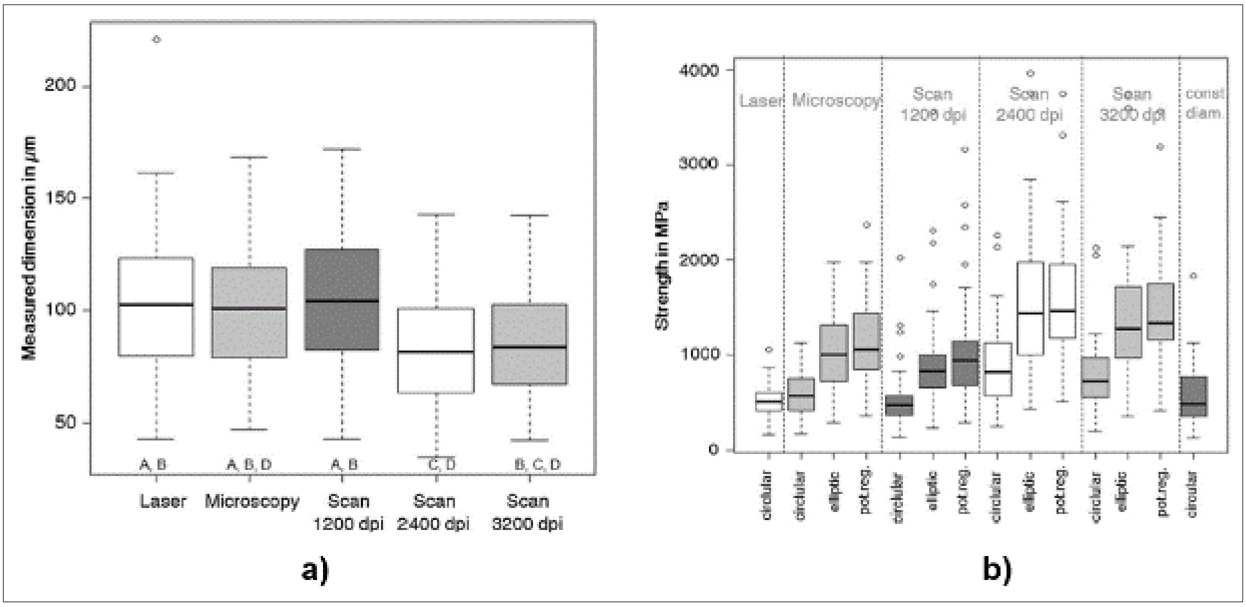


Figure 8 a) Influence of the cross-sectional geometric model on the calculation of the stress at break of flax fibre bundles; b) Effect of the technique applied to measure cross-sectional dimensions.[6]

Using FDAS, Haag and Müssig characterized the *CSA* of flax fibre bundles, comparing it with various measurement techniques such as scanning electron microscopy (SEM) and high-resolution scanner [6]. The authors also studied the influence of the geometric model adopted to calculate the cross-section. For the high-resolution scanner method and electron microscopy, circular and elliptical models were used, while the circular model was applied for laser scanning. The findings of the study show that the geometric model used has a significant influence on the stresses at fracture of the samples. For the same batch of samples, the stress at fracture varied from (470 ± 147) MPa (section measured by SEM) to (1465 ± 638) MPa (section measured by high-resolution scanner), a total deviation of 300%. The use of the automated laser, which allows the sample to be rotated, makes it possible to take better account of cross-sectional heterogeneities and gives a median diameter of around 100 µm. The choice of geometric model used to calculate the cross-section has little influence on the dispersion of diameter values (Figure 8).

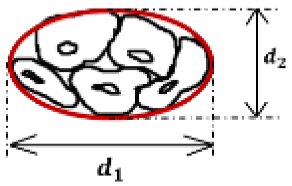
Fraunhofer diffraction

Fraunhofer diffraction is also used to characterize plant fibre from different angles, allowing non-circular shape assumptions to be applied. Recent studies have shown that the cross-sectional shape of flax and hemp fibre bundles is better modeled by an ellipse than by a circle. In their morphological analysis, Gogoli e*t al.* considered the cross-sections of flax fibre bundles to be elliptical [13]. To ensure reliable calculation of the CSA in accordance with the geometric definition of an ellipse, the axes d_1 and d_2 (minor and major axis) of the ellipse must be determined in two orthogonal directions. To achieve this, a device developed in the laboratory is used: first, the laser beam is focused on a point on the sample, and the width δ_1 of the central diffraction spot on the screen is measured. Then, keeping the beam focused on the same point, the sample is rotated 90° about its longitudinal axis. The width δ_2 of the central spot of the new diffraction pattern is then measured. This procedure is repeated for each of the six cross-sections characterized along the sample.

This gives a total of twelve axis measurements per sample. The two orthogonal directions, named "Direction 1" and "Direction 2", are fixed in advance and are therefore identical for all samples. The dimensions d_1 and d_2 of the ellipse axes are inversely proportional to the widths of the central diffraction spots δ_1 and δ_2 respectively, according to Equation 4.

Next, the CSA is determined using Equation 6 (Table 2). For comparison, circular cross-sections were also calculated. Specifically, for each cross-section, two circular cross-sections are calculated using Equation 7 (Table 1), considering the circular cross-section of diameter d_1 and d_2, respectively. A total of 100 samples were characterized and 593 cross-sections each represented by two axes d_1 and d_2 were analyzed.

Table 1 Geometric model and methods for calculating $\boldsymbol{CSA}$ [7]

Real cross-section	Geometric model	CSA Calculation formula	
	 Elliptical model	$CSA = \pi \times \frac{d_1}{2} \times \frac{d_2}{2}$	(6)
	 Circular model	$CSA = \pi \times \frac{d_i^2}{4}$ d_i being one of the axis d_1 or d_2	(7)

The simultaneous longitudinal evolution of the axes highlights the non-circularity of the fibre bundles. If the section were considered circular, there would be an average apparent diameter in two different directions. However, Figure 9a shows that the average apparent diameter depends on the direction of measurement, indicating non circularity of the cross-section. This confirms that the circular model is not suitable for plant fibres with high ellipticity in cross-section, such as flax and hemp, but would be better suited to bundles of sisal, palm and nettle fibres nettle, whose cross-sections are more circular [5].

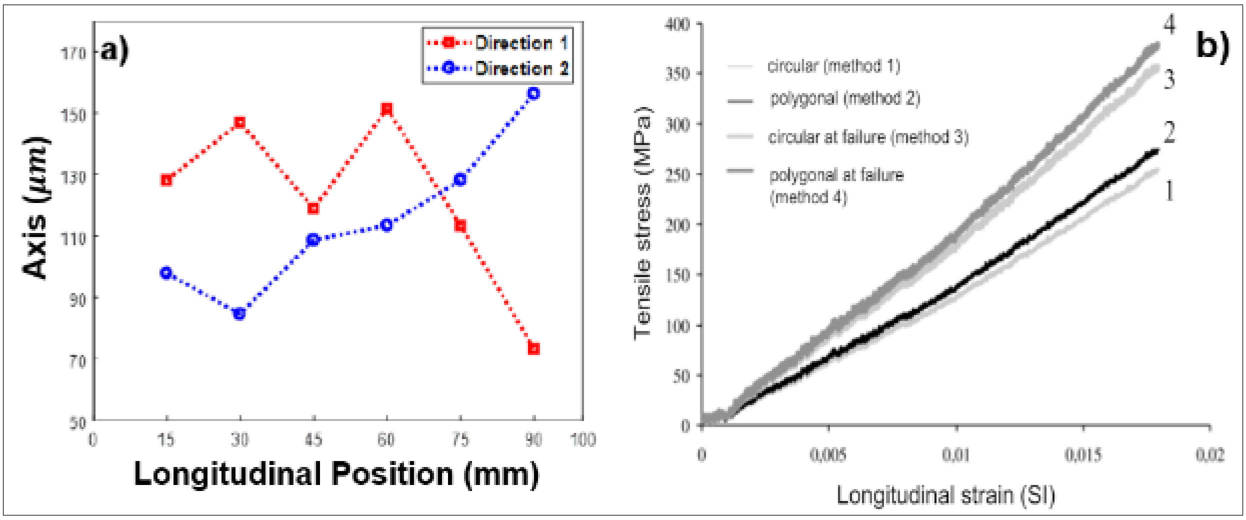


Figure 9 a) Longitudinal and simultaneous evolution of the axis of the cross-sections of a flax fibre bundles [7]; b) Evolution of tensile strength as a function of the method applied and the assumption of cross-sectional shape [30].

To assess the impact of the measurement direction on the mean apparent diameter of the fibre bundle, in the event that a circular model is used, the relative difference between the two mean apparent diameters obtained according to Direction 1 and Direction 2 was calculated for each of the hundred samples. This difference is approximately (20 ± 17) %. This means that the mechanical properties of the fibre bundle obtained from a tensile test using each of the mean apparent diameters will be affected by this difference. The random choice of the characterization angle by the experimenter, when the measurement depends on the orientation of the sample, thus becomes a source of variability, as several studies have pointed out [7,12]. This variability concerns not only the calculation of cross-sectional dimensions, but also the mechanical properties attributed to the plant fibre (be it the elementary fibre or the fibres bundle). Indeed, stress at break and Young's modulus depend directly on the average *CSA* of the sample.

The error introduced by using a circular model instead of an elliptical model to calculate the average *CSA* of fibre bundles was quantified. The results indicate that, on average, the circular model tends to overestimate the mean *CSA* fibre bundle cross-section by 1.13 ± 0.25 times compared to the elliptical model. These results confirm that the geometric model will directly influence the mechanical mechanical properties of the sample by calculating the stress at break and Young's modulus [26,31]. This dependence on the geometric model, if not taken into account, also contributes to the dispersion of mechanical properties reported in the various studies. According to Haag and Müssig the application of different geometric models can induce an error (Figure 9b) of up to 180% on macroscopic tensile stress calculation [6].

4 Conclusion

This mini-review of the methodologies employed to measure the *CSA* of plant fibres has highlighted the inherent diversity and complexity of measurement techniques – from traditional optical methods to sophisticated digital reconstructions – but also highlights the profound impact these methodologies have on the mechanical properties of plant-based fibres. It is clear that each method has intrinsic aspects that can significantly influence the results. However, it appears that the ability of a method to take into account the non-circularity of the cross-sectional contour and the longitudinal variation of the *CSA* is crucial. In this respect, our study highlights the laser scanning method as the most effective, allowing the sample to be rotated at a desired angle and translated to perform several longitudinal measurements. However, it should be noted that this method is time-consuming. Consequently, the choice of which method to use will depend on the precision required for the measurements, the specific objectives of the study, the number of samples to be characterized and, above all, the resources available to the experimenter.

In conclusion, this study calls for a harmonized approach to measuring the *CSA* of plant fibres, advocating the establishment of standardized protocols that would mitigate variability and promote comparability between studies. The adoption of such standards could make the dimensioning of plant fibre structures more reliable and catalyze progress in their use in industrial contexts, promoting their viability as sustainable alternatives to synthetic materials. Future research should continue to refine measurement techniques, ensuring that they are both accurate and accessible, while exploring the integration of new technologies such as machine learning for predictive modeling and improving the accuracy of morphological assessments.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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