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Effect of the Morphology Measuring Methods of Coconut Fiber on the Determination of Mechanical Tensile Properties

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ABSTRACT

This study is intended to improve the methodology to calculate the tensile properties of natural coconut fiber and increasing confidence in the use of sustainable natural fibers as reinforcing in composites used for different components in the naval industry. Coconut fiber is an agribusiness by-product that has been shown to have the potential for the development of new materials. The morphology of different species of coconut fiber was studied, and their mechanical tensile properties were determined. The coconut fibers were tested under direct tension in a universal testing machine, and the cross-sectional area of the fibers was calculated using images obtained in an optical microscope and a scanning electron microscope. The study assessed the incidence of key factors to determine coconut fiber mechanical tensile properties including different area measuring methods and the percentage of lumens present through an image analysis software called ImageJ. The results indicate that coconut fiber has a round-shaped cross-section, and the percentage of lumens is between 15% and 27%. Therefore, the effective area is reduced increasing the fiber's ultimate resistance to tension.

摘要

这项研究旨在改进计算天然椰子纤维拉伸性能的方法, 并增加使用可持续天然纤维作为增强材料用于海军工业不同组件的信心. 椰子纤维是农业综合企业的副产品, 已被证明具有开发新材料的潜力. 研究了不同种类椰子纤维的形态, 测定了椰子纤维的机械拉伸性能. 椰子纤维在万能试验机上进行直接拉伸试验, 并使用光学显微镜和扫描电子显微镜获得的图像计算纤维的横截面积. 该研究评估了确定椰子纤维机械拉伸性能的关键因素的发生率, 包括不同的面积测量方法, 以及通过名为 的图像分析软件显示的管腔百分比. 结果表明, 椰子纤维的横截面呈圆形, 管腔百分比在15%到27%之间. 因此, 有效面积减小, 增加了纤维的极限抗张力.

关键词

椰子; 纤维直径; 流明; 二值化; 力学性能; 最大应力; 杨氏模量

KEYWORDS

Coconut; fiber diameter; lumen; binarization; mechanical properties; maximum stress; young´s modulus

Introduction

The growing trend of new material for product development has drawn the attention of the naval industry for application in ship components. Colombia's national navy is promoting the development of a maritime cluster to foster greater interrelations between agribusiness, academia, and shipbuilders aimed at strengthening the development of this industry. Natural fiber worldwide studies have proven to be a prominent alternative as a partial substitute to traditional glass or carbon reinforcing material

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Table 1. Tensile properties of some various natural fibers (Chokshi et al. 2020).

Fiber	Tensile strength (MPa)	Young´s modulus (GPa)
Abaca	400–980	6,2–72
Flax	343–2.000	23,9–103
Bamboo	140–800	11–35,91
Hemp	270–1.110	3–90
Jute	187–800	3–55
Sisal	350–840	9–38
Coir	95–593	2,8–6

in composites. Although it is true, resistance and durability are unbeatable aspects, researchers are focusing on reducing the use of nonrenewable materials without considerable harm to the characteristics necessary for each application. Numerous studies have assessed the morphology, chemical composition, and mechanical properties of fiber (Chandramohan and Marimuthu 2011; Chokshi et al. 2020).

Natural fibers have several advantages compared to synthetic fibers including: lower levels of skin irritation and respiratory system during handling, less tool wear during processing, good recyclability, abundant supply, low cost, low density, high specific strength in relation to weight, non-toxicity, and good biodegradability (Chandramohan and Marimuthu 2011).

The morphology of natural fibers features irregular and nonuniform shapes around the longitudinal axis of the fiber. The conventional method to calculate the cross-sectional area of the fiber uses the area equation of a circle where the average diameter is obtained by an optical microscope or analysis software. This method produces inaccurate results with large standard deviations because it fails to consider the voids (called lumens) and the non-circular cross-section of the natural fiber's morphology (Carvalho et al. 2010). The function of these lumens is to serve as ducts for the circulation of certain nutrients. However, these lumens do not contribute either to the cross-sectional area or to its mechanical properties, and therefore should not be taken into account in the calculation of the area. Thus, the lack of an effective method for the morphological characterization of the natural fibers produces results with large standard deviations in the calculations of their mechanical properties, making it impossible to compare different types of fibers and to accurately predict the mechanical properties of their composite materials (Wei et al. 2009).

Natural fibers have different structures depending on the source, and consequently, the studies display wide variability of results in determining their properties. Perhaps the most important morphological aspect that influences the determination of a material's mechanical properties is the shape of its cross-section, since it defines the area and therefore the different stresses that the fiber supports during the mechanical tests. Many fibers may have circular or non-circular cross-sections, with different cell wall sizes and lumens (Alves et al. 2013).

Despite being the most influential feature, the methods used to determine the effective cross-sectional area of the fiber have not been fully studied to date (Munawar, Umemura, and Kawai 2007). In general, researchers assume three aspects for the calculation of this area: 1) that the area of the fiber is solid (without holes), 2) that the cross-section is circular, and 3) that the circular cross-section is uniform throughout the fiber length, and that the diameter remains constant (Thomason and Carruthers 2012). These assumptions lead to deviations in the real value of the mechanical properties of the fiber. Consequently, many of the properties of these fibers are erroneously characterized, which makes it difficult to determine their properties. One of the main consequences of this issue is the wide range of values for many mechanical properties of the same fiber (Pickering, Aruan, and Le 2016). Significant variations of the tensile property values of natural fibers have been reported (Table 1).

Table 1. Tensile properties of some various natural fibers (Chokshi et al. 2020).

The above indicates the necessity to improve methods and processes for obtaining morphological data for the study of the mechanical characteristics of the fibers in order to obtain more reliable and accurate values. Consequently, this research focuses on establishing the effect of lumens and reducing the percentage error in calculating the mechanical properties of the fiber.

Materials and methods

Materials

The coconuts were obtained from the Colombian Caribbean region, near the city of Barranquilla. This coconut variety, called Philippine, is large in size and has a yellow appearance. The coconuts were peeled manually to extract the fibers from the husk. Two groups were created: one was untreated, and the other was treated by submerging it in a solution with 5 wt% acetic acid for 30 minutes. Acid treatment removes hemicellulose and surface impurities (Verma, Lim, and Vimal 2020). In addition, being an organic material, acetic acid is used to prevent the growth of bacteria and fungi. Mechanical properties could improve with acid treatment on natural fibers, removing surface impurities and facilitating mechanical interlocking and the bonding reaction with matrix adhesion (Kommula et al. 2013). The extracted fiber was sun-dried for 24 hours, as shown in Figure 1.

Optical microscope (OM) and scanning electron microscope (SEM)

The characterization of the morphology was performed by means of an optical microscope (OM) and a Jeol JSM-5600 LV scanning electron microscope (SEM). For determining the minimum sample size, standard deviation was taken from a previous study (Kommula et al. 2013), and confidence level is 95%. The minimum sample size is 50. However, we prepared and tested 100 samples for each method and treatment. But, some samples were discarded because sample damage or test errors. Thereby, more than 50 samples were selected for longitudinal photos using the OM. Close to 200 samples were selected for SEM. These fiber samples were cut after freezing with liquid nitrogen. This prevents crushing the cross-section when cutting the fiber. The samples were covered with a thin layer of gold using a sputter coater prior to SEM scanning. Several fiber samples were taken in order to analyze their morphology. Other fibers were observed after the tensile test in order to analyze the cross-sections and determine their mechanical properties.

(a) Philippine coconut. (b) Extracted fibers. (c) Sun dried fibers.

Figure 1. Coconut fiber extraction untreated. (a) Philippine coconut; (b) Extracted fibers; (c) Sun-dried fibers.

Morphology measurement methods

Four methods were used to determine the area of the fiber.

(1) First method. The average diameter was determined from several measurements of the longitudinal diameter of the fiber, using photos taken by the OM. With this average diameter, the cross-sectional area was calculated, assuming a perfect circle.
(2) Second method. The second method is similar, but this time the average diameter is measured using the SEM photos. The average diameter is calculated by measuring diameters in different positions, using intervals of 36° between each measurement. The area is calculated according to Equation (1).

$$A = \frac{\pi}{4}\left[\frac{\sum_{i=1}^{n} d_n}{n}\right]^2 \tag{1}$$

(3) Third method. The cross-sectional area is determined directly by rounding the cross-section of the fiber, delimiting the circumference of the cross-section with a contour line. ImageJ software was used to calculate the area automatically.
(4) Fourth method. The area is estimated taking into consideration the lumens (hollow spaces) in the cross-sectional area through image binarization. ImageJ software was used to calculate the area excluding the lumens.

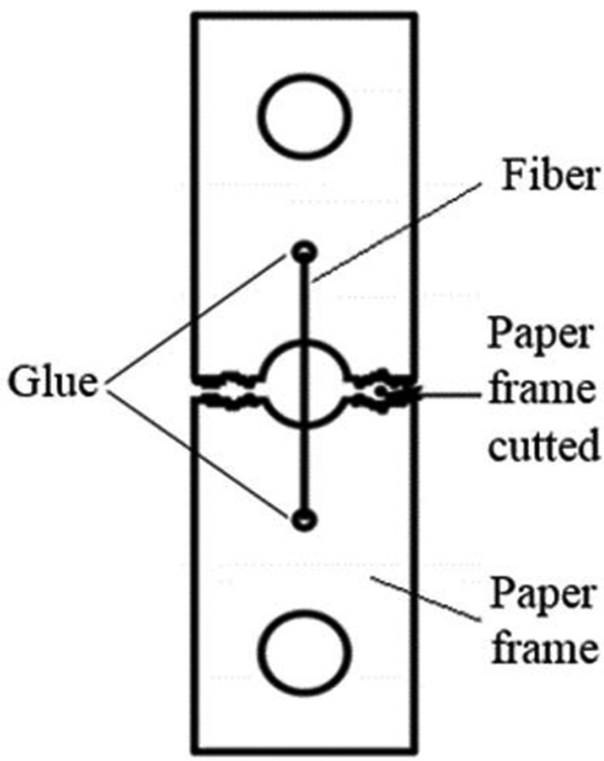

Figure 2. Preparation of specimens for tensile test.

Tensile test

Tensile tests were performed according to ASTM D3379-75 using a Shimadzu universal test machine AG-100NKX with a crosshead speed of 5 mm/min. To maintain consistency, fibers were glued individually to paper frames with 50 mm gauge length, as shown in Figure 2. The paper frame set-up was adopted to maintain consistency. Sides of the paper frames were cut with a scissor before the test to eliminate any pre-tension effects on the measured force.

To correct strain values for the slip effects between glue and paper, we had some considerations. Fiber-glued was delimited on the paper using a marker. Special metal grips were fabricated for pressing the fiber bonded section. After each test was performed, displacement of the fiber-glued related on marks were measured using a caliper. Finally, the elongation of the fiber can be corrected according to Equation (2).

$$\Delta L_C = \Delta L_{CHD} - \Delta L_{MC} \quad (2)$$

Where ΔL_C is the elongation of the fiber corrected, ΔLCHD is the cross-head displacement and ΔL_{MC} is the displacement caused by slippage measured with a caliper.

To obtain a representative set of results, more than 50 single fibers of each type were tested. During the test, the force-strain values were recorded and these values were used to measure the fiber tensile strength properties. All tested samples were analyzed by OM and SEM, and average values and standard deviations were reported.

Statistical analysis

Based on the numerical results obtained for each area, the yield and tensile strength were determined. Since a strength value was calculated for each method, four strength values are reported for each tested fiber. Data obtained from the physical and mechanical measurements were statistically assessed using analysis of variance ($P < 0,05$). ANOVA was run using IBM SPSS Statistics 23,0. These tests display significant differences between the four strength values obtained. Finally, an additional test was performed to prove whether the diameter obtained by OM can be used without losing precision and accuracy in the results instead of using the diameter obtained by SEM.

Results and discussion

Morphology analysis

The shape of the longitudinal fiber was observed using an optical microscope. For the scanning electron microscope, both longitudinal and transverse photos of the fibers were obtained, which provide detailed morphological information. During this process, one OM photo was taken of more than 50 samples, and one or two SEM photos were taken of more than 150 samples. However, not all photos were used for the analysis, because in many cases the fiber was not in good condition, and some had been crushed.

For each fiber, all four measurement methods are displayed in Figure 3. The average diameter was 394,3 μm determined by OM (Figure 3a). However, the average using method 2 was 259,3 μm (Figure 3(b)), which reduces the cross-section area by 2,29 times. Applying methods 3 and 4 (Figure 3(c) and 3(d), the cross-section area decreases even more by up to 2,77 times, from 0,122 mm^2 in method 1 to 0,044 mm^2 in method 4. Smaller values of diameters and cross-sectional area were determined using method 4, as shown in Table 2. This correction affects the determination of the mechanical properties of the fiber.

Table 2. Average diameters and cross-sectional area determined by different methods (standard deviations in parentheses).

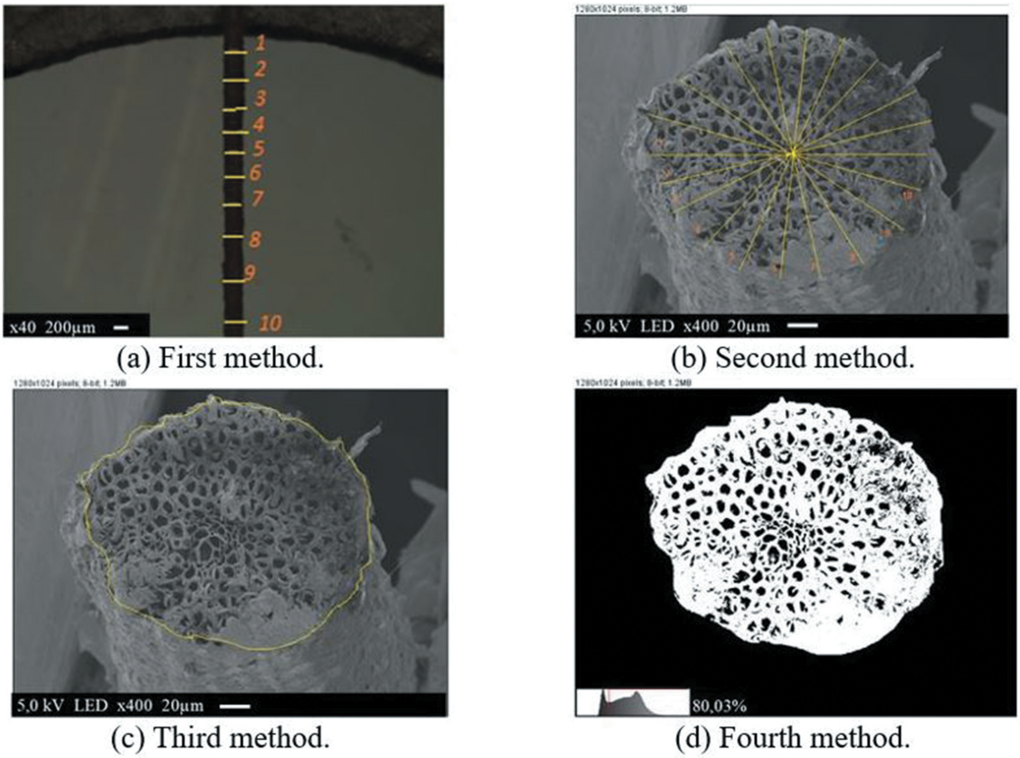

Figure 3. Morphology measurement methods.

Table 2. Average diameters and cross-sectional area determined by different methods (standard deviations in parentheses).

Method	Average diameter (μm)	Cross-sectional area (mm^2)
First	394,3 (92,97)	0,122 (7,33x10-3)
Second	259,3 (34,95)	0,053 (1,81x10-3)
Third	259,3 (34,95)	0,049 (1,95x10-3)
Forth	259,3 (34,95)	0,044 (2,18x10-3)

The second method was used to identify morphological distribution. One hundred images were selected through SEM observation for further morphological analysis. It was found that the average diameter is 259,3 μm, and the distribution is presented in Figure 4.

As for the measurements taken with the OM, it was observed that for Philippine coconuts the OM/SEM diameters ratio is less than 1,5, indicating that OM measurements will produce 1,5 times the diameter observed with SEM images (as shown in Table 3). These results indicate that the optical microscope does not provide reliable measurements to determine the diameter of a fiber, as it overestimates the actual diameter.

Table 3. Comparison of average diameters.

The linear density is an important parameter related to the diameter of the fiber. The linear density of coconut fibers was obtained by fiber weighing method, defined in ASTM D1577. Many of the samples were stored for 48 h at 21°C and cut at 1 in length. After that time, some groups of fiber were weighed on an analytic scale (Adventure pro, Ohaus), and densities were determined by calculating the rate between fiber´s weight, length, and number of fibers in each group. The linear density of coconut fiber is 71,61 Tex (SD = 15,60 Tex). Finally, the estimated diameter of coconut fiber was calculated using Equation (3).

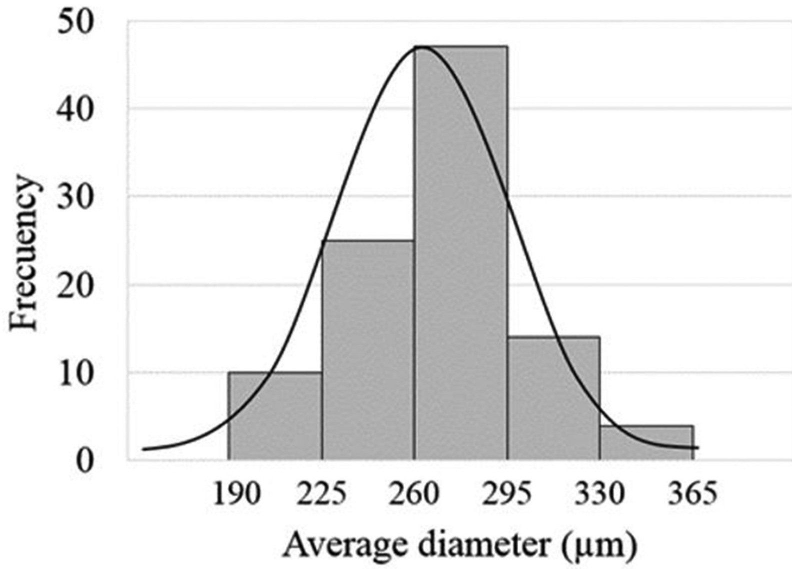

Figure 4. Average diameter distribution using SEM.

Table 3. Comparison of average diameters.

Method	Average diameter (µm)
OM	394,3 (92,97)
SEM	259,3 (34,95)
Estimated	274,0 (29,74)

$$d_{estimated} = 1000\left(\frac{T}{\sqrt{\pi\rho}}\right) \quad (3)$$

Where T stands for linear density in Tex and ρ is the volumetric density of coconut fiber (1,2 g/cm^3), in this case, it was taken from results reported by (Yusoff, Takagi, and Nakagaito 2016). Table 2 shows the average diameter for coconut fibers using SEM and calculated from density values.

A typical micrograph of an untreated Philippine fiber is shown in Figure 5. The cross-section has a rounded appearance, with clearly defined fiber cells and lumens. The SEM images of the cross-sections of flax, jute, ramie, and sisal fibers observed by (Hamad et al. 2017) are similar. Fibers exhibit a variation in terms of the fiber cross-sectional area, internal lumen shape, and size. This variation affects the tensile properties of the fibers. Lumens are empty spaces that could act as stress concentrators, and the fiber cells would be the ones that transmit and support the loads on the fibers.

The percentage of lumens (hollow spaces) was determined for 21 samples and results are shown in Figure 6. These generally range between 15% and 30% area of the fiber's cross-section. Therefore, calculating the area as if the fiber were solid (without lumens) over-estimates the actual area, which in turn produces calculated resistance values that are much lower than the actual resistance.

The porosity of coconut fibers is determined for many samples. Assuming that the fiber cross-section and the lumen are uniform along the fiber. The porosity of the fiber is then calculated based on the percentage of lumens and the total area of the fiber cross-section. The results show that the fiber porosity is in the range from 20,4 to 40,8%. Figure 7 shows the

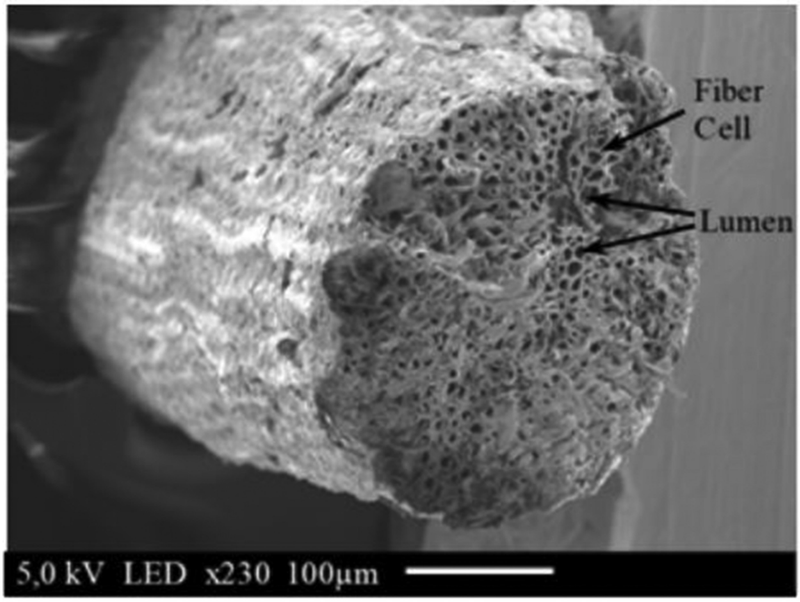

Figure 5. Scanning electron micrograph displays the typical shape of cross section of Philippine coconut fibers at x230 magnification.

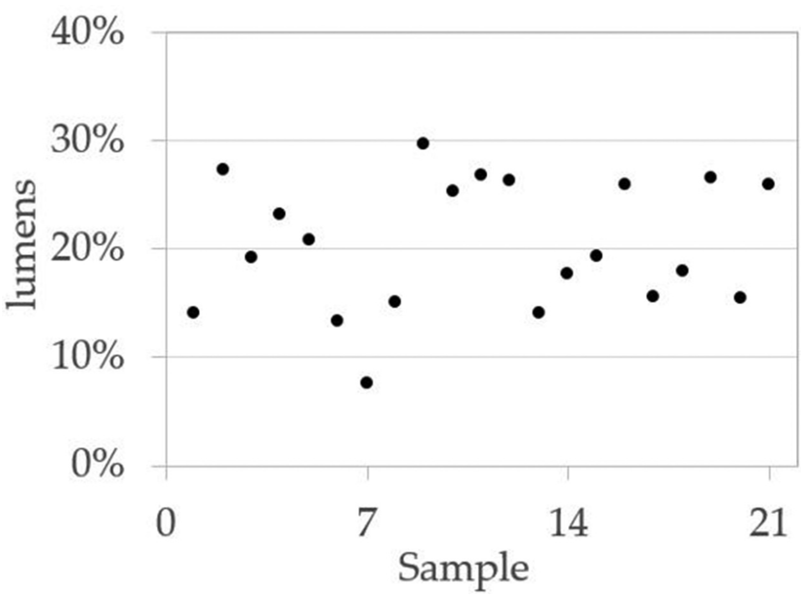

Figure 6. Percentage of lumens for Philippine fiber.

porosity of fiber samples and cross-section area reduction from method 1 to method 4. It can be seen that the greater the percentage of porosity, there is higher a reduction in the cross-sectional area.

Another important aspect in this study is the roundness of the fiber's cross-section. Using the second and third methods, a roundness factor "C" is defined as the ratio of the total area using the average diameter and the total area using circumference rounding. This factor indicates

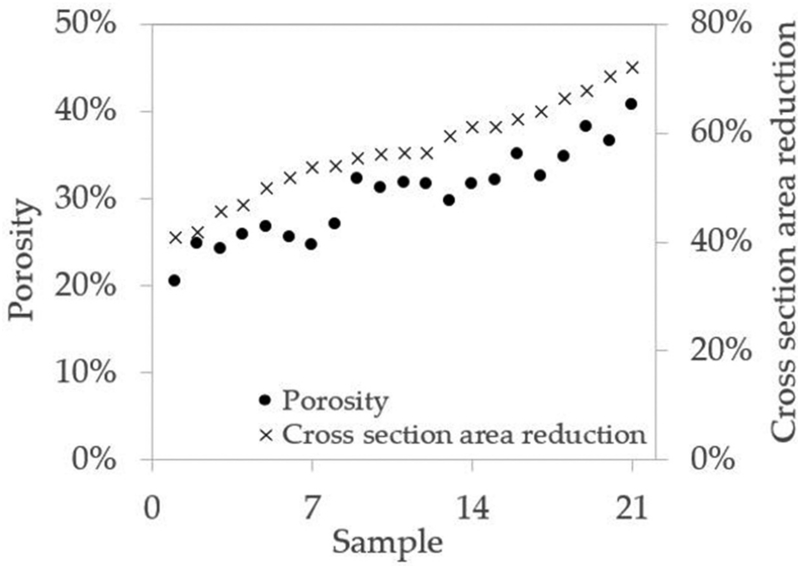

Figure 7. Porosity and cross section reduction for Philippine fiber.

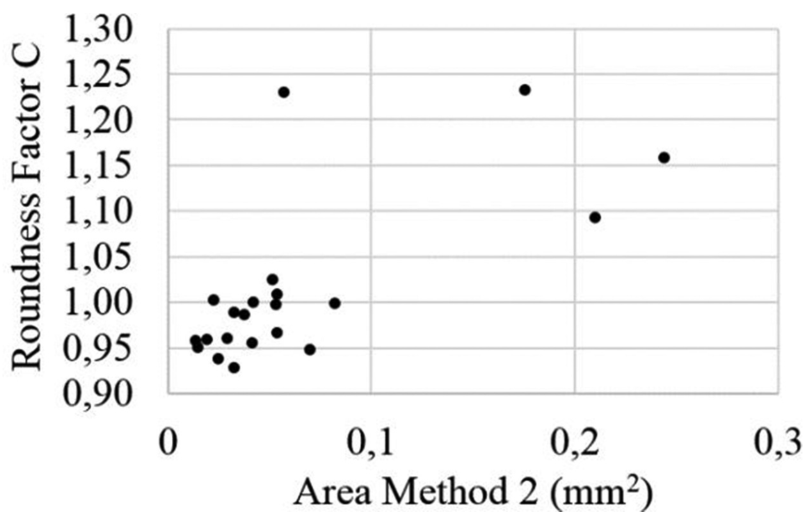

Figure 8. Roundness factor for every type of fiber.

how closely the fiber approaches a completely circular cross-section, where a value of 1 means that the fiber is perfectly circular. The actual values, as displayed in Figure 8, are mostly between 0,95 and 1.

Based on the values obtained for the roundness factor of the fiber, we can infer that the coconut fiber does have a circular cross-section. Consequently, the area can be calculated assuming a perfect circle. However, the lumens in the fiber must be taken into account, as calculated in the fourth method. Statistical analysis was used to observe differences between the third and fourth methods (binarization). According to these results, there are significant differences between both methods, which indicates that it is necessary to use a more accurate method for area calculation, such as the fourth method.

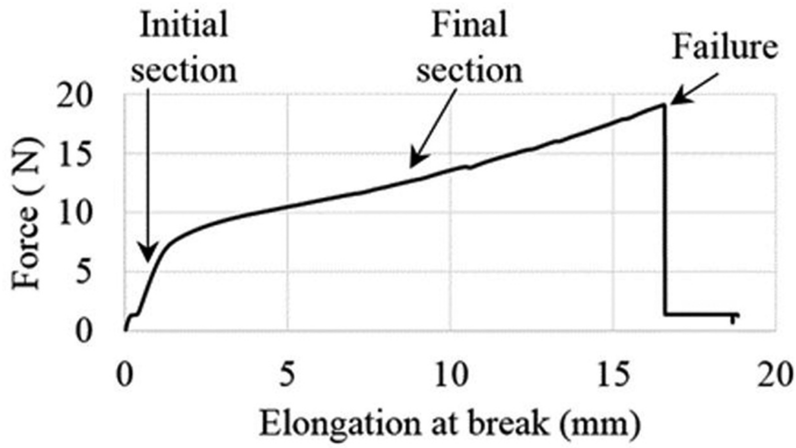

Figure 9. Typical force-elongation curve for the coconut fiber.

Tensile properties

The tested fibers display two different behaviors before failure. According to (Mukherjee and Satyanarayana 1984) many natural fibers showed two regions (nonlinear) of the stress-strain curve. The graphs obtained are comparable to those obtained in other studies of coconut fiber (Waifielate and Oluseun 2008). Figure 9 shows a typical force-elongation curve for a coconut fiber. It can be observed that the curves have two sections with different slopes. This change in slope can be explained by the collapse of the weak primary cell wall and the delamination between the fiber's cells (Silva, Nikhilesh, and Toledo 2008). It should be noted that not all natural fibers have two sections similar to the coconut fiber. Other fibers such as jute, sissal, and curauá, have a uniform linear behavior up to the point of fracture (Alves et al. 2013).

The end of the initial section is the point at which the fiber's primary cell wall breaks. The end of the second section is the point where the fiber breaks, i.e., the point of failure, which is also the point used to calculate its resistance to tension. Similar behavior and shape of plots were found for natural fibers reported in other works (Eveirtt, Aboulkhair, and Clifford 2013; Hamad et al. 2017; Shahzad 2013). The morphology of coconut fiber (referred to lumens) is the reason for variations in the mechanical properties. According to (Silva, Nikhilesh, and Toledo 2008), this behavior is probably due to the variability in the fiber cells.

The fiber´s tensile strength and young´s modulus are summarized in Table 4. The standard deviation of the fourth method is smaller than the first, which indicates that when lumens are taken into consideration for area calculation, the level of uncertainty in the determination of mechanical properties is lower. This was also found by (Hossain et al. 2014), whose research was able to reduce the standard deviation using the real area for jute fiber.

Table 4. Tensile properties of coconut fibers (standard deviations in parentheses).

Table 4. Tensile properties of coconut fibers (standard deviations in parentheses).

	Number of samples		Maximum stress (MPa)		Young´s modulus (GPa)	
Method	Untreated	Acid treated	Untreated	Acid treated	Untreated	Acid treated
First	51	63	222,04 (81,85)	226,01 (118,32)	4,32 (2,38)	5,57 (1,47)
Second	81	77	235,89 (37,49)	210,88 (91,74)	3,79 (2,61)	4,66 (0,39)
Third	81	77	238,15 (48,13)	203,94 (51,30)	3,83 (2,7)	4,51 (0,29)
Fourth	81	77	285,82 (34,84)	252,93 (34,99)	4,57 (1,82)	5,6 (0,45)

Table 5. One-way ANOVA results.

	Untreated		Acid treated	
Source of variations	F	P-value	F	P-value
Max strees	22,5170	<0,0001	5,8403	0,0007
Young´s modulus	19,3924	<0,0001	43,1834	<0,0001

The mechanical properties determined by the different methods, in particular maximum stress and young´s modulus, increase when the cross-sectional area of the fiber is calculated based on the fibers' actual morphology. This variation is caused by lumens, that reduce the cross-section area, and this can be reflected on the mechanical properties (Babu et al. 2019).

Statistical analysis

A one-way between subjects ANOVA was conducted to compare the effect of cross-sectional area measurement methods of coconut fiber on mechanical tensile properties. There is a significant effect of morphology measurement methods on maximum stress and young´s modulus, both untreated and acid treated at the $p < 0,05$ level for the conditions F and p-values.

Table 5. One-way ANOVA results.

Post hoc comparisons using Games-Howell test indicated that mean values for the fourth method are significantly different than the other methods. Taken together, these results suggest that using SEM for determining the cross-sectional area of coconut fiber does have an effect on calculating the real values for mechanical tensile properties. Moreover, porosity should be taken into consideration to obtain the effective area used to determine the maximum stress and young´s modulus.

Conclusions

The method of calculating the area of the fibers through binarization with is an interesting and useful technique for the calculation of the effective area of the fibers. This method enables obtaining the percentage of effective area and the lumens that are present in the fibers studied. However, the finding of this effective area does not produce significant results in terms of calculating the mechanical properties of these samples. Even so, this method is neither difficult nor time-consuming (it takes only 1–2 minutes longer than the traditional method 2), which implies that there is no justification for nor using it, since method 4 produces more reliable and accurate results in the long term. If the same method were consistently used, it would be possible to reduce the wide variability of values for this fiber's ultimate resistance to tension.

The binarization method produces higher standard deviations for the calculated stress values; however, these results are similar to those of other studies in the reference literature (Ali 2011; Alves et al. 2013; Defoirdt et al. 2010; Fernandez 2012; Munawar, Umemura, and Kawai 2007; Satyanarayana 2010). In those studies, the standard deviations were 25%-35% from the mean values. The effective area percentage obtained through binary analysis is between 73% and 85%, which indicate that the actual fiber stresses are between 15% and 27% higher than the normal stresses.

The ANOVA analysis provides strong evidence that the method for determining the cross-sectional area of coconut fiber has a significant effect in the mechanical properties. The tensile strength and modulus of elasticity for coconut fiber untreated are 285 MPa and 4,57 GPa, respectively. It was also verified that the chemical treatment decreases tension strength, due to the weakening of the cellular wall of the fiber. While young´s modulus increase when coconut fiber was acid treated.

Regarding the morphological characteristics of the fiber, it was determined that the coconut fiber does have a circular, with roundness factors of between 0,95 and 1,03, and lumens presence suggests that method 4 is a more accurate method for measuring the area. Results show that the morphology of the coconut fiber affects the fiber's real mechanical properties, in terms of maximum stress and young ´s modulus.

This study's results strengthen the theoretical design of calculations of composite materials, to enable the implementation of natural fibers in the manufacture of naval components. It therefore promotes business confidence for the implementation of the first maritime cluster in Colombia.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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