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Study of the Physical, Mechanical and Biochemical Characteristics of Coconut Fibres

Herve Zetome*, Sedjro Yvette Kiki Tankpinou

Processes and Technological Innovation Laboratory (LaPIT), National University of Science, Technology, Engineering and Mathematics (UNSTIM), Lokossa, Benin
Email: *hervzet85@gmail.com, yvettestankpinou@gmail.com

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Abstract

Waste management after coconut harvesting is a real problem at a national level. Coconut production in Benin increases considerably every year, especially in the coastal and Atlantic departments. To help manage this waste while helping to preserve the environment, the development of a composite material based on coconut fibres is one of the alternatives for moving towards renewable and biodegradable eco-materials. This article describes the physical, mechanical, chemical and biochemical characteristics of coconut fibres in their natural state. Once the fibres had been extracted, their morphological and physical characteristics were determined (absolute density, fibre diameter and length). A biochemical analysis of the fibres determined the proportion of chemical elements they contain. These results are supported by those obtained by X-ray diffraction on the fibres. Physical test results show that the average fibre density is 1.05 g/cm^3, the average fibre diameter is 417 µm and the average fibre length is between 24 and 26 cm. Biochemical analysis shows that the fibres are composed of 47.20% cellulose, 1.25% hemicellulose, 45.25% lignin and 6.30% pectin. The diffractogram obtained from the X-ray diffraction peaks shows that the fibres are essentially made up of cellulose. According to the results of mechanical tests (simple tensile test), the average tensile strength of the fibres is 125.45 MPa. These results show that coconut fibres have a low density and very good tensile strength, and can be used to reinforce the mechanical performance of soils.

Keywords

Eco-Materials, Fibre Extraction, Physical and Mechanical Characteristics

1. Introduction

The modernisation of industrial technology over the last few decades has affected

a number of sectors; the civil engineering sector is marked by the construction of large-scale structures using high-performance materials [1]. This progress in the world of construction calls for heavy exploitation of available natural resources and the release of large quantities of greenhouse gases into the environment as a result of the production of chemical and synthetic products. Studies carried out by a group of experts show that the construction sector is the second largest emitter of greenhouse gases [2]. With a view to tackling these types of environmental problems and preserving the environment for sustainable development, natural-fiber composite materials, as substitutes for conventional materials in the various fields of construction, are gaining in importance due to their renewable and biodegradable nature; they appear to be materials capable of making a greater contribution to achieving better environmental management, and their valorization could be promoted by scientific research [3]-[6]. On a national and international scale, the application of natural fiber composites has progressed in several fields over the last few decades [7], due to their low production and energy costs [8] [9]. To this end, several studies have been carried out on plant fibers such as sisal, esparto, pineapple and coconut fibers.

Benin has opted for the development of coconut production in order to combat coastal erosion, and producing areas include Ouidah, Cotonou, Tori-Bossito, Comè, Gand-popo, Abomey-Calavi and Kpomassè [10]. Once the coconuts have been harvested, the husks are burned in the fields. Disposing of them, therefore, creates environmental problems due to the pollution of the atmosphere and soil through the emission of greenhouse gases [11]. The introduction of coconut fibers in civil engineering applications could contribute to the management of agricultural waste and the preservation of the environment [12].

This research focused on the characterization of plant fibers in order to fully valorize them.

2. Materials and Methods

This section describes the materials and methods used in this work.

2.1. Materials

Coconut fibers are the materials used in this study.

2.1.1. Coconut Fibres

Southern Benin is the ideal zone for coconut production (**Photo 1**). Production is most pronounced in the communes of Ouidah, Grand-Popo and Abomey-Calavi. The coconut flocks used in this study were harvested at Pahou in the commune of Ouidah (**Figure 1**). Located between 2˚ and 2˚15'N latitude and between 6˚15 and 6˚30'E longitude, the commune of Ouidah is one of the eight (08) communes of the Atlantic department, covering an area of 364 km^2. Rainfall in this area is influenced by the southern climate. Rainfall averages 1100 to 1220 mm per year [13].

Photo 1. Coconut trees—coconut fibers.

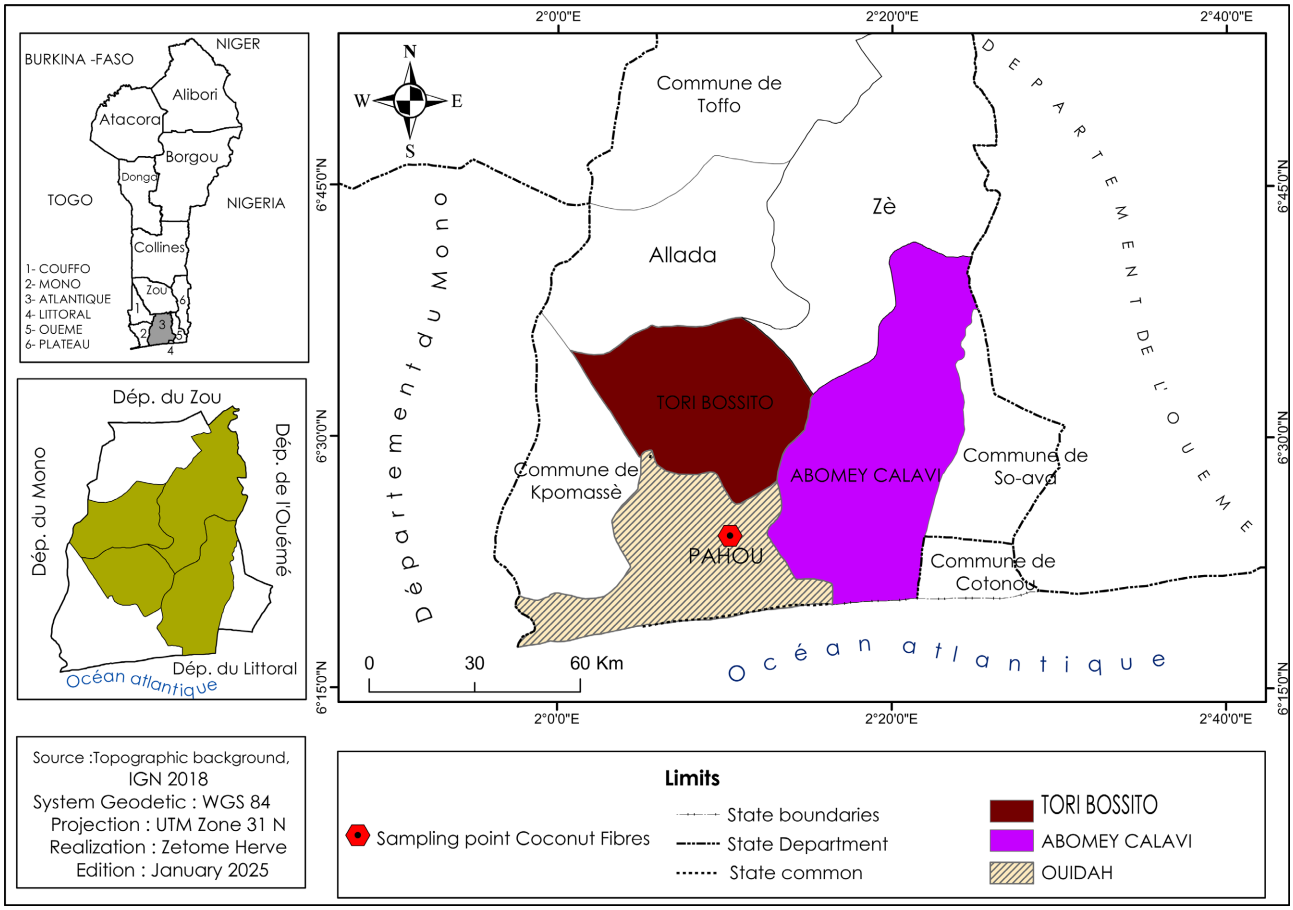


Figure 1. Coconut fiber collection area. (Source: IGN (National Geographic Institute))

2.1.2. Manual Extraction of Coconut Fibers

Fiber is extracted from the coconut's mesocarp (Figure 2). The seed or albumen is collected for food or nursery purposes, while the shell that covers it (the mesocarp) is processed, once the fibers have been extracted, they are selected according to length and characteristics for one market or another. For example, the finest fibers and dusts are destined for agriculture, while the longest go to the textile industry.

After the mature coconuts have been harvested, the fibers are extracted after the coconut husk has been softened with a hammer (Figure 3). The extracted fi-

bers are then cleaned to remove impurities, and dried before use. This traditional extraction method produces both long and short fibers. Long fibers are used for a wide range of applications, especially in the construction and transport sectors.

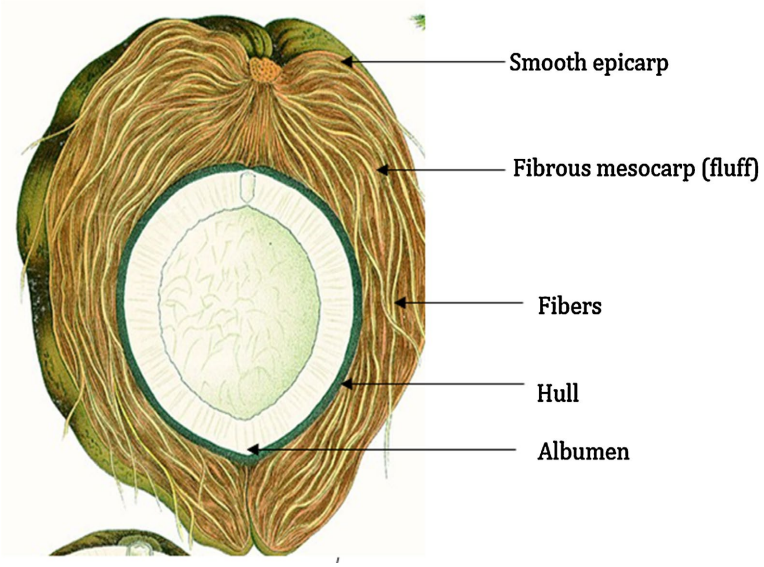


Figure 2. coconut cross-section.

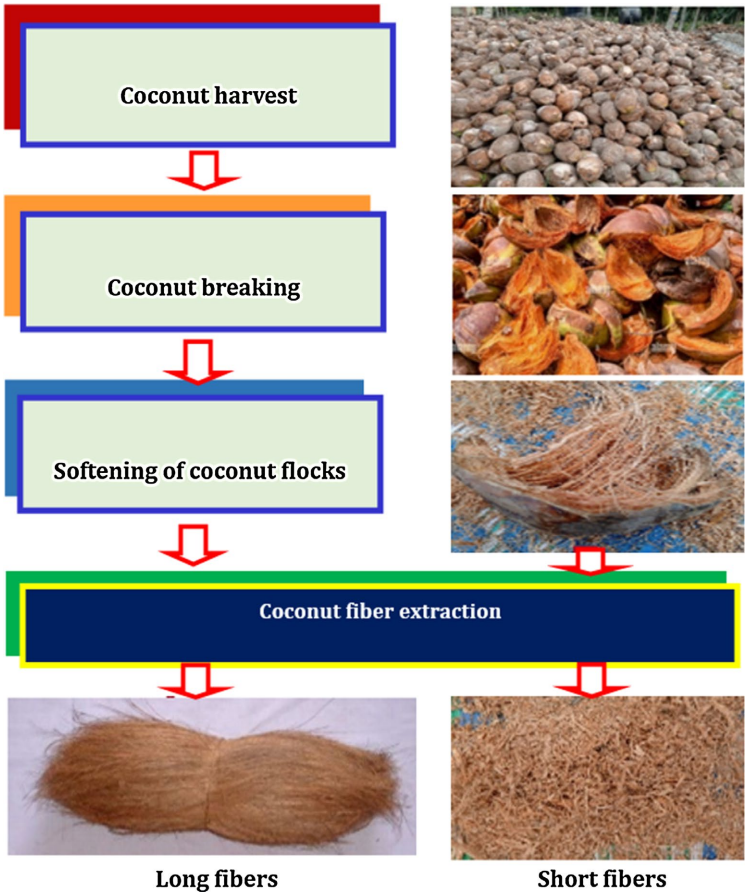


Figure 3. Coconut fiber extraction process.

2.1.3. Mechanical Extraction of Coconut Fibres Method

A machine has been designed for the mechanical extraction of coconut fibers (Figure 4). This machine allows long and short fibers. The long fibers are in the form of single bundles, while the short fibers are in powder form in powder form (also known as wax). This extraction method is faster, and therefore enables us to obtain a large quantity of fibers in record time. The fibers obtained using this method do not have the same lengths as those obtained using the manual method. This method of mechanically extracting coconut fibers is a new one, with very few developments in the literature.

2.2. Methods

2.2.1. Morphological Analysis

Morphological analysis allowed to determinate the transverse dimensions (diameter) of the fibers and the length distribution over one hundred (100) fibers. The diameter of coconut fibers was determined using a Leica M80 microscope with two 1× objectives and a 16× eyepiece. It has an 8:1 zoom range, a continuous zoom from 7.5× to 60× and activatable notches (Figure 5). The principle of diameter

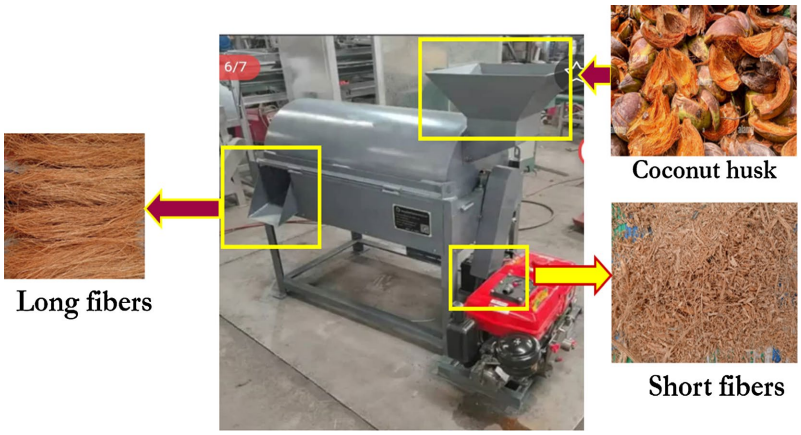


Figure 4. Mechanical extraction of coconut fibres method.

Figure 5. Unpolarized image of fibers using a Leica M80 microscope.

determination involves cutting the fibers to a length of one centimeter (01 cm) and arranging them over a width of one centimeter (01 cm) on a slide, which is then placed on the microscope stage for observation.

Fiber length is a very important morphological characteristic for complete fiber characterization. It is greatly affected by the extraction method used. Fiber length distribution is determined in accordance with standard NF G 07-007 on a sample of 100 coconut fibers.

2.2.2. Determining Absolute Fiber Density

Absolute fiber density was determined using the helium pycnometer method (**Figure 6**). The small size of the helium atoms allows access to the finest pores, making density measurement highly accurate. The sample to be analyzed is introduced into the pycnometer cell. The gas is confined (pressure P1) in a cell of known volume (Vc). It is then released into an expansion volume (V2). The result is a pressure P2 [14]. This test was repeated three times.

(a) (b) (c)

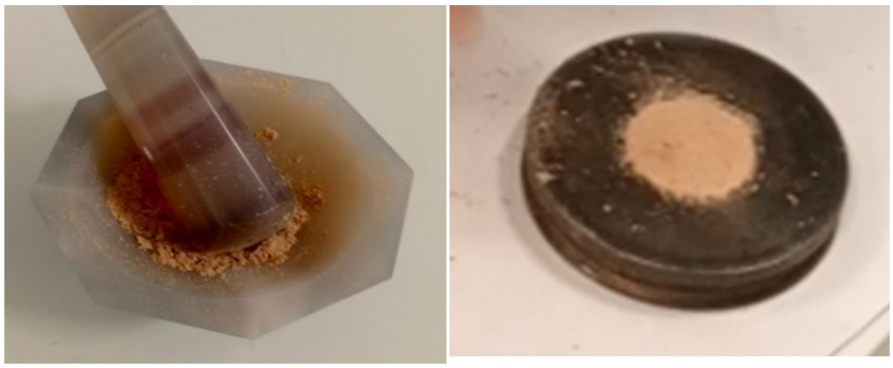
Figure 6. Experimental protocol for determining absolute fiber density.

2.2.3. X-Ray Diffraction Testing of Fibers

X-ray diffraction (XRD) is a structural analysis technique used to analyze the crystallographic structure of crystalline materials and determine the size of crystalline domains. The test involves preparing a specimen by grinding fibers into fine particles using an agate mortar and pestle (**Figure 7**). The specimen holder is filled and subjected to the diffractometer. The X-ray source sends X-ray beams onto the sample, which are scattered by the crystalline planes, and the intensities of the diffraction peaks are recorded on a computer for processing. The diffraction peaks together form the diffractogram. The intensity detected is recorded as a function of the beam's deflection angle 2θ. The intensity and position of diffraction peaks determine the different crystalline phases of the material. The X-rays diffracted during the test obey Bragg's law.

The powder diffractometer used in this study is PANalytical Empyrean (**Figure 8**). It consists of a diffractometry system with a Ka1 monochromatic beam of wavelength λ = 1.5406Å recorded between 0.5 and 140˚ 2θ, with a minimum angular step of 0.002˚ 2θ. Diffracted X-rays are recorded by a 3D pixel detector. The

material used for the anode is copper.



(a) (b)

Figure 7. (a) Agate mortar and pestle; (b) sample holder.

Figure 8. Powder diffractometer (PANalytical Empyrean).

2.2.4. Biochemical Analysis of Coconut Fibres Fibers

- **Determination of moisture and volatile matter content**

The moisture and volatile matter content of biomass is determined using the method recommended by AOAC in 2000 [15]. This method involves pre-drying an empty porcelain crucible and its lid in an oven at a temperature of 105˚C for 3 hours to remove all traces of moisture. The material is then transferred to a desiccator for cooling before weighing to the nearest 0.0001 g. A 3 g specimen of powdered biomass is introduced into the crucible and spread out evenly, then dried in an oven for 3 hours at 105˚C. After drying, the covered crucible is cooled in the desiccator before re-weighing to obtain the mass m2 (sample mass + crucible).

Moisture and volatile matter content were calculated according to the equation below.

- **Hemicellulose content**

Hemicellulose content is determined from a 0.5 g sample weighed to the nearest 0.0001 g of dried biomass. The specimen is subjected to the action of 0.5 mL of aqueous sodium hydroxide solution taken in an 80 mL quantity. This alkaline treatment is carried out at a temperature of 80˚C for 3.5 hours. The mixture was then filtered and the solid residue was washed with distilled water to neutral pH, then dried at 105˚C for 24 hours. The hemicellulose content was calculated according to the following equation [16].

- **Lignin content**

To determine lignin content, 0.5 g of dried biomass, weighed to the nearest 0.0001 g, was subjected to the action of 72% sulfuric acid in 15 mL. This treatment was carried out at room temperature with stirring.

Next, 135 mL of distilled water was added to the mixture and the whole was heated to reflux in a water bath at 100˚C for 2 hours. After cooling to room temperature, the mixture was neutralized with a 5% (w/w) sodium bicarbonate solution, then filtered and rinsed with distilled water. The residue was dried at 105˚C for 24 hours before being weighed. Lignin content was calculated according to the equation:

$$\text{Lignin, \% (m/m)} = (m1 - (m2 - m3) \times 100)/m1$$

where m1 represents the test sample mass of dried biomass without extractables, m2 is the mass of the biomass residue after treatment and filter paper after drying and m3 is the mass of filter paper [16].

- **Cellulose content**

Cellulose content is calculated using the method employed by Mansor *et al.* in 2019 [16]. This method consists of determining the cellulose content from the respective contents of extractives, hemicellulose and lignin, each corresponding to a mass fraction in relation to 4.5 g of dried biomass initially taken.

$$Q\,(\text{Etanol extractable}+)\; Q\,(\text{Hemicellulose} + Q\,(\text{Lignin}) + Q\,(\text{Cellulose}) = 4.5\ \text{g}$$

where Q represents the mass in grams of each component.

From this equation, in which 4.5 g of the test sample represents 100%, the respective percentages of the various constituents are deduced [16].

- **Determination of pectin content**

The sample was dispersed with 0.1 M citric acid (1:10; (w/v); pH = 1 - 1.5), homogenized, and pectin extraction was carried out according to the citric acid (CA) method with a few modifications. The prepared samples were heated to 90˚C for 60 min, cooled and centrifuged (Sigma 4-16 KS, Germany) at 6000 rpm for 20 min. The collected supernatants were treated with absolute ethanol. Pectin precipitates were collected by re-centrifugation, then washed twice with 80% (v/v) and 90% (v/v) ethanol, and the resulting pectin was conventionally hot-air dried (T = 40˚C ± 2˚C; t = 12 - 14 h) to a constant moisture content ≤ 9% [17].

Biochemical analysis of the fibers was carried out three times, and the results were compared with those in the literature.

The different materials used for carrying out biochemical analysis tests on the fibers are present in Figure 9.

(a) desiccator (b) water bath (c) precision balance

Figure 9. Equipment for biochemical fiber analysis.

2.2.5. Mechanical Testing

For a complete characterization of coconut fibers, samples of single fiber bundles were subjected to uniaxial tensile testing in accordance with ASTM-D7269 [18]. Prior to the tensile test, the cross-sectional area of the fibers was determined from their density, length and mass. The tests were carried out under ambient conditions.

Fibers 20 cm long were used to determine tensile strength. To prevent damage to the fibers in contact with the jaw, both ends of the fiber were protected by adhesive tape over a length of 3 cm on each side. Puller jaws (Figure 10).

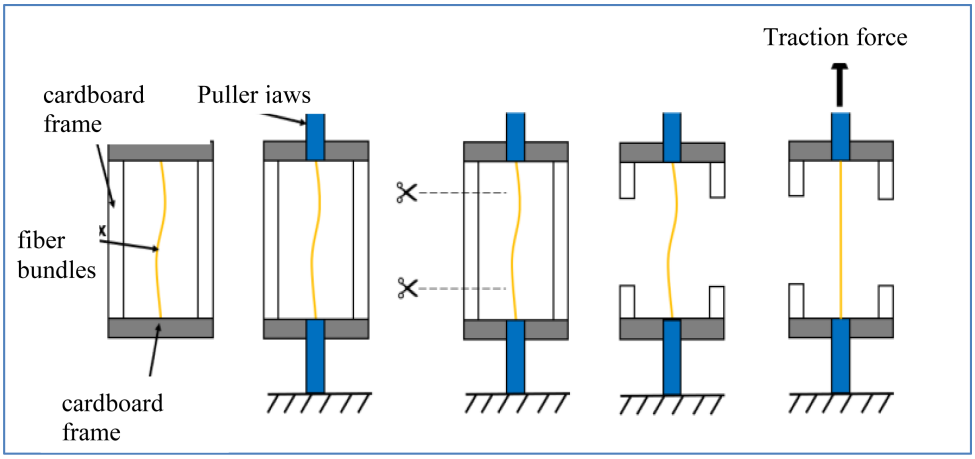
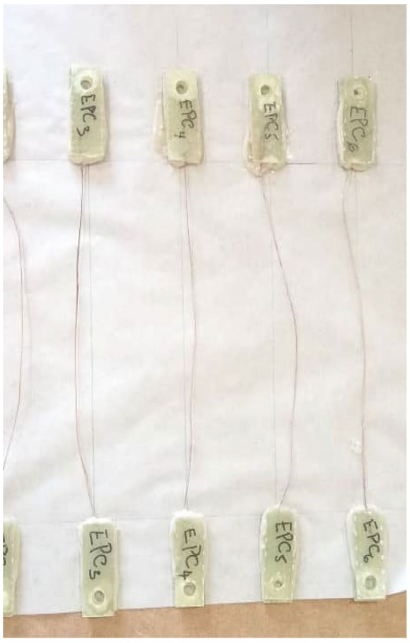
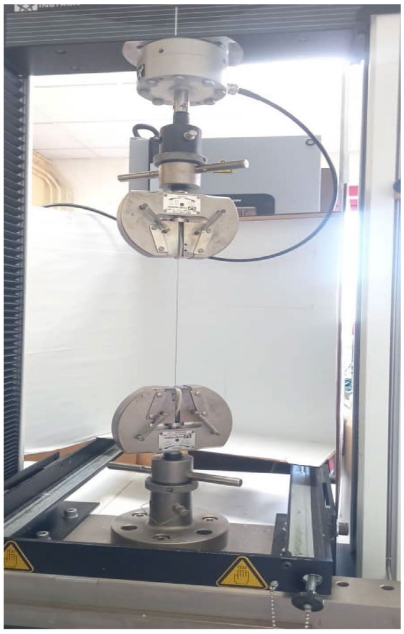


Figure 10. Graphic illustration of the main sequences required to perform a tensile test on coconut fibres [19].

The loading speed was set at 2 mm/minute throughout the test (Figure 11). The dimensions of the fiber bundles did not allow deformations to be measured with an extensometer during the test. Therefore, fiber elongation is considered equal to displacement during the test. Applied force and displacements are recorded to calculate stress and strain. The tensile test on the fibers was repeated 5 times.



(a) Tensile test specimens



(b) Instron 5867 traction machine

Figure 11. Tensile test on coconut fibres.

$$E = \frac{\Delta\sigma}{\Delta\varepsilon}$$

3. Results and Discussions

3.1. Morphological Characteristics of Fibers

The morphological characteristics of the fibers mainly concern their cross-section (diameter) and average length. The results of length measurements on a sample of 100 fibers are translated into Figure 12, showing the distribution of fiber lengths.

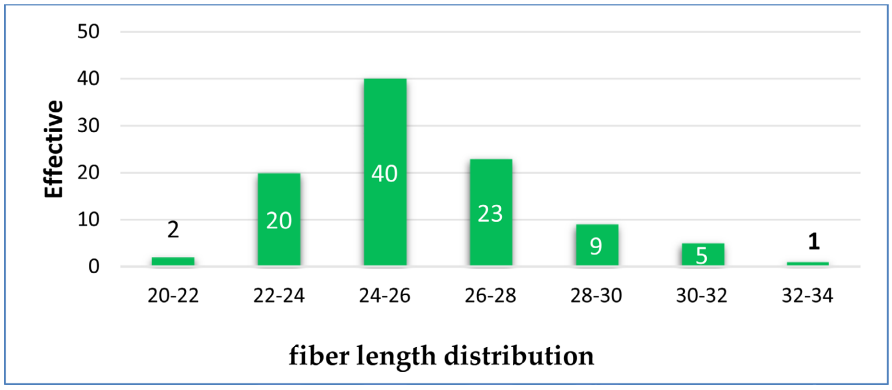


Figure 12. Fiber length distribution.

This graph reveals that the average length of coconut fibers ranges from 24 to 26 cm. The average diameter obtained from Leica M80 microscopy is 417 µm. These two parameters are highly variable and depend essentially on the type of

coconut plant, the extraction and processing methods used and the destination of the fibers. In the case of this study, fibers are extracted manually, and only long fibers are taken into account during characterization. In addition, the flocks used for fiber extraction come from large coconut palms. This fully justifies the dispersion of fiber diameter and length values.

3.2. Fiber Density

Test results for determining fiber density using the helium pycnometer method show that fibers have an average density of 1.05 g/cm^3. In the literature, coir fibers have a density of around 1.15 to 1.45 g·cm^{-3} [3]. The absolute density value is in line with that obtained in the literature.

3.3. Fiber Chemical Characterization Results

In a crystalline structure, the organization and stacking of atoms in the lattice results in crystal planes. In the diffraction test, the X-ray beams sent by the diffractometer source are diffracted by the crystalline planes at an angle of 2θ according to Bragg's law. The literature reveals that plant fibers are made up of cellulose, hemicellulose, lignin and pectin. Hemicellulose, lignin and pectin are amorphous elements, but cellulose is made up of a crystalline and an amorphous zone (**Figure 12**).

The diffraction peak intensities recorded yielded the diffractogram shown in **Figure 13**. The diagram is analyzed using X'Pert HighScore software.

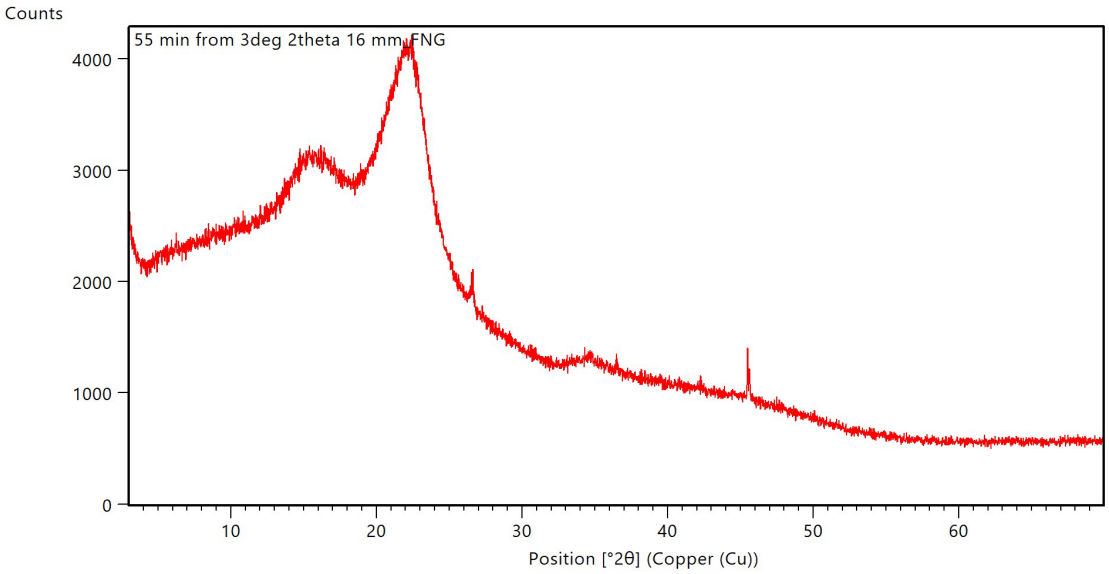


Figure 13. Diffractogram.

The coconut fiber diffractogram shows two major peaks. The first peak is recorded at $2\theta = 16^\circ$ and the second at $2\theta = 22.5^\circ$. These peaks recorded on the diffractogram are very close to those found in the literature in the case of date palm Lif fibers [20]. The peak at $2\theta = 16.00^\circ$ indicates the existence of amorphous con-

stituents in coconut fibers [20]. The peak at $2\theta = 22.5^{\circ}$ indicates the crystalline nature of coconut fibers.

Analysis of the diagram using X'Pert HighScore software revealed that the major recorded at $2\theta = 22.5^{\circ}$ corresponds to the [1 1 1] crystal plane of cellulose [21]. The results obtained are in line with those of work carried out by Mohamed D. in 2013 [21] and those of DJEBLOUN Y. in 2018 [22].

A crystallinity index (CrI) has been calculated from this information, using Segal's method. The crystallinity index can be estimated from the diffraction intensity values of the crystalline structure and those of the amorphous structure.

$$C_r I(\%) = \frac{I_{(2\theta=22.5^{\circ})} - I_{(2\theta=216^{\circ})}}{I_{(2\theta=22.5^{\circ})}} \times 100$$

I ($2\theta = 16^{\circ}$) = Diffraction peak intensity at $2\theta = 16^{\circ}$

I ($2\theta = 22.5$) = Diffraction peak intensity at $2\theta = 22.5^{\circ}$.

The diffractogram shows that the intensities of the major diffraction peaks recorded at $2\theta = 16^{\circ}$ and $2\theta = 22.5^{\circ}$ are equal to 3200 and 4300, respectively.

Thus, the crystallinity index obtained is equal to 27.72%. This result is very close to that obtained by KHELIFI Zakia in 2017 [3], $C_rI(\%)$ = 33% in the case of untreated esparto fibers. The crystallinity of plant fibers is related to the proportion of cellulose, so we can conclude that coconut fibers contain a lesser amount of cellulose than lignin (**Figure 14**).

3.4. Results of Biochemical Fiber Analysis

Biochemical analysis was used to determine the proportion of each chemical element.

Biochemical analysis shows that coconut fibers are composed of 47.20% cellulose, 1.25% hemicellulose, 45.25% lignin and 6.30% pectin. The different proportions of chemical elements obtained are in line with those obtained by other researchers in the literature [24].

Both the results obtained and those reported in the literature show that coconut fibres have the highest lignin content. Lignin is responsible for water resistance, resistance to micro-organisms and hardness. The high proportion of lignin in coconut fibers makes them rot-proof. This has led India to use them as a base layer for sustainable roads. Hemicelluloses are more easily degraded than cellulose by enzymes and micro-organisms, and are more water-soluble, meaning they absorb water more readily. Coconut fibers contain a low proportion of hemicellulose, so this has no remarkable influence on the durability of coconut fibers. Cellulose degrades over time, so coconut fibers should be treated with soda solution prior to formulation of composite materials to reduce the proportion of cellulose.

3.5. Tensile Test on Fibers

The single tensile test was performed on the fiber bundles.

Analysis of the results of the simple tensile test is based on the evolution of force as a function of displacement (**Figure 15**). The force-displacement curves for the specimens tested show an initial non-linear phase. Then, the drop in force

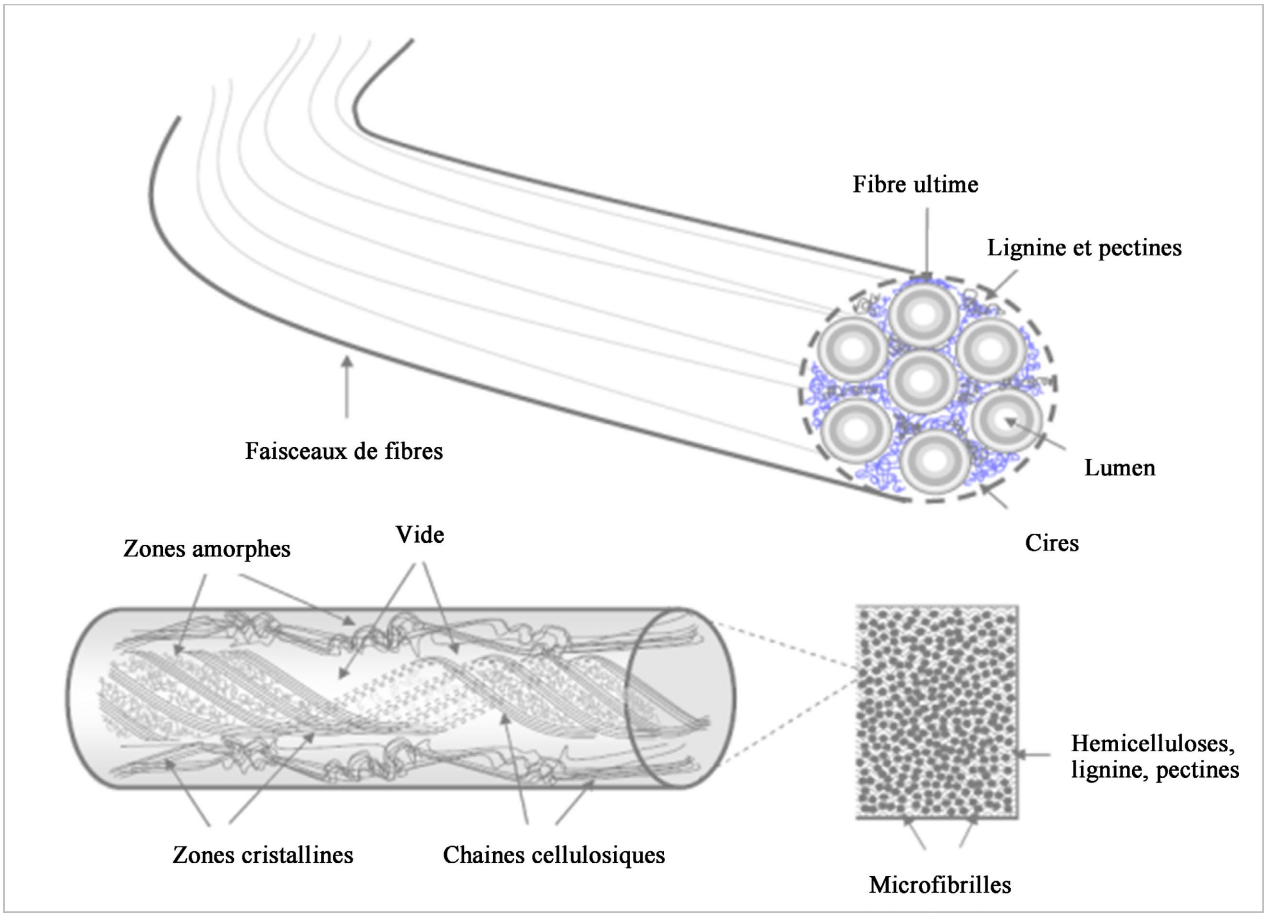
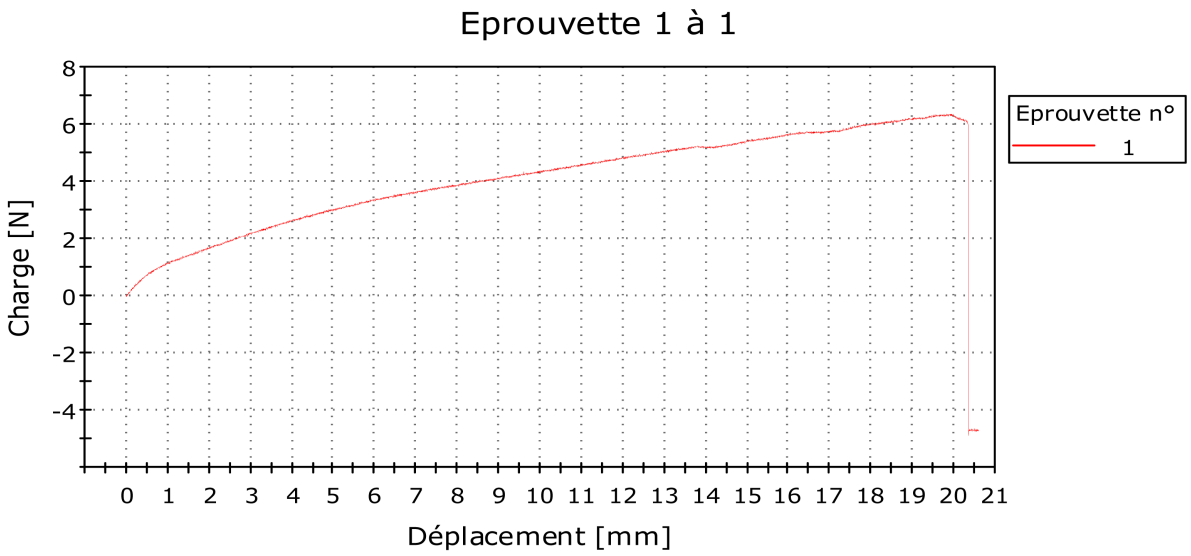


Figure 14. Lignocellulosic and crystalline structure of a cellulose fiber [23].



	Déformation de traction (Déplacement) longueur initiale [mm]
1	100,00000

Figure 15. Force-displacement curve.

reflects the onset of damage in the fibers. In the final phase, the fibers behave linearly until they suddenly break (see **Figure 6**). The Young's modulus of the fibers is determined on the linear part according to Hooke's law. Tensile test results show an average fiber tensile strength of 125.45 MPa. According to the literature, the tensile strength of coconut fibers varies from 54 to 250 MPa [25]-[27]. The average strength of coconut fibers obtained is in line with those obtained in the literature.

4. Conclusions

This research work on the physical, mechanical, chemical and biochemical characterization of coconut fibers highlighted several results. Physical tests show that coconut fibers have a low density, with an average length varying between 24 and 26 cm in the case of fibers obtained from manual extraction. In contrast, fibers obtained by mechanical extraction have different morphological characteristics (length and diameter). We can therefore conclude that the physical characteristics of coconut fibers are more influenced by the fiber extraction method. Biochemical analysis results show that, in their natural state, coconut fibers contain more lignin than cellulose. This makes them more resistant to microbial action (good resistance to decay). Mechanical studies show that coconut fibers have high tensile strength and can be used for soil reinforcement without undergoing any prior treatment.

This work highlights the extraction method for coconut fibers, which is not well developed in the literature, and emphasizes the dispersion of the physical characteristics of the fibers according to the extraction method. Finally, it confirms the rot-proof character of coconut fibers due to the presence of a high proportion of lignin. Coconut fibers are the richest vegetable fibers in lignin and can be used in road construction.

However, this work needs to be complemented by a study of fiber biodegradation kinetics for large-scale fiber valorization.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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