

Influence of Solvent onto Chemical Extraction of America-Type Coconut (*Coco nucifera L.*) Fbers: Analysis of Physicochemical, Mechanical and Morphological Properties

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Abstract

In this study, the natural fibers from Coconuts of the species *Coco nucifera L.* were Chemically extracted in different solvents such as sodium hydroxide (SH), acetone (AC) and sodium hydroxide-acetone (SHA) for their applications in the textile industries. Structural, morphological and physico-mechanical characterizations such as X-ray diffraction (XRD), Fourier transform infrared spectrometry (FTIR), scanned electron microscopy (SEM), measurements of density, Young's modulus, water absorption rate and humidity were evaluated. The XRD and FTIR results show that *Coco nucifera L.* fibers contains type I cellulose. Mechanical characterizations were also carried out. These results show that by varying the different solvents used, the physico-chemical, mechanical and morphological properties of the fibers change, which implies that the solvent has an influence on the properties of these fibers. The fibers extracted by the sodium hydroxide-acetone mixture have a linear density of 1.636, the percentage of water absorption is 62.428%, the percentage of moisture absorption 9.605% compared to other values in the literature shows that this solvent mixture improves the properties of coconut fibers which contain type I cellulose. The tensile stress is 0.013 GPa, the percentage strain is 49.836% and the Young's modulus is 0.114 GPa as well as the percentage elongation show that these fibers are elasto-plastic. The values obtained mean that these fibers are suitable for use in textiles.

Keywords

Chemical Extraction, Cellulose, *Coco nucifera L.* Fibers, Elasto-Plastic, Textiles

1. Introduction

Natural fibers account for a third of the world's fiber production [1] [2]. Most of these fibers are obtained from vegetable plants, animal hair or by chemical synthesis [3]-[7]. The various transformations undergone by these fibers enable them to be used in a variety of sectors, including the clothing industry [8] [9], civil engineering [10] [11], transport [12], medicine [13] [14], sport [15] [16], furniture [17] [18] and agriculture [19].

The design of environmentally friendly materials is nowadays a requirement of the scientific community, as this helps to protect the environment [20]. The incorporation of new environmentally friendly materials is an inevitable necessity, along with improved extraction techniques that comply with the increasingly stringent environmental standards. The interesting specific properties of these materials, their low density, their thermal insulation properties, and their mechanical properties, particularly their biodegradability, open up promising prospects [21]-[23]. Traditionally, coir fiber has been used to make rope for cordage, sparterie (a woven, plaited or braided object), as a source of energy, fishing nets, canvas, carpets, brushes, brooms or mattress padding. More recently, geotextiles made entirely from coir are being used to combat soil erosion. There is also insulation in the form of flexible panels and plywood (fibers and resins) [24]-[26].

Plant fibers are varied and can be used in a wide range of applications because, for certain specific applications, they represent materials with technical performances that are sometimes superior to those of traditional materials [26] [27].

These new materials have developed very rapidly in recent decades. Numerous studies have highlighted the advantages and intrinsic limitations of these materials.

The textile industry is therefore not immune to this requirement, and the various methods of extracting natural fibers, such as chemical extraction using bisulphite [28] [29], acid [30] [31], sodium hydroxide-anthraquinone [32] [33] and neutral sodium sulphate [34], are often used, but these methods have their limitations, being difficult and expensive to implement, and the various processes used are often not controlled. The sodium hydroxide and acetone extraction technique developed in this work have a number of advantages, such as ease of implementation, and the fibers obtained have good physicochemical and mechanical properties [35]-[37].

The aim of this work is to extract America-type Coconut (*Coco nucifera L.*) fibers using sodium hydroxide, acetone to assess the influence of solvent onto

different extraction methods and the physicochemical, mechanical and morphological properties are analyzed.

2. Materials and Methods

2.1. Sampling

Coconut fibers were extracted from *Cocos nucifera L.* fruits from Edea (Cameroon), in the Sanaga-Maritime department, in the sampling area with geographical coordinates of 3°29' longitude North and 10°06' latitude East. The reagents used in this work are analytically reliable, therefore have not undergone any prior purification. Sodium hydroxide, acetone, xylene and acetic acid used are purchased from Sisco Research Laboratories pvt. Ltd., India. The extraction of fibers involves three stages: pre-treatment, extraction and post-treatment.

2.2. Methods

2.2.1. Pre-Treatment

After sampling, *Cocos nucifera L.* fibers are pre-treated by simple scutching, which removes impurities from the fibers using a knife. This operation is carried out in two stages:

The first operation consists of cleaning the outer part of the coconut.

The second operation consists of separating the fibers from the outer part.

2.2.2. Extraction

1) Chemical Extraction with Sodium Hydroxide

For this treatment, 1g of *Coco nucifera L.* fibers flock was taken, making a total of nine (9) samples which were weighed. Another solution prepared with the mixture sodium-hydroxide is used. The proportions of 50/50 are used for experimentation. Each of the weighed *Coco nucifera L.* fibers samples was then soaked in the different solutions contained in a beaker and sealed with aluminium foil for 72 hours.

After this resting time, the samples are taken and rinsed with distilled water to remove the residual pectin and hemicelluloses deposited on the fibers surface. The fibers were then immersed in a neutralization bath containing 5 mL of 0.1 M acetic acid, followed by a second rinse. After these, the fibers were rinsed and then dried in an oven at 65°C for 24 hours.

3. Characterization Techniques for *Coco nucifera L.* Fibers

3.1. Physical Characteristics

3.1.1. Density Measurement

Two methods are used to measure the density and porosity of *Coco nucifera L.* fibers.

- Pycnometer method

Various density measurements are carried out using a pycnometer (20 cm³). The fibers were dried for 48 hours in a desiccator containing regenerated silica.

The fibers are then cut into lengths of 4 to 5 mm and placed in the pycnometer, which in turn is placed in the desiccators for at least 24 hours. Before weighing with water, the fibers are impregnated for 2 hours. The micro-bubbles between the fibers are practically evacuated. Each sample is weighed three times using a Sartorius balance (1/1000).

The apparent density is expressed by:

$$d_e \left(\text{g/cm}^3 \right) = \frac{M_e \times d_E}{M_E - [-M_T - M_e]} \quad (1)$$

M_e : sample mass (alone) (g);

M_E : mass of water (20 cm³) (g);

M_T : sample mass plus water mass (all 20 cm³) (g);

d_E : density of water = 0.997 (g/cm³) at 25°C.

- Arthur's method

This method consists of weighing the samples in air, then placing them in a desiccator to release the air contained in the pores. A pump sucks the air out of the desiccators for 30 minutes. They were then immersed in xylene for 10 minutes. The xylene-soaked samples are removed and lightly wiped dry. They were then weighed in air and distilled water. The density and porosity are respectively given by the relationships:

$$d_e \left(\text{g/cm}^3 \right) = \frac{M_e \times d_E}{M_E - [-M_T - M_e]} \quad (2)$$

$$P_0 (\%) = \frac{M_{xa} - M_e}{M_{xa} - M_x} \times \frac{d_E}{d_x} \times 100 \quad (3)$$

With:

M_e : mass of the sample in air (g);

M_{xa} : mass of the sample impregnated with xylene in air (g);

M_{xc} : mass of the sample impregnated with xylene in water (g);

d_x : density of xylene = 0.880 (g/cm³).

3.1.2. Measuring the Rate of Water Uptake and Absorption

Knowing the water content of a textile material is very important. From an industrial point of view, this has an influence on the smooth running of the conversion and manufacturing process. From a commercial point of view, this parameter can modify the mass of a good from one environment to another, and therefore from one country to another, hence the need to understand and control it in order to establish a common reference. And from a structural point of view, humidity, in parallel with temperature, can alter the physical, chemical and mechanical properties of the material.

1) The water absorption rate

The water absorption rate is defined as the quantity of water present in the air that 100 g of dry matter can absorb under well-defined hygrometric conditions. The gravimetric method is used in accordance with French standard NF G 08-

001-4. The sample is dehydrated in an oven for 12 hours at 60°C until it has a constant anhydrous mass (M_s). The sample is then placed in a room where the temperature is 22°C and the relative humidity is 62%. The sample was weighed every 15 minutes. The measurement is considered complete when two successive weighing give a difference of less than or equal to 5% of the sample mass.

2) Absorption rate

The moisture content is assessed by the total saturation method, in accordance with the standard (AATCC 20A). The sample is placed in a desiccator for 30 days at a humidity of 65% ± 5% and a temperature of 25°C. After weighing (M_m), the sample was dried in an oven at 105°C ± 5°C for 15 h. It was then cooled in desiccators and weighed (M_s). The samples are weighed repeatedly until the weight (M_s) is constant due to drying and cooling. The moisture content (TH%) of the fiber is calculated by :

$$TH(\%) = \frac{M_m - M_s}{M_s} \times 100\% \quad (4)$$

With:

M_m : initial mass of the sample.

M_s : mass of the sample after exposure to the humid environment.

3.2. Structural Characterization

Numerous techniques are used to characterize the microstructure of *Coco nucifera L.* fibers.

3.2.1. X-Ray Diffraction

X-ray diffraction tests are carried out using a Bruker D8 diffractometer. Samples are dry ground in a ceramic mortar to a size of less than 125 μm. The anticathode is copper ($K\alpha = 1.54 \text{ \AA}$). The scan angle (2θ) is between 5° and 75° with a step size of 0.02°.

Various physical methods have been proposed in the literature for determining the crystallinity ratio of cellulosic fibers. According to Segal's method [38] the crystallinity index I_c is determined by:

$$I_{cr} = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (5)$$

I_{002} is the maximum intensity of the spectrum (amorphous and crystalline).

I_{am} is the maximum intensity of the amorphous part.

The crystallite size is calculated using Sherrer equation below:

$$D(\text{nm}) = \frac{k\lambda}{\beta \cos \theta} \quad (6)$$

where:

k is Sherrer constant, 0.68 to 2.08, 0.94 for spherical crystallites with cubic symmetry;

λ is the X-ray wavelength equal to 1.5406 Å;

β is the line broadening at FWHM in radians;

θ is the Bragg's angle in degrees.

3.2.2. Fourier Transform Infrared Spectrometry (FTIR)

Samples of *Coco nucifera L.* fibers were ground and dried in an oven to remove absorbed moisture. These samples are then mixed with potassium bromide (5% KBr) and pressed into a small pellet approximately 1 mm thick. Infrared spectra were taken using a Perkin Elmer spectrometer. The samples were scanned at 400 and 4000 cm^{-1} with a resolution of 2 cm^{-1} .

3.2.3. Morphological Analysis

Scanning Electron Microscopy (SEM) micrographs of the coir fibers were taken using a Hitachi (Japan) S-3000H electron microscope with an accelerating voltage of 15 kV. SEM is a widely used technique for investigating surface morphology such as roughness or porosity (from secondary electrons) and chemical composition (from backscattered electrons) of most solid materials. A beam of electrons emitted by a cathode bombards a sample, then the interaction between these electrons and the surface provides signals that are detected, amplified and used to reconstruct the image seen on the screen.

3.2.4. Fiber Tensile Tests

The fibers were tested on an MTS tensile testing machine in accordance with ASTM D3379-75 "Standard Tensile Method" [39]. The tensile testing of coir fibers was carried out in accordance with ISO 5079, using a universal testing machine and Test Master software for machine control and data processing.

The diameter of each sample was first measured using a micrometer, and then recorded in the machine. The samples are then glued to the sample holders and fixed to the jaws of the testing machine. The edges of the sample holders are cut so that only the fiber is stressed in tension, and a laying tension is applied to the fiber. All the machine's sensors are then set to zero, and a load of 5 kN is applied to the sample at a speed of 5 mm/min. After the test, the data and all the technical parameters (Young's modulus, maximum resistance, elastic resistance, deformation, etc.) are recorded.

4. Results and Discussion

4.1. Structural and Functional Characterizations

- X-ray diffraction analysis

Figure 1 shows diffractograms of treated and untreated *coco nucifera L.* fibers. The major peak is recorded for *coco nucifera L.* fibers extracted by sodium hydroxide-acetone at a diffraction angle of $2\theta = 22.16^\circ$, which corresponds to the (002) crystallographic plane. Another peak also appeared in the diffractogram, one at an angle of $2\theta = 15.89^\circ$ which corresponds to the (111) crystallographic planes. The (002) crystallographic plane represents the crystalline part of the cellulose, while the amorphous part appears at $2\theta = 15.89^\circ$. According to

equation (5) the crystallinity index I_c for the higher peak is calculated and equal to 56.99% which shows that extraction using the sodium hydroxide-acetone mixture resulted in cellulose with a predominantly crystalline phase. The average of crystallite size is calculated according to Sherrer equation and is equal to $1.92 \times 10^{-3} \text{ \AA}$.

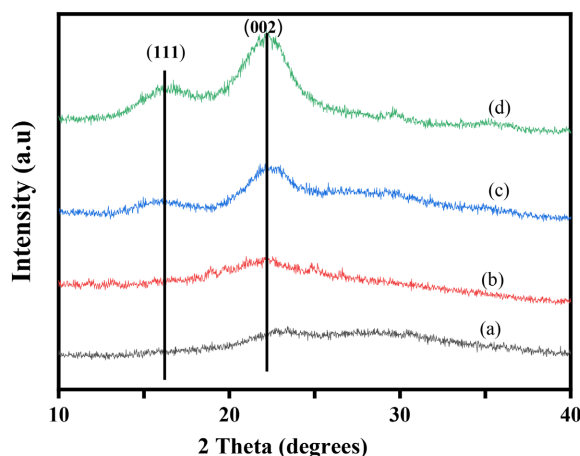


Figure 1. X-ray diffractograms of (a) Untreated *Coco nucifera L.* fibers and (b) *Coco nucifera L.* fibers extracted by acetone (c) *Coco nucifera L.* fibers extracted by sodium hydroxide (d) *Coco nucifera L.* fibers extracted by sodium hydroxide-acetone.

• Fourier Transform Infrared Spectroscopy (FTIR)

Figure 2 shows the FTIR spectra of untreated and treated *Coco nucifera L.* fibers. The different absorption bands are characteristic of *Coco nucifera L.* fibers. These different spectra do not show any significant differences between the peaks. The peak observed at a wavelength of 3347 cm^{-1} corresponds to the O-H group (stretching of the hydroxyl bond) of the cellulose. The peak at 2925 cm^{-1} corresponds to the C-H elongation vibrations of the cellulose. The peak at 1597 cm^{-1} shows the presence of the C-C group in lignin, which has been considerably reduced. Similarly, the peak at 1231 cm^{-1} , which corresponds to the C-O elongation of the acetyl groups in lignin, underwent a decrease. An intense band at 1032 cm^{-1} with a shoulder corresponding to the end of the stretching modes of the C-O acetyl groups increased sharply in intensity; partly because of the increase in the proportion of cellulose in the fibers. The spectra relating to the different IR treatments have a significant influence on the structure of the fibers treatments. The presence of lignin peaks in the fibers spectra shows that the fibers still contain lignin.

• Morphological analysis

According to the images obtained by scanning electron microscopy (SEM) in **Figure 3(a)**, *Coco nucifera L.* fibers has a very clean, smooth surface, free of non-cellulosic matter. Several authors [40] [41] have shown that treating fibers with basic solutions cleans the surface by degrading amorphous constituents such as lignins, hemicelluloses, waxes and fats, since they are soluble in an

aqueous NaOH solution. This treatment cleaned the micropores in the fibers and made the surface smoother with a reduction in diameter. **Figure 3(b)**, **Figure 3(c)** and **Figure 3(d)**, on the other hand, show the presence of debris with a rough surface. This always explains the presence of other bodies such as lignins, hemicelluloses or pectins, waxes and fats. Removal of the hemicellulose gives the cellulose fibrils freedom of movement in the direction of tensile deformation, which increases the tensile strength and surface roughness of the fiber.

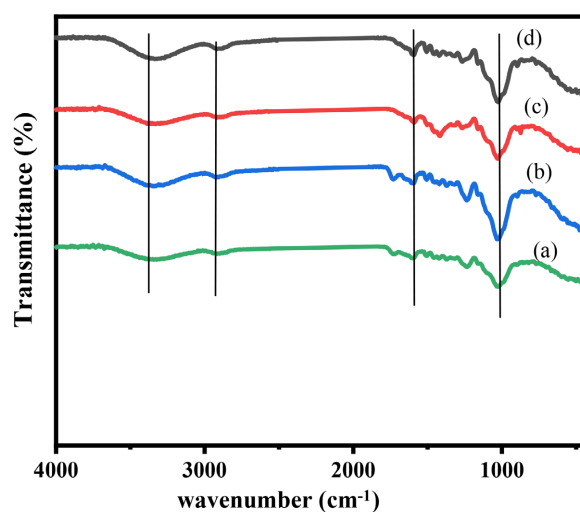


Figure 2. IR spectra of (a) Untreated *Coco nucifera L.* fibers and (b) *Coco nucifera L.* fibers extracted by sodium hydroxide (c) *Coco nucifera L.* fibers extracted by acetone (d) *Coco nucifera L.* fibers extracted by sodium hydroxide-acetone.

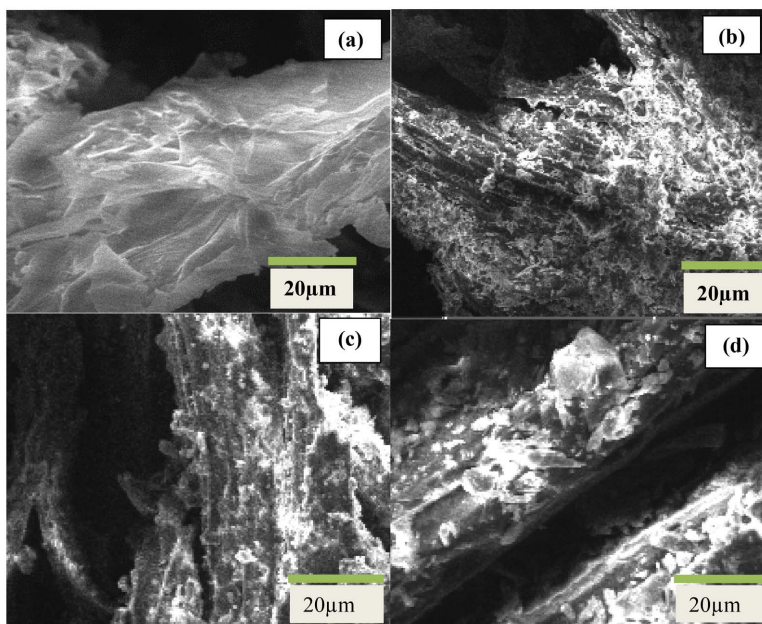


Figure 3. SEM analysis of (a) Untreated *Coco nucifera L.* fibers and (b) *Coco nucifera L.* fibers extracted by sodium hydroxide (c) *Coco nucifera L.* fibers extracted by acetone (d) *Coco nucifera L.* fibers extracted by sodium hydroxide-acetone.

4.2. Physical Characteristics

- **Density**

Figure 4 shows the different densities of fibers treated with different solvents. These density values of between 1.636 and 1.066 are not too far removed from the densities of other plant fibers such as cotton = 1.55, flax = 1.33, sisal = 1.53, alfa = 1.51, jute = 1.44, coir = 1.2, hemp = 1.07 [42]. When lignin, pectins and hemicelluloses are less present in the sample, its density is higher. This density measurement is therefore also a means of verifying the elimination of non-cellulosic matter. Comparison of the values obtained with those for plant fibers shows that the density of coconut fibers is within the average range for this type of fiber. It should be noted that these fibers show interesting results due to the absence of lignin, pectins and hemicelluloses.

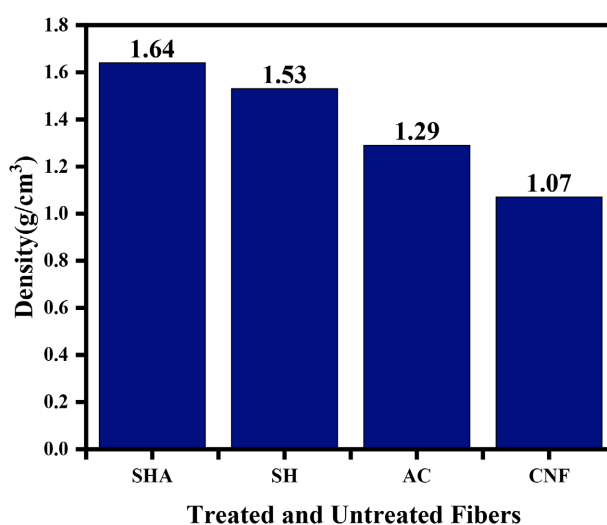


Figure 4. Density of different *coco nucifera L.* fibers.

- **Measurement of water absorption percentage**

Figure 5 below shows the histograms of the water absorption percentages of the fibers treated with the different solvents. These results show that the water absorption rate is 62.428% which can be explained by the fact that during this chemical extraction based on the sodium hydroxide-acetone mixture, these two solvents being polar favoured a better extraction of the fibers allowing the cellulose contained in these fibers to be less encumbered and thus to easily absorb water.

- **Measurement of moisture uptake**

The histograms presented in **Figure 6** below show the moisture content of the different fibers after extraction in different solvents. These results show that the moisture content varies from 9.605 to 7.793, which are values close to those found in the literature [43]-[45]. The values found indicate that a product made from these fibers will offer a fairly good feeling of comfort, since it will absorb up to 9% moisture, a value very close to that of cotton (7% - 8%) and jute (12%).

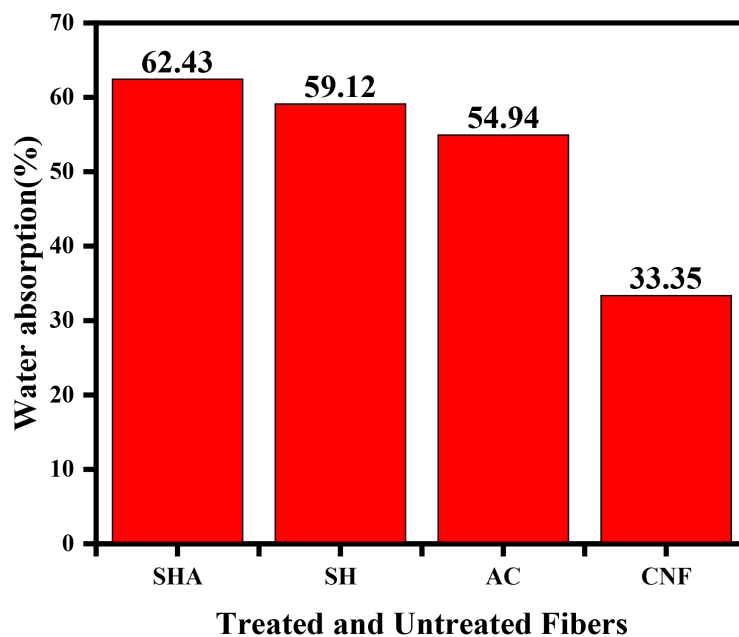


Figure 5. Water absorption percentage of different *coco nucifera L.* fibers.

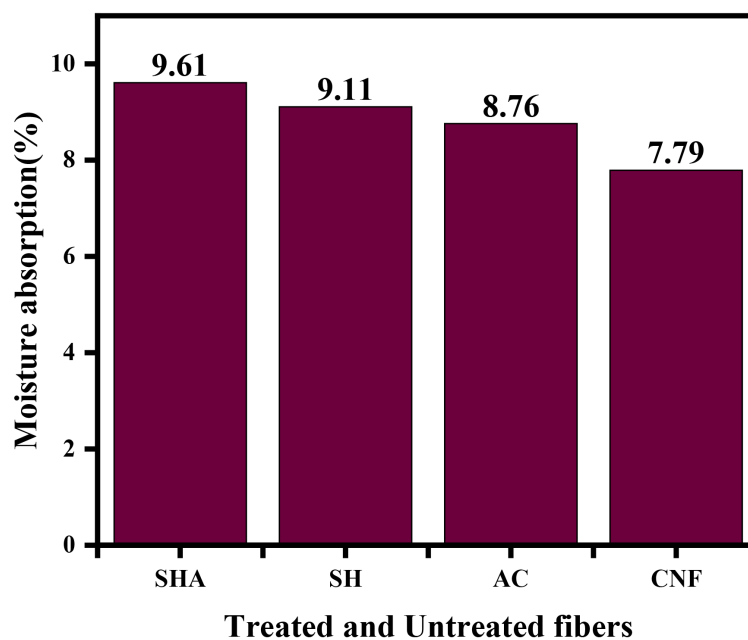


Figure 6. Moisture absorption of different *coco nucifera L.* fibers.

4.3. Mechanical Characterization of Coconut Fibers

Figure 7 shows the values for mechanical strength of untreated and treated of *coco nucifera L.* fibers. Sodium hydroxide-Acetone (SHA) extraction of *coco nucifera L.* fibers increases their mechanical strength.

As for fiber deformation, the difference between these values means that the treatment has effect on fiber deformation (see **Figure 8**).

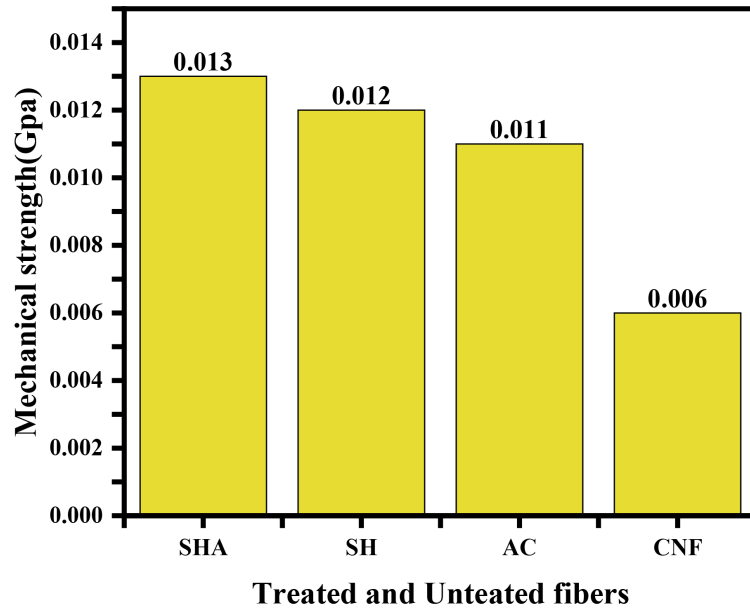


Figure 7. Mechanical strength of different coco *nucifera L.* fibers.

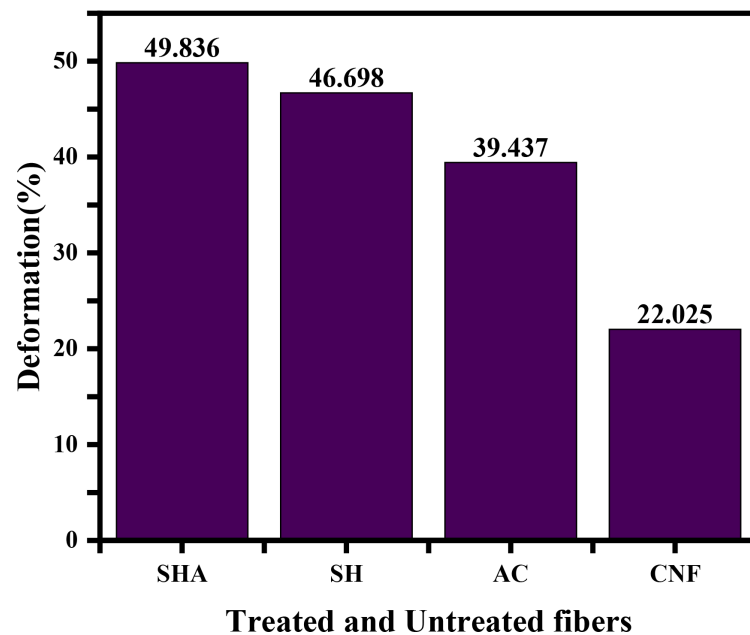


Figure 8. Deformation of coco *nucifera L.* fibers.

Young's modulus, which is the fibers resistance to deformation, shows a significant difference in the values obtained between the different treated fibers (see Figure 9). This reflects the beneficial effect of the treatment, which makes the fibers more resistant to deformation. This is undoubtedly due to the presence of hydroxyl groups on the surface areas of these fibers, as shown by the FTIR analyses. These groups are responsible for the formation of strong bonds and Van Der Valls bonds, and favour the establishment of mechanical bonds.

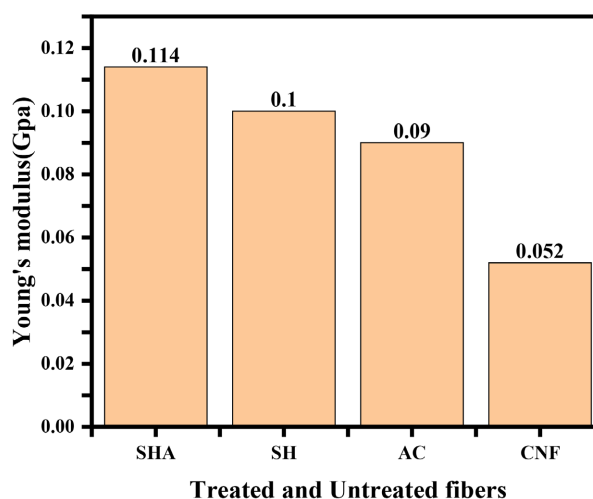


Figure 9. Young's modulus different *coco nucifera L.* fibers.

The force required to break the fiber is known as its tensile strength. The more resistant a fiber is before breaking, the higher its tensile strength. The mechanical tests carried out on the coconut fibers are tensile tests which gave the result of the tensile force and the elongation of the fiber during traction. These results are expressed by curves giving force as a function of elongation as shown in **Figure 10**. The slopes of these curves enabled us to determine the different Young's moduli of each type of fiber extracted.

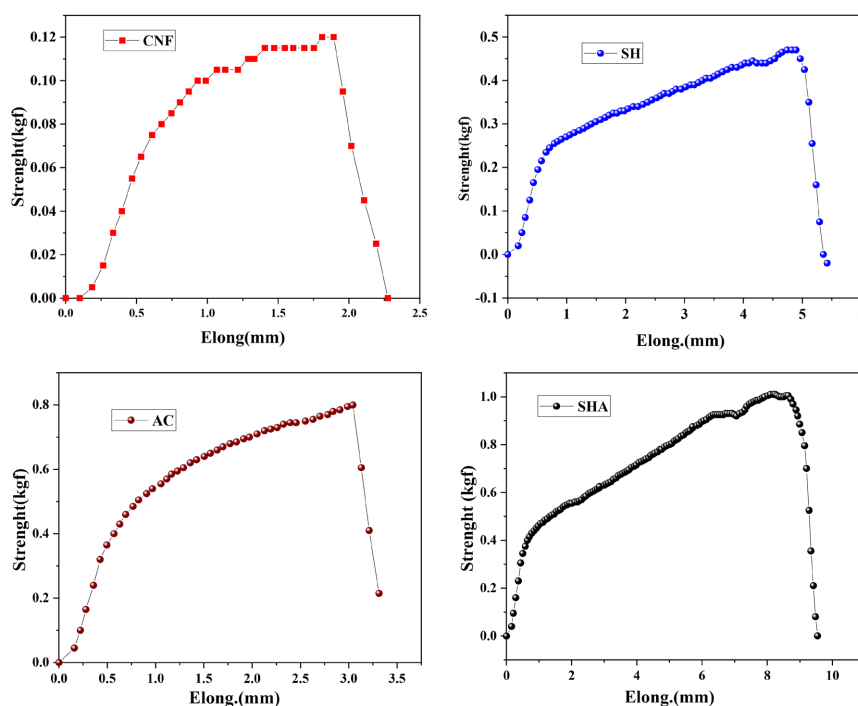


Figure 10. Tensile curves for *coco nucifera L.* fiber(CNF), *coco nucifera L.* fiber extracted by Sodium Hydroxyde (SH), *coco nucifera L.* fiber extracted by Acetone (AC) and *coco nucifera L.* fiber extracted by mixture Sodium Hydroxyde-Acetone (SHA).

The results of the tensile test showed that the tensile strength of coconut fibers (see **Figure 10**) is 0.006 GPa, 0.012 GPa, 0.011 GPa, and 0.013 GPa respectively for coco *nucifera L.* fibers (CNF), coco *nucifera L.* fiber extracted by Sodium Hydroxyde (SH), coco *nucifera L.* fibers extracted by Acetone (AC) and coco *nucifera L.* fibers extracted by mixture Sodium Hydroxyde-Acetone (SHA (**Table 1**)). These values are much lower than those of the other fibers presented in **Table 1**, obtained from the literature, such as cotton, kenaf, sisal and hemp, whose tensile strength is 0.287 - 0.597 GPa, 0.7 GPa, 0.6 GPa, 0.69 GPa and GPa respectively. On the other hand, the elasticity of coconut fibers was higher compared with the same fibers. Coconut fibers appear to be more flexible although they have a low tensile strength. Mechanical properties are directly related to molecular structure properties, fiber length, microfibrillar angle, cellulose content, fiber orientation and cellulose crystallinity. The mechanical characterization carried out is tensile strength to better understand the behaviour of different fibers. It was found that the best extraction of Coco *nucifera L.* fibers was achieved with sodium hydroxide (0.1 mol/L) diluted with acetone in proportions of 50/50, while the best.

Table 1. Mechanical properties of coconut fibers obtained by different treatments and some vegetable fibers.

Fibers	Tensile Stress (GPa)	Elongation (%)	Young's Modulus (GPa)	References
Cotton	0.287 - 0.597	7 - 8	0.005 - 0.012	[46]
Kenaf	0.7	3	0.055	[47]
Linen	0.345 - 1.035	1.3 - 3.3	0.027	[48]
Sisal	0.6	3	0.012	[49]
Jute	0.396 - 0.773	1.5 - 1.8	0.026	[50]
Hemp	0.690	1.6	0.03 - 0.06	[48]
SHA	0.013	49.84	0.114	Present work
SH	0.012	46.70	0.100	Present work
AC	0.011	39.44	0.090	Present work
CNF	0.006	22.03	0.052	Present work

5. Conclusion

The chemical extraction of coconut fibers has been carried out. However, the experiments focused on chemical extraction with the aim of proposing an appropriate method for this fiber. The work involved cleaning the outer part of the coconut fiber flock, then separating the fibers from the inner part. The fibers extracted by the sodium hydroxide-acetone mixture present a linear density of 1.636, the percentage of water absorption of 62.428%, the percentage of moisture absorption of 9.605% compared to other values in the literature shows that this solvent mixture improves the properties of coconut fibers which contains type I cellulose. The tensile stress is 0.013 GPa, the percentage of deformation is 49.836% and the Young's modulus is 0.114 GPa as well as the percentage of

elongation show that these fibers are elasto-plastic. After physical, structural and mechanical characterization, the results show that these fibers can be used in textiles and in other applications as composite materials. The perspectives of this work can be focused on the reaction mechanism of chemical extraction, carry out a thermodynamic and kinetic study.

Conflicts of Interest

The authors unanimously declare that they have no known competitive financial interests or personal connections likely to influence the work reported in this article.

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