Physiochemical, morphological and mechanical characteristics of long fibers extracted from date palm spadix stem

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Abstract

In this work, we improve the process of extracting fibers from date palm spadix stem in order to obtain them long and flexible without lignin, with simple steps and in a short time. As well as the tensile mechanical characterization of fibers treated chemically with sodium hydroxide solution (NaOH) , compared to sisal fibers (Filasse). The results show that the mechanical tensile strength of date palm fibers was higher than that of Sisal ones. This reinforces its suggestion as an alternative to filasse. Further more, infrared spectroscopy (FT-IR), XRD and SEM were used to characterize these fibers.

Keywords: Date Palm, Natural fibers, Chemical extraction, Sisal Fibers, Tensile Strength, FT-IR, XRD, SEM.

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Introduction

Due to the increased demand for sustainable and renewable materials, interest in natural fibers has increased due to their high mechanical properties, light weight and environmental friendliness. Palms are among the most important sources of natural fibers that can be extracted fibers from its various parts, namely the mesh, midribs, leaflets and spadix stems, which are the subject of current research because of a large number of byproducts of the annual pruning, making them one of the most abundant sources of natural fiber available.[1]

The date palm is à member of the palm family, usually distributed in the Middle East, North Africa, the Canary Islands, Pakistan, India and the United States (California). The stems of palm trees are covered with a net made of single fibers. Generally, these fibers form a natural woven mat of intersecting fibers with different diameters. The usability of date palm fiber (DPF) in fiber composites will open up new markets for products that are usually considered waste and low-value products.[2]

The natural fibers are derived from lignocellulose and are therefore inevitably hydrophilic, as they contain strong polar hydroxyl groups. Cellulose is one of the most important biorenewable elements, and it is abundantly available in a non-toxic and cheap form, which has made it possible to use it widely in various fields and is one of the most competitive ingredients for synthetic polymers, as it is included in a wide range of industries such as textiles, pharmaceuticals, paper and food, in addition to composite materials and additives[3], [4].Since ancient times, plant fibers taken from the processing chain of the textile industry have been approved for use in the reinforcement of composite materials, and then the techniques used to separate the fibers were developed to produce the mechanical, biological and chemical extraction technique adopted during this research.[5][6]

In recent years, the study of characterizing plant fibers has become more and more important[7]. They studied the possibility of incorporating date palm fibers into composite materials similar to geosynthetics. For this study the stems was taken from the Deghlet Nour date palm (local names). Most of the previous studies found that a low concentration of sodium hydroxide solution during the extraction process produced fibers with good mechanical properties, so we adopted 1% from sodium hydroxide. [8][9][10]

The main purpose of this research is to extract long and flexible stretched fibers in a short time by optimizing the chemical treatment conditions.

Materials and Methods

Spadix Stem fiber

The word fiber is often used to denote the basic components of the organizational structure. There are many kinds of them according to their properties, such as shorts[11],trousers[12], natural[13], [14], synthetic fibers, etc. In the current research, the fiber is extracted from the by-products of date palm, especially from the spadix stems of Deghlet Noor harvested in October 2020, which comes from the university farm of Ouargla in south-eastern Algeria.

Fiber extraction and Optimization

During this research, it was, mainly, adopted chemical extraction methods with some mechanical steps, to save time and energy.

Mechanical Separation

Date palm spadix stem was cut with a saw into pieces of 50 to 120 cm in length. Then all the pieces cut into thin sticks so that the thickness of one stick is about 2 mm.

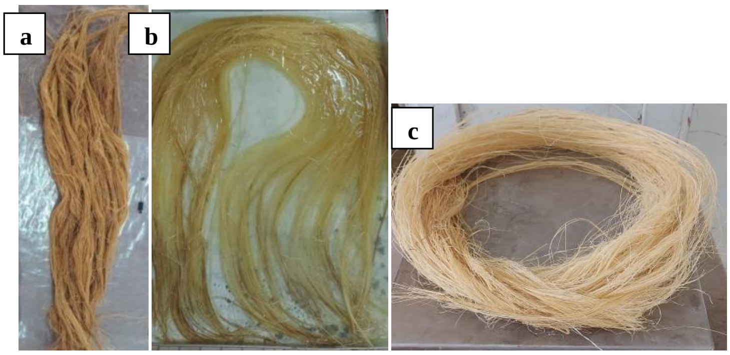
Pretreatment

This stage aims to sterilize the stems of the date palm spadix and facilitate its dissolution in the next step. Leave the sticks in the water for an hour and at a temperature of 100°C. Then the sticks were left to dry naturally at room temperature.

Chemical dissociation

This is the most important step. The pre-dried fibers were placed in a bowl of NaOH solution (01%) for 3 hours.

After completing the chemical dissociation, we get dark brown fibers, so we searched for a bleaching substance that helps to remove the lignin layer. The fibers were immersed in sodium hydrochloride (NaOCl) solution, and then washed well with distilled water. The extraction protocol is detailed within (Patent: deposit N° 220315 of 31/03/2022 INAPI Algeria, It is in the process of being published). The extracted fibers are shown in picture 1. It is worth noting that 20g of spadix stem produce 11g of fibers, either an extraction rate of 55%.



Picture 1. Spadix Stem fibers: (a) Treated fibers, (b) fibres after bleaching, (c) fibers dried after 3 days

Sisal fibers (Filasse)

Sisal fiber or what is called in the market as Filasse were purchased from the city of Ouargla (Affiliate of the import company southeast of Algeria SARL SO.CO.FI). These fibers were approximately 0.04 to 0.14 millimeters in diameter and 1 to 1.5m[15]. Sisal fibers used are shown in picture 02.

Picture 2. Sisal fibers (Filasse)

Chemical composition analysis

The chemical composition of spadix stem fibers was analyzed As follows:

1.00 g of palm fibers recovered after treatment with NaOH solution. Then 560 ml of water is added to reduce the acid content to 3% and the solution is heated to boiling and refluxes for 4 hours. The residue is then filtered and washed with 500 ml of water, dried at 100°C and weighed. Since lignin is insoluble in 75% sulphuric acid (H_2SO_4),this residue consists essentially of lignin.

% Lignin = $W_2/W_1*(100 – W_3)$

W_1 and W_2 being the masses of date palm spadix stem fibers before and after treatment. W_3 being the result obtained after the fiber solubility test in the ethanol-toluene mixture (%)[16].

The hemicellulose of the fibers was determined by using a Hydrogen peroxide (H_2O_2) solution.
% Holocellulose=W2/W1*(100 – W3)

W_1 and W_2 being the masses of DPSSF before and after treatment.

W_3 being the result obtained after the fiber solubility test in the ethanol-toluene mixture (%)[17].

3.00 g of dried holocellulose, obtained at the end of the previous test, are treated with a sodium hydroxide solution at 17.5% by weight, for 30min. 50 ml of distilled water are then added and the35mixture is stirred for 5 min. Then, the residue is filtered and washed with an 8.3% sodium hydroxide solution then with 40 ml of 10% acetic acid, with 1000 ml of water and finally dried at 100°C until weight constant.

% Cellulose = W2/W1*W3

W_1 and W_2 being the masses of date palm spadix stem fibers before and after treatment.

W_3 being the percentage of holocellulose obtained at the end of the previous test.[18]

Fourier Transform infrared analysis (FT-IR)

The analysis of FTIR spectra was conducted using the Cary 630 FTIR spectrophotometer, desperately recorded the samples on the **Spectral range** KBr 6300–350 cm^{-1},-1cm^{-1} area and an accumulation of 10 scans per analysis. One of the main advantages of this technique is that samples can be analyzed without special preparation[19]. FTIR can identify chemical compounds by identifying functional groups within the sample[20].

Scanning Electron Microscopy (SEM)

In this work, The Prisma E device from Quanta SEM was used to investigate the morphology and the chemical composition of treated fiber. The diameter of the fiber is between 20 and 60 micrometer and the length is 0.3 millimeters, where was taken the morphology of longitudinal direction and cross-section from the dried spadix stem fiber.

X-ray diffraction (XRD)

The crystallinity of Spadix stem fibers was examined using D8-Advance (Bruker) XRD with CuKα Radiation (=0.1541 nm) in the 2θ range of 5°–60° at a scan speed of 2 °C/min.

Tensile strength on individual fiber

Using a universal stretching machine (Multi Test 2.5i Mecmesin) the tensile strength of the extracted and filasse fibers was measured, the displacement velocity of which was set (V = 20 mm/min). We conducted experiments under climatic conditions (T=30°c) and (RH=20). The fiber area is then calculated from the largest and smallest diameters by following the ellipse model. As William GARRAT explained in his article, unlike the circular model, this model can calculate an area close to the actual area of the fiber. We can determine the diameters with an accuracy of 0.01 µm. But it should be noted that concave surfaces cannot be measured using this system[12].

Results and discussion

Chemical composition analysis

The Sisal fibers is generally composed of 65.8%cellulose, 12% hemicellulose and 9.9% lignin [21]–[23],While the chemical composition of spadix stem fibers showed a change in the proportions of the compounds after extraction, and they are presented in the table below. Where can be observed the increase in the parentage of cellulose and hemicellulose.

Table 1. Chemical composition of fiber before and after treatment

	Before treatment	After treatment
Lignin (%)	29.47	26.29
Hemicellulose (%)	26.2	13.17
Cellulose(%)	43.46	73.46

Fourier Transform infrared analysis (FT-IR)

Figure 1 shows the spectrum of the treated fiber, which shows a broad band at 3340 cm^{-1}, mainly due to the presence of OH groups in the fiber structure. The peak around 2890 cm^{-1} corresponds to the vibration of the aliphatic C-H chain. We also noticed the presence of peaks at 1600 cm^{-1} and 1030 cm^{-1}, reflecting the presence of double C=O and single C-O bonds, respectively [24]. In fact, this has been demonstrated by [25]. These fibers were extracted from date palm spadix stem (DPSS) treated with sodium hydroxide solution (5% for 24 h at 23°C) and showed similar spectra to those found in this work. Similarly,[8], studies on natural fibrous tissue from the date palm showed that treatment of fibers with soda solutions at concentrations of 5%, 2.5%, 1.5%, 1% and 0.5%, such as with untreated Holding at 100°C for 1 hour had no significant effect on the FTIR recording bands of the treated fibers compared to the treated fibers. Whereas, the FTIR of date palm spadix stem fibers before treatment is reported in the following study[18]

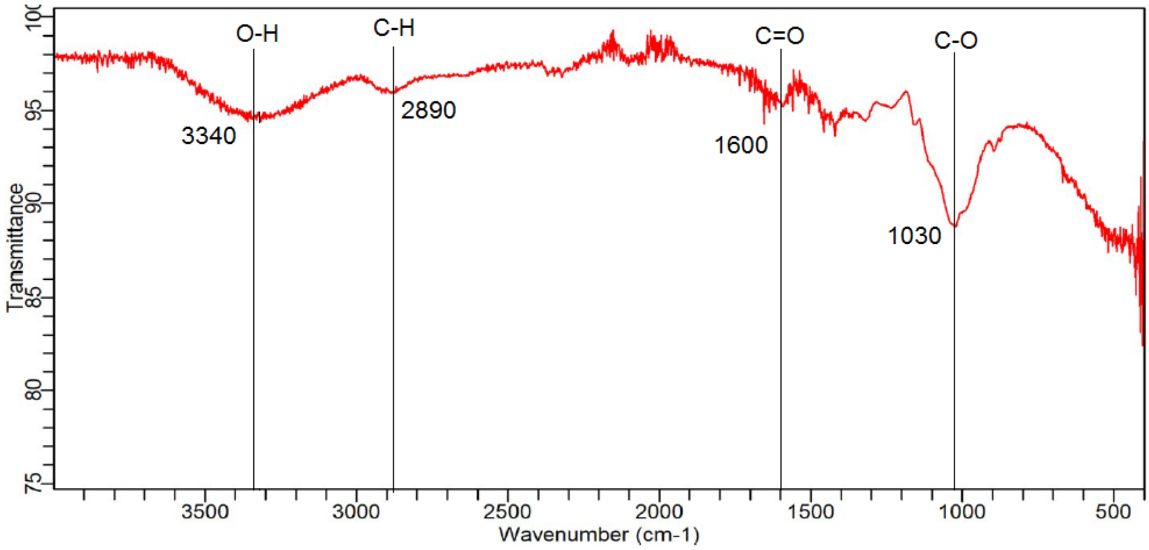


Figure 1. FTIR spectra of Alkali treated date palm spadix stem (DPSS)

Table 2.The main observed IR bands and their assignments.

Wavenumber (cm^{-1})	Assignment	Ref.
1030	Aromatic C-O groups in polysaccharides	[26]
1600	C=O carbonyl groups	[27]
2890	C–H bands in methyl and methylene groups	[3]
3340	H-bonded OH groups stretching	[28]

Scanning Electron Microscopy (SEM)

Scanning electron microscope images are shown in Figures 2, 3 and 4. Or the Figure 2 shows a SEM micrograph of individual cellulose microfibers before treatment with alkaline solution and bleaching. Figures03 and 04 present the scanning electron microscopy of treated spadix stem fiber.

Untreated fibers consist of bundles of fibrous tubes, longitudinal and cross-sectional views shown in Figure 2a [29]. The fiber surface is covered with lignin and hemicellulose structures[30]. However, the cross-sectional view of the untreated fiber in Figure 2b shows unfibrillated fibro-vascular bundles[31]. In the case of the alkali-treated stem fibers shown in Fig. 3a,b, the alkali treatment was found to have a positive effect on the cleaning of the fiber surface[32].The cross-section is in the form of a thin slice, as shown in Fig. 4a,b, noting that it contains a tiny lumen. According to reports, these micro-sized cellulose fibers are composed of strong hydrogen-bonded nano-fibers.[28]

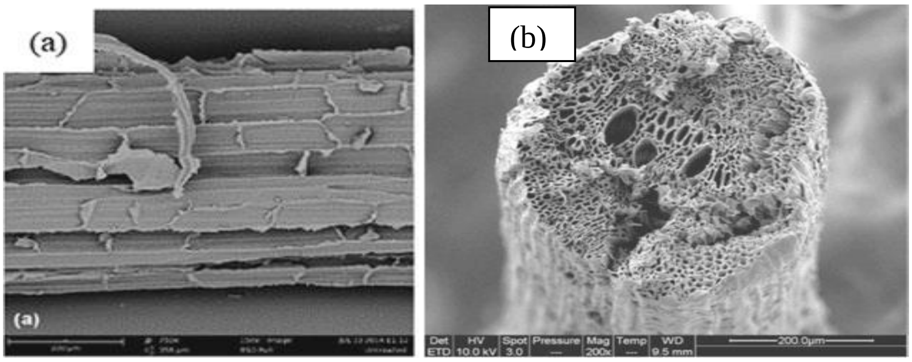


Figure 2.SEM images showing the untreated date palm spadix stems fibers: **a** longitudinal views of the untreated fiber [29] , and **b** cross-section of the fractured untreated fiber [31]

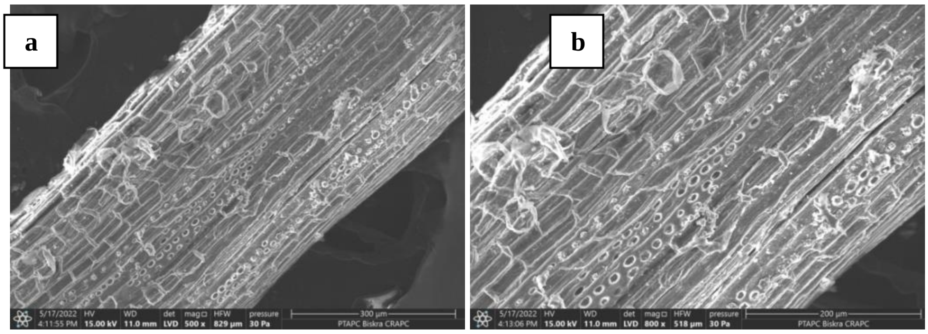


Figure 3.SEM images showing the longitudinal direction of treated date palm spadix stem fiber magnifications: (a) x500, (b) x800.

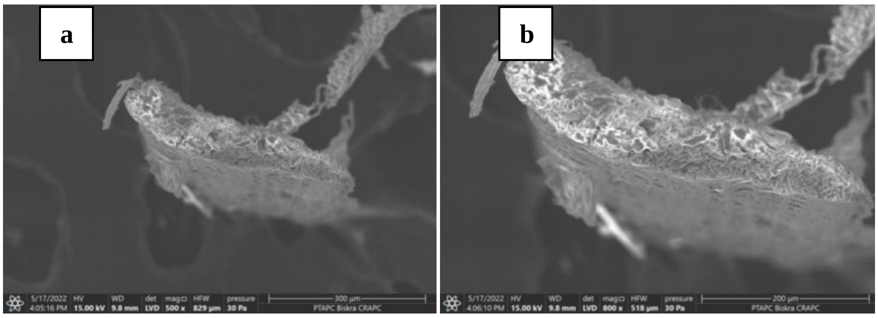


Figure 4.SEM images showing cross section of treated date palm spadix stem fiber magnifications: (a) x500, (b) x800.

Figure 5 shows the EDX spectra of the date palm treated fiber samples, while the elemental compositions of the samples were determined and summarized in Table 03. Determined from the data in Table 03.

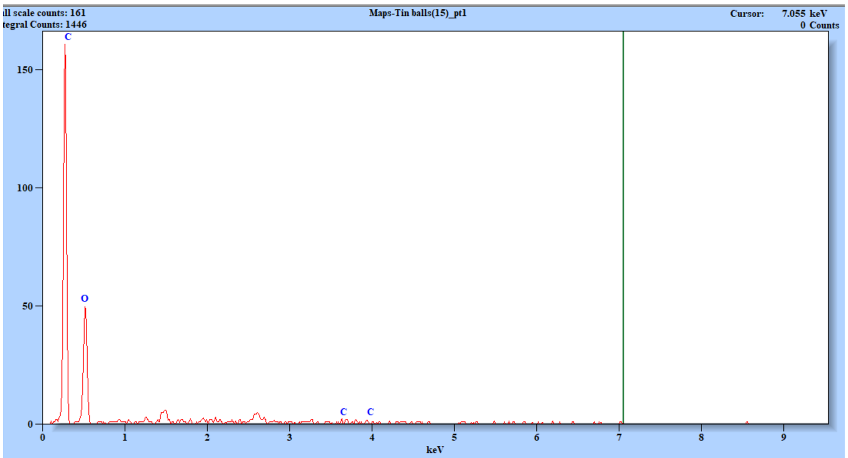


Figure 5. EDX spectra of Date Palm Spadix Stem fibre sample

Table 3.The elemental composition of treated fiber

Element	Weight (%)	Atom (%)
O	94.36	97.67
C	5.64	2.33

The amount of carbon is relatively high in the fiber sample, compared with oxygen as shown in fig.5 which was expected, because as we know, the structural formula of cellulose shows that it contains oxygen and carbon atoms, in addition to hydrogen, which did not appear due to the difficulty of detecting because it contains one electron. Accordingly, there are no results available in the literature to compare the final composition of date palm fibers, as determined in this study.

X-ray diffraction (XRD)

The XRD data allow studying the crystallization of the sample due to the diffraction peaks of cellulose crystals. Fig 6 Shows the X-ray diffraction curves of the treated fibers. There are two peaks at 2θ = 22° and 16°, indicating the crystal structure of cellulose[33].

An empirical procedure was used to obtain the crystallinity index (I_{Cry}) of the sample, as given in Equation (1): $Icry = (I_{max} - I_{min})/I_{max} \times 100$ ....(1)

Where I_{200} is the crystalline peak corresponding to 22° intensity and I_{am} is the amorphous peak corresponding to 16° intensity[34].

Basically, the crystallinity index (Icry) indicates the degree of crystallinity. This finding is consistent with the results of SEM analysis (Fig. 3.4 (a-b)). Therefore, the removal of non-cellulose content was achieved, the amorphous region was degraded, and a more ordered crystal structure was obtained. Whereas, the XRD of date palm spadix stem fibers before treatment is reported in the following study [18].

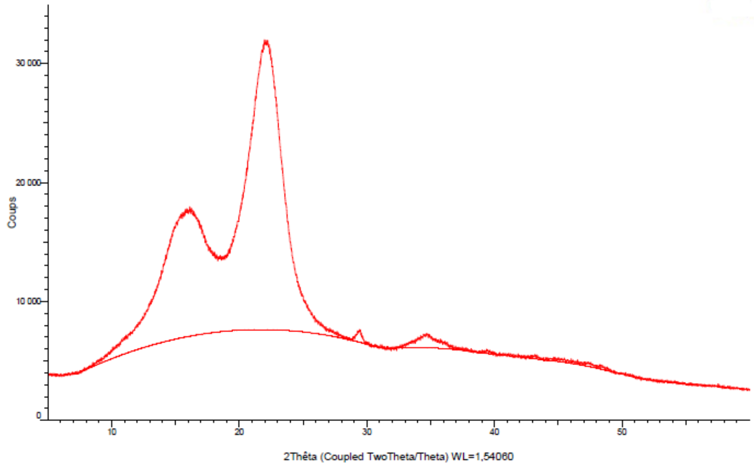


Figure 6. X-ray diffraction curves of treated date palm spadix stem fibers (DPSSF).

Tensile strength

We prepared many samples for each type of fiber with equal length (10 cm) and measured their diameters with a digital caliper device after that we selected several samples with diameters between 0.09 and 0.13 mm of each type of fiber for comparative accuracy, then conducted the test and recorded the results. The values of the force and displacement of the moving crosses are recorded simultaneously. Young's modulus and constraints can be estimated using classical relations of RDM and continuum mechanics. Since the fibers are very thin, we glued each fiber with rough paper from both ends to be fixed in the tensioning machine. As shown in picture number 03.

Table analysis shows that the dispersion of the tested fiber diameter and the mechanical property values obtained are very important. This confirms the heterogeneity of the materials used. The test parameters that may affect the results can be: the accuracy of the instrument, the measurement length, the deformation speed, the type of jaws (fixtures) of the machine, and the accuracy of the machine test. The fiber characteristics, age, processing type (fiber extraction method), its arrangement in the cluster arm and its microstructure are influencing factors[35], [36].

The filasse shows average mechanical characteristics especially of the ultimate stress and young's modulus. While we recorded relatively higher value for treated palm fibers, on the other side the deformation in the treated fibers, it showed fewer results than the filasse, and through it, we conclude that the elastic domain of filasse are wider than spadix stem fibers. Table 04 highlighted the results of the tensile experiment for both date palm spadix stem and filasse while a figure 7 and 8 shows the fibers resistance curve.

Picture 03: Date palm Spadix stem samples fibers mounted on a strip of paper for tensile testing

Table 4. Tensile strength of date palm spadix stem fibers (DPSSF) and Filasse individual fiber Results

	DPSSF	Filasse
Ø (mm)	0.09-0.13	0.09-0.12
L (mm)	100	100
F_{moy} (N)	15	11
σ_{moy} (MPa)	**1486.85 ± 514**	**1224.77±252**
ε (%)	3.25±0.9	5,58± 0.5
E (GPa)	45.72±12.23	21.95±6.78

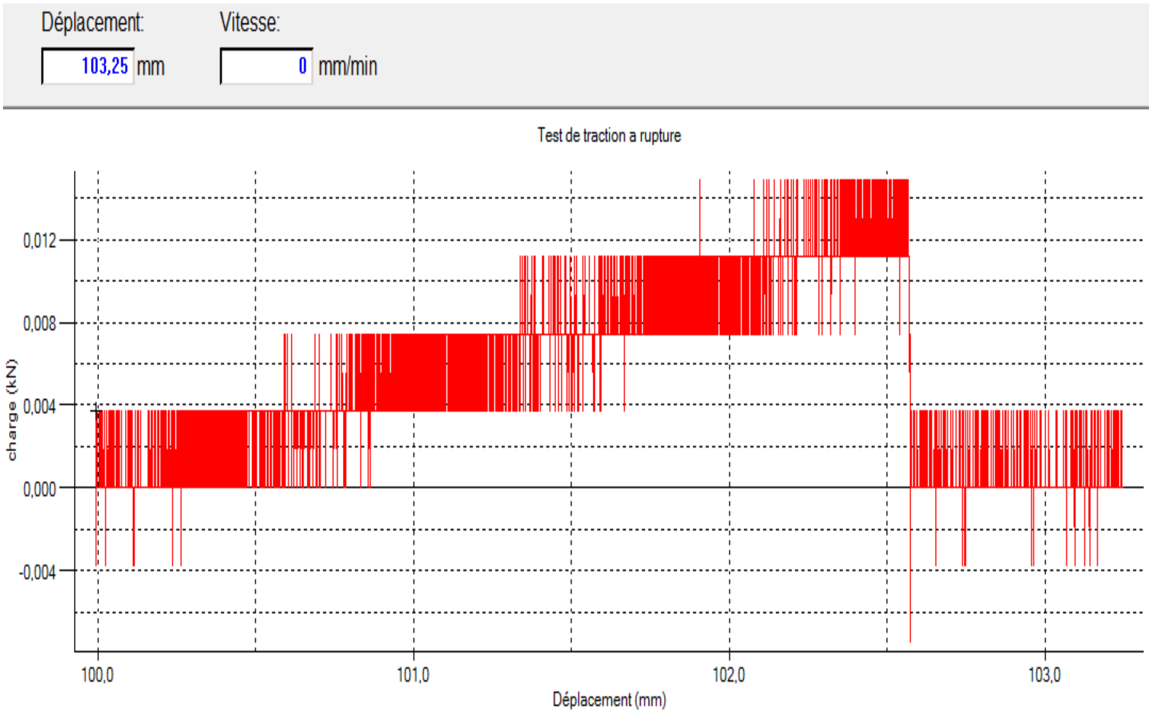


Figure 7. Curve (Force-deformation) in tensile strength of date palm spadix stem fibers

(DPSSF)

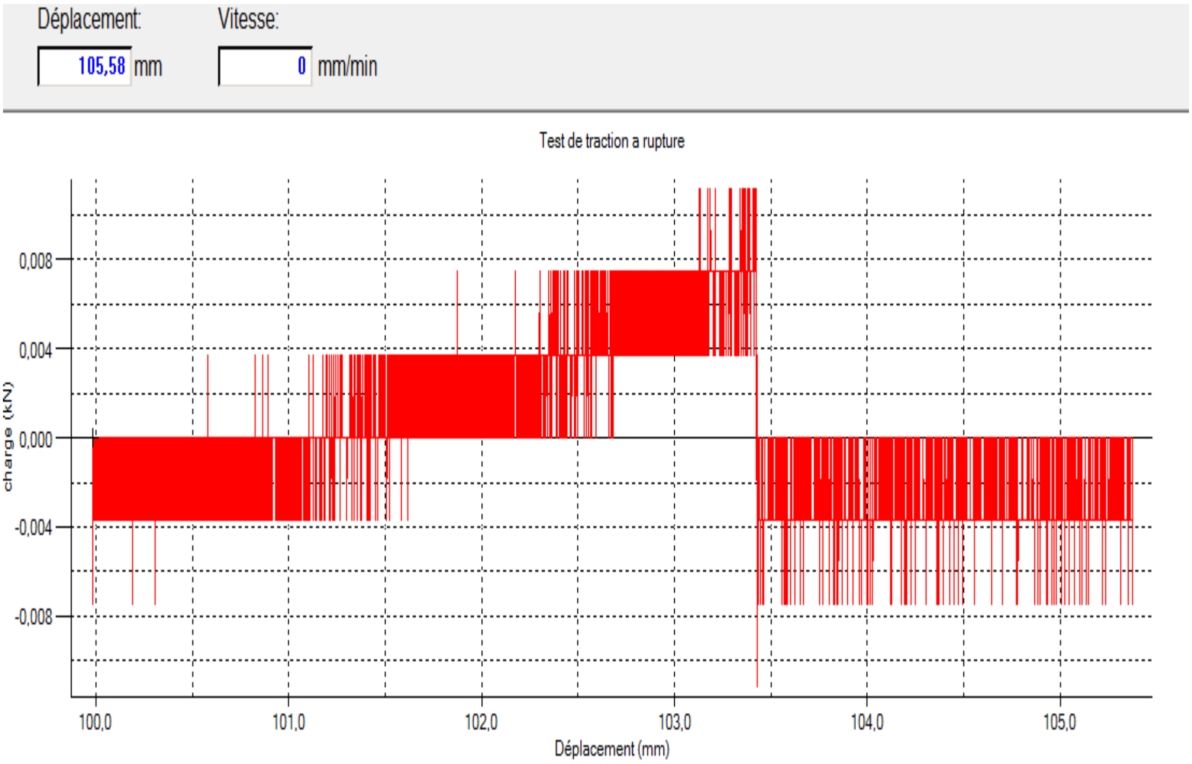


Figure 8. Curve (Force-deformation) in tensile strength of Filasse

Results evaluation

Figure 9 and Table 5 show a comparison of the tensile strength behavior results of the tests performed on date palm spadix stem fibers in several regions in the world. The analysis of the results shows that the fibers extracted from Ouargla palms (Deghlet Nour) have much higher properties (tensile strength) compared to the rest of the regions. This can be attributed to the composition of the soil in which the date palm trees were grown (Ouargla –Algeria in this study).

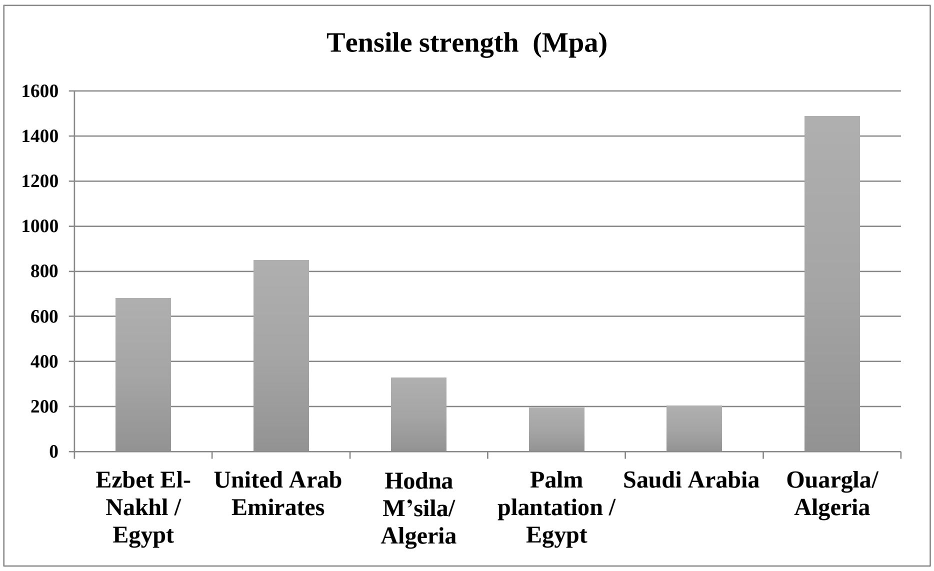


Figure 9. Comparison between tensile strength of date palm Spadix stem fibers

Table 5. Comparison between tensile strength of spadix stem fibers extracting chemically in several regions

Region	σ (MPa)	E (GPa)	Treatment	References
Ezbet El-Nakhl / Egypt	680	13-19	(2%for 2h) NaOH	
United Arab Emirates	850 ± 247	**150 ± 191.5**	(1% for 1h) NaOH	
Hodna M'sila/Algeria	328 ± 119	11.6 ± 4.8	(0.5% for 12h) NaOH	[31]
palm plantation / Egypt	195 ± 28.8	8.81 ± 1.5	(5% for 3h) NaOH	[37]
Saudi Arabia	203	-	(1% for 1h)NaOH	[9]
Ouargla/Algéria	**1487± 514**	45.72± 12.23	(1% for 3h) NaOH	current study

Conclusions

This study is concerned with defining an easy and fast method while maintaining permissible physico-mechanical properties of date palm Spadix stem where was giving a full explanation about the process of chemical extraction for spadix stem fibers using sodium hydroxide NaOH and sodium hypochlorite (NaOCl)solution.

Long fibers were obtained with the aim of using them in the production of composite materials such as geocomposites. Once the fibers were extracted, they were characterized using FTIR,XDR and SEM techniques in addition to conducting a tensile test on it, and the results were compared with Filasse fibers and with the same type of fibers in different areas to confirm the extent of its resistance. It was found from the results that the fibers extracted from the spadix stems of Deglet Nour palms have high tensile strength compared to the other selected fibers, as these techniques proved the success of the extraction process.

The results of this study lead us to commercial dimensions which allow using date palm Spadix stem fibers as an excellent alternative to filasse in the reinforcement of plaster at hot-dry climate.

The experimental data of this study are available in Figshare with the identifier https://figshare.com/account/articles/22692211 Example from: https://doi.org/10.6084/m9.figshare.22692211

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