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EFFECT OF COMBINED CHEMICAL TREATMENT ON PHYSICAL, MECHANICAL AND CHEMICAL PROPERTIES OF POSIDONIA FIBER

S. Zannen[1], L. Ghali[1], M.T. Halimi[1] and M. Ben Hassen[1], [2]

[1]Textile Engineering Laboratory, Monastir University, Monastir, Tunisia

[2] College of Engineering: Industrial Engineering Department, Taibah University Saudi Arabia

ABSTRACT :

The aim of this study is to investigate the effect of chemical treatment method on the properties of Posidonia fibers. The chemical treatment which was carried out is a combined hydrogen peroxide and sodium hydroxide treatment. First, an investigation of the treatment processes was undertaken. Secondly, the physical properties (linear density, diameter and ratio length per diameter), the mechanical properties (tenacity, elongation) and chemical properties (FT-IR spectra and X ray diffraction) of posidonia fibers were investigated. The optimum operating conditions were indentified using a factorial design.

KEYWORDS: Posidonia fiber, combined treatment, physical properties, mechanical properties, chemical properties.

1. INTRODUCTION

Posidonia oceanic is a species of seagrass that is endemic to the Mediterranean Sea. This marine plant loses leaves in autumn, and the cast litter deposits can be found mainly along sandy coasts, forming wedge structures, from a few centimetres to several meters thick, denominated 'banquettes'. In the Mediterranean region, where summer tourism is an important income, the banquette is often removed because it is believed to reduce the value of beaches and every summer the beaches must be cleaned. The valorization of this available biomass can be the solution of that problem [1,2]. On the other hand, environmental concerns have led to an increased demand for renewable materials in many industrial applications. Then, this aquatic biomass represents an abundant, inexpensive, and readily available source of renewable lignocellulosic biomass for the production of environmentally friendly industrial products and has received increasing attention. In the last few years, P. oceanic has been studied as: a low-cost adsorbent for removing dyes or phenol [3], a substrate for papermaking [4], a starting material for cellulose derivatives [5] and a reinforcement on composites [6]. However, to the best of our knowledge, no data has been reported yet regarding the chemical treatment of these materials in order to improve their morphological and mechanical properties as fillers in composite materials.

Many chemical treatments such as sodium hydroxide, silane, acetic acid, acrylic acid, maleated coupling agents, isocyanates, potassium permanganate, peroxide, both sodium hydroxide and peroxide hydrogen, etc., were widely used for modification of fiber surface [7-15]. Moreover, several researches show a significant influence of these chemical treatments on physical, chemical and mechanical properties of natural fibres. Then, the effect of combined chemical treatment on physical, mechanical and chemical properties of Posidonia fiber was studied.

2. MATERIALS AND METHODS

2.1. Materials

The balls of Posidonia are collected from Hergla's beach. It was manually frayed and placed on a horizontal opener. The balls opened manually are driven by a rolling lurking and then they are engaged in a threshing cylinder. Subsequently, they are driven by means of a toothed roller in order to separate fibers. By centrifugal force and aspiration the good fibers are driven

upwardly and the waste falls down. After this mechanical treatment we obtain the fibers shown in figure 1.

Figure 1: Raw Posidonia fibers

2.2. Methods

The fibers were treated by means of a chemical treatment under pressure and agitation using a Datacolor AHIBA MSTRI. For this, we used the Tagauchi L16 design (Table 1.). An experimental database has been elaborated by varying the treatment parameters. In this database (16 tests), we used as input variables the temperature, the extraction time, and the soda concentration. The outputs are the fiber yield, the diameter, the density, the linear density and mechanical properties.

The raw fibers were immersed in the following extraction bath:

- 5 g of raw fibers
- Liquor ratio = 1/40
- Hydrogen peroxide : 25 ml/L
- Temperature (T (℃)) ranges from 60 ℃ to 120 ℃.
- Duration (D (min)) of treatment ranges from 30 to 120 min.
- Sodium hydroxide concentration (C (N)) ranges from 0.2 N to 0.8 N.

Table 1: Features of the Tagauchi L16 design

	Levels			
Factors	1	2	3	4
Soda Concentration (N)	0.2	0.4	0.6	0.8
Temperature (℃)	60	80	100	120
Time (min)	30	60	90	120

After treating the raw Posidonia fibers, it is rinsed with water several times and the obtained fibers are dried to the ambient air for 48h. These fibers were characterized by means of physical, mechanical and chemical analysis in order to define their properties. The tests must carry out on a batch of conditioned fibers to a normal atmosphere (relative humidity: 65 % ± 4 %, temperature: 20 ℃ ± 2 ℃).

2.2.1. Morphological properties

The technical Posidonia fibers obtained are morphologically characterized. The specimens were observed using a Scanning Electron Microscope (SEM) to characterize the morphology of treated and untreated fibers.

2.2.2. Linear density

The measurement of linear density (title) of Posidonia fibers is described according to the standard ISO 1973 while weighing known lengths of the fibers.

2.2.3. Fineness measurement

The measurement of the fineness of Posidonia fibers is given by measuring the ratio of length by diameter. The average apparent diameter was measured with the profile projector according to the French standards NF G 07.004. The test is carried out on 300 fibers chosen at random.

2.2.4. Strength and elongation at break

We determined tenacity (cN/Tex) and elongation (%) of Posidonia fibers by determining the fracture toughness of the fiber bundles according to French standard NFG 07-080. We used the steleometer.

The tensile test is carried out on a batch of 50 fibers according to ISO 5079 relating to the determination of the strength and elongation at break under tensile stress. The length between clamps is taken equal to 10 mm. These tests were conducted on a FAVIMAT Fiber Test with a constant speed equal to 10 mm/min and a measurement cell of 32 N.

2.2.5. Yield measurement

Yield of fibers (R %) is measured by the ratio between the final mass of the fibers after chemical extraction process (Mf) and that of the Posidonia fiber before chemical extraction process (Mi).

The measurement of these two weights is performed using the gravimetric method in accordance with standard NF G 08-001.

$$\mathbf{R\ (\%) = \frac{Mf}{Mi} * 100 \quad (1)}$$

2.2.6. Fourier transform infrared spectroscopy and X ray diffraction of Posidonia fibers

The FTIR spectra of raw and surface treated natural fibers were recorded in a Perkin-Elmer FT-IR spectrometer Frontier. Absorbance was measured over a range of wave number from 4000 to 400 cm^{-1}.

Wide angle X-ray diffraction (XRD) analysis was carried out with a Panalytical X' Pert PRO MPD to investigate the crystallinity of Posidonia fibers. XRD patterns were obtained under Cu Kα radiation at 40 kV and 150 mA in reflection mode, with 0.017 ° step and 22 s of counting time. The angle ranges between 5,006 ° and 45 °. The crystallinity index (CI) was calculated by using equation (2), where I002 is the maximum intensity of the I002 lattice reflection and I101is the maximum intensity of X-ray scattering broad band due to amorphous region of the sample.

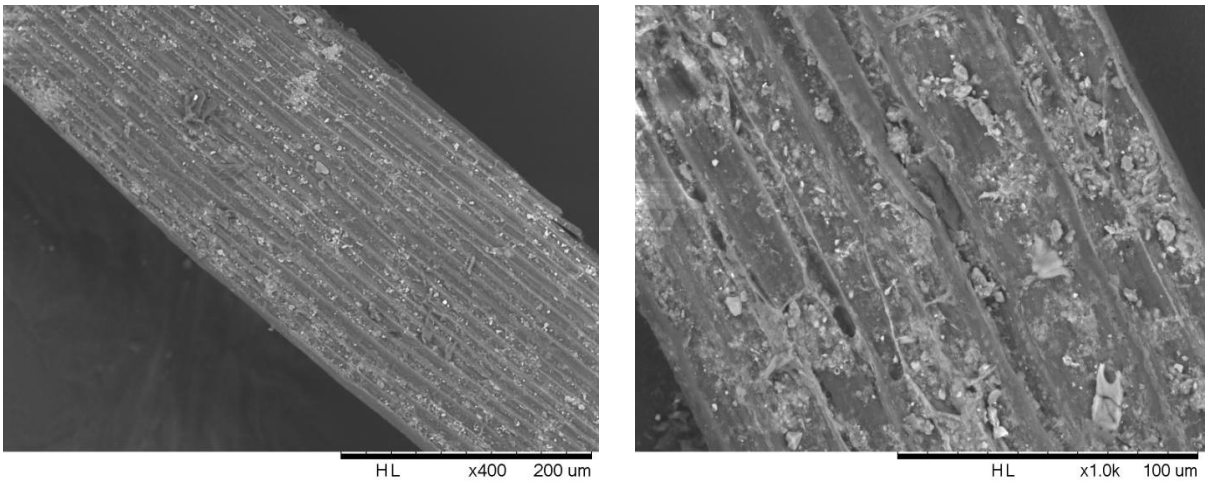
$$\mathbf{CI\ (\%) = [(I_{002} - I_{101})/I_{002}] \times 100 \quad (2)}$$

3. RESULTS AND DISCUSSIONS

3.1. Effect of the treatment processes on the morphological properties of Posidonia fibers

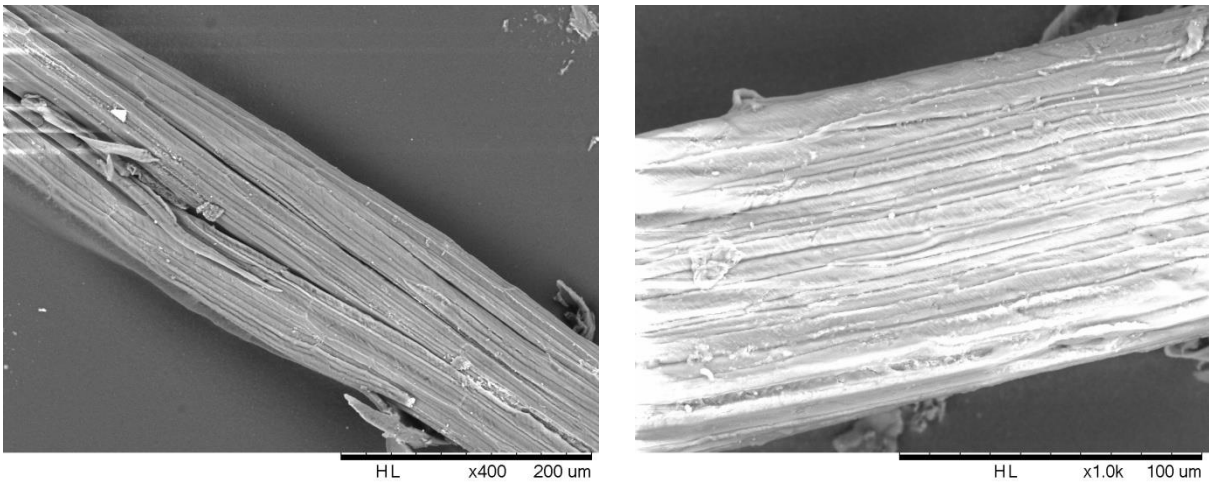
The characterization of fiber morphology is important since its influence on other processing methods of the fibers and the quality of products from it.

All fibers have a common structure, but their physical properties can vary in a substantial way depending on the method and conditions carried out for extraction.



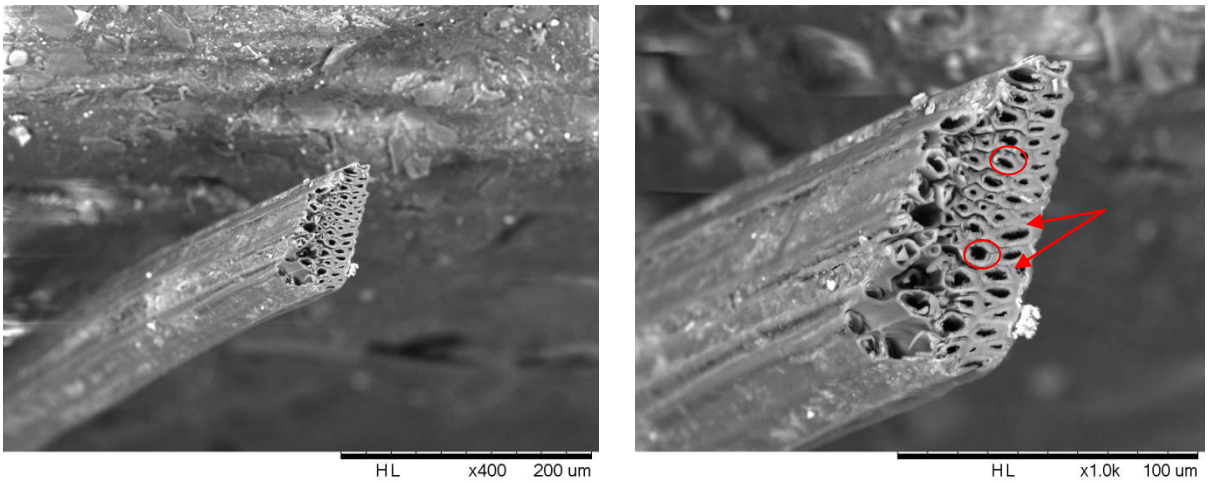
(a) **(b)**

Figure 2. SEM micrographics of untreated Posidonia fibers



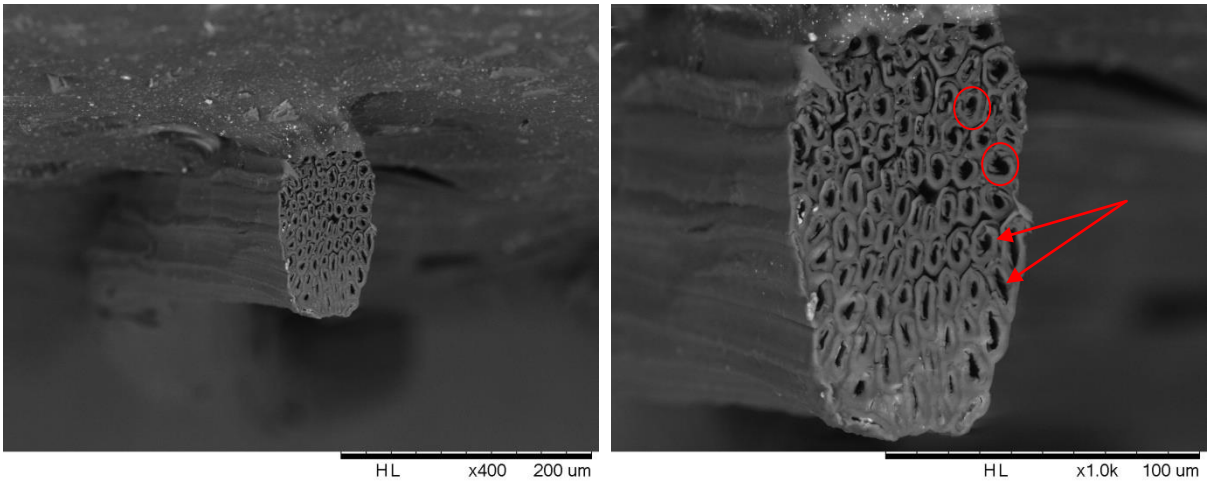
(a) **(b)**

Figure 3. SEM micrographics of treated Posidonia fibers



(a) **(b)**

Figure 4. SEM micrographics of cross section of untreated Posidonia fibers



(a) **(b)**

Figure 5. SEM micrographics of cross section of treated Posidonia fibers

Figure 4 and Figure 5 represent transversal views of the Posidonia fibers studied. These figures show that this technical fiber have an oval shape. Their structure is similar to a natural composite composed of ultimate fiber bundles of cellulose, thus forming the fibrous reinforcement, linked together by gummy and waxy substances, constituting the matrix. We can notice also that the ultimate fibers present a void as shown in figures 4 (b) and 5 (b), which means that the fibers are porous. This porosity could explain the use of posidonia fibers in thermal isolation and soundproofing. [16-17].

As shown in Figure 2(b), the untreated fibers present on their surface woody and gummy substance. After the combined chemical treatment, SEM micrographics show (Figure 3(b)) an

improvement in surface morphology. Then using soda treatment cleans the fiber surface of a large amount of impurities (gummy and waxy substances) and causes fibrillation.

The chemical treatment using sodium hydroxide and hydrogen peroxide allows the separation of fibers. In fact, the important modification done by alkaline and peroxide treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites.

3.2. Effect of the treatment processes on the physical properties of Posidonia fibers

To better visualize the effect of extraction conditions on physical properties of treated fibers, main effect plots were drawn.

As shown in Figure 6, the fibers linear density decreases when increasing treatment conditions (concentration of soda, temperature, duration of treatment). In fact, the untreated fibers present a linear density of 9.31 Tex. However the linear density of treated fibers ranges from 8.81 to 3.69 Tex. This reduce of mass per unit of length could be attributed to the removal of waxy and gummy materials present between the ultimate fibers. The lower linear density was obtained in the combination (120 ℃, 90 min and 0.4 N) which confirms result obtained of diameter. In addition to that, the lower yield obtained in this case (38.22 %) proved this fine structure. As shown in yield main effect plot (Figure 7), higher yields were obtained when proceeding in the lowest conditions of treatment (temperature= 60 ℃ and duration = 30 mn). Then, in these lower conditions this chemical treatment was not effective to remove gummy and waxy materials from technical Posidonia fibres. On the other hand, the removal of foreign substances is improved while increasing temperature and duration of treatment.

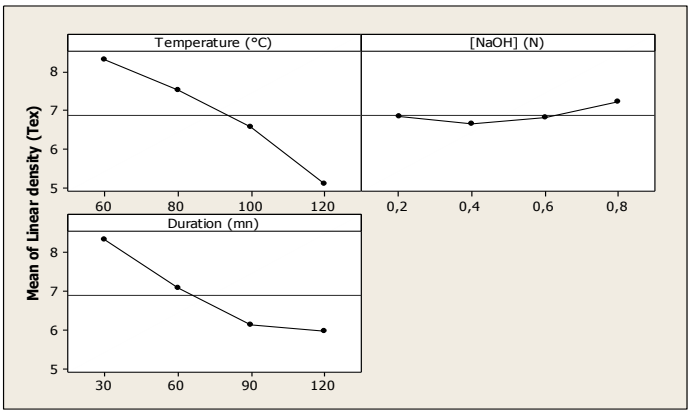


Figure 6: Main effect plot of linear density

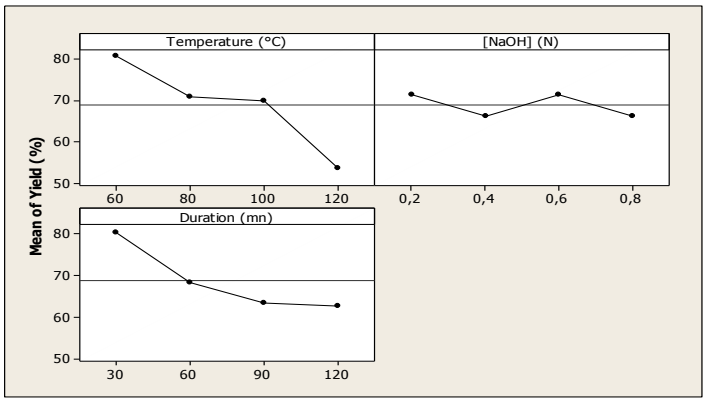


Figure 7: Main effect plot of yield

Concerning the ratio L/D, we can notice, as shown in Figure 8, the influence of treatment conditions changes from condition to another. In fact, this property is strongly influenced within temperature and weakly affected with sodium hydroxide concentration. Thereafter, it drops for 140 up to 110 within duration of treatment and it becomes constant between 90 mn and 120 mn. As given, the raw biomass has a lignocellulosic fibrous structure. Indeed, like all lignocellulosic-based fibres, the posidonia ones are formed by several holocellulosic microfibres, which are linked together via lignin (Mohamed Chaker et al., 2009). Then, this reduce of ratio L/D could be attributed to the decrease of length which is linked to the action of combined treatment (sodium hydroxide and hydrogen peroxide) while removing lignin [18]. In fact, this chemical treatment has removed lignin and separate posidonia microfibres which led to the reduction in length of these fibers. The stability of ratio L/D fibers (between 90-120 min) is due to the reactivity of the oxygen molecules, present into the treatment bath, in the early stages of the process when there is a lot of lignin. While the concentration of the latter decreases, there is no reactivity and therefore no degradation in this property.

In conclusion we can say that the most influential parameter on the physical properties of these fibers is first the temperature and secondly the duration of treatment.

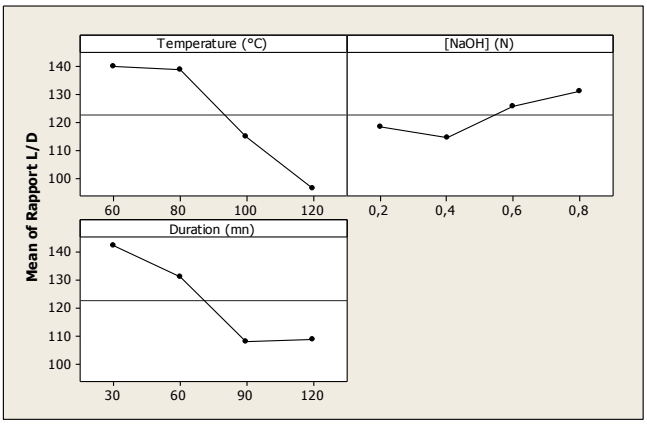


Figure 8: Main effect plot of ratio L/D

3.3. Effect of the treatment processes on the mechanical properties of Posidonia fibers

Chemically treated fibers can show a considerable decrease in tensile properties [19]. The extension at break of these fibers does not change much [20].

In our case, the combined chemical treatment has strongly influenced posidonia tensile properties. In fact, as shown in strength main effect plot (Figure 9), the fibers toughness is strongly influenced within temperature and treatment duration. However it is weakly influenced towards soda concentration. The tenacity of untreated fibres is higher than those treated (untreated posidonia = 11.19 cN/Tex; treated posidonia = 10.72- 5.31 cN/Tex). Then there is a decrease in fibres tenacity after combined chemical treatment (hydrogen peroxide and sodium hydroxide). This decrease attributed to the substantial delignification and degradation of cellulosic chains during chemical treatment. Moreover, this phenomenon of strength decrease becomes faster while increasing temperature and duration of treatment. Therefore, temperature and duration of treatment could be considered as catalyst of combined chemical treatment reaction. In fact, the lower tenacity obtained at higher temperature and duration is attributed to the damage induced in the cell walls and the excessive extraction of lignin and hemicellulose, which play a cementing role in the structure of the fibers. This result is confirmed by the lower yield obtained in these treatment conditions which explain the large amount of noncellulosic materials (lignin and hemicellulose) removed from raw and technical posidonia fibres. Then, combined chemical treatments have been found to decrease the fiber strength due to breakage of the bond structure, and disintegration of the noncellulosic materials [20]. As consequence, in order to obtain good mechanical properties we must operate to moderate proceeding conditions which should not exceed 100 ℃ for temperature and 60 minutes for treatment duration.

Duration of treatment has not a great influence on the elongation of posidonia fibers. However, a large increase in the concentration of sodium hydroxide (when concentration goes over 0.6 N) reduces the fibres elongation. Also, an increase of temperature leads to decrease of elongation (Figure 10).

Elongation of these fibres for different treatment conditions does not exceed 8.4 %, which confirms the property of natural fibres having generally low elongation.

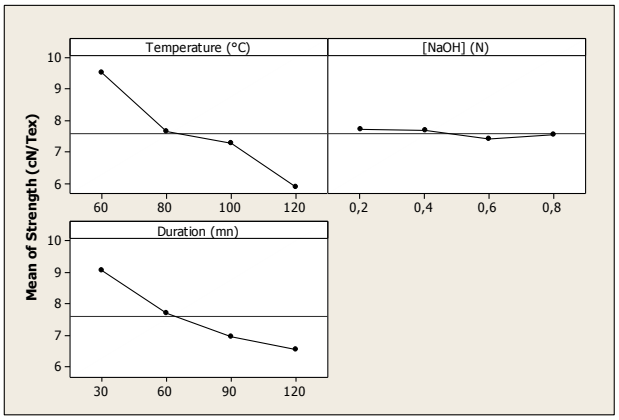


Figure 9: Main effect plot of Strength

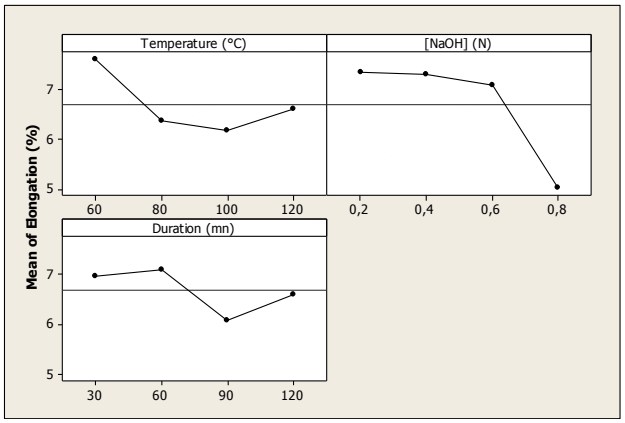


Figure 10: Main effect plot of elongation

3.4. Degree of control factors influence on the physical and mechanical properties

In order to conclude on the importance of extraction conditions, a statistical analysis of the effect of temperature, soda concentration and duration of the treatment on the various properties was developed.

The p-value used in hypothesis tests to help you decide whether to reject or fail to reject a null hypothesis. The p-value is the probability of obtaining a test statistic that is at least as extreme as the actual calculated value, if the null hypothesis is true. A commonly used cut-off value for the p-value is 0.05. For example, if the calculated p-value of a test statistic is less than

0.05, you reject the null hypothesis. This null hypothesis in our case is the the factor has not a significant influence on the fibres' property. [21-22]

Results of p-values meaning are shown in Table 2.

Table 2: p-values meaning

Dependent variables	Linear density (Tex)	Ratio (L/D)	Yield (%)	Strength (cN/Tex)	Elongation (%)
[NaOH] (N)	*	*	*	*	**
Temperature (°C)	**	**	**	**	**
Duration (mn)	**	**	**	**	*

*: insignificant influence ($p > 0.05$); **: significant influence ($p < 0.05$).

From this table, the most influent parameter on the measured properties was temperature and duration which affects mostly the majority of its (linear density, ratio (L/D), strength and yield).

3.5. Optimisation of treatment conditions

In order to optimise the treatment conditions we have used the desirability functions shown in figures 11 and 12 in wich we took into account the target "Y_{target}", the importance of every property "Y_i" in the definition of global desirability. [23]

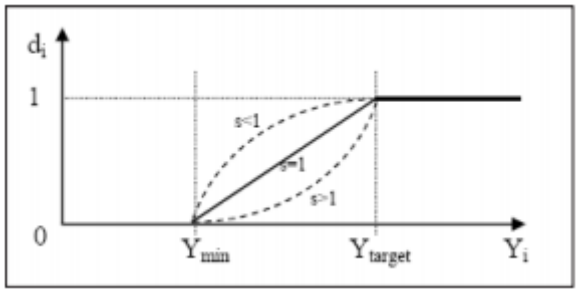


Figure 11: Desirability function to maximize.

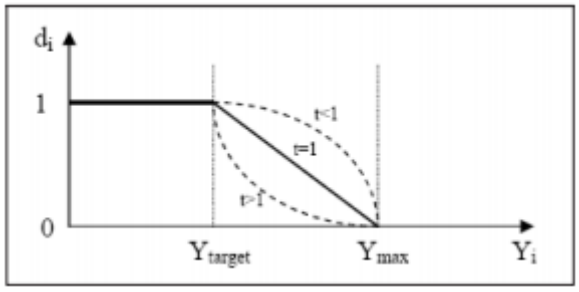


Figure 11: Desirability function to minimize.

In this study, we used two types of desirability functions "d_i": desirability function to maximize and to minimize. Thus, to maximize a property "Y_i", such as the yield, strength and elongation, the desirability function (shown in Figure 11) had to be used, where d_i was calculated as follows:

$$d_i = 0 \text{ if } Y_i \leq Y_{min}$$

$$d_i = \left[\frac{Yi - Ymin}{Ytarget - Ymin}\right]^S \text{ if } Y_{min} \leq Y_i \leq Y_{target}$$

$$d_i = 1 \text{ if } Y_i \geq Y_{target}$$

To minimize a property "Y_i", such as linear density, the desirability function (shown in Figure 12) had to be used, where d_i was calculated as follows:

$$d_i = 1 \text{ if } Y_i \leq Y_{target}$$

$$d_i = \left[\frac{Yi - Ymax}{Ytarget - Ymax}\right]^t \text{ if } Y_{target} \leq Y_i \leq Y_{max}$$

$$d_i = 0 \text{ if } Y_i \geq Y_{max}$$

For each property affecting the global desirability, we calculated the satisfaction degree "d_i" and we attributed a relative weight to indicate the property's importance. We grouped these different satisfaction degrees by using the Derringer and Suich desirability function [10] defined as follows:

$$d_g = \sqrt[w]{{d_1}^{w_1} \times {d_2}^{w_2} \times \ldots \times {d_n}^{w_n}}$$

Where d_i is the individual property's desirability function Y_i, $i \in [1 \ldots n]$, w_i is the weight of the property Yi in the "d_g" desirability function, w is the sum of w_i and n is the number of properties.

The compromise between the properties (minimize fiber linear density, maximize yield, strength and elongation) was better when "d_g" increased; it became "perfect" when "d_g" was equal to 1. When the satisfaction degree "d_i" of the property Y_i was equal to 0, the response had a value outside of tolerance the function "d_g" was equal to 0 and so the compromise was rejected.

To define the desirability function, we had to fix the objective of every property. These different objectives are reported in Table 3.

Table 3: The optimum levels of properties

Dependent variables	Objective	Min	Max
Linear density (Tex)	Minimise	-	7,5
Ratio (L/D)	Target	75	165
Yield (%)	Maximise	60	-
Strength (cN/Tex)	Maximise	5	-
Elongation (%)	Maximise	3	-

The results of desirability for each property and the optimum values for the independent variables are presented in Tables 4 and 5, respectively.

Table 4: desirability values for the dependent variables

Dependent variables	Value	Desirability (d_i) %	Weight
Linear density (Tex)	7,2	79	1
Ratio (L/D)	124,92	88	1
Yield (%)	70,7	100	1
Strength (cN/Tex)	8,24	100	1
Elongation (%)	7,2	100	1
Global desirability (d_g)	93,15		

di denotes desirability of dependent variables (yield, linear density, ratio (L/D), strength and elongation).

Table 5: Optimum values for the independent variables

Value	Normalized value	Real value
Temperature (℃)	3	100
Soda concentration (N)	2,5	0,5
Duration (mn)	1,5	45

The statistical study determined the optimum treatment conditions which are: 100 ℃ as temperature, 0.5 N as soda concentration and during 45 minutes.

3.6. Characterization of fibers treated with optimum conditions

The physical and mechanical properties of treated posidonia fiber in the optimum conditions ($F_{P\ optimum}$) and those untreated are presented in the table 6. According to this table, we can notice that the properties of $F_{P\ optimum}$ are similar as those predicted in table 4. Therefore the optimum conditions are valid.

In comparison with untreated fibers, the $F_{P\ optimum}$ present a cristallinity index less important. This is affirmed by the less important strength of treated compared to untreated posidonia fibers.

Table 6: Properties of the optimum

Properties	Linear density (Tex)	Ratio (L/D)	Strength (cN/Tex)	Elongation (%)	Cristallinity index (%)
Untreated fiber	9,31	176,51	11,19	11,9	31,19
$F_{P\ optimum}$	7,24	125,01	8,2	7,4	23,59

3.7. Effect of the treatment processes on the chemical structure of Posidonia fibers

FT-IR spectroscopy has been extensively used to visualize the chemical modifications of that occur during various chemical treatments. T-IR spectra of raw and treated fibers determined at 500-4000 cm^{-1} wave number are shown in Figure 11.

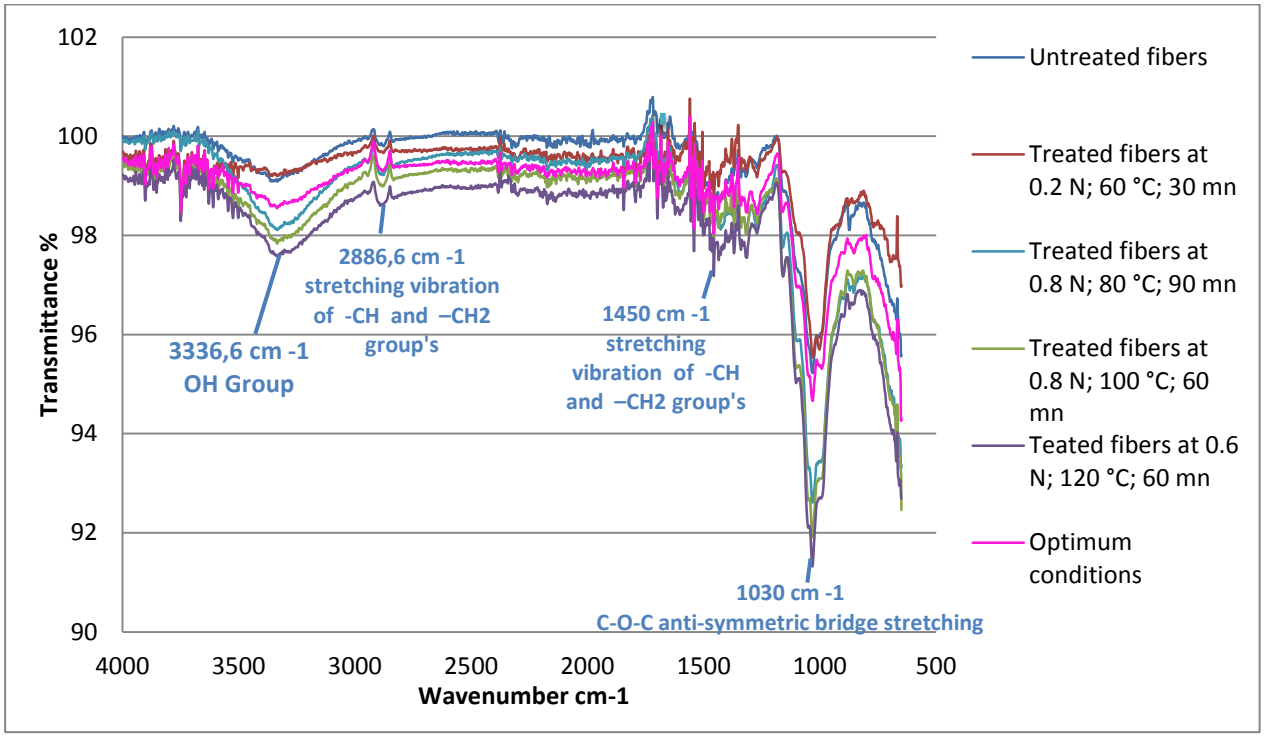


Figure 11: FT-IR spectra of untreated and treated posidonia fibers

Similar absorption bands in the spectra are generally found in the fibers having the same chemical composition. The figure shows that the intensity of transmittance of the treated fibers is less than those untreated. As well as, transmittance intensity decrease while increasing temperature, concentration and duration of treatment. This is could be explained by the fact that the structure become less opaque after chemical treatment. This transparence

could be attributed to the elimination of certain amount of lignin, hemicelluloses and other fatty and gammy substances. Moreover, the fibers absorbance is improved while increasing processing conditions. In fact, while proceeding in higher conditions of treatment (Temperature ≥ 100 °C, Duration ≥ 60 mn) there is a large amount of noncellulosic materials removed confirmed by the lower yield (≤ 70 %) obtained in these conditions.

The transmittance peaks of interest in this study are identified and shown in Figure 11. The occurrence of majority peaks did not change. The figure shows a broad band observed at 3000-3500 cm^{-1} in the spectra indicating the presence of OH group. The second band was observed at 2857-2926 cm^{-1} indicating the stretching vibration of the groups -CH and –CH2 of cellulose and another band at 1450 cm^{-1}, which also indicates the presence of -CH produced by a symmetrical deformation of lignin and alpha cellulose. Furthermore, the ratio of the intensities of the transmittance peaks at 3343 cm^{-1} (–OH) and 2900 cm^{-1} (>CH2, >CH-) for the raw (0.9937) (0.9985) and treated (0.987) (0,993) fibers indicated the presence of more –OH groups in the treated fiber than in the virgin sample. This was more likely due to the generation of new –OH groups on cellulose during alkaline treatment via the cleavage of phenolic ether links existing between cellulose and lignin moieties. In addition to that, the phenomenon of new –OH groups' generation is enhanced while increasing temperature, soda concentration and duration of treatment. This is expressed by the transmittance intensities decrease while increasing input parameters (temperature, duration and soda concentration). The sharp peak at 1031-1033cm^{-1} has been attributed to C-O-C anti-symmetric bridge stretching in cellulose and hemicelluloses [24; 7].

The spectrum of fibers treated under optimal conditions is in the middle of other spectra of fibers treated within other conditions of treatment. This spectrum shows that the optimal conditions of currying ensure the appearance of new OH and CH groups which confirms the effectiveness of treatment in these conditions.

The analysis of the IR spectra of the untreated posidonia fiber showed characteristic features of lignin and hemicellulose components, which indicated that the fiber was lignocellulosic in nature. The IR analyses clarified the elimination of a large amount of hemicellulose and lignin by combined chemical treatment.

4. CONCLUSION

Posidonia fiber is a natural vegetable fiber that is derived from the leaves of Tunisian P. Oceanica variety and harvested on the coasts of Tunisia. The study of the fibers treatment conditions seems to have an important role on the fibers properties. Posidonia fiber shows a linear density between 3.69 and 9.31 tex with a ratio length per diameter (L/D) between 74 – 165. Tenacity of fibers was extended between 5.31 and 11.19 cN/tex it seems to be suitable (>= means of tenacities which equal to 7.5 cN/tex) while proceeding in conditions of temperature ≤ 100°C and duration of treatment ≤ 60 minutes. The optimal properties of treated fibers are obtained when proceeding at 100 °C, using 0.5 N as soda concentration and during 45 minutes.

The FTIR spectra reveal the lignocellulosic structure of these fibers and their modification after chemical treatment. This change in structure is due to the increase of the cellulose amount exposed on the fiber surface, which increases the number of possible reaction sites (OH and CH groups) when reinforced composites.

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