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Study of Extraction and Characterization of Ultimate Date Palm Fibers

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Abstract: Natural fibers are generally hydrophilic in nature, as they are in fact derived from lignocellulose, which contains strongly polarized hydroxyl groups. During the last few decades, cellulose has been one of the most abundant, inexpensive, non-toxic, and renewable biomacromolecules in nature and has been widely applied in diverse fields. In this study, we choose to optimize the extraction process of date palm in order to obtain ultimate fibers with minimum rate lignin, minimum degradation, a high yield and degree of whiteness. To characterize these fibers infrared spectroscopy (IR), XRD, EDX, SEM and XPS were used.

Keywords: Date Palm Fiber, Hydroxide of Sodium, Hydrogen Peroxide, EDX, XPS

1. Introduction

Numerous recent researches study the idea of exploiting natural fibers and particularly lignocellulosic fibers. Ecological concern has resulted in interest in natural fibers. It has resulted in a renewed interest in cellulosic fibers. In fact, many natural fibers, such as alfa, sisal, luffa, and date palm, are of interest because they are environmentally friendly and are not hazardous for the environment. In addition to their multiple uses, natural fibers have shown many efficient properties such as lightness, resistance or flexibility which give them a wide perspective of application in textile field. In fact they are recyclable and nature-friendly and nowadays they are exploited in automobile and medical applications. Hence, in the last few years, lignocellulosic fibers in general have been widely applied in many fields such as textile [1-3] technical [4-9] or medical [10], [11] applications, because of their renewability, biocompatibility, and natural abundance.

Natural fibers are used for wide range of applications in particular in the field of composite materials, nonwoven and in the paper industry.

Many researches [12-15] illustrated the application of agave fibers. Ben marzoug and Saieb [16], [17] presented the utility of technical esparto fibers. Valcineide and Tanobe Thais [18] have studied luffa fibers. Thompson [19] has investigated henequen fibers, especially their absorbent properties. According to their extraction methods, natural fibers have very different qualities. Indeed, one can obtain discontinuous and short fibers or continuous and long fiber.

In this survey, date palm fibers are obtained from a chemical extraction. To characterize these fibers, some tests were made like crystallinity, morphological structure, chemical composition and thermal stability. In this perspective, we are interested in date palm fiber which confirmed its application in filtration domain [20] and composite materials, they can provide an absorbent potential to water or colorants [21]. In this study, we optimized the extraction process of these fibers to obtain ultimate cellulosic fibers. We used a mixed process which combines NaOH extraction process and H_2O_2 bleaching process [22], [23].

Our goal is to get the optimum experimental conditions to find the highest yield of cellulosic fibers extracted with the best whiteness, low lignin and less degradation. We do not forget the requirement to use an industrial process, economic and ecological.

In this study, we attempted to characterize fiber obtained from a chemical extraction using sodium hydroxide and hydrogen peroxide. The characterization was based on the study of the crystallinity, the morphological structure, the

chemical composition and the thermal stability.

2. Experimental

2.1. Materials

2.1.1. Fibers

The date palm used in the current work belongs to the species Dactylifera. The date palm fiber used in this study is composed of (32%-35%) of cellulose, 25% of hemicelluloses and 27% of lignin [24].

2.1.2. Extraction Optimization

The date palm fibers were extracted using the same method used for esparto fibers. In a preheated solution at 90 °C containing sodium hydroxide, hydrogen peroxide, , 3 g/L of a wetting agent (Subitol LSN BEZEMA), and 25 mL/L of stabilizer of hydrogen peroxide (contavan GAL), date palm sheet was treated for 90 min.

To optimize extraction, we choose the optimum NaOH and H2O2 concentrations. For extraction we started by setting the appropriate amount of sodium hydroxide for the regulation of pH. Then we choose the optimum concentration of NaOH that allow achieving the maximum Yield. Finally we optimize the concentration of hydrogen peroxide that ensuring the higher yield of extraction, the minimum rate of lignin and the maximum whiteness degree.

2.2. Methods

2.2.1. Data Color (Whiteness)

The whiteness is determined using Data Color and with rule given by Harisson, as indicated in Cegarra et al.'s (1976) work [25]. Indeed, fibers were exposed to the spectrocalorimeter light issued from Data Color. We stopped taking measurement once the tolerance value was equal to 0.035%.

2.2.2. Lignin Rate

Lignin rate is measured using destruction with sulfuric acid in two steps in two concentrations equal to 72 % then 3 % as reported by K. subraiman [2].

2.2.3. Infrared Spectroscopy

In this study, a Perkin Elmer 398 IR-Transmission Spectrometer ranging from 400 to 4000 cm-1 was used. Five milligrams of fibers was mixed with 200 mg of analytical-grade KBr. The fibers were then ground and pressed into KBr pellets.

2.2.4. X-ray Diffraction (XRD)

X-ray diffractograms are obtained with an analytical X Pert PRO MPD diffractometer, having an X-ray tube producing monochromatic Cu Kα (λ= 1,789 A°) radiation.

2.2.5. Scanning Electron Microscopy (SEM)

SEM analysis of date palm fiber was performed using XL30 ESEM model from SEI.

2.2.6. SEM-EDX

Qualitative SEM-EDX spectra were obtained on a Quanta 200 environmental scanning electron microscope from FEI, in the low vacuum mode (at a pressure of 1.0 Torr i.e. 133 Pa).

2.2.7. X-ray Photoelectron Scanning (XPS)

The X-ray photoelectron spectroscopy (XPS) experiments were performed with a XR3E2 apparatus from Vacuum Generators, UK.

3. Results and Discussion

3.1. Extraction Optimization

For tests, as seen in Table1 and Table2, the pH and the yield R(%) change depending on the concentration of NaOH.

The yield was determined by the percent decrease in weight as follows:

$$R(\%) = \frac{(Wi - Wf)x100}{Wf} \quad (1)$$

Where R is the yield of extraction date palm fiber process (%), Wi (g) and Wf (g) represent the weights of the initial leaves and the final fibers respectively.

Table 1. *The effect of NaOH concentration on the pH value.*

NaOH(g)	0	1	2	4	7	15
pH	2	7.7	8.6	9	9.4	9.4

Table 2. *The effect of NaOH concentration on the yield.*

Essais	T (°C)	D (min)	NaOH(g)	H_2O_2 (ml)	R(%)
1	120	90	7	3	78
2	120	90	15	3	73

Different parameter values are summarized in Table3. According to this result, tests have contributed to optimization process extraction by setting the optimum concentrations for the high yield and whiteness degree, the minimum lignin rate and minimal oxidation.

Table 3. *Effect of H_2O_2 concentration on the whiteness, yield and lignin rate.*

Essai	T (°C)	D (min)	NaOH (g)	H_2O_2 (ml)	Whiteness(%)	Yield(%)	Lignin rate(%)
1	120	90	7	5	54	71	11
2	120	90	7	7	57	63	7
3	120	90	7	9	57	63	7
4	120	90	7	11	87	57	4
5	120	90	7	13	87	54	2
6	120	90	7	15	92	41	-

Figure 1 shows the influence of varying the amount of sodium peroxide on the lignin, the extraction yield and the

degree of white. Note that the yield and lignin decreased with increasing the amount of hydrogen peroxide in the degree of white increases. We chose an amount of H_2O_2 equal to 11mL because it allows minimum lignin-free cellulose degradation (formation of oxycellulose). Indeed, Test No. 5 (H_2O_2 = 13mL) gives a rate equal to 2% lignin but with this amount the whiteness remains constant and after a test oxycellulose we note that there is a huge formation of oxycellulose

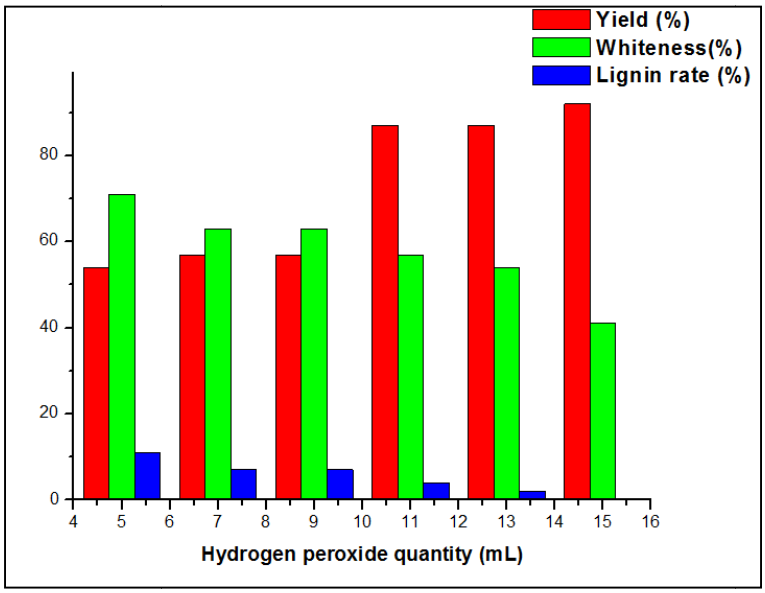


Figure 1. *Result of extraction optimization.*

During the extraction treatment, we have used a combined alkali and hydrogen peroxide process.

In fact, the whitening power of the extracted fibers is obtained by activating the hydrogen peroxide. Hydrogen peroxide is regarded as a very weak acid whose dissociation reaction product hydroperoxide ion (HO^{2-}) which is the bleaching ion according to the following reaction:

But this dissociation also produces H^+ cation which makes the most acid bath as possible, it is therefore necessary to drive bleaching with an alkalinity reserve otherwise the H^+ unite with HO^{2-} anions. Under alkaline conditions, the reaction rate increases with temperature. The bleaching ion (perhydroxyl ion) reacts with lignin and hemicelluloses and converts these compounds by oxidation onto soluble compounds.

For the success of cellulose extraction, one must choose auxiliary products to ensure maximum wetting and stabilization of the maximum use of hydrogen peroxide.

Thus, all bleaching techniques to hydrogen peroxide include a total impregnation of the material to bleach in an aqueous solution whose constituents are the following:

- Hydrogen peroxide is a bleaching agent
- The water must be clean, free of any metal and organic impurities.
- An activator which aims to transform the hydrogen peroxide perhydroxyl ions. In made, sodium hydroxide is the most common activator but alkali salts such as sodium carbonate, trisodium phosphate are used.
- A wetting agent that its primary role is to promote the penetration of the bleaching bath in the matter.
- A stabilizer agent for protecting the hydrogen peroxide the catalytic decomposition by capturing the metal ions present.

Indeed, a catalytic decomposition under the action of certain metals, including iron and copper influence on the quality of treatment, or a loss of bleach, or by a degradation of the material (causing degradation cellulose and therefore a loss of resistance and premature wear of the article).

In general, bleaching of cellulose fibers with hydrogen peroxide is optimum at a pH of 10.5 to 11, at temperatures between 80 ° C and 120 ° C and for duration between 45 minutes until 5 to 6 hours.

Since hydrogen peroxide is not stable in basic medium and oxide cellulose when in contact with oxygen in air, it is necessary to select the best conditions of cellulose extraction process.

The right choice of method of extracting cellulose from date palm depends primarily on the origin, nature and chemical composition of the leaves used. Indeed, the choice of operating conditions of the extraction method is critical to ensure the reliability of the subsequent results (grafting of itaconic acid). Certainly, extraction must meet three basic criteria: quantity, quality and not alterative. Thus, we managed to develop a combined process (NaOH = 35 g / L H2O2 = 55 mL / L) of the cellulose extraction from date palm and we achieved a yield of 57%, a lignin content of 4% and a whiteness of 87% without too much cellulose oxidation.

We also note that the material is very heterogeneous and we can by no means get the same physicochemical characteristics fibers. We have not been able to characterize the fiber obtained in a satisfactory manner, but it is certain that this fiber has good absorption seen its high cellulose

3.2. XRD Results

Surface state of fibre is a fundamental characteristic which determines liquid contact; however it can't define the absorption capacity. The crystallinity studies the microstructure of the fiber which is intimately linked to the absorption capacity property.

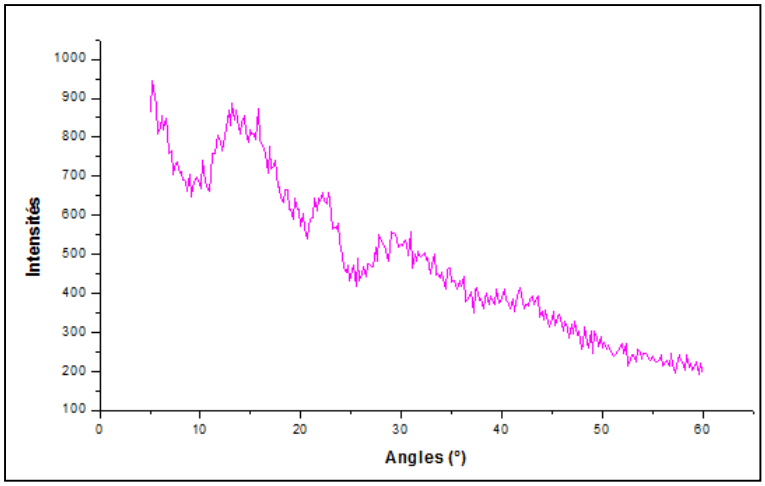


Figure 2. *XRD diffractogram of date palm ultimate fiber.*

The X-ray patterns of ultimate date palm fiber are shown in Figure 2. The diffraction peaks from linocellulosic fibers at 2θ = 14.67°, 16.39° and 22.53° for (110), $(\overline{1}10)$ and (200) planes are characteristic for cellulose I crystal as also reported in literature [26].

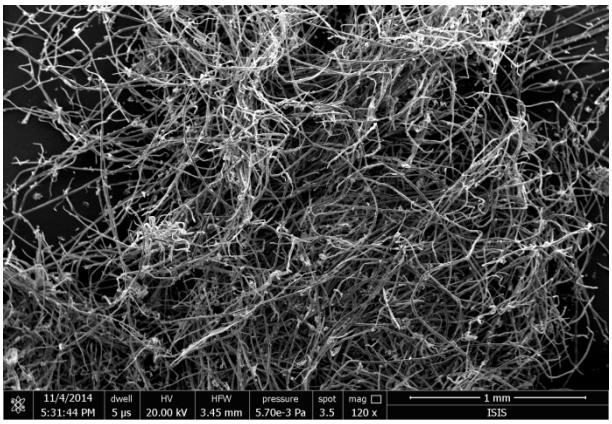
Lignin and hemicelluloses are more amorphous than cellulose but amorphous zones offer more free volume which can store more water.

3.3. SEM Results

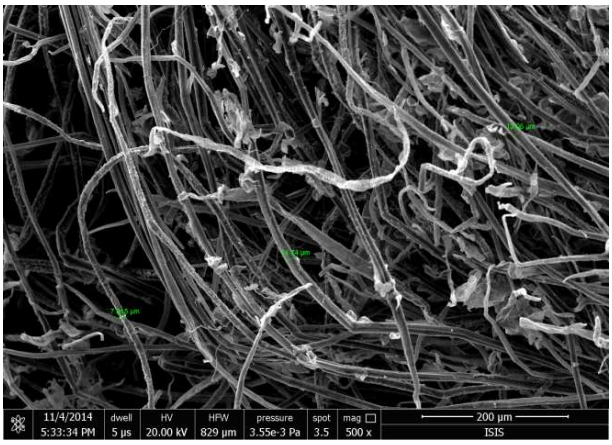
Studies of morphologic characteristics can ensure a comparison between different extraction methods give more information about surface and evaluate a fiber diameter.

Figure 3 illustrates a treated sample with 55 ml/L of hydrogen peroxide and 35 g/L of sodium hydroxide and demonstrates that fibers contain some quantities of lignin.

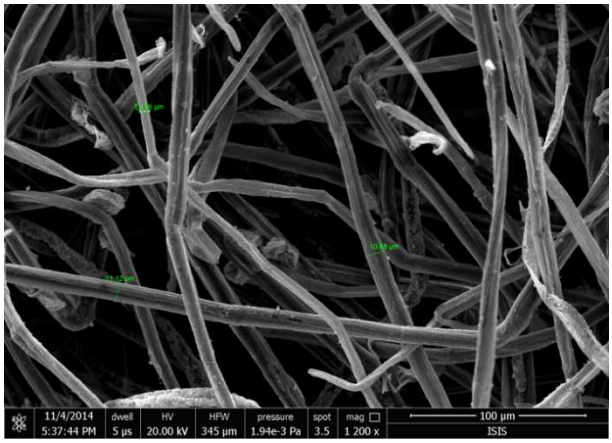
Figure 3(a–c) are SEM images of ultimate date palm fibers under various magnifications. Figure 3a clearly shows the shape and size distribution of the ultimate date palm fibers. These well-separated fibers have lengths of 1– 3 mm and diameters of 5–15 µm. The SEM images (Figure3 c) of surface fiber at larger magnification show that the surface of ultimate date palm fiber is almost free of defects.



(a)



(b)



(c)

Figure 3. *SEM images showing untreated date palm fibers. Different magnifications: a) x120, b) x500, c) x1200.*

The wide spectra of the surface of all the samples were recorded. As expected, for the ultimate date palm fiber, the spectra showed the presence of two main atoms, as depicted in Figure4. The corresponding peaks were situated at 285 and 531 eV, and attributed to carbon and oxygen atoms, respectively.

The chemical composition of date palm fiber surface was evaluated by X-ray photoelectron spectroscopy Figure 5. As previously shown, the XPS spectrum of cellulose, with deconvolution in the spectral region of carbon 1s line, contained 4 individual components with binding energies (Ebound) corresponding to the carbon in groups C-H (Ebound = 285.0 eV), C-OH and C-O-C (Ebound = 286.6 eV), O-C-O and C = O (Ebound = 287.7 eV) and carboxyl (Ebound = 289.1 eV). According to the spectra, the surface of cellulose contains mostly hydroxyl, ether and carbonyl groups, also attended by a small number of carboxyl groups.

3.4. XPS Results

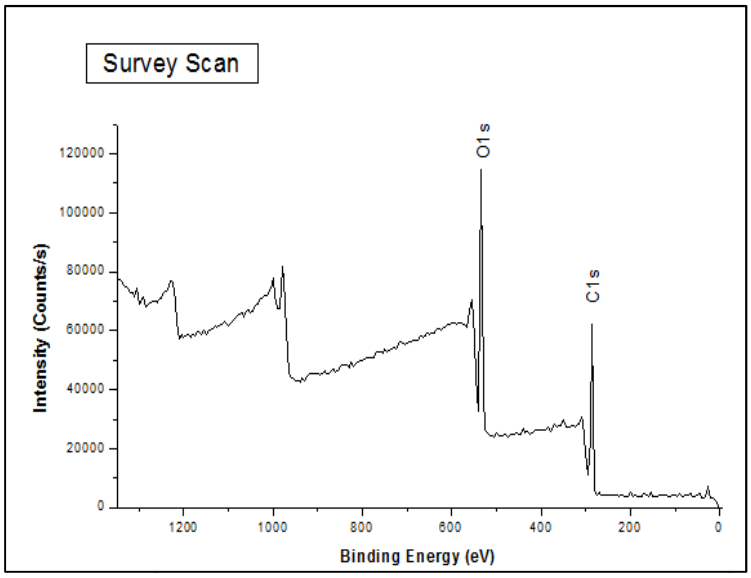


Figure 4. *XPS result: Survey scan.*

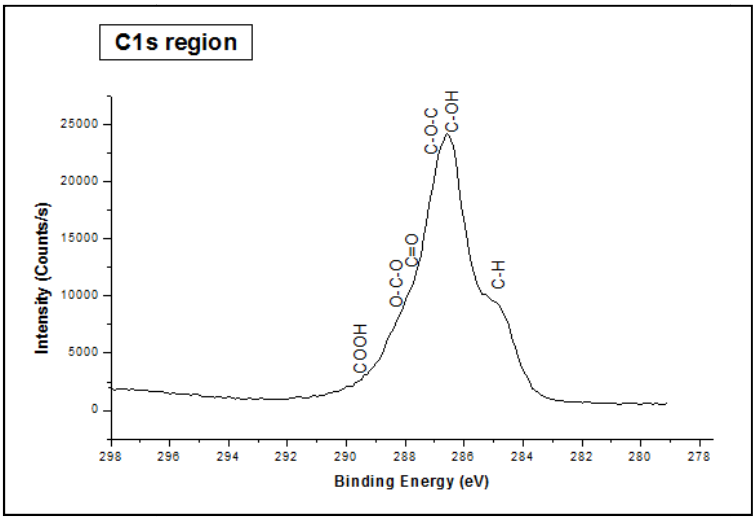


Figure 5. *XPS Results: C1s region.*

3.5. EDX Results

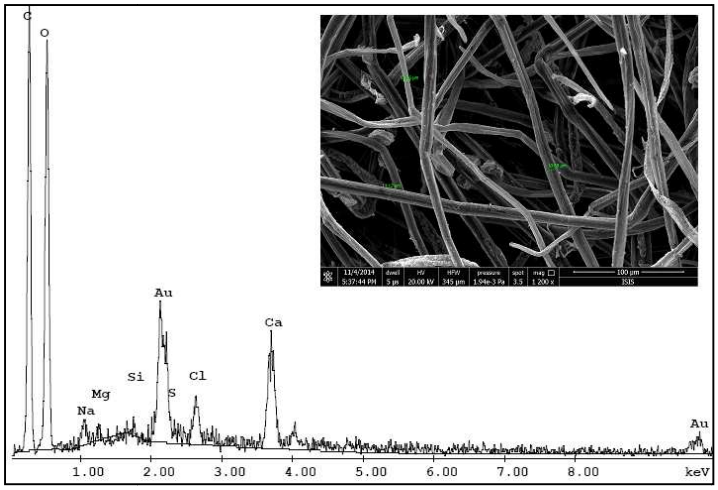


Figure 6. *EDX spectra of date palm fiber.*

The EDX spectrum of the date palm fiber is shown in Figure6. It appears that: silicon is detected in significant amount in the sample which was not submitted to any treatment. This result can be explained by the extraction process.

Table4 reports the surface atomic composition of the samples studied, as deduced from the EDX measurements

Table 4. *The surface atomic composition of the date palm fiber.*

Element	Wt(%)	At(%)
C	38.20	57.01
O	33.01	36.98
Na	0.89	0.69
Mg	0.31	0.23
Si	0.15	0.10
S	0.38	0.21
Cl	1.57	0.79
Ca	4.68	2.09
Au	20.83	1.90

3.6. Infrared Spectroscopy Results

The IR spectrum of the ultimate date palm fiber is illustrated in Figure 7.

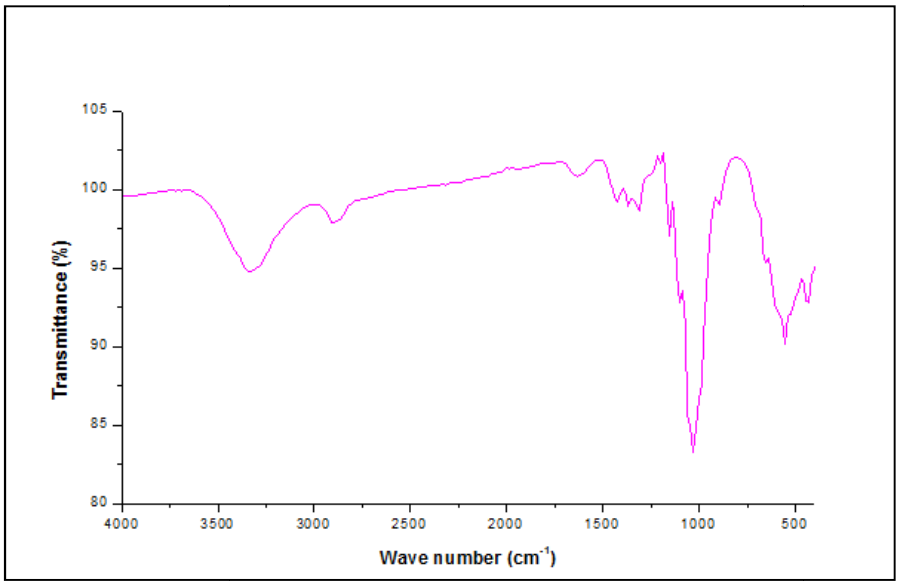


Figure 7. *Infrared spectrum of date palm fiber.*

The spectrum of the cellulose of date palm fibers shows the same basic structure as all cellulosic samples. These spectrograms show the presence of relative bands at 612, 1070, 1431, 1320, 1637, 2915, and 3400 cm^{-1}.

They were identified in other lingocellulosic fibers, as reported by Subramanian et al [2]. As noted by Bessadok [13] , the ν C–O–C symmetric glycosidic stretch or a ring stretching mode at 1100 cm^{-1} and the C–OH stretching vibration of the cellulose backbone at 1050 cm^{-1} (ν C–O secondary alcohol) arise from the polysaccharide components

(that is, largely cellulose). The 1360 cm^{-1} band is attributed to the CH2 bending of cellulose. Peaks due to alcohol group of cellulose (δ OH deformation) are characterized by the peaks located at 1370 and 1335 cm^{-1} while the peak at 1316 cm^{-1} corresponds to the CH2 wagging of cellulose. Peak at 900 cm^{-1} assigned to the out-of-phase ring stretching is a characteristic of the cellulose backbone. The region 3500–2500 cm^{-1} is mainly related to OH and CH2 groups. The peaks located in an approximate way at 3000 and 2850 cm^{-1} are attributed to ν (CH) and ν (CH2) groups, respectively [27]. These results show the absence of peaks relative to aromatic vibration of lignin and also peaks corresponding to the presence of hemicelluloses. Results also present peaks at 1637 cm^{-1} that show the presence of pectin.

4. Conclusion

In other studies, many characteristics of ultimate date palm fibers extracted with combined method are investigated. This method is confirmed to give a total separation at adequate quantities of sodium hydroxide and hydrogen peroxide. The obtained fibers are so fine and short. With the aim of exploiting and studying date palm fibers potential, we tried in this study to focus on fiber process extraction in order to obtain ultimate fibers. For this purpose, we choose to work with a mixed extraction process that combines a NaOH extraction process and an oxygen peroxide bleaching process. We started by optimizing the extraction process, conducting in a first step a preliminary study that has been interesting with respect to NaOH and H_2O_2 concentration variation. In a second step we investigated control test (whiteness, lignin rate, yield, oxycellulose formation, crystallinity, etc.).

Once extraction was achieved, characterization of date palm fibers is done with effective techniques like EDX, XPS, IR, SEM and DRX. These techniques proved the extraction of date palm fiber successfully.

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