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**Alkaline Treatment Effect on Mechanical Properties of Date palm Wood Fiber**

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**Abstract**

The date Palm tree (*Phoenix dactylifera* L.) is a providential tree for oases inhabitants, growing in wild state around the Mediterranean North Africa and dry areas. In the past it was used as building material because of its mechanical and hydrous qualities. The use of such a fibrous plant in cementitious matrix leads to lightweight materials with very attractive tensile behavior that can be used as advantageous filling materials for structures.

This paper is focused on the optimisation of mechanical properties of this kind of materials by a prior alkaline treatment of the Date palm fiber before its using as reinforcement in a given matrix. The treatment was carried out using sodium hydroxide (NaOH) solution at three different concentration 0.5%, 0.75% et 1% NaOH, we submit after that the untreated and treated specimens to a thermogravimetric analysis (TGA) to measure the influence of soda treatment on the mass loss. Afterward, the samples were tested for mechanical properties determination. Consistent results were obtained for tensile strength and young modulus, which indicates that the NaOH treatment led to a change in the chemical composition of the Palm fiber and succeeded to improve its mechanical properties and therefore proved the effectiveness of the treatment.

**Keywords:** *Phoenix dactylifera* L; Mechanical properties; Palm fiber; Tensile strength; Young modulus

**Introduction**

In the recent years, with the strong emphasis on environmental awareness, scientists and technologists have placed so much importance on the application of natural materials. This move has encouraged industries like furniture, automotive, building construction, and packaging to search for new form of berk composites that can substitute the conventional composite materials.

Unfortunately, some drawbacks such as poor wet ability, incompatibility with some matrix and high moisture absorption by the fibers make them undesirable for certain applications [1, 2, 3]. The main problem often encountered in their use is the fiber –matrix adhesion problem due to the incompatibility between the hydrophilic natural fibers and the hydrophobic matrix. This problem may be improved by a chemically treating fiber surface. Therefore, alkaline treatment is a common method to clean and modify the fiber surface to lower surface tension and enhance interfacial adhesion between the natural fiber and the matrix [4]. That is why several publications have discussed the effects of alkaline treatment on structure and properties of natural fibers, such as kenaf [5], hemp [5], flax [6], jute [7] and sisal [8].

In this context this study was prepared and divided into two major parts:

- In the first part stability and durability of the Date Palm Frond (DPF) Fibers are investigated. It is worth noting that one of the difficulties and disadvantages which impedes the development of natural fiber use in industry and in the manufacture of composites is their poor dimensional stability due to water absorption, that is why several authors do have to study the effects of chemical treatments on the properties of natural fibers to improve their characteristics and whence comes the utility of thermo gravimetric analysis performed on samples types. These analyses were carried out on crude and treated fiber to characterize the degradation of the DPF palm fibers and consequently measure the samples mass variation as a function of time or temperature. The mode chosen to analyze the DPF fibers in this case is the isothermal mode in which measurement is done at constant temperature and the measured parameter is the evolution of the mass. The samples proposed for thermo gravimetric analysis, were extracted from the Date Palm Frond (DPF) (figure 1).

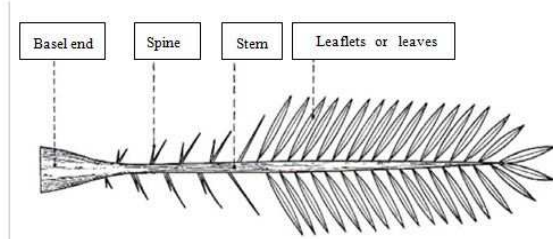


Fig. 1. Schematic representation of a Date Palm Frond

This extraction comes from the Stem, the Cluster and the Basal End of the DPF (figure 2).



Fig. 2. Test Piece of Basal End DPF

And subsequently treated with a sodium hydroxide (NaOH) solution. This alkaline treatment was conducted with concentration of 0.5%, 0.75% and 1% NaOH. The variation of concentration was made to optimize the treatment parameter. The DPF palm fibers were immersed in NaOH solution at various concentrations for an hour and at temperature of  $100^{\circ}\text{C}$ , and after that they were rinsed with distilled water until the rinsed solution reached neutral (PH 7). Then, fibers were dried at room under atmospheric pressure, temperature  $23 \pm 2^{\circ}\text{C}$  and humidity  $50\% \pm 5\%$  for ten days.

- While the Second part discusses the influence of chemical treatment on the mechanical properties of the Date Palm Frond Fibers. Therefore, fore specimens of date palm Frond, untreated and treated with concentrations of sodium hydroxide (NaOH: 0.5 %, 0.75 % and 1% ) were tested for tensile property determination and were examined under scanning electron microscope (MEB) to study the microstructure of the materials.

### Dimensional Stability of the Date Palm Frond (DPF)

#### Alkalization treatment (NaOH treatment)

Alkalization is a common preprocessing technique used on base natural fiber to remove hemicelluloses, fats and waxes that may reduce the interfacial strength between the resin and matrix

when processed into composite form and often results in a change in fiber surface energy in a polar or dispersive manner. Hemicelluloses, which is thought to consist principally of xylan, polyuronide and hexosan, has been shown to be very sensitive to Caustic Soda. The Caustic Soda (Sodium Hydroxide) is said to exert only minimal influence on the lignin in the fibers and the high strength alpha-cellulose. Therefore, It is of great interest to understand the effect this treatment has on the base fiber mechanical properties, Indeed, the major effect was the increasing of the resultant composite strength through increasing fiber matrix adhesion. It is additionally beneficial to investigate current literature to aid in understanding other effects that alkalization may have. These include transformation of cellulose type, and also improved ability for microfibrils to rearrange to accommodate loading of the fiber [9-14].

#### Thermo gravimetric Analyses

In order to characterize the stability and durability of the Date palm Frond (DPF), a (TGA) Thermo gravimetric analysis was performed on crude and treated fibers with concentrations of sodium hydroxide (NaOH:

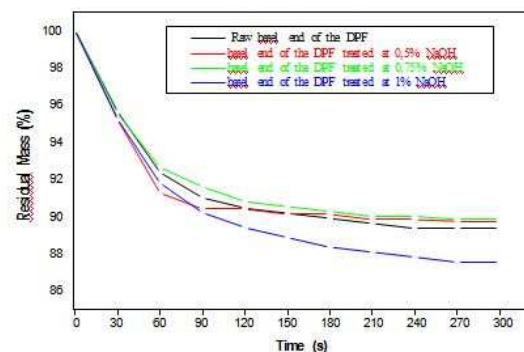
0.5%, 0.75% and 1%). This measure is to characterize the degradation of a material with increasing temperature in which the mass variation of a sample is measured as a function of time. The measured parameter is the evolution of the mass.

#### Objective

The main purpose of this section was to measure the change in DPF remaining residual mass as the concentration of the NaOH treatment changes

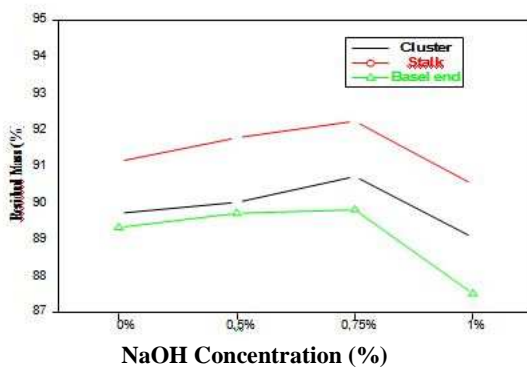
#### Thermal Characterization

Thermo gravimetric analysis (TGA) measurements were performed in air atmosphere using (TA instrument GA 2950) at a heating rate of  $10^{\circ}\text{C}/\text{min}$ . At the end of assessing the effect of treatment on different types of samples, we plotted as an example the curve figure (3) which represents the residual mass versus time for different concentration of sodium hydroxide treatment.



**Fig. 3. Residual Mass variation of Raw and Treated Basel end DPF with Soda solution (0.5%-0.75%-1%) Versus Time**

This figure shows the variations of mass ratios versus time respectively for typical DPF fibers samples of Basel End Palm Wood. In another hand, the figure (4) inform us on how vary the remaining residual mass percentage of the different DPF samples when the amount of NaOH used for the treatment increase. These curves are represented respectively in black, blue, red and green. The dark curve shows the response of an untreated fiber, the blue curve shows the response of a fiber previously treated at a concentration of 1% NaOH, the red one at 0.5% NaOH while the last green one at 0.75% NaOH.



**Fig. 4. Variation of the Residual mass of Cluster, The Stalk and The Basel End DPF Versus Sodium Hydroxide concentration NaOH(%)**

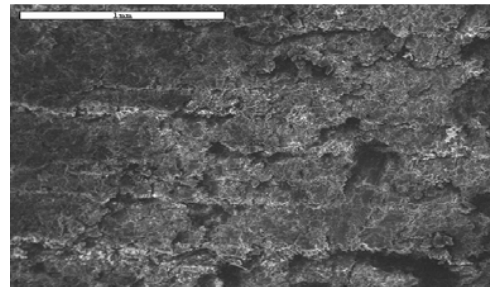
By analyzing the curves of the figures mentioned above, we note the following:

- The mass decreases with time until a stable minimum value corresponding to the maximum heating.
- The variation of sodium hydroxide treatment concentration influences the value of the residual mass. In fact, for the three samples of DPF, Basel End, Stem and Cluster, the residual mass increase from 0% NaOH (untreated DPF sample) to 0.75 % NaOH which corresponds to the minimum mass loss. However it decreases for 1% NaOH.
- After stabilization, the difference between the residual mass of an untreated sample (0%) and treated with 0.75% is approximately: 0.486% for the Basel End DPF, 1.06% for the cluster DPF and 1.125% for the stem DPF.
- Beyond a certain concentration of NaOH, we note that mass loss increased significantly ( blue curve: concentration of 1

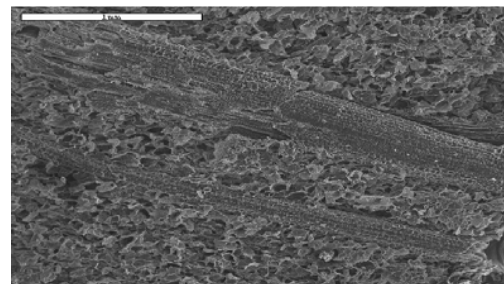
% NaOH ), this is explained by the fact that the internal structure of wood is quite drops till and starts to degrade. Thus the optimal concentration for treatment in alkaline NaOH is of the order of 0.75 %.

### Morphology Analysis

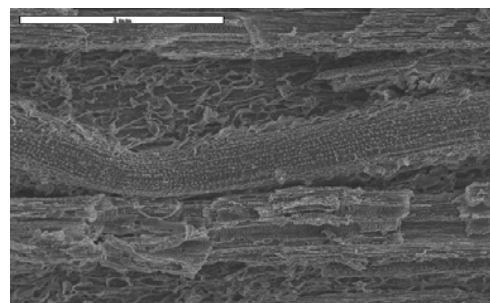
Microscopic examinations were carried out using a HITACHI S3200N scanning electron microscope (SEM) to study fiber morphology. Prior to those analysis, the first step is to take a part of the sample of the wood without altering the structure, the material is immersed in nitrogen liquid for a minute, then a piece of a few centimetres is collected by breaking the structure of the material. This technique helps to avoid the formation of ridges that can interfere with observation so that sample, be well observed in the electron microscope. The SEM micrographs for untreated and treated DPF samples were analyzed, some examples of micrographs for Basel end DPF Fiber are shown in figures (5a, 5b, 5c, 5d)



**Fig. 5(a). Untreated Basel End DPF with 0% NaOH**



**Fig. 5(b). Basel End DPF treated 0.5% NaOH**



**Fig. 5(c). Basel End DPF treated 0.75%NaOH**



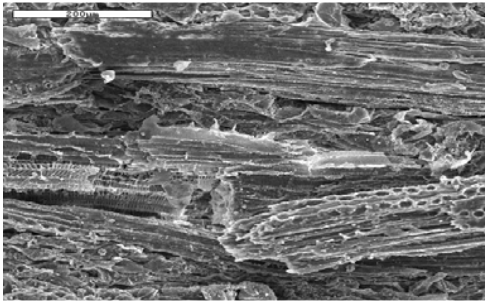


Fig. 5(d). Basel End DPF treated 1%NaOH

## Mechanical Properties of Alkalinized Fiber Date Palm Frond

### Tensile Test

The trials of push-ups are made according to the standard ISO37/2005 and the method used is (Dynamométrie sur ZWICK), the mechanical properties measured were tensile strength, Young's modulus, and elongation to break of the DPF specimens. This ISO 37/2005 Standard is typically used to quantify the mechanical properties. Tensile tests were performed using a universal testing machine 1455 WN model 116942. A load-cell with a capacity of 2 KN was used to monitor the applied load to the alkalinized fiber; the specimens were tested at 2 mm/min rate. The room temperature tests were carried out at 23°C ±2 with a controlled room humidity of 50 ±5%. Each sample of DPF included three or more specimens. The dimension of the specimen used to carry out test was adapted from ISO 37/2005, for tensile testing. All these testing were carried out for untreated and treated DPF. The last step is to calculate the elastic modulus and tensile strength from the stress-strain curve.

### Tensile Strength

The specimens were tested for tensile property determination. Consistent results were obtained for tensile Strengths, which proved the effectiveness of the treatment. The mechanical properties of the DPF before and after NaOH treatment at different concentrations are shown in Figure (6)

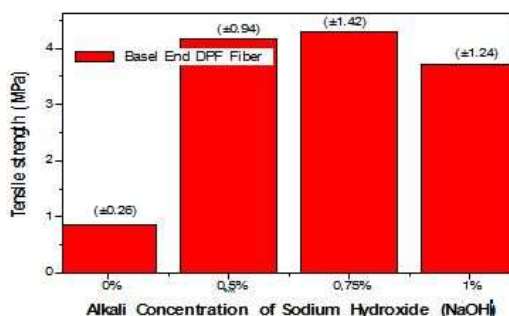


Fig. 6. Average tensile strength of Basel End DPF Fiber Versus alkali concentration (NaOH)

The maximum tensile strength was reported at 0,75% NaOH treatment. As soda concentration increases the fiber become cleaner of its impurities and later improves the tensile strength from 0% NaOH through 0.5% NaOH to 0,75% NaOH treatment to exceed 4,28 Mpa for Basel End DPF Fiber, 62,04 Mpa for Cluster DPF Fiber and 135,04 Mpa for Stalk or Stem DPF Fiber.

However, it is interesting to note, as soda concentration increases and reaches 1% NaOH, the solution attacks the main construction components of the fiber and more grooves appear on the surface of the fiber. Improvement in tensile strength of DPF was observed when soda treatment was applied. This results in further weakening in fiber strength, so the tensile strength start to decrease. As it is known, natural fibers are usually composed of cellulosic materials cemented together with weaker materials. The deterioration mechanism has been explained to be due to the attack of the cementing materials rendering the cellulose chains unconnected and hence unable to carry any load. Eichhorn et al. reported in there review that high concentration of caustic soda results in a decrease of fiber tensile strength due to notched grooves at the plant fibers surface [15].

### Tensile Modulus

Tensile modulus is a measure of rigidity of the material. The effect of the alkali treatment for the DPF fiber provides enhancement of their rigidity for all conditions (different alkali concentrations). It means, there is a significantly increasing in tensile modulus with the increase in the alkali concentration. Figure 7 show that the maximum tensile modulus was provided by 0, 75% alkali concentration. But, the most important conclusion from these results is the significant enhancement of the tensile modulus of DPF fiber with the alkali treatment.

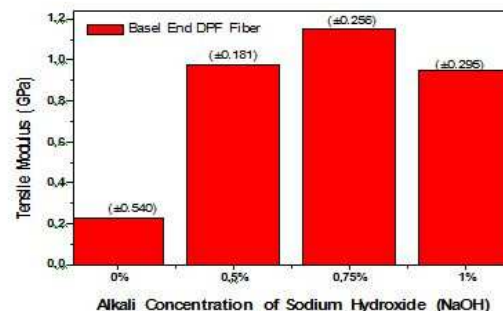


Fig. 7. Average tensile modulus of Basel End DPF Fiber Versus alkali concentration (NaOH)

Rong et al. [16] reported that the alkali treatment for sisal fibers provides the improved crystallinity of cellulose and remove the hemicellulose and lignin content. Then, it suggests that sisal fiber becomes relatively ductile after the removal of some hemicellulose and lignin. The fibers can result in higher fiber stiffness due to the increased crystallinity of hard cellulose. For the case of DPF fiber, similar reason for the improvement in the tensile modulus is viewed.

The average tensile strength, tensile modulus and elongation failure for each sample of groups untreated and treated DPF Fibers were calculated as the mean value of the carried out measurement on all the specimens tested and shown for example for Basel End DPF Palm Fibetr in table 1.

**Table 1. Mechanical properties of Raw and treated Basel End DPF with Soda Solution (0, 5%-0, 75% - 1 %)**

Concentration of NaOH treatment	Tensile Modulus (GPa)	Tensile Strength (MPa)	Elongation Failure (%)
Basel/End DPF at 0% (Untreated)	0.23±0.005	0.73±0.260	3.39±1.01
Basel End DPF at 0.5%	0.975±0.181	3.70±0.940	1.06±0.34
Basel End DPF at 0.75%	1.150±0.256	4.28±1.420	0.96±0.58
Basel End DPF at 1%	0.951±0.295	4.16±1.240	0.69±0.51

## Conclusion

The aim of this work is to optimize the mechanical properties of date fiber (DPF) by an alkaline treatment.

In fact two specimens untreated and treated with NaOH solution at three different concentrations, were tested for mechanical properties. All the results leads to conclude that Date Palm mechanical properties can be improved by NaOH treatment of the fiber. This enhancement in tensile strength and tensile modulus is attributed to the improved wetting of alkali treated fiber by removal of impurities and waxy substances from the fiber surface and the creation of a rougher topography after alkalization, thus the mechanical interlocking and the interface quality will be promoted.

The hydrophilic nature of DPF palm fiber has been reduced to this treatment, the content of hemicellulose and lignin decreased, thereby an increase on the effectiveness of the orientated cellulose fibers and a considerable improvement in surface morphology were observed. The result indicates that the treatment at the condition of 0.75% NaOH is the optimum treatment which gives the maximum tensile strength, tensile modulus, the minimum mass loss and the better surface morphology of the DPF palm fiber. Thermal analysis of DPF fiber shows that soda treated fibers have

better thermal resistance compared to raw fibers which due to the repellent action of the treatment of the sample to the phenomenon of water absorption. However, at higher alkaline concentrations 1% NaOH, the effect of these parameters on tensile properties is so pronounced because at this condition, fiber damages may have been dominant. The results obtained in this study encourage us to integrate DPF Palm fiber as reinforcement in a given matrix by a prior chemical treatment at 0.75% NaOH that we can remedy to its reliable mechanical performance before its integration.

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