Study of Structure and Properties of Tunisian Typha Leaf Fibers

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***Abstract* —Typha fiber is derived from the leaves of Typha plant, which belongs to the family Typhaceae. The fiber has been obtained using a hot alkali treatment. Some properties of this fiber, such as diameter, linear density, tensile properties and thermal properties have been determined.**
Using scanning electron microscope, the morphological structure of the obtained fibers has been investigated and shows a structure similar to a natural composite of ultimate fiber bundles of cellulose. As a result, measured properties have been compared with untreated typha fibers and other lignocellulosic fibers in order to conclude on the importance of the textile potential of the Typha leaf fibers.

Keywords—Typha fiber; hot alkali treatment; thermal properties; tensile properties; morphological structure.

I. INTRODUCTION

The problem of the shortage of resources and environmental pollution increases the development of new natural fibers. The natural cellulose fibers are the most abundant renewable resources in the world [1]. A series of researches on new type of natural fibers have been carried out in the last few years, such as, pineapple leaf fibers [2], palm fibers [3], and bamboo fiber [4].

So, a myriad of plants remain to be explored. Typha or cattail ("marsh" in Greek), is considered as a new vegetable source of fiber and it constitutes the subject matter of this study. Typha, is a monocotyledonous plant belonging to the family Typhaceae, with about 12 species distributed throughout the tropical and temperate regions of the world in marshes and wetlands [5].

Leaves are thick, having a spongy section due to the presence of the air channels. It may exceed 3 m in height. Because its leaf fiber is similar to that of hemp or jute, it can be used in the textile industry for the same purposes [6]. All parts of this plant are extensively used: Cattail roots rich in starch are eaten raw or boiled. Pollens are yet another source of protein used as additive in making bread. Moreover, the leaves are diuretic. The pollen is astringent, desiccant, diuretic, haemostatic and vulnerary, and, used in the treatment of nose bleeds, uterine bleeding, dysmenorrheal, postpartum abdominal pain and gastralgia, scrofula and abscesses. Other researches show that Typha stands may be used to clear sewage and industrial wastes [5]. Typha fibers uses are restricted as a natural fiber for composite reinforcement [7] and paper making [8]. Although some researchers have tried to extract and develop Typha fibers of different varieties in the world, the Tunisian Typha variety has not been described in the literature.

In this study, we attempted to extract the Typha fibers and remove non-cellulosic materials using a hot alkali treatment. Fiber quality has been evaluated in terms of extraction yield, diameter measurement, linear density, morphological and mechanical properties. Thermal analysis and Infrared spectrum have also been investigated, since fiber performance depends on its chemical composition and physical properties. As a result, measured properties have been compared with untreated Typha fibers and other lignocellulosic fibers in order to conclude on the importance of the textile potential of the Typha leaf fibers. The importance of the extraction conditions has also been discussed in terms of their influences on measured properties.

II. MATERIALS AND METHODS

A. Materials

Typha plant as shown in Fig. 1was collected from the river in Moknine (Tunisia). Leaves (Fig. 2) were washed, dried and cut to 10-12 cm lengths. For the experiments, sodium hydroxide and acetic acid were used for fibers extraction and neutralization [9].

Fig. 1: *Typha plant*

B. Methods

1) Fiber extraction

The template Vegetable fibers can be extracted by various methods based on either the mechanical action, or the dissolution of the constituents of the sheet other than the fibers by chemical or microbial action [10]; [11]. Several studies have revealed how various methods such as silane, alkali, peroxide, and isocyanate treatments affect the properties of natural fibers [12]; [13]. Out of these methods, it has been observed that one of the simplest, most economical and effective forms of treatment with least environmental impact, is alkali treatment particularly using NaOH [14].

Literature has shown the effect of varying alkali concentrations, treatement temperatures and times during natural fiber mercerization [15]; [16]. So, several experiments were carried out to study the suitable conditions for extracting fibers from Typha leaves. The fibers were extracted using a chemical treatment in a Mathis LABOMAT. For this, we used the Box–Behnken design (Table I.) which is a spherical, revolving design. Viewed as a cube, it consists of a central point and the middle points of the edges. It has been applied for optimization of several chemical and physical processes [17]; [18]; [19]. All statistical analyses have been carried out using the statistical software, Minitab 14 [20]. An experimental database has been elaborated by varying the Typha extraction parameters. In this database (15 tests), we used as input variables the temperature, the extraction time, and the soda concentration. The outputs are the fiber yield, the diameter, the linear density and mechanical properties.

The extraction bath is as follows:

- 5g of typha leaves.
- Liquor ratio = 1/40.
- Temperature (T (°C)) varies from80°C to 120°C.
- Duration (D (h)) of treatment varies from 2 to 4h.
- Concentration of soda(NaOH (g/L))from 10 to 30g/L.

The treated fibers were thoroughly washed in warm water to remove dissolved substances.

The fibers were then neutralized with 10ml/L acetic acid, rinsed in water, and dried under ambient conditions [6].

2) Morphological properties

The morphological structure of the typha fibers was studied using the scanning electron microscopy (SEM).

Fig. 2: *Typha leaves*

TABLE I: FEATURES OF THE BOX BEHENKEN DESIGN

Factors	Min	Mean	Max
Time (h)	2	3	4
Temperature (°C)	80	100	120
NaOH concentration (g/L)	10	20	30

3) Fineness measurment

The fineness of Typha fibers is given by measuring the diameter. It is measured using an optic microscope Leica. The test is carried out on 300 fibers chosen at random. We are based on the French standard NF G 07-004, which determines the method used to measure the diameter of wool samples using a projection microscope.

4) Linear density

We determined the linear density with the standard ISO 1973 by weighing known lengths of the fibers. In this study, the gravimetric method was used.

5) Yield measurement

Yield of fibers (R %) is measured by the percentage of the ratio between the final mass of the fibers after chemical extraction process (Mf) and that of the Typha leaves 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. The material is placed in a special oven equipped with a ventilator and a thermostat. The drying temperature is of course depending on the nature of the fiber. It is usually slightly greater than 100°C (about 105°C). Dehydration by heating is carried out for at least 3 hours until a dry or almost dry constant weight (Mf).

Desiccation is considered complete when two successive weightings made at 15 minute intervals give a less than 0.05% of the mass of the sample difference. We proceed in the same way to calculate the initial and final mass to obtain the fiber yield.

$$R\ (\%) = (Mf / Mi) * 100 \qquad (1)$$

6) Strength and elongation at break

The tensile tests of the fibers were performed under standard conditions with a LLOYD dynamometer according to NF G07-002 relating to the determination of the strength and elongation at break under tensile stress. The length between clamps is taken equal to 20 mm; the crosshead speed was 20 mm/min and a measurement cell of 10 N. The values are reported as averages of 50 test replicates.

7) Chemical structure

In the research presented below, we have used IR spectroscopy to characterize Typha leaf fibers. Infrared radiation will excite molecular vibrations within a material; the frequencies of these vibrations, and hence the absorption peaks in the spectrum, are characteristic of the chemical composition of the specimen.

The IR spectra of Typha fibers were recorded using an apparatus type Shimatzu 8400 FTIR equipped with a software hyper1.57. A pellet was made from dry KBr (potassium bromide) and the powdered sample was placed in the sample

holder of the instrument. The spectra were recorded over the range 400–4000 cm_1. The IR spectroscopy was applied to determine the functional groups and chemical structure of Typha fibers

8) Thermal stability

Extracted fiber samples were analyzed with an apparatus Perkin Elmer Diamond TG/DTA. Thermograms are obtained with a heating rate of 10°C/min; samples are treated under nitrogen stream and at a temperature ranging from 30 to 550°C. Differential thermal analysis (DTA) curves and thermo gravimetric analysis (TGA) curves were obtained.

III. RESULTS AND DISCUSSION

The characterization of these fibers is an essential step in the study of their textile potential to find the appropriate uses. Before starting any measurement, the fibers must be packed in a standard atmosphere with 20°C ± 2°C for temperature and or 65% ±4 % relative humidity.

For our study, we attempt to determine the most efficient conditions, the lowest for economic reasons while having good properties measured.

A. Morphological and physical properties

Fig. 3 represents longitudinal views of the studied Typha fibers. 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, the same result has been showed by other researcher [9].

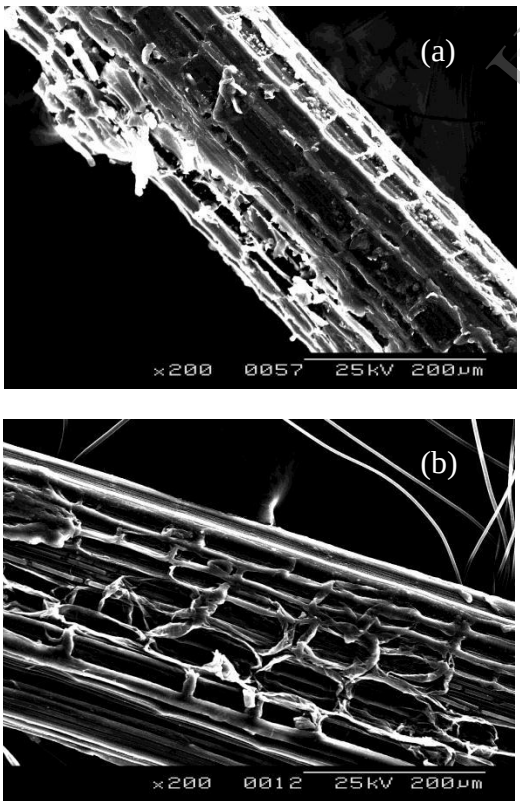


Fig. 3: *SEM of the surface of the surface of untreated typha fiber (a) and alkali treated Typha fiber (b)*

As shown in Fig. 3 (a), the untreated fibers had smooth surfaces having some impurities like wax and gummy substances whereas the surfaces of the treated fibers were rough. After soda treatment, SEM micrographics show (Fig.3 (b)) an improvement in surface morphology. In addition, the following reaction takes place as a result of alkali treatment [21]; [22]; [23]:

$$\text{Fiber} - \text{OH} + \text{NaOH}\ \text{Fiber} - \text{O-} - \text{Na+} + \text{H2O} + \text{impurity} \quad (2)$$

In this structure, the OH groups of the cellulose are converted into O – Na-groups, expanding the dimensions of molecules. Subsequent rinsing with water will remove the linked Na-ions and convert the cellulose to a new crystalline structure [24]. After alkali treatment, the crystallinity of fibers increases [20], which might be attributed to the removal of the cementing materials, leading to a better packing of cellulose chains. Additionally, treatment of the fibers with NaOH might lead to a decrease in the spiral angle and increase in molecule orientation. Then using soda treatment cleans the fiber surface of a large amount of impurities (gummy and waxy substances). This is approved by the appearance of clean structure on the surface of the fibers after alkali treatment as shown in Fig. 3 (b). This figure shows that the chemical process of extraction using sodium hydroxide allows the separation of ultimate fibers. Fineness in textiles is one of the most important characteristics that affect application and quality of the final products. Alkali treatment was found to change fiber surface topography, and the fiber diameter was reported to be decreased with increased concentration of sodium hydroxide concentration [25].

To better visualize the effect of extraction conditions on the fiber fineness of Typha, contour plots were drawn. The evolution of different properties depending on the treatment time and concentration are given for a temperature of 100°C. As shown in the diameter contour plot (Fig. 4), fiber extracted at high concentration and for longer times resulted in fine fibers due to removal of more impurities and gummy materials which held the fibers in bundles such as pectin, lignin, hemicellulose, wax, and fat materials which confirms results shown by the SEM figures.

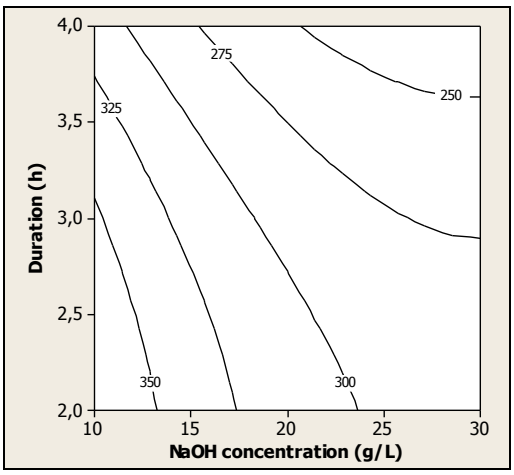


Fig. 4: *Contour plot of Diameter (µm); T= 100°C*

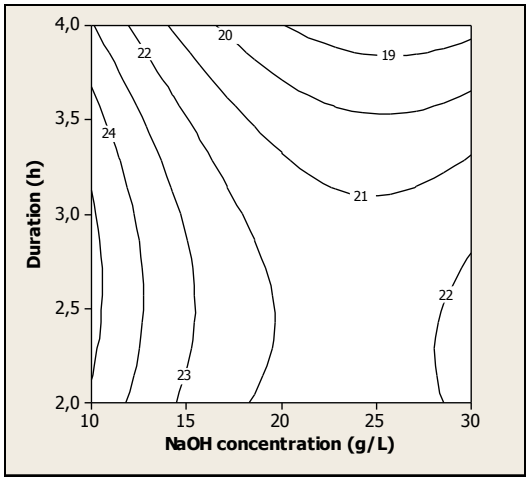


Fig. 5: *Contour plot of Linear density (tex); T=100°C*

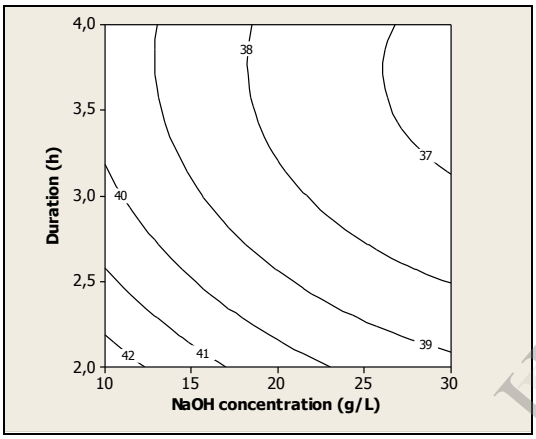
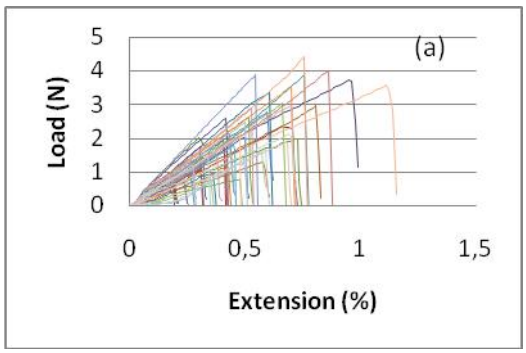


Fig. 6: *Contour plot of Yield (%); T=100°C*



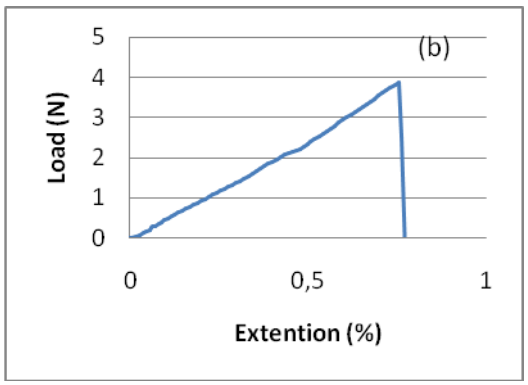


Fig. 7*: Load-elongation diagrams for fifty treated fibers (a) and for individual treated fiber (b)*

Fibers extracted at 120°C for 4 h were observed to be thin (141 µm). Against, thick fibers are obtained by proceeding with minimum temperature and concentration of caustic soda for 3 hours which also shows the important role in the choice of parameters of Typha fiber extraction.

Treatment duration, soda concentration and temperature favor the separation of cattail fibers and their cleanings while removing foreign matter deposited on the fibers which decrease their diameters. The average diameter of untreated fibers is 486 µm. This bigger value resulted from layers of wax and fatty substances on their surface and a large numbers of micro fibrils in one fiber bundle.So, in order to obtain a thin fiber, extraction conditions needs to be severe. In fact, the principle of the NaOH treatment is that the gum is removed layer by layer, and the inner gum remains intact; then the coarsest fiber comes into being. Besides, as shown in the contour plot of linear density (Fig. 5), the linear density also varies depending on the fiber extraction parameters. The average linear density of untreated Typha fibers was 32 Tex. After alkali treatment, there is a reduction in the fiber mass per unit length which can be explained by the removal of foreign material of the fiber. The finest fibers are obtained by working in the maximum temperature and duration of treatment with a concentration of 20 g/L of sodium hydroxide. The linear density in this case is equal to 10 Tex which confirms the previous results of diameter.

From Table II., treated Typha leaf fibers were found to be very thick particularly with cotton fibers. Compared to untreated Typha leaf fibers, fineness increases with alkali treatment. Moreover, a clear difference between the measured properties of Tunisian

Typha variety and the Iranian one was given. This one seems obvious because the properties of vegetable fibers vary within the variety and even in the same plant. In fact, studies have shown that the chemical composition and physical properties vary depending on several parameters such as the level of fiber in the sheet (top or bottom of the plant), the layout of the sheet from which the fiber is extracted (from the heart of the plant). Fiber extracted at 80°C shows low yield results that some of the leaves remain after the fiber extraction process. So, in this condition, it was ineffective to remove impurities on fiber surface. When the temperature increases, the performance improves, it is about 46% for T = 100°C and C = 10 g/L for 2 hours extraction. Whereas, it decreases by increasing the

processing conditions (Fig.6). This may be explained by the fact that for severe processing conditions, the removal of non cellulosic components occurred at a faster rate with deterioration of fibers.

B. Strength and elongation at break

As reported in much literature, natural fiber chemical constituent consists of cellulose and other non cellulosic constituent like hemicellulose, lignin, pectin and impurities such as wax, ash and natural oil [26]; [27]. This non cellulosic material could be removed by appropriate alkali treatments, which affect the tensile characteristic of the [23]; [16]. Tensile properties of chemical fibers extracted were determined by a LLOYD LRX tensile tester. The load-elongation diagrams on fifty controlled tensile fibers are shown in Fig. 7 (a). From the figure, we note that the relative arrangement of different curves

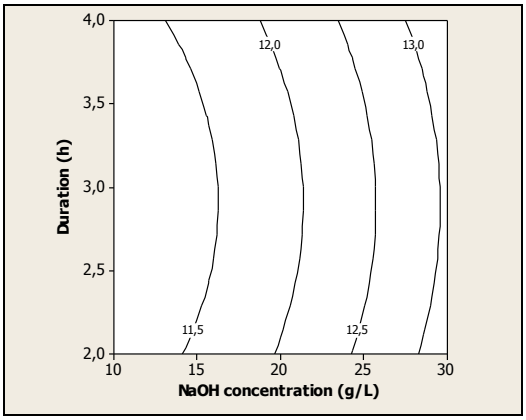


Fig. 8*: Contour plot of Tenacity (cN/tex); T=100°C*

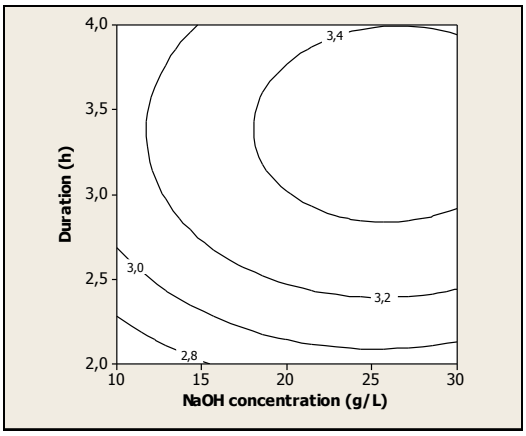


Fig. 9: *Contour plot of elongation (%); T=100°C*

obtained, confirms the great dispersion described by high values of the coefficients of variation of all the mechanical characteristics of the tensile test, which can be accepted in the case of natural cellulosic fibers (CV%~30-45), unlike the synthetic fibers [28].

To better describe the behavior of Typha fiber strength, we will discuss in more detail a loadelongation diagram obtained from an individual fiber. This diagram is shown in Fig.7 (b), the load-extention curve for individual Typha fiber. It is observed that this fiber has higher modulus value without a yielding point. The same behavior has been shown by linen and jute fibers [9].

Studies depending on the temperature alkali treatment have shown how fibrillation is affected with increased treatment temperatures [15]. At higher temperatures, it was discovered that fibrillation and the removal of non cellulosic components occurred at a faster rate. On the other hand, if the treatment temperature is too high, it is possible to deteriorate cellulose, resulting in lower mechanical properties of the fibers.

From Figure8, the results show that the tenacity of fibers increased with sodium hydroxide concentration due to the removal of impurities and the arrangement of the cellulosic chain.

The addition of NaOH improved fiber separation, possibly by neutralizing the acid produced and removing more impurities. However, higher concentrations of sodium hydroxide reduced fiber tenacity as a result of destruction of cellulosic structure and greater impurity removal (C=20g/L, T=120°C, D=4h).

TABLE II. : PHYSICAL AND MECHANICAL ROPERTIES OF SOME VEGETABLE FIBERS

Fiber	Diameter (µm)	Linear Density (tex)	Tenacity (cN/tex)	Elongation (%)	Reference
Nettle	30	___	59.3	5.6	[35]
Kenaf (10% alkali treated)	50- 80	4.5	40	2.57	[10]
Jute	47	___	31	1.8	[36]
Cotton	12- 25	___	32	7.1	[36]
Tunisian Typha (untreated)	520	32	7.76	3.34	This paper
Iranian Typha	___	3.92- 7.3	18.32 - 40.95	1.23- 3.36	[9]
Tunisian Alkali treated Typha	141- 506	10- 30	7- 16	1.96- 4.60	This paper

As consequence, the best fibers are obtained in a moderate condition of Typha extraction in order to have good mechanical properties. Besides, as we can see from Table II, alkali treated Typha fibers are less resistant compared to others fibers like cotton, kenaf,jute fibers whereas, it seems to be resistant when compared to untreated Typha fiber. This confirms that alkali treatment affects the mechanical properties of the fibers by arranging the cellulose molecular chains.

Contour plot of Elongation of these fibers for different extraction conditions does not exceed 4.6 %, which confirms the property of natural fibers having generally low elongation (Table II.).

C. Chemical structure

Lignocellulosic fibers have a heterogeneous structure. IR spectra are characterized by their relatively sharp absorption

bands. Similar absorption bands in the spectra are generally found in the fibers having the same chemical composition. Fig. 10 shows the whole IR spectra of untreated and alkali treated Typha leaf fiber sample determined at 400-4000 cm-1 wave number.

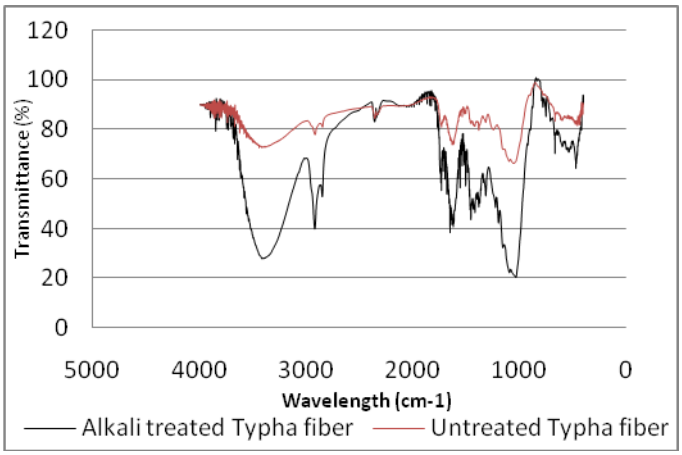


Fig. 10: *IR spectra of the Typha leaf fibers*

The transmittance peaks of interest in this study are identified and shown in Fig. 10. 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 2852-2921 cm-1 indicating the stretching vibration of the groups -CH and –CH_2 of cellulose and another band at 1383 cm-1, which also indicates the presence of -CH produced by a symmetrical deformation of lignin and alpha cellulose. The medium-intensity transmittance band at 1730 cm-1 is assigned to the C=O stretching of carboxyl and acetyl groups in the hemicelluloses content of the fiber, which appeared as a strong peak at 1739 cm-1 in the case of untreated Typha fibers and which indicated a higher hemicellulose content. But, it is noted that this absorption peak, was almost missing in the spectrum of the alkali treated fiber, indicating the elimination of hemicellulose occurred by alkali treatment.The observed band at 1635 cm-1, due to vibrations of adsorbed water molecules in the no crystalline region of cellulose, appeared as a shoulder in the spectra. The peak at 1506 cm-1 was caused by aromatic C=C skeletal in-plane vibrations and were diagnostic of the aromatic structure of the lignin present in the fiber and indicated their lignocellulosic characteristics. Furthermore, there was no change in this peak, which shows no structural change of the lignin component in the Typha fibers after alkali treatment.

The peak at 1157 cm-1 in untreated fiber, which can be attributed to C-O-C anti-symmetric bridge stretching in cellulose and hemicellulose [29] no longer exists for NaOH treated fibers. During alkali treatment, a substantial portion of uronic acid and fatty substances might be removed resulting in disappearance of this [30]. The band at 900 cm-1 is attributed to β- glucosidic linkages between the sugar units in hemicelluloses and celluloses while the band at 658 cm-1 can be linked to OH out-of-plane bending [31] and/or atmospheric CO_2 (deformation vibration) contamination [32].

The analysis of the IR spectra of the untreated Typha 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 hemicellulose by alkali treatment, which results in the untreated fiber bundle broken down into smaller ones. This phenomenon of fibrillation is the reason that fiber diameter of the treated fiber decreased. Same observation on Typha australis (Iranian variety), kenaf, jute and plam fiber has been reported [9]; [30] and [33].

D. Thermal stability

The thermal stability of the Typha fibers was studied to assess the possibility of their being used as reinforcement. The peaks shown in Fig. 12 are peaks in the DTA curves that corresponded to points of maximum slope the original TGA curves (Fig. 11). For each of these peaks, the mass loss associated with it was calculated, and the corresponding peak temperatures are reported in Tables III and IV. The DTA curves were found to have three stages of weight loss coinciding with regions of weight loss observed through TGA, as shown in Fig. 11 and 12 for the extracted fibers. Where M1is weight loss of fiber corresponding to the peak 1 of DTA

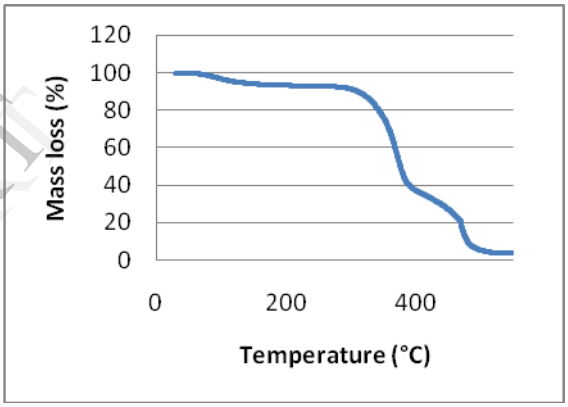


Fig. 11: *DTA curve of Typha leaf fiber*

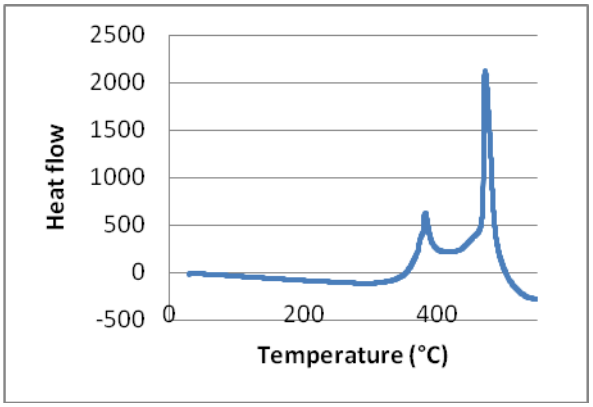


Fig. 12: *DTA mass loss of Typha leaf fiber*

peak temperatures; M2 is weight loss of fiber corresponding to the peak 2 of DTA peak temperatures and M3 is weight loss of fiber corresponding to the peak 3 of DTA peak temperatures.
From a room temperature until 120°C, the TGA thermograms (Fig. 12) present the loss of mass due to water evaporation. The second peak at 381.16°C and the third one at 471.66°C were due to the decomposition of the hemicelluloses and

cellulose, respectively. So, thermal analysis indicates that the Typha fibers were thermally stable below 381°C and that, as such, the fibers could be effectively used as reinforcement when the molding temperature was set under this temperature. Thermal stability of Typha seems to be higher than kenaf fiber (below 218°C) [34]. This confirms the significant thermal insulation properties of Typha which can be used for electronic package, auto parts…

TABLE III : *DTA peak temperature of Typha leaf fiber*

	Temperature (°C)		
	Peak1	***Peak 2***	***Peak 3***
Typha fiber	46.16	381.83	471.66

TABLE IV*: DTA mass loss of Typha leaf fiber*

	Mass loss (%)		
	M1	***M2***	***M3***
Typha fiber	99.71	44.10	16.28

TABLE V: *p-values meaning*

	Linear Density	**Diameter**	**Yield**	**Tenacity**	**Elongation**
NaOH (g/L)	*	*	*	*	*
D (h)	*	*	*	*	*
T (°C)	*	*	**	**	*

* : Insignificant influence (p> 0.05)
**: Significant influence (p< 0,05)

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. Results of p-values are shown in Table v. From this table, the most influent parameter on the measured properties was temperature which affects mostly the Tenacity and Yield measurement.

IV. CONCLUSION

Typha fiber is a natural vegetable fiber that is derived from the leaves of Tunisian Typha Variety, which belongs to the family Typhaceae. The study of the fibers extraction conditions seems to have an important role on the fibers properties. Typha leaf fiber shows a linear density between 10 and 30 tex with a diameter between 141 – 496 µm and Yield variant from 25.1 to 46.6%. Tenacity of fibers was optimal (15.1 cN/tex) for T=100°C; soda concentration of 30g/L during 4hours. After soda treatment, SEM micrographics show an improvement in surface morphology. Moreover, Infrared spectrum of the fibers showed that the whole patterns of Typha fiber were similar to other natural cellulosic fibers, confirming the cellulosic structure and lignocellulosic nature of the extracted fibers. As a first study, in order to have fibers with good quality in terms of measured properties, we must work in intermediate temperature, concentrations of caustic soda and duration of treatment; otherwise degradation of cellulose takes place which gives weak fibers. The Typha leaf fibers from Tunisian variety represent another source of natural fibers which need more research and development in the future to valorize its textile potential. The study of its chemical composition, cristallinity, density, better extraction methods optimization of the different extraction condition etc, will be reported in the future.

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