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OPTIMIZATION OF PROCESSING PARAMETERS FOR EXTRACTION OF TYPHA STEM FIBERS

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ABSTRACT

The influence of temperature, duration and soda concentration on the extraction yield, linear density, diameter, Tenacity and lignin ratio of Typha Stem fibers has been studied. A factorial design of experience has been used to identify the optimum operating conditions, equations relating the dependent variables to the operational variables of the extraction process were established. The optimum extraction condition was determined by the statistical study using response surface and desirability function. The morphology of the obtained fibers and chemical constituents were determined. Fibers, extracted from stems of Typha with the optimum process, have a lignin content value about 22,05% like alfa fibers and Typha Domingensis, alpha-cellulose value about 51,3% similar to jute bast fibers, extractives content value about 3,6%, starches content value about 4,25% and ash content value about 3,2%. Finally, the characteristics of the optimum Typha fiber were compared to those of other vegetable fibers showing high diameter and low mechanical properties.

KEYWORDS

Typha Stem fibers; Extraction process; Physical properties; Chemical properties; Design of experiment.

1. INTRODUCTION

The natural cellulosic fibers are the most abundant renewable resources in the world (L. Y. Liu et al., 2011). A series of researches on natural fibers have been carried out, such as, milk weed stem fiber, linen, Hop stem fiber, hemp fibers (Narendra, Yiqi 2009; Sfiligoj et al., 2013)… Typha (“marsh” in Greek), shown in Figure1, is considered as a vegetable source of fiber, represents the subject of this study. Typha, is a monocotyledonous plant belonging to the family Typhaceae, with about 12 species distributed in the tropical and temperate regions of the world in marshes and wetlands (Kumar et al., 2012). Typha fibers uses are restricted as a natural fiber for composite reinforcement (Wuzellaa et al., 2011) and paper making (Sarwar et al., 2007).

In a previous study (Rezig et al., 2014), we tried to study the extraction of fibers from Typha leaves, in which, the same extraction method was used and the effects of the extraction parameters on the measured properties were studied. Therefore, the purpose of this study was to extract the Typha fibers and remove non-cellulosic materials using a hot alkali treatment. The objective of this paper was to optimize the extraction process. Fiber quality has been evaluated in terms of extraction yield, fiber diameter, linear density, tenacity and lignin ratio and the optimal extraction condition was determined. This study was completed by a characterization of the fibers extracted in the optimal condition: chemical composition and morphological properties. The obtained characteristics were compared to other vegetable fibers and to conclude on the importance of the textile potential of the Typha stem fibers.

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Figure 1: Typha plant and stems

2. MATERIALS AND METHODS

2.1. Fiber extraction process

To extract fibers from the organic matrix, different methods can be used. These methods have a great influence on the fine structure of the obtained fibers (Hawley et al., 2008), (Joseph et al., 1996). In this study, we attempted to extract the Typha stem fibers and remove non-cellulosic materials using a hot alkali treatment (Williams et al., 2011), particularly using NaOH (Xioa et al., 2001)

Several experiments were carried out to study the suitable conditions for extracting fibers from Typha stems. The fibers were extracted using a chemical treatment in a Mathis LABOMAT. The extraction bath is as follows:

- 5 g of typha stems.
- Liquor ratio = 1/40.
- Temperature (T) from 80°C to 120°C.
- Duration (d) of treatment from 2h to 4h.
- Concentration of soda (C) from 10g/L to 30g/L.

We have noticed that at a temperature below 80°C, Typha fibers remains bonded to each other. Besides, the use of an amount less than 10 g/L of NaOH for less than 2 hours, gives a partial stems extraction. 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 (Mortasavi, Moghadam, 2009).

2.2. Experimental design and desirability function

For the fiber extraction, we used the Box–Behnken design of experimnts based on three-level incomplete factorial designs. 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 (Abdelmouleh, 2006; Eddine et al., 2004; Iniguez, 2001). All statistical analyses have been carried out using the statistical software Minitab 14 (Evans et al., 1995). An experimental database has been elaborated by varying the Typha extraction parameters as presented above (extraction bath composition). In this database (15 tests), we used as input variables the temperature (T), the extraction time (d), and the soda concentration (C). The outputs are the extraction yield (Y), the fiber diameter (D), the fiber linear density (LD), the fiber tenacity (T) and lignin ratio (L).

The independent variables were normalized to values from –1 to +1 in order to make easy direct comparison of the coefficients of the resulting polynomial equation and an understanding of the effects of the individual independent variables on the dependent variables considered (yield, linear density, diameter, fiber tenacity and lignin ratio).

Experimental data were fitted to the following second-order polynomial equation:

$$Y_i = a_0 + a_1X_C + a_2X_d + a_3X_T + a_{11}X_C^2 + a_{22}X_d^2 + a_{33}X_T^2 + a_{12}X_CX_d + a_{13}X_CX_T + a_{23}X_dX_T \quad (1)$$

where Y_i is the response of dependent variable [yield (Y_Y), diameter (Y_D), the linear density (Y_{LD}), the tenacity (Y_T) and lignin ratio (Y_L)]; X_T, X_d and X_c are the normalized values of T, d and C, respectively and a_0, a_i and a_{ij} are unknown characteristic constants estimated from the experimental data.

Therefore, this experimental design would be used in order to optimize the extraction conditions of Typha fibers, where it is necessary to achieve the right combination (C, T, and d) that:

- Minimizes D, LD, L
- Maximizes Y and T.

Among the optimization tools that can be used, there is the desirability function (Mounir Jaouadi 2009). In this study, we used two types of desirability functions "d_i": desirability function to maximize and to minimize (figure 3 and 4).

Using this mathematical functions we will calculate individual desirability for each property d_i (i = D, LD, L, Y and T). We calculated the global desirability using the Derringer and Suich desirability function[17] defined as follows:

$$dg = \sqrt[w]{(dD^{wD} * dLD^{wLD} * dL^{wL} * dY^{wY} * dT^{wT})} \quad (2)$$

Where w_i (i = D, LD, L, Y and T) is a relative weight to indicate the property's (Y_i) importance in the "d_g" desirability function, w is the sum of w_i.

The compromise between the properties 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 the compromise was rejected (Mounir Jaouadi 2009). So, the optimum combination of (C, T, D) is the one that gives "d_g" close to 1.

Thus, to maximize a property "Y_i", for example the extraction Yield (Y_y), the desirability function (shown in Figure 2) had to be used, where the individual desirability (d_D) was calculated as follows:

dy= 0 if Yy < Yymin dD = 0 if YD ≤ Yymin (3)

$$dy = \left(\frac{Yy - Yymin}{Yytarget - Yymin}\right)^s \quad if \quad Ymin \le Yy \le Yytarget$$
(4)

dy=1 if Yy≥Yytarget *(5)*

To minimize a property "Y_i", such as the Linear Density (Y_{LD}), the desirability function (shown in Figure 3) had to be used, where the individual desirability (d_{LD}) was calculated as follows:

dLD= 1 if $Y_{LD} \le Y_{LDtarget}$ *(6)*

$$dLD = \left(\frac{yLD - y\,LDmax}{y\,LDtarget - y\,LDmax}\right)^s \quad if \quad y\,LD\,target \le yLD \le y\,LDmax$$
(7)

dLD = 0 if $Y_{LD} \ge Y_{LDmax}$ *(8)*

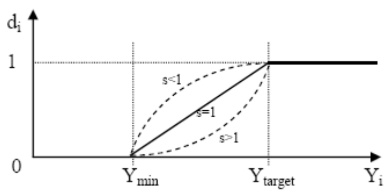

Figure 2: Desirability function to maximize

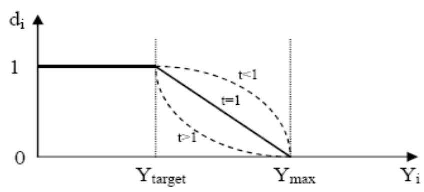

Figure 3: Desirability function to minimize

2.3. Characterization of the extracted fibers

A search for significant effects on quality parameters is demanding (Calado et al., 2014). The properties of the Typha stem fibers are determined by the physical, mechanical and chemical properties of the morphological constituents and their interfaces.

2.3.1. Chemical composition

The hemical composition of natural fibers varies depending upon the type of fiber. Primarily, fibers contain cellulose, hemicelluloses, pectin and lignin. The properties of each constituent contribute to the overall properties of the fiber (JOG et al., 1999). Lignin plays the role of binding the fibers of cellulose. Alkaline treatment is used for the release of fibers just as it is one of the standard procedures in the pulp and paper industries for lignin removal, lignin can be dissolved in sodium hydroxide (NaOH) solution and the cellulosic fibers can be extracted with relative ease. NaOH causes dissolution of lignin by breaking it into smaller segments whose sodium salts are soluble in the medium (Ebisike et al., 2013). In order to optimize the fiber extraction process, ratio of Lignin was determined according to ASTM standard Test method D 1106 – 96 (2001).

Besides, chemical constituents of obtained fibers under optimum conditions: alpha-cellulose, lignin, starches, extractives and ash content were determined following TAPPI ans ASTM standard methods: T 203 cm-09; T 222 om-11/ASTMD 1106 (2001); ASTMD 1110 (2001) / T207 cm-08; ASTMD 1107(2001); and T 211 om-12/ ASTMD 1102 (2001) respectively.

2.3.2. Yield measurement

Natural fiber extraction processes could be employed in different procedures, including mechanical, biological and chemical methods. Different techniques offered advantages and difficulties according to the quality and amount of fibers obtained (Ebisike et al., 2013; Ladkrabang, 2009). So, yield of fibers (Y %) seems to be an important factor in the optimization of fiber extraction parameters. It 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 stems 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.

$$Y (\%) = (Mf / Mi) * 100 \qquad (9)$$

2.3.3. Fineness measurement

Fineness in textile is one of the most important characteristics that affect application and quality of the final products. Therefore, it is important to determine the fineness of natural fibers since it's considered an important factor to define their cost and quality. The fineness of Typha stem fibers was given by measuring the diameter and the linear density. Diameter was measured using an optic microscope Leica, in accordance with the French standard NF G 07-004 (1983). Also, we determined the linear density with the application of the standard ISO 1973(1995) by weighing known lengths of the fibers in application of the gravimetric method.

To observe the yarns surface, Scanning Electron Microscopy (SEM, Hitachi S-2360N, Elexience, Verrières le Buisson, France) was then used at low and high magnification. The observations were made on Typha stem fiber treated at the optimum conditions. The metallized specimens were analyzed in partial vacuum conditions (0.1–0.15 torr), and under an accelerating voltage ranging from 8 to 25 KV

2.3.4. Strength at break

The quality of any textile fiber largely depends on its two important properties, namely fineness and strength. The tensile tests of the fibers were performed under standard conditions with a LLOYD dynamometer according to NF EN ISO 5079 relating to the determination of the tenacity and elongation at break of individual fibers.

3. RESULTS AND DISCUSSION

3.1. Optimization of extraction process

Table 1 summarizes the statistics of the measured properties. The Minitab software was used to conduct a multiple linear regression analysis involving all terms in equation (1). The coefficient of variation for each dependent variable was calculated and the results are between 6% and 32% and they are considered acceptable. In fact, unlike synthetic fibers, the coefficient variance of natural cellulosic fibers properties is high (CV%~30-45) (S. M. Mortasavi, Moghadam, 2010). In addition, Natural fibers have little resistance towards environmental influences and show an intrinsic variability of their properties. Hence, the use of natural fibers depends on the environmental conditions which are likely to influence ageing and degradation effects.

Table 1: Statistics of the measured properties

	Mean	Minimum	Maximum	Coefficient of variation (%)
Linear Density (tex)	42.39	31.3	48.27	11.10
Diameter (µm)	351.21	205.1	505.86	25.33
Yield (%)	24.81	15.27	39.53	14.26
Tenacity (cN/tex)	7.15	5.76	8.68	31.69
Lignin (%)	22.34	20.52	25.45	6.66

In the evaluation of experimental designs, a mathematical model is provided to relate the response variable with the factor effects. The terms of the corresponding equations (accompanied by their corresponding R^2 value (coefficient of determination)), and the p-value (p-value is the significance level for the hypothesis that the coefficient is zero) for their terms at a 95% confidence limit are shown in Table 2. The equations were subjected to non-linear programming using Minitab software in order to determine the optimum values of the dependent variables.

Table 2: Values of the coefficients and statistical parameters for the different dependent variables

Coefficients and statistical parameters	Linear Density (tex) (LD)	Diameter (µm) (D)	Yield (%) (Y)	Tenacity (cN/tex) (T)	Lignin (%) (L)
a0	42.81	346.13	25	7.03	20.78
a1	-5.22	-109.74	8.33	0.85	-0.63
a2	-1.94	-25.85	2.98	0.58	-0.57
a3	-1.58	-22.6	4.44	-0.13	-1.1
a11	-0.6	19.69	1.22	---	1.44
a22	0.95	9.09	-0.95	0.22	1.41
a33	-1.14	-19.24	-0.64	---	0.08
a12	-1.18	2.21	-0.18	---	0.1
a13	-1.4	3.89	1.8	---	-0.13
a23	-0.51	4.1	1.94	---	0.18
R2	94.5	98.6	95.2	60.7	95.5
p-value	0.012	0.000	0.008	0.038	0.007

--- denotes non significant value.

The regression coefficients of the models for Y_{LD}, Y_D, Y_Y, Y_T and Y_L are given in Table 2. The significance of each model was determined by the p-value. The smaller the p-value is, the more significant is the corresponding coefficient. The results indicated that the model was significant ($p < 0.05$). As shown in Table 2, the R^2 of each second-order polynomial regression was, 94,5; 98,6; 95,2; 60,7 and 95,5% for Y_{LD}, Y_D, Y_Y, Y_T and Y_L, respectively. The results indicated that the model used to fit the response variables were all significant and adequate to represent the relationship between the response and the independent variables.

For second order models, optimization using the desirability function technique is the recommended tool (Cruz et al., 2010; Granato et al., 2010). It is based on the idea that the 'quality' of a product or process has multiple quality characteristics. The desirability approach, proposed initially by Derringer and Suich (1980), seems very promising for optimizing simultaneous response variables, besides being easily performed (Reis, 2008). The aim was to convert each response into an individual desirability. The optimum values and corresponding normalized values for the independent variables are shown in Table 3.

Table 3: Optimum value of the properties

Property	Optimum value	XC	XD	XT
Linear Density (tex)	30.19	1	1	1
Diameter (µm)	207.66	1	1	1
Yield (%)	43.96	1	1	1
Tenacity(cN/tex)	9.01	1	1	-1
Lignin (%)	20.52	0	0	0.244

From the data in Table 3, it followed that the optimum levels of the dependent variables for the fiber extraction entailed using a low to high soda concentration, duration and temperature. In fact:

- Ensuring good values for the yield, diameter and linear density properties entailed using a high duration, temperature and soda concentration (1).
- Optimum lignin was obtained medium to high duration and medium temperature and soda concentration.
- Good values for fiber tenacity entailed using high soda concentration and duration (1) and low temperature (-1).

The operational conditions for the dependent variables determined in Table 3 were used to calculate the predicted values for the fiber extracted properties and the deviation from the optimum levels, as shown in Table 4.

Table 4: Values of the dependent variables for the fiber obtained under the stated conditions

	LD deviation (%)	D deviation (%)	Y deviation (%)	T deviation (%)	L deviation (%)
High temperature, duration and soda concentration	30.21-0.07	207.65- (-0.01)	43.97-0	8.56- (-2.33)	21.56-5.1
Low temperature, high duration and soda concentration	45.79-51.67	414.95- 99.81	24.05- (- 45.3)	6.86- (-21.72)	22.87-11.47
Medium to high duration, high temperature and soda concentration	42.36 -40.30	339.47- 63.64	26.04- (-40.76)	7- (-20.14)	20.52- (-0.016)

As we can see from Table 4, obtaining good quality and resistant Typha stem fibers, entailed using high duration and soda concentration (4hours and 30g/L respectively) and low temperature (80°C). With these conditions, the values of the extracted fibers (Tenacity and Lignin) departed by only 11 - 22% from their optimum levels. However, the deviation of the diameter, yield and linear density obtained was too high (100, 46 and 52% respectively), which varied widely with the extraction parameters. In fact, studies on kenaf fiber show that the mean diameter was reduced by 30.12% to 42.92% after alkali treatment. In addition, alkali concentration had a higher impact on diameter changes compared to alkali treatment immerse temperature (Mohd et al., 2013). Besides, they showed that kenaf fiber bundle mean cross sectional area was reduced from 6.77% to 29.88% after alkali treatment compared to kenaf mean cross sectional area. The decrease was due to swelling reaction during alkali treatment process at different conditions setting which affect the fiber structure, dimension and morphology (Mohd et al., 2013). Same results were obtained in the case of Banana Stem fibers (Samrat al., 2008). For the yield deviation, this is due to the great variability in natural fibers. In fact, it depends on the position of the fibers during the extraction. In addition, a study on the number of fibers throughout a leaf of agave sheet cutted in sections of 10 cm was studied. The results show that in the first 20 cm, the sheet contains a high percentage of pulp but also fibers which are stacked and bonded to each other. These proportions decrease as one moves away from the base of the leaf (Lock, 1962).

Once the models have been developed and checked for adequacy, the optimization criteria can be set to find out the optimum extraction process. In this investigation, the optimization criteria were implemented to maximize fiber T and Y and to minimize D, LD and L. In this criterion, the goal was to reach this objective at minimum NaOH concentration, time, and temperature.

Solving multiple response optimization equations using this technique involves combining multiple responses into a dimensionless measure of performance called the overall desirability function. The desirability approach involves transforming each estimated response, Yi, into a unitless utility bounded by 0 < di < 1. A higher di value indicates that the response value Yi is more desirable, with di =0 signifying a completely undesired response (Aly et al., 2012). So, for every experience, we got an individual desirability function for each property that allowed us to know if the property was satisfying or not. For every extraction, we also got a global desirability function by affecting the relative weight "wi" and by using the Derringer and Suich function that represented the degree of global satisfaction of the properties.

Fixing our target for each parameter studied we obtained the optimization diagram giving the optimal case and the optimal values of the properties of the fibers studied which are: a diameter of the order of 207,67 microns, a linear density of the order of 30,19 tex, a lignin ratio of about 21,57 %, a tenacity in the range of 8,65 cN/tex and a yield of about 43,96%, with a desirable value of all individual desirability functions.

These optimal characteristics are achieved if we work in optimal conditions of temperature, duration and soda concentration which are: 120°C, 4h and 30g/L respectively. The global desirability obtained was equal to 94,71% which is very significant. The statistical study determined the optimum extraction conditions that minimized the wasting energy, time and soda concentration. Therefore, the extraction cost would be reduced.

So, optimum values obtained were found to be the extreme conditions of the treatment conditions.

3.2. Characterization of the optimum fiber

In accordance with the optimization results obtained from response surface methodology with desirability function verification experiments were carried out at the selected conditions (T=120°C, d=4h and C=30g/L). Indeed, all experimental results of fiber characteristics in the optimal conditions (1) and those given by the desirability function (2) are shown in the following table:

From this table, we note that the experimental results (1) are in good agreement with the statistical conditions (2) that we proposed and the sample is validated. Thus, it can be seen that the second-order model was adequate to determine the optimum values of the dependent variables.

Table 5: Comparision of extracted typha fiber with experimental (1) and statistical method (2)

	1	2	Algerian alfa Stem fiber (Abdelhak et al., 2013)	Banana Stem fiber (Samrat et al., 2008)	Milkweed stem fiber (Narendra, Yiqi, 2009)	Linen (Narendra, Yiqi, 2009)	Typha Domingensis (Tarig et al., 2012)
Tenacity (cN/tex)	9.42	8.65	---	---	30.9±17.66	40.61-53.86	---
Yield (%)	39.68	43.96	---	---	---	---	38.5-40.6
Linear density (tex)	29.65	30.19	---	---	11.55±1.89	0.19-1.98	---
Diameter (µm)	208.12	207.67	---	80-290	---	---	---
Lignin (%)	22.05	21.57	18.2	---	4.1±0.8	2-3	---

Besides, comparing to others vegetable fibers, alkali treated Typha stem fibers are less resistant. The observed variation in mechanical properties of these fibers can be explained in terms of structural variables such as the number of cells, cell wall thickness, microfibrillar angle, cellulose content and molecular structure. Other variables such as source, age and processing presumably remain constant in view of the collection of the fibers from the same place.

Table 6: Comparative chemical composition of Typha stem fiber in per cent

	Typha stem fiber	Algerian alfa stem fiber (Abdelhak et al., 2013)	Banana stem fiber (Samrat et al., 2008)	Jute bast fiber (Krishnan, Dhas, 2012)	Typha Domingensis stem fiber (Tarig et al., 2012)
Solubility in hot water (starches)	4.25	---	---	---	7.5
Amount of extractives	3,6	---	4.46	---	2.6
Lignin	22.05	18.2	15.07	12.94	21.5
Alpha-cellulose	51.3	---	---	56.25	49
Ash	3.2	5.75	8.65	2.15	4.3

From the following table, it is clear that Typha stem fiber has a chemical composition similar to other vegetable fibers with a 51.3% of alpha cellulose like jute bast fiber and Typha Domingensis fibers and 22.05% of Lignin comparable to other varieties of Typha Domingensis from Sudan 21.5% and alfa fibers, but more than Jute, alfa and banana stem fibers.

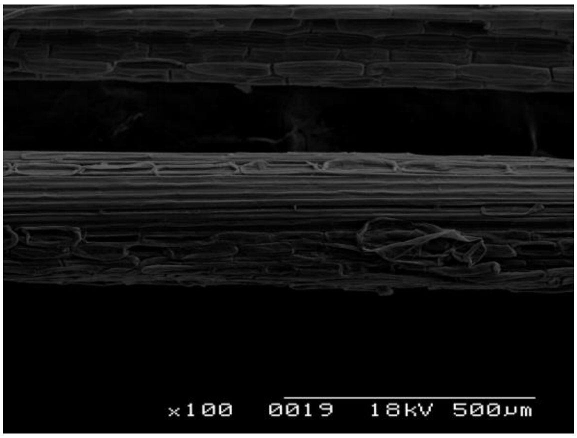

Figure 4: Longitudinal view of Typha stem fiber treated at the optimum conditions

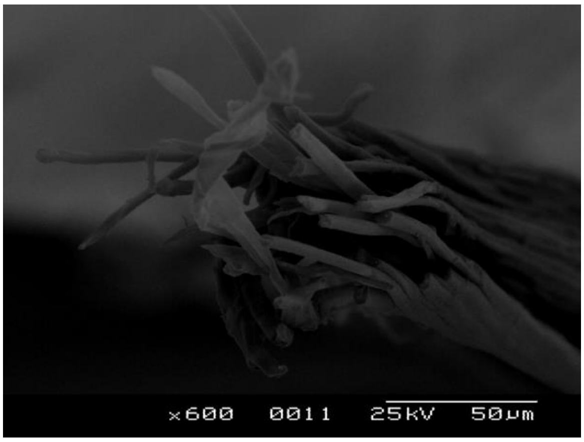

Figure 5: cross-section of Typha stem fiber at the optimum conditions

Figure 4 represents longitudinal views of the studied Typha stem fibers. Their structure is similar to a natural composite 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 researchers (Mortasavi , Moghadam, 2010; Samrat et al., 2008; Abdelhak et al., 2013; Tarig et al., 2012; Krishnan, Dhas, 2012).

After soda treatment, SEM micrographics show modification in surface morphology. In addition, the following reaction takes place as a result of alkali treatment (Li et al., 2007; Mwaikambo et al., 2002; Sreenivasanet al., 1996):

Fiber– OH+ NaOH $\longrightarrow$ Fiber – O^- –Na^++ H_2O + impurity (10)

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 (MY Hashimet al., 2012). After alkali treatment, the crystallinity of fibers increases (Gassan, Bledzki, 1999), which might be attributed to the removal of the cementing materials, leading to a better packing of cellulose chains. The soda treatment of the fiber cleans its surface from a large amount of impurities (gummy and waxy substances). Figure 5 show that the chemical process of extraction using sodium hydroxide allows the separation of ultimate fibers.

It can also be seen that a number of lumpy strips existed on the surfaces of the treated fibers (figure 4), which might be due to mercerization function of NaOH resulting in the partial removal of wax or fatty substances. It is a well-known fact (Liu et al., 2007) that there are binder lignin and fatty substances which hold the unit cells firmly in a fiber as reported in the case of jute fiber (Seong et al., 2010).

The cross-sectional investigation of Typha stem fibers showed initially intact bundles (Figure 5). It seems that some material was still attached to several bundles and formed a relatively large fragment. This finding shows that the obtained fiber is multicellular in nature just like jute bast fibers. Thus, we conclude that Typha fibers are held by gum in bundles with different fiber numbers and sizes.

4. CONCLUSION

The optimum extraction conditions were found to be the extreme parameters of the extraction process with 30g/L soda concentration and 120°C for 4 hours. At these conditions, experiments results show an extraction yield of 39,68%, a tenacity of 9,42cN/tex, a linear density of 29,65tex, a diameter of 208,12µm and a lignin ratio of 22,05% and the experiment results were in good agreement with the predicted value. The Typha stem fibers obtained quality must be compared to Typha Leaf fibers to know about the variability in term of fiber quality and exraction process. Obtained fibers must be more characterized by

DRX, IR ... in order to identify the fine structure and its relation in the obtained properties, and the appropriate use of the fibers.

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