

## Optimization of extraction process of Typha leaf fibres

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The influence of temperature, duration and soda (NaOH) concentration on the extraction yield, linear density, diameter, tenacity and lignin ratio of Typha leaf fibres has been studied. A factorial design of experience has been used to identify the optimum operating conditions, and equations relating to the dependent variables to the operational variables of the extraction process are established. The optimum extraction condition has been determined by the statistical study using response surface and desirability function. The morphology of the obtained fibres and chemical constituents are determined. Fibres, extracted from leaves of Typha with the optimum process, have a lignin content value of about 14% like jute, alpha-cellulose value of about 67% similar to pineapple and jute fibres, extractives content value of about 1%, starches content value of about 2% and ash content value of about 1%. Finally, the characteristics of the optimum Typha fibre are compared with those of other vegetable fibres, showing large diameter and low mechanical properties as compared to other vegetable fibres.

**Keywords:** Chemical properties, Factorial design, Fibre extraction, Physical properties, Typha fibres

### 1 Introduction

Typha, is a monocotyledonous plant belonging to the family Typhaceae, with about twelve different species distributed throughout the tropical and temperate regions of the world in marshes and wetlands. Leaves are thick, having a spongy section due to the presence of the air channels<sup>1</sup>. Because its leaf fibre is similar to that of hemp or jute, it can be used in the textile industry for the same purposes<sup>2</sup>. Researches show that Typha stands may be used to clear sewage and industrial wastes<sup>1</sup>. Some others have tried to extract and develop Typha fibres as a natural fibre for composite reinforcement<sup>3</sup> and paper making<sup>4</sup>. In a previous study<sup>5</sup>, we tried to study the extraction of fibres from Typha leaves and the effect of extraction parameters on the properties of the obtained fibres. Results show that the fibres extraction conditions seem to have an important role on the fibre properties. In order to have fibres with good quality, moderate conditions may be required. Otherwise, degradation of cellulose takes a place which gives weak fibres.

In this study, we attempted to optimize the Typha fibres extraction process. Fibre quality has been evaluated in terms of extraction yield, fibre diameter,

linear density, tenacity and lignin ratio, and the optimal extraction conditions are determined. This study is completed by a characterization of the fibres extracted under the optimal condition, chemical composition and morphological properties. The obtained characteristics are then compared to other vegetable fibres to conclude on the importance of its textile potential.

### 2 Materials and Methods

Typha fibres were extracted using the procedure as reported earlier<sup>5</sup>. Experimental design and characterisation of extracted fibres are discussed here under.

#### 2.1 Experimental Design and Desirability Function

For the fibre extraction, we used the Box–Behnken design of experience 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 the optimization of several chemical and physical processes. All statistical analyses have been carried out using the statistical software Minitab 14<sup>6-9</sup>. An experimental database has been elaborated by varying the Typha extraction parameters. In this database (15 tests), input variables were taken as temperature ( $t$ ), extraction time ( $d$ ), and soda concentration ( $C$ ). The outputs are the extraction yield

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(*Y*), diameter (*D*), linear density (*LD*), tenacity (*T*) and lignin ratio (*L*). The independent variables were normalized to values from -1 to +1 in order to make an easy direct comparison of the coefficients of the resulting polynomial equation and an understanding of the effects of individual independent variable on the dependent variables considered (yield, linear density, diameter, fibre tenacity and lignin ratio). Experimental data were fitted to the following second-order polynomial equation:

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

where  $Y_i$  is the response of dependent variable [yield ( $Y_Y$ ), diameter ( $Y_D$ ), linear density ( $Y_{LD}$ ), 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 the unknown characteristic constants estimated from the experimental data.

Therefore, this experimental design would be used to optimize the extraction conditions of Typha fibres, where it is necessary to achieve the right combination of parameters ( $C$ ,  $T$ , and  $d$ ) as shown below:

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

Among the optimization tools that can be used, there is the desirability function. In this study, we used two types of desirability functions ( $d_i$ ) to maximize and minimize (Fig. 1). 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

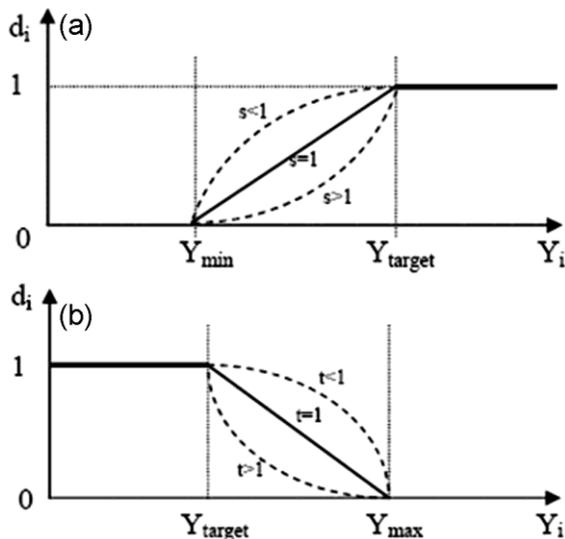


Fig. 1— Desirability function to (a) maximize and (b) minimize

the Derringer and Suich desirability function<sup>10</sup>, as defined below:

$$d_g = \sqrt[w]{(dD^{wD} \times dLD^{wLD} \times dL^{wL} \times dY^{wY} \times dT^{wT})} \dots (2)$$

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

The compromise between the properties was better when  $d_g$  is 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 has a value outside of tolerance, the function  $d_g$  was equal to 0 and the compromise was rejected<sup>11</sup>. So, the optimum combination of  $C$ ,  $t$  and  $d$  is the one that gives  $d_g$  close to 1.

Thus, to maximize a property  $Y_i$ , the desirability function [(Fig. 1(a)] has to be used, where the individual desirability ( $d_i$ ) was calculated as follows:

$$d_i = \begin{cases} 0 & \text{if } y_i \leq y_{i,\min} \\ \left[ \frac{y_i - y_{i,\min}}{y_{i,\max} - y_{i,\min}} \right]^{r_i} & \text{if } y_{i,\min} < y_i < y_{i,\max} \\ 1 & \text{if } y_i \geq y_{i,\max} \end{cases} \dots (3)$$

To minimize a property  $Y_i$ , the desirability function [(Fig. 1(b)] has to be used, where the individual desirability ( $d_i$ ) was calculated as follows:

$$d_i = \begin{cases} 1 & \text{if } y_i \leq y_{i,\min} \\ \left[ \frac{y_i - y_{i,\max}}{y_{i,\min} - y_{i,\max}} \right]^{r_i} & \text{if } y_{i,\min} < y_i < y_{i,\max} \\ 0 & \text{if } y_i \geq y_{i,\max} \end{cases} \dots (4)$$

**2.2 Characterization of Extracted Fibres**

The use of optimization procedures to improve the process conditions and to obtain a product with certain characteristics is essential when a low production costs and low use of energy are desired. A search for significant effects on quality parameters is demanding<sup>12</sup>. The properties of the Typha leaf fibres are determined by the physical, mechanical and chemical properties of the morphological constituents and their interfaces.

**2.2.1 Chemical Composition**

The chemical composition of natural fibres varies depending upon the type of fibre. Primarily, cellulosic

fibres contain cellulose, hemicelluloses, pectin and lignin. The properties of each constituent contribute to the overall properties of the fiber<sup>13</sup>. Lignin plays the role of binding the fibres of cellulose. Alkaline treatment is used for the release of fibres 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 fibres 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<sup>14</sup>. In order to optimize the fibre extraction process, ratio of Lignin was determined according to ASTM standard Test method D 1106 – 96 (2001).

Besides, chemical constituents of obtained fibres under optimum conditions, such as alpha-cellulose, lignin, starches, extractives and ash content were determined following TAPPI and ASTM standard methods such as 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.

### 2.2.2 Yield Measurement

Natural fibre 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 fibres obtained<sup>14, 15</sup>. So, yield of fibres (Y %) seems to be an important factor in the optimization of fibre extraction parameters. It is measured using the procedure as reported earlier<sup>5</sup>.

### 2.2.3 Fineness Measurement

Fineness in textile is one of the most important characteristics that affects application and quality of the final products. Therefore, it is important to determine the fineness of natural fibres since it's considered an important factor to define their cost and quality. With natural materials the fibre diameter does not have a single value but it has a fairly wide distribution of sizes even in material of one type, for example wool from a single sheep<sup>16</sup>. The fineness of Typha leaf fibres was given by measuring the diameter and the linear density. They were measured using the procedure as reported earlier<sup>5</sup>.

### 2.2.4 Strength at Break

The quality of jute or any textile fibre largely depends on its two important properties, namely fineness and

strength. The tensile tests of the fibres were performed using the procedure as reported earlier<sup>5</sup>.

## 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 Eq. (1). The coefficient of variation for each dependent variable was calculated and the results are found between 15% and 34%; they are considered acceptable. In fact, for sensory that uses consumers as panelists (subjective by nature), the coefficient of variation may reach high as 40% value and it can be still considered acceptable. Unlike synthetic fibres, the coefficient variance of natural cellulosic fibres properties is high (CV%~30-45)<sup>17</sup>. In fact, natural fibres have little resistance towards environmental influences and show an intrinsic variability of their properties. Hence, the use of natural fibres depends on the environmental conditions which are likely to influence ageing and degradation effects.

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<sup>2</sup> value and the p-value at a 95% confidence limit are shown in Table 2. The equations are subjected to non-linear programming using Minitab software in order to determine the optimum values of the dependent variables.

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 is determined by the p-value. The smaller the p-value, the more significant is the corresponding coefficient. The results indicate that the model is significant ( $p < 0.05$ ). As shown in Table 2, the R<sup>2</sup> of each second-order polynomial regression is 95.9, 96.8, 73.9, 74.6 and 94.6 for  $Y_{LD}$ ,  $Y_D$ ,  $Y_Y$ ,  $Y_T$  and  $Y_L$  respectively. The results also

Table 1—Statistics of measured properties

Property	Mean	Minimum	Maximum	Coefficient of variation %
Linear lensity, tex	21.51	10.02	29.56	23.33
Diameter, $\mu\text{m}$	307.84	141.3	495.46	33.98
Yield, %	36.02	25.12	46.65	15.32
Tenacity, cN/tex	10.11	7.23	15.11	23.63
Lignin, %	17.53	11.83	25.25	20.70

indicate that the model used to fit the response variables is 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<sup>18,19</sup>. It is based on the idea that the ‘quality’ of a product or process has multiple quality characteristics<sup>20</sup>. The desirability approach, proposed initially by Derringer and Suich (1980), seems very promising for optimizing simultaneous response variables, besides being easily performed<sup>20</sup>.

The aim is to convert each response into an individual desirability. The optimum values and corresponding normalized values for the independent variables are shown in Table 3. The optimum levels

of the dependent variables for the fibre extraction entailed using a low to high soda concentration, duration and temperature have been used, considering the following:

- (i) Good values for the diameter and linear density properties are obtained using a high duration and temperature (1) and medium to high soda concentration (0.571 - 1).
- (ii) Optimum yield is obtained using low soda concentration and duration, and medium to high temperature.
- (iii) Good values for fibre tenacity is obtained using high soda concentration and duration (1), and a low to medium temperature.
- (iv) Efficient use of the raw material (i.e. obtaining a low lignin ratio) is achieved using medium to high values of the three independent variables.

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

Coefficients and statistical parameters	Linear density tex (LD)	Diameter µm (D)	Yield % (Y)	Tenacity cN/tex (T)	Lignin % (L)
a0	21.563	292.373	38.240	11.860	13.906
a1	-1.723	-40.610	-1.546	1.023	-1.750
a2	-1.347	-29.988	-1.3437	0.043	-0.810
a3	-5.833	-124.750	4.505	-0.125	-2.342
a11	1.842	20.358	0.451	0.177	1.492
a22	-1.175	-9.817	0.876	0.222	0.337
a33	-0.767	18.453	-5.491	-3.675	4.957
a12	-0.465	0.955	NS	NS	-0.002
a13	0.087	-1.785	NS	NS	0.997
a23	-1.270	6.115	NS	NS	0.592
R <sup>2</sup>	95.9	96.8	73.9	74.6	94.6
p-value	0.006	0.003	0.044	0.04	0.011

NS – Non significant value.

Table 3 — Optimum value of properties

Property	Optimum value	X <sub>C</sub>	X <sub>d</sub>	X <sub>t</sub>
Linear density, tex	10.57	0.571	1	1
Diameter, µm	131.306	1	1	1
Yield, %	46.170	-1	-1	0.784
Tenacity, cN/tex	13.43	1	1	-0.14
Lignin, %	12.915	0.344	0.983	0.178

X<sub>C</sub>, X<sub>d</sub>, X<sub>t</sub>, correspond to the standard values of C, d and t respectively.

The operational conditions for the dependent variables (Table 3) were used to calculate the predicted values for the properties of extracted fibre and the deviation from the optimum levels (Table 4).

As we can see from Table 4, good quality and resistant Typha leaf fibres, are obtained using high duration (4h) and soda concentration (30g/L) and low to medium temperature (°C). With these conditions, the values of the extracted fibres (yield, tenacity and lignin) are departed by only 0 - 20% from their optimum levels. However, the deviation of the diameter and linear density obtained is found too high (90 % and 86% respectively), which varies widely with the extraction parameters. In fact, natural fibres are known to vary in their diameter and cross sectional area along the fibre length. Study conducted by Hashim *et al.*<sup>21</sup> shows that kenaf fibre mean diameter is reduced by 30.12- 42.92% after alkali treatment. They mention that alkali concentration has a higher impact on diameter change, as compared to temperature of alkali treatment. In another paper, they showed that kenaf fibre bundle mean cross-sectional area is reduced from 6.77% to 29.88% after alkali treatment as compared to kenaf mean cross-sectional area. The decrease is due to

Table 4 — Values of dependent variables for the fibre obtained under the stated conditions

Parameter	LD deviation, %	D deviation, %	Y deviation, %	T deviation, %	L deviation, %
High temp., duration & medium-to-high soda conc.	10.56-0	135.36-3.09	32.6-29.4	8.1 -39.67	16.7-29.35
High temp., duration & soda conc.	10.9-3.12	131.3- 0	31.39-32.01	8.44-37.15	17.38-34.62
Low duration, soda conc. & medium-to-high temp.	20.71-95.93	284.61-116.76	46.17-0	9.7-27.8	18.26-41.44
High duration, soda conc. & low-to-medium temp.	19.66-86	250.5-90.78	36.66-20.6	13.43-0	13.38-3.64
Medium-to-high temp., duration & soda conc.	17.29-63.57	221.51-68.7	37.42-18.95	12.22-9	12.91-0

swelling reaction during alkali treatment process at different conditions which affect the fibre structure, dimension and morphology<sup>22</sup>.

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 are implemented to maximize fibre  $T$  and  $Y$  and to minimize  $D$ ,  $LD$  and  $L$ . In this criterion, the goal is 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 ( $Y_i$ ) into a unitless utility bounded by  $0 < d_i < 1$ . A higher  $d_i$  value indicates that the response value  $Y_i$  is more desirable, with  $d_i = 0$  signifying a completely undesired response<sup>23</sup>. So, for every experience, we get an individual desirability function for each property that allowed us to know if the property is satisfying or not. For every extraction, we also achieved a global desirability function by affecting the relative weight " $w_i$ " 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 fibres studied such as a diameter of the order of 292.37 microns, a linear density of the order of 21.56 tex, a lignin ratio of about 13.9 %, a tenacity of 11.86 cN / tex and a yield of about 38.24%, with a desirable values of all individual desirability functions.

These optimal characteristics are achieved only under optimal conditions of temperature (100°C), duration (2h) and soda concentration (20g/L). The global desirability obtained is found to be equal to 99.86% which is very significant. The statistical study shows the optimum extraction conditions that minimize the wasting energy, time and soda concentration. Therefore, the extraction cost would be

reduced. So, optimum values obtained are found to be the central point of the box-behnen design.

### 3.2 Characterization of Optimum Fibre

In accordance with the optimization results obtained from response surface methodology with desirability function, verification experiments have been carried out at the selected conditions of  $T$  (100°C),  $d$  (2h) and  $C$  (20g/L). Indeed, all experimental results of fibre characteristics in the optimal conditions (1) and those given by the desirability function (2) are shown in Table 5.

From Table 5, it is found that the experimental results (1) are in good agreement with the statistical conditions (2) as proposed and the sample is validated. Thus, it can be seen that the second-order model is adequate to determine the optimum values of the dependent variables.

Besides, as compared to others vegetable fibres, alkali- treated Typha fibres are less resistant. The observed variation in mechanical properties of these fibres can be explained in terms of structural variables such as 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 fibres from the same place.

#### 3.2.1 Chemical Composition and Morphological Characterization

Table 6 shows that Typha leaf fibre has a chemical composition similar to other vegetable fibres with a 67.3% of alpha cellulose like jute and pineapple leaf fibres and 13.65% of lignin comparable to jute fibres but more than cotton, ramie and pineapple leaf fibres.

Figure 2 represents longitudinal views of the studied Typha fibres. Their structure is similar to a natural composite composed of ultimate fibre bundles of cellulose, thus forming the fibrous reinforcement, linked together by gummy and waxy substances, constituting the matrix, as also shown by other researcher<sup>17</sup>. After soda treatment, SEM micrographics show modification in surface morphology. In addition, the following reaction takes place as a result of alkali treatment<sup>24-26</sup>:

Table 5 — Comparison of extracted typha fibre with experimental (1) and statistical (2) method

Property	1	2	Nettle <sup>30</sup>	Kenaf(10%alkali treated) <sup>6</sup>	Jute <sup>31</sup>	Cotton <sup>31</sup>	Iranian Typha <sup>32</sup>
Tenacity, cN/tex	12.41	11.86	59.3	40	31	32	18.32- 40.95
Yield, %	37.82	38.24	-	-	-	-	-
Linear density, tex	22.47	21.56	-	4.5	-	-	3.92- 7.3
Diameter, $\mu\text{m}$	303.86	292.37	30	50- 80	47	12- 25	-
Lignin, %	13.65	13.90	-	-	-	-	-

Table 6 — Comparative chemical composition of Typha leaf fibre in per cent<sup>33</sup>

Parameter	Typha leaf fibre	Pinneapple leaf fibre	Capsularis jute	Ramie	Cotton
Solubility in hot water (starches)	2.06	-	-	-	-
Amount of extractives	1.24	3.3	0.9	0.3	0
Lignin	13.65	4.4	13.2	0.5	0
Alpha-cellulose	67.3	69.5	61	86.9	94
Ash	1.07	0.9	0.5	1.1	1.2

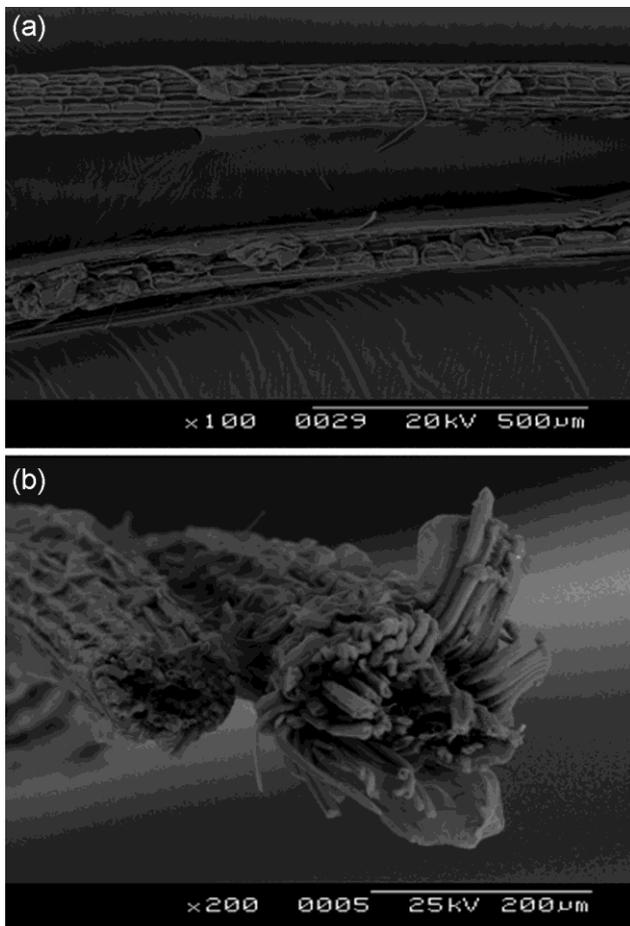
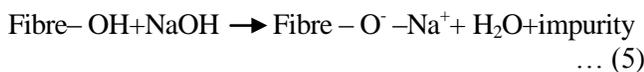


Fig. 2—SEM of Typha leaf fibre treated at the optimum conditions (a) longitudinal and (b) cross- sectional views



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<sup>27</sup>. After alkali treatment, the crystallinity of fibres increases<sup>28</sup>, which might be attributed to the removal of the cementing materials, leading to a better packing of cellulose chains.

Additionally, treatment of the fibres with NaOH might lead to a decrease in the spiral angle and increase in molecule orientation. Then soda treatment cleans the fibre surface of a large amount of impurities (gummy and waxy substances). This figure shows that the chemical process of extraction using sodium hydroxide allows the separation of ultimate fibres.

It can also be seen that a number of lumpy strips exists on the surfaces of the treated fibers (Fig. 2), which might be due to mercerization of NaOH resulting in partial removal of wax or fatty substances. It is a well-known fact<sup>29</sup> that there are binder lignin and fatty substances which hold the unit cells firmly in a fiber as reported in the case of jute fibre<sup>30,31</sup>.

The cross-sectional investigation of Typha fibres shows initially intact bundles (Fig. 2). It seems that some materials are still attached to several bundles and form a relatively large fragment. This finding shows that the obtained fibre is multicellular in nature just like jute and pineapple leaf fibres. Thus, we conclude that Typha fibres are held by gum in bundles with different fibre numbers and sizes.

#### 4 Conclusion

The optimum extraction conditions are found to be the central point of the box-behken design with medium soda concentration, duration and temperature (time 3h; temperature 100°C; soda concentration 20g/L). At these conditions, experimental results show an extraction yield of 37.82%, a tenacity of 12.41 cN/tex, a linear density of 22.47 tex, a diameter of 303.86 µm and a lignin ratio of 13.65%. The experimental results are found in good agreement with the predicted value. The Typha leaf fibres obtained must be more characterized in the future using DRX, DMA, DTA/TGA... in order to identify its appropriate uses like paper products, nonwoven, geotextiles, and composites.

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