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RESEARCH ARTICLE

PHYSICAL AND MORPHOLOGICAL CHARACTERISATION OF TYPHA AUSTRALIS FIBRES

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Abstract

Typha australis is an invasive plant from Senegal that is also found in wetlands. In order to limit its proliferation, Typha australis is used in the energy sector but also in construction. This study aims to contribute to the valorisation of Typha australis in the field of biocomposites. For this purpose, Typha fibres were used. The fibres were extracted manually using a plastic comb, then subdivided into three zones, namely the bottom, middle and top of the plant, and dried. The obtained fibres, were characterized physically by measuring density, water content and absorption coefficient, and morphologically by determining the morphology of the section of the fibres.In many ways, the physical property results concerning density showed an average of the three areas of 1.53 g/cm^3 with a water content between 6 and 10% and an absorption coefficient showing two main absorption phases. Concerning the tomography results, we directly noticed the presence of fibre bundles and not of individual fibres due to the manual extraction method used. In conclusion, the obtained results confirma behaviour comparable to those of the most commonly used natural fibres, namely flax. Typha australis has a real potential as reinforcement in biocomposites.

Introduction:-

The use of plant fibres in the development of composite materials has increased in recent years due to their mechanical properties, good thermal insulation properties, low density, non-abrasive nature, easy availability from renewable sources, lower price and recyclability[1]which make them competitive with manufactured synthetic and natural fibres. The target sectors are automotive[2], aerospace and construction [3], [4], for semi-structural applications. Fibres can be extracted from Typha australis leaves, a highly prolific plant found in the wetlands of Senegal [5]that can grow up to 1 to 3 meters long with spongy, strap-like leaves[6], for processing into composite reinforcements.

The present study is a contribution to the valorisation of this plant, more precisely of the fibres obtained.

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A previous study on Typha australis fibres from a physical point of view by determining the density and water content was carried out[7]. The obtained results show a similarity to the properties observed on flax fibres.

The main objective is to study the physical and morphological characteristics of Typha fibres for a relevant comparison with other natural fibres. This will allow a better knowledge of the potential of Typha fibres in order to position them in the family of the most used natural fibres.

This paper will address the part relating to the obtaining of Typha fibres, as well as the determination of their physical properties involving the determination of the absolute density, the water content and the water absorption; and morphological consisting in identifying the morphology of the fibres following the extraction method used and at the same time determining the diameter of the Typha fibres.

Materials and Methods:-

Extraction of Typha australis fibres

Typha australis is harvested according to the NS 02 - 061 Senegalese cutting standard and subdivided into three zones: low, middle, high [8] as shown in Figure 1 [7].

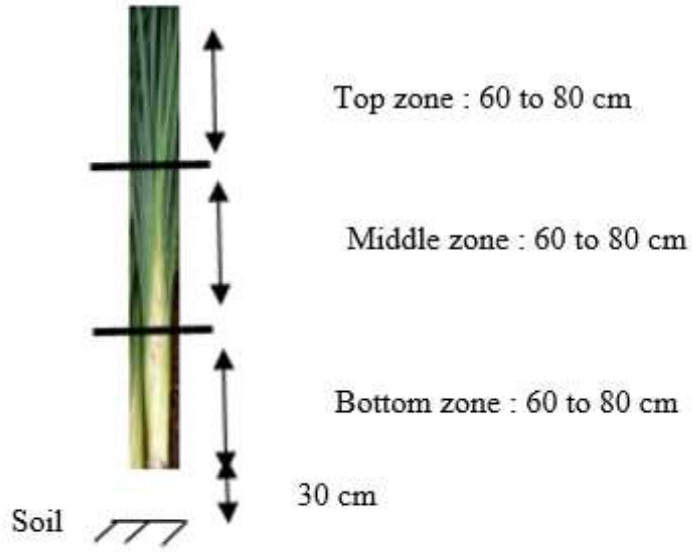

Fig. 1:- Method of subdividing Typha leaves [7].

The extraction of the fibres, shown in Figure 2, is done manually with a comb [9] with a tooth spacing of 1 mm. The resulting fibres are coarse and undamaged [7].

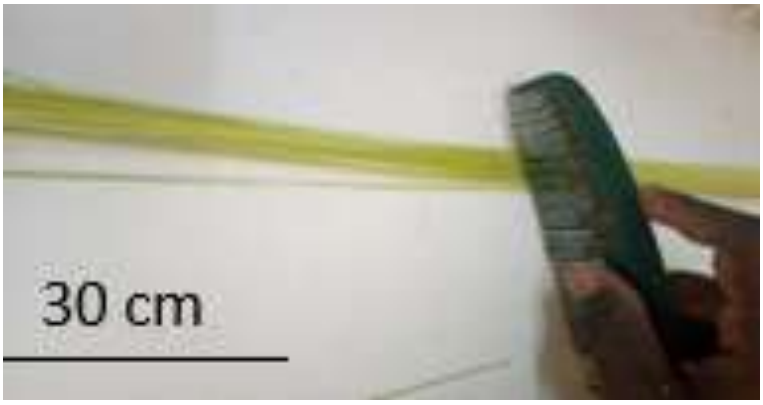

Fig. 2:- Combing.

Density

The tests were performed using a Micromeritics AccuPyc II 1340 helium pycnometer. The measurements, repeated ten times, are performed by applying a pressure of 19.5 bars with a stabilisation criterion of 0.0065 g/min. A sample holder of 10 cm^3 is used. The absolute fibre density (ρ) is given by Equation 1, where V represents the volume occupied by the fibre sample and m the fibre mass tested [7].

$$\rho = \frac{m}{V} \quad (1)$$

Water content

The water content (W) is determined according to the French standard NF EN ISO 665 [10]. It corresponds to the loss of mass that the sample undergoes when it is dried in an oven at 105 °C until it reaches a constant mass. According to the NF EN ISO 665 standard, the determination of the mass of cotton seeds begins after three hours of drying. On this basis, the drying times of the Typha samples were 120, 150, 180, 210, and 240 minutes. The tests were repeated three times [7].

Absorption coefficient

The purpose of this test is to quantify the mass of saturated water seeping into the pores of the fibres when they are immersed in water. Similarlyto the determination of the water content measurement carried out previously, the fibres are first dehydrated at 105°C for 4 hours in a ventilated oven. They were then immersed in distilled water for 5min, 15min, 30min, 60min, 2h, 4h, 24h and 48h ([11], [12]and[13]). Before weighing the immersed fibres to obtain their wet weight, they are first placed on a layer of absorbent paper for 10 min to remove surface water, as shown in Figure 3. This method was applied by Sibiath [11], but for only 5 min.

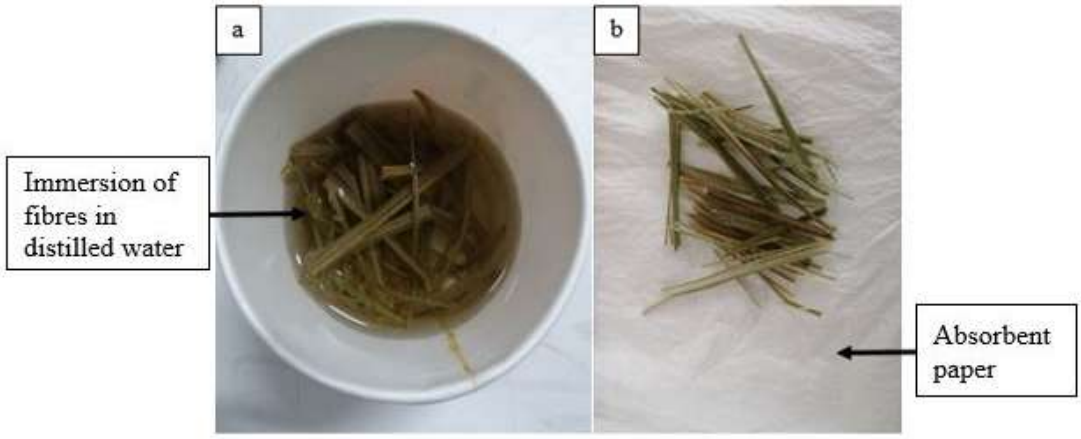

Fig. 3:- Steps of the water absorption measurement: a) Immersion of the fibres and b) deposition on absorbent paper before weighing.

The water absorption rate (Wabs) expressed as a percentage is the difference between the humid mass (mh) and the dry mass (ms) of the sample, related to the latter (Equation 2).

$$Wabs\ (\%) = \frac{m_h - m_s}{m_s} x100 \quad (2).$$

Morphology

The morphological study involves the use of the EasyTom XL micro tomograph (Figure 4). It has a measuring chamber of the order of one cubic metre, allowing the analysis of rather large volumes. The tomograph is capable of achieving a maximum resolution of 4 μm^3. It can reach a voltage of 150 kV and the power of its X-ray source is 75 W. It is important to note that among these parameters the focal length, which is the concentration level of the X-ray beam, is comparable to the magnification of a microscope.

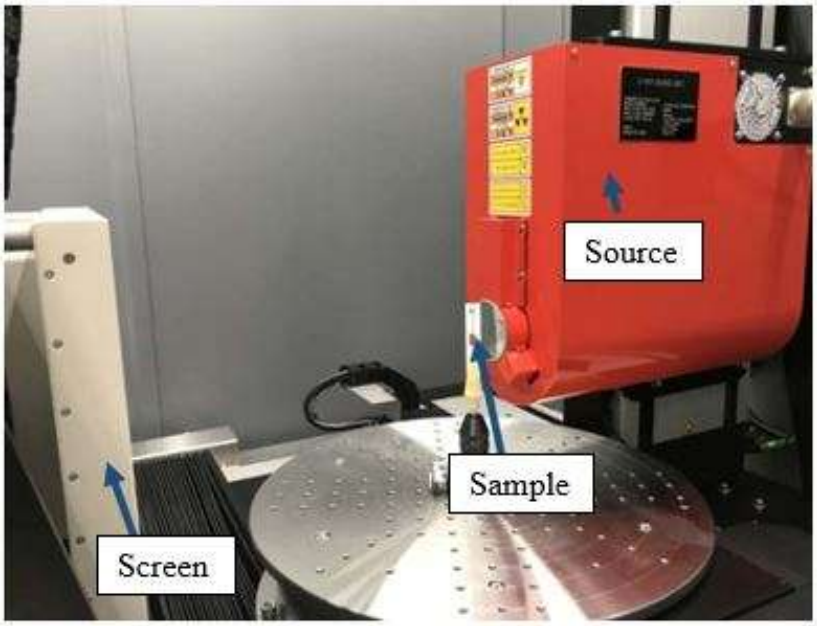

Fig. 4:- EasyTom XL micro tomography with sample holder, source and control screen.

To ensure that the fibre is straight when measured, the well-stretched fibre is laid out along the axis parallel to the length of a paper frame in the middle of the window (Figure 5). The fibre is then glued to the paper frame at its ends. The fibre-paper frame assembly is placed on the sample holder.

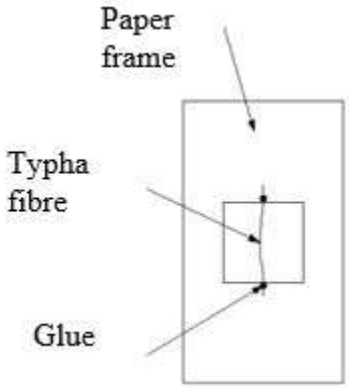

Fig. 5:- Paper support for the fibre to be analysed by tomography.

The parameters chosen during the tests were:

1. A small focal length for a more accurate analysis
2. A voltage of 150 kV
3. A current of 66 µA
4. A number of six images per second
5. One image is obtained from the average of 5 base images to improve quality
6. 1440 images are taken for each fibre sample tested, to have good accuracy in 3D reconstruction.

However, care must be taken not to overexpose the sample by automatically rotating it.

Thus we will be able to determine not only the cross-section at any point of the observed fibre, but also its morphology after reconstruction of the volume of the latter. Other software will be used to process the images for better accuracy, such as Image J [14]to process and determine the section of the fibre and ABViewer ([15], [16]) for the morphological study of the fibres.

Results:-

Density

Our previous studies yielded the results given in Table 1. The average absolute densities of Typha fibres are compared to those of other natural fibres is given.

Table 1:- Average density of Typha fibre samples with that of common plant fibres [7].

Fibres	Average density g/cm^3 (standard deviation)	References
Bottom Typha	1.605 (0.026)	[7]
Middle Typha	1.519 (0.016)	[7]
Top Typha	1.478 (0.023)	[7]
Sisal	1.450	[9]
Flax	1.540	[17]
Hemp	1.470	[17]
Cotton	1.550	[18]
Pineapple	1.440	[19]

It has been explained in our previous studies [7] that the different values obtained from the different fibres were also observed on flax fibres[8]. They explained that the bottom and top fibres have a higher porosity than the middle ones. In addition to that, the bottom ones are in direct contact with the soil hence exposed to harsh environmental conditions. However, the density of the middle fibre of Typha (1.519 g/cm^3) iscloser to that of flax.

The average obtained from the three Typha zones, which is 1.53 g/cm^3[7], is of the same order of magnitude as the density of flax and that of lignocellulosic fibres, which is about 1.53 g/cm^3[13].

Water Content

The results of water content are shown in Figure 6.

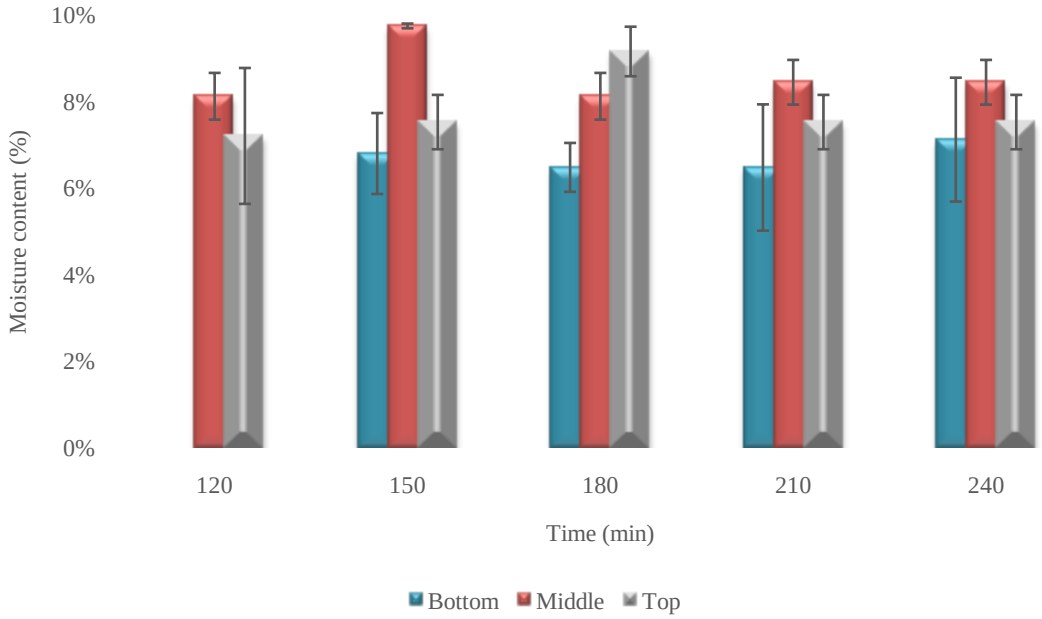

Fig. 6:- Wet-based moisture contents of Typha Australis fibres measured for different drying times [7].

The water content of our fibres ranges from 6 to 10% as shown in Figure 6 above[7]. In detail, the lowest contents are measured for the bottom fibres, despite their larger standard deviation. According to studies of Baley and al[20] and K. Charlet[21], water represents about 8-10% of the mass of flax fibre. These observations are verified for our middle fibre with an 8.44 ± 0.52% content beyond 3 hours of drying[7]. In addition, data available in works [22], [23], and [24] show that the mass moisture content of flax fibres stored under ambient conditions is generally between 6 and 10%, as shown in Figure 6 of our Typha Australis fibres.

Absorption coefficient

The results obtained for absorption coefficient are presented in Table 2. The measurements not carried out are due to the unavailability of the experimental set-up.

Table 2:- Water absorption of Typha fibres as a function of immersion time. Standard deviations are shown in brackets.

Time (min)	5	15	30	60	120	240	1440	2880
Bottom	273% (64)	362% (83)	399% (74)	456% (87)	507% (110)	562% (97)	646% (132)	646% (122)
Middle	208% (14)	250% (25)	300% (25)	308% (29)	325% (25)	375% (25)	442% (52)	442% (52)
Top	253% (26)	292% (38)	353% (50)	353% (26)	360% (30)	-	483% (14)	485% (13)

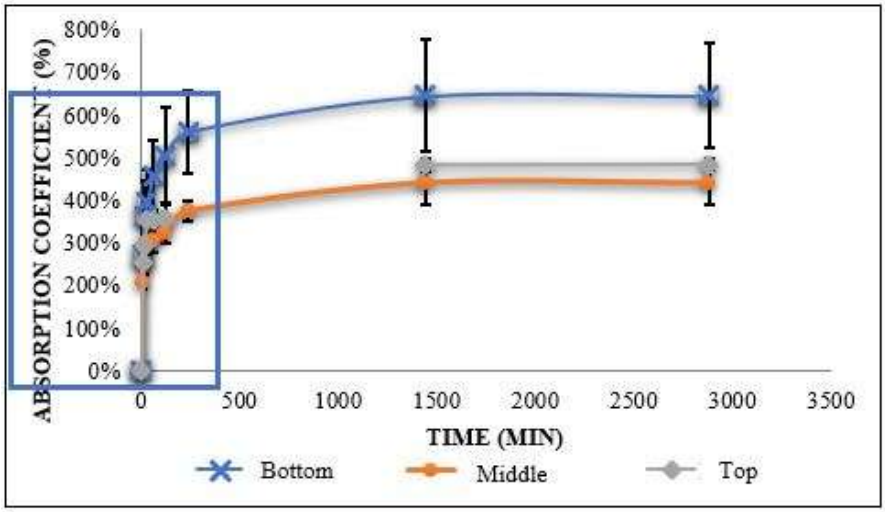

Fig. 7:- Evolution of the absorption coefficient of Typha Australis fibres as a function of the immersion time in a distilled water bath, for immersion times of up to 48 hours.

The measurements are plotted in Figure 7. The magnification of the measurements for durations of less than two hours is shown in Figure 8.

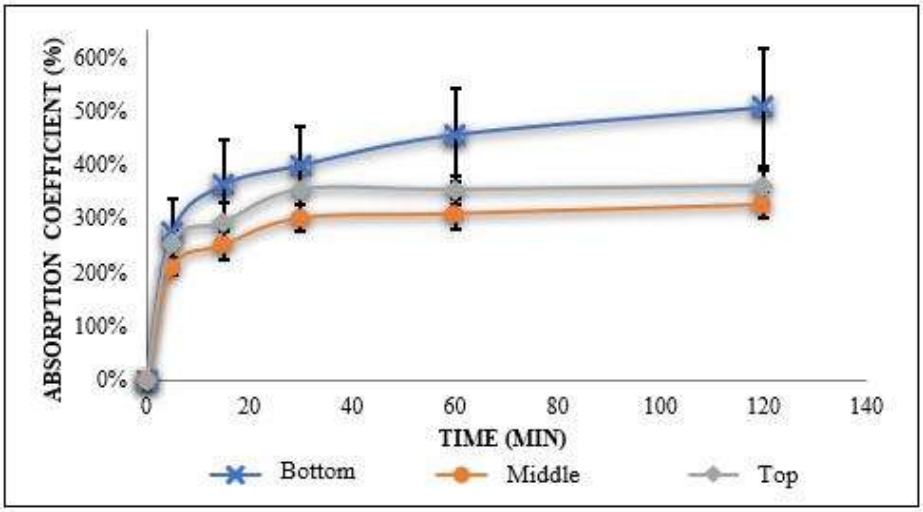

Fig. 8:- Evolution of the absorption coefficient of Typha Australis fibres for immersion times of less than 2 hours.

Figures 7 and 8 show an asymptotic behaviour ofTypha Australis fibres' water absorption. There are two absorption phases that are seen for all the fibresstudied. The primary corresponds to a fast increase in the mass of the immersed

fibres during the first half-hour with an increase of 32, 31 and 28% observed respectively for the bottom, middle and top fibres.Then, the increase in mass occurs more slowly and continuously, between 1 and 48 hours of immersion, for the second phase. We note that after 5 minutes, all the fibres have already absorbed more than 200% of their mass in water.After 24 hours of immersion, saturation of absorption is noted for all samples. At 48 hours of immersion, the bottom fibres show the highest water absorption, of about 600%, among all other fibres, with significant variability. This is followed by the top fibres, then the middle fibres with an absorption coefficient of 485 and 442% respectively. The better absorption and water retention capacity of the bottom fibres can be linked to the internal structure of these fibres, which have porosities, giving them a highly hydrophilic character. The presence of hemicelluloses, which are non-linear polymers that are very hydrophilic, is responsible for this water absorption. The absorption capacity of Typha fibres then depends on the location of the fibre.

The physical properties differ from one area to another. The middle fibres have properties most similar to lignocellulosic fibres, notably flax, with fairly equivalent values of density and moisture content.

When compared with commonly used plant reinforcements on the basis of density, water content and water absorption, the Typha fibres analysed do not appear to have any contraindications for use as reinforcements in fibre composites impregnated with a polymer matrix.

Tomography

As an illustration, images obtained after tomography and binarized images after processing with Image J are shown in Figure 9. The tomograph images (figures I.a, II.a, III.a and IV.a) show us the exact shape of the fibres in a grey background without giving the value of the cross-section. In image J (figures I.b, II.b, III.b and IV.b), these are binarized and we get black pixels for the background and white pixels for the fibre cross-section. Thus, the value of the cross-section is calculated by counting the number of white pixels.

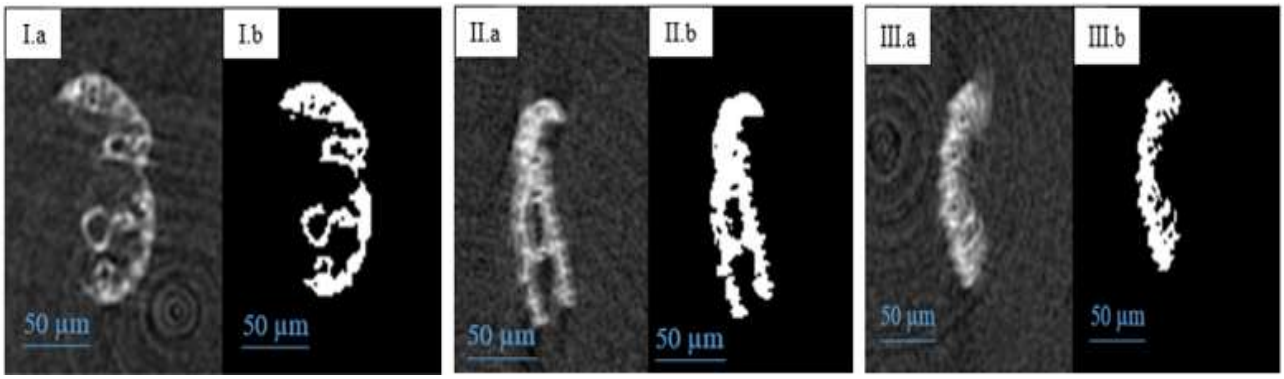

Fig.9:- Images of fibres I) bottom, II) top, III) middle and IV) treated with 3% NaOH for 2 hours, from the tomograph (index a) and binarized after processing on Image J (index b).

The fibre sections of subdivided Typha plants (bottom, middle and top) have a very irregular contour and cannot be associated by elementary geometric shapes (circular or polygonal). Some of the fibres appear to be individual, while others are more in the form of aggregates of fibres forming technical fibres. The large presence of fibre bundles, rather than individual fibres, is attributable to the combing of the harvested plants, which does not allow sufficient individualisation of the fibres. As a result, the measured cross-sections correspond to those of the technical fibres.

Table 3 shows the average cross-sectional values obtained for each batch of fibres, with an equivalent diameter (equation 3) in comparison to other plant fibres. The equivalent or average diameter is calculated with the assumption that the cross-sectional value obtained (S) corresponds to that of a disc of diameter Deq according to equation 3.

$$\mathrm{Deq} = \sqrt{\frac{4 \times S}{\pi}} \qquad (3).$$

For experimental reasons (lack of time and of usable fibres as the useful length must be twice the gauge length but also the fibres to be tested must be as homogeneous as possible) 22 fibres in the middle were chosen to determine their average cross-section whereas for the other fibres (bottom, top) the average cross-section is obtained on 6 fibres.

Table 3:- Average cross-sections and diameters of Typha Australis fibres compared to other plant reinforcements.

Fibres	Location	Cross-sections in mm^2(standard deviation)	SD (%)	Equivalent diameter in mm (standard deviation)	SD (%)	References
Typha Australis	Bottom	0.141 (0.068)	49	0.410 (0.115)	28	This study
	Middle	0.153 (0.083)	55	0.425 (0.121)	28	
	Top	0.126 (0.094)	75	0.376 (0.151)	41	
Flax	Bottom	-	-	0.0235 (0.0079)	-	[8]
	Middle	-	-	0.0213 (0.0063)	-	
	Top	-	-	0.0215 (0.0053)	-	
Hemp	-	-	-	0.088	-	[25]
Sisal	-	-	-	0.163	-	[25]
Banana	-	-	-	0.29	-	[11]
TunisianTyphatreated with 20g/l NaOH for 3h	-	-	-	0.3039 (0.0284)	-	[26]
UntreatedTunisianTypha	-			0.520	-	[27]

Our untreated and subdivided Typha Australis fibres from Senegal have larger diameters than the untreated and NaOH-treated Tunisian Typha fibres [26],and smaller diameters than for untreated Tunisian Typha[27]. Consequently, it is notable that among the effects of soda treatment is the reduction of fibre cross-section. In fact, this treatment removes some chemical constituents from the fibres, such as pectin, wax, lignin and some hemicellulose on the surface of the fibres as stated by Sumesh and al.[28]. However, the diameter of Typha Australis fibres is about 20 times the diameter of flax fibres. Charlet andal.[8]have shown that a flax fibre bundle has a diameter of between 0.1 and 0.3 mm whereas a single flax fibre is between 0.01 and 0.03 mm in diameter. So a bundle can contain up to about 30 unit fibres. Correlating this with our Typha Australis fibres; a fibre bundle with an equivalent diameter of between 0.2 and 0.4 mm can contain up to 40 Typhaaustralis unit fibres.This is valid if we consider that the Typha fibre has a diameter between 0.01 and 0.03 mm. However, the equivalent diameter will only make sense when we find the experimental means to individualise the fibres.

To observe the regularity of the cross-section along the fibre (20 mm gauge length), the 1400 tomographic cross-section images of a fibre sample were processed and the results of the variation are shown in Figure 10.

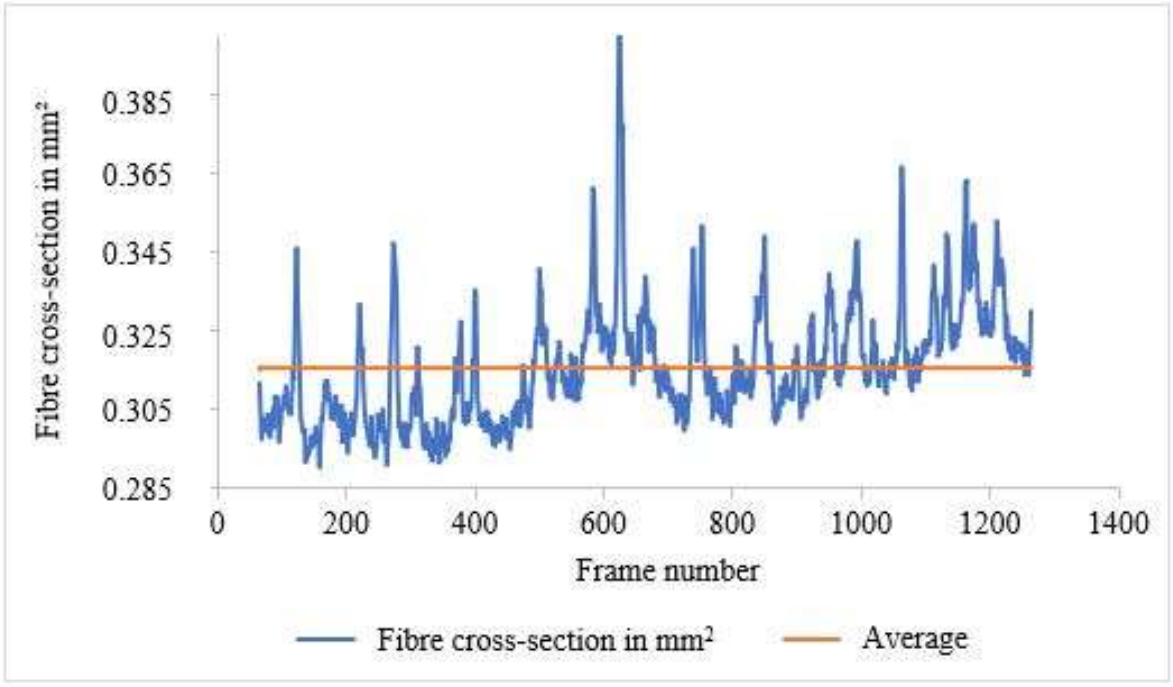

Fig.10:- Variation in cross-section along a medium fibre.

At first sight, we notice a non-constancy of the cross-section along the fibre. Although it is a straight section since it is perpendicular to the plane of the mean line of the fibre. An oscillation of the cross-section values is noted with a minimum of 0.29 mm^2 and a maximum of 0.40 mm^2. The calculated average cross-section (0.32 mm^2) shows a difference between the minimum and maximum cross-section of 0.03 and 0.08 mm^2 respectively. To the naked eye this variation in cross-section is very slightly noticeable along a fibre extracted with combing only.

Indeed, the use of a plastic comb tends to deform, with the resistance of the plant stem when combing.Thus, the gap between two successive teeth, which was initially 1mm, is likely to increase, even if the flexibility of the plastic teeth reduces tearing and favours separation into bundles.

The morphological aspect of the fibre is better identified by reconstructing the 1400 sections of the fibre obtained with the tomograph over a length of 20 mm. Figure 11.a shows section number 175 (corresponding to a height of about 3 mm from the glue point) of the Typha fibre bundle from the middle area, which will be used as an example of reconstruction.

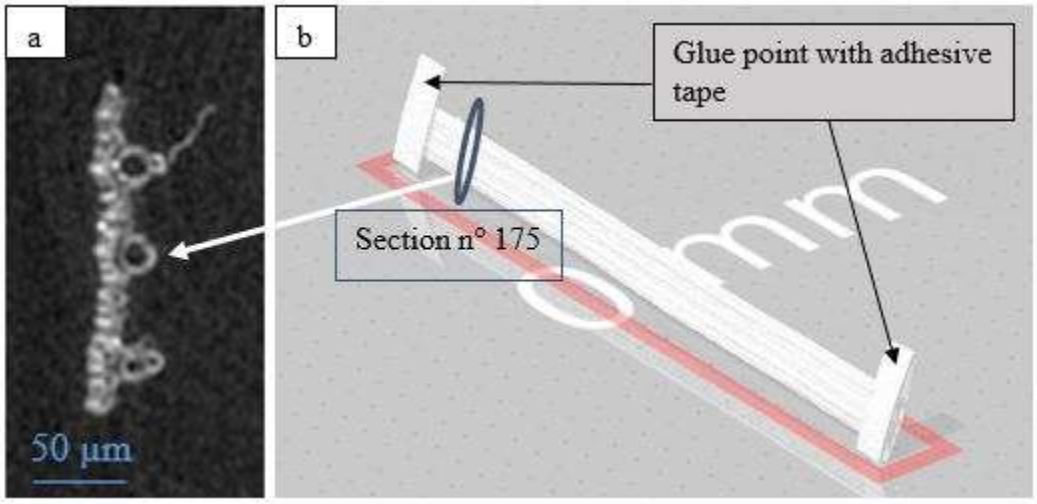

Fig.11:- Typha fibre bundle of the middle zone obtained by tomography: a) section, b) volume reconstruction.

A volume reconstruction of this fibre bundle is possible with tomography knowing the number of sections, the position of each section and its total length. Figure 11.b shows the obtained reconstruction. Using the ABViewer 14 software, the reconstructed fibre can be visualised as shown in Figure 12.

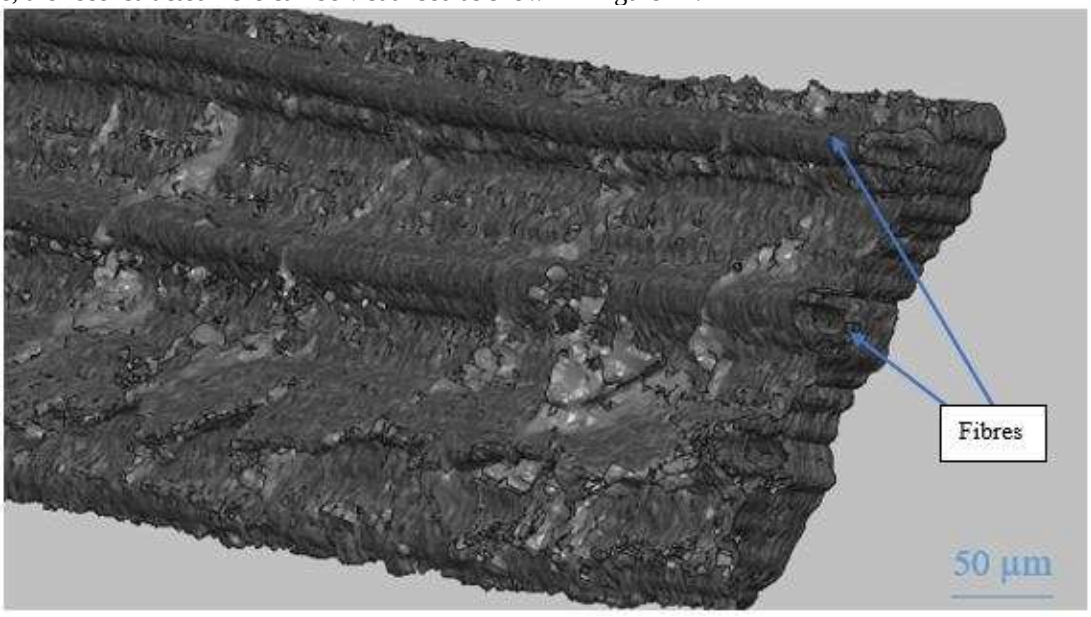

Fig.12:- 3D reconstruction of an untreated fibre from the middle area.

The 3D reconstruction does show the assembly of several fibres to form a bundle. However, it is difficult to give the number of fibres found. The almost flat shape of the structure can be attributed to combing. However, we note the presence of surface elements. It is explained that plant fibres are made up of hemicelluloses and celluloses fused together by a matrix which is either pectin or lignin[18]. This confirms the hypothesis of Sana andal [27]that chemical elements such as lignin, pectins and other non-cellulosic substances hold the fibres together to form a bundle that can contain, for example, flax between 10 and 30 unit fibres[8].

The morphological study makes it possible to observe the fibre and see what the naked eye is not able to perceive. It effectively shows the impact of the extraction method used on the final appearance of the fibres. It is clear then that a direct combing method allows to obtain bundles of fibres. Thus, to obtain better individualised, finer fibres, tending towards unitary fibres, it is important to optimise the extraction method, either by the chemical method for which the use of chemicals can be harmful to humans and the environment or mechanically, or by refining the combing by using adapted equipment.

Conclusion:-

Plant fibres have become an important part of the reinforcement of composite materials. Their knowledge in all aspects is becoming essential to meet the needs of composites.

More and more researchesare focused on the use of new local plant fibres to replace the most commonly used natural fibres such as flax, hemp, sisal, ...

Typha australis is one of the plants that is of increasing interest to researchers because of its diversity but also because of the problems it causes to the environment, especially in underdeveloped or developing countries. The example is taken from Senegal where this plant causes many problems in the public health and economic sectors. A use of the fibres of this plant could participate in the protection of the environment with the manufacture of biocomposites in sectors with high CO_2 consumption.

In order to validatethis potential, it is important to well know this material, that is why studies from a physical and morphological point of view have been carried out to determine the water content, the density, the absorption coefficient and the morphology.

The results obtained from a physical point of view show a certain resemblance to natural plant fibres, especially those of flax, taking into account the water content of between 6 and 10%, the average density of the three zones of 1.53 g/cm^3 and an absorption coefficient showing two main absorption phases, the first of which corresponds to a rapid increase in the mass of the fibres immersed during the first 30 minutes and the second of which shows a slower increase in mass in a continuous manner, between 1 and 48 hours of immersion. As for the morphology, it shows the important presence of bundles of fibres and not of individual fibres, which is attributable to the combing of the harvested plants, which does not allow sufficient individualisation of the fibres.

The determination of the cross-section is an essential introduction to the mechanical part as it allows the calculation of the stress at break and the Young's modulus following a tensile test for a confirmation of the potential use of this fibre as a mechanical reinforcement in a long fibre composite.

It is important to note that for the same Typhaaustralis sheet, the fibres differ from a physical point of view according to their location in the sheet.

The observed characteristics prove a possible use of these Typha australis fibres as reinforcements in composites. The next step is the elaboration of the biocomposites.

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