

Study of a Fibrous Annual Plant, *Luffa Cylindrica* for Paper Application Part I: Characterization of the Vegetal

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ABSTRACT: *This study is devoted to the morphological, chemical and physical characterization of raw material of the fruit of *Luffa Cylindrica*. By microscopic analysis, it can be observed that the fruit is composed of an assembly of dependent cords, where four parts are differentiated: external, internal wall, core and bond. Each cord consists of hollow and flexible cylindrical fibers. The external part is richest in cellulose (80%). The mean contents cellulose of the various parts appears higher than those of wood fibers, and the lower percentages lignin (10%). The crystallinity index of cords cellulose was characterized by x-rays diffractometry; the measured value (69%). The thermophysical analysis shows that various kinetic phases of the adsorbed water drainage process and heat capacities measured being close to those of wood celluloses.*

KEY WORDS: *Luffa Cylindrica, Morphological characterization, Crystallinity, Chemical and thermal analysis, Specific surface.*

INTRODUCTION

During the last years, various applications tentative of vegetal fibers were realized. Before undertaking the present study, we list many references dealing with the *luffa Cylindrica*, but none of them treated the paper mill [1-11]. *Luffa Cylindrica* is an annual herbaceous plant

from the cucurbitaceous family. It gives a fruit of cylindrical form slightly angular, right and bent, of a very variable size. Its structure has the form of the sponge type, made of various fibrous cords consolidated between them, this allow it early use in Japan between 1890 and

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Table 1. Principal references relating to the studies of *Luffa Cylindrica*.

Application(s)	References
Use as basic stamps for the chemical and biological immobilization and / or as support with fixed bed of biological culture or in chemical synthesis.	[8-10,20,21]
Use like support of discoloration of the reagents and / or thermal support	[15, 22]
Use as basic material for the insulation and the extraction of the chemical and biological compounds	[23-27]

1895 for manufacture of filters for steam engine and Diesel engine [12]. Japan became quickly a large producer of *Luffa Cylindrica* intended mainly for export towards the USA before the 2nd world war, *Luffa Cylindrica* imported by the USA was used to 60 % in the production of filters and to 40% in applications such as sponges of bath, and industrial [12, 13].

Some other studies characterized the *Luffa cylindrica* fibers for other technical applications such as reinforcing composite materials.

Tanobe et al. [14] prepared a data characterizing fibers extracted from Brazilian sponge gourd. It has been found that the physical properties of *Luffa cylindrica* fibers must be enhanced by chemical or physical modifications to make them more useful in composite [15].

Luffa cylindrica mainly composed of cellulose, hemicelluloses and lignin, is called as a lignocellulosic material.

Except the production of sponges, the principal applications are summarized in Table 1. In this study, these various approaches justify a morphological, chemical, physical and thermal through the study of this material, to be the subject of this article. The second article deals with to the paper application of fibers obtained by cooking the vegetal.

EXPERIMENTAL SECTION

Microscopic analysis

The microscopic observations were carried out at the French School of Paper mill and Graphic Industries: optical microscope on an apparatus OLYMPUS BH-2, software of image analysis OPIMAS, electron microscope on apparatus ABT-55. The insulation of fibers for observation in optical microscope is carried out by boiling the fibers in alkaline solution (NaOH, 0.5 M).

Chemical analysis

The elementary analyses were carried out by atomic absorption at the Central Laboratory of Analysis of the National Scientific Research Center, Vernaison and Paris.

The cellulose percentage was measured by Kürschner and Hoffnere method, corresponding to hydrolysis with the nitric acid in ethanolic medium.

The lignin percentage was measured by sulphuric hydrolysis, using the dimethylaniline as catalyst (method of Noll): in a becher, 5 mL of N, N dimethylaniline are added to 2 g of dry vegetal; this one was triturate during 5 min, then after addition of 25 mL of H₂SO₄ with 78% in weight, the trituration was continued during 10 min. The sample was then supplemented with 250 mL of distilled water addition, and carried with soft biling. The lignin that was separated in flocs which are recovered by filtration on crucible filtering of porosity 5 indexes. After the washing of the precipitate with hot distilled water, lignin is dried with 105°C and then it is weighed.

The hemicelluloses percentage was deduced by the index from furfural, corresponding to the NF T 12-008 standard.

The holocellulose corresponds to a delignification with sodium chlorite in acetic plug medium.

(pH 4.9) (NF B 51-104 standard).

Crystallinity index by x-rays diffractometry

Measurements were taken at the Higher National School of Electrochemistry and Electrometallurgy of Grenoble (ENSEEG) on a diffractometer Philips PW 1730, CuK α 1 anticathode: 1.5405Å, vertical goniometer (2 θ); the samples were beforehand crushed (mean granulometry) and the powders was deposited on the plastic carry-sample.

Thermophysical analyses

Analyses TGA, DTA and DSC were carried out on SETARAM TGA-DTA 92-12 and SETARAM DSC 92 apparatuses. The sampling is composed of 13.08 mg, under atmosphere of nitrogen. Measurements of heat-storage capacity (C_p) were calculated from the calorimetric signal resulting from analysis DSC; two tests were carried out with identical conditions: the first using

two blank cells, the second with the same cells and the same sample. The variation of the calorific signal between the two curves characterizes the heating capacity of the sample. This amplitude was directly converted into thermal power. The determination of C_p was direct for each temperature without use of a standard sample (sapphir).

Specific surfaces and hydrophilicity of fibers

The specific surface determined by adsorption of nitrogen to 77.3K was measured on Micromeritics apparatus 2100 D. The effectuated measurements are indicated on Fig. 7, where the quantity of nitrogen that is adsorbed at the temperature (T) was represented according to the equilibrium pressure (P) in the enclosure. The BET (Brunner, Emmer and Teller) theory gives the X-coordinate to which this quantity is equivalent to the saturation of the surface of the solid by full-course compact of adsorbate. The rather weak slopes of the isotherms at the end of the adsorption show that equation is well applied in the studied case.

Hydrodynamic specific surface was measured on a Pulmac apparatus according to the following protocol: a wet cake of paste (4 to 8 g of dry matter) was consisted of decantation and the pressure drop in laminar flow through the bed was measured for different thicknesses. The relation of Kozeny-Carman [13] was used to determine the hydrodynamic specific surface from the porous medium; the value of the Kozeny constant was taken equalizes to 5.55, the value generally applied to cellulose fiber.

WRV (Water Retention Value) corresponds to the water mass retained in material after centrifugation to 3000 G ($G = 9.81 \text{ m/s}^2$) during 15 min.

This water mass, reported to one gram, is expressed as a percentage:

$$\text{WRV}(\%) = \frac{m - m_s}{m_s} \times 100 \quad (2)$$

Where: m is the mass of the wet material after centrifugation and m_s is the mass of the dry material

The WRV is an index characterizing the degree of hydration of the fiber, by supposing that the swelling of fibers is not modified during the centrifugation: it makes it possible to differentiate the liquid extra - fiber (water or soda solution 0.5 M), retained by capillarity, and the liquid impregnating fibers.

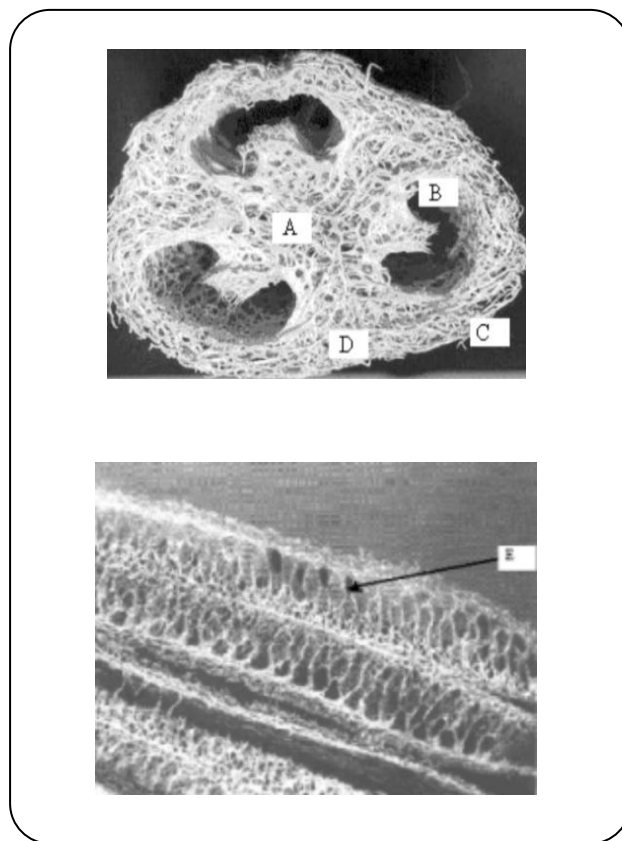


Fig. 1: Macroscopic observation of the *Luffa Cylindrica* fruit.

RESULTS AND DISCUSSION

Morphological characterization

The general aspect of the *Luffa Cylindrica* fruit can be observed on the Fig. 1a, 1b. From the inside towards outside, we distinguish the central part (called core or net), the internal and external walls, the bond between the core and walls.

The observation under the scanning electron microscope with weak enlargement of the different parts of *Luffa Cylindrica*, shows a texture made up of cords more or less consolidated between them, some being completely free (Fig. 2a-d).

The transverse section of the cords (fig. 3a) shows the fibrous organization of the structure. With stronger enlargement (Fig. 3b), it can be observed that the fibers are hollow and punctuated.

After boiling the fruit in medium alkaline, the elementary components of *Luffa Cylindrica* are dislocated out of fibers, vessels rings and punctuated spirals, sclerous in sticks, hairs in comma (Fig. 4a-c). The flexibility of fibers is revealed by their strong curve (Fig. 4a), and the average dimensions are easily visualized (Fig. 4b). By comparison with the

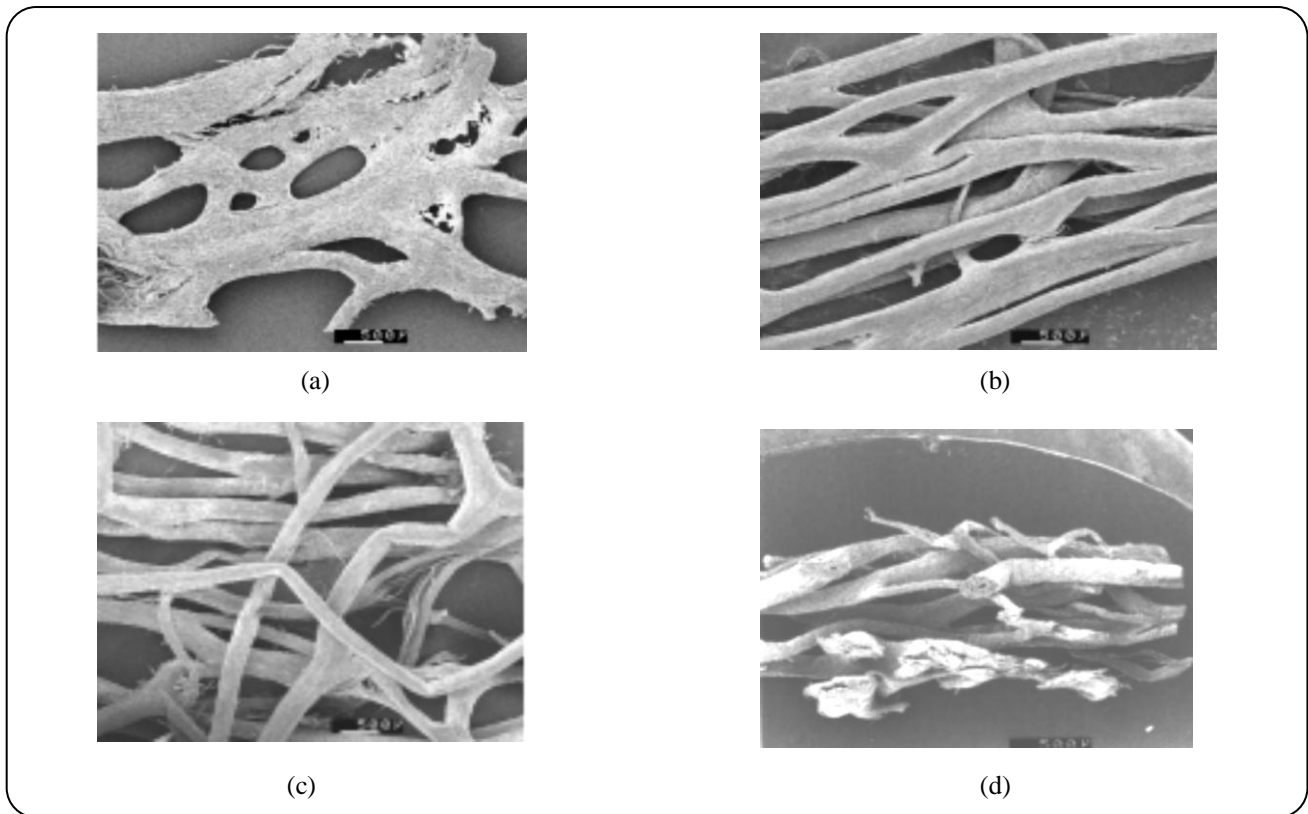


Fig. 2: Observation under the scanning electron microscopy of the *Luffa cylindrica* cords to weak enlargement – scale 500 μ .

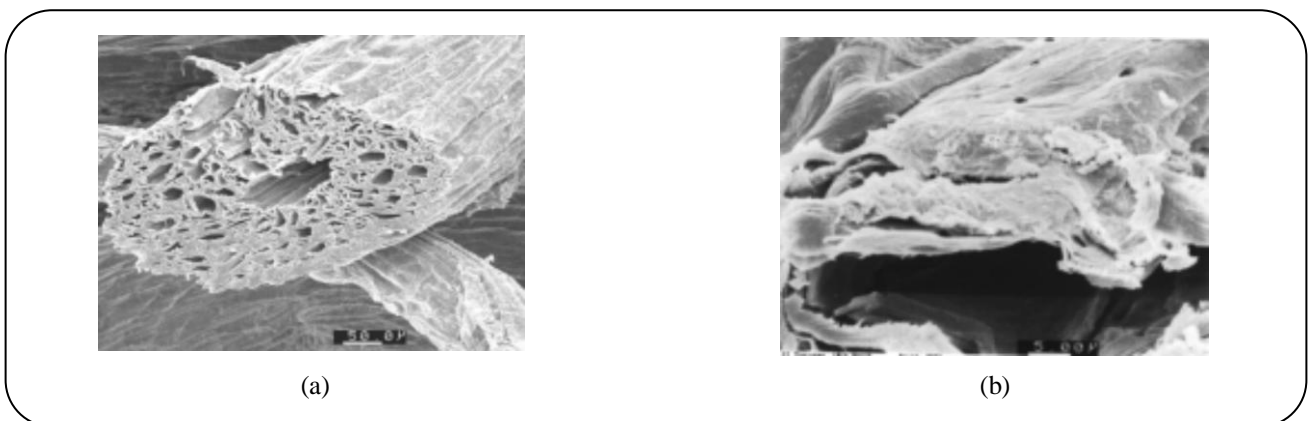


Fig. 3: Observation under the scanning electron microscopy of the *Luffa cylindrica* cords with high enlargement - scales 50 μ and 5 μ .

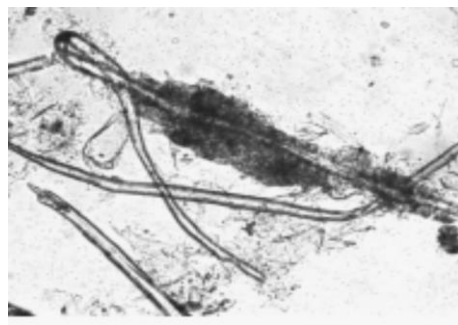
characteristics of cotton or wood fibers, the *Luffa cylindrica* fibers appear shorter but of similar diameter. Their density and moisture with the ambient air is also comparable (Table 2).

Chemical analysis

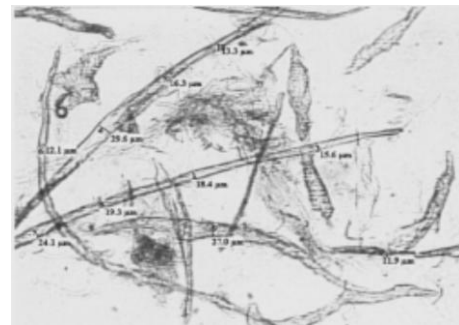
The results of the elementary analyses of cords extracted from the different anatomical parts that constitute the *Luffa cylindrica* are shown in Table 3. The characteristic point

is the significant variation of the rates of carbon and oxygen in the different parts of the fruit, whose evolution appears regular from the inside towards the outside of the fruit.

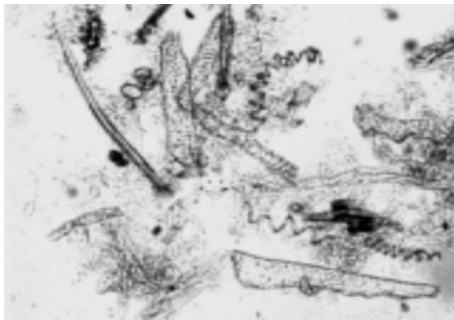
These measurements are to be composed with the cellulose percentages, indicated in the Table 4, whose raised values, reaching 80% significantly appear higher than the percentages of wood cellulose (resinous or leafy trees) and of the cellulose straw.



(a)



(b)



(c)



(d)



(e)



(f)

Fig. 2: Observation under the scanning electron microscopy of the *Luffa Cylindrica* cords to weak enlargement – scale 500 μ .

The cellulose percentage is in narrow correlation with the increase in the molar ratio oxygen/carbon, also indicated in Table 4.

The other components of the fruit are lignin and hemicelluloses; the values shown here (Table 5) were analyzed in the cord of the bond core-internal wall. The obtained figures appear in general agreement with those of the literature; however, the results fluctuate, probably because of the origin of the plants, the used methods of analysis, the climatic conditions to which harvest

was exposed, the nature of the ground as well as anatomical point of taking away within the fruit, as the present study tends to show it. In conclusion, the *Luffa Cylindrica* fruit is characterized by a strong cellulose percentage and a weak lignin rate, toward the fibers wood.

Physical characteristics

Crystallinity index measured by x-rays diffraction

The x-rays diffractometry analysis realize on a non-specific taking away of the plance in the fruit, reveals the presence

Table 2: Comparison of the characteristics of *Luffa Cylindrica* fibers to those of other vegetal fibers.

Origin of fibers	Nature	Length L (mm)	Diameter D (μm)	Density (g/cm ³)	Moisture to the ambient air (%)	L/D (1)
Annual plant (our results)	<i>Luffa Cylindrica</i>	0.9	8 - 30	1.48	7.5	48
	Rice, [28]	0.65-3.4	5-14	-	-	170
Annual plants	Bagasse, [28, 29]	1.0-1.5	20	-	-	65
	Cotton, [30]	5-65	10-40	1.54	8-9	1400
	Kenaf, [28, 29]	2.6	20	-	-	130
	Sorgho, [28]	1.0-1.5	20	-	-	65
	Leafy trees, [28] (moderate Zone).	0.7-1.6	20-40	-	-	40
	Coniferous tree, [30]	0.5 - 5	20 - 40	1.4-1.5	6-10	25-125

Table 3: Comparison of the elementary analysis of the different anatomical parts of *Luffa Cylindrica* cords to the straw; percentages expressed in (% w/w).

Vegetal	Anatomical part	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Oxygen (%)	Others (%)
<i>Luffa Cylindrica</i> (Our results).	External wall	46.5	7.7	3.8	37.6	4.4
	Internal wall	49.5	8.2	3.0	36.1	3.2
	Bond	59.6	8.1	3.0	26.5	2.8
	Core	66.1	6.8	5.0	21.3	0.8
Straw in an anhydrous state [31]	Total	48.0	5.8	-	42.2	4.0

Table 4: Analysis of cellulose and comparison of the molar ratios: Oxygen/Carbon (O/C) for the different anatomical parts of the *Luffa Cylindrica* vegetal and the straw.

Vegetal	Anatomical part	Cellulose rate (%)	Molar ratio (O/C)
<i>Luffa Cylindrica</i> (our results)	External wall	80.0	0.61
	Internal wall	75.0	0.55
	Bond	60.0	0.33
	Core	45.0	0.24
European straw [29]	Between nodes	39.5	0.66
	Nodes	30.6	
	Break into leaf	33.5	

of parts of crystalline cellulose (diffractogram characteristic of cellulose I) and amorphous (Fig. 5).

A rather precise value of the C_i can be estimated by the method of the surfaces [16], consisting in calculating the rate of the surface due to the crystalline peaks on the total surface (crystalline peaks + continuous bottom corresponding to the amorphous part):

$$C_i = \frac{\text{Surface of the crystalline peaks}}{\text{Totale surface}} \times 100 \quad (1)$$

We obtain as follows: $C_i = 69\% \pm 2\%$ value seeming higher than those usually obtained for wood fibers (between 32 % for the TMP and 55-60 % for the bleached chemical pastes (Kraft or sulphite of coniferous tree or leafy trees)) [17].

Thermophysical measurements

In the ThermoGravimetric Analysis (TGA), we observe (Fig. 6a) the loss in weight of the sample (in % brought

Table 5: Chemical composition of *Luffa Cylindrica*—comparison to wood, the annual plants and the straw.

Vegetal	Nature	Cellulose (% w/w)	Hemicellulose (% w/w)	Lignin (% w/w)
annual Plant (<i>Luffa Cylindrica</i>)	According to [12]	55	8	23
	According to [23]	90	-	10
	Our results ^a	60	22	10.6
Straws	Corn, [33]	33-45.5	21-28.5	10-21
	Rice, [34]	42-49.8	12.9-26.2	11.4-13.5
Annual plants	Hemp, [33]	74	18	4
	Cotton, [35]	92	6	-
wood	Coniferous tree, [33]	40-45	7-15	23-33
	Leafy trees, [36]	43-47	32-33	17-26

^a Analysis of the bond core internal wall

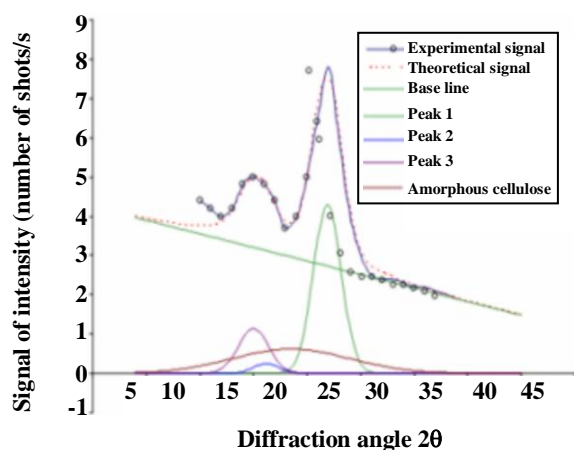


Fig. 5: Analysis of the crystallinity index of the *Luffa Cylindrica* cord by x-rays diffractometry: peak 1 ($2\theta=22.4^\circ$); peak 2 ($2\theta=16.2^\circ$); peak 3 ($2\theta=15^\circ$); corresponding to cellulose I; diffuse bottom (amorphous cellulose). Anode Coppers.

back to the initial weight) according to the temperature. We distinguish a phase from of evaporation of interstitial water then of bound water, for temperatures ranging between 100 °C and 260 °C, followed by a phase of decomposition (pyrolysis) beyond 260 °C. The latter one is characterized by various kinetics stages: slow kinetics between 260 and 320 °C, then fast between 320 and 380 °C; beyond 380 °C, the kinetics becomes again slow and stable.

The curve of Differential Thermal Analysis (DTA), represented on the same graph (expressing the heat flow emitted or absorbed by the sample according to the temperature), illustrates the various kinetic stages mentioned above, characterized by minor endothermic

reactions rather than 100 °C (corresponding to the adsorbed water loss), followed by an endothermic major sequence, then exothermic rather 320 °C. The analysis by calorimetry with differential Compensation (DSC (Fig. 6. b)) allows more precisely observing the water elimination adsorbed starting from 45 °C. Measurement also allows the determination of the heat storage capacity C_p of the raw vegetal obtained in the interval 80-180 °C. The obtained data that are recapitulated in Table 6 can be compared with those of various celluloses, or with pure glucose (according to [18]). The obtained values of C_p appear slightly closer to those of amorphous celluloses than those of crystalline celluloses, and comparable, even slightly higher, with the obtained values of wood celluloses.

Specific surface (BET) and hydrophilicity of the cords (WRV index)

The analysis carried out on the different constituent parts of the *Luffa Cylindrica* fruit show significant differences, at the same time in term of capacity of the adsorption, specific surface and degree of water retention after centrifugation (WRV index), representative of the hydrophilicity of the cords (Fig. 7, Table 7). Such differences are to be putting in relation to the measured cellulose percentages (Table 4), showing that the absence of lignin increases the hydrophilicity character and the fibers specific surface. The highest percentages of cellulose also contribute to a greater retention of soda compared to water, a phenomenon related to the swelling of the fibers (interaction of hydroxyl groupings).

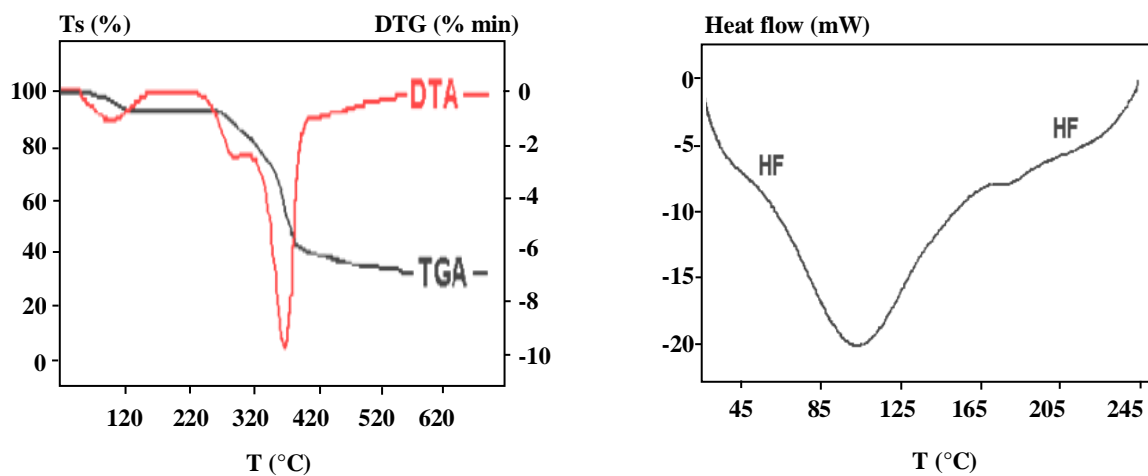
Table 6: Comparison of the heat-storage capacities of different celluloses and D-glucose to 350 K.

Sample	Crystallinity percentage (%)	($\Delta C_p/T$) (J/kg K ²)	C _p to 350 K (J/kg K)
Our results on <i>Luffa Cylindrica</i>	69	6.67	1506.2
Crystalline cellulose, [18]	100	1.42	1230.1
Amorphous cellulose, [18]	0	8.08	1414.2
Wood cellulose, [18]	38	5.06	1364.0

Table 7: Mass specific surface (Hydrodynamic and BET) and Water Retention Value measured on different samples of cord and on delignified fibers of *Luffa Cylindrica* comparison with the wood paste and straw.

Vegetal	Nature or form	Mass specific surface, a_m (m ² /g)		Water Retention Value (% / dry matter)	
		BET	Hydrodynamics (Pulmac apparatus).	Water	Soda
<i>Luffa Cylindrica</i>	Core	5.48±0.2	0.53±0.10	172.5±2.0	175.9±1.5
	Internal wall	7.22±0.15	0.70±0.12	175.7±1.5	205.6±1.8
	External wall	9.78±0.3	0.96±0.14	195.0±3.0	233.4±2.0
	Paste ^b	13.25±0.5	1.30±0.15	124.5±2.5	149.5±2.7
Wood	Paste	14.4±1.0 [19]	1.25±0.10 [19]	110±2.0 [37]	-
Straw	Paste [33]	-	-	200	-

^b Delignification by cooking the raw of *Luffa Cylindrica* cord in soda, [38].

**Fig. 6: Thermophysical analysis of the cord of *Luffa Cylindrica*.**

However, the values of specific surface (*BET*) of *Luffa Cylindrica* fibers appear of like usual size of the pastes kraft fibers;

the hydrodynamic specific surface is approximately ten times weaker than surface (*BET*) (Table 7), result commonly obtained on wood fibers [19].

CONCLUSIONS

- By microscopic analysis, it can be observed that the fruit is composed of an assembly of cords related to each other, forming a consolidated structure, where four parts are differentiated: external wall, internal wall, core and bond. Each cord consists of hollow and flexible

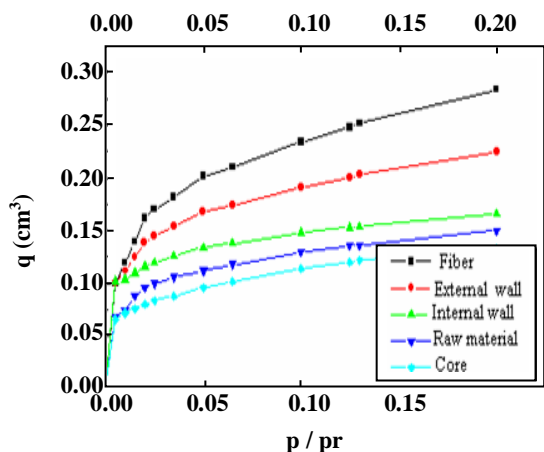


Fig. 7: Isotherm of adsorption B.E.T (gas: nitrogen) measured on different parts of the *Luffa Cylindrica* fruit: core; bond; interior cord; external cord; fibers of delignified cord ^a.

cylindrical fibers. From its structure, made up of vessels, cells of parenchyma and short fibers, *Luffa Cylindrica* approaches more to wood of leafy trees or annual plants such is sorghum, bagasse or the straw.

- The elementary chemical constitution varies between the different parts of the fruit. The external part of the fruit is richest in cellulose (80 %), in correlation with a high molar rate oxygen/carbon. The mean contents cellulose of the different anatomical parts appears higher than those of wood fibers and the lignin percentage are low ($\approx 10\%$).

- The crystallinity index of cellulose of the cords was characterized by x-rays diffractometry; the measured value (69 %) appears higher compared to the rather broad range of the values obtained from wood fibers of coniferous tree or leafy trees. This interesting result, coupled with the high rate of cellulose of the vegetal confers on fibers interesting properties.

- The thermophysical analysis (TGA, DTA and DSC) shows the various kinetic phases of the adsorbed water drainage processes and decomposition of material by pyrolysis, the heat storage capacities measured by DSC being close to those of wood celluloses

- The analyses of specific surface and hydrophilicity of the fibers (WRV index) appear also very differentiated in the various anatomical parts of the fruit, and in narrow correlation with the cellulose rates.

In accordance with this article relating to the analysis of the vegetal, a second article will deal to the

manufacture of a chemical paste of *Luffa Cylindrica* by cooking with soda, like with its paper characterization.

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