Study *of the Luffa Cylindrica* Part II: Pulp Elaboration and Paper Characterization

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ABSTRACT: *This study was devoted to the development of pulp Luffa Cclindrica and its characterization paper (annual plant fiber of the family Cucurbitaceae). Baking at soda helps to achieve a high level of delignification (Kappa ≤ 10) and high yield (≈ 67%). The beatability stack Valley is comparable to refined wood fibers, the fibers develop a normally time with good hydrophilic properties without excessive morphological variations. The Comparing to the same level of physical properties of refining pulp Luffa cylindrica with different pulps annual plants (straw, bagasse or kenaf), obtained by different processes, shows good mechanical properties of the fibers, in addition its behavior would tend to approach more the hardwood pulps. The measured thermal conductivity on the handsheets Luffa Cylindrica was found equal to 0.112 W / m K, value being also in the rather broad range of the data of the literature on different types of pulp, between 0.1 and 0.2 W / m K.*

KEY WORDS: *Luffa Cylindrica, Soda pulping, Mechanical properties, Optical properties, Thermal conductivity.*

INTRODUCTION

Many studies in the literature [1-9] relate the paper using annual plants as an alternative use of agricultural waste. Mechanical pulps of annual plants are often more energy efficient defibrizing of the ThermoMechanical Pulp (TMP) or Chimico ThermoMechanical Pulp (CTMP) wood. As for the pulp of annual plants obtained by chemical way, they can show such a high mechanical characteristics; even grater than those of pulps TMP or CTMP. The results were primarily shown on the straw, the Kenaf or Bagasse, but no study deals with

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the *Luffa Cylindrica*, annual plant fibrous of the cucurbitaceous family that grows in humid Mediterranean climate.

The valorization methods of the annual plants also relating to the chemical industry [6], the need for a study of the pulp elaboration of *Luffa Cylindrica* and its paper characterization thus seems also justified. After a first part devoted to the characterization of the plant [10], this second study addresses the development of pulp by cooking soda with or without additive (Anthraquinone), followed by a study of the paper potential of the pulp : aptitude for the refining and bleaching, physical properties of the formettes, and comparison with other pulps of annual plants.

EXPERIMENTAL SECTION

All the equipment used in this section are available to the French School of Papeterie and Graphics Industries in Grenoble (FSPG).

Pulping

Pulping were carried out on a rotary digester of with 6 chambers with 1.5 liters for each one; the chambers are individually heated by electrical resistors; the control system uses thermocouple immersed in the middle plunging. The conditions for obtaining the pulp pulping must be rewritten including the liquor ratio of the pulping process: Effective alkali: Ea = 32% / (NaOH); Anthraquinone : 0.1 % for cooking 1 only; temperature rise 90min; bearing temperature T=90 °C.

Kappa Index

The Kappa index is measured according to the AFNOR NF T 12-018. The Kappa index is an indication of the degree of delignification or bleaching ability pulp. For a given type of pulp, the kappa number is proportional to the amount of lignin contained in the pulp, according to the relationship:

$$[L](\%) = k.(X) \quad (1)$$

With k neighbor 0.15 - 0.20.

Mean degree of viscosimetric polymerization $\overline{DP_v}$

The $\overline{DP_v}$ is measured according to the AFNOR NF T 12-005. The viscosity index at a given solution of cellulose dissolved in the diamine curiéthylène temperature, is the quotient of the difference between the viscosity of the solution (η), and that of the solvent, (η_0), by the product of the viscosity of solvent (η_0), the concentration of the cellulose, C.

The Limiting Viscosity Index (LVI) at a given temperature is the limit value of the viscosity index when the concentration of cellulose becomes null:

$$LVI = [\eta] = \lim\left(\frac{\eta - \eta_0}{\eta_0\ C}\right) \quad (2)$$

With LVI expressed in ml/g

Calculate viscometric degree of polymerization, $\overline{DP_V}$, cellulose applying the formula

$$\overline{DP_V} \approx k^{\frac{1}{0.905}\log_{10}(0.75.LVI)} \quad (3)$$

With k =10

Scanning Electron Microscope (SEM)

The sample is held on a support by means of a brass double sided sticky then metallized prior to introduction into the chamber of the electron microscope balyage (ABT-55 apparatus (JEOL JMS 6100). The insulation fibers for microscopic observation are made by boiling in alkaline solution (NaOH, 0.5 M).

Refining

Refining was carried out in a stack Valley according to the section (I) of the AFNOR NF Q 50-008. Refining is a mechanical treatment that induces a change of both fiber geometry and physico - chemical, which will cause removal of the walls and greater flexibility of the fiber. That is introduced 30g of dry pulp in a liter of water, fiber solution is introduced into a ball mill refiner types (Lamper) The refiner has the following characteristics: 30g of dry pulp capacity, speed ω = 25 t / min , time (t = 1h 40min = 2500 rpm).

Degree Schopper – Riegler (°SR)

Measured according to the AFNOR NF Q 50-003 (ISO 5267-1). The Schopper - Riegler (°SR) is the number of water drained through a cake of pulp flowing through a reservoir centiliters. It represents a measure of the rate at which water can be extracted from a dilute suspension of pulp. The control of the refining takes place by measuring the Schopper-Riegler.

WRV (Water Retention Value)

WRV index corresponds to the water mass retained in material after centrifugation to *3000 G* $(G = 9.81\ m / s^2)$ during 15 min, measured according to the standard SCAN-C 62: 00.

Size distribution of fibers

The morphological analyses of the *Luffa Cylindrica* fibers are made on PQM 1000 device. Optical principles constitute a continuous observation through an optical device very dilute suspensions, which are fed into a capillary strength fibers arise in the direction of flow, the capillary is illuminated with polarized or non-white light that is used to detecting the passage of the fibers. For example Kajaani unit Fs -100 analyzer developed fibers allows the evaluation of the length of the fiber elements, shape, state of fibrillation and the tendency to flocculation and other more recent methods that use optical sensors and which does not require manual intervention. The Kajaani apparatus gives the results of weight distribution of fibers with length 21 for pulp loofah unrefined fiber unit 51850, the weight of the sample 2 g, length of 1.65 mm and a width of 26.7 microns. Linear density 0.109 mg / m and 9.4% flexibility.

Mechanical characteristics

The Handsheets manufacturing is performed according to the standard AFNORD NF Q50-002 (Section III) (Rapid-Köthen method) and begins by paying a quantity of paper pulp diluted equal to 2.36 g dry weight in a final mixer by regular suction will flatten the carpet fibers on a screen. Then, the wet sheet is recovered and allowed to dry. The handsheets were dried on a laboratory apparatus Frank dryers 2 or 3, then conditioned for 24 hours in an atmosphere at room temperature of 23 ° C and a relative humidity of 50%

Grammage; thickness; breaking length; bursting index; tearing index; tensile index; percent tensile elongation; air permeability (Bendtsen) are determined according to AFNOR standard: NF Q 03-019; NF Q 03-016; NF Q 03-004; NF Q 03-053; NF Q 03-11; NF Q 03-004; NF Q 03-002; NF Q 03-025, respectively.

Bleaching unbleached pulp:

The bleaching is made in a polyethylene bag by addition of sodium chlorite in buffered middle (pH 4.7), temperature of 70 °C, and consistency of the pulp 3 %.

Whiteness and opacity are measured according to the AFNOR standard : NF Q 03-039 and NF Q 03-040, respectively.

Thermal conductivity of handsheets:

The measurement method used here is an extension of the method "Sandwich" practiced for the measurement of thermal conductivities of insulating materials. A heating plan produced by photoengraving with a very low heat-storage capacity is arranged between the circular handsheets of the tested paper (diameter 9 cm); the assembly is pressed between two metallic plates, and thermally isolated from the external environment. After reaching a steady state, the thermal flow emitted by the plan warming as well as the difference of temperature between the central heating plate and the external patches are measured by thermocouples [11, 12].

MATHEMATICAL METHOD

As the heating plan is supplied with constant power (constant flow) when the steady state is established we can apply Fourier's law, taking into account the symmetry of the system:

$$\frac{\varphi}{2} = -K\,.\,A\ \mathrm{grad}T \tag{4}$$

Whence the thermal conductivity of the material:

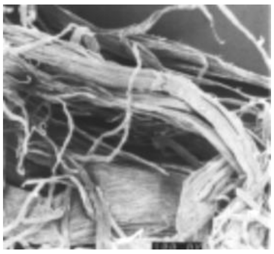
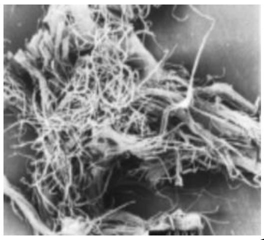
$$K = \frac{\varphi\,.\,e}{2\,.\,A\,.\,\Delta T} \tag{5}$$

With K is the thermal conductivity (W / m °C.), the dissipated power (W), A the surface of the sample (m^2), the thickness e of the sample (m) and the difference ΔT temperature between the two faces of the sample (°C). The temperatures are measured, the above equation becomes:

$$K = \frac{\varphi\,.\,e}{2\,.\,A\,.\,\Delta T}\left(1 + R_c\right) \tag{6}$$

Where R_c : a term without corrective dimensions.

Table 1: Pulping with the soda of Luffa Cylindrica.

	Bearing time (min)	Ratio liquor / plant(L/kg)	Active alkali (% NaOH / plant)	Pulp yield (% mass)	kappa index	Analysis at the SEM
Pulping 1 (with 0.1% AQ)	15	7/1	32	66.3	7.8	
Pulping 2 (without AQ)	32	7/1	32	68.0	10.8	

RESULTS AND DISCUSSION

Pulp elaboration

Pulping

Choice of a Soda Alkaline Cooking, with or without the addition of anthraquinone, is justified to the extent that is generally well adapted to annual plants. The results, summarized in Table 1, show a level of delignification and a high yield can be achieved with low cooking time and lower temperature. The addition of anthraquinone accelerates delignification significantly, allowing realize a gain of three 3 points of Kappa index, but at the expense of 2% of yield loss. However, given the low hold time and the high degree of delignification achieved with only soda, the addition of additive does not seem justified.

An optimum would be to seek as however the alkali rate used, standing at a high level compared to traditional cooking wood pulp.

By comparison to kraft cooking of leafy trees wood, the value of kappa index of the order of 11 or less obtained with *Luffa Cylindrica* is situated in the lower limit, and the efficiency of about 67% appears much higher. The $\overline{DP_v}$ index measured after delignification with sodium chlorite (conditions applied being those of the test holocellulose) on the pulp baking 2 (without Anthraquinone) is 1130. These Numbers demonstrate the good quality of cellulose obtained, high efficiency is due to the high cellulose percentage measured in the different anatomical parts of the fruit, ranging between 60% and 80%, and the strong crystallinity index of the cellulose (69%) [10]. After grinding, the paste did not contain unburnt or sticks and did not require screening. By observing the SEM, the fibers appear well separated and not degraded (Table 1).

Refining

The aptitude in the refining in stack Valley of the pulp resulting from cooking 2 was studied, on the one hand in terms of total indices (Freeness, *WRV*), and on the other hand, in term of morphological characteristics of the fibers (apparatus PQM 1000). The obtained results are summarized in Tables 2, 3 and in Fig. 1. The *Luffa Cylindrica* fibers demonstrate beatability stack Valley comparable to that of wood fibres, developing at a normal time the hydrophilic properties without effects of cutting or important morphological variations (Fig. 1, Table 3). note also that the percentage of the class of fibrous length [3mm - 7mm] is relatively high, measurement is confirmed after refining; what could to imply the existence of different populations of fibers, a significant fraction of long fibers.

The resulting values from the literature also reported in Table 3 can locate a range of comparison with different chemical pulp fibers. *Luffa Cylindrica* fibers would morphologically seem rather close to hardwood fibers;

Table 2: Effect of the refining on the characteristics of draining and water retention of the Luffa Cylindrica pulp cooked with soda (cooking 2).

Time of refining	°SR	WRV index with water	WRV index with soda 0.5 M
Not refined pulp	15	124	149
20 min	17	--	--
40 min	38	130	156
80 min	63	--	--

Table 3: Effect of the refining on the average morphological characteristics of Luffa Cylindrica fibers of the pulp (pulping 2).

	Luffa Cylindrica Pulp		Kenaf	Leafy Pulp	Coniferous tree Pulp
	Not refined	Refined with 38°SR			
Length *(mm)* [23]	1.65	1.52	4	1.1-1.2	2.5-3.5
Width *(µm)* [23]	26.7	29.2	22	25	35-40
Linear density *(mg/m)* [13]	0.109	0.104	-	0.075-0.10	0.15-0.35
Flexibility: (curve index)[b] *(%)* [13]	9.4	12.8	--	25	5-33

[b] *Curve index is defined by the following relation [13],S(%)=((l/L)-)1.100 with (l) the real length of fiber and (L) the geometrical length.*

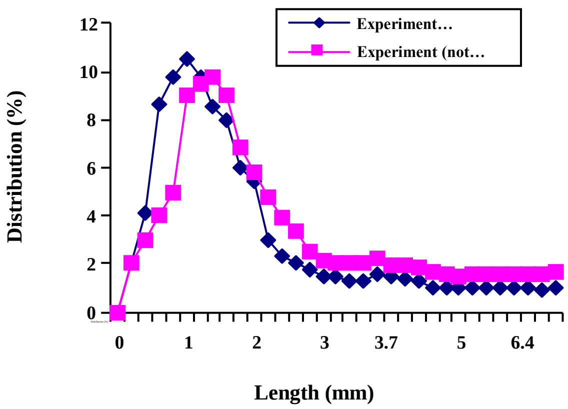


Fig. 1: Effect of the refining on the distribution lengths of Luffa Cylindrica fibers of pulp cooked with soda (pulping 2) – curve of distribution in weight according to the length of fibers – apparatus PQM-1000.

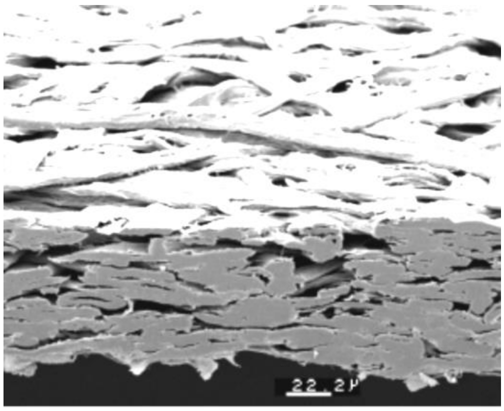


Fig. 2: Photography under the scanning electron microscopy of a paper cut worked out from the pulp of Luffa Cylindrica not refined resulting from pulping 2

however it is worth noting that comparisons of morphological characteristics of the fibers are often related to the measurement methods, and sometimes the very definition of measured variables, which makes rather difficult a very precise evaluation [11-13].

Manufacture of Mandsheets and Physical Proprietes

Mechanical and optical properties

Fig. 2 shows a section of handsheet photographed in a scanning electron microscope carried out starting from unrefined pulp (carried out on Handsheet Franck). Fibers will appear well individualized, flexible, flattened, thick walled and slightly degraded.

The physical properties measured on a set of Franck handsheet carried out starting from the pulp refined to 38°*SR*, resulting from pulping 1 and 2, are shown in Table 4. The pulpings are reproducible and physical properties do not differ from one cooking to another. We will be noticed the relatively high value of the tear index, index representative of the fibers flexibility, good binding

Table 4: Values of the mechanical properties, physical and optics for a pulp of Luffa Cylindrica refined with 38°SR.

Type of pulping	Pulping 2				Pulping Cooking 1			
	Mean	écart-type	Minimum	Maximum	Mean	écart-type	Minimum	Maximum
weighting (g/m^2)	75.4	1.70	73.9	78.2	71.5	3.2	69.1	77.1
Thickness (μm)	109.7	4.3	103.0	114.8	106.1	10	97.4	121.3
Density (g/ cm^3)	0.687	0.018	0.673	0.718	0.677	0.037	0.636	0.710
Specific volume (cm^3/g)	1.46	0.04	1.39	1.48	1.48	0.08	1.41	1.57
Bursting index ($kPa.m^2/g$)	4.69	0.88	3.15	5.37	4.78	0.94	3.16	5.60
% traction lengthening	5.30	0.50	4.60	6.10	5.2	0.3	4.7	5.5
Traction index (J/m^2)	220.1	26.6	191.9	258.7	213.5	12	201.5	232.1
Rupture length RL (m)	7977	267	7549	8271	8272	213	8042	8477
Tear index ($mN.m^2/g$)	13.9	0.7	13.2	14.9	13.7	0.8	12.8	14.5
Air Permeability (Bendtsen) (mL/min)	144	2	143	146	--	--	--	--
Whiteness [c] (% ISO)	85.8	0.3	85.4	86.1	--	--	--	--
Opacity[c] (λ=555 nm)	79.1	0.4	78.7	79.8	--	--	--	--

[c]*Values measured after bleaching in only one stage with sodium chlorite of the refined pulp.*

Table 5: Comparative table of properties of the Luffa Cylindrica pulp (issued of pulping 2) and the different straw pulps.

	Luffa Cylindrica	Straw pulp						
		[2]	[3]	[7]	[4]	[5]	[5]*	[9]
Pulp yield (%)	68	70	50.2	45.0	46.0	45.2	53.1	44.0
Kappa index	10.8	--	20.2	20	20	16.4	15.2	22.7
$\overline{DP_v}$	1130	--	1410	1600	--	--	--	1500
Freeness index (°SR)	38	39	40	40	50	40	44	40
WRV (%)	130	212	--	--	--	--	--	--
Air permeability (Bendtsen) (ml./min)	144	36	--	--	--	--	--	--
Density (g/cm^3)	0.69	0.81	0.7	0.82	-	0.68	0.60	0.81
Burst index ($kPa.m^2$./ g)	4.7	2.2	6.1	5.1	4.0	3.6	4.1	5.4
Tensile elongation (%)	5.3	2.7	--	--	--	--	--	--
Tensile index (J/m^2)	220	720	--	--	--	--	--	--
Breaking length R L (m)	7980	5700	9380	8000	7790	7060	7550	8100
Tear index ($mN.m^2/g$)	13.9	5.5	5.8	5.7	4.0	6.8	6.9	5.9

index and good resistance of walls after refining, as well as the high value of whiteness obtained by action of sodium chlorite in single one stage, result showing a priori a good bleaching ability.

Table 5 shows the comparison of the results of physical tests to those fibers of straw pulp (Table 5), Bagasse or Kenaf (Fig. 3) obtained by different processes, the data resulting from different sources of literature. We shall notice again the values of high performance and low kappa index for a chemical pulp, as well as the good performance of the mechanical properties of *Luffa Cylindrica* fibers. A remarkable fact appearing on the exploded straw pulp is exploded its high degree of hydration (correlated with low air permeability), although the comparison is limited here by the difference processes of obtaining pulp, this result does not appear on the *Luffa Cylindrica* fibers, whose behavior would tend, to approach more that of leafy trees fibers.

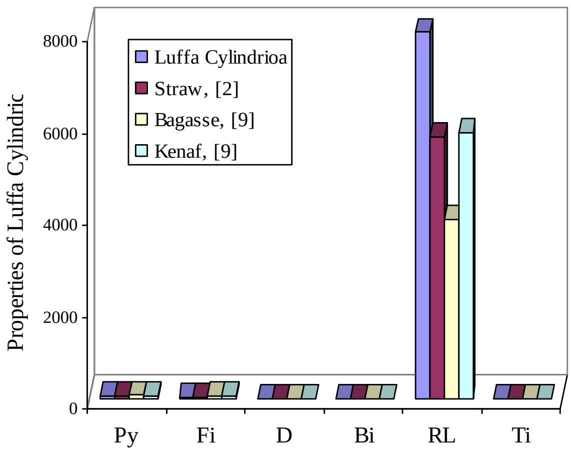


Fig. 3: Comparison of the properties of Luffa Cylindrica pulp (exit of pulping 2) with pulps of annual plants produced by process of explosion. (Py : Pulp yield (%), Fi : Freeness index (°SR), D : Density (g/cm^3), Bi : Bursting index (kPa.m^2/g), RL : Rupture length (m), Ti : Tear index (mN.m^2/g).)

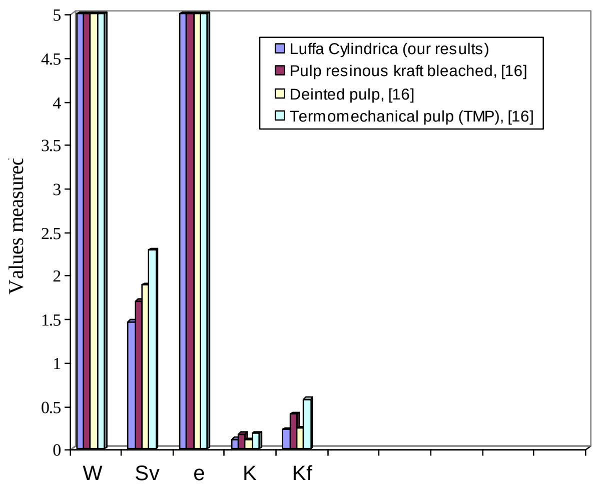


Fig. 4: Comparison of the values of the measured thermal conductivity of formettes of ecru pulp of Luffa Cylindrica (formettes realized on Franck apparatus, refined pulp with 38°SR resulting from pulping 2) with those of formettes of papers exist of the literature. (W : Weighting (g/m^2), Sv : Specific voluminal (cm^3/g), ε (%) : Porosity, K : Thermal conductivity measurement of formettes (W/m.K), k_f: Conductivity calculated thermal fibers (W / m.K).)

Thermal conductivity

This measure has an interest here because as has been reported *Luffa Cylindrica* fibers have been various attempts to use, and among these, the incorporation in composite materials [14, 15].

The thermal conductivity of the *Luffa Cylindrica* pulp, deduced from measurements taken on a variable stacking thickness handsheets Franck pulp cooking 2, is found equal to 0.11 W/m K. This value can be compared with some data stemming from the literature [16] (Fig. 4), undocked the dough appearing here nearest (0.1 W/m K).

The thermal conductivity measured on handsheets being connected to their porosity and internal structure [16-22], the thermal conductivity of fibers can be calculated from the following relation, from the hypothesis of the mediums with parallel pores:

$$k_{formette} = (1-\varepsilon)\, k_{fibres} + \varepsilon\, k_{air} \quad (7)$$

With: ε : porosity of the handsheet and k_{air} = 0.026 W / m K.

Thus, the values of thermal conductivity of fibers so calculated, also shown in Fig. 4 show results of the same order of magnitude, although it can vary from simple to double. Given the variety of the fiber sources mentioned as well as the different methods of measurement, it seems difficult to go further in the search for interpretation of observed variations.

CONCLUSIONS

The development of pulp of *Luffa Cylindrica* by pulping in the soda and paper characterization has established the following points:

- A good level of delignification and a relatively high yield can be reached at low temperature and in holding time; the pulp is free of unburnt, fibers appear well defibrated, flexible and little degraded.

- The suitability to stack refining Valley is good, fibers developing at a normal time the hydrophilic properties without important morphological degradation.

- The mechanical properties of handsheets *Luffa Cylindrica* are good, by comparison to those straw pulps, Bagasse or Kenaf at the same level of refining, obtained by different processes, and tend to get closer to those of hardwood fibers.

- The action of sodium chlorite in a single stage on pulp *Luffa Cylindrica* allows to reach a level of rather high enough whiteness, indicating a good aptitude for bleaching.

- The thermal conductivity measured on handsets of *Luffa Cylindrica* is situated in the range of the literature data concerning different types of pulp.

In summary, this study shows that the *Luffa Cylindrica* is fibrous resource high in cellulose interesting for the manufacture of paper pulp, allowing a high yield alkaline cooking and of short duration as well as fibers production of suited to the bleaching whose physical characteristics are comparable to wood pulp. Economically, we can estimate that the cultivation of *Luffa Cylindrica* would make it possible to reach a efficiency around ten tonnes per hectare, number who could represent a certain potential paper in the Maghreb countries where this plant could be easily implanted on the extended surfaces in humid zone.

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Nomenclatures

AFNOR	French Association for Standardization
CTMP	Chemico thermomechanical pulp
FSPG	French School of Papeterie Grenoble
ISO	International Organization for Standardization
TMP	Thermomechanical pulp

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