

IMPROVED HYDROPHILIC EXTRACTION METHOD (IHEM) OF BAMBUSA VULGARIS FIBER S AND EFFICIENT PHYSICO-CHEMICAL PROPERTIES.

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ABSTRACT

This work aims to propose an extraction technique for reliable bamboo fibers with improved hydrophilic property, for potential use in composites related to walls revetement and fences. "Bambusa vulgaris" aged five have been used for this purpose. Many fibers extraction methods were examined and compared to IHEM that consists to use the DMCN Art-150 press machine (8000 N/m²) on bamboo and immersing in a 3% caustic soda solution. Parameters like the diameter of the fiber s, their density, their percentage of celluloses, which influenced the hydrophilic character, are found. The chemical characterization, by ASTM D standards (American Society for Testing and Materials), has shown that the fiber s of "Bambusa vulgaris" extracted by the IHEM have 19.7% lignin, 54.1% cellulose. With the "pycnometry" method, we found a density of 1.494 g / cm³ and by measuring, the professional digital electronic caliper of YATO brand (YT - 7205) ranges 0-150 mm sensitive to ± 0.02 mm. The fiber s diameter has been determined using a "Brasser Biolux NV" optical microscope equipped with Balow Lens. An average diameter of fiber s equal to 0.140mm and 0.225 millimeters respectively on 25.2% and 22.2% of the extracted fiber s are found. The lateral surface of each fiber was observed with a magnification of 2, a 16-x eyepiece and a 60-x lens. Images acquired by computer, have been analyzed using Image J software, showing a very smooth morphology. The IHEM reduces extraction duration to less than five days with very little waste (less than 20%).

KEYWORDS: Characterization, "Bambusa vulgaris", fiber extraction, improved hydrophilic.

I. INTRODUCTION

Bamboo is a technological plant in the sense that all its components can be applied. Although bamboo fibers were studied by numerous researchers [1][2], the characterization of bamboo fibers always remains an axis of exploration. Bamboo fibers, like other vegetal fibers are very effective in the strengthening of polymer composites. They could be used as reinforcements in a composite to replace reinforced concrete on external walls decoration and fences. In the present case, some civil engineering structures are of composite materials with the reinforcement of steel and the concrete matrix [3]. Some buildings undergo several mechanical stresses (compression, bending, due to their architecture) and physical constraints (due to heat and rain cycles). Steel as a reinforcement that undergoes degradation due to oxidation is increasingly expensive; added to the matrix made up of cement, which also has limitations due to its unsustainable and polluting character. While plant materials such as bamboos abound in the swamps and constitute this day vegetation not sufficiently exploited in the Cameroonian cultural environment. In addition to that, bamboo is a lightweight bio composite with an interesting Young's module and its use is already marketed and industrialized in the world today [1] (Figure1).



Figure 1: B.N Mohanty, IFS Bamboo Industrial Processing in India dans « INDIAN PLYWOOD INDUSTRIES RESEARCH & TRAINING INSTITUTE ».

However, their hydrophilic character poses a problem with their characteristics which can thus lead to rapid aging of the composites. When penetrating water, the main constituent of bamboo which is cellulose (46-58%) day [1] loses its hydroxyl groups (-OH) as a replacement for hydrogen ions (Figure 2).

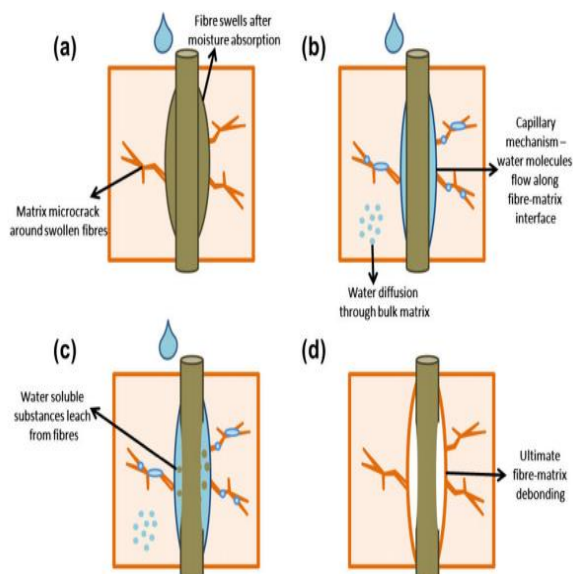


Figure 2: Mechanism of water absorption by composites [4]

Under normal conditions of use, cellulose can contain up to 70% more or less bound water. Replacement of part of the inter-chain interactions by hydrogen bonds between cellulose and water causes a plasticization of the material, therefore, a decrease in its mechanical characteristics. While the breaking stress of very crystalline cellulose fibers can reach 700 MPa dry, it can lose up to 30% of its value in a humid atmosphere [3][5]. Cellulose is resistant to strong alkaline but is easily hydrolyzed by acids [6]. Several treatments have been created to solve the problem of hydrophilicity of plant fibers and thus allow to increase the mechanical characteristics and a better adhesion with the polymer matrix, for better longevity at the work of the composite. Most researchers opted for alkaline pretreatment followed by chemical fiber cutting treatment [7] of these fibers; reduce their hydrophilic behavior compared to certain physical parameters. In this case, the diameter of the fibers is considered as cylindrical which is reduced when the surface is smoother and the angle of contact greater [6]. The diameter is as much smaller as the fiber is resistant [8]. Density and porosity that decreases [1], the cellulose composition that increases, for an increase in crystallinity. Some of these parameters are different from one fiber to another within the same species of fibers [9]. In addition, there are more than 1200 types and species of bamboo in the world; each type or species may have different mechanical characteristics from the others [10]. The density of the fibers also depends on the extraction process [11]. Tensile and bending resistance increases with cellulose density and ratio from the bottom of the thatch upwards and the inside to the outside [12].

The stubble must be mature (between 3 and 6 years) to have optimal resistance for structural applications. In addition to their species and their treatment, the method of extracting bamboo

fibers is a very determinant impact factor in the action of reducing their hydrophilic behavior [13]. Bamboo fibers extraction methods are multiple and limited by: a yield below 50%, a high cost and various inadequacies to solve the problem of hydrophilic. The sample is always global in bamboo [10] or just left on the inside and outside edge [6]. The main objective of this article is to propose a technique of extraction of "Bambusa vulgaris" fibers, which makes it possible to obtain improved fibers of certain characteristics such as diameter, density, porosity and cellulose level are optimal to the phenomenon of hydrophilicity. In the literature, some techniques for extraction of bamboo fibers are such: mechanical extraction, steam explosion, alkaline processing techniques and accelerated retting. The method discussed in this article is one that is closed to above techniques and which have made it possible to estimate the value of the characteristics sought to improve the hydrophobic character of extracted bamboo fibers.

- ✓ Masako ASHIMORI [14] proceeded extraction by mechanics (crushed fibers) and steam explosion then obtained fibers whose diameters range from 210 – 425 μm and from 125 – 210 μm. The fibers can also be fine resembling powder. Their diameter is from 40 μm to 120 μm.
- ✓ Through a mechanical means of pressing bamboos in a press for 30 seconds or laminated with a rotational speed of 60 rpm, DO THI VI [10] has extracted fibers with the parameters according to tables 1, 2 and 3.

Table 1: Diameter and length of bamboo fibers according to the extraction method of Do Thi Vi [15]

	Diameter d (mm)	Length l (cm)
Parallele rolling	0.5-1.7	3.0-4.5
Perpendicular rolling	0.8-2.6	0.5-2.0
Pressing	0.7-2.2	4.5-6.0

Table 2: Distribution of fiber size according to the Do Thi extraction method [10]

	Diameter (d) d ≤ 1.0 mm	Diameter (d) 1.0 < d ≤ 1.5 mm	Diameter (d) 1.5 < d ≤ 2.0 mm	Diameter (d) d < 2.0 mm
Parallele rolling	27%	70%	3%	0
perpendicular	15%	49%	16%	10%
Pressing	19%	66%	12%	3%

Table 3: Chemical composition of bamboo fibers according to the do Thi extraction method [10]

Component	Cellulose	Hémicellulose	Lignin	Pectin	Ash	Wax
Rate (%)	47.1	16.1	20.2	9.6	1.1	5.9

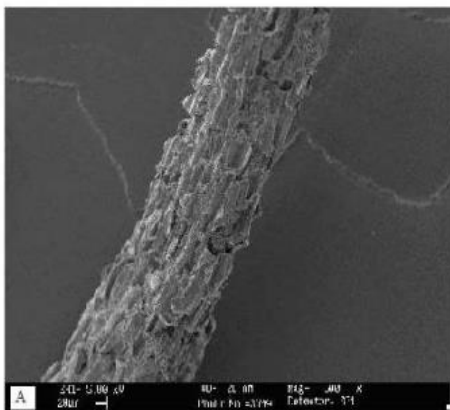
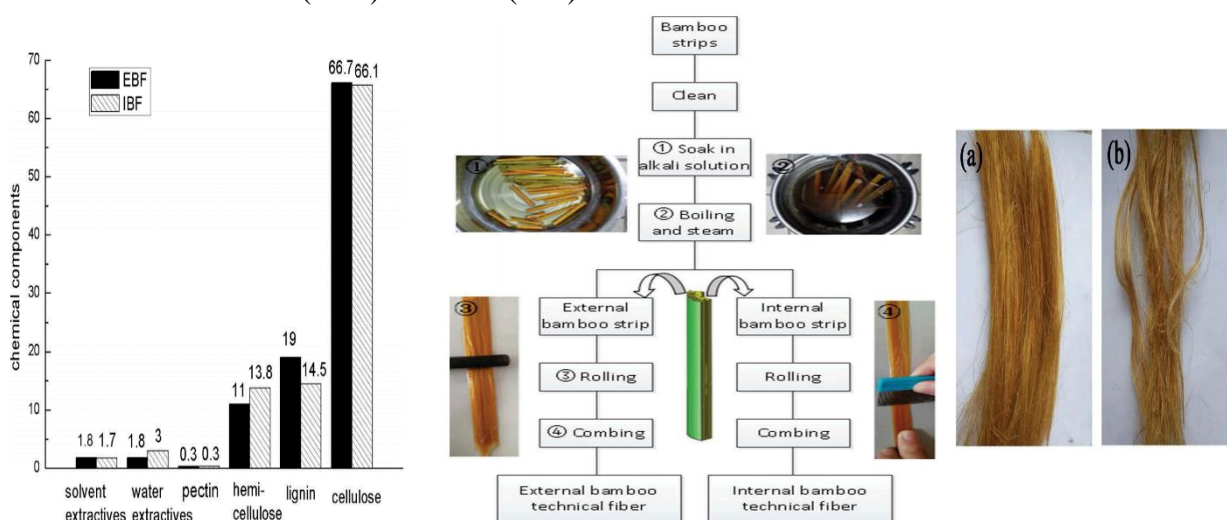


Figure 3: morphology of the surface observed under an optical microscope

- ✓ R. A. Ilyas [15] extracted fibers by the typical procedure for the production of cellulose nanocrystals.
- ✓ The extraction of Min Hu [16], by the mechanical-chemical technique, through bamboo slats placed in an alkaline solution for 24 hours at room temperature cleaned with water, and then immersed in boiling water for 1 hour, pressed and combed afterward with unidirectional force, has the average diameters 107.9 μm and 89.6 μm respectively for fibers taken from outside (EBF) and indoor (IBF), as well as the respective swelling coefficients of 18.6% (EBF) and 21.6 (IBF).



Chemical components of bamboo fiber.

Figure 4: Comparison of the chemical decomposition of bamboo fiber extracted according to Min Hu

Figure 5: Bamboo fibers. Extraction process by Min Hu [16]

Table 4: Comparison of the diameter and coefficient of variation

Fiber name	Average diameter (μm)	Coefficient of variation (%)
EBF	107.9	18.6
IBF	89.6	21.6

All these methods have participated in extracting the fibers for a particular utility with numerous advantages: the production is easy and takes only a few hours (Masako ASHIMORI [14]); the use of a parallel and perpendicular rolling mill with a rotational speed of 60 revolutions/min, the space between the two rolls being 0.1mm, allows a large production of fibers in a short time (Do Thi Vi) [10]; the function of the acids used lies in their ability to release H⁺ protons for the hydrolytic cleavage of glycosidic bonds in cellulose molecular chains at the level of amorphous regions. It allows separation between the fibers (R. A. Ilyas) [15]; NaOH treatment up to 2% reduces surface tension and increases the contact angle against hydrophilic, and there is the possibility of distinguishing fibers from the outside to the inside of the thatch; with good cellulose content (Min Hu) [16]. However, there are limitations of these methods in terms of their participation in the reduction of hydrophilic character. At Masako ASHIMORI, the diameter of bamboo fibers is as large as 0.5 mm even if they are extracted mechanically or by steam explosion. Fibers are damaged such that the defect of chemically pretreated does not promote the improvement of their hydrophilic character. We note in Do Thi Vi that the non-hybrid treatment with heat and alkaline does not give the fibers the desired qualities to improve the surfaces of the fibers against hydrophilicity as for R. A. Ilyas and Al. He experimented with extraction on corn cobs, not bamboos in Min Hu and Al where, the soaked bamboo slats cleaned with water and then immersed in boiling water for 1 hour does not facilitate the reduction of crystallinity, polymerization and the obvious separation of fibers (especially that of the outside) pretreated against their hydrophilic character.

To improve the possibility of reducing the hydrophilic character of bamboo fibers during extraction, we will first proceed to the choice of the species and the part of bamboo. Finally, we propose a new extraction method based on the knowledge in experimental characterization in laboratories that have made it possible to understand and analyze the hydrophilic behavior concerning their application in structural elements such as house facings and fences. It will simply remain to evaluate some parameters of the fibers due to the different modes of extraction of bamboo fibers in general, as presented by the previous authors.

II- MATERIALS AND METHODS

II-2- Equipment used

II-2-1- Equipment

- A Chinese brand DMCN Art-150 press machine, 8000 N/m² (Figure 6).
- A Bresser Biolux NV optical microscope equipped with a CeCaM® Barlow lens and a computer (Figure 7);
- An electronic scale mini pocket scale 0.01g -600 g brand (Figure 8-a) and an electronic scale of 1 g – 5 kg (Figure 8-b).



Figure 6: Bresser Biolux NV optical microscope



Figure 7: Press machine



Figure 8: 8-a) et 8-b): Electronic scale

II-2-2- Materials

The methodology we used here, matches with that of F. Ilczysyn [17]. The choice of the quality of fibers is oriented concerning their nature (place and conditions of cultivation, moisture content of the fiber, position of the fiber in the stem, density of the material and their location) thanks to the review of V.M. Ngwel [18] and F.N. Fokwa Didier [19]. We chose the fibers of "bambusa vulgaris" in maturity of about 5 years from the species of "graminae" harvested in a locality with marshy and warm area in the coast of Cameroon.



Figure 9: Longitudinal texture of « bambusa vulgaris »



Figure 10: Transverse texture of « bambusa vulgaris »



Figure 11: Transverse texture of « bambusa vulgaris »

Selection of samples

The fiber samples were selected according to the strips A', A''; B', B'' and C', C''; with the same technique as that of [F.N. Fokwa Didier \[19\]\[20\]](#): Bamboo is cut on the stem at its size delimited by two knots; then the bamboo is cut into three parts of length $l = 100$ mm such as that at the base of the plant "A", that above the plant "B", and that in the middle "C". These three parts are also split each of two.

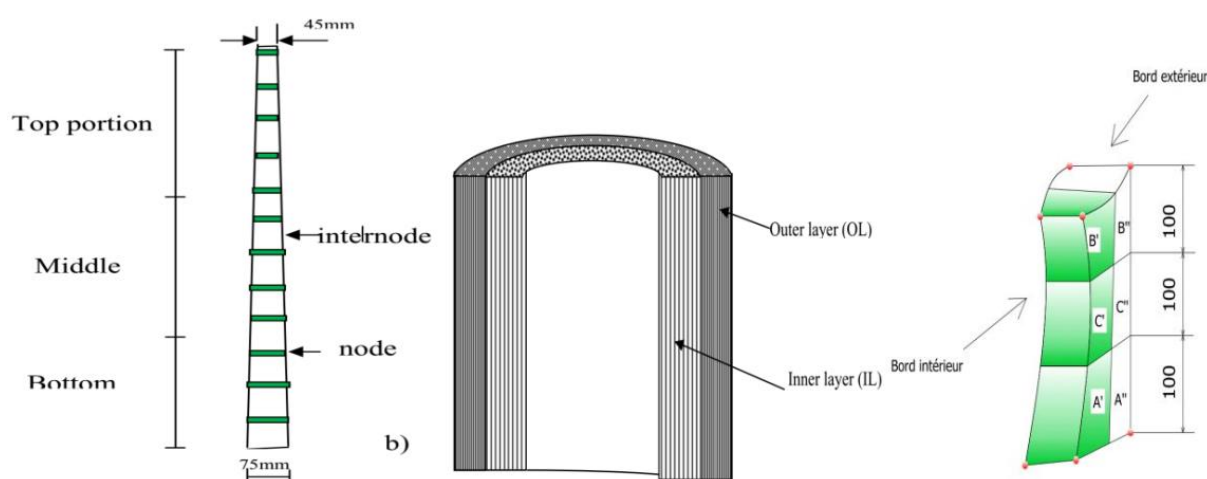


Figure 12. Bamboo laminated in six zones of study [20]

II-3- Method

II-3-1- Hypothesis:

- Extraction by alkaline processing techniques is the best method for treating fibers against hydrophilicity according to [Nguyen Tien Phong \[20\]](#).
- The density of the fibers also depends on the extraction process ([K, Murali \[11\]](#)).
- Stubble must be mature (between 3 and 6 years) to have optimal resistance for structural applications [20].
- The fibers of the outer part of the top thatch are those experienced because, according to [Trujillo et Al](#), the tensile and bending resistance increases with the density and rate of celluloses, from the bottom of the thatch upwards, and from the inside to the outside [12][21].

II-3-1 Extraction mode

The extraction method adopted here is a new mechano-chemical artisanal technique that we have called: extraction "IHEM" (Improved Hydrophilic Extraction Method)

➤ Equipment

The press consists of a DMCN Art-150 press machine with a tone of 20 on a surface of 9cm x 7 cm; or a pressure of 1312.5 N/m².

- Experimentation: The extraction of bamboo fibers followed the steps according to the following table:

Table 5: Process of "IHEM" of "bambusa vulgaris" fibers

N ^o	STEPS	JUSTIFICATION OF THE STAGE	INFLUENCE AND PURPOSE OF THE STAGE
1	Choice of « bambusa vulgaris »	Bamboos harvested in the Cameroon coastal region	- Exploitation of local Materials
2	Bamboos at the maturity of 5 years	Stubble must be mature (between 3 and 6 years) to have optimal resistance for structural applications [20]	- Use of fibers from the most resistant part of bamboo
3	Choice of the bamboo part (thatch)	Tensile and bending resistance increases with cellulose density and levels, from top to bottom [12]	
4	Sectioning and cutting into strips of about 15cm x 4 to 6 cm, eliminating the knot and integument	Split the inner and outer parts for better treatment against hydrophilic	- Exploitation of local materials
5	Solubility of the slats at decreasing temperature T=100 °C for 30 min in a caustic soda solution (NaOH 3%)	<ul style="list-style-type: none"> • NaOH reduces surface tension and increases contact angle against hydrophilic [22] • The water heated during extraction dissolves the lignin and part of the hemicellulose, leaving the cellulose reactive in the solid phase [23]. • • The level of the % of alkaline < 5% prevents the decrease in the tensile strength of the samples, as well as the breakage of the fibers [7] 	<ul style="list-style-type: none"> - Swelling of the bamboo and increase of its internal surface. - Decreasing in the degree of polymerization of bamboo cells. - Decrease in crystallinity of bamboo. - Dislocation of structural bondages. - Stabilization thermal effect. - Facilitation of hydrophobicity
6	Quenching in distilled water at room temperature for 2 days	Assurance of a homogeneous retting [24]	- Complete elimination of free acid molecules in amorphous regions
7	Grinding to the press	<ul style="list-style-type: none"> • Dispersion of internal and external bamboo strips, separately. • Cell wall rolling and individualization of nanofibrils [25] 	<ul style="list-style-type: none"> - Decrease in the thickness of the lamella and alignment of the fibers on a single layer - Hydraulic cleavage of bonds in molecular chains

8	Drying in the sun for 3 days with a temperature that varies from 30°C to 35°C	Increasing and maintaining temperature for 3 days, makes it possible to empty the water in the deep regions of the slats, in the littoral region of Cameroon during the dry season	<ul style="list-style-type: none"> - Elimination of water molecules in the amorphous regions of the lamellae - Creation of porosity in bamboo slats for better detachment of fibers
9	Threshing of the slats	Fiber bundle shaping	<ul style="list-style-type: none"> - Effective detachment of fiber bundles
10	Spinning with a sharp knife and then by hand after and combing	Fiber Shaping	<ul style="list-style-type: none"> - Detaching Unit Fibers
11	Treatment in a solution of sodium hypochlorite (bleach 5%) for one hour	<ul style="list-style-type: none"> ▪ Dilution with distilled water stops the reaction of repeated washes with successive centrifugations Removal of nanoparticles from cellulose [26][15].	<ul style="list-style-type: none"> - Reduction of the diameter of the fibers and regulation of the circularity of its surface
12	Dried fibers again and combed	Cleaning of amorphous regions of the cellulose polymer	Fiber smoothing

The fibers of "bambusa vulgaris" were obtained according to the process described in the figure below (Figure 12).

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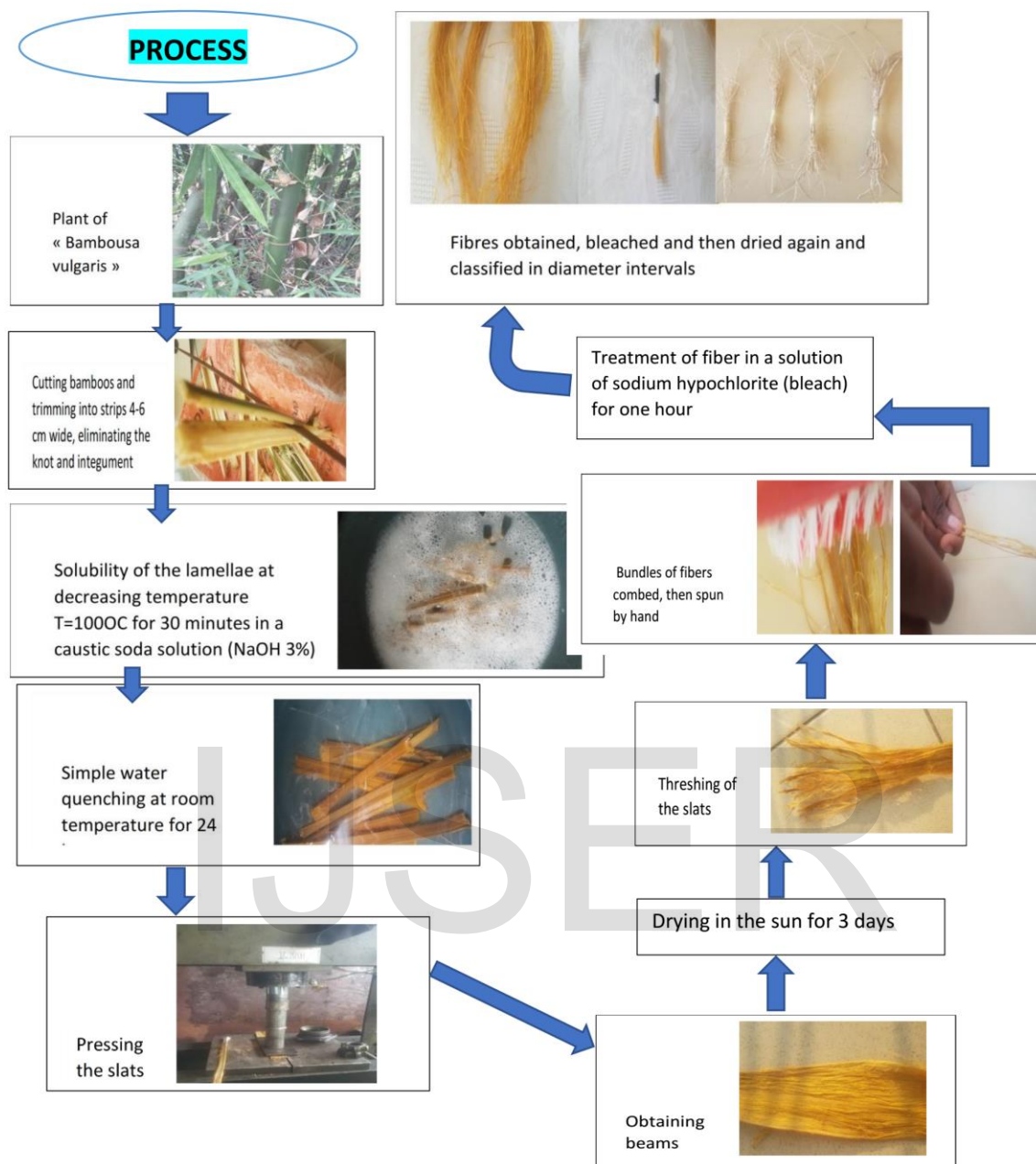


Figure 13: Extraction process "IHEM" of "Bambousa vulgaris"

II-4- Characteristics of the extracted fibers

The important characteristics (in this case: the diameter, density, porosity, cellulose level) which is the object of this article are those that allowed us to evaluate the impact of this new extraction technique "IHEM" on the hydrophilic character of the fibers of "bambusa vulgaris".

- The diameters were determined using a *Bresser Biolux NV* optical microscope equipped with a *Barlow lens from CECAM Lab*. The lateral surface of each fiber was observed with a magnification of 2, a 16-x eyepiece and 40-x objective. The image acquired by computer were analysed using the logical *ImageJ*. It's made over the length of the size of 100 short (less than 20 mm) and long (more than 20 mm) fibers. (Figure 13).

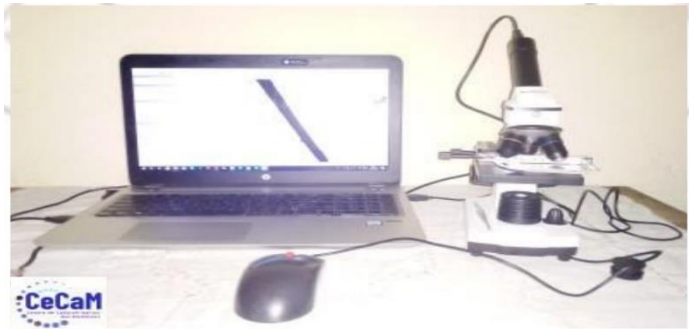


Figure 14: Measuring of the diameters of the extracted fibers

- The determination of the density of the fibers of the "bambusa vulgaris":

The experiment is carried out in a perfectly cylindrical pycnometer with a radius $r = 1.5$ mm, containing benzene at height $h_0 = 84$ mm. It immerses a mass $m = 100$ g of fiber of "bamboos with vulgaris" (Figure 14); it is noted that the liquid has risen to $h_1 = 94$ mm.

We find the volume of the fibers such that $V = \pi r^2 (h_1 - h_0)$.

This involves determining the density for this specimen : $P_B = \frac{mB}{VB}$;



Figure 15: 15-a) Content volume: content alone; 15-b) Volume of Benzene: Benzene-Fibers.



Figure 16:
Weighing of fibers

➤ The mass of an extracted fibers was measured by weighing 100 fibers with a length of 100 mm (Figure 15).

➤ Determination of the chemical composition of the extracted fibers

All tests were performed according to the procedures described in the literature [1][27] which incorporate the standards of the American Society for Testing and Materials (ASTM). A toluene solution is used instead of a benzene solution. The precise references of the standards followed for each chemical property are presented in Table 6 and each protocol is described in this section. Each test is performed 3 times (all concentrations are expressed in % by mass).

Table 6: Standards for chemical analysis [10]

Property	standard
Solubility Alcohol-Toluene	ASTMD 1107-56 (Reapproved 1972)
Solubility in hot water	ASTMD 1110-56 (Reapproved 1977)
Lignin	ASTMD 1106-56 (Reapproved 1977)
Holocellulose	ASTMD 1104-56 (Reapproved 1978)
Alpha-cellulose	ASTMD 1103-56 (Reapproved 1978)
Ash content	ASTMD 1102-56 (Reapproved 1990)

III- RESULTS AND DISCUSSION

- Extraction of bamboo fibers

In this new method of extraction "IHEM" of "bambusa vulgaris" fibers, it is possible to obtain bundles of fibers before spinning. There are also several other advantages such as:

- More economical and lasts less than five days.
- Less waste in bamboo, so good yield (almost 80%); hand spinning after combing details the fibers well to the nearest unit.

Although caustic soda tends to cause rapid breakage of the fibers in the tensile test, the "IHEM" method indicates that the immersion in the solution contains barely 3% soda and lasts only 30 minutes and after the fibers are well washed. The fibers appear smoother thanks to controlled hydrolysis. On the other hand, this new method also has a disadvantage because it requires a serious concentration and it produces little quantities.

- The diameter of the bambusa vulgaris fibers is deduced according to the following table:

Table 7: Summary of the diameters measured at three points of the size of the "bambusa vulgaris" fibers extracted

Fiber size	Quantity (%)	Diameter				Overall concentration
		[0 ; 0.10 mm] D _{m1} =0.05mm]0.10 ; 0.13 mm] D _{m2} = 0.115 mm]0.13 ; 0.15 mm] D _{m3} = 0.14 mm]0.15 ; 0.30 mm] D _{m4} = 0.225 mm	
Lower length to 20 mm	37 %	0 %	10 %	30 %	60 %	D _{m4} =0.225mm : 22.2 %
Upper length or equal to 20 mm	63 %	50 %	10 %	40 %	0 %	D _{m3} =0.14mm : 25.2 %

We found diameters ranging from 0.10 to 0.27 mm, with better overall concentrations of 22.2% (D_{m4} = 0.225 mm) and 25.2% (D_{m3} = 0.140 mm). It has been found that the diameters seen in the literature on bamboo fibers "Dang Ngà" (from 0.4 to 2mm) and "bambusa vulgaris" (from 0.3 and 1.9mm), are much higher. The reduction of the diameter thus leads to a standardization of the surface condition thus creating a large difference in contact angle which is an element impacting on the hydrophilic behavior of the fibers. This leads us to think that the extraction "IHEM" is favorable to the quality of the fibers against hydrophilicity.

▪ **Morphology of extracted fibers**

The surface of crude fibers is covered by fats, waxes or polysaccharides such as lignin, hemicelluloses or pectin and therefore, much rougher. In optical microscopy, the fibers extracted by the technique "IHEM" have smaller diameter than the bamboo fibers of the Thi Vi method, and their surface roughness is reduced due to the partial dissolution of the amorphous parts of fibers, so a decrease in hydrophilic behavior.

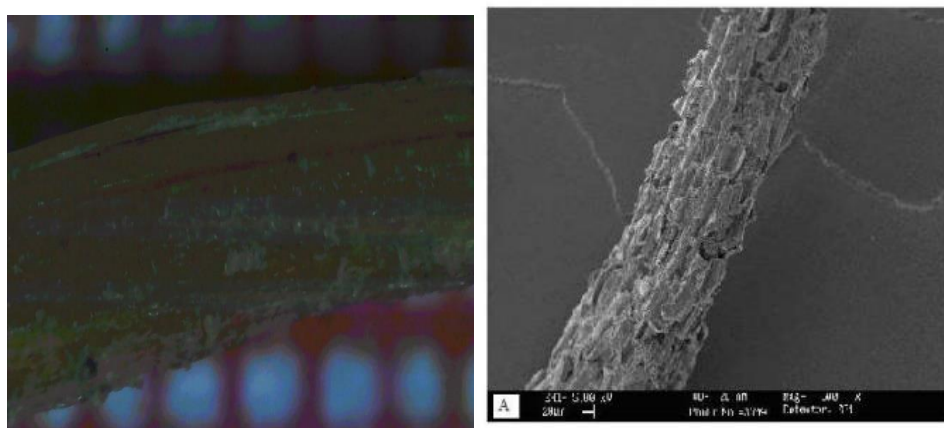


Figure4: Morphology of extracted fibers
a) by the extraction "IHEM" and b) that extracted fibers by Do Thi Vi

▪ **Density of extracted fibers**

Four experiments on specimens of "bambusa vulgaris" fibers yielded the following results: 1.476; 1.485; 1.491 and 1.513 g/cm³. So, our average density is $P_B = 1.494$ g/cm³. Value significantly smaller than that seen in literature.

Table 8: Chemical composition of "bambusa vulgaris" fibers

Component	Cellulose	Hemicellulose	Lignin	Pectine	Ash	Waxes
Percentage (%)	54.1	13.2	19.7	7.4	1.1	4.5

- **Cellulose level**

Kouchner's experiment resulted in a relatively improved cellulose composition of 2%. This allows the fiber a slight increase in crystallinity and therefore a decrease in the hydrophilic character of bamboo vulgaris fibers.

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IV- CONCLUSION AND OUTLOOK

The physical and chemical characterizations of bamboo fibers found following the search for a reliable extraction technique of "bambusa vulgaris" fibers to impact on the hydrophilic behavior of fibers. This led us to propose a mechano-chemical method called "artisanal" called "IHEM" method. The results made us to obtain smooth fibers by an ease of operations that lasts barely five days, with very little waste (less than 20%) and also to understand that the fibers extracted at the end and above the thatch (Sample B ") have interesting characteristics that allow to improve their hydrophilic behavior before any treatment. There is a clear increase in cellulose for better crystallinity, a decrease in density and diameter which makes the surface of the fibers less complex leading to an improved interfacial contact with a probable increase in the Young's modulus. The results obtained show that the extraction method "IHEM" could be favorable to the solution of the problem of hydrophilicity through the increase in the concentration of cellulose and the decrease in the density and diameter obtained on the fibers of bambusa vulgaris. Compared to other extraction techniques as seen in the literature and thanks to the alkaline solution (3% NaOH), this method prevents the condensation of lignin and prepares the bambusa vulgaris fiber for future treatments such as chemical coupling against the problem of hydrophilicity. In the future, the fibers of "bambusa vulgaris" can therefore easily undergo a chemical coupling before being used in the development of a polymer matrix composite for a better performance and longevity at work in the structures of facing houses of fences.

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