DEVELOPMENT AND CHARACTERIZATION OF POLYESTER RESIN/TAQUARA STICKS COMPOSITES

Carlos Alfredo Alves Junior, eq.carlos@gmail.com
Ricardo Pedro Bom, dem2rpb@joinville.udesc.br
Santa Catarina State University – Technological Science Center – Rua Paulo Malschitzki, S/N, Campus Universitário Prof. Avelino Marcante, Bairro Bom Retiro, CEP 89219-710, Joinville - SC

Abstract. *Environmental concerns lead to a search for replacements for fiberglass in the production of polymeric matrix composites. The aim of this study was to use cold-press molding to produce and characterize composites developed with taquara (Merostachys ternata) sticks and unsaturated orthophtalic polyester resin by means of flexural tests, and also to characterize taquara by means of tension tests. Taquara internodes with 150 mm were cut into sticks with a 3 mm blade opening cutting device. A mass distribution curve of the sticks was determined and the behavior was described by a Gauss curve, with an average stick mass of 0.79 ± 0.21 g. Average moisture content of 11.45% was achieved by drying three samples of sticks in a vacuum oven at 100ºC for 24 hours. Billets measuring 150 x 50 x 50 mm were prepared in a wood mold by compression. Tension tests were performed in the taquara sticks and the composites were characterized by flexural tests, according to ASTM D790. Using the results of the flexural modulus of the composite and the resin, the flexural modulus of taquara was calculated by Halpin-Tsai equation, and the result obtained was coherent with the elasticity modulus determined by tension tests.*

Keywords: *composites, taquara fibers, flexural tests, tension tests*

1. INTRODUCTION

Synthetic fibers, such as fiberglass, carbon and metallic fibers, are still used by the industry to obtain composites with good mechanical properties. However, these synthetic fibers have some disadvantages, such as high energy consumption required for their production, high cost when compared to other fillers, high specific weight, abrasivity for the equipment and non-degradability. Besides, some of them are toxic and may cause skin irritation during handling (ARAÚJO *et al*., 2010).

During the last years, environment concern has increased, highlighting to scientists the need for the use of natural materials. This movement has encouraged several industries, such as automotive, construction, furniture, packaging and others, to produce natural fiber composites instead of conventional composites with synthetic fibers (DEUS *et al*., 2005; MORAIS *et al*., 2006; BACHTIAR *et al*., 2008).

The application of natural fibers in polymeric matrix composites has several advantages in comparison with conventional composites, namely low abrasivity for the equipment, low specific mass, low cost and reduction of CO_2 emission (JOSEPH *et al*., 1999; JOSHI *et al*., 2004; DEUS *et al*., 2005; BACHTIAR *et al*., 2008; ARAÚJO *et al*., 2010).

The main components of natural fibers are cellulose, hemicellulose, lignin, pectin and waxes. Cellulose, hemicellulose and lignin are the basic components that give physical properties for the fibers (BLEDZKI and GASSAN, 1999).

Bamboo is a material that has been used for thousands of years and has followed human evolution in several activities, such as feeding, shelter, manufacturing of manual tools, utensils and objects (MOIZÉS, 2007). Like other natural fibers, bamboo is a natural composite with lignin and hemicellulose amorphous matrix and cellulose fiber reinforcement (RAO and RAO, 2007; JOHN and THOMAS, 2008). Bonilla and co-workers (2010) argue that bamboo has higher rupture strength and rigidity than timber in applications such as construction components, furniture pieces and agriculture tools.

Beraldo and Azzini (2004, *apud* BERNDSEN *et al*., 2009) said that in Brazil, bamboo is rarely used compared with tree or timber species. Timber account for almost all fibrous raw material utilized in several applications, from burning for energy production to industrial paper production. An increasing scarcity and value of timber observed in the last years has contributed to the increased interest in bamboo as raw material. An abundant bamboo species in Santa Catarina and Paraná regions, which is not commercially valuable, is the *Merostachys ternata* Nees, commonly known as “taquara-lixa” (SMITH et al., 1981). It has 4-8 m length, culms with 1.8-2.2 cm diameter, internodes shorter than 70 cm length, rough in the upright region of the culms (SCHMIDT, 2009).

The aim of this study was to use cold press molding to produce and characterize composites developed with taquara (*Merostachys ternata*) sticks and unsaturated orthophtalic polyester resin by means of flexural tests, and also to characterize taquara by means of tension tests.

2. MATERIALS AND METHODS

In this study, taquara fibers were used in a stick format with 150 mm length as reinforcement and a polyester resin was used as a bonding matrix.

2.1. Polyester resin

The unsaturated orthophtalic polyester resin used in this study is Arazyn 4.6, produced by AraAshland and distributed by Mastergel Compósitos. The initiator that comes with the resin is methyletylketone (MEK-P), used in a 1% wt. proportion for curing reaction. Flexural strength and flexural modulus provided by the manufacturer are 68.9 MPa and 2.8 GPa, respectively. The density of the resin is 1.1 g.cm^{-3}.

2.2. Production of sticks

Taquara sticks were extracted from bamboo internodes with 150 mm length. The internodes were cut in a cutting device with 3 mm blade opening.

2.2.1. Determination of the mass of the sticks

One hundred eighty-five sticks were weighed individually in a digital balance model AY220 from Shimadzu®, with 0.0001 g precision and maximum capacity of 220g. Then, a distribution curve of the sticks was obtained, as follows: with 0.1 g intervals, from 0.3 g to 1.4 g, the number of samples for each interval was counted. Thus, the distribution was represented by a histogram. The arithmetic averages of the sticks in each interval are indicated by blue spots on the histogram. Over the points Eq. 1 was interpolated, and then coefficients could be determined.

$$f(x) = \frac{A}{\sqrt{2\pi\sigma^2}} e^{-\frac{(x-\mu)^2}{2\sigma^2}} \tag{1}$$

In a normal curve, A is 1, μ represents the average, σ the standard deviation, and x the variable.

2.2.2. Moisture content of the sticks

Three samples with two hundred sticks each were previously weighed, and they were dried in a vacuum oven at 100ºC. Samples were weighed every hour for seven hours, at which point the variation in the mass of the samples was insignificant. After this period, samples were kept in oven for 24 hours for drying and they were weighed after that. The variation in the mass of the samples represents the loss of water during the test. Moisture content was calculated by Eq. 2:

$$\%m = \left(\frac{m_i - m_f}{m_i}\right) x\ 100 \tag{2}$$

Where m_i is the initial mass of the sample (g) in t = 0 h and m_f is the measured mass after a period of time.

2.3. Composite production

The composite was prepared by a cold-press molding process. First of all, 202.6 g (approximately 250 units) of dried taquara sticks were aligned longitudinally in a wood mold measuring 150 x 60 x 50 mm length, width and depth, respectively. After that, 200 g of polyester resin was weighed and its cure was started with 1% wt. of MEK-P initiator. Resin was poured carefully in the mold containing the sticks and a compression force was performed in order to ensure bonding of the sticks and adequate resin spreading through the composite, thus acquiring the desired composite shape for the specimen. There was a loss of resin during the process but after disregarding this loss, the matrix mass fraction was 0.33.

2.4. Preparing of composite specimens for flexural tests and taquara specimens for tension tests

Flexural samples of composites were based on ASTM D790. The samples were cut from composites that have the same shape as shown in Figure 1. Eight specimens were obtained with length ranging from 131.1 to 132.7 mm, width ranging from 20.1 to 20.4 mm and height from 6.7 to 7.4 mm. Nine taquara tension specimens were produced, with 150 mm length, width ranging from 2.5 to 4.0 mm and height from 2.0 to 2.3 mm. The corners were embedded in epoxy resin to improve placement in the universal machine and prevent shearing of the fibers by the claws.

Figure 1 – Produced composite

2.5. Tension and flexural tests

The specimens were first dried in a vacuum oven at 80ºC for 24 h and cooled at room temperature in a desiccator. Tests were carried out in an EMIC DL-30000 test machine. For tension tests, an extensometer with initial opening of 50 mm was used. Load cell used for composite flexural tests and taquara tension tests was 10000 N. Support span for flexural tests (L) was 105 mm for the first six samples and 111 mm for the other two. Crosshead speed for flexural and tension tests was 5 mm.min^{-1}. A three-bending test was carried out, and the flexural stress (MPa) and flexural modulus (GPa) were evaluated from the samples. After the test a little variation in the distances between the load point and the supports was found. For composites with L = 105 mm, the value of c_1 (distance between the first support and the load point) was 30 mm and c_2 (distance between the load point and the other support) was 40 mm; for composites with L = 111 mm, c_1 = 52.5 mm and c_2 = 58.5 mm. Flexural stress calculation was done by Eq. 3:

$$\sigma_f = \frac{6Pc_1c_2}{Lbh^2} \tag{3}$$

Where σ_f is flexural stress (MPa), P is the load applied to the specimen (N), c_1 is the distance between the first support and the load point (mm), c_2 is the distance between the load point and the other support (mm), L is the distance between the supports (mm), b is the specimen width (mm) and h is the specimen height (mm). Flexural modulus calculation was done with Eq. 4:

$$\boldsymbol{E} = \frac{2Pc_2[(L^2-c_2^2)c_1-c_1^3]}{\boldsymbol{vLbh^3}} \tag{4}$$

Where E is the flexural modulus (MPa), P is the load applied to the specimen (N), c_1 is the distance between the first support and the load point (mm), c_2 is the distance between the load point and the other support (mm), L is the distance between the supports (mm), v is the displacement, (mm), b is the specimen width (mm) and h is the specimen height (mm). To determine the taquara flexural modulus, Halpin-Tsai equation (Eq. 5) was used, as described below:

$$\frac{E_c}{E_m} = \frac{1+\xi\eta V_f}{1-\eta V_f} \tag{5}$$

Where E_c is the flexural modulus of the composite, E_m is the flexural modulus of the matrix, V_f is the taquara volumetric fraction in the composite, and η is calculated by Eq. 6:

$$\eta = \frac{\left(\frac{E_f}{E_m}\right)-1}{\left(\frac{E_f}{E_m}\right)+\xi} \tag{6}$$

Where E_f is the taquara flexural modulus and ξ is known as the reinforcement aspect ratio, defined by Eq. 7:

$$\xi = 2 \times \frac{a}{b} \tag{7}$$

Where a and b represent the dimensions of the cross section of a stick, where a is the dimension in the load direction given to the composite. The cross section of the sticks is randomly distributed. With the dimensions of its cross section, the area of each stick was calculated. A sum of the area of the sticks in the vertical position and horizontal position was carried out and from these values, calculation of the area contribution from the horizontal and vertical sticks was possible. Depending on inclination, stick was considered in the vertical or horizontal position. The aspect ratio was multiplied by each area contribution, according to Eq. 8:

$$\xi = 2 \times \left[\left(\frac{a}{b}\right)_v A_v + \left(\frac{a}{b}\right)_h A_h\right] \tag{8}$$

Where A_v e A_h correspond to the area contribution of the sticks in the vertical and horizontal position, respectively.

3. RESULTS

3.1. Characterization of taquara sticks

Figure 2 shows the sticks distribution histogram with the arithmetic average mass of each interval and the Gauss curve. The average mass and standard deviation of a stick were determined by Gauss equation and the result was 0.79 ± 0.21 g.

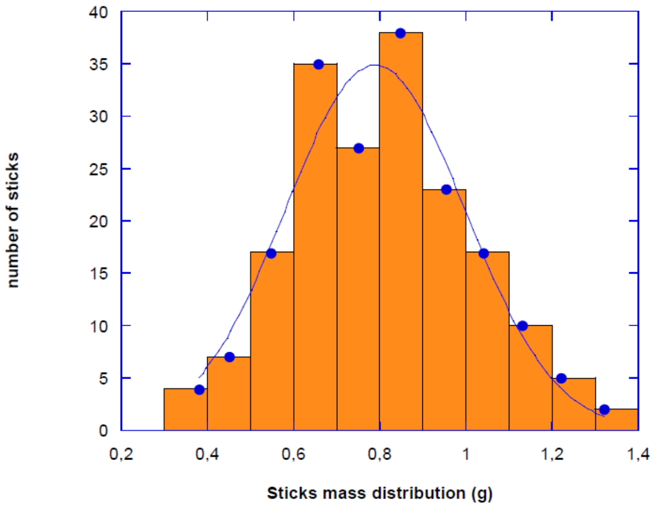

Figure 2 – Sticks histogram with the arithmetic average mass of each interval and the Gauss curve

Table 1 shows the values calculated by Eq. 1 for A, μ, σ and R.

Table 1 – Values of A, μ, σ and R obtained from the sticks mass distribution

	Value	Error
A	18.15	1.31
σ (g)	0.21	0.02
μ (g)	0.79	0.02
R	0.96	NA

Table 2 shows the mass variation of the samples measured during the drying experiment and the results for the percentage of mass loss determined by Eq. 2, using the data displayed in Table 2.

Table 2 – Mass (g) and percentage of mass loss (%) of the samples by drying time (h)

time (h)	sample 1 (g)	sample 1 (%)	sample 2 (g)	sample 2 (%)	sample 3 (g)	sample 3 (%)
0	158.4232	0.0000	157.6756	0.0000	155.5969	0.0000
1	152.2230	3.9137	150.3108	4.6708	147.1147	5.4514
2	148.6853	6.1468	146.5810	7.0363	143.6207	7.6969
3	146.3664	7.6105	145.3444	7.8206	140.9166	9.4348
4	144.2099	8.9717	143.8925	8.7414	138.0504	11.2769
5	142.7918	9.8669	142.5478	9.5942	137.7015	11.5011
6	141.8401	10.4676	141.5237	10.2437	136.9646	11.9747
7	140.9623	11.0217	141.0100	10.5695	136.9243	12.0006
24	140.7420	11.1607	140.3375	10.9961	136.6228	12.1944

Figure 3a shows the mass loss behavior of the sticks. Figure 3b shows the percentage mass loss behavior of the samples. A curve was adjusted over the points for simple viewing. As can be seen, mass loss is not significant after seven hours of drying. The average moisture content of the three samples was 11.45%.

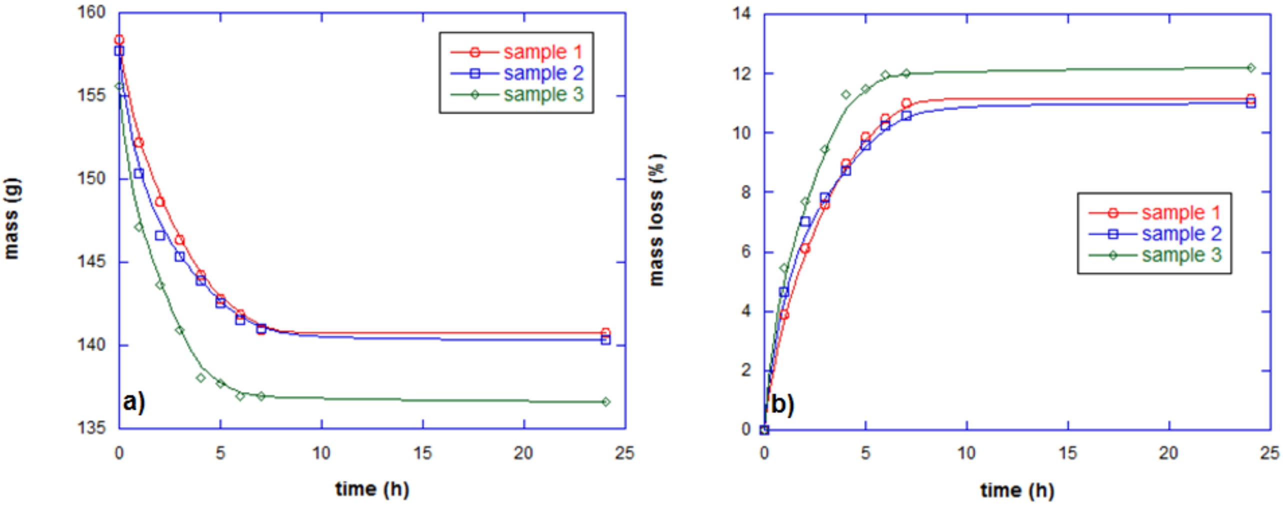

Figure 3 – a) Mass variation behavior of the samples and b) Percentage mass loss of the samples during drying

3.2. Tension and flexural tests

Figure 4a shows the behavior of a taquara subjected to a tension test. The curve presented a linear format and the maximum tension stress coincides with the material disruption, which is characteristic of a fragile material. Interruption in Fig. 4a is from the removal of the extensometer. The rupture of the first fibers can damage the extensometer. Taquara strain in the tension tests ranged from 0.4 to 1.6%. Determination of the maximum rupture tension tress was obtained by the average value of the samples 2, 5, 7 and 9, where the samples were effectively disrupted. In the other samples, there was slippage in the claw. The maximum tension stress was obtained without the extensometer. Figure 4b shows the behavior of the composite in a flexural test. The serrated format observed in Fig. 4b is from the delamination occurred between the taquara sticks and polyester resin, without the actual rupture of taquara sticks.

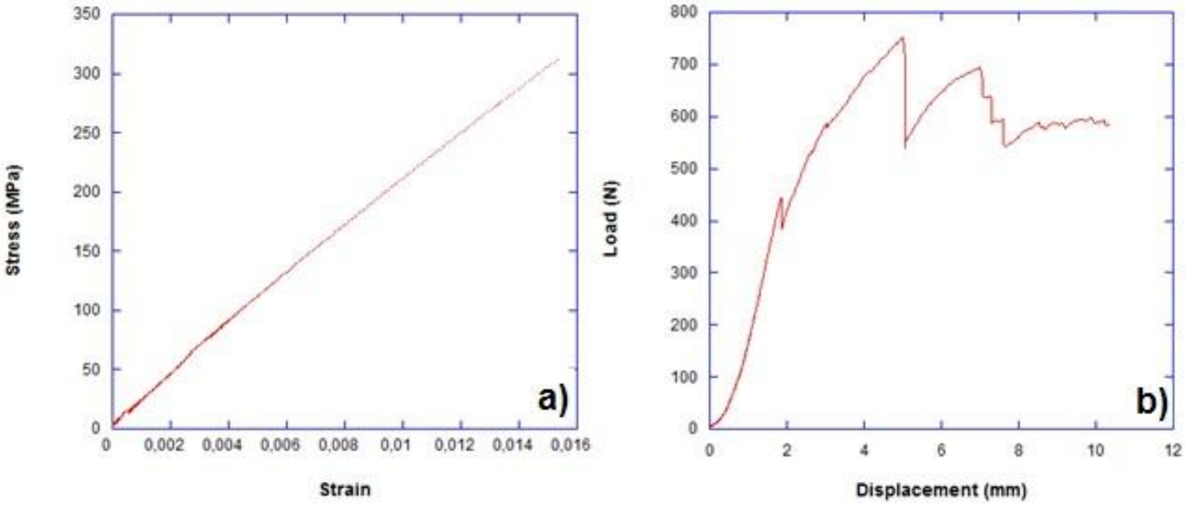

Figure 4 – a) Taquara behavior in a tension test and b) composite behavior in a flexural test

Rupture stress in tension and taquara elasticity modulus are indicated in Table 3.

Table 3 – Taquara tension test data

sample	depth (mm)	width (mm)	length (mm)	σ_T (MPa)	E (GPa)
1	2.0	3.3	50	310.6	21.50
2	2.0	2.5	50	316.6	19.91
3	2.3	4.0	50	175.6	23.92
4	2.1	3.2	50	257.6	20.66
5	2.2	3.2	50	329.4	25.74
6	2.2	3.3	50	261.7	20.34
7	2.1	3.5	50	327.0	21.86
8	2.2	3.7	50	108.4	22.66
9	2.2	3.1	50	243.9	21.74

The flexural stress and flexural modulus of the composites were determined by Eq. 3 and 4, using the data from Table 4.

Tabela 4 – Composite flexural test data

Sample	depth (mm)	width (mm)	Support span (mm)	A_{cross} (mm^2)	F_{max} (N)	σ_f (MPa)	E (GPa)
1	6.9	20.2	105	139.38	757.25	123.5	11.0
2	6.8	20.2	105	137.36	683.06	114.7	9.7
3	6.7	20.1	105	134.67	796.45	138.4	10.9
4	6.8	20.3	105	138.04	760.28	127.0	11.6
5	6.9	20.3	105	140.07	752.15	122.1	8.8
6	7.0	20.3	105	142.45	680.02	107.0	9.7
7	7.4	20.3	111	150.22	820.85	122.6	10.8
8	7.3	20.4	111	148.55	666.20	102.0	8.8

For taquara flexural modulus calculated by Halpin-Tsai equation, determining the volumetric fraction of taquara in the composite, the values of ξ and η, was necessary. For the taquara volumetric fraction calculation, the mass and volume of the composites (Table 5) was used, besides the resin mass fraction and density. Average taquara volumetric fraction was 0.72. For the determination of ξ, A_v was considered as 0.51 and A_h as 0.49, where ξ was 2.8. The values of η for each composite, calculated by Eq. 6, and the taquara flexural modulus by Halpin-Tsai for each composite can be found in Table 5.

Table 5 – Taquara flexural modulus by Halpin-Tsai

sample	length (mm)	mass (g)	volume (cm^3)	η	E (GPa)
1	131.6	16.8	18.34	0.67	21.5
2	131.1	16.7	18.01	0.61	17.3
3	131.7	17.0	17.74	0.67	21.3
4	131.2	16.8	18.11	0.69	23.8
5	132.7	17.0	18.59	0.57	14.9
6	132.2	17.6	18.83	0.61	17.3
7	132.5	18.7	19.90	0.66	21.1
8	132.1	17.9	19.62	0.56	14.8

The average flexural stress and flexural modulus of the composites, resin and taquara, and the tension stress and elasticity modulus of taquara are shown in Table 6.

Table 6 – Average values of the flexural stress (MPa) and flexural modulus (GPa) of the composite, taquara and polyester resin, and the rupture stress (MPa) and elasticity modulus (GPa) in tension of the taquara

Material	**σ (MPa)**	**E (GPa)**
Polyester resin	68.95	2.8
Taquara (tension)	304.2 ± 40.6	22.0 ± 1.8
Taquara (Halpin-Tsai)	----------------	19.0 ± 3.4
Composite	119.7 ± 11.5	10.2 ± 1.0

Figures 5a, 5b and 5c show the taquara cross section microstructure. In the picture, number 1 represents cellulose fibers, number 2 shows the lignin and hemicellulose matrix that bonds the fibers together, number 3 indicates the structure of the inner wall of the taquara, number 4 shows the porosities presented in the structure, known as xylem (RAY *et al.*, 2004) and number 5 the structure of the outer wall of the taquara. A similar electronic micrography of a bamboo cross section was published by Ray and coworkers (2004).

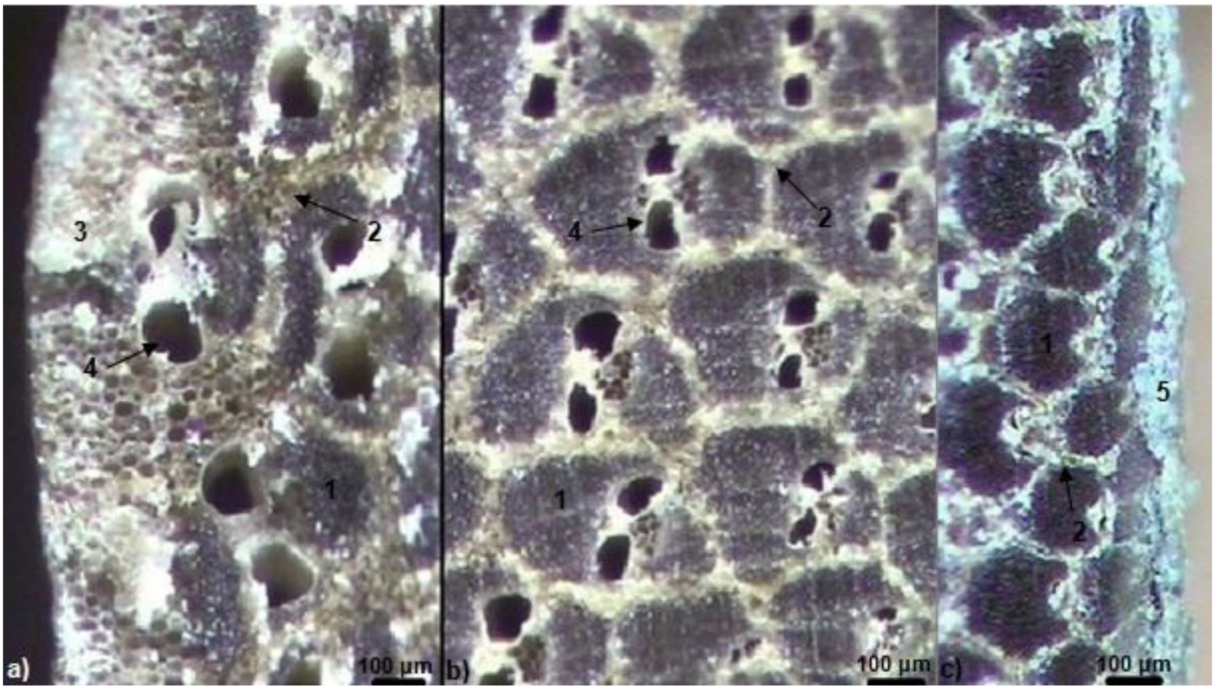

Figure 5 – Cross section taquara micrographs, being a) inner wall of a taquara; b) interior of a taquara; c) outer wall of a taquara

Jayaraman (2003) said that biofibers can be considered as composites with hollow cellulose fibrils (number 1) bonded by a hemicellulose and lignin matrix (number 2). Hemicellulose molecules are hydrogen-bonded to cellulose and act as a cementing matrix between the cellulose microfibrils, forming the cellulose-hemicellulose network, which is thought to be the main structural component of the fiber cell. The hydrophobic lignin acts as a bonding agent and increases the stiffness of the cellulose/hemicellulose composite (JOHN and THOMAS, 2007).

Micrographs show that there was a different structure between the inner and the outer wall of taquara, which shows its anisotropy. Being the distribution and the format of cellulose fibers in the shell region narrower and more compact than in the interior, this region presents little porosity, compared to the inner wall, as observed in Fig. 5. Flexural tests in taquara sticks did not present coherent values for the flexural stress and flexural modulus using the equations for isotropic materials, despite the repeatability in the results.

Several authors have worked with bamboo as reinforcement in polymeric matrix/natural fiber composites, but they did not specify the bamboo species used. Mechanical properties obtained by the authors were lower than those from the composites produced in this study. Wang and coworkers (2008) worked with three different particulate diameters of bamboo, 0.25, 0.42 and 0.84 mm, made composites with PVC and evaluated their flexural properties. Mass fraction of the fibers ranged from 0.5 to 0.7. The composites had low flexural resistance and modulus, and the best result was achieved with particulates of 0.42 mm diameter and mass fraction of 0.5, which resulted in 7.5 MPa of flexural resistance and 1.3 GPa of flexural modulus. Kushkawa and Kumar (2010) worked with laminated bamboo fibers with a cross section of 4.25 x 0.5 mm embedded in a polyester matrix, with a mass fraction of fibers 0.45, and achieved a flexural resistance of 107 MPa and a flexural modulus of 4.6 GPa.

4. CONCLUSION

In the development of this study it was possible to produce polyester resin/taquara sticks composites by cold-press molding in conjunction with transversal pressure of the sticks in order to improve compression. This process allowed for the fabrication of a composite with resin mass fraction of 0.33, which is an advantage over other conventional process, such as RTM, which normally presents an average resin mass fraction of 0.5.

The mass distribution of sticks can be described by a normal distribution curve. The use of statistical results will enable the production of billets with mass repeatability and an estimated quantity of sticks.

Average moisture content obtained was 11.45%, and this value depends on the initial taquara moisture content, which is dependent on cutting and drying time. Defining the minimum 7-hour drying time of the sticks at a given temperature was possible in this study. After this, production of billets was carried out.

Average rupture stress in tension of the taquara was 304.2 ± 40.6 MPa, and the elasticity modulus in tension was 22.0 ± 1.8 GPa.

Average flexural stress of the composites was 119.7 ± 11.5 MPa. Average flexural modulus of the composites was 10.2 ± 1.0 GPa.

Taquara flexural modulus, calculated by Halpin-Tsai equation, was 19.0 ± 3.4 GPa, which is coherent with the results obtained in the tension test. Moreover, the flexural modulus of the composite is an intermediate value of its components, as predicted by the theory.

Mechanical properties of the composite can be improved by eliminating delaminating effects.

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