

NATURAL FIBER FOR REINFORCEMENT IN MATRIX POLYMERIC

Raimundo Nonato Alves da Silva
Universidade do Estado do Amazonas - UEA, Portugal
E-mail: rnasilva@uea.edu.br

José Costa de Macedo Neto
Universidade do Estado do Amazonas - UEA, Brazil
E-mail: jotacostaneto@gmail.com

Solenise Pinto Rodrigues Kimura
Universidade do Estado do Amazonas - UEA, Brazil
E-mail: solenisekimura@yahoo.com.br

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ABSTRACT

The natural fiber market has been growing extraordinarily. Hereupon the current work presents the natural fiber of the periquiteira tree *Cochlospermum orinocense* of the Amazon forest. The chemical composition, physical aspects, morphology, thermal and mechanical properties of this fiber will be discussed. The thermal stability of the fiber samples was about 200 °C. The decomposition of cellulose and hemicelluloses in the fibers occurred at 300 °C and above, while the degradation of the fibers happened above 400 °C. This fiber had good specific strength and good binding properties due to their low weight and presence of high cellulose (60.15wt.%), low lignin (12.03wt.%). More pronounced mass loss indicated the degradation of the amorphous regions of the cellulose, and finally reached a peak of approximately 390 °C.

Keywords: Natural fiber; Chemical composition; Morphology Periquiteira

1. INTRODUCTION

Increasingly, demands for materials from natural, sustainable and environmentally sound sources are gaining more use in the industry as well as reducing the cost of finished products (Faruk *et al.*, 2014, 2002; Mohanty, Misr & Drzal, 2002). The reinforcement with natural fibers in the polymeric matrix was positioned as a source of constant research by several researchers (Nopparut & Amornsakchai, 2016; Ramezani *et al.*, 2013; Scalici, Fiore & Valenza, 2016).

Various natural fibers are available in nature, such as flax, hemp, jute, piaçava, sisal, splice, coconut, and they are also used as reinforcement in the polymer matrix (fávaro *et al.*, 2010; sood & dwivedi, 2018). Periquiteira fiber, scientific name *Cochlospermum orinocense*, has the potential to be used as reinforcement in polymers, especially in the applications where conditions of use are less mechanically required, and partially and/or completely replace synthetic fibers with natural resources. Natural fibers, when incorporated into polymers, can be processed by virtually all conventional methods extrusion, injection, and pressing, and have a lower density than synthetic fibers.

Over the past decade, the automotive industry has changed its strategy and started using natural fibers in place of conventional fibers such as glass and carbon to reinforce a polymer matrix (Ardanuy, Claramunt & Toledo Filho, 2015; Yan *et al.*, 2016). Factors such as increased prices of synthetic fibers have driven the search for the use of vegetable fibers as reinforcement in polymeric materials in the national and international markets. There are several examples of the use of natural fibers as reinforcement in the Brazilian automotive industry, such as internal coatings, panels, ceilings and doors made of plastic reinforced with fibers of cotton, jute, ramie, and coir (Joseph, Medeiros & Carvalho, 1999; Morassi, 1994).

Also in international industries such as Mercedes-Benz and Chrysler have already used natural fibers as components in their products meeting high-quality standards (Prasad *et al.*, 2018; Elanchezhian *et al.*, 2018). However, there are some drawbacks such as moisture absorption, instability at high temperatures and these are some limitations of use (Sawsen *et al.*, 2015). Natural fibers need to undergo chemical treatments and, in some cases, there is a need to use some products to improve the coupling between the polymer matrix and fiber (Fiore, Di Bella & Valenza, 2015; Manalo *et al.*, 2015; Sood, Dharmal & Gupta, 2015).

Biological-based fibers can be classified into two main categories: non-wood fibers and wood fibers bast fibers: flax, jute, and hemp are usually required for higher strength and the best-performing traction and flexural modulus (Biagiotti, Puglia & Kenny, 2004). Depending on their utility, (Faruk *et al.*, 2012) plant fibers are classified into primary and secondary services, e.g.,. hemp, jute, kenaf are used as fibers for primary services. The by-products of some plants, such as coconut, pineapple, etc. belong to the secondary group. The new natural periquiteira fiber is placed in the secondary group as well.

The goal of this work was the mechanical characterization of thermogravimetric, physical, chemical, morphological (scanning electron microscope - SEM) and tensile tests of

the natural periquiteira fiber (*Cochlospermum orinocense*). Therefore, this fiber could be another option in the context of reinforcement with natural fibers (Natural fiber composites - NFCs) for some industries, besides the application of it can be possible.

2. MATERIALS AND METHODS

2.1. Physical analysis of periquiteira fiber

Plant fibers can be found within the stems of monocotyledonous, dicotyledonous plants, and gymnosperm trees in different positions (Akin *et al.*, 2010). The natural fiber from the stem bark of the periquiteira tree (*Cochlospermum orinocense*), and evergreen tree (leaves fall after a prolonged drought) reaching dimensions of 30 m high and 60 cm DBH (diameter at breast height measured at 1.30 m from the ground) in adulthood. It has a shell up to 12 mm thick (contains several fibers). An optical microscope was used to investigate the shape of the fiber (Fonseca *et al.*, 2018). Figure 1 (a) shows the microscopic image of the natural fiber, Figure 1 (b) indicates where the tree fiber is taken, and Figure 1 (c) displays a micrograph.

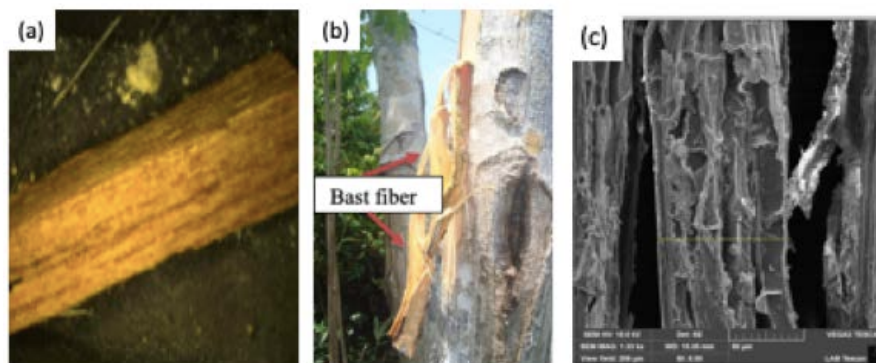
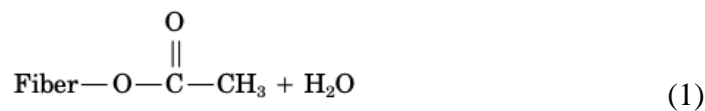


Figure 1: (a) Fiber microscopy (b), natural fiber (*Cochlospermum orinocense*) (c) Fiber cross section measurement

The surface of the outer shell is grey to dark coffee, fluted, rough, longitudinally crimped, easily detached in rectangular plates or strips. The inner bark is fibrous, pink with white streaks. The transverse sections of the fibers were irregular. To measure the fiber cross-sectional area, a sample was obtained before tensile testing and examined using the SEM. To evaluate the variability of the cross-sectional area along the length, 10 fibers were used, and micrographs were made. The average cross section of the fiber is 3.61 mm Figure 1 (c), and the area were determined.

2.2. Chemical analysis of the fiber

This analysis served to determine the content of Cochlospermum orinocense cellulose, lignin, hemicelluloses in addition to conventional chemical and FTIR analyses. The analyses for fiber lignin and cellulose determination were done as follows: As fiber samples were cleaned and oven-dried for 48 hours at temperatures of 55 °C to 60 °C, soon after they were placed in the dissector. In Figure 2 (c), the periquiteira fiber bleached with NaOH + H₂O₂ solution. Treatment with alkaline peroxide (H₂O₂) was due to the need to leave the organophilic fiber. Bonding the fiber with the chemical compound, see Equation 1.



First, 10 g of fiber was added to the hydrogen peroxide solution, next, the sample was stirred at 55 °C for an hour and a half, after that, cooled to room temperature and vacuum filtered, then washed with water distilled to neutral pH, finally, oven drying at 50 °C until reaches a constant mass (weighing every 10 minutes).

2.3. Mechanical Proprieties

After oven drying, the fibers were cut with dimensions of 3.0 mm in width and 10.0 mm in length. Then the fibers were glued with adhesive at the ends of a paper (grammage 180 g/m²) with dimensions of 80.0x25.0 mm. Using a stylus, a cut was made in the center of this paper with dimensions 20.0x20.0 mm. The paper sides were cut to avoid interfering with the assay. The confection of the specimens was done based on the literature (Fonseca *et al.*, 2018). The Figure 3 (b) shows the samples and dimensions for the tensile test.

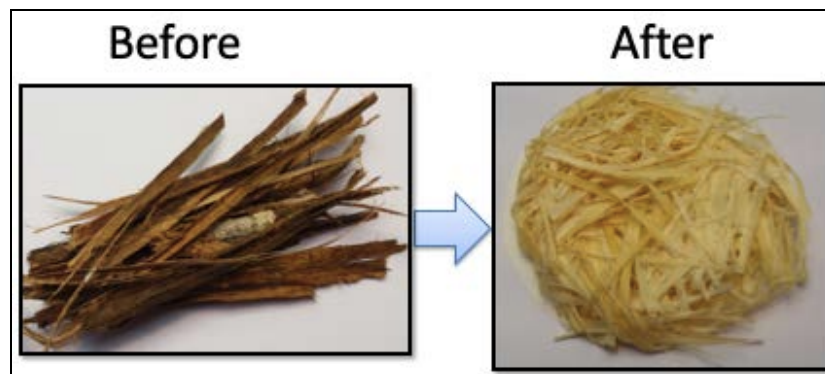


Figure 2: (a): Natural fibers after treatment NaOH + H₂O₂.



Figure 3: (b): Sample preparation for tensile testing

Breaking stress and breaking strain were obtained using a universal tensile Instron machine, model 2630-100, with a load cell of ± 10 kN with 0.01 kgf resolution, test speed 0.5 mm/min. The dimensions of each fiber were inserted into the traction machine software for each test. The Young's modulus was determined from the proportion between the stress at break (σ_b) and Strain at break (ϵ_b) (linear part) obtained from curves of the tensile test (Motta & Agopyan, 2007).

Table 2 show the test method and the results of the tensile test of the fiber compared to the periquiteira. The 1.0 kN load cell was used for tensile testing. Fiber displacement was measured by a short-stroke transducer. Due to the variability of natural fibers, 27 samples were tested, and the mean value was recorded. All tests were performed at room temperature (~ 20 °C) and relative humidity of about 65 %.

3. RESULTS AND DISCUSSIONS

3.1. Fourier transform infrared spectroscopy – FTIR

The FTIR allowed characterizing the chemical structure by identifying the functional groups presented in each sample. The infrared spectra of the natural fiber and chemically treated fiber are shown in Figure 4 (Silva *et al.*, 2020), Figure 5 and Figure 6. Comparatively, they evaluated the jute and Piaçava fibers of the Amazon with the periquiteira fiber and show axial vibrations characteristic of hydroxyl groups of cellulose and hemicellulose. It is possible to notice the presence of the functional groups of the fiber components (Jute, Piaçava and periquiteira) which are composed of alkene, alcohol, aromatics, ketones, and esters, with different functional groups containing oxygen, for example, O-H $3400 - 3200$ cm^{-1} , C=O $1765 - 1715$ cm^{-1} , C-O-C 1270 cm^{-1} , and C-O-H 1050 cm^{-1} (Morán *et al.*, 2008).

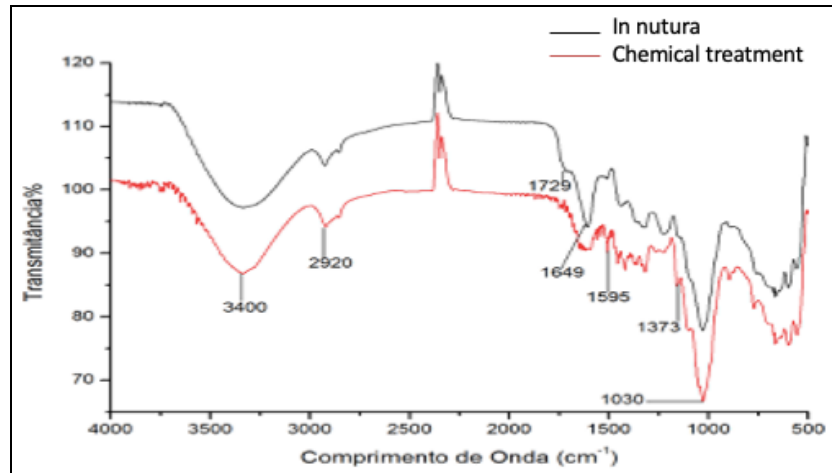


Figure 4: FTIR spectra of periquiteira fiber.

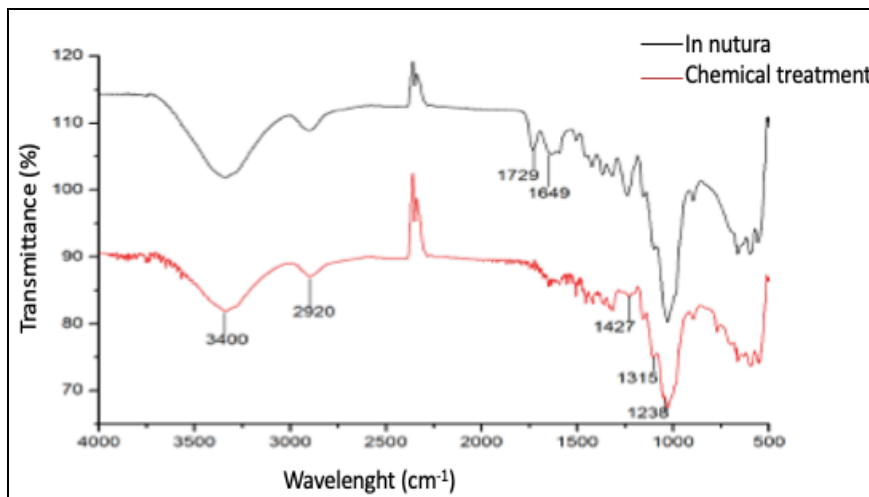


Figure 5: FTIR spectra of jute natural fibers.

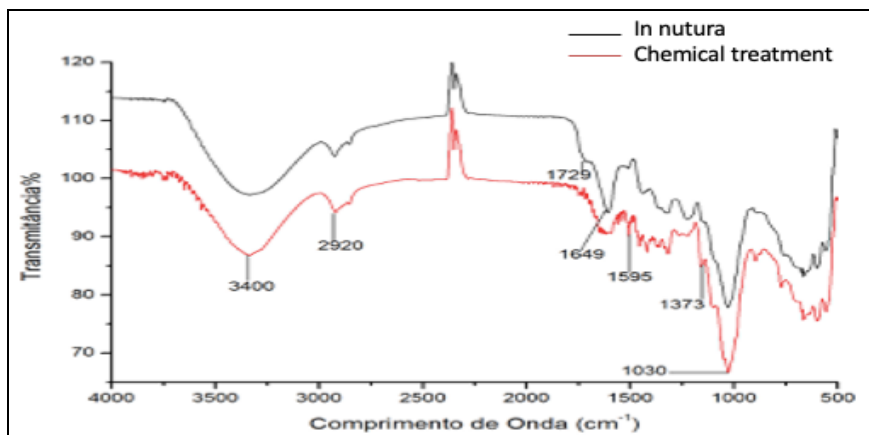


Figure 6: FTIR spectra of Piaçava from amazon

The spectral values of natural fibers start from the 3400 cm^{-1} bands using the FTIR, referring to the vibrational elongation of OH groups presented in cellulose and water. The 2920 cm^{-1} bands are related to the aliphatic C-H portion of the methyl (CH_3) and methylene (CH_2) groups, respectively. The bands of 1507 cm^{-1} , 1450 cm^{-1} and 1238 cm^{-1} are the

removal of lignin in the treated fibers. It is observed that the 1649 cm⁻¹ values of fresh fibers refer to the elongation of carbonyl groups (C=O) presented in lignin. There was no change of cellulose structure as the 1373 cm⁻¹ bands of the periquiteira fiber identify the C-OH flat stretch of crystalline cellulose due to the low intensity of these bands.

3.2. Chemical Analysis

To determine the percentages of thread and cellulose, they were used at (Van Soest, 1963) and measured at 80 °C - 90 °C by a thermometer. The NaOH alkaline treatment was performed on 20 g of fiber were used for 400 ml of 20 g sodium hydroxide solution, then the samples were stirred at 90 ° C an hour and a half, immediately after vacuum filtration, washing in distilled water until it reaches neutral pH and drying in an oven at 50 ° C. Table 1 show the average values in percentage of the components of the periquiteira fiber. Table 2 show the constituents of various natural fibers, which are publicly known as periquiteira fiber. Equation 2, bonding the fiber with a sodium hydroxide molecule.

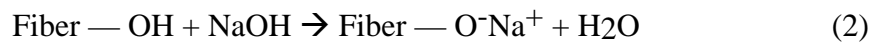


Table 1: Chemical composition

Components	Avg content, %
Fiber (ADF - lignin + cellulose)	71.95
Lignin	12.03
Cellulose	60.15

Table 2: Constituents of Periquiteira natural fibers (wt.%) and important natural fibers

Fiber type	Cellulose (wt.%)	Lignin (wt.%)	Hemicellulose (wt.%)	Pectin (wt.%)	Wax (wt.%)
Coir	46	45	0.3	4	-
Piçava	82.7	45	5.7	5.7	0.6
Abaca	60.8 - 64	12	21	0.8	-
Bamboo	26-43	21-31	15-26	-	-
Musa sepientum	50.15±1.09	17.44±0.19	0.77±0.58	-	-
Bagasse	40	20	30	10	-
Banana	60 - 68	5 -10	6 -19	3 - 5	-
Cotton	82.7±1.09	0.7-1.6	5.7	-	0.6
Kenaf	44-57	15 - 19	21	2	-
Flax	71	2.2	18.6-20.6	2.3	1.7
Palf	70 - 82	5-12	-	-	-
Kapok	13.16	-	-	-	-
Hemp	70.2 - 74.4	3.7-5.7	17.9-22.4	0.0	0.8
Ramie	68.6 - 76.2	0.6-7	13.1-16.7	1.9	0.3
Sisal	67 - 78	8-11	10-14.2	10	2
Jute	61 - 71.5	12-13	13.6-20.6	2.3	1.7
Periquiteira (present work)	60.15	12.03	-	-	-

The lignin and cellulose that corresponded to the sample fractions were weighed. Fiber treatment with solutions is widely used as pretreatment or coating of natural fibers, i.e., removing impurities and increasing defibrillation in the fiber surface area through

solubilization of hemicellulose and lignin improves wettability and adhesion of the fiber-matrix (Miranda *et al.*, 2014; Tita, Paiva & Frollini, 2002), allowing efficient stress transfer between the polymeric matrix and the natural fibers, besides allowing the increase of the stiffness of the fibers.

With the alkaline treatment, under specific conditions of concentration, temperature and agitation, the cellulose expands, and its chains are rearranged, changing the crystalline structure of the cellulose, consequently increasing its amorphous area giving greater resistance, thus producing a rough surface topography and adhesion of the fiber with its polymeric matrix.

3.3. Morphology studies of fiber

The fiber sections were observed using a scanning electron microscope (SEM) model HITACHI S3000H. The fibers were evenly coated with gold on all sides except the cross-sections to make the surfaces conductive. The cross-sections of the fiber were verified at different magnifications according to Figure 8. Surface morphology was also evaluated at different magnifications, as shown in Figure 7. SEM micrographs reveal that the fibers were multicellular in structure and then closed with cellulose and lignin coated hemicelluloses. Using alkaline treatment, under specific conditions of concentration, temperature, and agitation, the cellulose expands, and its chains are rearranged, altering the crystalline structure of the cellulose and, consequently, increasing its amorphous area, providing greater fiber strength and adhesion.

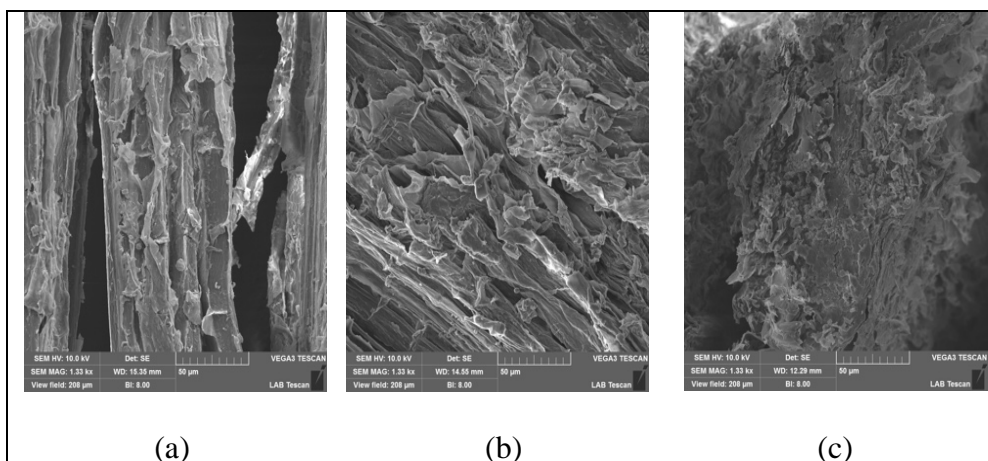


Figure 7: Morphology of lignocellulosic of fiber by SEM: (a) upper view 1300X; surface view 50X; upper view 50X

Cellular structures with small voids were observed on one side of the fiber and microfibrils. The fiber surface is sufficient to give more bonding force between the fiber and the matrix in the polymer composite fabrication.

3.4. Mechanical propriety Test

The results found in the tensile test have presented the variation of modulus of elasticity, tensile stress, and elongation at break (Figure 8). It is worth to note that modulus of elasticity, tensile stress, and elongations at break were selected to study the mechanical properties, in Table 3.

Table 3: Proprieties of the fiber (adapted)

	Width (mm)	Thickness (mm)	Stress (MPa)	Strain (mm)	Young's (GPa)
Avg	3.61	0.29	168.19	0.81	7.09
S-D	0.71	0.08	83,93	0.19	4.04

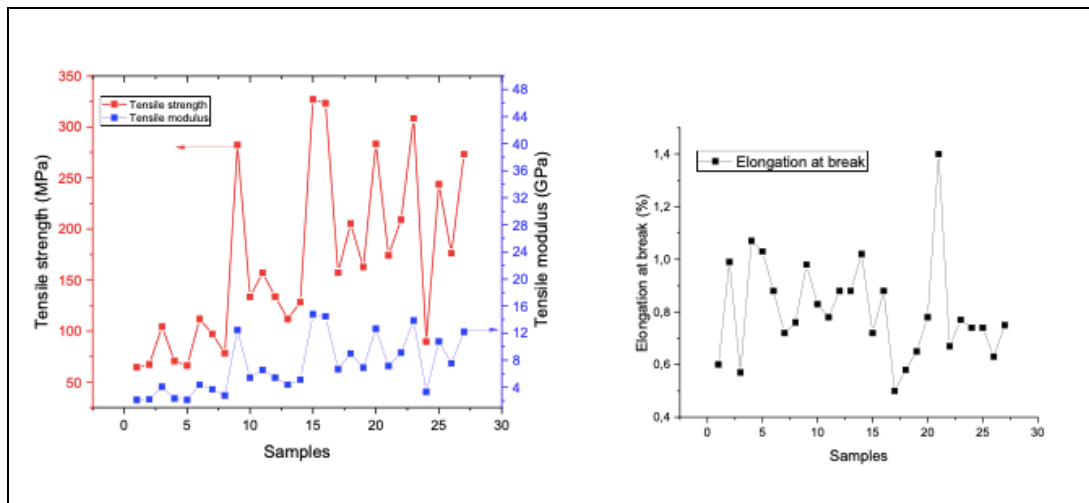


Figure 8: Mechanical properties of fibers; (blue) modulus of elasticity, (red) tensile stress, (black) elongation at break.

Significant values were observed by comparing the values of modulus of elasticity, tensile strength, and elongation with several natural fibres. Table 4 shows the mechanical properties of some lignocellulosic fibers (Satyanarayana, Guimarães & Wypych, 2007), and the data were compared to the fiber of the periquiteira (*Cochlospermum orinocense*).

Table 4: Comparison of the tensile properties of periquiteira with various natural fibers

Natural fiber	Tensile modulus (GPa)	Tensile strength (MPa)	Elongation (%)	Reference
Piaçava	5.6	143	5.6	(AQUINO, [s. d.])
Bamboo	27	575	-	(PERRY; FARNFIELD, 1975)
Kapok	4	93.3	6-8	(MWAIKAMBO; ANSELL, 1999)
Rami	44-128	220-938	2-3	(JAWAID; ABDUL KHALIL, 2011)
Flax	27.6-80	345-1500	1.2-3.2	(JAWAID; ABDUL KHALIL, 2011)
Hemp	70	550-900	1.6	(JAWAID; ABDUL KHALIL, 2011)
Jute	10-30	400-773	1.5-1.8	(HYNESS <i>et al.</i> , 2018)
Sisal	9.4-22	511-635	-	(HYNESS <i>et al.</i> , 2018)
Kenaf	4.3	250	-	(JAWAID; ABDUL KHALIL, 2011)
Coir	4.6	108-252	4.6	(JAWAID; ABDUL KHALIL, 2011)
Musa sepientum	32.703	779.078	2.750	(GUIMARÃES <i>et al.</i> , 2009)
Malva	24.97- 32.94	284.13 – 523.01	1.19	(OLIVEIRA, [s. d.])
Periquiteira	4.04 -7.09	83.93 -168.19	0.19-0.81	Present work

However, the tensile properties of fibers depend on the diameter (mainly in the tensile calculations), angle of the fibers, their volume, their chemical bonds, density, structure failures and the amount of cellulose, and lignin (Satyanarayana, Guimarães & Wypych, 2007; Tomczak, Satyanarayana & Sydenstricker, 2007).

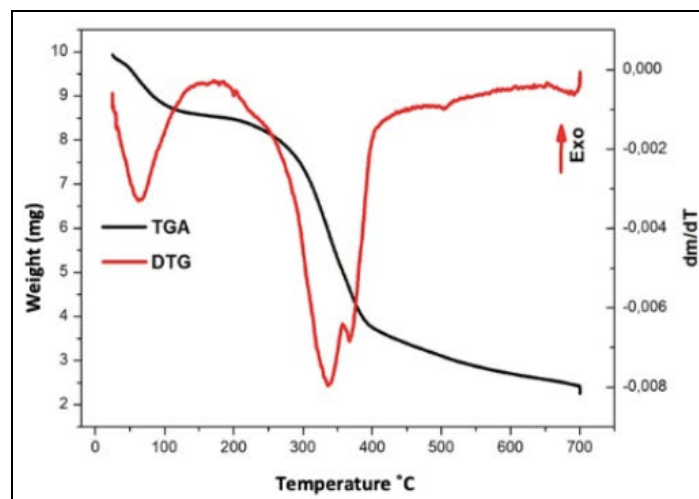


Figure 9: Thermal behavior of the periquiteira fiber

A thermal analysis test was performed to evaluate the behavior of the tree stem extracted fiber (*Cochlospermum orinocense*) employing Thermogravimetric (TG) analysis and the derivative thermo-gravimetric (DTG), on the following behaviors: the first related stowage peak, and there is a slight loss of water-related mass around 100 °C, although the material is fresh, the fiber was thermally stable and at approximately 340 °C another loss was observed. The mass at this temperature leads to the degradation of the cellulose amorphous regions, is related to the degradation of the crystalline phase of the fiber as thermal

phenomena, according to the literature (Nopparut & Amornsakchai, 2016; Yang *et al.*, 2007). This test was carried out under temperature conditions ranging from 10 to 500 °C, in a nitrogen atmosphere, with a heating rate of 10 °C / min, in a thermal analyzer STA 449 F3 Jupiter (NETZSCH).

4. CONCLUSIONS

Physical, chemical, mechanical proprieties and morphological analyses were performed to characterize the fiber as its shape and percentages of cellulose, lignin, and hemicellulose. The chemical analysis performed showed that periquiteira is a lignin rich fiber, showing similarities with lignin rich sisal and jute fibers. Twenty-seven samples of the periquiteira fiber were evaluated in tensile tests and compared with other fibers, obtaining 83.93-168.19 MPa tensile strength, these results are similar to those found in other fibers already researched.

There is good fiber orientation in the same direction, this factor is quite significant for reinforcement materials. In the thermogravimetric analysis (TG) and its thermogravimetric derivatives (DTG), obtaining the following information: the first peak presents a slight water-related mass loss around 100 °C, although the material was fresh, the fiber was thermally stable at 340 °C. The mass at this temperature (~ 340 °C) suffers from the degradation of the cellulose amorphous regions, at approximately 390 °C. According to the literature, the crystalline phase of the fiber is another factor that favors the use of natural fiber as reinforcement.

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