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CHARACTERIZATION OF PLANT FIBERS FROM THE AMAZON REGION FOR USE IN ECO-COMPOSITES

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Abstract: Human-caused climate change is being felt in all regions of the planet. This scenario leads to impacts with losses and damage to all communities. Waste from the construction industry is a cause for great concern due to the large volume wasted and the long degradation time. As a result, scientists are studying ways to mitigate the damage caused to nature, both in terms of the depletion of raw materials and the correct way to dispose of them, in search of renewable resources that can be used to produce new materials that serve this industry and do not severely damage the environment. Plant fibers from the Amazon region are widely used in local handicrafts, textiles and other industries, and are of great interest in studies as reinforcements in polymer, ceramic and cement ecocomposites for application, for example, in the area of coating design in civil construction works. The ongoing study has characterized fibres from coconut (*Cocos nucifera*), curauá (*Ananas comosus var. erectifolius*) and timbo-açú (*Heteropsis flexuosa Araceae*), *in natura*, by the techniques of Stereoscopy, Energy Dispersive Spectroscopy (EDS), X-ray Fluorescence (XRF), X-ray Diffraction (XRD), Mercerization, Scanning Electron Microscopy (SEM) and traction for morphological analysis, chemical composition and application in eco-composites. The results indicate the feasibility of using the fibers studied in eco-composites, as the analyses indicate good interaction between the fibers and a possible matrix.
Keywords: Amazonian plant fibers, Characterization, Ecocomposites, Construction waste

INTRODUCTION

In 2023, the Intergovernmental Panel on Climate Change (IPCC) compiled a report summarizing the state of knowledge on climate change, its widespread impacts and risks, and mitigation and adaptation to these changes. Climate change has caused substantial damage in all regions of the planet and some ecosystems are approaching irreversibility. However, mitigation options, in particular solar energy, wind energy, electrification of urban systems, urban green infrastructure, energy efficiency, demand-side management, better management of forests and plantations/pastures and reduction of food waste and loss, are technically feasible and are becoming increasingly cost-effective and have the support of the general public (IPCC 2023).

The construction industry in Brazil produces around 30% of all the waste generated in the country. According to the Panorama of Solid Waste in Brazil, published in 2022 by the Brazilian Association of Public Cleaning and Special Waste Companies (Abrelpe), Brazil produced around 48 million tons of construction and demolition waste (CDW) in 2021, which is equivalent to 227 kilos of rubble per inhabitant. The report also pointed out that some of this material is abandoned in public areas, causing great impact and concern (Abrelpe 2022).

In the search for alternatives to minimize the environmental impacts caused by construction activities, be it the depletion of raw materials or the improper disposal of materials that are wasted in building works, the scientific community is studying the use of renewable resources that are available in nature or can be cultivated to make new materials that can partially or totally replace the traditional materials used by this industry. In this way, plant fibers offer advantages not only due to their environmental and

socio-economic aspects, but also due to the characteristics of their constitution, low cost, reduced energy consumption in production, biodegradability and stimulation of job creation, especially for socially vulnerable communities (Rajeshkumar *et al*., 2021; Kumar *et al.*, 2021; Queiroz *et al* 2023).

Plant fibers, specifically lignocellulosic fibers, are mainly composed of cellulose, hemicellulose, lignin and other substances such as pectin, inorganic salts, nitrogenous substances and natural dyes. The lignocellulosic constitution is very important for understanding the physical and chemical properties of each plant fiber, as it is used to classify their mechanical, chemical and biological properties, among others. Plant fibers are still used today by the native peoples of Brazil for medicinal, ornamental and artifact purposes, but in large centers they are widely used for handicrafts, clothing, weaving and other purposes (Oliveira *et al*. 2014; Scalioni *et al*. 2017).

The curauá plant (*Ananas comosus var. erectifolius* (L.B.Sm.) Coppens & F.Leal, Bromeliaceae) has been cultivated, generating employment and income through the development of new technologies with socio-economic and ecological benefits. The same is true of plantations and species that germinate through the cycle of nature, such as the coconut (*Cocos nucifera*) and the timbó-açu or titica liana (*Heteropsis Araceae*), which are typical of the Amazon region of Brazil. The particular characteristics of these species have led to their industrial use in the manufacture of paper, ropes, string, handicrafts, geotextiles and automobiles for the production of seat components and car coverings (Germano 2014; Raj, S. *et al*., 2020; Brasil 2022).

Fibers extracted from the husk of the coconut fruit (*Cocos nucifera*), fibers extracted from the curauá leaf (*Ananas comosus var. erectifolius*) and fibres extracted from the timbo-açú vine (*Heteropsis Araceae*) *in natura* were characterized using the following techniques: Stereoscopy, Energy Dispersive Spectroscopy (EDS), X-ray Fluorescence (XRF), X-ray Diffraction (XRD), Mercerization, Scanning Electron Microscopy (SEM) and traction for morphological analysis, chemical composition and feasibility of use in ecocomposites with polymeric, ceramic and cementitious matrices for possible application in the area of coating design in construction works.

DATA AND METHODS

The coconut fruit and the timbó-açú vine were purchased in the city of Belém do Pará at the Ver-o-Peso market, and the curauá leaves, which had been passed through a machine to remove the liquid part of the leaf, were purchased in the municipality of Aurora do Pará (Figure 1). The fibers were removed manually from both plants after proper drying and storage. The average fiber diameters were obtained from 100 samples of each fiber using a Carl Zeiss Stemi 508 optical stereomicroscope with a magnification of up to 50X. The specific mass, in triplicate, of the fibers cut to 10 mm in length and dried in an oven at 70^0 C until a variation of 0.1% of the dry mass was obtained by pycnometry according to ASTM D 854, after 24 hours of immersion in distilled water at 25^0 C ± 1 (ASTM D 854:2014; Queiroz *et al*. 2021). The morphological analyses were carried out using a TESCAN Vega 3.LM scanning electron microscope (SEM) and a preliminary analysis of some of its chemical constituents using a SHIMADZU Energy Dispersive Spectrometer (EDS), which allows the composition of the sample to be identified, albeit qualitatively, at specific points in the image. The chemical elements present in the fibres were also analyzed by X-ray fluorescence (XRF) in a Bruker S2 Ranger X-ray fluorescence machine. X-ray diffraction

(XRD) analyses were carried out to identify the amorphous and crystalline phases of the fibers in a BRUKER D2 PHASER diffractometer with a copper tube (λ=1.5406Å) and an angle of 2θ:5-75°. In the mercerization process, 10 grams of each fiber was treated with a 5% NaOH solution for 30 minutes of constant stirring at 50°C. After the treatment, the fibers were dried in an oven at 70°C for 12 hours and examined using a scanning electron microscope. Ten of the 100 fibers examined under the stereomicroscope were used in the tensile strength tests. The tests were carried out on an AROTEC Model WDW 100E universal machine at a speed of 0.2 mm/min in accordance with ASTM C 1557 (ASTM C 1557:2003).

RESULTS AND DISCUSSION

DIAMETER AND SPECIFIC MASS

The results of the average diameter and specific mass of each fiber are shown in Table 1. The diameter of the fibers, verified by Stereoscopy, varies according to the species analyzed and can vary within the same species, Kumar *et al*. 2021 and Queiroz *et al*. 2021 found similar values in their studies with plant fibers as reinforcement in composites. Figure 2 shows images of the coconut, curauá and timbó-açú fibers captured during the tests and randomly selected from the 100 analyzed to elucidate their dimensions and morphology.

Fibers	Coconut	Curauá	Timbó-açu
Diameter (mm)	0,41 ±0,046	0,15 ±0,031	0,79 ±0,146
Specific mass $(g/cm)^3$	1,3 ±0,12	1,4 ±0,01	1,54 ±0,03

Table 1 - Average diameter and specific mass of fibers

ENERGY DISPERSIVE SPECTROSCOPY - EDS

Table 2 shows a preliminary analysis of some of the chemical constituents of the fibers using the EDS equipment. Elements present in the material such as C, O, Na, Mg, Si, P, K, Ca were found and are common in plant fibers, which vary between species, including in the cell wall of the same fiber. Raj *et al.* 2020 and Queiroz *et al*. 2021 found similar percentages in their studies of plant fibers as reinforcements in composite materials.

Figure 3 shows the chemical element maps and the spectra of these elements obtained by EDS. The map shows the distribution of the elements that prevail in the structure of the fibers and the spectra highlight this quantity in the characteristic peaks. The red coloration of the element carbon, C, which is the main constituent of the walls of plant cells called lignocellulosic, prevails throughout the structure of the fibers and is in agreement with the literature by Mannheimer 2002; Mano 2013 and Seyam 2017 who found similar results in their studies with plant fibers.

X-RAY FLUORESCENCE (FRX)

The data on the chemical composition of coconut, curauá and timbó-açú fibers, obtained by X-ray fluorescence (XRF), is shown in Table 3. We can see the presence of the elements C, O, Na, Mg, Si, P, K, Ca and TI, which are always found in plant fibres, as observed in the EDS analysis, but we can also see the presence of elements such as Fe, Al, Ca, Ni, Cu, among others, which can be found in the fibres in small quantities depending on the soil and climatic conditions of their origin or can be elements that contaminated the fibres during harvesting, transport, storage or characterization techniques. The results are in line with those found by Scipione *et al.* 2012, Pinto *et al.* 2021 and Elfaleh *et al.*2023.

Fibers	Elements (%)											
Elements	C	O	Mg	Yes	P	S	K	Ca	Ti	Fe	Al	Total
Coconut	61,85	37,39	-	0,44	-	-	0,31	-	-	-	-	99,99
Curauá	57,18	42,33	0,1	-	-	0,29	0,53	0,44	-	-	0,06	99,78
Timbó-açu	60,33	39,17	-	-	-	-	-	0,28	-	-	-	99,92

Table 2 - Previous analysis of the chemical composition of some fiber constituents by EDS

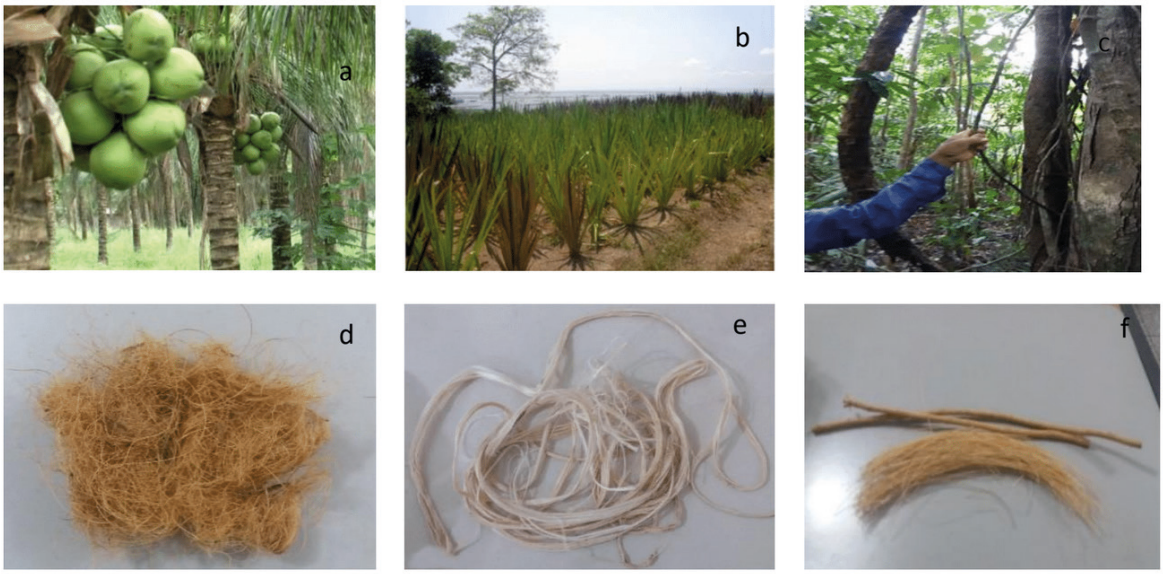

Figure 1 - Plants (a) Coconut - Agrodebate 2015, (b) Curauá - Pentagronomiaunb 2012, (c) Timbó-açú - Pinto 2017, and fibers (d) coconut, (e) curauá, and (f) timbó-açú.

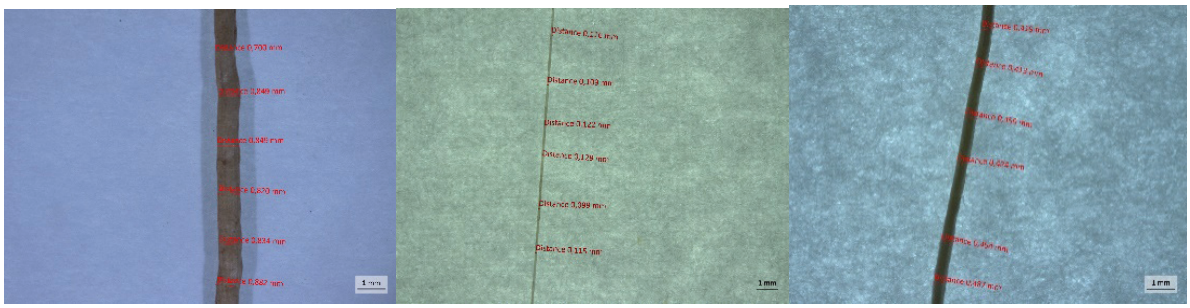

Figure 2 - Stereoscopy of the fibers (a) coconut, (b) curauá and (c) timbó-açú

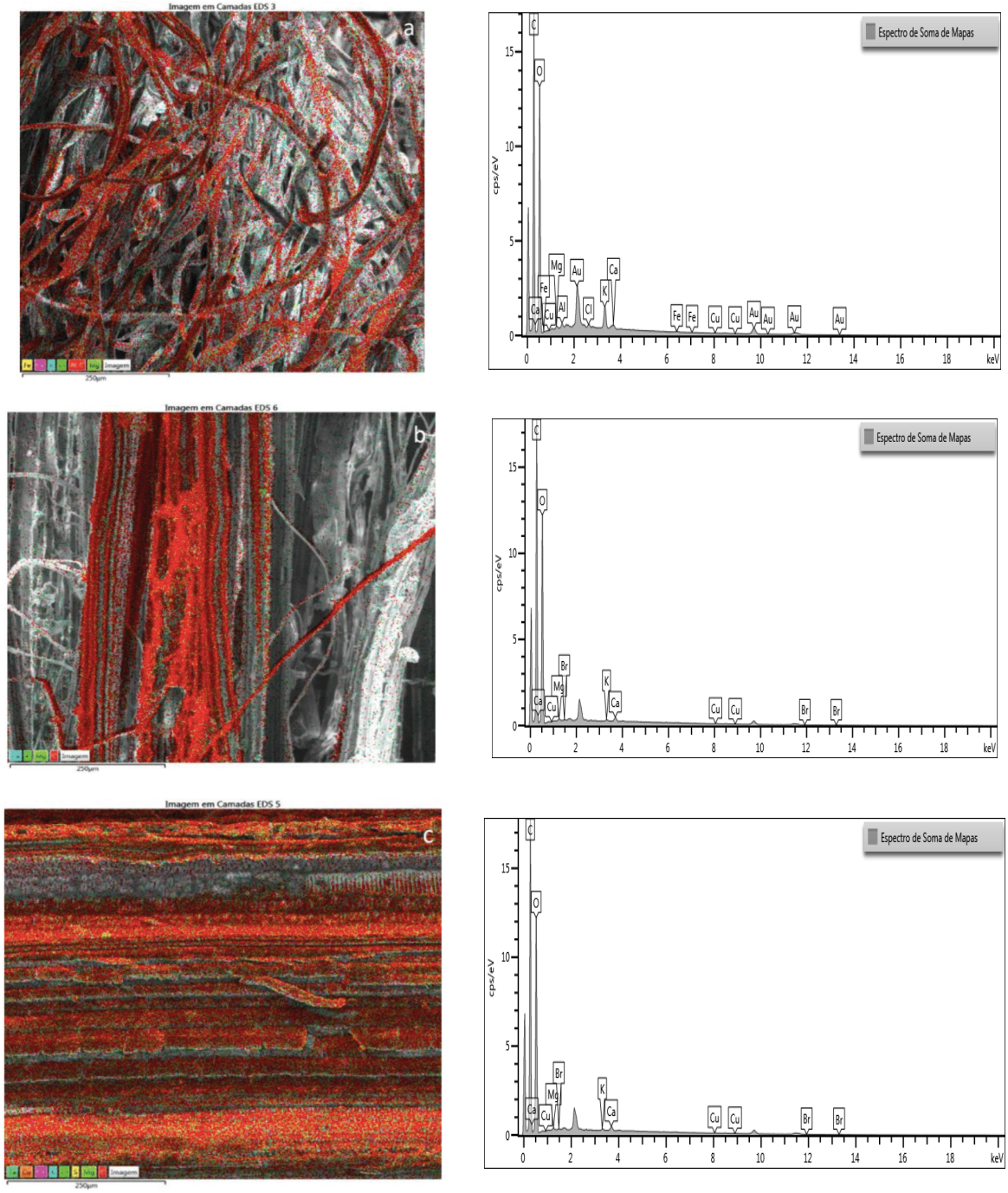

Figure 3 - Map of chemical elements and spectra obtained by EDS. (a) coconut fibers, (b) curauá fibers, and (c) timbó-açú fibers.

Elements (%)	Coconut	Curauá	Timbó-açú
Mg	-	0,0771%	0,0374%
Al	-	0,0215%	0,0134%
Yes	0,2382%	0,0223%	0,0107%
P	0,0269%	0,0465%	0,0175%
S	0,0428%	0,0431%	0,0186%
Cl	0,2079%	0,0414%	0,0039%
K	0,4837%	0,3410%	0,1254%
Ca	0,0868%	0,1336%	0,1203%
Ti	0,0062%	0,0034%	0,0013%
V	-	0,0015%	0,0004%
Cr	0,0039%	0,0052%	0,0010%
Mn	-	0,0132%	0,0006%
Fe	0,0646%	0,0449%	0,0044%
Co	-	0,0007%	0,0003%
Ni	0,0357%	0,0027%	0,0002%
Cu	0,0019%	0,0013%	0,0011%
Zn	0,0238%	0,0059%	0,0008%
Br	0,0010%	0,0004%	-
Sum	100%	100%	100%

Table 3 - X-ray fluorescence of plant fibers

X-RAY DIFFRACTION (DRX)

The coconut, curaua and timbó-açú fibres were characterized by X-ray diffraction to identify the amorphous and crystalline phases shown in Table 4 and Figure 4. The highest Icr (62%) was for the curaua fibre and the lowest Icr (30) for the coconut fibre. The amorphous phase predominates in the first phase, while the quartz phase also occurs in the second phase. Timbo-Açu fibers show an Icr of 49%. Vegetable fibers with a higher crystallinity index tend to have greater mechanical strength. The results found are similar to the literature by Costa *et al.* 2020; Queiroz *et al.* 2021; Queiroz *et al.* 2023 and Elfaleh et al., 2023 who studied plant fibers for use in composites.

Fibers	Amorphous phase	Crystalline phase	I_{cr} (%)
Coconut	Amorphous	Quartz	30
Curauá	Amorphous	-	62
Timbo-açú	Amorphous	Phases not identified.	49

Table 4 - Phases and crystallinity index

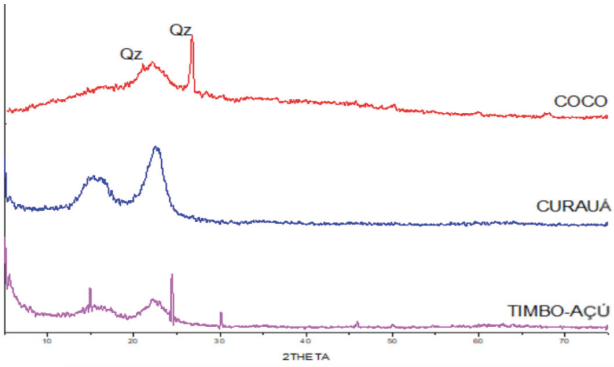

Figure 4 - Diffractograms of plant fibers

MERCERIZATION

Figures 5, 6 and 7 show the fractures obtained by scanning electron microscopy of the coconut, curauá and timbó-açú fibers *in natura* and Figures 8, 9 and 10 after treatment with 5% NaOH. It can be seen that after treatment, the surface of the outermost fibers showed greater roughness, allowing the fibrils to be exposed. Mercerization can cause irreversible morphological changes in the fibres. The treatment causes the fibers to break down into microfibrils. This is due to the partial removal of the lignin and hemicellulose present in the fiber structure, which has the function of a matrix and provides the bonding of the cellulose fibrils. The results indicate that the surface of the mercerized fibers should have a better interaction with a possible matrix, since according to the studies by Mano 2013; Silva, E. *et al.* 2015; Jesus *et al* 2015; Jariwala *et* al 2019 and Elfaleh et al, 2023, the surfaces of plant fibers have become more reactive and tend to improve fiber/matrix adhesion.

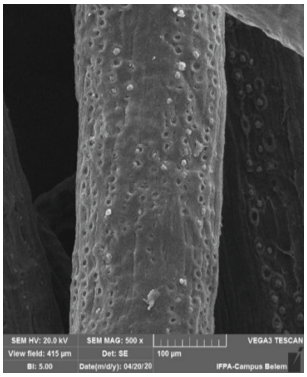

Figure 5 - Fractography of coconut fiber. In natura. 500x

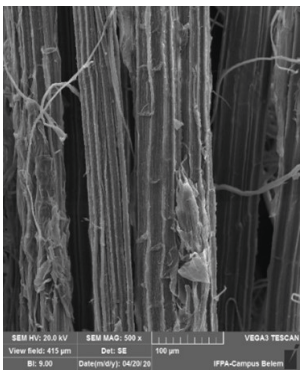

Figure 6 - Fractography of Curauá fiber. In natura. 500x

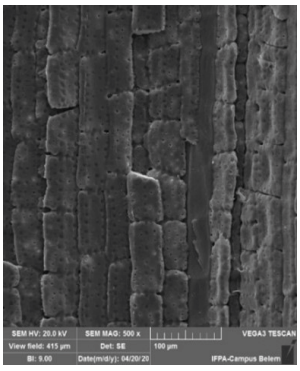

Figure 7 - Fractography of timbó-açu fiber. In natura. 500x

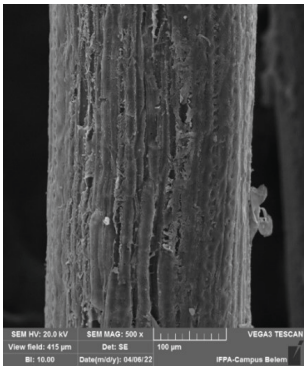

Figure 8 - Fractography of coconut fiber after mercerization. 500x

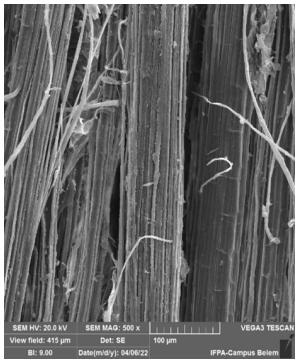

Figure 9 - Fractography of Curauá fiber after mercerization. 500x

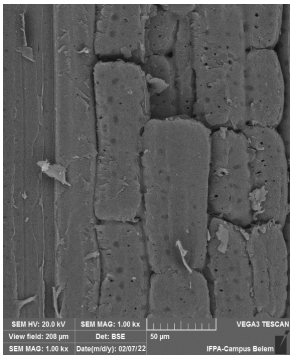

Figure 10 - Fractography of timbó-açu fiber after mercerization. 500x

SCANNING ELECTRON MICROSCOPY (SEM)

The composition of plant fibers defines their physical properties, which in turn depend on their chemical structure. The main chemical components of plant fibers are polar substances such as cellulose, hemicellulose and lignin, and others in smaller percentages such as pectin, wax and water-soluble substances. Each fiber has its own appearance when subjected to the scanning electron microscope, as can be seen in Figures 11, 12 and 13, which show the SEM images of the longitudinal section of coconut, curauá and timbó-açu fibers, respectively. The images show very heterogeneous aspects that vary and depend on the type of soil, the climatic conditions, the fertilizers used, the type of harvest, the size of the leaves, fruit or stem of these plants and are in line with what is found in the literature. All these details define the morphology and chemical composition of the fibers. Coconut fibers have globular particles, cavities and cuticles throughout their structure. Curauá fibers have similar characteristics of straight filaments in the direction parallel to the fibrils, but with some roughness that is typical of these fibers in their natural state. The timbó-açu fibers have a smooth surface on their straight filaments with a rigid appearance, as well as the presence of protruding globules along these filaments. The results are in line with those found by Pereira 2012; Germano 2014; Levy Neto 2012; Pinto *et al.* 2016; Queiroz *et al.* 2021; Queiroz *et* al. 2023 and Elfaleh et al., 2023 in their studies with plant fibers.

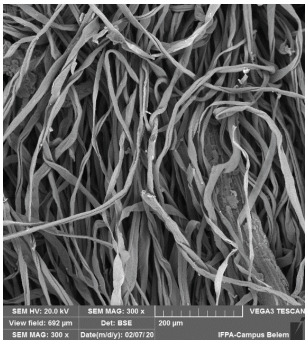

Figure 11 - Fractography of coconut fiber. 300x

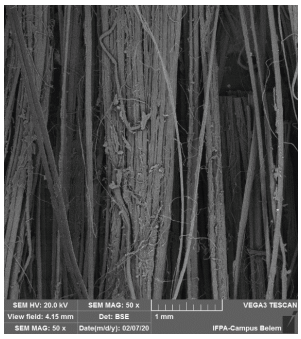

Figure 12 - Fractography of curauá fiber. 300x

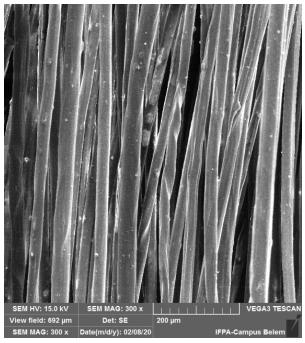

Figure 13 - Fractography of timbó-açu fiber. 300x

The images in Figures 14, 15 and 16 show the cross-sections of the fibers with a 45º angle of inclination of the sample holder. It can be seen that although the natural fibres have a standardized morphology, they differ from each other in terms of their total cross-section, the number of fibrocells and the size of their cell walls. The curauá and timbó-açú fibers have a smaller total area compared to coconut fiber, which has a larger total area, i.e. a greater number of fibrocells, but its cell walls are thinner when compared to the other fibers. The aspects analyzed are in line with those found by Silva *et al.* 2012; Germano 2014; Monteiro *et al.* 2014; Pinto *et al.* 2016 and Queiroz *et al.* 2023 in their characterizations of plant fibres for use in composites, and influence the mechanical strength of the fibres, such as tensile strength. The analyses indicate that curauá and timbó-açú fibers should have better mechanical performance in tensile strength.

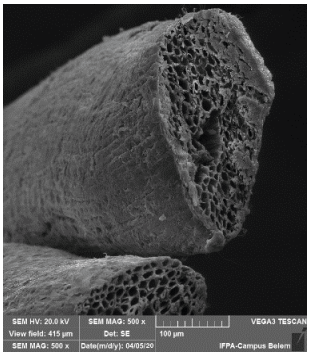

Figure 14 - Fractography of coconut fiber. 500x

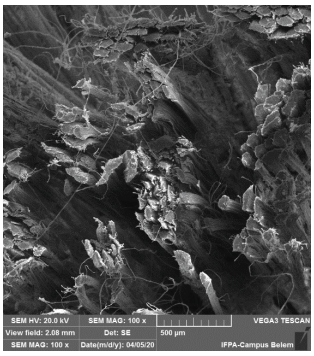

Figure 15 - Fractography of Curauá fiber. 500x

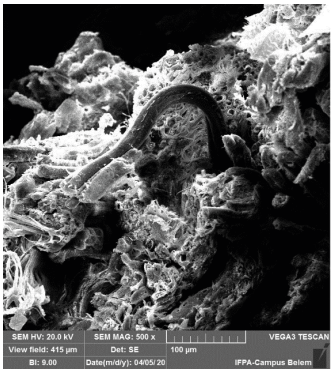

Figure 16 - Fractography of timbó-açu fiber. 500x

TENSILE STRENGTH OF PLANT FIBERS (RTFV)

The curauá and timbó-açú fibers are among the lignocellulosic fibers that showed the highest mechanical tensile strength in this study. Tensile values can also vary between fibers of the same species and are related to the morphological aspects of fiber origin analyzed in sections 3.1 to 3.6. Table 5 shows random images of typical curves generated during the tests and the average tensile strength values of coconut, curauá and timbó-açú fibers, respectively. Curauá fiber was the strongest of the fibers studied. The tensile strength values are similar to those found in the literature by Peças *et al.* 2018 (coconut fiber, 175 MPA); Spinacé *et al.*2011 (curauá fibers, 509 MPA) and Pinto *et al.* 2021 (timbó-açú fibers, 433.96). The results indicate the possibility of using fibers in various components that require greater or lesser mechanical tensile

strength as eco-composite materials that can be applied in civil construction works depending on the choice of a suitable matrix (Mano 2000; Monteiro *et al*.,2008; Levy Neto 2012; Queiroz *et al*., 2023).

Coconut fiber	Curauá fiber	Timbó-açú fiber
173.85±002 MPa	502±104 MPa	432±009 MPa

Table 5 - Typical curves and tensile strength of plant fibers

CONCLUSIONS

The morphological and mechanical aspects of the fibers were observed through the techniques used for the evaluation and allowed us to understand their behavior in order to predict the use of plant fibers from the Amazon region in eco-composites. The chemical composition was verified in the Energy Dispersive Spectroscopy and X-ray Fluorescence analyses and is in agreement with what is found in the literature. The X-ray diffraction analyses will have to be repeated to compare and verify the data as crystalline phases not identified in this study. The aspects analyzed, morphology and chemical composition of the plants directly influence their mechanical characteristics such as tensile strength. The planting and cultivation of the plants studied could generate employment and income in poor communities where the plants are widespread or can easily adapt to soil and climate conditions. The results indicate good interaction between the fibers and a possible matrix and the possibility of using plant fibers, in future work, as reinforcement in the production of ecocomposites with ceramic, polymeric or cementitious matrices that allow them to be used in civil construction works in areas such as cladding design.

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