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Article

Different Fiber Types from the Banana Tree. Characterization and Possible Impacts in Polymeric Composites

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Abstract: The fibers of logs of *Musa sapientum* and *Musa balbisiana* were separated into three parts and analyzed through density, lignin content, moisture, and chemical structure, using Fourier Transform infrared spectrometry (FTIR) and solid low field nuclear magnetic resonance (NMR). NMR analysis showed morphological differences between the two kinds of banana fibers and a longer relaxation time in the Musa balbisiana fibers than the *Musa sapientum* fibers, which can be due to the lower moisture values detected in the former. However, the FTIR technique exhibited no chemical composition differences between the banana fibers spectra and coconut fibers.

Keywords: Natural fiber; banana fiber; chemical characterization; physical characterization.

1. Introduction

The waste valorization and circular economy have been one of the most important approaches to put the many countries into a sustainable path, mainly for an important agribusiness player like Brazil[1,2]. Thinking about waste valorization, several new products have been designed and prepared from wasted biomass such as solid antioxidants [3], products for nanotechnology devices[4], supercapacitors [5,6] or traditional applications as composites[7–11].

Since Brazil is an important banana producer and consumer [12], a large amount of natural fiber has been wasted from banana trees residues. These residues can be different depending on the banana variety, in the same way, that differences in appearance, flavor, and length are observed in the fruit and the pseudo-stem [13]. As well as the organoleptic properties, the structural, physical, chemical, and thermal properties of the fibers from many species can have noted differences. These properties can affect the fibers' behavior in several applications as a reinforcement in composites [14,15], which has also been done with other agricultural wastes [16–19].

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In this article, we analyzed the fibers' chemical and physical characteristics of the most popular banana tree varieties from Brazil, the *Musa balbisiana,* and *Musa sapientum was* known by Brazilians as "banana d'água" and "banana prata" respectively. The fibers were separated from different parts of the pseudo-stem: "external part fiber", "intermediary part fiber," and "internal part fiber".

The focus was to analyze the properties that impact the application of banana fibers in the production of polymeric composites. The samples were characterized by Fourier Transform infrared spectrometry (FTIR) and solid low field nuclear magnetic resonance (NMR), as well as lignin content, moisture, and density.

The introduction should briefly place the study in a broad context and highlight why it is important. It should define the purpose of the work and its significance.

2. Materials and Methods

2.1. Obtaining the banana fibers

The banana tree logs, called pseudo-stems, are formed by layers. For each layer, it is possible to identify five different types of fibers which, in this work, were called "Limit part fiber" (F_{lim}) (Figure 2a), "External lateral part fiber" (F_{LExt}) (Figure 2b), "Internal part fiber" (F_{int}) (Figure 2c), "Intermediary part fiber" (F_{interm}) (Figure 2d) and "External part fiber" (F_{ext}) (Figura 2e). In the present study, the three types of fibers, "Internal part fiber" (F_{int}), "Intermediary part fiber" (F_{interm}), and "External part fiber" (F_{ext}) were used.

Figure 1. Banana fibers milled in knives miller: (a) internal fiber, (b) intermediary fiber, and (c) external fiber.

Figure 2. A sequence of obtaining and separation of the different types of fibers in the banana tree log: (a) "Limit part fiber" (Flim), (b) "External lateral part fiber" (FLext), (c) "Internal part fiber" (Fint), (d) "Intermediary part fiber" (Finterm) and (e) "External part fiber" (Fext).

The *Musa sapientum* and *Musa balbisiana* fibers were unbundled in a forage crusher by Trapp brand, model JK-700, and sequentially milled in a knife's miller by Primotécnica brand, model LP 1003, then sieved until a size of 0.15 mm was reached (Figure 1).

2.2. The fibers characterizations

The *Musa sapientum* and *Musa balbisiana* fibers were characterized by lignin content, moisture content and density measurements, Fourier Transform infrared spectrometry (FTIR), and solid low field nuclear magnetic resonance (NMR).

2.3. Lignin quantification

The lignin was extracted from the banana fibers and quantified through the Klason Method [20]. This method aims at removing the polysaccharides through dissolution in ethanol, extracting the lignin as a residue by acid hydrolysis. The fibers without lignin were weighted after drying at 100°C for 16 h, and the lignin content was quantified by mass difference between the initial fiber and the post-treatment fiber.

The lignin content is directly associated with the hardness of the fiber and moisture content [21].

2.4. Moisture content

The moisture percentage present in the fibers' structure was measured in triplicate assays, following the ASTM D1348 standard.

The moisture content of the crushed samples of the banana fibers was measured by heating in an oven at 80°C of 5,00 g of fiber until a constant mass value was achieved. After the heating process, the moisture content was calculated using Equation 1:

$$Moisture\ (\%) = \frac{M_h - M_d}{M_d} \times 100$$

Where: M_h = sample mass (g); M_d = dry sample mass (g).

2.5. Density

The banana fibers powders densities were determined using pycnometer assays. This method determines the apparent density of solid materials

employing indirect mass and volume measurements in a flat bottom flask (pycnometer), using a known density liquid (ethanol).

This pycnometer method was carried on in triplicate, following the ISO 8962 standard, and the density was calculated using Equation 2:

$$Density\ (g/mL) = \frac{\rho(M_3 - M_1)}{M_3 - M_1 + M_2 - M_4}$$

Where: ρ = density of ethanol under test temperature (g/mL); M_1 = mass of the clean and dry pycnometer (g); M_2 = mass of the pycnometer filled with ethanol (g); M_3 = mass of the pycnometer with 2g of fibers (g); M_4 = mass of the pycnometer with both ethanol and fibers (g).

2.6. Fourier Transform infrared spectrometry (FTIR)

The samples' analysis was carried on using an absorption spectrometer in the region of infrared, from the brand Nicolet Magna 750, with Fourier Transform, scanning the range from 4,000 to 400 cm^{-1}. The fibers were characterized in the form of tablets with KBr.

2.7. Solid low field nuclear magnetic resonance (NMR) – Relaxation time measurements.

The measurements of longitudinal relaxation times for the ^1H nuclei of the fibers were obtained through the inversion-recovery technique in a Maran Ultra spectrometer at 23 MHz, with a rotation speed of 6 kHz and the probe at room temperature (25°C). Forty points were obtained, and the analysis was made in duplicate. The analysis time was from 100 to $5x10^6$µs. The interval time between each waiting time was 5s, and four repetitions were made.

3. Results and Discussion

3.1. Lignin content, moisture, and density

Figure 3 presents the results of lignin content, moisture, and density of fibers from *Musa sapientum* and *Musa balbisiana*. The *Musa balbisiana* has twice more lignin than *Musa sapientum* reaching close to 20% for external fibers. This value is meaningfully higher than has been reported by other researches elsewhere, with a typical lignin content around 11-12% [22–25].

This variation of the lignin contents between this article and the other authors can be explained using different cultivars. However, there is an additional reason for this behavior.

Observing Figure 3, it can be noted that the lignin content increases from internal to external fibers for both species of banana. This behavior is much more evident for the *Musa sapientum* species. The external fiber F_{ext} has twice more lignin per gram than the internal fiber. Considering that in most articles in the literature, the authors use all fibers together, the values obtained at the end of their works are a ponderous average of each fiber's contribution. The *Musa balbisiana* presents similar behavior, but the three fiber types' lignin content is closer and reaching the maximum value of 8.5% for F_{ext} and a minimum lignin content for the internal fiber with 5.0%. The application on composites of the higher lignin content of *Musa sapientum* fibers results in a higher hardness[26].

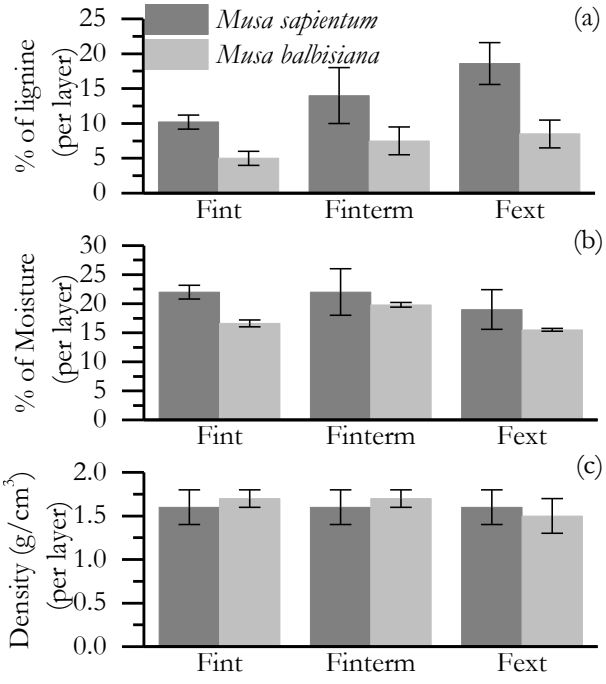

Figure 3. Lignin content, moisture content, and density of the three types of the *Musa sapientum* and *Musa balbisiana* fibers.

3.2. Assessment of moisture

The samples' moisture contents are presented in Figure 3b, and it can be seen from the results that the *Musa sapientum* fibers have slightly higher moisture content than the ones of the *Musa balbisiana*. Moisture values which were found for the *Musa sapientum* fibers (average value of 21.0% in

weight) and the *Musa balbisiana* fibers (average value of 17.3% in weight) are greater than the ones which were reported by other authors in the literature [27–29].

This is due, probably, to the reabsorption of water by the fibers, although all the material was dried by 48 h and placed under 80°C in a stove, and also due to the high relative moisture in the place from which the banana trees were obtained, namely Rio de Janeiro. The relative humidity of Rio de Janeiro in 2011, the year during which the fibers were collected, was of 65–75%. The moisture is important because it tends to improve the biological degradation of fibers.

3.3. Density

The densities of the materials (Figure 3c) are related to its chemical structure and molecular organization. The experimental values for the density of *Musa sapientum* and *Musa balbisiana* fibers were virtually equal in the range of 1.5–1.7 g/cm³. The values found are comparable to most fibers described in the literature and used as composites reinforcement such as bamboo (1.23 g/cm³) [30], sisal (1.3g/cm³)[31], coconut fibers (1.28 g/cm³) [32] and jute fibers (1.4 g/cm³) [33]. The authors have reported that the density varies depending on the part of the fiber, which was analyzed and the harvesting season. Thus, there is no meaningful difference in densities regarding the type of fibers or the banana species.

3.4. Fourier Transform infrared spectrometry (FTIR)

FTIR analyses were carried out to characterize the Musa sapientum and Musa balbisiana fibers' main chemical components. It is known that the components which are found in the fibers are cellulose, hemicellulose, and lignin, and these components present in their structure the hydroxyl functional group. Some of the treatments of fibers (such as mercerization) depend on the reaction of the hydroxyl groups with chemical agents [34].

Figure 4 shows the FTIR specters of the 3 types of *Musa sapientum* fibers compared to coconut fibers, which is broadly known in the literature. The FTIR spectra of *Musa balbisiana* fibers are not shown because there was no meaningful difference between *Musa balbisiana* and *Musa sapientum* spectra

The hydroxyl groups can be observed using the intensity and broad absorption band in the range of 3,500 to 3,000 cm^{-1}. The presence of lignin is confirmed by the characteristic aromatic ring signs in 2,900 and 750 cm^{-1} [35,36].

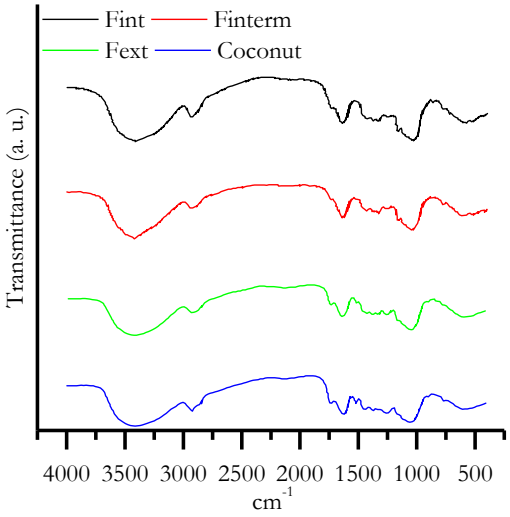

Figure 4. Infrared spectra of the Musa sapientum and coconut fibers.

Although the FTIR difference in the composition of the natural banana and coconut fibers presents few differences among them.

3.5. Solid low field nuclear magnetic resonance (NMR) – Relaxation time measurements

The solid-state NMR technique was used to identify the different chemical structures present in the fibers and verify the crystallinity and the structural organization of these structures.

Table 1. Relaxation time (T^1H) of *Musa sapientum* and *Musa balbisiana* fibers.

Banana variety	Layer of fiber	T_1H
Musa sapientum	"F_{int}"	17.0
	"F_{interm}"	12.0
	"F_{ext}"	28.0
Musa balbisiana	"F_{int}"	23.2
	"F_{interm}"	35.0
	"F_{ext}"	45.0

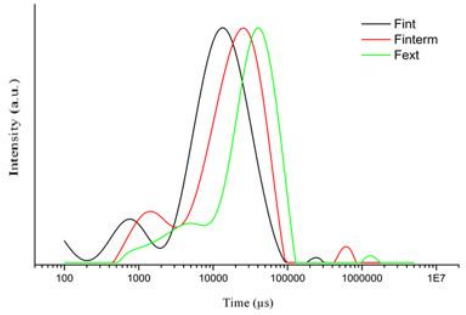

Figure 5. Relaxation domain curves for the Musa sapientum (a) "Fint"; (b) "Finterm"; and (c) "Fext".

Table 1 shows the relaxation time (T_1H) results, which were obtained for the *Musa sapientum* and *Musa balbisiana* fibers. Figure 5 shows the curves with the different domains which were found for each type of fiber present in *Musa sapientum*, and Figure 6 shows the curves with the different domains which were found for each type of fiber present in *Musa balbisiana*.

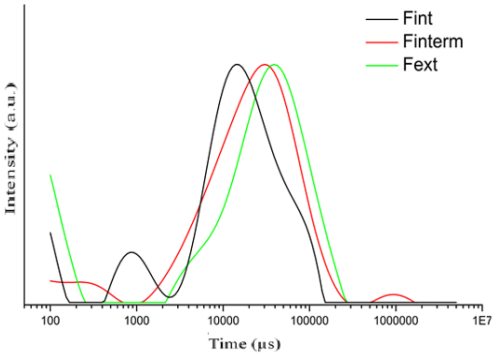

Figure 6. Relaxation domain curves for the Musa balbisiana (a) "Fint"; (b) "Finterm"; and (c) "Fext".

The relaxation times obtained by mono-exponential decay for the *Musa sapientum* and *Musa balbisiana* fibers presented three main domains. Due to the structure of its components, the first domain, which is more shifting, happens due to the moisture which is present in the natural fibers; the second domain, large and with less resolution, is related to the cellulose and hemicellulose of the fibers; and the third domain, less shifting and with longer relaxation time, is related to the lignin content [37,38]. Employing the relaxation times of the ^1H nuclei of the banana fibers (*Musa sapientum* and *Musa balbisiana*), it is possible to follow the changes occurring in the fibers' structure. This also renders possible to relate the lignin contents in the fibers with the structural capacity to relax, which is carried out by measuring the excited spin's times to return to its fundamental stage.

The presence of water in the fiber structure provides higher mobility, which means that the water acts like a plastifying agent in the structure of the natural polymer [39]. Water has only two hydrogens (^1H), thus having a more shifting domain and a lower T_1H time. According to the literature, higher moisture contents in the fibers are connected with greater molecular mobility, due to the greater molecular freedom degree[40]. The moisture contents could not be significantly related to the T_1H results and the molecular chains' mobility because the measured contents were too close regarding the *Musa sapientum* (average moisture of 21%) and *Musa balbisiana* (average moisture of 17%) fibers.

The *Musa sapientum* and *Musa balbisiana* fibers presented lower relaxation times when compared to the coconut fibers T_1H (78.5 ms). This is explained by the presence of higher lignin content (44%) in the coconut fibers composition than in both varieties of banana fibers (5.0% to 18.6%, depending on the fiber types).

Lignin has a much more rigid structure, which is connected to higher relaxation times and thus, to lower chain mobility. This is related to the presence of aromatic rings in its structure [41]. Higher lignin contents in the fibers lead to the increase of the relaxation times, due to a greater stiffness of the chains. The lignin's phenolic constitution acts as a strengthening agent for the cellulose molecules inside the fiber walls, keeping them united [39,41].

The *Musa sapientum* and *Musa balbisiana* fibers of the type "External wall fiber" presented a slightly greater T_1H time than the "F_{int}" and "F_{interm}" fibers. This result is following the lignin content measured in the fibers, where the "F_{ext}" fiber showed a higher lignin content. The fact that this type of fiber has a higher percentage of lignin conferred to this fiber a greater chain stiffness and, consequently, lower mobility in its molecular structure, leading to greater relaxation times of the domains.

The analyses of the T_1H values showed that the fibers have a complex molecular structure, which means that they possess some constituents with a high degree of rigid chains and low mobility, which results in superposed peaks and low definition in the assessments. Due to the peaks' superposition, it was not possible to identify the domains related to cellulose and hemicellulose. The only domain which presented a satisfactory separation was that related to lignin.

4. Conclusions

The results have shown that the techniques which were used to differentiate between the types of banana fibers are efficient for their characterization. Through the solid-state NMR analysis, it was possible to observe that greater lignin contents present nuclear relaxation times, which are bigger than that of the fibers with lower lignin content. The FTIR technique has

not proven to be efficient in differentiating the *Musa sapientum* and *Musa balbisiana* fibers due to the equipment's sensibility, which allows observing the functional groups but not the overall structure.

The banana fibers represent an additional source of biomass for composites reinforcement, which is renewable and has attractive properties, as well as a low cost. They are available in abundance and remain not yet deeply explored.

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Conflicts of Interest

The authors declare no conflict of interest.

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