

Characterization of Brazil Nut Fibers

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Abstract The fruit from Brazil nut (*Bertholletia excelsa*) is characteristically a spherical capsule. When ripe, the capsule releases seeds through its lower portion. In this work, an attempt has been made to characterize the Brazil nut capsule or bur and shell fibers by various techniques. The organic composition was established and elementary composition was determined by Instrumental Neutron Activation Analysis (INAA) followed by gamma-ray spectrometry and Wavelength Dispersive X-ray Fluorescence Spectrometry (WDXRF). Possible applications of these fibers must be considered as novel markets for lignocellulosics have been identified in recent years, representing an exceptional opportunity for sustainable technological development.

Keywords Brazil nut · Lignocellulosics · INAA · WDXRF

Introduction

The agro-industry generates innumerable sources of biomass that are not sufficiently nor adequately utilized. The use of lignocellulosic fibers and their constituents as raw materials in the production of polymeric and composite materials represent an exceptional opportunity for sustainable technological development (Bastioli 1998; Silva et al. 2010). In order to think about potential and future exploitation and applications of lignocellulosics, properties and performance of fibers under environmental or other conditions must be known. The necessity of developing a detailed chemical composition and physical properties data bases of the many natural fibers which are potentially available in Rowell et al. (2000).

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Many lignocellulosic fibers were studied and several of their properties, such as chemical and physical nature and mechanical strength have been already evaluated. The chemical composition of fibers from piassava, sisal, coir, flax, hemp, sugarcane bagasse, banana, curauá, pineapple, cotton lint, bamboo, jute, ramie and rice straw, for instance, were subjected to different important studies (D’Almeida et al. 2006; Satyanarayana et al. 2007; Tomczak et al. 2007; Moura et al. 2010; Corrêa et al. 2010). The available properties of some of these fibers reported so far can be understood in terms of their observed structure.

Besides a small utilization in artistic handicraft, very few works on Brazil nut shell and bur fibers were performed which revealed some further application. One that is possible to mention comes from our laboratory (Inamura et al. 2010).

The Brazil nut (*Bertholletia excelsa*) tree occurs only in the Amazon basin river, thus it is produced in Brazil and also in parts of Bolivia and Peru. The Brazil nut is one of the most valuable non-timber forest products of the Brazilian Amazon and one of the few which reaches the international market, and come entirely from wild collection rather than from plantations.

Brazil nuts, the commercially harvested, edible seed, are considered to be one of the most valuable products that can be harvested from undisturbed rainforest. The tree is large and stands out of other trees tops in the forest and can reach up to 50 m, with a trunk diameter that reaches from 10 to 12 m and it lives over 500 years. The fruits, known to Brazilians as *Castanha do Pará*, are characteristically a spherical capsule, with a thick, hard, dark brown surface (Fig. 1). The fruits are difficult to be

Fig. 1 Brazil nut (*Bertholletia excelsa*) shell and bur fiber



collected: each hard outer bur can weigh over 1 kg and contain between 14 and 25 seeds (nuts) inside, surrounded by yellow pulp that is rich in vitamins, fats and proteins (Vianna et al. 1972; APIZ—Zoro Indigenous People Association 2008).

Brazil nut is an angular nut with a very hard hull. Their nut almond is very white, with a dark brown tegument with high energy content and rich in proteins of high biological values. A tree may produce more than 150 kg of nuts a year and the species is propagated naturally. It is difficult to estimate the nut production because there is a significant variation in burs number from one year to another and among the trees. For example, new Brazil nut trees produce 30–50 burs per year, whereas mature trees, 200–400 years old, can reach up to 1000 burs produce in just one year (APIZ—Zoro Indigenous People Association 2008). In this work, an attempt has been made to characterize the Brazil nut bur and shell fibers by several techniques.

Materials and Methods

Material

Brazil nut shell and bur fibers from the residues disposed by the processing industries of Brazil nuts were provided by Amazon Brazil Nuts. The dry fiber was reduced to fine powder, with particle sizes equal or lower than 200 μm by using ball mill.

Fiber Characterization

The Brazil nut shell and bur fibers were evaluated following the standard procedures and all analyses were performed at least in triplicate. The extractives present in the fiber were removed via Soxhlet extraction with toluene/ethanol for 4 h/70 °C, subsequently replaced by water (100 °C/4 h).

The lignin content was determined by Klason method. This method is one among many others used to evaluate the lignin content, which is based on acid hydrolysis of polysaccharides. Subsequently, the gravimetric determination of soluble and insoluble lignin, according to TAPPI T13M-54 was carried out. For insoluble lignin content, the extractive-free fiber was weighed; after that, 72 % w/v sulfuric acid solution was added, homogenized and maintained in an incubator at 25.0 ± 0.5 °C for 24 h. The solution was transferred to a 1 L flask and 560 mL distilled water was added, followed by a 4 h reflux. The insoluble lignin was washed several times with water and dried in an oven at 105 ± 2 °C until constant weight.

The soluble lignin content was determined by the filtrate obtained from the insoluble lignin percolation. This filtrate was analyzed by UV-Visible Spectrophotometer, model UV-1601, Shimadzu and the absorbance was measured at wavelengths from 280 to 215 nm. The lignin concentrations (g L^{-1}) in diluted samples were calculated by the following expression:

$$C(\text{g L}^{-1}) = \frac{4.53(A_{215}) - A_{280}}{300} \quad (1)$$

$C(\text{g L}^{-1})$ concentration in g/L of soluble lignin Klason in diluted samples
 A_{215} absorbance value at 215 nm
 A_{280} absorbance value at 280 nm.

The total lignin content of the sample was quantified by the sum of soluble and insoluble lignin determinations.

For cellulose content determination, 25 mL of nitric acid-glacial acetic acid were added in 1 g of extractive-free, dried Brazil nut bur and shell fiber. The sample was maintained under reflux at 120 ± 3 °C for 25 min. The system was cooled to room temperature and the cellulose residues were percolated in sintered glass, washed with 500 mL of hot water and then with 25 mL of ethanol. The sample was dried in oven at 105 ± 2 °C until constant weight and then cooled and weighed.

The moisture content of Brazil nuts bur and shell fibers was performed in triplicate. Samples of 1 g were weighed and then placed in an oven at 105 ± 2 °C until constant weight. The samples were cooled in a desiccator and then weighed and quantified.

The determination of density for porous materials of varying composition such as lignocellulosic materials, must consider the pores and microcracks of the samples. The measurement was performed in triplicate. The Brazil nuts bur and shell fibers were dried in the oven at 105 ± 2 °C until constant weight. After dried, the 5 g of samples were completely submerged in graduated cylinders partially filled with distilled water. The assays were maintained in solution for 24 h to determine the variation of the water.

Thermogravimetric Analysis (TG)

Brazil nut shell and bur fibers thermal stability was evaluated by a thermogravimetric analyzer TGA-50 (Shimadzu, Japan). Sample with 5.0 ± 1.0 mg was placed in a platinum cell, under air atmosphere with a flow rate of 50 mL min^{-1} . The experiment was conducted from ambient temperature (25 °C) to 600 °C at a heating rate of 10 °C min^{-1} .

Scanning Electron Microscopy (SEM)

The Brazil nut shell and bur fibers morphological characterizations was accomplished by means of a Philips, XL 30 SEM. Powder sample was fixed in carbon, introduced into a vacuum sputter coater until reach the critical point drying and then coated with gold. Therefore, the analysis was performed using secondary electrons. The scanning electron microscope was equipped with an energy-dispersive X-rays spectroscopy (EDS) system that gathered a spectrum of elements.

Wavelength Dispersive X-ray Fluorescence (WDXRF) Analysis

The pressed powdered samples (grain size ca 100 μm) were prepared according to Scapin et al. (2009). A Rigaku Co. X-ray fluorescence spectrometer, model RIX 3000 was used with the proper measurement conditions (Scapin et al. 2009).

Instrumental Neutron Activation Analysis (INAA)

Aliquots of approximately 200 mg were transferred to polyethylene bags, which had been previously cleaned by leaching with a diluted HNO_3 (1:5) and purified water.

Certified standard solutions (Spex Certiprep) of Br, Cr, Cs, Co, Na, Rb, Sb, Sc, Se, Th were used to prepare elemental synthetic standards. Aliquots (50–100 μL) were pipette on to small sheets of analytical filter paper (Whatman 42) for irradiation. After drying, these filter papers were placed into polyethylene bags.

Irradiations were carried out at the IEA-R1 nuclear research reactor. The thermal neutron flux utilized ranged from 1 to $4 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$. Sample and standards (Br, Cr, Cs, Co, Na, Rb, Sb, Sc, Se, Th) were irradiated together in an aluminum container for 8 h. The ^{82}Br and ^{24}Na were measured after 3 days of decay time, while ^{51}Cr , ^{134}Cs , ^{60}Co , ^{86}Rb , ^{122}Sb , ^{75}Se and ^{233}Pa ($^{233}\text{Th} \rightarrow ^{233}\text{Pa}$) were measured after, at least, 10 days of decay time.

The equipment used to measure the gamma-radiation was a Canberra Model GX2020 hyperpure Ge detector, coupled to a Model 1510 Integrated Signal Processor and MCA System 100, both from Canberra. The detector used had a resolution (FWHM) of 0.9 keV for 122 keV gamma-rays of ^{57}Co and 1.9 keV for 1332 keV gamma-ray of ^{60}Co .

Analysis of the certified reference material IAEA Soil-7 was also carried out simultaneously and, the results obtained were within the range of certified values.

Table 1 Chemical composition (% w/w) of Brazil nut bur and shell fiber

	Brazil nut bur fiber	Brazil nut shell fiber
Cellulose (%)	53.3 (1.1)	38.2 (1.0)
Lignin (%)	35.8 (0.8)	54.1 (1.1)
Extractives (%)	7.5 (0.2)	3.9 (0.2)

Data expressed as mean (standard deviation)

Results and Discussion

Fiber chemical components are distributed through outer cell wall, which is composed of primary and secondary wall layers. Chemical composition differs between different plants and also different parts of the same plant, according to the geographic location, age, climate and soil conditions, among other factors (Rowell et al. 2000).

The Brazil nut shell and bur fibers chemical composition is presented in Table 1. The cellulose content found to Brazil nut bur fiber, 53.3 %, is in the range for jute fiber, 45–63 % (Rowell et al. 2000) and somewhat higher was found for sugarcane bagasse, 54.3–55.2 % (Satyanarayana et al. 2007). For Brazil nut shell fiber, the cellulose content of 40.0 % was similar to the coir fiber (Bledzki et al. 1996).

The lignin content of Brazil nut bur and shell fiber, 35.8 % and 59.5 %, respectively, found in the present work was considerably higher than that from bamboo, 20–25 % (Kozłowski et al. 2004) and 36.14 % for coir fiber (Espert et al. 2004), but inferior to that found in piassava, ~48 % (D’Almeida et al. 2006).

The moisture content and density of Brazil fibers are presented in Table 2. Similar results for moisture were found for cotton, jute or flax, 10.0 % (Bledzki et al. 1996) and curauá fiber, 9.10 % (Spinacé et al. 2009). The density values were in the same range found for banana fiber, 1350 kg m⁻³ (Idicula et al. 2005) and not so far from that of curauá fiber, 1100 ± 91 kg m⁻³ (Spinacé et al. 2009). A major limitation for all cellulosic fibers is the hydrophilic nature if used as reinforcement in plastic. This hydrophilicity influences the all mechanical properties as well as other physical properties of the fiber itself (Bledzki et al. 1996). For that reason moisture determination is an important issue as moisture presence can prevent composite formation (Spinacé et al. 2009).

In general terms, the chemical composition and density values obtained here are consistent with data from other fibers reported in the literature (Rowell et al. 2000; Satyanarayana et al. 2007; Tomczak et al. 2007).

Table 2 Moisture content and density of Brazil fibers

	Moisture content (%)	Density (kg m ⁻³)
Brazil nut shell fiber	14.73 (0.09)	1336 (1)
Brazil nut bur fiber	10.29 (0.03)	1299 (1)

Data expressed as mean (standard deviation)

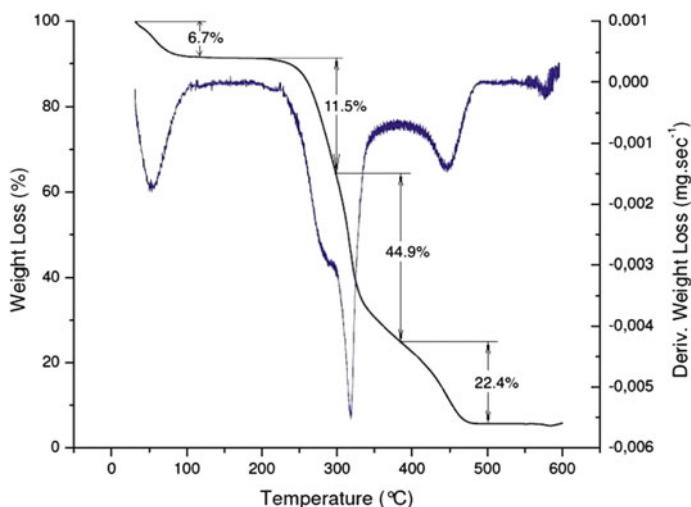


Fig. 2 Thermogravimetric analysis of Brazil nut bur fiber

The thermogravimetric analyses (TG) of Brazil nut bur and shell fiber are presented in Figs. 2 and 3, respectively, as an instrumental output of mass versus increasing temperature. The numerical derivative TG trace is also shown. Distinct transformation regions, all indicating mass loss, can be observed. The first one indicates a weight loss at the beginning of the analysis (about 55–75 °C) and then a thermal stability until 230 °C. The weight loss of 6.7 % at the lower temperatures

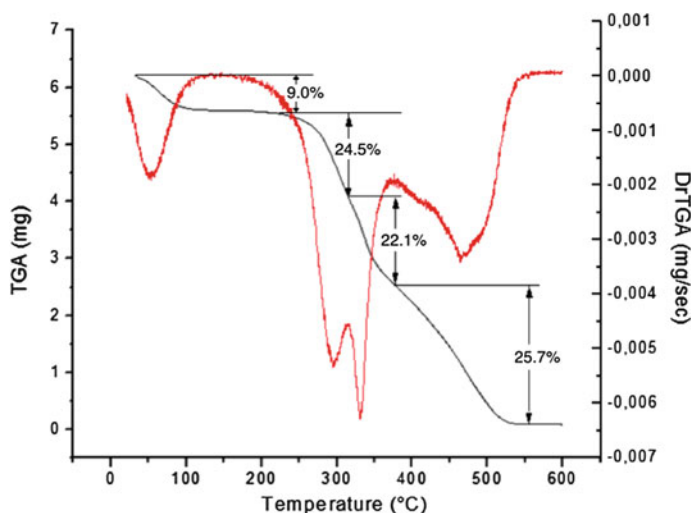


Fig. 3 Thermogravimetric analysis of Brazil nut shell fiber

can be related to water evaporation (D'Almeida et al. 2006; Tomczak et al. 2007; Corrêa et al. 2010). Weight losses (11.5 %) were showed at 260 and 310 °C (44.9 %), and at 426 °C, another weight loss of 22.4 % can also be seen. According to the literature (D'Almeida et al. 2006; Tomczak et al. 2007; Ramiah 1970) the degradation temperature showed at about 260 °C can be attributed to decomposition of hemicellulose. The lignin onset temperatures at 426 °C (Fig. 2) and 451 °C (Fig. 3) are similar as those reported before for lignin degradation (Ramiah 1970). Silva et al. (2010) showed similar degradation temperature stages for sisal fiber/polyurethane resin-based composites and other authors obtained similar results for other lignocellulosic fibers (Satyanarayana et al. 2007; Tomczak et al. 2007).

Figures 4 and 5 shows some cross and longitudinal section microphotographs of the Brazil nut shell fiber. Can be observed that shell fiber present different morphology, depending of the fiber layers. And Figs. 6 and 7 presents cross and longitudinal section microphotographs of the Brazil nut bur fiber. In Figs. 6b and 7 could be observed that the bur fiber presents irregular fibers, in different directions, like polymers cross-linking. And this quality can be given by the difficulty to fracture the fiber. It is possible to see an array of protruded features. These protrusions, shown in detail in Fig. 4d, would be, according to some authors, Si rich particles (Calado et al. 2000).

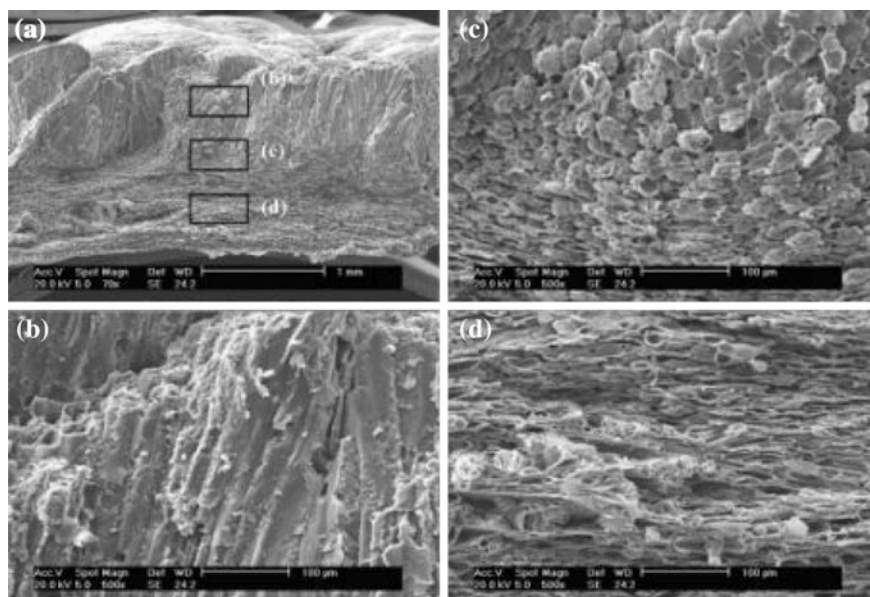


Fig. 4 SEM cross-section images of Brazil nut shell fiber: **a** 70×; **b** superior, **c** central and **d** inferior parts of fiber, 500×

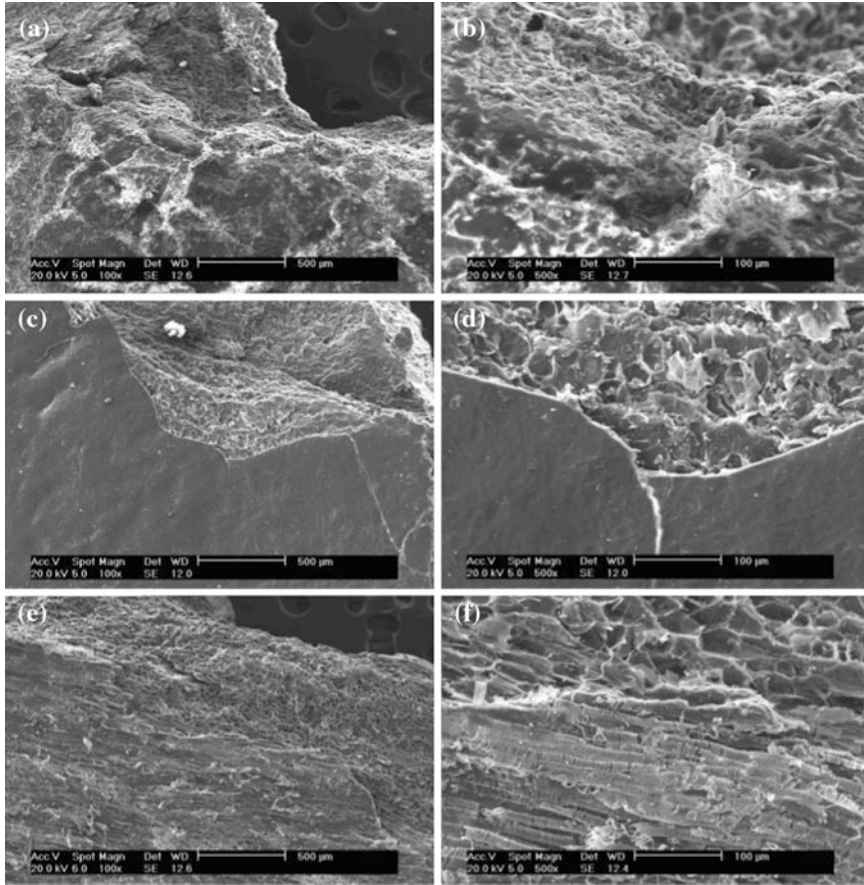


Fig. 5 SEM longitudinal section images of Brazil nut shell fiber: **a, b** external micrograph of fiber, 100× and 500×; **c, d** internal micrograph of fiber, 100× and 500×; **e, f** central micrograph of fiber, 100× and 500×, respectively

Although EDS has reduced accuracy in inhomogeneous and rough samples, the elements identified (Figs. 8 and 9) are consistent with the data found by other methods presented as follows in Tables 3 and 4.

The results obtained by INAA and WDXRF for the elemental composition in both kinds of fiber from Brazilian nut are shown in Tables 3 and 4. Each value is expressed with its uncertainty in parenthesis. In the case of INAA, the uncertainty was evaluated considering the error from statistical counting.

As Skinner and Jahren (2003) pointed out, the process of biomineralization creates heterogeneous accumulations in vegetal tissues with inhomogeneous

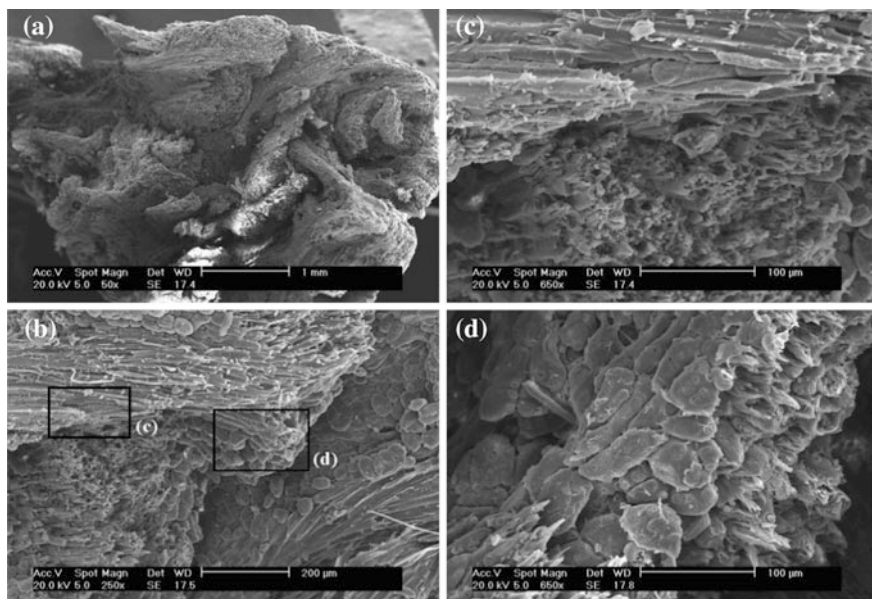


Fig. 6 SEM cross-section images of Brazil nut bur fiber: **a** 50×; **b** 250×; **c** 650×; and **d** 650×

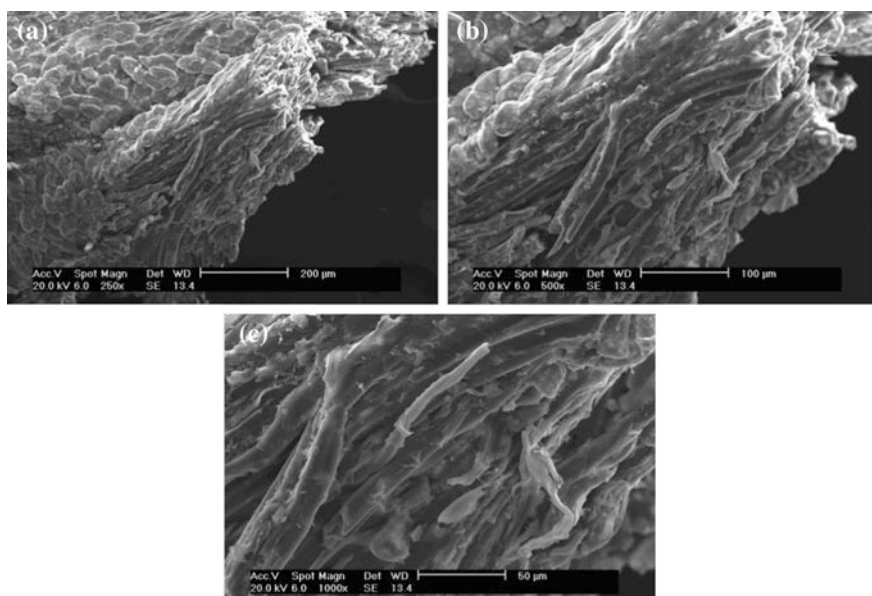


Fig. 7 SEM longitudinal section images of Brazil nut bur fiber: **a** 250×; **b** 500×; **c** 1000×

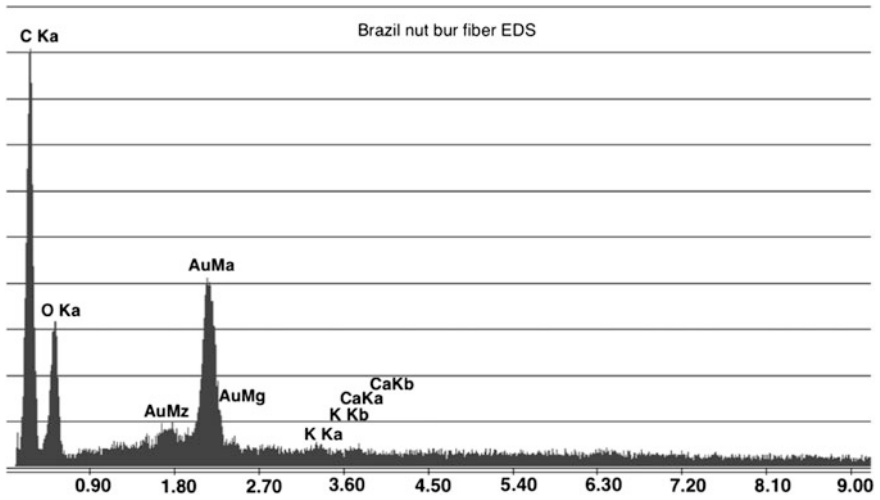


Fig. 8 EDS spectrum of Brazil nut bur fiber

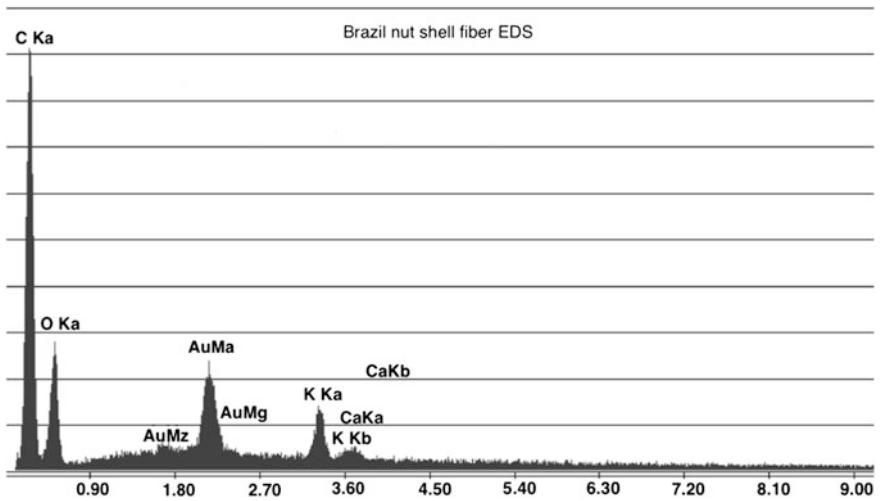


Fig. 9 EDS spectrum of Brazil nut shell fiber

distributions that reflect the environment in which they are formed. Vegetal material can contain carbonate, phosphate, oxalate, silica, iron, or sulfur-containing minerals with particular chemistries. The knowledge of the mineral composition of the fiber

Table 3 Inorganic composition of Brazil nut bur fiber

Element/compound (unit)	Concentration (uncertainty)
Al ₂ O ₃ (μg/g) ^a	1651 (100)
As ₂ O ₃ (μg/g) ^a	16 (10)
Br (μg/g) ^b	87 (4)
CaO (μg/g) ^a	1080 (100)
Cl (μg/g) ^a	697 (30)
Cr (μg/g) ^b	39 (1)
Cs (μg/g) ^b	1.74 (0.04)
Co (μg/kg) ^b	1609 (58)
CuO (μg/g) ^a	25 (10)
Fe ₂ O ₃ (μg/g) ^a	14462 (300)
K ₂ O (μg/g) ^a	8229 (100)
MgO (μg/g) ^a	555 (30)
MnO (μg/g) ^a	67 (10)
Na (μg/g) ^b	49 (5)
NiO (μg/g) ^a	31 (10)
P ₂ O ₅ (μg/g) ^a	447 (30)
Rb (μg/g) ^b	16.3 (0.6)
Sb (μg/kg) ^b	36 (4)
Sc (μg/kg) ^b	9.7 (0.6)
Se (μg/kg) ^b	198 (44)
SiO ₂ (μg/g) ^a	625 (30)
SO ₃ (μg/g) ^a	1996 (100)
SnO ₂ (μg/g) ^a	<10
SrO (μg/g) ^a	<10
Th (μg/kg) ^b	19 (4)
ZnO (μg/g) ^a	21 (10)
ZrO ₂ (μg/g) ^a	<10

^aDetermined by WDXRF^bDetermined by INAA

can be important for the correct analysis of some properties of this material, as inorganic elements are related to metabolism of living organisms.

Comparing present data from bur and shell fibers, it is possible to perceive that the first is richer in Fe₂O₃, and this fact correlate with a stronger red color of that fiber. On the other hand, shell fiber presented a higher content of CaO and K₂O and SiO₂.

Table 4 Inorganic composition of Brazil nut shell fiber

Element/compound (unit)	Concentration (uncertainty)
Al ₂ O ₃ (μg/g) ^a	6533 (100)
As ₂ O ₃ (μg/g) ^a	37 (10)
Br (μg/g) ^b	30 (1)
CaO (μg/g) ^a	6684 (100)
Cl (μg/g) ^a	290 (30)
Cr (μg/g) ^b	2.83 (0.09)
Cs (μg/g) ^b	1.1 (0.1)
Co (μg/kg) ^b	1272 (22)
CuO (μg/g) ^a	111 (20)
Fe ₂ O ₃ (μg/g) ^a	6380 (100)
K ₂ O (μg/g) ^a	34381 (500)
MgO (μg/g) ^a	3214 (100)
MnO (μg/g) ^a	160 (20)
Na (μg/g) ^b	62.3 (0.5)
NiO (μg/g) ^a	83 (10)
P ₂ O ₅ (μg/g) ^a	1735 (100)
Rb (μg/g) ^b	41 (1)
Sb (μg/kg) ^b	20 (2)
Sc (μg/kg) ^b	12.4 (0.4)
Se (μg/kg) ^b	456 (46)
SiO ₂ (μg/g) ^a	2019 (100)
SO ₃ (μg/g) ^a	9356 (100)
SnO ₂ (μg/g) ^a	<10
SrO (μg/g) ^a	34 (10)
Th (μg/kg) ^b	19 (3)
ZnO (μg/g) ^a	87 (10)
ZrO ₂ (μg/g) ^a	<10

^aDetermined by WDXRF^bDetermined by INAA

Conclusion

This work presents an investigation on the thermal behavior, chemical and elementary composition of Brazil nut fibers. Their characteristics are comparable to those of fibers already studied by other groups. The chemical analysis showed that Brazil nut bur fiber is a lignin-rich fiber. This material, nowadays a leftover, could result to be useful as other lignin-rich fibers, considering that many lignocellulosic materials are suited for composite material development. As poor information is available on this kind of fiber until now, the present paper is a contribution to elucidate characteristics of Brazil nut shell and bur fibers.

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