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Waste açaí (*Euterpe precatoria* Mart.) seeds as a new alternative source of cellulose: Extraction and characterization

Sementes de açaí (*Euterpe precatoria* Mart.) como uma nova fonte alternativa de celulose: Extração e caracterização

Semillas de açaí (*Euterpe precatoria* Mart.) como nueva fuente alternativa de celulosa: Extracción y caracterización

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Abstract
Açaí *(Euterpe precatoria* Mart.) is a plant widely cultivated in the northern region of Brazil. Its fruits have been gaining worldwide prominence due to their countless benefits for human health. Consequently, pulp production has increased considerably in recent years, generating significant amounts of waste (mainly seeds). As these residues do not have proper disposal, they are either discarded in the environment or incinerated, causing numerous environmental impacts. To present alternative applications of these residues, this study aimed to evaluate the lignocellulosic contents of açaí seeds – extracts, ash, lignin, cellulose (α-cellulose and hemicellulose) – in addition to extracting and characterizing the cellulose obtained from this abundant residue. The seeds and the extracted cellulose were characterized by several techniques: X-ray fluorescence (XRF), X-ray diffraction (XRD), Attenuated total reflectance with Fourier transform infrared spectroscopy (ATR/FTIR), and thermogravimetry (TGA). In this study, the high potential of using açaí seeds as an alternative source of cellulose was confirmed, since presents 45.5% of this polymer and all the characterization techniques show the purity of the extracted cellulose (type I).
Keywords: Agro-industrial waste; Açaí seed; Cellulose.

Resumo

O açaí (*Euterpe precatoria* Mart.) é uma planta amplamente cultivada na região norte do Brasil. Seus frutos vêm ganhando destaque mundial devido aos inúmeros benefícios para a saúde humana. Consequentemente, a produção de celulose aumentou consideravelmente nos últimos anos, gerando uma quantidade significativa de resíduos (principalmente sementes). Como esses resíduos não têm uma destinação adequada, eles são descartados no meio ambiente ou incinerados, causando inúmeros impactos ambientais. Com o objetivo de apresentar alternativas de aplicação desses resíduos, este trabalho teve como objetivo avaliar o conteúdo lignocelulósico das sementes de açaí - extratos, cinzas, lignina, celulose (α-celulose e hemicelulose) - além de extrair e caracterizar a celulose obtida desse abundante resíduo. As sementes e a celulose extraída foram caracterizadas por diversas técnicas: fluorescência de raios X (XRF), difração de raios X (XRD), refletância total atenuada com espectroscopia de infravermelho por transformada de Fourier (ATR / FTIR) e termogravimetria (TGA). Neste estudo, foi confirmado o alto potencial do uso da semente de açaí como fonte alternativa de celulose, uma vez que apresenta 45,5% desse polímero e todas as técnicas de caracterização mostram a pureza da celulose extraída (tipo I).
Palavras-chave: Resíduo agroindustrial; Semente de açaí; Celulose.

Resumen

El acaí (*Euterpe precatoria* Mart.) es una planta ampliamente cultivada en la región norte de Brasil. Sus frutos han ido ganando protagonismo a nivel mundial debido a sus innumerables beneficios para la salud humana. En consecuencia, la producción de celulosa ha aumentado considerablemente en los últimos años, generando importantes cantidades de residuos (principalmente semillas). Como estos residuos no tienen una disposición adecuada, o son descartados al medio ambiente o incinerados, provocando numerosos impactos ambientales. Para presentar aplicaciones alternativas de estos residuos, este estudio tuvo como objetivo evaluar los contenidos lignocelulósicos de semillas de açaí - extractos, cenizas, lignina, celulosa (α-celulosa y hemicelulosa) - además de extraer y caracterizar la celulosa obtenida de este abundante residuo. Las semillas y la celulosa extraída se caracterizaron mediante varias técnicas: fluorescencia de rayos X (XRF), difracción de rayos X (XRD), reflectancia total atenuada con espectroscopia infrarroja por transformada de Fourier (ATR / FTIR) y termogravimetría (TGA). En este estudio se confirmó el alto potencial de utilizar semillas de açaí como fuente alternativa de celulosa, ya que presenta un 45,5% de este polímero y todas las técnicas de caracterización muestran la pureza de la celulosa extraída (tipo I).
Palabras clave: Residuos agroindustriales; Semilla de açaí; Celulosa.

1. Introduction

Açaí (*Euterpe precatoria* Mart.) is a spherical fruit that contains a light brown seed covered by a thin layer of purple-colored pulp. It is obtained from a palm tree widely found in the Amazon rainforest, which also produces an edible palm heart (Batista, Rapôso, & Liberato, 2017; Lopes et al., 2019).

This fruit, due to its high anthocyanin content, has been termed a superfruit due to its numerous benefits to human health (Boeira et al., 2020). The Northern region of Brazil is the main producer, with Pará and Amazonas states producing around 68% and 22%, respectively, of national production. In 2018, the fruit production reached 221 thousand tons (V. O. Santos et al., 2020), where approximately 180 thousand tons of açaí pulp are consumed annually in Pará state (Buratto, Cocero, & Martín, 2021).

The amount of açaí produced increases year by year, consequently, some problems are inherent to this culture. The pulp and peel represent only 10% of the weight of the fruit, with about 90% of the seed (Rossetto, Maciel, Bortolini, Ribeiro, & Haminiuk, 2020). These seeds end up generating a large amount of waste, where only in 2018, the amount of waste reached 176 thousand tons (Melo et al., 2021).

The main sectors that try to make use of açaí seeds are energy generation and agribusiness, where they are used in boilers and as fertilizers for the soil, but are not able to use the large volume of waste generated (Queiroz et al., 2020). Thus, an immense amount of waste is usually incinerated, resulting in the formation of gases that contribute to the greenhouse effect (de S. Barros et al., 2020), or erroneously discarded, polluting soil and groundwater, as well as causing the proliferation of diseases and pests (Sato et al., 2019).

Due to these environmental issues, several researchers have been studying possible applications for this waste: energy generation using as activated carbon, bio-absorbent to remove metal ions from water (de Souza et al., 2020; do Nascimento et al., 2020; Nagata, Souto, Perazzini, & Perazzini, 2020; Queiroz et al., 2020), transformed into biochar and applied as a soil

conditioner (da Silva et al., 2021; Sato et al., 2019), as a catalyst for biofuel production (Araujo et al., 2021), as downdraft aerator (Itai et al., 2014), as a model of *in vitro* simulated gastrointestinal digestion (Melo et al., 2020), among others.

The use of biomass as raw material for the development of new products is very promising, as it can go beyond adding value, reducing both demands for fossil fuels and emission of greenhouse gases, as well as minimizing solid urban waste (Melo et al., 2021; Oliveira, Aguilar-Galvez, Campos, & Rogez, 2019; Santos, Silva, & Alves, 2017).

Another way of using agro-industrial residues that has been very widespread is the extraction of cellulose, a linear homopolymer with a long chain composed of glycosidic bonds with β-D-glucopyranose units (Turner & Kumar, 2018). The cellulose used industrially is usually extracted from trees, such as pine, eucalyptus, and cotton (Gabriel, Belete, Syrowatka, Neubert, & Gebre-Mariam, 2020a). However, the application of these woods in the generation of energy and construction has increased the need to obtain new alternative sources of cellulose, in addition to the growing concern with deforestation.

This cellulose can be easily extracted from lignocellulosic wastes that are widely available at a low cost, in a sustainable way, in addition to presenting several characteristics – renewable, non-toxic, biodegradable origin, enabling thermal and acoustic insulation (de Azevedo et al., 2021; Tonoli et al., 2012) - enabling subsequent applications. Due to its versatility and for presenting several advantages, this biomaterial has been applied in several areas: medicine, pharmaceutical, and cosmetic industries; it is also used for fuel production, applied as a filler in many thermoplastic products, adhesives, in civil construction, and used as a polymeric reinforcement (Alain, 2013; Han, Yu, & Wang, 2018; Mondal, 2017; Tamilselvi et al., 2019).

The açaí seed has already been characterized in some studies, being mainly composed of cellulose (32 - 45%), hemicellulose (19 - 25%), and lignin (14 - 26%); although depending on the region, these levels may vary considerably (Nagata et al., 2020; Santos et al., 2020). Therefore, it has a high potential for adding value through the production of cellulose. However, to the best of our knowledge, no study has evaluated the viability of the cellulose extraction from this waste, as well as the characterization of this material with high added value.

Thus, the objective of this work was to evaluate the lignocellulosic contents present in açaí seeds, in addition to extracting cellulose by a simple and efficient route, and to characterize the product, confirming a new alternative source to produce this important polymer.

2. Methodology

2.1 Materials

The reagents used were glacial acetic acid (> 99.85%, Sigma-Aldric), sulfuric acid (> 96%, Vetec), ethanol (> 95% vol, Vetec), sodium chlorite (> 80%, Sigma-Aldrich) and sodium hydroxide P.A (98%, Merck).

2.2 Obtaining the raw material

The açaí seeds were donated by a private fruit pulps company in the state of Amazonas. These were dried for 3 h at a temperature of 65 °C in a circulation oven (CIENLAB, model CE 81). Then, they were ground in a knife mill (WILLYW, STAR FT-50) for 3 h and sieved through a 40-mesh sieve.

2.3 Assessments of lignocellulose content and extraction

Extractive content

It was used the methodology applied by the Technical Association of Pulp and Paper Industry, 1997 to quantify the extractives content, where 8 g of the ground açaí seeds were weighed on a paper roll. Then, the rollers were added to the Soxhlet apparatus, where the material was washed with 200 mL of ethanol for 5 h. At the end of this period, the flask with the extract

was added to a rotary evaporator to recover ethanol. After that, the flask was placed in an air-circulating oven (Shimadzu, AUW220D) up to constant weight to determine the content calculation.

Ash content

To calculate the ash content, we followed the methodology described by Tappi, 2007, before adding the ground seeds, the crucibles were weighed to determine the crucible mass. Subsequently, the crucible/seeds were added in a muffle (Quimis, Q318M) with a temperature of 650 °C for 5 h in air atmosphere. In the end, the crucibles were weighed in order to know their mass with the ashes and calculate their content.

Lignin content

To determine the lignin content present in the seeds, 1 g of the seeds were added to a glass beaker with 8.5 mL of sulfuric acid (72%) (w/w) cooled in the refrigerator and stirred for 20 min, and then, left for digestion for 24 h, after this time the digestion mixture was added in a 500 mL flask, adding 150 mL of distilled water. in a heating blanket (70 °C for 5 h), remaining at reflux. After that, the contents of the flask were washed/filtered through a sintered glass filter and a 10 µ pore opening with distilled water to a pH close to that of the water. Subsequently, the resulting filtrate was added to an oven at 100 °C until constant weight (Technical Association of the Pulp and Paper Industry, 2011).

Cellulose extraction

The cellulose content was quantified according to Kumode et al., 2017, where it was used a 500 mL Erlenmeyer with 5 g of the ground sample without extracts, 1 mL of glacial acetic acid, 5 g of sodium chlorite ($NaClO_2$), 180 mL of distilled water, remaining at 75 ºC with stirring. After 1 h of reaction, 1 more glacial acetic acid (mL) and $NaClO_2$ (5 g) were added, repeating this cycle for another 1 h. In the end, the solution remained for 5 h under magnetic stirring. Afterward, the contents remained in an ice bath for 30 min. After that, the contents were filtered and washed with distilled water until reaching a pH close to that of the water in a 40-mesh sieve. Subsequently transferred to Petri dishes, where they were dried in an oven at 80 °C for 6 h.

Alpha-cellulose content

To discover the alpha-cellulose content, 1 g of the extracted cellulose was used and 8 mL of 17.5 wt% sodium hydroxide solution were placed in a mortar. After that, the contents were macerated for 3 min and then filtered through a Büchner funnel (Type 2). In the end, the resultant was washed with water until it reached a pH value close to that of the water and added in a 100 °C circulation oven to dry to constant weight, (Technical Association of Pulp and Paper Industry, 1997).

2.4 Cellulose characterization

The extracted cellulose and ground seeds were characterized by the techniques to verify their composition, as well as the purity of the product obtained using the following techniques:

X-ray fluorescence (XRF)

The ground seeds were characterized by X-ray fluorescence (equipment of the Panalytical brand model Epsilon 3-XL), with a maximum voltage of 50 kV, a maximum current of 3 mA, and helium gas (pressure 10 atm/10 kgf/cm^2) to evaluate the inorganic composition of the raw material.

X-ray diffraction (XRD)

X-ray diffraction measurements were performed on a diffractometer (Panalytical, Empyrem model), operating with Cuka radiation (l = 1.54056 Å), at a voltage of 40kV and a current of 40mA, being controlled by software. The measurements of the samples performed on this equipment are analyzed in the Bragg-Brentano HD mirror mode, in the angular range from 3º to 70º with steps of 0.01313 with 60 seconds each step.

The sample data were normalized and plotted, generating diffractometry graphs with the chart. For the calculation of the Crystallinity Index (CI) of cellulose and the fresh sample, we use Equation 1 (Segal, Creely, Martin, & Conrad, 1959):

$$CI = \frac{CI - Iam}{CI} x100 \qquad (1)$$

where CI is the crystallinity index, C_I is the intensity of the crystalline peak of cellulose (around 2θ: 22°) represented by the crystallinity of the material and I_{am} is the intensity of the amorphous part.

Attenuated total reflectance with Fourier transform infrared spectroscopy (ATR/FTIR)

The ground açaí seeds and fresh cellulose were characterized by Fourier transform infrared spectroscopy with attenuated reflectance spectrophotometer (FTIR-ATR Cary 630 - Agilent). The analyzed spectra were in the range of 4000 to 650 cm^{-1}, with a resolution of 8 cm-1 with 128 scans per sample.

Thermogravimetric analysis (TGA)

Thermogravimetric analyzes (TG-DTG) were used to evaluate the purity and thermal stability of the samples, where approximately 3 mg of sample was used and added in a platinum crucible and inserted in a thermal analyzer (Shimadzu, model TGA-50). The analysis was performed with a heating rate of 10 °C/min, starting from room temperature to 600 °C in a nitrogen atmosphere.

3. Results and discussion

X-ray fluorescence (XRF)

When analyzing the inorganic components present in açaí seeds using the XRF technique, we observe the majority constitution of potassium, silicon, and calcium, as can be seen in Table 1. This high concentration of silicon is probably linked to the silica present in the residue, which is solubilized during the alkaline treatment. (Gabriel et al., 2020a). Similar results were observed for açaí (Buratto et al., 2021; Melo et al., 2021) and other lignocellulosic residues (de S. Barros et al., 2020; Mendonça et al., 2019; Mendonça et al., 2019). It is worth mentioning that the concentration of these elements can vary depending on several factors: location of where the plant is inserted, climate, soil, temperature, and humidity.

Table 1: Concentration of the inorganic elements present in the ground açaí seeds.

Elements	Mg	Al	Si	P	S	Cl	K	Fe	Cu	Br	Ca
wt. %	3.89	4.77	23.67	4.43	4.67	2.78	30.64	1.79	5.15	2.18	16.04

Source: Authors.

It is worth highlighting in Table 2 that the high percentage of potassium and silicon favors the application of this material in the development of catalysts for biodiesel synthesis, and there may be other applications of this residue.

Chemical and physical analysis

When evaluating the lignocellulosic contents present in açaí seeds and comparing them with those obtained in other studies (Table 2), it is possible to observe a significant cellulose content of 66% (45.4% consisting of α-cellulose and 21.1% hemicellulose). Melo et al., 2021, when quantifying cellulosic contents also in açaí seeds, they found an even higher value (85%). Thus, the açaí seed shows itself as an interesting alternative source of cellulose, since the amount of cellulose that constitutes this residue is higher than that observed in other important agro-industrial residues, i.e., sugarcane bagasse, coffee hull, peanuts husks, among others (Table 2). The contents of cellulose and hemicellulose are also important in agro-industrial residues, given the possibility of obtaining compounds with high added value: sugars through their hydrolysis of these polymers (Akhlisah, Yunus, Abidin, Lim, & Kania, 2021; Javed, Ansari, Aman, & Ul Qader, 2019; Pereira & Arantes, 2020), bioethanol (Malik et al., 2021; Zoubiri, Rihani, & Bentahar, 2020) and other derivatives such as 5-hydroxymethylfurfural and levulinic acid (Freitas et al., 2016; Malik et al., 2021; Melro et al., 2020).

The lignin content in açaí seed (24.36%) was similar to that observed in sugarcane bagasse and lower than that observed in coffee husks (Table 2). Several researches are also developed in order to transform this portion into products with added value, such as levulinic acid (Melro et al., 2020), biodiesel (Bhatia et al., 2019; Zhang et al., 2019), and energy generation (Buratto et al., 2021). However, to obtain quality cellulose, the alkaline treatment performed must be highly efficient, since it is at this stage that the cell wall breaks when dissolving the lignin, through the hydrolysis of the esters of acetic and uronic acid and breaks the bonds alpha-ether between lignin and hemicellulose, swelling cellulose (Gabriel et al., 2020a).

The ash content showed a high amount in the açaí seeds, and as the seeds present a high number in the percentage of potassium seen through the XRF analysis, this material has several applications, among them it can be applied as a catalyst in studies in the biodiesel synthesis (Barros et al., 2020; Mendonça et al., 2019). The moisture content (7.17%) presented was similar to that observed in other studies (Table 2).

Table 1: Composition (wt. %) of various agro-industrial wastes.

Wastes	Cellulose	Hemicellulose	Lignin	Extractive	Ash	Moisture	Reference
Açaí seeds	45.49±1.85	21.08±1.59	24.36±2.12	4.48 ±0.11	1.01±0.16	7.17±0.40	This work
Açaí seeds	85.21	-	-	-	1.36	7.91	Melo et al., 2021
Sugarcane bagasse	39.5	23.4	21.0	12.9	3.2	-	Gabriel et al., 2020
Apricot-industrial waste	41.2	29.6	1.85	-	0.81	86.75	Zoubiri et al., 2020
Coffee hull	35.5	14.8	30.7	17.6	1.5	-	Gabriel et al., 2020
Peanut husks	34.18	20.83	31.60	-	2.34	10.95	Julie Chandra et al., 2016
Corn grain	71.77	23.56	0.41	-	1.62	-	Xu et al., 2009
Date seed	49.78	25.03	30.55	15.85	1.33	-	Abu-Thabit et al., 2020
Buriti fibers	58	1	19	6	2	9	Da Cruz Demosthenes et al., 2020

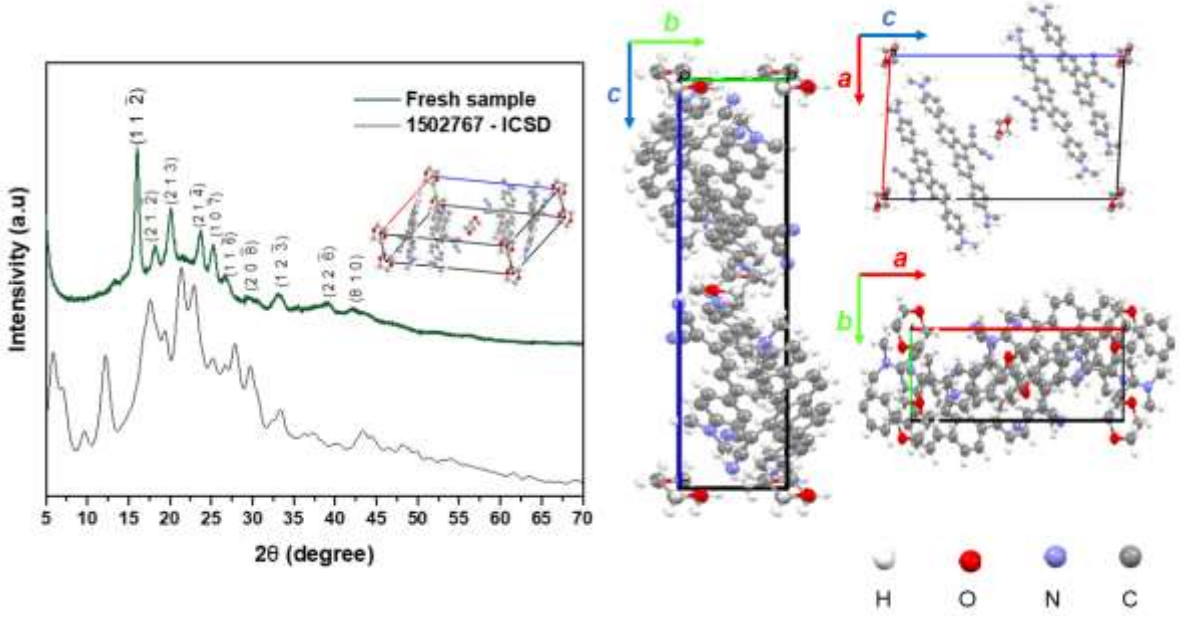
Source: Authors.

We can also see in Table 2 that the açaí residue, compared to other residues from the agroindustry, presents a high percentage of cellulose and a low percentage of extractives, important parameters for paper production, for example.

X-ray diffraction (XRD)

Figure 1 shows the diffractogram of the ground açaí seed and a crystallographic pattern - Crystallographic Information File (CIF) – was associated, identifying the diffraction peaks observed in 2θ values: 15.62°, 18.24°, 20.11°, 23.56°, 25.03°, 26.50°, 29.68°, 32.87°, 38.86°, 42.44° corresponding to (1 1 -2), (2 1 -2), (2 1 3), (2 1 -4), (1 0 -7), (1 1 -6), (2 0 8), (1 2 -3), (2 2 -6), (8 1 0) crystallographic planes, respectively. They were combined with registration 1502767 – Inorganic Crystal Structure Database (ICSD), indicating the formation of monoclinic materials. The diffraction patterns of the material have wide diffraction peaks, indicating a semi-crystalline nature. The reflections observed between 15° and 23° refer to the contribution of crystallographic planes with a crystalline structure of cellulose, which are superimposed on the extended reflection of the non-crystalline structure (amorphous material, i.e., hemicellulose and lignin) (Salama, 2020).

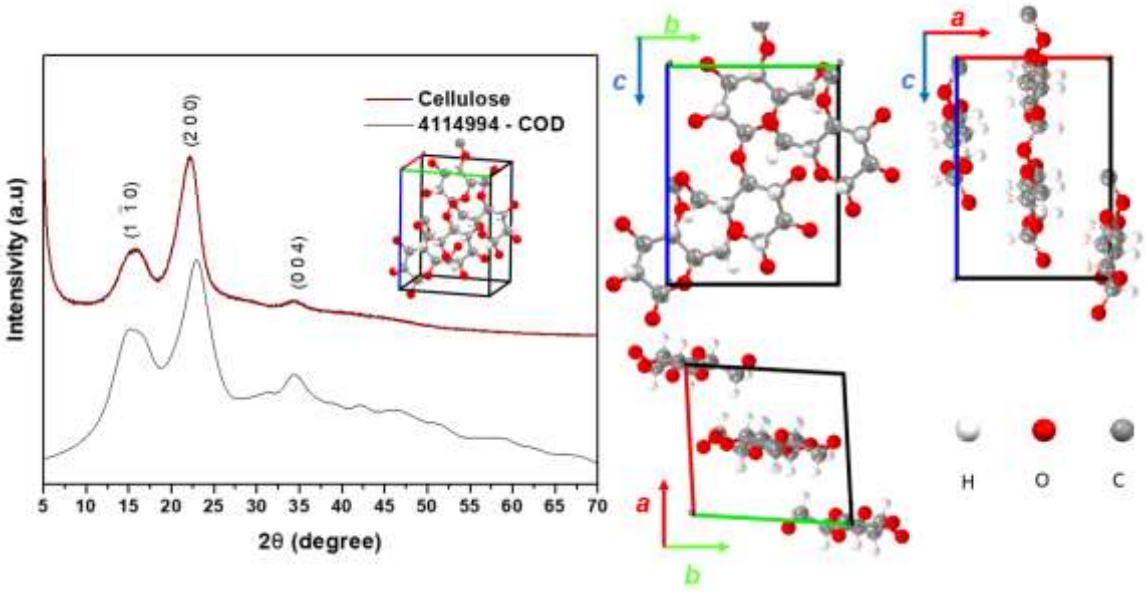
Figure 1: Diffractometry of ground açaí seeds with the respective Crystallographic Information File (CIF), and lattice parameters: a, b, and c.



Source: Authors.

When analyzing the extracted cellulose (Fig. 2) it was evident the intense peaks at the 2θ angles: 15.67°, 22.17°, and 34.37° and the crystallographic planes (1 -1 0), (2 0 0), and (0 0 4). By combining the diffractogram obtained with the registration of 4114994 – ICSD, we confirm that the extracted cellulose presented a semicrystalline structure with characteristics of type I cellulose (TAPPI, 1999). It is important to note the absence of peaks around 29°, normally associated with the presence of mineral impurities such as silica and weddellite (Gabriel et al., 2020a).

Figure 2: Diffractometry of the extracted cellulose, its respective Crystallographic Information File (CIF), and lattice parameters: a, b, and c.



Source: Authors.

Comparing the XRD from the extracted cellulose (Figure 2) with the fresh seeds (Figure 1.), it is possible to observe the removal of amorphous materials and the elevation of the characteristic peaks of cellulose, showing successful isolation of cellulose and increased crystallinity.

Depending on the treatment applied to the residue for cellulose extraction (i.e. thermal, mechanical, and/or chemical), changes in unit cell dimensions and crystalline structure in large or smaller proportions can be observed, which can result in different cellulose polymorphs, such as Iα, Iβ, II, III, IV, and V (Silva & D'Almeida, 2009).

Table 3 describes the lattice parameters of the structures obtained through the XRD, wherein seeds the dominant structure is monoclinic with the following lattice parameters: a = 18,382 Å, b = 5.7303 Å, c = 25.034 Å with volume 2.634.01 Å and spatial group P 1 1 2_1 (4) in three projections a, b, and c. For the extracted cellulose, the crystalline structure is also monoclinic. However, the lattice parameters are quite different: a = 7,784 Å, b = 8,201 Å, c = 10,380 Å resulting in a smaller cell volume (658,299 Å) and space group P 2_1/n (14).

Table 2: Lattice parameters for cellulose and açaí seed.

Sample	ICSD*	Spacial group	Formula	a (Å)	b (Å)	c (Å)	Volume (Å^3)	Reference
Açaí Seed	1502767	P 2_1/n (14)	C_{32} H_{26} N_4,0.5(C_4 H_8 O_2)	18.382(4)	5.7303(11)	25.034(5)	2634.01	Xu et al., 2010
Cellulose	4114994	P 1 1 2_1 (4)	C_{12} H_{14} O_{10}	7.784(8)	8.201(8)	10.380(10)	658.299	Nishiyama et al., 2002

*Inorganic Crystal Structure Database
Source: Nishiyama et al., 2002 and J. Xu et al. (2010).

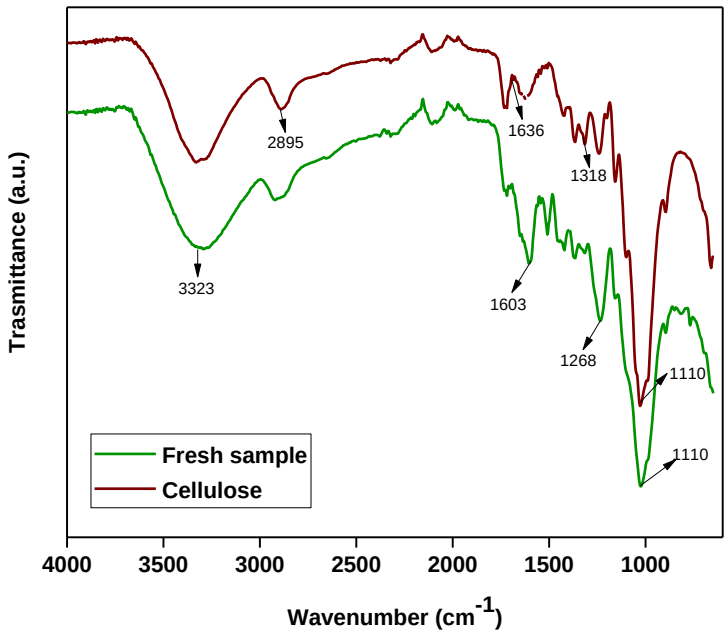
Applying the Segal equation, we calculated the crystallinity of the two samples (ground seed and cellulose), where the first showed crystallinity of 34%, with an increase in the crystallinity of the material to 46% after the cellulose isolation. These

data prove that it is possible to obtain pure cellulose, gaining crystallinity. The intensity and increased crystallinity of the peaks corroborate the events observed in the FTIR analysis, where the lignin bands in the cellulose sample disappeared. They were also observed in the degradation events in the thermogravimetric analysis, since, for the sample for cellulose, the presence of lignin was not identified. When comparing them with data from similar studies, we observed a similar behavior for cellulose extracted from the curacao fiber, pineapple leaf and date seeds (Corrêa, Teixeira, Marconcini, Pessan, & Mattoso, 2009; Ana Carolina Corrêa, de Teixeira, Pessan, & Mattoso, 2010; Fethiza Tedjani, Ben Mya, & Rebiai, 2020; R. M. dos Santos et al., 2013). Therefore, it is evident that the method used to isolate the cellulose was carried out successfully.

Attenuated total reflectance with Fourier transform infrared spectroscopy (ATR/FTIR)

At Fig.3, it is possible to observe some characteristic bands of plant materials: two bands related to the stretching vibrations of the -OH group in cellulose molecules that have intra and intermolecular hydrogen bonds in 3323 cm^{-1} (Wu et al., 2021); stretching vibrations of groups O – C and C – H in 2895 cm^{-1} In seeds, the 1603 cm^{-1} band referring to the asymmetric deformation of the CH_3 groups is attributed to lignin and xylem (Kassab, Abdellaoui, Salim, & El Achaby, 2020). The bands at 1636 cm^{-1} and 1318 cm^{-1} for the cellulose sample are characteristic of vibrations of elongation of the glucose ring referring to cellulose (Cheng, Huang, Wang, & Zhang, 2016). The 1603 cm^{-1} band for the fresh sample refers to lignin that is very evident in the material before treatment, a band that is not seen in the cellulose sample (Cheng et al., 2016). When observing the same band on the cellulose spectrum, a decrease was observed, which is related to the removal of the lignin from the sample, evidencing success in the method used for the extraction, such results can be confirmed through the XRD analysis. due to the increase in the crystallinity of the sample for cellulose. Another characteristic band of lignin is observed at 1268 cm^{-1}, which refers to the elongation of the C-O-C ether bond evident in the fresh sample and is not remarkable in cellulose after hydrolysis treatment (Abu-Thabit et al., 2020). At 1110 cm-1, it corresponds to the C – O elongation and C – H vibrations of the cellulose indicate cellulosic structure after chemical treatments for the extraction of cellulose. The band around 890 cm-1 indicates the cellulose structure due to the presence of β-glycosidic bonds of the cellulose glucose ring (Naduparambath et al., 2018).

Figure 3: Normalized FTIR spectra of samples of fresh seeds and extracted cellulose.

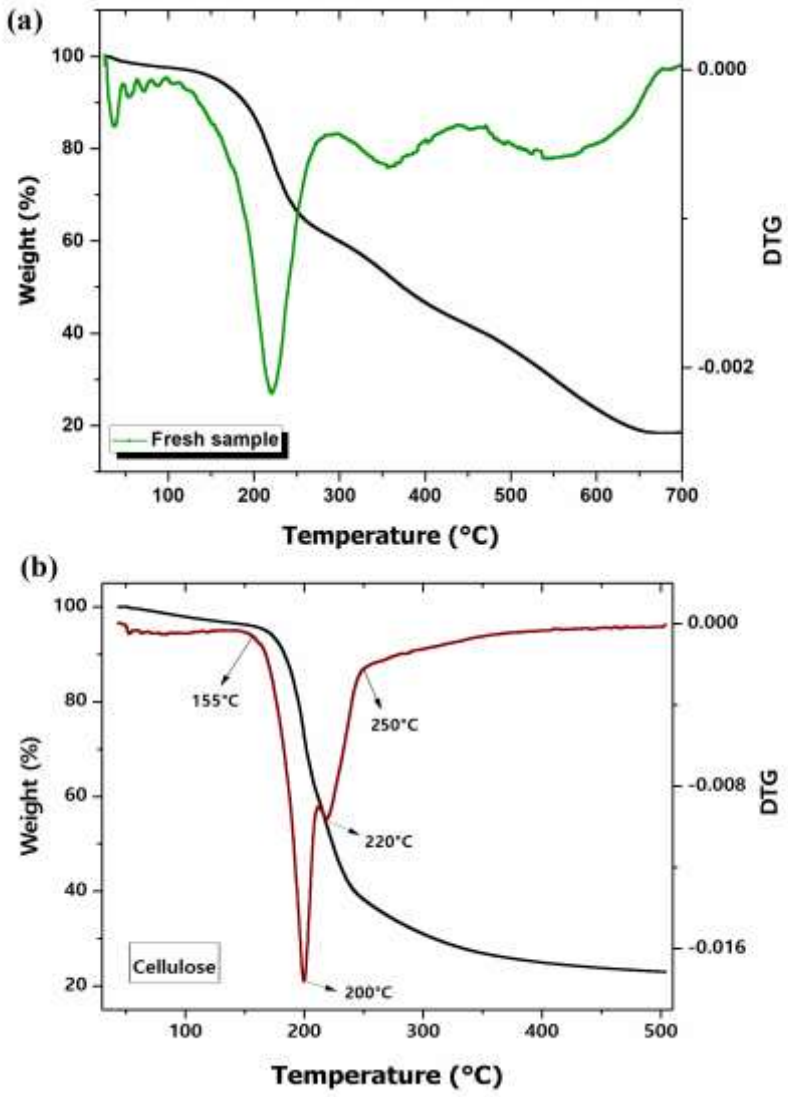


Source: Authors.

Thermogravimetric analysis (TGA)

When observing the mass loss curves shown in Fig. 4a, we see four stages of thermal degradation in the seeds and three for cellulose: 1st stage occurs at 45 °C, attributed to the loss of moisture and water in the two samples. This behavior is common in several materials and can be observed in cellulose extracted from different agro-industrial waste (Table 4); 2nd stage was also observed in both samples – around 180 °C with the highest rate of degradation at 200 °C, related to hemicellulose and cellulose decompositions (Galiwango, Abdel Rahman, Al-Marzouqi, Abu-Omar, & Khaleel, 2019). This result is similar to that found for the degradation of the date palm fiber (180 °C), rose stem (188 °C), and coffee hull with sugarcane bagasse (192 °C) (Gabriel, Belete, Syrowatka, Neubert, & Gebre-Mariam, 2020b; Galiwango et al., 2019; Ventura-Cruz, Flores-Alamo, & Tecante, 2020). It is worth mentioning that below these temperatures, cellulose is thermally stable, but its decomposition can occur between 150 to 455 °C, depending on the source of the biomass (Galiwango et al., 2019); 3rd and 4th stages of the seeds occur at 350 and 540 °C, characteristic of lignin degradation. Due to its rigid and stable structure, this polymer needs higher temperatures to degrade than cellulose (V. O. Santos et al., 2020); 3th stage for extracted cellulose occurs at 220 °C, related to the degradation of long-chain cellulose. Cellulose extracted from different biomass (brown algae, sunflower oil cake, and cotton) also showed a similar peak of degradation (Bessa et al., 2021; Kassab et al., 2019; Maache, Bezazi, Amroune, Scarpa, & Dufresne, 2017; Tarchoun, Trache, & Klapötke, 2019) (see Table 4). It is important to note that in the cellulose TG/DTG there is no degradation at temperatures above 250 °C, confirming the absence of lignin in the sample, and that the DTG curves showed some oscillations, caused by the purging of the gas during the analysis, moving low-density material during analysis (Da Cruz Demosthenes et al., 2020). Therefore, these results are consistent with those presented in the previous characterizations, proving the efficiency of the extraction method, as well as the purity of the extracted cellulose, which presents good thermal stability when compared to Table 4.

Figure 4: Thermogravimetric curves (TG and DTG) of a) fresh seeds and b) extracted cellulose.



Source: Authors.

Table 4: Thermal stability of cellulose obtained from different agro-industrial wastes residues and their stages of degradation.

	Cellulose degradation stage			
Wastes	**Initial (°C)**	**Intermediate (°C)**	**Final (°C)**	**Reference**
Açaí seeds	180	200/220	400	This work
Cotton	100	160	310	(Bessa et al., 2021)
Coffee hull/sugarcane bagasse	192	-	320	(Gabriel et al., 2020a)
Brown algae	80	200	390	(Tarchoun et al., 2019)
Sunflower oil cake	100	185	400	(Kassab et al., 2019)
Aerial roots of banyan tree	150	300	370	(Ganapathy, Sathiskumar, Senthamaraikannan, Saravanakumar, & Khan, 2019)
Tree *(Ficus religiosa)*	234	325	400	(Moshi et al., 2020)
Juncus effusus L.	220	360	455	(Maache et al., 2017)
Rose stem (*Rosa* spp.)	188	-	402	(Ventura-Cruz et al., 2020)
Pistachio shells	250	370	450	(Kasiri & Fathi, 2018)
Date palm biomass	180	-	350	(Galiwango et al., 2019)

Source: Authors.

In Table 4 it is also possible to observe the intermediate degradation and final degradation, primordial parameters to know up to what temperature the cellulose can withstand and, thus, study the possible applications. The cellulose extracted in this work presented an intermediate temperature higher than that extracted from cotton and sunflower oil cake. The final degradation temperature found for cellulose extracted from açai seeds was also superior to cellulose extracted from other residues (see Table 4), which allows several applications even at high temperatures.

4. Conclusions

The present research showed the high potential of açaí seeds for cellulose extraction through the Kumode method, in view of the good cellulose content obtained (45.5% of cellulose). Analyzing the high consumption of the product and the high availability of this residue in the state of Amazonas, we can consider that the reuse of açaí residues is highly promising in the production of cellulose. In addition to the extraction, this research carried out the analysis and characterization of cellulose by

means of various physical-chemical techniques to know its purity and characteristics. Through this, it was clear that the extracted cellulose is similar to cellulose extracted from different agro-residues and, in some cases, showing superior properties. Thus, this research enables the use of açaí seed as a possible source of cellulose. As future work, it is worth mentioning studies for nanocellulose obtention and its application, development of catalysts from the seeds for biodiesel synthesis, and the preparation of different composites.

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