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PHYSICO-CHEMICAL CHARACTERIZATION OF AÇAÍ SEED AND FIBER (EUTERPE OLERACEA MART.) THROUGH CLASSICAL AND INSTRUMENTAL METHODS

CARACTERIZAÇÃO FÍSICO-QUÍMICA DO CAROÇO E DA FIBRA DO AÇAÍ (EUTERPE OLERACEA MART.) VIA MÉTODOS CLÁSSICOS E INSTRUMENTAIS

CARACTERIZACIÓN FISICOQUÍMICA DEL NÚCLEO Y LA FIBRA DE AÇAÍ (EUTERPE OLERACEA MART.) MEDIANTE MÉTODOS CLÁSICOS E INSTRUMENTALES

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ABSTRACT

The açaí tree fruit (Euterpe oleracea Mart.) has great importance in the Brazilian agroindustrial sector, being widely consumed in the country. The cultivation of açaí generates residues such as the seed and fiber, which are usually discarded without previous treatments. There are few data regarding the physical-chemical characterization of these residues, and the possibilities of its reuse have not been defined yet. In this context, this work aimed to study the physical -chemical and thermal characterization of the seed, the fiber and its extractives, through the analysis such as absolutely dried percentage, extractives in water, sodium hydroxide and organic solvent, ash content, lignocellulosic percentage, density and thermogravimetric analysis. The absolutely dried percentage was 90% for fiber and 85.1% for seed, that showed a higher amount of extractives compared to the fiber, and both showed low levels of soluble extracts in organic solvent. The ash content was 1.40% for fiber and 3.41% for seed. The lignin contents obtained for seed and fiber were 25.1% and 24.8%, respectively. The cellulose content was 46.2% for fiber and 18.6% for seed. The average seed density was 1.27 g/cm^3. All residues showed four stages of degradation in the TGA.

RESUMO

O açaí, fruto do açaizeiro (Euterpe oleracea Mart.), tem grande importância no setor agroindustrial brasileiro, sendo largamente consumido no país. A cultura do açaí gera como resíduos o caroço e a fibra, que geralmente são descartados sem tratamentos prévios. Existem poucos dados relativos à caracterização físico-química desses resíduos e possibilidades de reutilização ainda não foram definidas. Nesse contexto, este trabalho objetivou estudar a caracterização físico-química e térmica do caroço, da fibra e dos seus extrativos, por meio de análises como percentual absolutamente seco, extrativos em água, hidróxido de sódio e solvente orgânico, teor de cinzas, percentuais lignocelulósicos, densidade e análise termogravimétrica. O percentual absolutamente seco foi de 90% para a fibra e 85,1% para o caroço. O caroço apresentou maior quantidade de extrativos comparado à fibra, e ambos apresentaram baixos teores de extrativos solúveis em solvente orgânico. O teor de cinzas foi de 1,4% para a fibra e 3,41% para o caroço. Os teores de lignina obtidos para caroço e para a fibra foram 25,1% e 24,8%, respectivamente. A fibra apresentou teor de celulose de 46,2% e o caroço 18,6%. A densidade média do caroço foi de 1,27 g/cm^3. Todos os resíduos apresentaram quatro etapas de degradação na TGA.

RESUMEN

Asaí, el fruto del árbol de açaí (Euterpe oleracea Mart.), es de gran importancia en el sector agroindustrial brasileño, siendo ampliamente consumido en el país. El cultivo genera como residuos la semilla y la fibra, que generalmente se desechan sin tratamiento previo. Hay pocos datos sobre la caracterización físico-química de estos residuos y aún no se han definido las posibilidades de reutilización. En ese contexto, este trabajo tuvo como objetivo estudiar la caracterización físico-química y térmica del núcleo, fibra y sus extractivos, a través de análisis como porcentaje absolutamente seco, extractivos en agua, hidróxido de sodio y solvente orgánico, contenido de cenizas, porcentajes lignocelulósicos, Densidad y Análisis Termogravimétrico. El porcentaje absolutamente seco fue de 90,0% para la fibra y 85,1% para la semilla. La semilla presentó mayor cantidad de extractivos en comparación con la fibra, y ambas presentaron bajos niveles de extractivos solubles en solvente orgánico. El contenido de cenizas fue del 1,40% para la fibra y del 3,41% para el núcleo. Los contenidos de lignina obtenidos para el núcleo y para la fibra fueron 25,1% y 24,8%, respectivamente. La fibra tenía un contenido de celulosa de 46,2% y el núcleo de 18,6%. La densidad de semilla promedio fue de 1,27 g/cm3. Todos los residuos mostraron cuatro etapas de degradación en el TGA.

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1. INTRODUCTION

The açaí tree (Euterpe oleracea Mart.) is a palm tree native to the Amazon that is widely exploited for the extraction of palm heart and fruits. According to the Brazilian Institute of Geography and Statistics (IBGE, 2020), the North region produced 1,395,141 tons of the fruit in 2019, corresponding to 99.8% of national production. Pará is the Brazilian state that most produces and consumes açaí, being responsible for the production of 1,320,150 tons in 2019. The states of Amazonas, Roraima and Rondônia also stand out for their high productivity, and together they produced 74,152 tons in 2019, which generated a 9.6% increase in production compared to the previous year (IBGE, 2020).

The production of açaí has spread to other regions of Brazil, such as the states of Espírito Santo and Bahia, due to the fact that it is an easily cultivated plant and due to the similarities in terms of climate. According to IBGE (2020), the two states together produced 1,295 tons of the fruit in 2019. The most favorable areas for açaí planting are those with medium temperature and precipitation. In Espírito Santo, the municipalities that stand out for the highest productivity are Linhares, São Mateus and Jaguaré, in the north of the state, (Gasparini, Fonseca, Pastro, Lacerda & Santos, 2015) and in the state of Bahia, the municipalities of Ilhéus and Itabuna stand out.

The açaí crop stands out among the plant species produced in Brazil due to its abundance in planting and its ability to produce a food with a high energy source (Cordeiro, Paula, Sousa & Amorim, 2017). The fruit has the potential to be used by pharmaceutical, cellulosic and animal feed production industries (Goulart et al., 2016).

The extraction of açaí generates the pit and fiber as solid waste. According to federal law No. 12.305/10, which instituted the National Solid Waste Policy (PNRS, intials in Portuguese), açaí seeds are commercial activity residues and their collection and disposal are the responsibility of their generator — in this case, the beaters, not being able to be collected by the companies bidding for the collection of urban solid waste from the cities. However, the pits are usually discarded in the open, without prior treatment. The lack of treatment of this waste can lead to environmental problems, as improper disposal generates debris in the streets and on vacant lots, which can lead to the formation of clandestine deposits; and when discarded in cannals it can cause silting and flooding (Oliveira, Passos & Conceição, 2020). This can generate problems related to climate change by increasing the emission of greenhouse gases, mainly methane (CH_4) and carbon dioxide (CO_2) (Sadh, Duhan & Duhan, 2018).

The use of natural fibers as a raw material in several sectors has been the object of study because they are a material obtained from renewable sources. When compared with synthetic fibers, the natural ones stand out for their reuse and reduced waste generation, besiedes presenting excellent properties such as: being biodegradable, having good tenacity, high rigidity, lower processing cost and ease of processing because they cause less wear on machinery (Junior, Novack, Botaro, Protásio & Couto, 2013).

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Sato et al. (2019) studied the use of biochar produced from açaí pit as a solid's conditioner. For the characterization of the residue, the contents of soluble extractives in ethanol-toluene organic solvent, total lignin and ash were quantified. There was an achievement of 2.3% of extractive content in organic solvent, 37.2% of total lignin content were obtained, which was a value considered satisfactory since the complex structure of lignin gives rigidity to the material, affecting the stability of biochar. The ash content of 2.51% was considered low, suggesting high energy efficiency because it has a large amount of organic matter. The pyrolysis of this residue resulted in biochar of an extremely hydrophobic character, which indicated the presence of non-polar compounds in the composition of the açaí seed (Sato et al*.,* 2019).

Oliveira et al. (2020) studied the pretreatment of açaí pits with a 4% hydrogen peroxide solution alkalized to pH 11.5 with sodium hydroxide for the production of fermented sugars and ethanol. Analyzes of the açaí seed were carried out to determine the cellulose, hemicellulose, and lignin contents, obtaining values of 40.3%, 18.3% and 16.2%, respectively. After pretreatment of the açaí seed with a 4% hydrogen peroxide solution at 60°C, 5.95 g of glucose was obtained from 100 g of the seed, which was considered a high conversion rate. When submitted to enzymatic hydrolysis and fermentation, this residue resulted in an 80.1% conversion rate. This is because high values of lignin, cellulose and hemicellulose indicate a high amount of carbon in the cell structure, which is a source of fermentation and ethanol production (Oliveira et al., 2020).

In this context, the need to develop research aimed at reusing agro-industrial residues from the açaí crop was observed. A broad characterization of the açaí seed and fiber is important from a technical-scientific point of view, since when all the properties related to these materials are known, new forms of reuse can be proposed in order to generate greater added value to the product residues, and thus transform it into raw material for new industrial segments. Therefore, the objective of this work was the characterization of the açaí peel and fiber using classical and instrumental methods of chemical analysis to determine the physical-chemical and thermal profile.

2. MATERIALS AND METHODS

Five kilos of pulped açaí were obtained from the local market. The sample was washed in running water to eliminate solid residues, such as sand and insects. Then, sanitization was performed by immersing the sample in a 30ppm sodium hypochlorite solution for 15 minutes, with subsequent rinsing in running water. After rinsing, the seeds were dried in an oven at 105°C for 10 hours, and then the seed and fiber were manually separated. Crushed seeds were used in all analyzes. The procedures were performed in triplicate, so that the results presented are the arithmetic average. The analyzes were carried out for the açaí seed and fiber and are described in items 2.1 to 2.12. All analyzes performed are shown in Figure 1.

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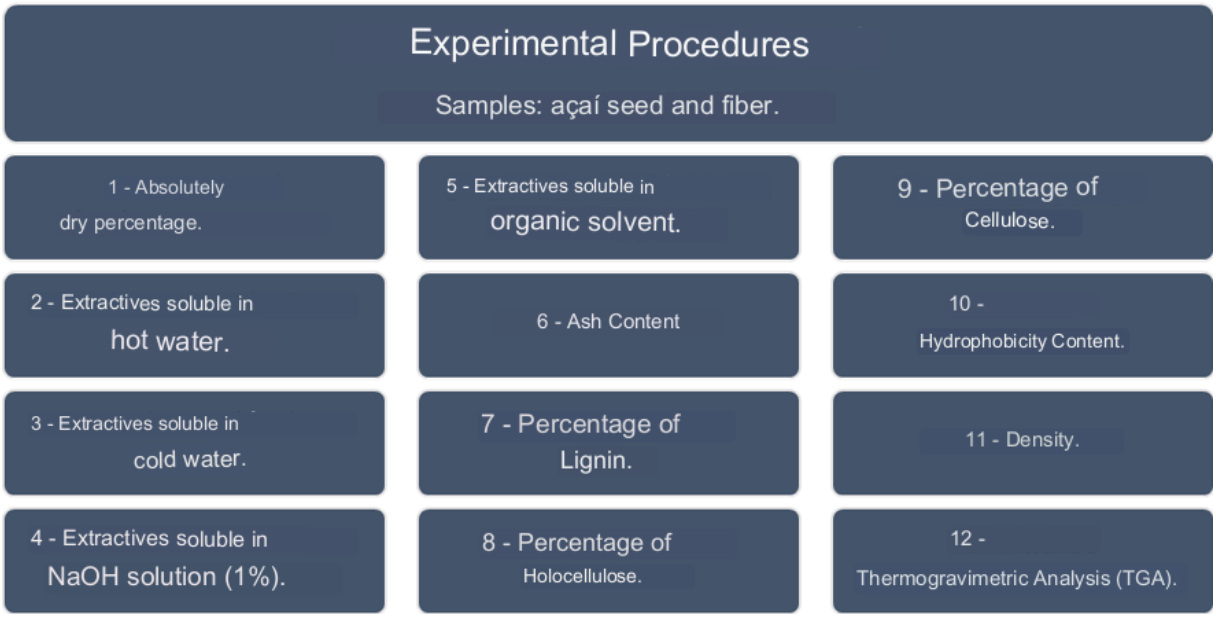
Figure 1. Analyzes carried out for the açaí fiber and seed.



Source: Authors (2023).

2.1. Absolutely Dry Percentage

About 2.0 g of sample (core/fiber) were weighed and transferred to a BEL moisture-determining scale, model i-Thermo 163L, at a temperature of 105°C, until constant weight.

2.2. Hot Water-Soluble Extracts

About 2.0 g of sample (core/fiber) were weighed in an Erlenmeyer flask and then 200 mL of distilled water were added and kept in a thermostatic bath at boiling temperature of 100 ± 5°C. The solution was manually stirred every 15 minutes for 3 hours, then filtered under vacuum under continuous washings with 250 mL of hot distilled water. Finally, the sample was dried in an oven at 103°C for 18 hours. Hot water-soluble extractives were calculated using Equation 1.

Equation 1. Calculation of soluble extractives. Where: Mi is the initial mass of the sample (g) and Mf the final mass of the sample (g).

$$\% \text{ extractives} = [(Mi - Mf) / Mi] * 100$$

2.3. Cold Water-Soluble Extracts

Two grams of sample (seed/fiber) were weighed and then 200 mL of distilled water at room temperature was added. During the first hour of immersion, the solution was manually shaken every 15 minutes. After remaining at rest for 48 hours, it was filtered under vacuum under continuous washings with 250 mL of distilled water and, finally, the sample was dried in an oven at 103°C for 18 hours. Cold water-soluble extractives were calculated according to Equation 1.

2.4. Extractives soluble in 1% NaOH solution (w/v)

Two grams of sample were weighed and then 100 mL of 1% sodium hydroxide (NaOH) solution (w/v) at room temperature was added. The mixture was manually shaken after 10, 15 and 25 minutes from the beginning of the extraction. The total extraction time was one hour, and at the end of the procedure, the mixture was filtered under vacuum and washed with 250 mL of

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hot distilled water, 50 mL of acetic acid (CH 3 COOH) 9% (w/v) and again 250 mL of hot water, and finally dried in an oven at 103°C for 18 hours. The extractives soluble in 1% NaOH solution (w/v) were calculated according to Equation 1.

2.5. Extractives soluble in organic solvent

Weighed separately 1.0 g of dry and ground seed and 2.0 g of dry fiber. These were subjected to extraction using a Soxhlet extractor with 150 mL of ethyl alcohol–toluene solution (1:2) for 6 hours. At the end of the extraction, the solvent was recovered and the flask containing the extract was dried in an oven at 103°C for 24 hours. Extractives soluble in organic solvent were calculated using Equation 2.

Equation 2. Calculation of extractives soluble in organic solvent. Where: Me is the mass of the extract (g) and Mi is the initial mass of the sample (g).

$$\% \text{ extractives} = [(Me / Mi] * 100$$

2.6. Ash Content

The ash content was determined from the moisture-free sample (core and fiber), which was subjected to a calcination process in a muffle furnace preheated at a temperature of 750°C, calcined for 2 hours, cooled, and then conditioned in a desiccator and its final mass determined by weighing in an analytical balance. The results were calculated using Equation 3.

Equation 3. Calculation of ash content. Where: %C is the ash content, Mi is the initial mass of the sample, Mf the final mass of the sample.

$$\%C = (Mf / Mi) \times 100$$

The equipment used in the process was a Quimis digital muffle furnace, model Q318M24, at a working temperature of 300 to 1200°C

2.7. Percentage Of Lignin

To determine the lignin and holocellulose content, the core and fiber samples were prepared as follows. First, the extraction was performed (from the core sample and then from the fiber) with a Soxhlet extractor using 150 mL of 96° alcohol for 6 hours at a temperature of 80 ± 5°C. Then, the filter paper containing each sample was immersed in hot water for 3 hours and then dried at room temperature for three days.

To determine the percentage of lignin, approximately 1.0 g of sample (core/fiber) was weighed in an Erlenmeyer flask and cooled to a temperature of 18 ± 2°C with 15 mL of sulfuric acid solution (H_2SO_4) 72% (v/v) and kept in an ice bath for 2 hours. 500 mL of distilled water were added, keeping the solution boiling for 4 hours, and then left to rest for 18 hours. The solution was vacuum filtered and dried at room temperature for 48 hours. The percentage of lignin was calculated using Equation 4.

Equation 4. Calculation of lignin percentage. Where: Mi is the initial mass of the sample (g) and Mf the final mass of the sample (g).

$$\% \text{ lignin} = [(Mi – Mf) / Mi] * 100$$

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2.8. PERCENTAGE OF HOLOCELLULOSE

Holocellulose is an empirical term used to refer to the mixture of hemicellulose and cellulose. Two grams of sample (core/fiber) were weighed in an Erlenmeyer flask, then 100 mL of 6% (v/v) sodium hypochlorite solution (NaClO) and 50% (v/v) hydrogen peroxide (H_2O_2) were added for 6 hours at room temperature. Soon after, the sample was caustically washed with 50 mL of 0.6% (w/v) sodium hydroxide (NaOH) solution, filtered and dried at room temperature for 48 hours. The percentage of holocellulose was calculated using Equation 5.

Equation 5. Calculation of the percentage of holocellulose. Where: Mi is the initial mass of the sample (g) and Mf the final mass of the sample (g).

$$\% \text{ holocellulose} = (Mf / Mi) \times 100$$

2.9. CELLULOSE PERCENTAGE

After weighing 1.0 g of holocellulose, resulting from the holocellulose determination procedure, 15 mL of potassium hydroxide solution (KOH) 24% (w/v) was added and kept under mechanical agitation for 15 hours in an Erlenmeyer flask. Then, the sample was washed with a 1% (v/v) acetic acid (CH_3COOH) solution and ethanol (CH_2CH_3OH), filtered, and dried at room temperature for 48 hours. The percentage of cellulose was calculated using Equation 6.

Equation 6. Calculation of cellulose percentage. Where: Mi is the initial mass of the sample (g) and Mf the final mass of the sample (g).

$$\% \text{ cellulose} = (Mf / Mi) \times 100$$

Knowing that holocellulose is the mixture of hemicellulose and cellulose, the hemicellulose content was calculated using Equation 7, by subtracting the holocellulose content from the cellulose content.

Equation 7. Calculation of hemicellulose content.

$$\text{hemicellulose content} = \text{holocellulose content} - \text{cellulose content}$$

2.10. HYDROPHOBICITY CONTENT

At this time, 1.0 g of sample (seed/fiber) was weighed, and then 40 mL of a solution of distilled water and hexane with a ratio of (1:1) (v:v) was added. The mixture was subjected to magnetic stirring for 3 minutes. Then the sample immersed in the solution was left to rest for 5 minutes to separate the hydrophilic and hydrophobic phases, and then filtered with filter paper. The material transferred to the organic phase was dried in an oven at 103°C for 18 hours. The hydrophobicity content was calculated using Equation 7.

Equation 7. Calculation of the hydrophobicity content. Where: Mh is the mass of the material transferred to the organic phase (g) and Mi is the initial mass of the sample (g).

$$\% \text{ hydrophobicity} = (Mh / Mi) * 100$$

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2.11. DENSITY DETERMINATION

The apparent density of the seed was determined through the ratio between mass and volume. The samples were previously dried in an oven at 105°C for 2 hours. The average diameter of the pit was determined with the aid of a caliper, by the arithmetic average of its three-dimensional measurements, and the volume was obtained by approximating the pit to a spherical geometry. Thus, the density was calculated using Equation 8.

Equation 8. Density calculation. Where: ρ is the core density (g/cm3), M is the mass (g), and $D$the average diameter (cm).

$$\rho = 6 \times M / (\pi \times D^{3})$$

2.12. THERMOGRAVIMETRIC ANALYSIS (TGA)

Approximately 10mg of fiber sample, crushed core and extractives soluble in organic solvent were weighed and submitted to a heating program of 20ºC/min under nitrogen atmosphere (N 2), in the temperature range between 20 and 600° C, using the Shimadzu DTG-60 equipment.

3. RESULTS AND DISCUSSION

3.1. ABSOLUTELY DRY PERCENTAGE

The absolutely dry percentage obtained for the seed was 85.1%. This value is close to the result obtained by Martins, Konagano, Souza and Lopes (2020) who determined the absolutely dry percentage for the açaí seed (88.3%). For the fiber, the value determined for the percentage absolutely dry was 90.0%, the same result obtained by Quirino (2010), who analyzed the açaí fiber under the same conditions. The values obtained show that the fiber had a higher absolutely dry percentage than the seed. Such difference may be related to the shape and volume of the analyzed sample, and also to the washing and drying processes to which the sample was submitted before the experiments. The core has a spherical body and the fiber has an elongated and narrow shape, so that the drying process used in the initial treatment given to the samples may have been more efficient for the fiber than for the core.

However, analyzing the data obtained and the literature references, it can be stated that the core and fiber analyzed in this experiment were dried efficiently and that, in this condition, they can be ground in a way that does not compromise the physical-chemical characterization tests (Pereira, Anjos & Magnago, 2019). Another important fact is that the moisture content is related to the biodegradation property that the material presents, so when this type of material is properly dried, there will be an increase in durability and consequently its state of preservation will be increased.

3.2. SOLUBLE EXTRACTIVES

The value obtained for extractives soluble in cold water for the seed was 17.4% and for the fiber was 3.71%. The comparison of these results was performed using a hypothesis test, considering the scenario in which the population standard deviations are unknown. The critical region was defined with degrees of freedom GL = 2, and with a significance level of α = 5%. Thus, the values for rejecting the hypothesis of equality of means (H_0) are $t < -4.3127$ or $t > 4.3127$. The t value calculated in the test was 29.23, being in the rejection region of H_0.

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Therefore, we considered, at a significance level of 5%, that the values of extractives soluble in cold water for the core and for the fiber showed significant differences.

The data related to the content of extractives soluble in hot water reproduced the same differences in relation to the behavior of the core and the fiber, these values being 11.6% and 3.22%, respectively. Table 1 presents the results of the analyzes of extractive contents for the açaí seed and fiber, with the respective means and standard deviation (in parentheses).

Table 1. Results of extractive contents for the core and fiber.

Sample	Cold water soluble extractives (%)	Hot water soluble extractives (%)	Extractives soluble in sodium hydroxide 1% (%)	Extractives soluble in organic solvent (%)
Lump	17,4 (0,02)	11,6 (2,89)	18,5 (0,35)	10,7 (0,75)
Fiber	3,71 (0,811)	3,22 (0,477)	9,45 (2,459)	1,67 (0,139)

Source: Authors (2023).

Extraction with cold water is used to remove compounds such as inorganic salts, resins, sugars and dyes, which influence the morphological characterization and microbiological protection of the sample (Moreira, Fazion & Ribeiro, 2016), among other factors. Hot water extraction, in addition to removing inorganic compounds, also removes starch. Therefore, the results of these two analyzes suggest a greater presence of these components in the core when compared to the fiber, which is an indicator that the core may have greater resistance against natural biodegradation.

Santos et al. (2020) determined the extractive content in African mahogany wood using cold water (3.91%) and hot water (4.97%). The açaí seed had higher levels of extractives in both conditions: hot (11.6%) and cold (17.4%) water. This may be due to its morphological structure, because unlike African mahogany wood, the core is coated with açaí pulp. The pulp, the edible part of the fruit, is composed of sugars, starches and dyes, substances that can be absorbed by the stone and increase the percentage of extractives.

Junior et al. (2013) determined the content of extractives soluble in hot water in bamboo fibers (7.40%). The açaí fiber (3.22%) had less sugar and starch in its structure. These data suggest that açaí fiber is a more efficient alternative for application in the production of polymeric reinforcements when compared with bamboo fiber, since the high content of extractives hinders the adherence between composite and matrix.

The data relating to the analysis of the soluble extractive's contents in 1% (w/v) sodium hydroxide solution is presented in Table 1, which for the core was 18.5% and for the fiber was 9.45%. The extraction process using 1% sodium hydroxide solution removes substances of acidic character, such as greases and oils, also providing the fragmentation of lignin and hemicellulose, along with terpenes, phenols and cresols (Moreira, 2016; Pereira, 2019). During the analysis, the darkening of the solutions containing the samples, which were initially without color, was observed after 15 minutes. This behavior may occur because when alkaline treatments are applied to lignocellulosic materials, fragmentation of lignin and hemicellulose structures occurs. Residual lignin has hydroxyphenylpropane units, a component with a phenolic character that is responsible for the darkening of the reaction medium. Thus, the

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data obtained for the contents of extractives in 1% sodium hydroxide solution showed that there was fragmentation of lignocellulosic materials since there was mass variation and also due to the darkening observed in the solutions. The açaí stone (18.5%), although similar, contains lower amounts of grease and oil when compared to mahogany wood.

The analyzes concerning the determination of the extractive's contents in organic solvent (Table 1) showed that the core presented a value of 10.7% and the fiber 1.67%. The compounds that are extracted in this analysis are acids, fatty esters, phenolic compounds, waxes, fats and ethers insoluble compounds which can influence the permeability and hardness properties of the sample (Moreira et al., 2016). Thus, the difference between the levels of extractives in organic solvent for the core and the fiber suggests that the samples have different behaviors in terms of water absorption and durability. Since the seed has a higher content of these extractives — which have, in their majority, non-polar character — it has a lower capacity for water absorption when compared to the fiber. On the other hand, as the extractives are long-chain components, they are more resistant to breakage, increasing the durability of the seed.

Santos et al. (2020) analyzed the content of extractives in organic solvent using a method similar to that used in this work for African mahogany wood of varying heights, obtaining an average value of 6.35%. When comparing this data with that obtained for the açaí seed, whose value was 10.7%, it was observed that the amount of this extractive was lower in African mahogany. This result may be related to the substances extracted in this analysis. The organic solvent extracts long-chain and non-polar organic substances, which increases the impermeability of the material since the water molecule is polar. Thus, the açaí stone has a lower water absorption capacity when compared to African mahogany wood.

When making a comparison with the value of soluble extractives in organic solvent obtained by Junior et al. (2013) analyzing bamboo fibers whose value was 5.69%, it was observed that the açaí fiber had a lower content of this component, with the obtained content of 1.67%. This difference may be related to the different needs of the samples — while the açaí fiber is responsible for enveloping the seed, the bamboo fiber provides support to the plant, indicating that the bamboo fiber has greater rigidity when compared to the açaí fiber.

3.3. Ash Content

In general, the ashes are composed of inorganic residues that are left over from the burning of the organic matter of a given sample, and are mainly composed of elements such as potassium, sodium, calcium, silicon and magnesium, and may contain in smaller amounts elements such as aluminum, copper, iron, manganese and zinc. With the knowledge of the ash content of a material, its energy potential can be evaluated, in which the higher the ash content, the lower the energy potential, due to the smaller amount of organic matter.

To determine the ash content, experiments were carried out in triplicate. The result obtained for the fiber was 1.40%, with a standard deviation of 0.043, and for the core it was 3.41% with a standard deviation of 0.004.

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Comparing the values of the ash content of the fiber and the core, it is observed that the fiber had a lower percentage, as expected, since it is mostly made up of cellulose, hemicellulose and lignin, thus characterizing a higher energy potential compared to the seed.

3.4. Lignocellulosic Contents

3.4.1 Lignin Content

The value of lignin content determined for the core was 25.1% and for the fiber was 24.8%. The comparison of these results was also performed using a hypothesis test, considering the scenario in which the population standard deviations are unknown. The critical region was defined with degrees of freedom GL = 3, and with a significance level of α = 5%. Thus, the values for rejecting the hypothesis of equality of means (H_0) are t < -3.1825 or t > 3.1825. The t value calculated in the test was 0.22, being within the H_0 acceptance region. Therefore, we considered, at a significance level of 5%, that the values of lignin contents for the core and for the fiber did not show significant differences.

Pereira, et al. (2019) studied the chemical processes of cellulose extraction in lignocellulosic residues from banana cultivation. Through this bibliographic review, it was observed that, in general, the concentrations of lignocellulosic constituents in biomass vary greatly from one plant species to another, or even within the same species. Table 2 presents the results of the analyzes of the lignocellulosic contents for the açaí seed and fiber, with the respective means and standard deviation (in parentheses).

Table 2. Results of lignocellulosic contents for the core and fiber.

Sample	Lignin Content (%)	Holocellulose Content (%)	Hemicellulose Content (%)	Cellulose Content (%)
lump	25,1 (2,08)	76,7 (2,15)	58,1	18,6 (1,44)
Fiber	24,8 (1,06)	73,0 (1,20)	26,8	46,2 (5,30)

Source: Authors (2023).

When comparing the lignin content in the work by Oliveira et al. (2020) for the açaí seed, whose value was 18.3%, it is possible to observe that the seed analyzed in this study presented a higher amount of this component (25.1%). This difference may be related to the fact that the residue studied in this work comes from a plantation cultivated in the state of Bahia, while Oliveira et al. (2020) used samples from the state of Pará. Differences in terms of geographic conditions, time and temperature in which the samples were cultivated confer different biological needs for the same species, resulting in different amounts of the same component.

When analyzing the lignin content for the fiber, whose value was 24.8%, it was observed that this was higher than that determined by Quirino (2010) when he analyzed açaí fibers of the Euterpe precatoria species, whose value was 10.2%. This result suggests that lignin contents may vary between different species, since the Euterpe oleracea species was used for the present study. This data confirms the hypothesis suggested by Pereira et al. (2019), that lignocellulosic materials may vary for the same material by studying different species.

It is important to analyze lignin, as it is an amorphous, highly complex and branched polymer, which has a phenolic character. The lignin content is directly related to the difficulty of degrading the material (Oliveira et al., 2020), so the data obtained in this analysis indicate that

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the seed and the fiber have the potential to be used in the synthesis of new materials because they have a high content of lignin and, consequently, high level of durability. In addition, the data also suggest that due to the fact that these are very close, the lignin content determined for both samples should not vary the chemical durability due to the presence of this component.

3.4.2 Holocellulose Content

Measures were taken to determine the holocellulose content of the açaí seed and fiber, whose values were 76.7% and 73.0%, respectively (Table 2). The analysis of these results suggests that this difference can be attributed to the different percentages of cellulose and hemicellulose in the samples, since holocellulose is an empirical term used to refer to the mixture of hemicellulose and cellulose (Mesquita, 2013).

When comparing the values determined in relation to the holocellulose content for the seed (76.7%) with the data obtained by Oliveira et al. (2020) which were 56.5% in relation to the açaí seed, it was possible to notice that the values determined in this work were higher. Regarding the holocellulose content obtained for the açaí fiber (73.0%), the same behavior was observed for the seed, which was higher than the value of 67.6% determined by Junior et al. (2013) analyzing natural bamboo fibers using a similar method.

Hemicellulose is formed by a heterogeneous class of low molecular weight polysaccharides, the main one being xylose (Pereira et al., 2019). The deconstruction of hemicellulose for its constituent sugars provides data about the carbon content in the analyzed biomass, which is an indication for the potential use of this biomass in the production of biofuel (Oliveira et al., 2020).

Using equation 7, the hemicellulose content obtained for the core was 58.1% and for the fiber the value was 26.8% (Table 2). These data suggest different amounts of this component in the analyzed samples, indicating a higher carbon content in the seed.

3.4.3 Cellulose content

The values obtained for the cellulose content of the seed and the açaí fiber were different, as shown in Table 2. The determined value of the cellulose content for the seed was 18.6%, and for the fiber a content of 46.2% was determined. The main characteristic of cellulose is to provide rigidity to the cell wall, therefore, the data obtained suggest that the fiber presents greater hardness when used for the production of composites due to the greater amount of this component.

When comparing the cellulose content of 40.3%, obtained for the seed in the work carried out by Oliveira et al. (2020), it was observed that the seed analyzed in this work had a lower amount of cellulose, whose value was 18.6%. This difference can be explained because the chemical composition of lignocellulosic materials varies according to the growing region, soil type and climatic conditions (Mesquita, 2013).

Cellulose, formed by glucose monomers, is widely used as a raw material in papermaking. In addition, it is used as a reinforcing agent for composites, in the synthesis of cellulose acetate

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for the pharmaceutical industry, of semipermeable membranes for the extraction of heavy metals, and in the manufacture of biofuels. Thus, cellulose extraction is an important alternative to add value to lignocellulosic waste (Pereira et al., 2019).

3.5. HYDROPHOBICITY CONTENT

The results referring to the hydrophobicity analyzes for the açaí seed and fiber are shown in Table 3. The hydrophobicity levels obtained were 98.3% for the seed and 92.5% for the fiber. The data suggest that the core had greater hydrophobic properties compared to the fiber, which may be related to the differences between the structures of the samples. The core has a body of greater volume and thickness when compared to the fiber, which has a narrow and thin shape. This morphological structure of the core makes water absorption difficult, thus increasing the hydrophobicity content in relation to the fiber.

Comparing the hydrophobicity value obtained for the açaí fiber, used in this work, with the hydrophobicity content of coconut fibers of 38.6%, obtained by Annunciado, Sydenstricker and Amico (2005), using similar methods of analysis, it was observed that the açaí fiber had a hydrophobicity content 2.4 times higher than that reported in the literature. This is a data that characterizes the açaí fiber with a high hydrophobicity value, which suggests a potential use of this natural fiber in adsorbent materials in oil spills in water bodies (Annunciado et al., 2005).

Table 3 presents the results of the hydrophobicity contents for the açaí seed and fiber, with the respective means and standard deviation (in parentheses).

Table 3. Results of the hydrophobicity content for the core and the fiber.

Sample	Hydrophobicity content (%)
lump	98,3 (2,08)
Fiber	92,5 (0,52)

Source: Authors (2023).

3.6. DENSITY

The average diameter of each pit was obtained by averaging its three-dimensional measurements, obtained using a caliper. After determining the diameter and weighing the seed on an analytical balance, Equation 8 was used to calculate the density. The result obtained was an average density of 1.27 g/cm3 for the açaí seed, with a standard deviation of 0.123.

The analysis of the density of the açaí seed is fundamental for its physical, chemical, thermal and mechanical properties to be understood, as these properties are influenced by the empty spaces between the particles of the material (Barbosa, Rebelo, Martorano & Giacon, 2019). In addition, this characteristic influences the transport and storage of the material, since the denser the material, the smaller the volume occupied by a given mass (Faustino, Santana, Cerqueira, Ataíde & Cardoso, 2019).

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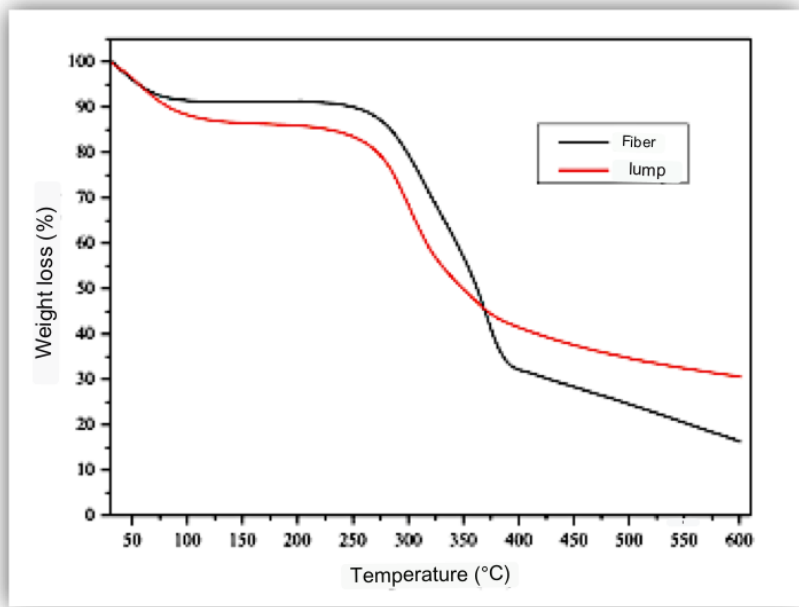
3.7. THERMOGRAVIMETRIC ANALYSIS (TGA)

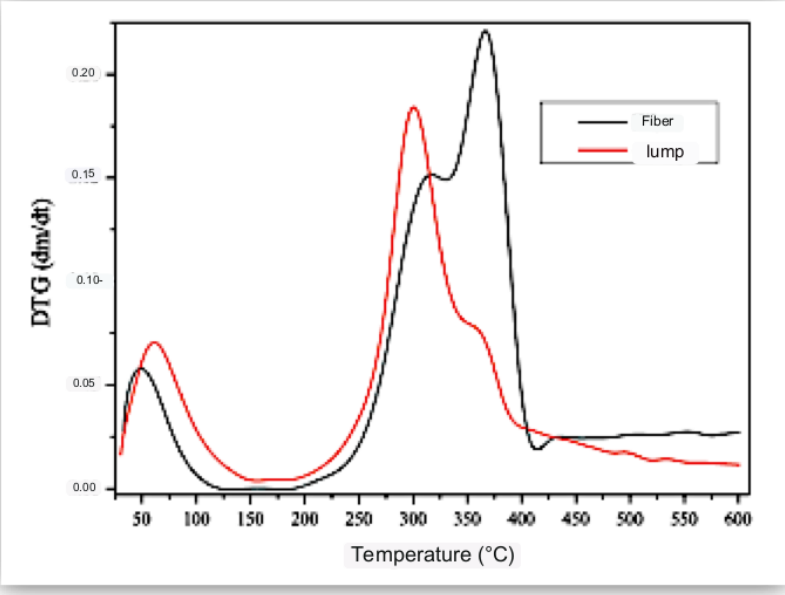
Figure 2 shows the TGA curves in which the percentage of mass loss as a function of temperature during sample heating up to 600°C is described. Figure 3 shows the DTG curves that represent the first derivative of the TGA curves and reflect the variation of mass in relation to time, expressed as a function of temperature. Table 4 shows the data resulting from the interpretation of the TGA and DTG graphs, in which the main stages of degradation with the respective percentages of mass loss for each temperature range analyzed are highlighted.

Figure 2. TGA curves for the fiber and seed of Acai.



Source: Authors (2023).

Figure 3. DTG curves of Açaí fiber and seed.



Source: Authors (2023).

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Table 4. Results of the Thermogravimetric Analysis of the core and fiber.

Samples	Temperature Range (°C)	Maximum Temperature (°C)	Weight loss (%)
Fiber	30 - 120	50	9
	190 - 330	315	25
	330 - 415	365	35
	415 - 600	-	15
lump	30 - 155	60	14
	185 - 350	300	36
	350 - 400	360	9
	400 - 600	-	10

Source: Authors (2023).

In Figures 2 and 3, it is possible to observe that the fiber presented four stages of mass loss, in which the first stage occurred in the range from room temperature to a temperature of 120°C, with a loss of 9% of mass, which corresponds to moisture loss. The second stage occurred in the temperature range from 190°C to 330°C, in which there was a loss of 25% of mass, related to the decomposition of the hemicellulose and the breaking of the cellulose bonds. The third stage occurred in the temperature range of 330°C to 415°C, with a loss of 35% of mass, which is due to the final decomposition of cellulose, and partial decomposition of lignin. The fourth stage occurred in the temperature range of 415°C to 600°C, with a loss of 15% of mass, related to the final decomposition of lignin and residues (Martins, Mattoso & Pessoa, 2009).

Regarding the seed, it is also possible to observe, in Figures 2 and 3, four stages of degradation, the first stage occurring in the range from room temperature to 155°C, with a loss of 14% of mass, corresponding to moisture loss. The second stage occurred in the temperature range of 185°C to 350°C, in which there was a loss of 36% of mass, related to the degradation of the hemicellulose and the breaking of the cellulose bonds. The third stage occurred in the temperature range of 350°C to 400°C, with a loss of 9% of mass, which is due to the final decomposition of cellulose, and partial decomposition of lignin. The fourth stage occurred in the temperature range of 400°C to 600°C, with a loss of 10% of mass, related to the final decomposition of lignin and residues (Martins et al., 2009).

Analyzing the temperature range between 185°C and 350°C attributed to hemicellulose degradation (data shown in Figure 3 and Table 4) it is possible to state that there is a higher percentage of hemicellulose in the seed (36%) than in the fiber (26 %). Regarding the amount of cellulose, which degraded in the temperature range between 330°C and 415°C, it is possible to conclude that the seed has a lower amount of cellulose (9%) than the fiber (35%). These data are in line with the ones presented in Table 2, which show the results of the extraction

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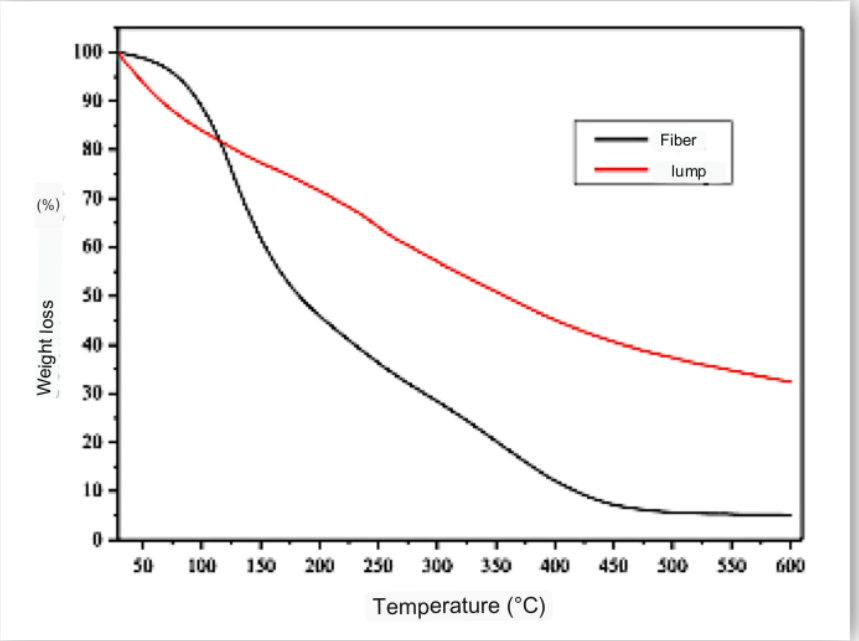
of lignocellulosic contents, in which the core presented 58.1% of hemicellulose, while the fiber presented 26.8%. And the fiber had 46.2% of cellulose while the core had 18.6%.

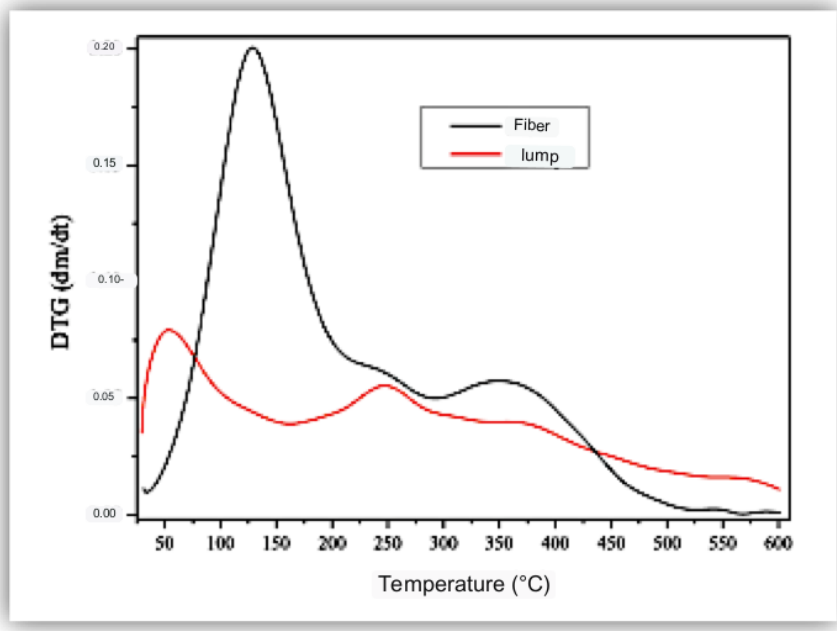
The results obtained referring to the thermogravimetric analysis of the extractives soluble in organic solvent for the fiber and for the core are shown in Figures 4 and 5. The data resulting from their interpretation are shown in Table 5.

Figure 4. TGA curves of the organic extracts of the Açaí fiber and seed.



Source: Authors (2023).

Figure 5. DTG curves of the organic extracts of the Açaí fiber and seed.



Source: Authors (2023).

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Table 5. Results of Thermogravimetric Analysis of the extractives soluble in organic solvent for the fiber and the core.

Samples	Temperature Range (°C)	Maximum Temperature (°C)	Weight loss (%)
Fiber Extractives	30 - 220	130	58
	220 - 290	245	12
	290 - 520	350	24
	520 - 600	-	1
Core Extractives	30 - 160	55	24
	160 - 345	245	25
	345 - 550	380	17
	550 - 600	-	2

Source: Authors (2023).

By analyzing the TGA (Figure 4) and DTG (Figure 5) graphs and the data in Table 5, four degradation stages can be observed for the fiber extractives. The first step occurred between room temperature and 220°C, with a loss of 58% of mass. The second stage occurred between 220°C and 290°C, with a loss of 12% of mass. The third step occurred between 290°C and 520°C, with a loss of 24% of mass. The final step occurred between 520°C and 600°C, with a loss of 1% of mass.

Regarding the seed extracts, it can be observed that there were also four stages of mass loss, in which the first one occurred between room temperature and 160°C with a loss of 24% of mass. The second stage occurred between 160°C and 345°C with a 25% loss of mass. The third step occurred between 345°C and 550°C with a loss of 17% of mass. The last step occurred between 550°C and 600°C with a 2% mass loss.

In general, it can be stated that the first stage of mass loss, both for the extractives from the fiber and the core, observed in Figure 5, suggests that a large part of the mass of extractives initially lost can be related to the presence of solvents used in the extraction and also by the presence of moisture. The solvents used were Toluene and Ethanol, with boiling points of 110.6°C and 78.3°C, respectively. Analyzing Figure 4, it can be observed that up to a temperature of 110°C, there was a loss of 16% of mass of the organic fiber extractives, and 17% of the extractives of the core.

4. CONCLUSION

The absolutely dry percentage obtained was 85.1% for the core and 90.0% for the fiber. It is desirable that lignocellulosic materials have a moisture content of less than 15% for further analysis, therefore the drying process used in the samples was adequate, eliminating possible interference in subsequent analysis.

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The analysis of the extractives content showed a higher amount of extractives in the core when compared to the açaí fiber, which suggests that the fiber has greater potential use for the production of materials such as composites or polymeric reinforcements, since the high content of extractives makes it difficult for the adhesion of the particles between matrix and reinforcement. The result of the analysis of the ash content obtained was 1.4 0% for the fiber and 3.41% for the core, demonstrating the potential of these residues for energy generation, as they had a low ash content.

The lignin contents obtained for core and fiber, 25.1% and 24.8%, respectively, did not show significant differences, which suggests that both samples have the same potential for use in the synthesis of new materials. The high content of hemicellulose for the core (58.1%) when compared to the content obtained for the fiber (26.8%) indicates that the core has a higher carbon content in its composition. This characteristic suggests that the seed could be used for the synthesis of biochar, since a high amount of carbon in raw materials used for this purpose is desirable. The fiber had a higher cellulose content (46.2%) compared to the core (18.6%). This indicates that the fiber can be exploited for cellulose extraction and subsequent application in the most diverse branches of industry, such as paper manufacturing, adhesives and bioplastics. The hydrophobicity contents between core and fiber were approximate, being 98.3% and 92.5%, respectively. This result indicated that both samples can be used as adsorbent materials in oil spills in water bodies, however further studies would be needed for the application of the seed for this purpose.

The average density of the core was 1.27 g/cm ^{3}, making it possible to understand, with this value, other physical-chemical and mechanical properties, as well as to predict transportation and storage conditions for the material. The açaí fiber showed thermal stability up to 190°C, and the seed showed thermal stability up to 185°C and both degradation processes occurred in four stages. The degradation process of the core and fiber extractives also occurred in four stages. The extractives soluble in organic solvent from the fiber and the core showed thermal behavior similar to the behavior of vegetable oils, as it was possible to observe degradation ranges that correspond to the decomposition of fatty acids.

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