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Physical and Thermal Characterization of Achira (*Canna edulis Ker*) Fiber Obtained from Food Industry Waste in the Department of Cundinamarca, Colombia

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Abstract: In recent years, there has been a growing interest in effectively managing agro-industrial waste. One promising approach that has gained attention is exploring this waste to develop new composite materials, especially polymeric materials, with diverse applications across various industries. This study focuses on comprehending the physical and thermal properties of fibrous residues derived from achira (*Canna edulis Ker*). To achieve this, several analyses, including thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and Fourier transform infrared spectrometry (FTIR), have been conducted. Additionally, parameters such as moisture percentage, moisture absorption, bulk density, and lignin percentage have been calculated. The results indicate similarities between achira fibers and other characterized fibers, such as bamboo and other natural fibers studied in scientific research. Based on these findings, it is evident that integrating achira fibers into polymeric matrices is a feasible option. The results of this research offer an opportunity to utilize these materials and contribute to the advancement and strengthening of the recycled raw materials market, promoting sustainability and the circular economy.

Keywords: achira fiber; physical characterization; thermal characterization; polymeric matrices

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1. Introduction

Using natural fibers to create composite materials is a topic of great interest, as recent studies have shown that natural fibers have properties that facilitate their possible use as reinforcement materials to improve the mechanical properties and strength conditions of the composite materials in which they are integrated [1]. In addition, natural fiber composite materials offer benefits in terms of environmental impacts, such as recyclability, renewability, and biodegradability. They also minimize waste during manufacturing, have lower raw material costs, and are lightweight [2,3]. These reinforced composites possess various other advantages, such as lower weight, reduced cost, and excellent mechanical properties [4–7].

In the field of engineering, the use of natural fiber-reinforced composites has increased as alternatives to conventional materials due to their good specific strength, specific stiffness, and adjustable properties [8]. The substitution of harmful materials, such as asbestos, with natural fibers has yielded positive results [9,10]. Some more specific examples include the use of banana fiber as a reinforcement material in a polyester resin matrix, resulting in improved mechanical properties compared to using only polyester resin [11]; guadua fiber has been considered as a possible reinforcement material due to its physical and mechanical

properties [12]; sugar palm shows potential for advanced industrial applications like automotive, defense, and packaging [13]; and abaca, hemp, sisal, and jute fibers have been used as reinforcement materials in cement-based composites to produce fiber cement boards and fiber-reinforced concrete [14].

In Colombia, studies have been conducted to characterize native natural fibers, such as guadua (*Guadua Angustifolia Kunth*) [15], esparto (*Juncus ramboi* subsp. *colombianus*) [16], damagua (*Poulsenia armata*), quérregue (*Astrocaryum standleyanum*), palm mat (*Astrocaryum malybo*), wild cane (*Gynerium sagitatum*), and iraca (*Carludovica palmata*) [17]. These studies have demonstrated that these fibers can be used as reinforcement materials in polymeric matrices. This is due to their favorable morphological properties, as well as their strength and thermal stability, as observed in physical and thermal characterization tests and trials.

However, in addition to the aforementioned fibers, there are other natural fibers derived from the residual biomass generated in the country through industrial processes that are currently not being properly managed or utilized. One such example is the fiber derived from edible canna (*Canna edulis Ker*), which is the focus of this study. There are very few characterization studies available in the scientific literature that provide insights into potential alternative uses of this fiber for reinforcing composite materials and improving their suitability for industrial applications.

Edible canna, one of the Andean root and tuber crops known as achira, is a native crop of South America [18,19]. This crop is distributed in many regions of the world and is known by a variety of local names, such as "Chisgua" in Colombia, "Capacho" in Venezuela, "Imbirg" in Brazil, "Tous les mois" in the West Indies, "Queensland arrowroot" in Australia, "Zembu" in the Philippines, "Lotus tuber" in Taiwan, and "Sagu" in Thailand [20,21].

In Colombia, the cultivation of achira and the extraction of its starch are carried out in areas located at altitudes below 2700 m above mean sea level (MAMSL). This is primarily performed in the department of Cundinamarca and Southern Huila. Additionally, there are municipalities in the departments of Nariño, Cauca, and Tolima where these processes are undertaken, although in a more isolated manner. The estimated annual demand for achira starch in the country is around 2000 tons [22].

In the department of Cundinamarca, the achira fiber is derived as a byproduct from the food industry while sifting rhizomes to extract raw materials for achira starch production. It is estimated that approximately 17.7 g to 36.7 g of bran or achira fiber is generated per kilogram of rhizome [23]. Taking into account the total annual volume of achira fiber produced in the country, it has been calculated that approximately 4284 kg of dry bran is generated as food industry waste annually [24]. According to the results of the animal nutrition laboratory analyses conducted by CORPOICA, achira fiber has high values of crude fiber content (36.5%), in situ digestibility of organic matter (57.01), and crude protein content (3.6%). These characteristics make achira fiber suitable for breeding red Californian earthworms and producing humus [23]. However, no other applications for this waste have been identified.

The rhizomes of achira are a lignocellulosic material whose fibers can be used as a filler to improve the properties of biodegradable films [25], thanks to their high content of amylose and phosphorus [26,27]. Several studies have indicated that the rhizomes of achira have significant technological potential as a source of industrial biopolymers [28,29] and for the generation of biodegradable materials [30,31].

There are studies that show that achira starch can be used as a filtering agent controller in water-based drilling mud, with better performance than other additives commonly used for this purpose [32]. It is capable of removing contaminants, such as perchlorate, from wastewater [33]. This opens up the possibility of using achira fibers as reinforcement in filtering compounds for the treatment of contaminated wastewater as an alternative to other synthetic materials. One possible practical application being studied by the authors of this work is the treatment of leachate from municipal solid waste landfills. Leachate production is the final result of a hydrological process related to the infiltration of precipitation through the MSW mass forming a complex organic liquid. This fluid shows the characteristics of a

wastewater concentrate and may induce extremely negative effects on the surface water and groundwater quality if it is released into the environment. Leachate contains soluble organic, inorganic, and bacteriological constituents, and suspended solids [34].

The advantage of using Sagú bran compared to other fibers depends on the specific application for this material. Sagú bran is generated in various tropical regions, where there can be a significant availability of this fiber. The improper disposal and underutilization of this fiber can lead to environmental impacts. In these regions, valorizing this residue can be a feasible and economically viable option compared to other fibers that may be scarcer or harder to obtain. Additionally, this residue requires analysis of its physical and chemical properties, as it holds potential for various sectors or areas, such as biogas production, paper and cardboard manufacturing, and construction materials. However, it has not been extensively studied, necessitating research to identify the best applications based on its specific characteristics.

This study presents a characterization of the physical and thermal properties of achira fiber to propose it as an alternative to be integrated as a reinforcement material in polymeric matrices. Currently, this information is not available, and several studies have found that natural fibers have stable properties that make them suitable for use in polymeric matrices.

2. Materials and Methods

The achira fiber is obtained during the process of extracting achira starch through a sieving procedure. The achira (*Canna edulis Ker*), also known as Sagú in the east of Cundinamarca, is a plant of Andean origin cultivated in Colombia primarily for the extraction of starch from its rhizomes, which is used as a raw material for making traditional products, such as "achira biscuits", "sagú bread", and "sponge cake". Cundinamarca concentrates around 70% of these producers and 95% of the starch production [35].

The starch extraction process involves a series of operations following the harvest of the achira rhizomes in the following order: collecting the rhizomes to prepare the suspension from which the starch is extracted, washing the rhizomes to achieve the highest degree of cleanliness, and preparing them for shredding. They are washed to remove impurities embedded in the rhizome's interstices. To release the starch present in the cells that form the rhizome, it is necessary to break them, which is mainly achieved through shredding, where the fibers are cut, breaking the cell walls to release the starch. Subsequently, sieving is carried out to separate the bran or fiber from the rhizome and the starch slurry. The result of sieving is separated from the starch through decantation. The obtained starch is washed to remove residues or impurities, and this process is repeated as many times as necessary until it is completely clean [36]. The main byproducts of the process are bran and "mogolla" or stain. The bran is obtained after separating the starch in the sieving operation, and it has a high crude fiber content (36%), a dry matter digestibility of 57%, a crude protein content of 3.6%, and low mineral contents [35]. This bran is the material that will be incorporated into the polymer matrices in the present research.

In order to conduct this study, samples of achira fiber were obtained from industrial food waste. The equipment and methods used in the laboratory tests performed during this research were as follows:

- TGA Test: TGA 550 Equipment, TA Instruments.
- DSC Test: DSC25, TA Instruments.
- FTIR Test: IRTracer-100, Shimadzu.
- Zeiss Stemi 305 Stereoscope.
- Moisture Balance PCE-MB 120C.

Figure 1 shows the bran obtained from the extraction process of achira starch collected in situ and Figure 2 shows the achira fiber observed under a stereoscope at 45× magnification.

Figure 1. Bran obtained from the extraction process of achira starch collected in situ.

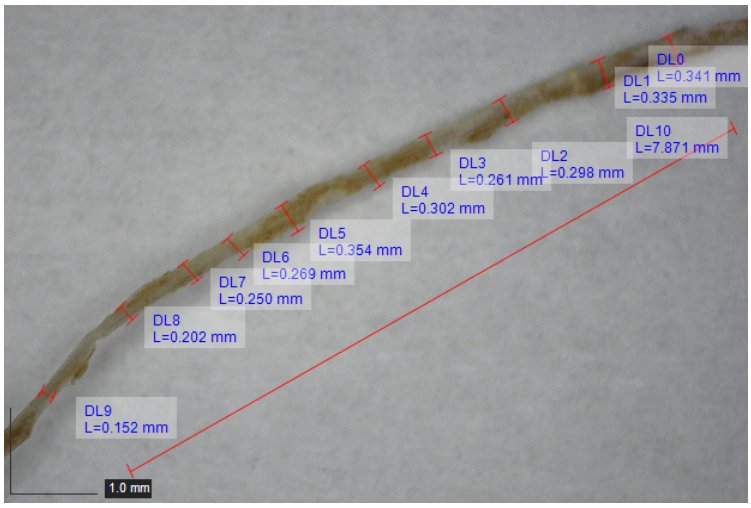

Figure 2. Achira fiber observed under a stereoscope at 45× magnification.

2.1. Thermogravimetric Analysis (TGA)

The thermal stability of the fiber was evaluated using the TA Instruments TGA 5500 thermogravimetric analyzer, with a heating rate of 10 °C/min up to 900 °C, under a controlled inert atmosphere.

The test was conducted on an achira fiber sample, with ASTM E1131-20 [37] serving as a reference for this test.

2.2. Differential Scanning Calorimetry (DSC)

For the differential scanning calorimetry, TA Instruments DSC 250 was used, considering ASTM D3418-21 [38] as a reference. The achira fiber sample underwent temperature variations from −40 °C to 398 °C to measure the material's response. The DSC curve was obtained during the second heating.

2.3. Fourier Transform Infrared Spectrometry (FTIR)

ASTM E1252-98 [39] was utilized as a reference for performing this test, which was conducted on two achira fiber samples. The spectra of the samples were acquired using an IRTracer-100 Shimadzu infrared spectrophotometer within a range of 400–4000 cm^{-1}. The

measurements involved 32 scans at a resolution of 8 cm^{-1}, and the data were recorded in terms of transmittance.

2.4. Moisture Content

The ASTM E871-82 [40] standard was employed to determine the moisture content of the achira fiber samples.

Seven samples of 1 g of achira fiber were utilized in the test, using a Sartorius Entris 224-15 digital balance. The samples underwent a drying process for 16 h at 103 ± 1 °C. Afterward, the sample was placed back in the oven for 2 h until the weight change was less than 0.2%. The moisture percentage was calculated using Equation (1) [41]:

$$\% \, moisture = \frac{W_i - W_f}{W_i} \cdot 100 \quad (1)$$

where W_i is the initial weight of the samples and W_f is the final weight of the sample.

2.5. Moisture Absorption

There are studies that conclude that the fibrous residues of achira rhizomes have a high-water retention capacity, which is beneficial for avoiding syneresis [42,43].

To determine the percentage of moisture absorption, seven 1 g samples of achira fiber were taken using a Sartorius Entris 224-15 digital scale. Subsequently, they were immersed in distilled water (pH 6.5) for 24 h and then weighed again to record their saturation point weight. For this measurement, any water adhering superficially to the fiber was removed using absorbent paper. Equation (2) was used to calculate the absorption percentage [41]:

$$\% \, absorption = \frac{W_i - W_s}{W_i} \cdot 100 \quad (2)$$

where W_i is the initial weight of the saturated sample and W_s is the weight of the dry sample.

2.6. Bulk Density

The bulk density of the fiber bundles was determined using the volume displacement method, which involves measuring the weight of the fiber bundles and the volume of water displaced. To conduct the measurements, seven fiber samples of equal weight were taken. This allowed us to observe and quantify the variation in the volume of water in a 100 mL test tube immediately after immersing the fiber samples.

Two types of bulk density measurements were performed, taking into account the varying moisture content.

- Fiber samples with ambient humidity.
- Fiber samples at the point of moisture saturation.

The total mass of submerged fiber was calculated. After submerging the samples, the dislodged volume was quantified using Equation (3) [41].

$$\delta = \frac{m}{vd} \quad (3)$$

where δ is the bulk density, m is the mass of the submerged fiber, and vd is the dislodged volume.

2.7. Lignin Percentage Determination

The Klason lignin extraction method was employed as a reference to determine the percentage of lignin [44]. Hydrolysis was initiated by adding 5 mL of 72% sulfuric acid to the fiber for two hours in a water bath at 20 °C. Next, a second hydrolysis was performed by diluting the acid to 3% and heating the mixture at 120 °C for 1 h. The filtered acid-insoluble residue corresponds to Klason lignin [45,46].

3. Results

3.1. Thermogravimetric Analysis (TGA)

The thermogram in Figure 3 depicts a blue curve (TGA) indicating the initial mass loss and a green curve (DTG) representing the derivative of the mass loss. The first change in the slope corresponds to the loss of water and highly volatile extractives present in the achira fiber sample, resulting in an 8.8% weight reduction at 128 °C. This was followed by the degradation of hemicellulose and cellulose glycosidic bonds at 230 °C, resulting in a weight loss of 58.61% at the end of this phase at 349 °C. In the third phase, the fibers begin to burn and α-cellulose degrades, yielding similar results to those reported by Indran and Raj [47]. Pyrolysis of the fiber occurred at 470 °C, resulting in a loss of 10.28% of the mass.

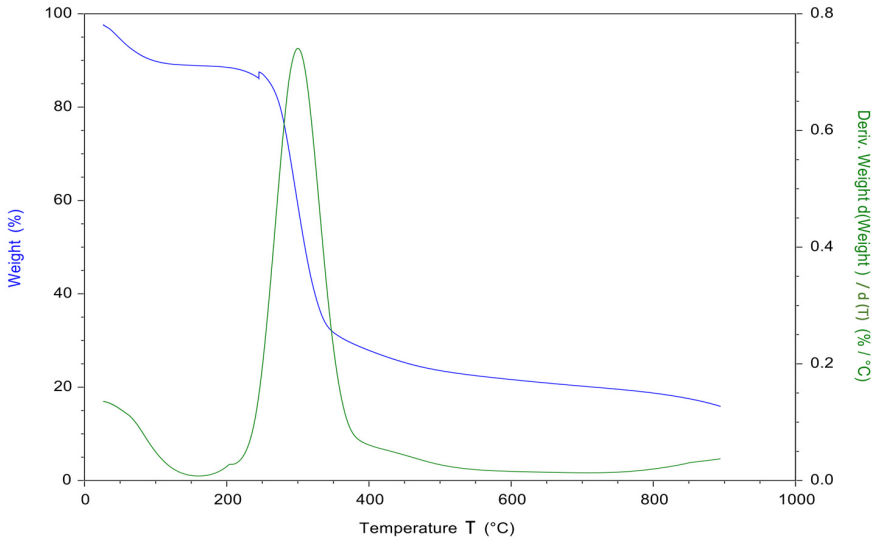

Figure 3. Thermogravimetric analysis (TGA) of achira fiber.

3.2. Differential Scanning Calorimetry (DSC)

Figure 4 illustrates the characteristic DSC of achira fiber up to 400 °C, offering insights into the fiber's structural modifications as a reaction to fluctuations in temperature. The plot depicts the alteration in heat flow relative to temperature. An endothermic peak is noticeable, signifying heat absorption during the fiber's melting phase, along with an exothermic process indicating the liberation of heat linked to the fiber's degradation or combustion.

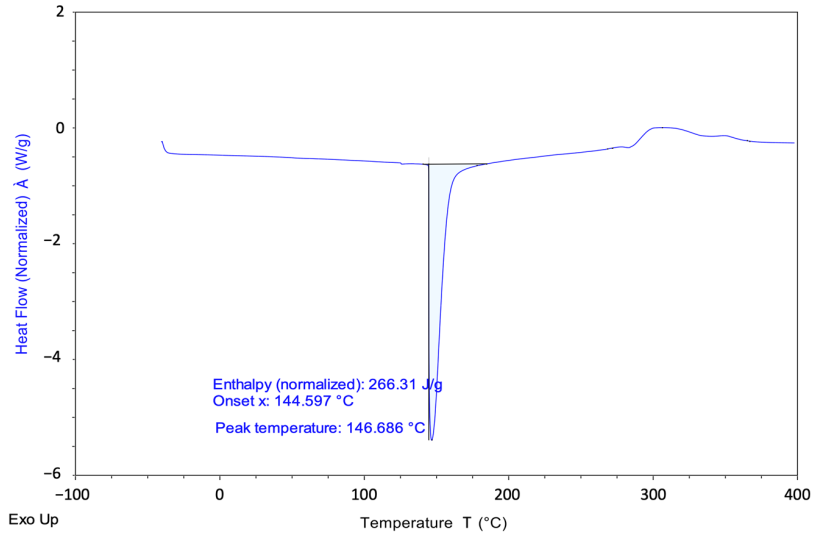

Figure 4. Differential scanning calorimetry.

The peak on the graph corresponds to the highest rate of material melting. As illustrated in Figure 4, the melting temperature of achira fiber is measured at 146.68 °C. Additionally, being an endothermic process, the heat flow necessary for the fiber's melting was calculated to be 266.31 J/g. One possible reason for the lack of Tg (glass transition temperature) visibility is that the thermal transition may exhibit small amplitudes in certain natural fibers, rendering it undetectable in the DSC.

3.3. Fourier Transform Infrared Spectrometry (FTIR)

Infrared spectroscopy is used to identify the bonds present in organic molecules, allowing for the determination of functional groups based on their characteristic vibrations at specific wavelengths [48]. It is important to highlight that obtaining the FTIR spectrum of all the compounds present in the fiber was not possible due to constraints in research funding.

Figure 5 shows the results of FTIR obtained for two samples of the achira fiber, showing absorption peaks at 3329.14 cm^{-1} and 3321.42 cm^{-1}, indicating axial deformations of the hydroxyl (OH) bond due to the presence of cellulose. The peaks at 2920.23 cm^{-1} and 2924.09 cm^{-1} are observed due to the stretching of the SP3 C-H bonds from cellulose vibration [47]. The peak value at 1616 cm^{-1} indicates the stretching of aromatic C=C with a strong conjugated C-C bond, attributed to the lignin content in the fiber [49]. The peak in the band at 1319 cm^{-1} is attributed to a strong acyl C-O with a superimposed C-H stretching of the phenols and esters. The bands in the natural fibers may vary around $\pm$16 cm^{-1} from their position [47].

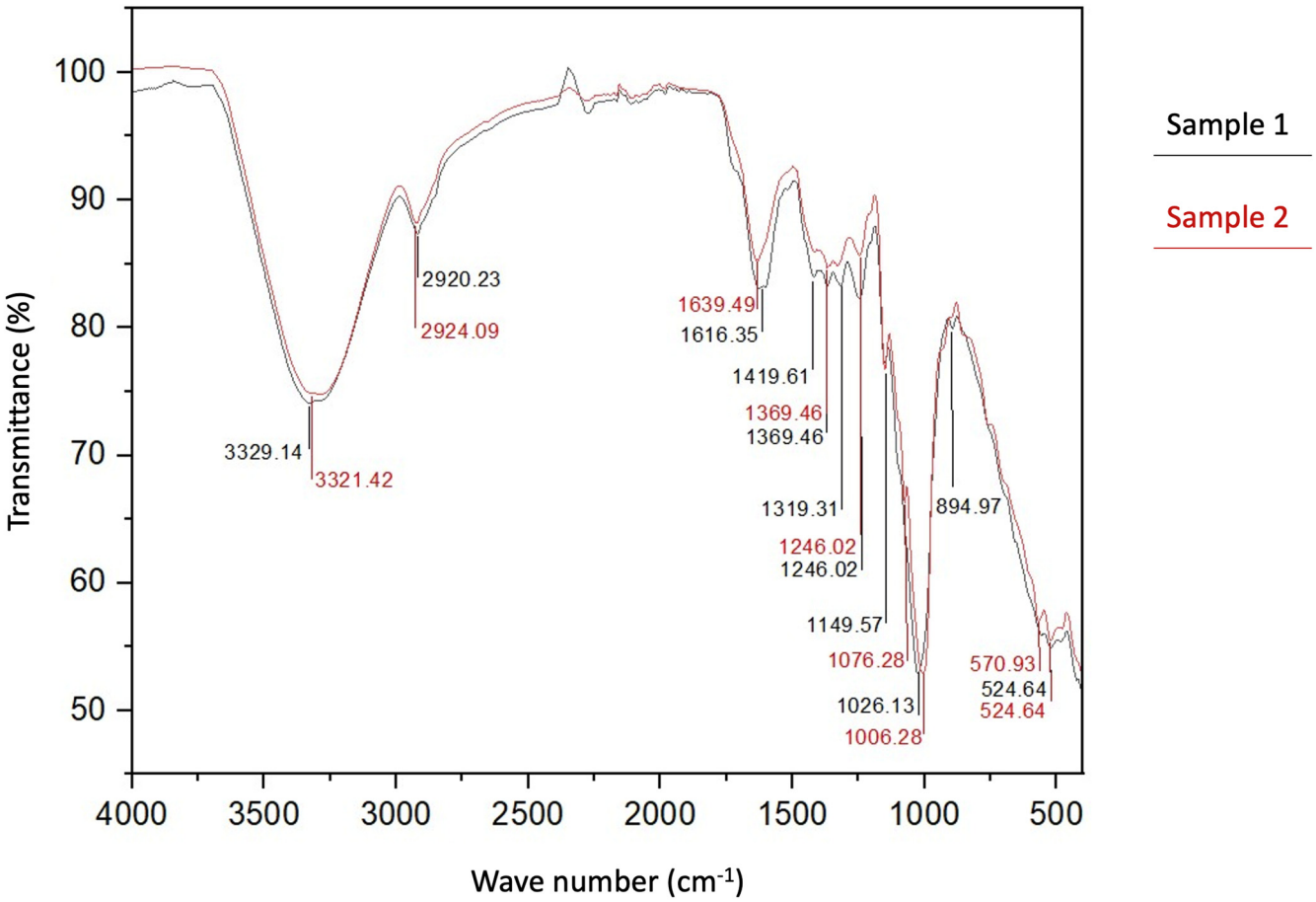

Figure 5. Fourier Transform Infrared Spectrometry (FTIR) for Sample 1 and Sample 2.

Lignin is one of the three main components that constitute the plant cell wall, along with hemicellulose and cellulose, which serve as its raw material. The presence of this natural adhesive (biopolymer) in different areas of the plant's natural fiber weakens the

mechanical properties of the composite materials, as it hinders optimal bonding between the matrix and the fiber [50].

3.4. Moisture Content

The moisture percentage of natural fibers is a significant characteristic of their subsequent applications, enabling them to be utilized in various processes. Controlling this variable enhances adhesion and performance due to the influence of elasticity, flexibility, and mechanical strength [51].

As indicated in Table 1, the average moisture content of the samples amounts to 11.007% of the fiber weight.

Table 1. Achira fiber moisture content in each sample.

Sample	Moisture Content (%)
1	10.56
2	10.00
3	11.37
4	11.01
5	11.53
6	11.23
7	11.31

3.5. Determining the Moisture Absorption Percentage

According to the data presented in Table 2, the moisture absorption percentage of the seven samples was 81.58%. The higher the porosity of the fibers, the greater their moisture absorption, which leads to a decrease in fiber strength.

Table 2. Moisture absorption percentage in each sample.

Sample	Moisture Absorption (%)
1	80.06
2	81.85
3	82.35
4	82.08
5	81.01
6	81.48
7	82.24

3.6. Bulk Density

At zero humidity, the bulk density of the achira fiber averaged 0.64 g/mg, while at its saturation point, it averaged 0.23 g/m^3 (Table 3).

Table 3. Bulk density results.

Sample	Density at Zero Humidity g/m^3	Density at the Saturation Point of Humidity g/m^3
1	0.67	0.23
2	0.77	0.23
3	0.72	0.23
4	0.63	0.23
5	0.46	0.24
6	0.72	0.22
7	0.50	0.24

3.7. Determination of the Percentage of Lignin

The percentage of lignin extracted from the 0.8 g fiber sample was 8%, resulting in the acquisition of 0.064 g of lignin. This lignin value is comparable to that of other natural fibers like hemp, which has a lignin percentage ranging from 8 to 10% [52].

4. Discussion

Most thermal degradation tests on biomass aim to analyze their potential as biofuels [19,53]. The study of the basic behavior of materials against thermal degradation and a simple reaction model are sufficient for practical engineering applications [54]. However, in recent years, these investigations have focused on analyzing the synthesis process of polymeric compounds with natural fibers [55–58]. It has been seen that the weight loss of the fibers during the heating process is due to the deconstruction of cellulose, hemicellulose, and lignin constituents [56,59,60].

Currently, most studies on natural fiber-reinforced compounds include thermal characterization using DSC, both for the composite material itself [61–66] and for the natural fibers [67–69]. The FTIR analysis method is used to detect characteristic functional groups in the fibers by passing infrared light through the sample fibers [70,71].

Natural fibers derived from crop residues can be used to reinforce a variety of thermoplastics after being processed and treated through mechanical, thermomechanical, semichemical, and chemical treatments [72]. These treatments modify the lignin content, thus affecting the cellulose and hemicellulose content in the treated fiber [73].

The bulk density of other fibers, such as cotton, jute, sisal, and coconut, is higher than the bulk density of the achira fiber. However, bamboo fiber has a similar density to that of achira fiber [74]. The composition of cellulose, lignin, pectin, and hemicellulose in plant fibers affects their properties. It is a common practice to remove lignin and pectin to enhance the reinforcing effect of natural fibers. Previous studies have shown that flax fiber demonstrated superior thermal resistance among the natural fibers examined, mainly due to its low lignin content of 2%. In comparison, jute had a lignin content of 11.8%, and sisal had a lignin content of 9.9% [75].

The first phase in creating mixtures with polymeric and agro-industrial waste through mechanical recycling is to determine the mechanical and thermal properties of the materials. "In recent years, chemically recyclable polymers have been designed and developed to manage plastic waste at the end of its useable life" [76], and including natural fibrous waste within its structures is a good solution to support circular economy (CE) policies. Waste management technologies are evidently one of the most important aspects currently being considered for CE [77]. This helps maximize the properties of the materials.

In regard to the fiber moisture content of 11.007% that was found, it is important to note that the presence of moisture in the fibers can affect their use as reinforcement due to the hydrophobic nature of polymeric resins. This diminishes interfacial bonding, resulting in a reduction in the mechanical properties of the composite [78]. Therefore, it is suggested that the fibers undergo chemical or thermal treatment to optimize bonding when used as a reinforcement material with polymeric matrices.

The moisture percentage obtained for the achira fiber presents values similar to those found in other studies for banana, pineapple, and sisal fibers [79]. The results of the moisture absorption percentage showed an average of 81.58% for the seven samples. One of the main challenges in using natural fibers in polymeric matrices is their strong polar character, which creates incompatibility with the matrix. Surface treatments, both chemical and physical, can help alleviate this issue while simultaneously reducing moisture absorption.

When the fiber has zero moisture, its density (0.64 g/mg) is higher than its saturation point density (0.23 g/m^3). This suggests that in order to optimize fiber transportation and storage costs, it is preferable for the fiber to have the least amount of moisture possible to optimize space. The results from the TGA test reveal important aspects, including the fiber's hydrophilic character, with a significant retained moisture percentage of 8.8%.

This indicates that the fiber should undergo chemical or thermal treatment to improve its adhesion and make it suitable for use as a reinforcement material in polymeric matrices. In terms of thermal stability, the decomposition temperature begins at 230 °C, which is similar to other reported fibers, such as bamboo [80]. It should be noted that the selected polymer for processing the composite material has a lower working temperature. Therefore, once this limit is exceeded, the achira fiber will start to degrade.

5. Conclusions

Considering the characteristics of an achira fiber and its similarities to other natural fibers mentioned in the scientific literature throughout this article, it is feasible to integrate it into polymeric matrices. The average apparent density of an achira fiber at ambient humidity was 0.64 g/mg, and it averaged 0.23 g/m^3 at its saturation point. This suggests that to optimize transportation and storage costs, it is preferable for the fiber to have as little moisture as possible to save space. Compared to cotton, jute, sisal, and coconut fibers, the achira fiber has a lower density, while the bamboo fiber has a similar density. The moisture content in the fiber was found to be 11.007%. It is important to note that the presence of moisture in the fibers affects their use as reinforcements due to the hydrophobic behavior of polymeric resins. This diminishes interfacial bonding and reduces the mechanical properties of the composite. Therefore, it is recommended that the fibers undergo chemical or thermal treatments to optimize adhesion when used as reinforcement material with polymeric matrices.

Based on the TGA test results, the degradation temperature of the fiber begins at 230 °C, which is similar to other reported fibers, such as the bamboo fiber. It should be mentioned that the selected polymer for processing the composite material has a working temperature lower than 230 °C. Therefore, once this limit is exceeded, the achira fiber will start to degrade. The melting temperature of the achira fiber was determined to be 146.68 °C. Additionally, as an endothermic process, the heat flux required for the melting of the crystalline phase is 266.31 J/g. The FTIR characterization revealed the presence of a small amount of lignin in the achira fiber. This could be advantageous since lignin is a natural adhesive that can enhance the physical and/or chemical interaction between polymers and fibers.

The residues from the starch extraction process, in this case, Sagú bran, have the potential to serve as a reinforcement for composite materials, acting as a possible substitute for conventional plastics, contributing to energy production, paper manufacturing, soil stabilization, and primarily composting. Sagú products have found extensive use in the food industry. Nevertheless, it is crucial to properly characterize the resulting process residues to explore their potential applications. Moreover, addressing the issue of bran accumulation in the starch extraction chain results found in this research provide an opportunity to utilize these materials and contribute to the advancement and strengthening of the recycled raw materials market, fostering sustainability and the circular economy.

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