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Improving textile circular economy through banana fibers from the leaves central rib: effect of different extraction methods

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ABSTRACT

In the last decades, the use of agricultural wastes as a source of natural cellulosic fibers has become urgent, given the growing demand for natural and synthetic fiberss. Cellulose is a renewable natural resource and the most abundant in nature, being obtained from biomass such as wood, cotton and vegetables. Banana fiber is of great interest as bananas are one of the most consumed fruits in the world. Banana fiber is extracted from the banana pseudo-stems and leaves that remain after the fruit is harvested. Added value products based on banana fiber are an innovative material with strong potential in the market. The extraction of fibers from the banana plant can be carried out mechanically, chemically, or biologically. A combination of these methods is also possible, meaning that mechanical extraction can be followed by other treatments. In this work, the extraction of banana fibers was carried out using different methods, namely, manual extraction, chemical extraction (sodium hydroxide (NaOH)), biological extraction (retting in water at room temperature and 35 ºC) and boiling water. All the extracted fibers were analyzed using Optical Microscopy, Fourier-Transform Infrared Spectroscopy coupled with an Attenuated Total Reflectance accessory (ATR-FTIR), Thermogravimetric Analysis (TGA), Field Emission Scanning Electron Microscopy (FESEM), X-ray Diffraction (XRD) and their mechanical properties were also evaluated. Fibers with diameters between 27.46 and 240.89 μm were obtained. Chemical extraction increased the tensile strength of the fibers by effectively removing non-cellulosic components, but some cellulose degradation was observed. Biological extractions removed lignin and hemicellulose, resulting in increased fiber individualization and homogeneous fiber surfaces with improved thermal properties.

1. Introduction

Since the textile industry is one of the most polluting industries in the world, the sustainability of its processes and products has become more and more important. Nowadays, with the environmental growing consciousness, there has been an increasing interest in the use of natural fibers in replacement of synthetic ones, due to their biodegradability, biocompatibility, low-weight, high abundance, low-cost, good electrical resistance and sound insulation (Abral et al., 2019; Ilyas et al., 2019; Kenned et al., 2020; Araújo et al., 2021). Thus, the textile industry has been exploring and opting for natural fibers of animal and plant origin (Patel and Patel, 2022).

Agricultural processing generates millions of tonnes of lignocellulosic biomass annually worldwide. These lignocellulosic biomasses are derived from the processing of wood, forest plants and food crop residues such as wheat straw, corn husk fiber, pineapple leaves, banana leaves and stems, between others (Diarsa and Gupte, 2021). Banana fiber is of great interest given that bananas are one of the most widely consumed fruits worldwide, generating large amounts of lignocellulosic waste (Balda et al., 2021).

The banana plant belongs to the family of plants Musaceae and it regrows in the same location, not requiring additional water, soil, or fertilizers, and their strong roots help maintain soil stability, preventing landslides (Patel and Patel, 2022; Balda et al., 2021). India, China, Indonesia and Brazil are the largest producers of bananas (Food and Agriculture Organization of the United Nations, 2023). Although not on such a large scale, the Azores and Madeira in Portugal also grow bananas, which are one of the most profitable agricultural activities,

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especially on the island of Madeira, where 20 % of the economy is linked to the production of tropical fruits (National Institute of Biological Resources, 2008).

The pseudostem of the banana plant is similar to the trunk found in most fruit trees. It provides and transports nutrients from the soil to the fruits. After banana harvesting, the pseudostem is cut down, becoming residual biomass, as the banana plant becomes unusable for the next harvest (Subagyo and Chafidz, 2018).

Most of the time, the banana plant residues are left to decompose in the soil to act as fertilizers, releasing a significant amount of carbon dioxide or methane gas or they up in landfills. Therefore, they have a negative impact on the environment (Akinyemi et al., 2019). Approximately 220 tonnes of lignocellulosic residues are generated per hectare of banana plantation (Diarsa and Gupte, 2021). In an attempt to make use of them, some of these residues have been applied in product development, while others are used as biomass for energy production (Balda et al., 2021; Akinyemi and Dai, 2020). However, one of the most promising approaches may involve the extraction of fibers from these residues (pseudostems and leaves) for the development of new sustainable materials (Manickam and Kandhavadivu, 2022; Priyadarshana et al., 2022; Sivaranjana and Arumugaprabu, 2021).

The extraction of fibers from the banana plant can be carried out mechanically, chemically, or biologically. A combination of these methods is also possible, meaning that mechanical extraction can be followed by chemical or biological treatments (Balda et al., 2021). The extraction method of the fiber has an impact on its quality, chemical composition, structure, and properties (Ramamoorthy et al., 2015). Although there are many publications on the extraction and processing of banana fiber, they almost always focus on the use of fiber from the pseudostem and publications that make use of fiber from other banana crop residues, such as leaves, are rare. This potential therefore remains untapped. Additionally, reports of the extraction of these fibers from european banana plants, especially from Portugal, are scarce.

In this work, fibers were extracted from the central rib of banana leaves from Portugal. As far as the authors know, this is the first report of fibers extraction from Portuguese banana leaves, increasing even more the novelty of this work. Firstly, they were extracted only by manual scraping to serve as a control for the other extractions. Parts of the central rib of banana leaves were extracted in a solution with sodium hydroxide (NaOH) (chemical extraction) and in water (biological extraction) under different conditions, namely water retting at room temperature, water retting at 35 ºC and boiling water. At the end of the maceration period, manual scraping was carried out. The fibers obtained were analysed using different techniques and also evaluated for their mechanical properties.

2. Experimental

2.1. Materials

Banana leaves were collected with different lengths and from different parts of banana trees cultivated in Guimarães, Portugal. NaOH with 98 % purity in pellet form was used for the extraction of the banana fibers and purchased from Sigma-Aldrich. Distilled water was used in all procedures.

2.2. Methods

In order to extract the central rib from banana leaves, the lateral sections of the leaves were removed, as illustrated in Fig. 1. The central ribs were washed with running water to remove impurities. The leaves were of different colours, ranging from light green to dark green. They were about 30 cm wide and 1 m long.

Procedures were selected and adapted from methods reported in literature for banana fiber extraction (Mumthas et al., 2019a; Adamu, 2021; Soraisham et al., 2021; Khan et al., 2022; Motaleb et al., 2020). The extraction methods used are summarized in Fig. 2.

In the chemical extraction, the central rib of the banana leaves was cut into several sections of 30 cm long. A solution of NaOH 5 % (w/w) relative to the material was prepared. The banana rib to NaOH solution ratio used was 1:20. The samples were kept in the NaOH solution at 90 ºC for one hour before hand scraping extraction. Extracted fibers were washed many times with hot water until complete removing of the NaOH.

For water retting at room temperature, several sections of the central rib of the banana leaves were cut into 10 cm long pieces. After cutting, they were placed in a beaker with 900 mL of water, covered to keep the samples submerged, sealed with parafilm and left at room temperature. Samples were taken after 7 and 21 days of retting. After extraction, the fibers were washed with water and dried in an oven at 70 ºC for 24 hours. Water retting at 35 ºC follow a similar procedure to the water retting at room temperature. However, in this method, the temperature

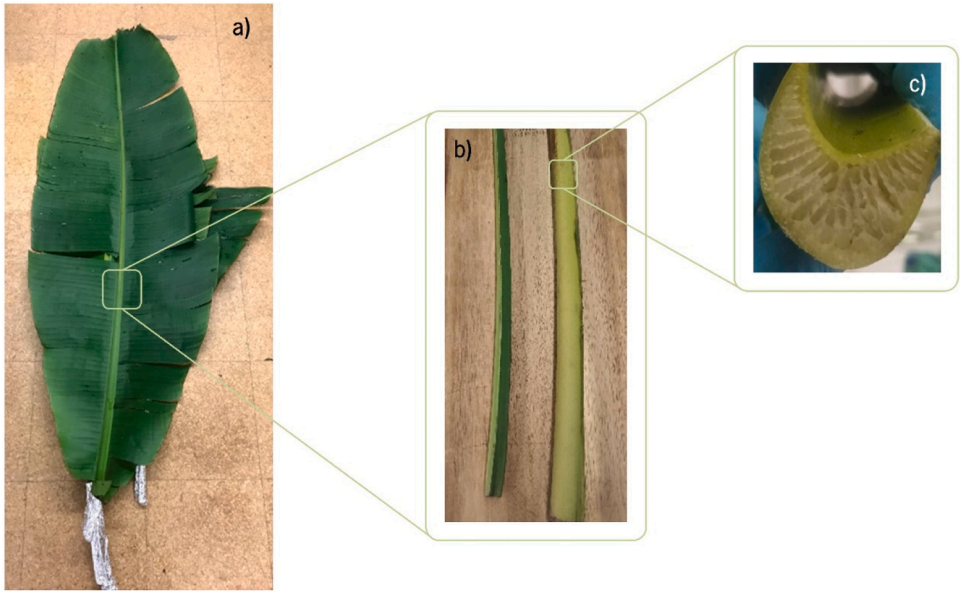

Fig. 1. a) Acquired leaves, b) Central rib of the leaf and c) Cross section of the central rib.

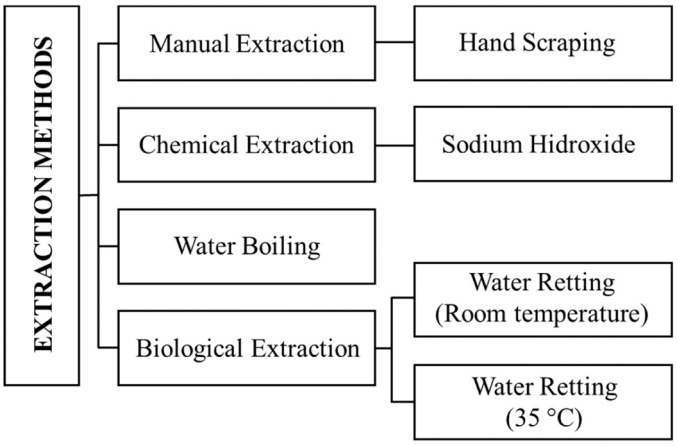

Fig. 2. Schematic diagram of the extraction methods applied to banana leaves.

was kept at 35 ºC and samples were taken after 14 and 28 days.

For the extraction by water boiling, several sections of the central rib of the banana leaves were cut to pieces of 25 cm. The samples were placed in a container with water, at 100 ºC for one hour.

In the manual extraction, the central rib of the banana leaves was hand scraped until fibers were obtained, with the help of knives and spatulas. Then, they were washed and left to dry at room temperature for 5 days.

2.3. Characterization of the extracted fibers

2.3.1. Optical microscopy (OM)

Optical Microscopy was used to observe the morphology of extracted banana fibers and for measure their diameter. The analysis of the samples was performed at 5x magnification using a Leica DM750 M Microscope (bright-field), equipped with a high-definition digital camera. After extracted for each method, 15 individual fibers were carefully separated and fixed on a slide. Each individual fiber was observed at various points along its length, with 50 diameter values recorded for each method studied, using ImageJ software.

2.3.2. Field emission scanning electron microscopy (FESEM)

Field Emission Scanning Electron Microscopy (FESEM) was used to analyse the surface and cross-sectional morphology of the obtained fibers. The sample analysis was conducted using a high-resolution Field Emission Scanning Electron Microscope NOVA 200 Nano SEM from FEI Company (Hillsboro, OR, USA), at an acceleration voltage of 10 kV. Prior to imaging, the samples were coated with a very thin film (10 nm) of Au-Pd (80–20 % w/w) using a sputter coater 208 HR Cressington Company (Watford, United Kingdom), coupled with a high-resolution coating thickness controller MTM-20 Cressington.

2.3.3. Attenuated total reflectance - fourier transform infrared spectroscopy (ATR-FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) coupled with an Attenuated Total Reflectance (ATR) accessory was used to evaluate changes in the chemical composition of the fibers after extraction. The sample analysis was performed using the IRAffinity S1 equipment from SHIMADZU (Kyoto, Japan), equipped with an ATR accessory. Each spectrum was acquired in transmittance mode in a diamond ATR cell, accumulating 64 scan cycles with a resolution of 4 cm^{-1} in a spectral range from 400 to 4000 cm^{-1}. A bundle of fibers from each method performed was submitted for analysis. Three measurements were made on each beam.

2.3.4. Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA) was performed to assess the thermal stability of the obtained fibers. The study of the thermal behaviour of the fibers was conducted using the HITACHI STA7200RV equipment. The samples were heated from 30 ºC to 600 ºC at a heating rate of 10 ºC/min under a nitrogen atmosphere. For the analysis, the samples were reduced to powder and placed in a crucible, connected to a microbalance, which was heated under controlled conditions and the mass losses were monitored.

2.3.5. X-ray diffraction (XRD)

X-ray Diffraction (XRD) was used to analyse the crystal structure of the raw and extracted fiber by different methods. The sample analysis was performed using a Bruker AXS D8 Discover diffractometer, using a Cu-Kα radiation at a voltage of 40 kV and a current of 40 mA. Data were recorded for 2θ values ranging from 5º to 60º. For the analysis, the samples were reduced to powder, suspended in ethanol and placed on a slide through ethanol drying. The crystallinity index of the different samples was calculated from the diffraction intensity values corresponding to specific peaks observed in the X-ray diffractograms, accordingly with the Eq. 1:

$$\mathrm{CrI}(\%) = (\mathrm{I\_002} - \mathrm{I\_am})/\mathrm{I\_002} \quad \times 100 \tag{1}$$

Where, *CrI* is the crystallinity index, I_{002} represents the maximum intensity, typically found between $2\theta = 21^{\circ}$ and $2\theta = 23^{\circ}$, for crystalline cellulose and I_{am} is the intensity of the peak at $2\theta = 18^{\circ}$ for amorphous cellulose (Soraisham et al., 2021; Das et al., 2017a).

By measuring the intensities of these peaks and applying the formula to calculate the crystallinity index, it is possible to determine the crystallinity of the cellulose fraction into the fibers under study. The crystallinity index provides information about the degree of crystalline and amorphous regions present in the cellulose material (Das et al., 2017a).

2.4. Mechanical properties

To evaluate the mechanical properties of the extracted fibers, tensile tests were performed. These tests were carried out according to D 5035, 2003 (D, 5035–95, 2003) standard, using a Hounsfield H100KS dynamometer and a 250 N load cell. The samples used had a minimum length of 70 mm. The initial distance between the jaws was set to 50 mm, with a displacement speed of 1 mm/min. Ten repetitions of each extraction or treatment method were performed. Tensile strength, elongation at break and Young's modulus were calculated from the stress-strain curves.

The diameter and linear density of the fibers were measured prior to testing. The average diameter value obtained for each method was used for the diameter measurement. To determine the linear density, the fiber samples were conditioned in an environmental chamber at 20 ± 2 °C and 65 ± 2 % relative humidity for at least 24 hours according to NP EN 20139, 1996 (NP EN 20139, 1996) standard. After conditioning, each sample was weighed using an analytical balance and its length was measured using a crimp tester according to the ISO 07211–5–1994, 2002 standard. Fiber classification establishes differences between them and serves as a guideline for the commercialization and production of specific fabrics or for comparing fibers. To meet this purpose, a form of expression to measure the fiber diameter was created, known as linear density or yarn count. Yarn count is represented by a value that expresses the relationship between mass and length. The results represent the relationship between the weight and the length of the fiber yarn in Tex units (Costa et al., 2013a).

3. Results and discussion

Looking at the visible aspect of the extracted fibers different color shades were obtained, depending on the extraction process, as shown in Fig. 3.

Given that this process was carried out manually and not automatically, it was observed that it was difficult to remove all the impurities

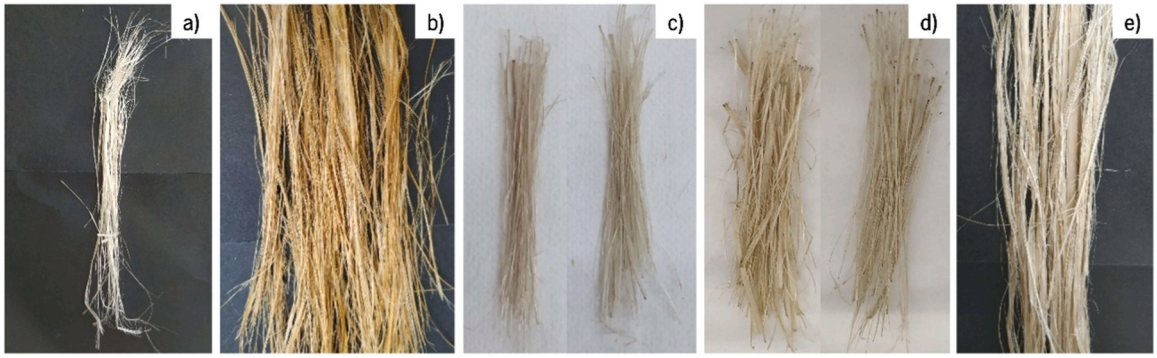

Fig. 3. Fibers obtained from the different extraction procedures: a) hand scraping only; b) NaOH; c) water retting at room temperature (7 and 21 days, respectively); d) water retting at 35 ºC (14 and 28 days, respectively) and e) boiling water.

present in the fibers (Fonseca-Pinheiro et al., 2022). In the chemical extraction with NaOH and in the biological extractions, a certain improvement in the scraping of the fibers was observed compared to the hand scraping only. However, in water retting at room temperature, no positive effect was observed, neither after 7 nor after 21 days, which led to a second process with a longer time and higher temperature (14 and 28 days at 35 ºC). In this second process, it seems that an improvement in fiber extraction was observed. The fibers that appear to be easier to extract after 28 days compared to 14 days. The boiling water process also seems to improve slightly the fiber extraction. However, chemical extraction with NaOH seems to be the process that allowed a more efficient extraction.

3.1. *Surface and morphologic analysis of extracted fibers samples*

Several fibers from each method were observed under the optical microscope to evaluate their morphology, mainly their uniformity and diameter. Images of the fibers extracted by different methods are exhibited in Fig. 4.

Considering the characteristic diversity of natural fibers, the OM images presented are a demonstration of the most frequent features observed for each method performed.

Looking at the image of a fibers extracted by hand scraping only (Fig. 4-a), it is possible to notice an elongated and slender structure, resembling a long, thin cylindrical thread, as intended. However, some irregularities are visible on its surface, indicating possible non-fibrous components that might not have been completely removed by the scraping process. The surface of the fibers extracted by chemical retting (*i.e.*, NaOH) appears to be more uniform (Fig. 4-b). Fibers extracted by water retting at room temperature show irregularities and the presence of non-fibrous residues (Figs. 4-c and -d) and no improvement was noticed compared to the fibers extracted by hand scraping only (Fig. 4-a). Fibers extracted after water retting at 35 ºC (Figs. 4-e and 4-f), unrelated of the retting time, as well as after boiling water extraction (Fig. 4-g), shown uniform and homogeneous surface almost throughout their entire length. These results illustrate the possible effectiveness of both methods in removing non-fibrous matter without the use of chemical reactants.

All histograms corresponding to the diameters of the fibers obtained in each method are presented in a more detailed manner in Fig. 5 and Table 1.

The mean and standard deviation are also shown. Diameters of the studied banana fibers ranged from 11.36 μm to 204.89 μm. The calculated mean values were 92.33 μm for extraction by boiling water, followed by 105.20 μm for water retting at room temperature, 107.01 μm for water retting at 35 °C, considering the average diameter values at the end of both retting procedures, i.e., at 21 days and 28 days, respectively. 110.86 μm for chemical extraction with NaOH and finally, 113.65 μm for hand scraping. These results show that the extractions reduced the diameter of the fibers compared to hand scraping alone. These reductions may indicate that chemical and biological extraction methods are removing non-cellulosic materials from the fibers (Mumthas et al., 2019). The water retting method, specially at 35°C, and at room temperature for 21 days resulted in fibers with higher diameters when compared with the hand scraped ones. This is mostly likely related to the swelling of the fibers in contact with water. The average values obtained are in line with the study carried out by Mukhopadhyay et al (Mukhopadhyay et al., 2008). where they recorded fiber diameters ranging from

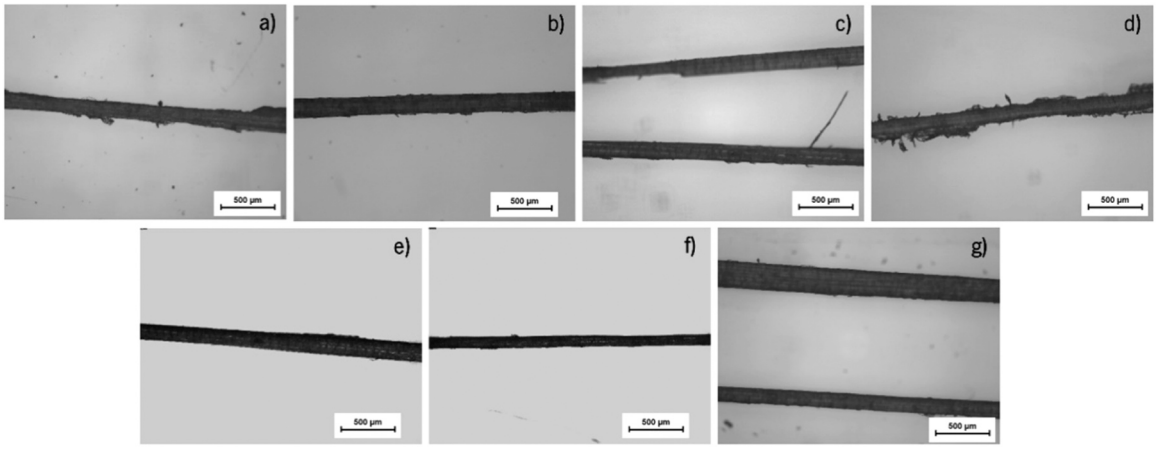

Fig. 4. Images of OM of banana fibers after a) hand scraping only; b) NaOH extraction; c) 7 days and d) 21 days of water retting at room temperature; e) 14 days and f) 28 days of water retting at 35 ºC and g) boiling water extraction.

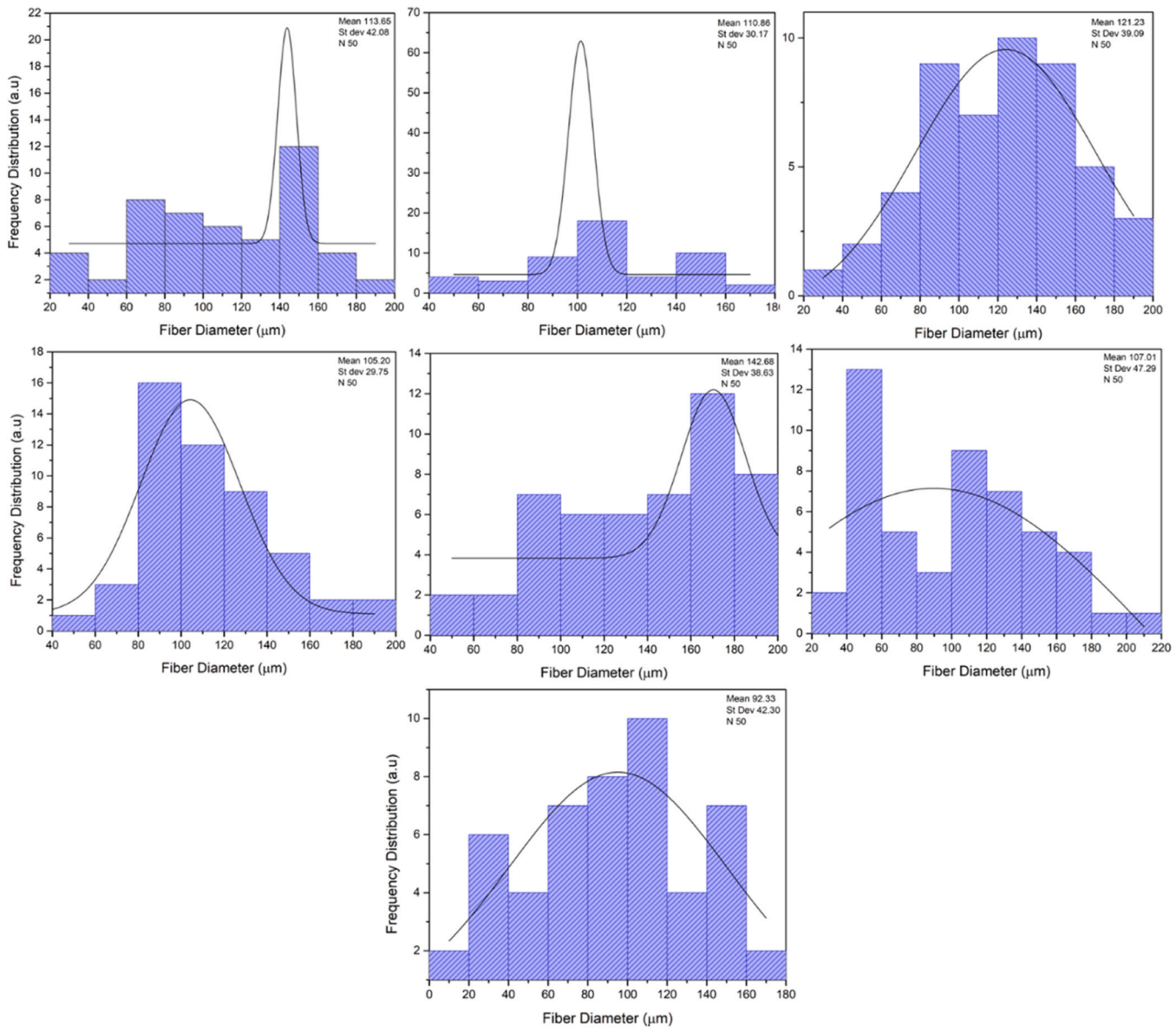

Fig. 5. Histograms of banana fibers diameters after a) hand scraping only; b) NaOH extraction; c) 7 days and d) 21 days of water retting at room temperature; e) 14 days and f) 28 days of water retting at 35 ºC and g) boiling water extraction.

Table 1
Effect of the extraction method in the banana fibers diameter.

Fibers samples	Range of diameters (μm)
Hand scraped	27.46 – 191.86 (113.65)
NaOH extraction	47.35 – 162.92 (110.86)
Retted at room temperature (7 days)	29.47 – 185.28 (121.23)
Retted at room temperature (21 days)	41.67– 181.86 (105.20)
Retted at 35 ºC (14 days)	53.03– 199.39 (142.68)
Retted at 35 ºC (28 days)	53.03– 199.39 (142.68)
Water boiled	34.09 – 240.89 (107.01)

80 μm to 320 μm for mechanically extracted banana fibers.

The FESEM analysis was carried out to evaluate the surface and morphology of the cross-section of banana fibers samples obtained through hand scraping, chemical extraction with NaOH and biological extraction, specifically water retting at 35 °C for 28 days and boiling water. The obtained images are presented in Fig. 6.

By using FESEM images at different magnifications, it is possible to assess the quality of the fibers, as it allows for the observation of the presence or absence of scales or irregular shapes along their length. Additionally, it allows for the determination of the fibers grouping, structure, longitudinal and transversal cuts, and other relevant parameters (Costa et al., 2013a).

In the longitudinal section images (Fig. 6 a), b), c)), it is possible to observe unitary cells oriented in parallel, forming tubular structures composed of lignin, hemicellulose, and cellulose. This organization may confer the structure a high rigidity, making it highly resistant and difficult to break (Patel and Patel, 2022).

The cross-sectional view of the banana fiber (Fig. 6 d), e), f)) demonstrates that the fiber is composed of several smaller fibers grouped together. According to Mumthas *et al* (Mumthas et al., 2019)., these smaller fibers can vary in number from 10 to 100 and are similar to cotton fibers. It was also observed that the cross-section of the banana fiber is multicellular and has a porous structure with a hexagonal shape, i.e. it has voids within the fiber structure. This characteristic can provide the fiber with good insulation and absorption properties (Soraisham et al., 2022).

The analysis of the images revealed that partially separated

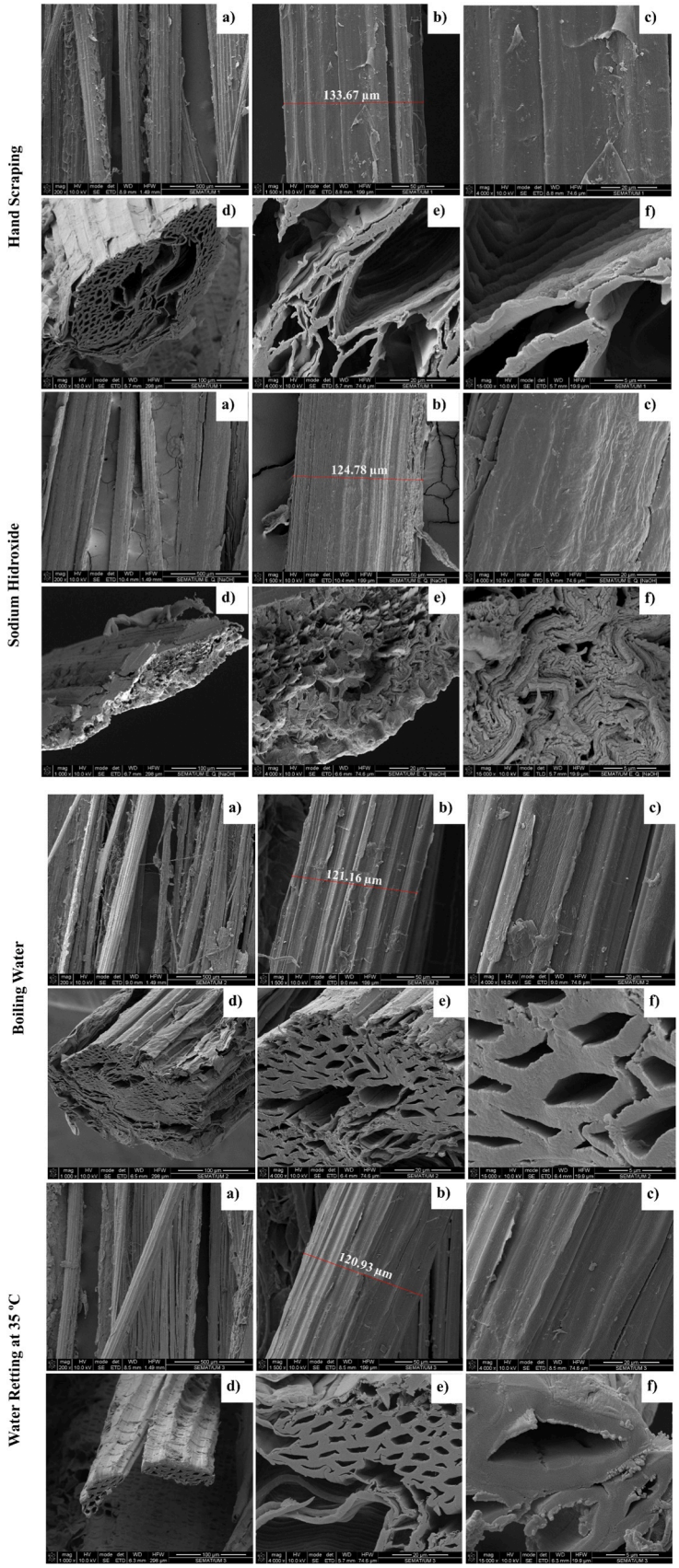

Fig. 6. FESEM images of banana fiber samples using different magnifications: a) 200x (500 µm), b) 1500x (50 µm), c) 4000x (20 µm), d) 1000x (100 µm), e) 4000x (20 µm) and f) 15000x (5 µm).

agglutinating materials were present on the surface of the extracted fibers. The removal of these agglutinating materials brings the fibers closer to each other, resulting in a denser fiber structure with less empty space between them, leading to a smaller diameter. These characteristics can contribute to the improvement of the mechanical properties and quality of the extracted fibers (Mumthas et al., 2019).

It was found that the fibers obtained by both chemical and biological methods not only had a cleaner surface, but were also smoother, due to the removal of non-cellulosic materials, than those obtained by hand scraping only (Soraisham et al., 2022). In addition, the final individual fibers show a slight separation. This is due to the removal of the outer layer of the fiber by dissolution in solution during the extraction process (Patel and Patel, 2022). Furthermore, Elanthikkal et al (Costa et al., 2013b). stated that manual scraping can damage the surface of the fibers, therefore it is advantageous to use other methods.

3.2. ATR-FTIR analysis

The ATR-FTIR spectra of the extracted fibers are displayed in Fig. 7.

The analysis of spectra of the fiber reveals distinct bands corresponding to the components of cellulose, hemicellulose, and lignin fractions, characteristic of lignocellulosic fibers (Das et al., 2017a).

The broad absorption band at 3334 cm^{-1} is attributed to the O-H stretching vibration of the hydroxyl group, indicating the presence of absorbed water, free phenols, primary and secondary aliphatic alcohols found in cellulose, hemicellulose and lignin (Mumthas et al., 2019; Balakrishnan et al., 2021). The presence of the hydroxyl group stretching band, associated with the presence of cellulose, may result in fibers with higher strength (Mumthas et al., 2019).

The absorption bands at 2920 cm^{-1} and 2850 cm^{-1} correspond to the stretching vibrations of the aliphatic C-H group, specifically CH_2, indicating the existence of cellulose and hemicellulose (Patel and Patel, 2022; Diarsa and Gupte, 2021).

The absorption band at 1730 cm^{-1} corresponds to the stretching vibration of the carbonyl group C═O and can be attributed to the presence the acetyl groups, typically found in hemicellulose (Balakrishnan et al., 2021). The absorption bands at 1595 cm^{-1}, 1315 cm^{-1}, and 1240 cm^{-1} correspond to the angular deformation vibration of C═C associated with aromatic rings, the asymmetric deformation vibration of C-H group and the stretching vibration of C-O, respectively, due to the presence of lignin (Diarsa and Gupte, 2021; Das et al., 2017a; Fonseca-Pinheiro et al., 2022; Elanthikkal et al., 2010a).

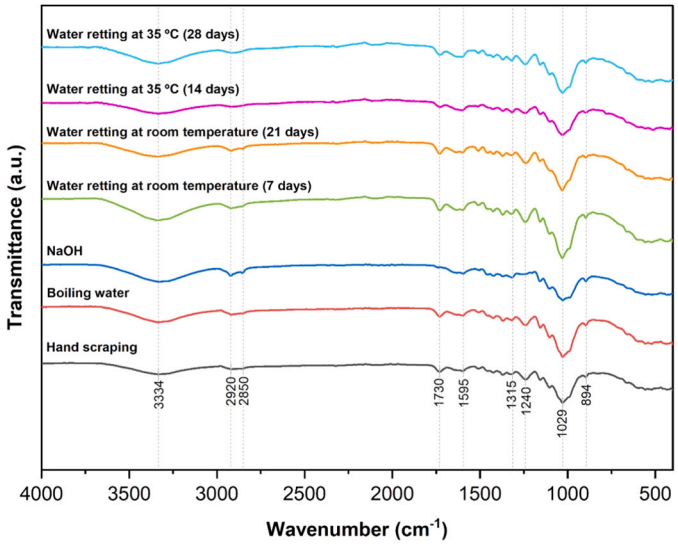

Fig. 7. ATR-FTIR spectra of banana fiber samples after hand scraping only, NaOH, water retting at room temperature for 7 and 21 days, water retting at 35 °C for 14 and 28 days and boiling water extraction.

The absorption bands at 1029 cm^{-1} and 894 cm^{-1} correspond to the stretching vibration of O-H and the deformation vibration of C-H, respectively, associated with cellulose (Diarsa and Gupte, 2021; Fonseca-Pinheiro et al., 2022; Balakrishnan et al., 2021; Elanthikkal et al., 2010a).

All the mentioned bands are summarized in Table 2.

All spectra are quite similar. However, they present some divergences, indicating changes in functional groups and fiber structure composition during the respective extraction processes (Diarsa and Gupte, 2021; Elanthikkal et al., 2010a). Analysing the spectrum of the fibers extracted by chemical extraction with NaOH, the absence of some characteristic bands of hemicellulose and lignin can be observed, leading to a reduction in the intensity of cellulose bands, suggesting the removal of non-cellulosic components, triggering cellulose degradation. More pronounced degradation of lignin, hemicellulose, and cellulose can adversely affect obtaining high-quality fibers, rendering them unsuitable for use in the textile industry (Patel and Patel, 2022).

When comparing the extractions by biological methods and analysing the resulting spectra from water retting at 35 °C, an increase in bands between 14 and 28 days suggests that retting for 28 days under these conditions gives better results, since an increase in cellulose content may be associated with the reduction of non-cellulosic components (Patel and Patel, 2022). On the other hand, in the case of water retting at room temperature, the bands decreased between 7 and 21 days. Therefore, it is likely that 21 days may have been excessive under these conditions, indicating that in the absence of temperature control, it may be difficult to properly manage the retting time, leading to unexpected results. It is worth noting that retting at 35 °C proved to be more efficient in terms of fiber extraction during the scraping process, in contrast to water retting at room temperature, where no significant improvement was observed. These observations indicate that temperature plays a crucial role in the biological extraction process and its control can significantly influence extraction efficiency and results. Therefore, careful control and consideration of temperature as an important variable is recommended to achieve more consistent and reliable results.

Chemical extraction seems to be excessively aggressive, degrading not only the non-cellulosic components of the fibers, but also the cellulosic ones, which can be very prejudicial to fibers quality. On the other hand, water retting at 35 °C allows the removal of non-cellulosic components, increasing the cellulosic content.

3.3. TGA analysis

The TGA was carried out to evaluate the thermal stability and understanding the degradation process of the banana fibers obtained by hand scraping, chemical extraction with NaOH and biological extraction methods (*i.e.* water retting at 35 °C for 28 days and boiling water). In view of the previous results, the biological extraction method using water retting at room temperature was not included in this and subsequent characterisation analyses, as it involves variables that we cannot control, making it difficult to replicate. The TGA together with differential thermogravimetric analysis (DTG), which is the derivative of TG) results are shown in Fig. 8.

These data represent the resultant effect of heating and pyrolysis

Table 2
Attributions of the ATR-FTIR absorption bands observed in the spectra of the extracted fibers.

Wavenumber (cm^{-1})	Allocation	Element
3334	O-H	Cellulose, hemicellulose, lignin
2920, 2850	C-H	Hemicellulose, cellulose
1730	C═O	Hemicellulose
1595, 1315, 1240	C═C/C-H/C-O	Lignin
1029, 894	O-H/C-H	Cellulose

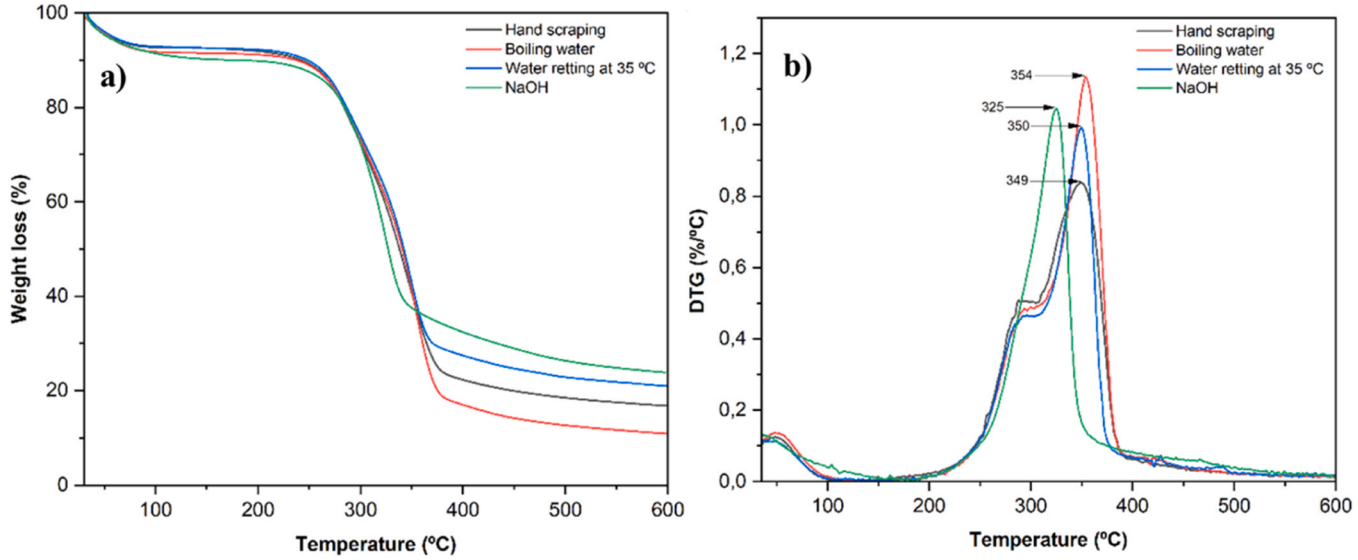

Fig. 8. a) TG curves and b) DTG curves of banana fiber after hand scraping only, NaOH, water retting at 35 °C and boiling water extraction.

degradation of the banana fibers (Soraisham et al., 2022). The weight loss of the fiber during the heating process is due to the breakdown of the cellulose, hemicellulose and lignin components. There are four stages of fiber degradation during the TGA process. The stages are moisture evaporation, hemicellulose degradation, cellulose degradation and lignin degradation (Lai et al., 2023).

The samples of banana fibers exhibited three main stages of mass loss in all extractions when subjected to temperatures from 30 °C to 600 °C. The first stage, up to 100°C, was observed in all samples and may be related to moisture loss due to the hydrophilic nature of the fibers (Diarsa and Gupte, 2021; Das et al., 2017a).

The second, close to 300 °C, can be attributed to the loss of hydroxyl and polar components, related to the degradation of hemicellulose, which is absent in the DTG curve for the chemically extracted fibers. This confirms the total or partial removal of hemicellulose, as verified by ATR-FTIR analysis.

The third and main peak near 350 °C, is associated with cellulose thermal degradation (Lai et al., 2023). Through the DTG analysis, degradation peaks of cellulose are observed with maximum at 325 °C, 349 °C, 350 °C, and 354 °C, corresponding to the chemically extracted, hand scraped, water retted at 35 °C for 28 days, and boiling water extracted fibers, respectively. Therefore, chemical extraction with NaOH has a lower thermal stability as cellulose degrades at lower temperatures.

In comparison to hemicellulose and cellulose, lignin is described as the most difficult component to decompose. Lignin decomposition can initiate at 160 °C and slowly decompose up to 900 °C (Das et al., 2017a).

The differences in mass loss can be attributed to differences in crystallinity, associated with the cellulose content, where higher crystallinity requires a higher degradation temperature (Diarsa and Gupte, 2021; Costa et al., 2013b). Cellulose plays a crucial role as the most significant factor in determining fiber quality, as it is the main structural component that provides strength and stability to plant cell walls. Therefore, the amount of cellulose present in a fiber is a determining factor for its suitability in various applications (Patel and Patel, 2022).

3.4. XRD analysis of extracted fibers samples

XRD analysis was performed to study the crystallinity of banana fibers obtained by the same methods as classified in the TGA analysis. The diffractograms are shown in Fig. 9.

The X-ray diffraction patterns revealed characteristic peaks of crystalline cellulose I at 2θ=15° and 2θ=22°, corresponding to reflections

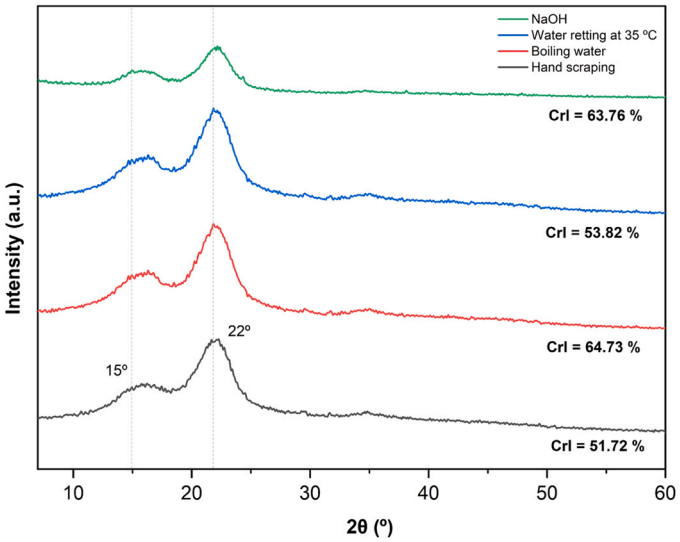

Fig. 9. The X-ray difraction (XRD) patterns of banana fiber samples after hand scraping, NaOH, water retting at 35 ºC and boiling water.

from the (110) and (002) planes, respectively (Das et al., 2017a; Costa et al., 2021).

The crystallinity index of each fiber sample obtained by the different extraction methods was calculated on the basis of Eq. 1 above, which gave values of 64.73 % for boiling water, 63.76 % for NaOH, 53.82 % for retting at 35 °C for 28 days and 51.72 % for hand scraping.

The diffraction pattern showed that the crystallinity index of the banana fibers obtained by different extraction methods was higher than the crystallinity index of the fibers obtained by manual scraping only. An increase in the crystallinity index may be related to an increase in cellulose and removal of non-cellulosic components. Therefore, the use of different extractions instead of direct manual scraping is more feasible to obtain fibers of higher quality, since a higher quality of the fibers is associated with a higher percentage of cellulose in their constitution.

This indicates that banana fibers obtained through hand scraping contain more amorphous or disordered regions. The increased fiber ordering can be attributed to the removal of hemicelluloses and lignin during the extraction (Diarsa and Gupte, 2021; Elanthikkal et al., 2010a). Once again, the results of ATR-FTIR and TGA analysis for the

biological methods are confirmed. The crystalline and amorphous regions found in cellulose fibers exist in different proportions when extracted by different methods (Das et al., 2017b). All samples provided similar diffraction patterns. The only difference lies in small changes in peak intensity, representing slight variations in the degree of fiber ordering among the samples (Elanthikkal et al., 2010b).

3.5. Evaluation of the mechanical properties of the extracted fibers

To evaluate the mechanical properties of the obtained fibers, tensile tests were conducted for the samples which presented higher quality according to the ATR-FTIR analysis (*i.e.* hand scraping, chemical extraction with NaOH and biological extraction, namely boiling water and water retting at 35 °C for 28 days).

As shown in Table 3, the mechanical properties such as linear density, elongation at break, tensile strength, and Young's modulus of the fibers varied from 0.90 to 24.18 Tex, 0.16–5.15 %, 22.94–479.14 MPa, and 2.91–23.80 GPa, respectively.

Fibers obtained by hand scraping only obtained higher values of linear density than those obtained by chemical or biological extraction, due to a greater quantity of non-cellulosic components, reaching a value of 24.18 Tex. The components were confirmed by ATR-FTIR analysis, which showed the presence of characteristic lignin and hemicellulose bands. Furthermore, a TGA analysis showed a significantly higher peak in the hemicellulose treatment temperature (around 300 °C) compared to other extractions, indicating that the presence of hemicellulose in these fibers could be higher. Fibers obtained by biological extraction, specifically by boiling water, had the lowest linear density value, around 7.29 Tex. This result is more suitable for the use of banana fibers in the textile industry, since a lower linear density can be associated with a lower content of non-cellulosic components and, consequently, a higher cellulose content (Patel and Patel, 2022).

Among the different extractions, the fibers obtained by chemical extraction showed the highest tensile strength, with a maximum value of 479.14 MPa, as well as the highest Young's modulus, 23.80 GPa, and the lowest elongation at break, 2.72 %. The increase in tensile strength and Young's modulus of NaOH extracted fibers, as well as the elongation at break of chemically and biologically extracted fibers, is related to the delignification of the fibers (Patel and Patel, 2022). Once the lignin is removed, the cellulose is more exposed. The different fibrils present in a fiber are made up of multiple cellulose chains composed of multiple glucose molecules. The hydroxyl groups present in each glucose molecule form strong hydrogen bonds with adjacent chains and also between the different fibrils, which are responsible for the mechanical properties of the fibers (Simbaña et al., 2020). However, the decrease in tensile strength and Young's modulus of fibers extracted by biological methods, compared to fibers hand scraping only, may be related to some manipulation of the cellulose during the extraction process. This manipulation may have been caused by the breaking of hydrogen bonds between cellulose chains, leading to the disruption of cellulose fibrils and weakening the fiber structure (Costa et al., 2021). In addition, the frictional forces during the scraping process after retting and boiling water may cause wear of the fiber structure. In the case of fibers obtained by water retting at 35 °C, the action of microorganisms present in the water from the retting process may have caused the destruction of the cell wall of the fibers, leading to a decrease in their tensile strength (Mumthas et al., 2019).

Table 3
Mechanical properties of extracted fiber samples.

Properties	Hand scraping	NaOH	Boiling water	Water retting at 35 °C (28 days)
Linear Density (Tex)	1.30 – 24.18	1.33 – 13.95	0.90 – 7.29	1.22 – 19.44
Elongation at Break (%)	0.46 – 5.14	0.16 – 2.72	0.86 – 3.18	0.35 – 2.98
Tensile Strenght (MPa)	57.13 – 252.00	39.22 – 479.14	46.92 – 134.30	22.94 – 102.50
Young's Modulus (GPa)	8.25 – 16.80	7.81 – 23.80	2.91 – 9.00	3.28 – 7.40

4. Conclusions

This work presents the various extractions performed to obtain banana fibers. The fibers were extracted by manual extraction, involving only scraping, by chemical extraction using NaOH as the alkaline reagent and by biological extraction through two different methods: retting at room temperature, retting at 35 ºC and boiling in distilled water.

Optical microscopy analysis revealed that the fibers obtained through biological extraction resulted in a greater reduction in the diameter of banana fibers, in the order of boiling, retting, and retting at 35°C, followed by chemical extraction, and finally, manual extraction. FESEM images confirmed the diameter reduction in fibers obtained through biological extraction, as compared to manual extraction, due to the removal of non-cellulosic materials. Cross-sectional images of the fibers showed reduced spaces between the fibers, justifying the diameter reduction. ATR-FTIR spectroscopy revealed that chemical extraction was the most effective in removing non-cellulosic components from banana fibers, but it also showed higher degradation of cellulose. TGA analysis indicated that fibers obtained through chemical extraction required a lower temperature for cellulose degradation, which may be associated with lower crystallinity. Thus boiling and retting at 35°C, followed by manual extraction, may provide fibers with a higher cellulose content and consequently superior quality. XRD analysis indicated a higher crystallinity index in boiling extraction, followed by manual extraction, and then biological extraction. A higher crystallinity index means an increase in crystalline regions and a decrease in amorphous regions, i.e., an increase in cellulose content.

In the evaluation of mechanical properties, fibers obtained through chemical extraction showed the highest values for tensile strength and Young's modulus. They also presented very low density values, as intended. The evaluation of mechanical properties refuted the results obtained in the characterization of the samples. Fibers obtained through chemical extraction had shown greater cellulose degradation and less diameter reduction, suggesting that they could potentially exhibit worse mechanical performance.

After extraction, banana fibers can be washed, dried, and processed for use in various applications, such as the textile industry, paper manufacturing, composite materials production, among others. Banana fiber is highly valued as it is a sustainable, low-cost, and biodegradable material, making it a promising alternative to replace synthetic materials in various industrial applications. In the future, it would be interesting to study other parts of the Portuguese banana plant for fiber extraction, for comparison studies, as well as to test the spinnability of the extracted fibers, to produce 100 % banana fiber yarns.

Authors' contributions

Joana C. Araújo was responsible for the experimental part and wrote the paper. Diana P. Ferreira supervised the work and also wrote the paper. Pilar Teixeira performed the antibacterial activity tests and participated in the writing of the paper. Raul Fangueiro also supervised the work.

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CRediT authorship contribution statement

Diana P. Ferreira: Writing – review & editing, Supervision, Project administration. **Carina V. Gomes:** Writing – original draft, Methodology, Investigation. **Joana C. Araújo:** Writing – review & editing, Validation, Investigation, Conceptualization. **Diego M. Chaves:** Writing – review & editing, Methodology. **Raul Fangueiro:** Supervision, Funding acquisition.

Declaration of Competing Interest

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