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Article

Sustainable extraction of cellulosic microfibers from agro-industrial residues of banana

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Abstract

Natural cellulose fibers have properties and structures that make them suitable for a variety of uses, such as the manufacture of textiles, composites, pulp and paper. In addition to this, there is a growing interest in the use of alternative materials with low environmental impact due to the problems related to the environment; therefore, it is necessary to implement new sources and processes to obtain cellulose. The research began in 2024. The purpose was to obtain crystalline cellulose microfibers from agro-industrial residues from banana cultivation through processes with a lower environmental impact. The cellulose fibers were obtained from the fibers of the pseudo-stem of bananas, variety *Musa cavendish*, using steam explosion treatments carried out in a thermal reactor and bleaching processes with 2% NaClO, obtaining soft and fragile cellulose microfibers. The TGA analysis showed that the cellulose fibers obtained from banana fibers presented greater thermal stability due to the removal of hemicellulose, which could be corroborated by FT-IR. Steam explosion treatment is a viable and environmentally friendly alternative for obtaining cellulose microfibers from banana fibers and other agro-industrial waste.

Keywords:

Musa cavendish, reactor,steam explosion, treatment.

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Introduction

Natural fibers have favorable properties such as low cost, easy availability, low density, and recyclability, etc. The disadvantages of natural reinforcement, as is the case with fibers, are increased moisture absorption and poor compatibility of the reinforcement and matrix. Therefore, there are a variety of chemical or physical treatments that are used to modify the surface of the fiber and improve the adhesion between the matrix and the fiber (Godara, 2019).

Natural fibers have some advantages over artificial fibers, including low cost, high availability, low density, the ability not to damage processing equipment, and being recyclable and biodegradable (Rajwade *et al*., 2020; Reddy *et al*., 2020). Natural cellulose fibers have physical and chemical properties that make them suitable for various uses, such as the manufacture of textiles, polymer composites, pulp, and paper. Cellulosic fibers for textile and paper pulp production remain important non-food trade products (Sheng *et al*., 2014).

The niche market for natural cellulose has remained steady, and numerous new markets are emerging due to the 'green' ecological image and low carbon footprint of cellulosic fibers. Depending on the type of lignocellulosic biomass, natural fibers usually contain between 25 and 45% of cellulose by weight, between 20 and 40% of hemicellulose by weight, and between 10 and 25% of lignin by weight (Sarker *et al*., 2021).

Cellulose is a homopolysaccharide consisting of linear chains of D-glucose subunits linked by β-(1-4) glycosidic bonds. Hemicellulose is a heterogeneous branched polysaccharide consisting of galactose, arabinose, mannose, glucose and xylose. For its part, lignin is an aromatic polymer of phenylpropane, the precursors of which are mainly made from coniferyl alcohol, p-coumaryl alcohol, and sinapyl alcohol. It has a structural function in the plant cell, joining cellulose and hemicellulose (Gupta *et al*., 2020).

It also contains extracts of the non-structural components of biomass, such as essential oils, fats, waxes, phenolics, and fatty acids, among others. Hemicelluloses, lignin, and other non-cellulosic components must be separated, dissolved, and partially broken down during the maceration process of cellulose fibers for textile use (Xu *et al*., 2017). Nevertheless, the complexity and variability of the lignocellulosic structure hinder biodegradation, particularly the hydrolysis of organic matter, it is complex to convert it into soluble compounds, which is the limiting step of the degradation rate (Capári *et al*., 2016).

This structural strength can be broken by physical, chemical, and biological pretreatment methods. Hydrothermal pretreatment is a method in aqueous conditions that combines physical and chemical processes (Barciela *et al*., 2023). One of them is steam explosion, the mechanism of which is based on the depolymerization of lignin and the explosion of cellulosic fibrils by treating biomass with pressurized steam at high temperature, which usually ranges between 160 and 280 °C.

To extract the cellulose fibers and maintain their integrity, the hydrothermal temperature must be below 240 °C. The water contained in the substrate evaporates and swells rapidly, resulting in some degree of hydrolysis (Pérez-Limiñana *et al*., 2022). Therefore, this research work presented a study on hydrothermal pretreatment, specifically by steam explosion, to improve the properties of natural fibers in polymeric composites.

The research focuses on how the control of temperature and time conditions influences the depolymerization of lignin and the preservation of cellulose, thus optimizing fiber-matrix compatibility and the biodegradability of the material. This contribution offers a more efficient and sustainable strategy for the use of natural fibers in ecological composite material applications.

Materials and methods

For this study, fibers were collected from the pseudo stem of bananas, variety *Musa cavendish*, from the state of Tabasco. The bleaching process was performed using analytical-grade sodium hypochlorite (NaClO) from Sigma-Aldrich. Steam explosion (SE) treatment was performed in a Parr 4842 reactor with a capacity of 2 L.

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Steam explosion (SE) treatment

Ten grams of previously moistened banana fiber were placed in the sample holder of the Parr reactor, which prevents the fibers from being in direct contact with the walls of the reactor and the water, preventing their carbonization. Three hundred milliliters of distilled water were placed in the reactor and heated to 185 °C for 10 min at a pressure of 170 psi.

The sample was exploded by a pressure drop when it was released through the opening of a relief valve. The exploded fibers were dried at 70 °C for 24 h in an oven. The design of the reactor with the sample holder adapted for the fiber treatment process is shown in Figure 1; this design was based on a work previously reported by Shamsudin *et al*. (2024).

Figure 1. Reactor with a sample holder for the treatment of fibers by steam explosion.

Cellulose microfiber extraction process

To extract the microfibers, a bleaching process was carried out in a beaker and a heating plate; for that purpose, a 2% NaClO solution was previously prepared and heated at 50 °C with constant stirring. Once the temperature was reached, 5 g of exploded fiber was placed in 250 ml of solution for 1 h. At the end of the reaction, the recovered microfibers were filtered by gravity and washed with plenty of water until the excess NaClO was removed and then were dried at room temperature for 24 h.

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Characterization

The chemical analysis was carried out in a Nicolet Nexus 470 ESP FT-IR spectrophotometer in a range of 600 to 4 000 cm^{-1}, and its purpose was to determine the functional chemical groups present in both the untreated and treated fibers and to perform a comparative analysis on the possible effects of the treatment on the fibers.

The thermogravimetric analysis (TGA) was performed in a Linseis thermal analyzer in order to evaluate the thermal stability of the fibers; the samples (13 mg ±3 mg) were heated in a nitrogen atmosphere from temperatures of 30 to 700 °C at a heating rate of 10 °C min. For the superficial analysis of the fibers, a PrimoStar optical microscope was used with magnifications of 100 and 200 X.

Results and discussion

After steam explosion and NaClO treatment, banana fibers only showed a color change, because hypochlorite is a bleaching agent that can remove hemicellulose and lignin from the fiber (Aridi *et al*., 2021). On the other hand, the fibers treated only with SE presented a dark brown coloration because the temperatures used tend to carbonize the fibers partially; on the other hand, the fibers treated with NaClO and SE showed a white hue and a soft and fragile appearance, resulting from the effect of hypochlorite.

This situation was because the SE process evaporates the water contained in the fibers and expands rapidly, which causes the rupture of the cell wall, forming pores, and promotes delignification and hydrolyzation of hemicelluloses (Ma *et al*., 2022); in contrast, the NaClO treatment applied to the exploded fibers tends to separate them into microfibers during the removal of lignin and hemicellulose, resulting in a soft and thin consistency, as can be seen in Figure 2d.

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Figure 2. a) untreated banana fiber; b) NaClO-treated fiber; c) SE-treated fiber and d) fiber treated with SE and NaClO.

Infrared (FT-IR spectroscopy analysis

Figure 3 shows the FT-IR spectra of untreated banana fiber, NaClO-treated fiber, SE-treated fiber and SE and NaClO-treated fiber. Both untreated and treated fibers presented the typical bands corresponding to cellulose, hemicellulose and lignin. In all cases, the stretching absorption band of O-H can be observed around 3 331 cm^{-1}; these groups can come from the water absorbed as a result of the hygroscopic nature of the fibers, and from the primary and secondary aliphatic alcohols present in cellulose, hemicellulose and carboxylic acids (Ibrahim *et al*., 2010).

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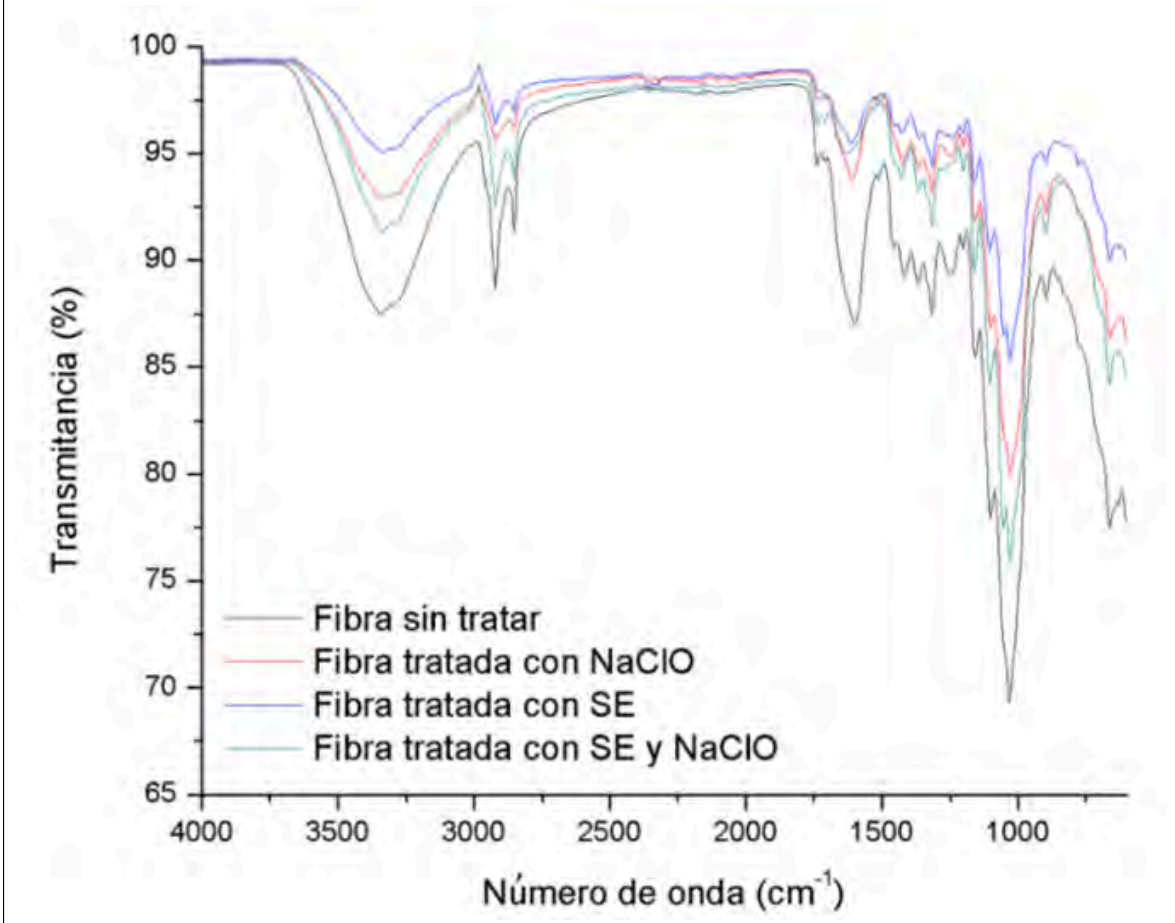
Figure 3. Infrared spectrum of treated and untreated fibers.



The absorption peaks present at 2 918 cm^{-1} and 2 850 cm^{-1} are attributed to the asymmetric stretching vibration of C-H in methyl groups and the symmetrical stretching vibration of C-H in methylene groups belonging to residual cellulose and hemicelluloses, respectively. Peaks located around 1 390 and 1 410 cm^{-1} in the symmetrical deformation spectrum belong to the C-H stretches of cellulose (Parre *et al*., 2020).

Peaks between 1 000 and 1 200 cm^{-1} indicated an asymmetrical C-O-C stretching caused by cellulose and hemicellulose, as well as a C-O and C-C stretching vibration. The absorbance intensity of the 1 608 cm^{-1} peak, characteristic of hemicelluloses and attributed to C=O stretching, decreased in all cases compared to untreated fiber. This is due to the decrease in hemicellulose and lignin content as a result of the treatments (Ronald Aseer *et al*., 2013).

Thermogravimetric analysis (TGA)

In order to compare the characteristics of treated and untreated fibers, banana fiber samples were analyzed by thermogravimetric analysis. The thermogravimetric (TG) and derived thermogravimetric (DTG) curves of treated and untreated fibers are shown in Figure 4a and 4b. A slight loss in weight could be observed between 50 and 100 °C, indicative of moisture loss due to the water absorbed by the fiber.

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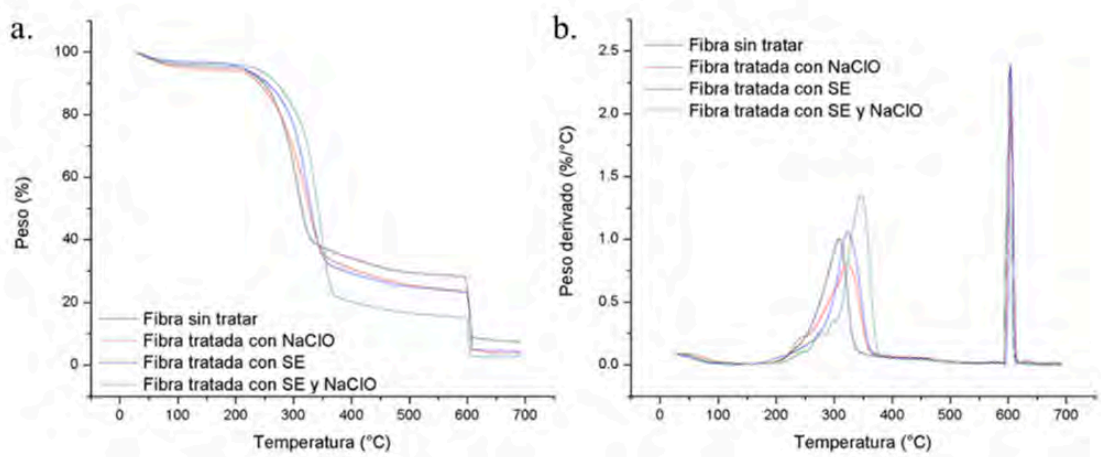
Figure 4. a) thermogram (TG) of treated and untreated fibers and b) derived thermogram (DTG) of treated and untreated fibers.



The primary decomposition signal of untreated fibers occurs between 200 and 350 °C, which is attributed to the breakdown of cellulose and lignin components; the DTG curve of untreated banana fiber (Figure 4b) shows a peak at 309.91 °C (mass loss of 63.44%), which is due to the thermal decomposition of α-cellulose (Deepa *et al*., 2011).

After 400 °C, weight loss was slow until 800 °C, with small signs probably due to residual lignin (Kataria *et al*., 2017). The DTG of the fibers treated with NaClO presented two peaks; the initial peak at approximately 85 °C corresponds to a loss of adsorbed water mass; however, the main breakdown peak in NaClO-treated fibers increased to approximately 325.33 °C (mass loss of 66.73%), mainly due to the removal of hemicellulose and lignin during treatment.

Sodium hypochlorite at 5% has been reported to be the best bleaching agent for removing hemicellulose and lignin from the fiber (Aridi *et al*., 2020). From the DTG curve of the SE-treated fibers, a significant decomposition peak is observed at 322.51 °C (mass loss of 68.91%), mainly due to the decomposition of α-cellulose.

On the other hand, the DTG curve of the fiber treated with SE and NaClO presented a majority peak at 346.78 °C (mass loss of 78.36%) as a result of the decomposition of the α-cellulose; it is evident that there is a change in the main decomposition temperature from 309.91 to 346.78 °C while the transition from the untreated fiber to the fiber treated with SE and NaClO occurs.

The treated fibers show in all cases an increase in the mass loss temperature. The NaClO bleaching process helps to remove residual lignin after the process by steam explosion. The bleaching mechanism involves the oxidation of lignin, which leads to the depolymerization of lignin, leaving #-cellulose as a residual material, which has been reported to have crystalline properties. Increases in degradation temperature occur due to the higher cellulose content and high crystallinity of the treated fibers, which allows the fibers to withstand higher decomposition temperatures (Meng *et al*., 2019).

Optical microscopy (POM analysis

Figure 5 showed that fibers treated with SE and NaClO range in size from 9 µm to 39 µm thick, this being the main reason why the fibers have a soft and pliable consistency compared to untreated fibers; however, even with the treatment carried out, fibers with sizes greater than 100 µm could be found in a minority proportion, indicating that the defibrillation process is not entirely homogeneous; it is known that the operating conditions in high-temperature steam treatments and the concentrations of the bleaching agent are highly determinant for the release of microfibers.

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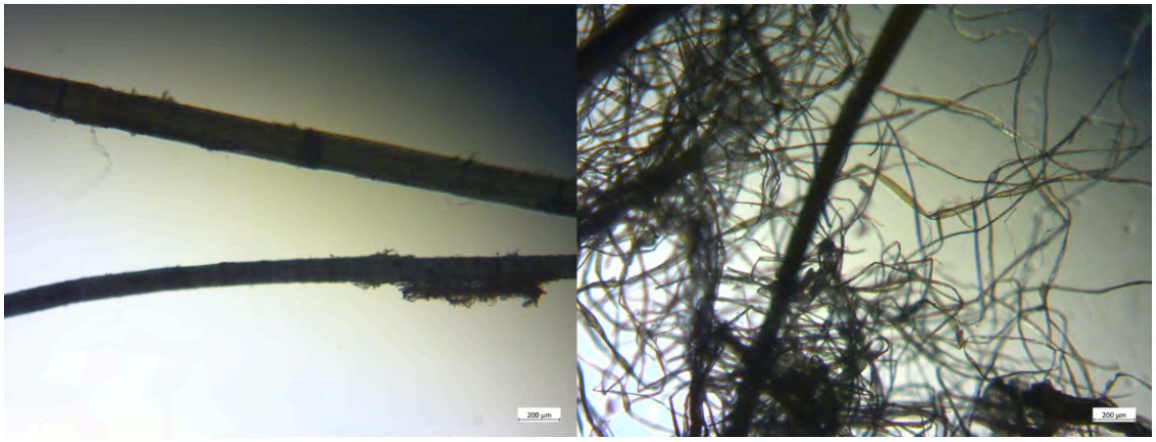
Figure 5. Micrographs obtained from untreated fibers (left) and fibers treated with SE and NaClO (right).



Conclusions

Steam explosion treatments combined with hypochlorite have proven to be highly effective in drastically reducing the long packed fiber chains to microfibrils by acting mainly on the interfibrillar region during the high-pressure steam explosion. This structural breakdown facilitates greater accessibility and dispersion of microfibrils, significantly improving their properties.

The results obtained indicate that the microfibers produced show a notable improvement in their chemical and thermal characteristics compared to the untreated fibers. These improved properties allow for a larger contact surface area with various polymeric matrices, making them highly suitable for use as reinforcement materials in composite manufacturing, thus offering a promising path towards the production of more sustainable and high-performance materials.

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