

# CORN LEAF FIBERS PREPARATION AND CHARACTERIZATION FOR COMPOSITE OBTENTION

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## Summary

There is a global trend in seeking plant fibers to replace the synthetic fibers to obtain reinforced composites, aiming the use of renewable resources. In this context, this paper objective is to develop the process of corn leaf fibers preparations, characterize and adapt them for applications in the construction industry. Corn leaves were dried in room temperature, treated by mercerizing (chemical process of attack on the fiber surface making it rough); then neutralized with acid solution and washed in running water. The characterization of the corn leaf fibers was performed by X-ray diffraction, X-ray fluorescence Spectrometry, Scanning Electron Microscopy, Thermogravimetry, specific mass and Fourier Transform Infrared Spectrometry. The mercerizing treatment was effective, because the corn fibers have similar characteristics to synthetic fibers, leading to a possibility of new technological uses, besides the application in gas pipe manufacturing.

## Introduction

New agricultural waste uses are important to the environment, reducing the dependency on raw materials from petroleum.

The characterization of these materials opens prospects of noble uses of a product considered agricultural waste, enhancing the production in small communities and promoting sustainable development.

In the last two decades, reducing the generation of waste and environmental preservation has been the subject of challenges to society, focusing on recycling. There is a global trend in seeking substitute natural resources, as exchange for synthetic fibers, that are environmentally friendly, developing new products, generating jobs and income, developing new technologies for the construction industry and for other areas where it is possible. [1]

Although polymers and its derivatives have, historically, contributed immensely to the global technological development, enhancing the modern man life quality, the continued use of these materials has brought concerns to society. This is because, most of it, has low recyclability and large cumulative power in the biosphere, particularly due to its low biodegradability and non-renewable sources origin, such as petroleum. [2]

In this context, the search for alternative materials with high technical, social, environmental and economic performance is increasingly necessary and the research with plant fibers becomes more frequent. The replacement of synthetic fibers, a non-renewable resource, for vegetable fibers, that are provenient of renewable resources, is an interesting point. [3]

The study underlies in the use of available vegetable residues, with the possibility of generating considerable waste volumes.

The relevance of the research is the opportunity to generate another source of income and jobs, with the exploration of a subproduct, for people who survive of the corn harvest in Brazil, and the combined economic benefit of several industries recycling the waste, corn

straw. In this logic, this study aims to develop the preparation of corn fiber and characterize it. The fibers are fundamental for preparing a polymeric composite for the manufacture of gas pipes.

### Materials and methods

The leaves used in this study were those that coat the corn cob,, purchased in street markets of São Paulo, they are previously dried by sun exposure. It was found that the concentration of 3% to 5% (weight percentage) of sodium hydroxide and stirring time from 4 to 7 days in immersion, depending on the leaf thickness,are suitable conditions to a higher extraction efficiency.

During the immersion step, the leaves are submitted to stirring, causing the removal of corn leaf pulp ,obtaining the fibers (Figure 1).



Figure 1 - Corn leaves immersed in a sodium hydroxide solution while stirring.

After the alkaline attack (mercerizing), the fibers are washed in running water and rubbed on a sieve for cleaning. Then, the fibers are subjected to the acetic acid solution 4% (volume percentage) for neutralization of the mercerization process and are stirred for 1 hour, and then the fibers are washed again with running water and taken to drying in an stove at approximately 50 °C. Figure 2 shows the fibers obtained after sodium hydroxide attack.



Figure 2 – Fibers obtained after alkaline attack.

Due to the high solubility of cellulose, even at low alkali concentrations, treatment with sodium hydroxide promotes a greater surface roughness in the fiber, improving the mechanical adhesion between fiber and matrix. Thus, generally, the alkali treatment causes the fibers swelling and partial removal of hemicellulose and lignin, which promotes a better packing of the cellulose chains, which are responsible for the crystallinity of the fiber. Consequently, the treatment causes an increase in crystallinity and a reduction of the fibers diameter and density [3].

Figure 3 shows clean dry fibers obtained by two routes. It is observed that in the chemical route, the fibers are free of pulp.

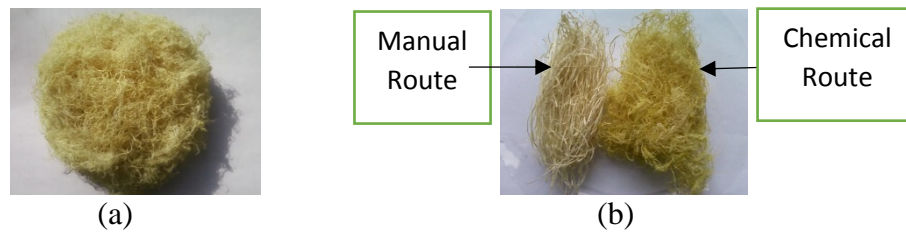


Figure 3 - Clean and dry fibers (a) and difference of fibers obtained by two routes (b).

The density of the fibers was determined by helium pycnometry using a powder sample. It was used 4,5 helium (99.995% purity), in a Micromeritics equipment, model AccuPyc 1330.

The specific surface area was determined by BET analysis; The equipment used was a Quanta-Chrome Corporation, New 1200BET Surface Area Analyser, version 3.11. First the sample was degassed for 12 hours in a sand bath at 150 ° C for removal of the volatile and interfering gases, then there was adsorption of nitrogen to ensure an inert atmosphere, and then the analysis was performed by the adsorption and desorption of nitrogen in the sample at vacuum conditions of 0,1mmHg.

To check the morphology, the fibers were placed in metallic sample holder, and sprayed with a thin layer of gold to make them conductive and generate an image with better resolution. The equipment used was a scanning electron microscope, from Jeol JSM, model - 6010LA - Series No. MP11000026.

Thermogravimetry analysis of the samples determined fiber mass degradation, in a Shimadzu equipment TGA - 51, AT control system - 60WS.

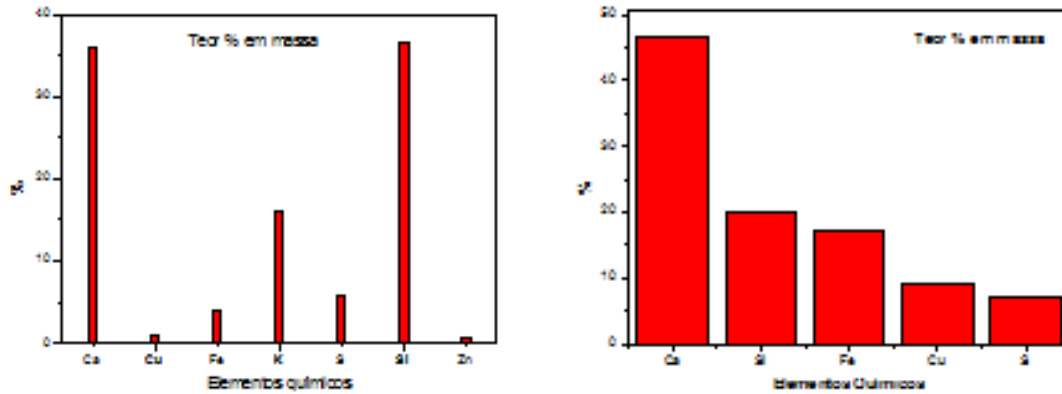
The crystallinity of the fibers was identified by X-ray diffraction. The compacted fiber powder sample was placed in a stainless steel sample holder and analyzed in an X-ray diffractometer of Rigaku, model Multiflex monochromator using Cu-K  $\alpha$  radiation, at 40kV and 20mA. The scan rate was 0.5 ° / min and varied from 10° to 90° $\theta$ . The identification of crystalline phases is/was done with the aid of crystallographic data available in the International Centre for Diffraction Data System / Joint Committee on Powder Diffraction Standards (ICDD / JCPDS).

The Fourier transform Infrared Spectroscopy (FTIR) was used to identify the main characteristics of the fibers. The spectra in KBr were made in a Nexus 670 FTIR spectrophotometer of Thermo Nicolet.

## Results and discussion

The results of X-ray fluorescence assay before the chemical treatment (Figure 4a), show a high percentage of potassium (17%) and calcium (37%), the amount of silicon is about 45% (weight percentage) and elements such as copper, iron, sulfur and zinc are in minor amounts.

The chemical composition of corn leaf fibers after chemical treatment indicates presence high concentration of calcium (47.8%), followed by Silicon (19.2%), Iron (17.2%), Copper (9, 2%) and sulfur (6.9%).



(a) (b)  
 Figure 4 - Chemical composition of the corn leaf fibers before (a) and after (b) treatment with mercerizing.

Other fiber characteristics are essential for the preparation of composites such as density (table 1) and specific surface area (Table 2).

Table 1 - Density of corn leaf fibers

<b>Corn leaf fibers before surface treatment with chemical attack</b>
(1.3607 ± 0.0037) g / cm <sup>3</sup>
<b>Corn leaf fibers after surface treatment with chemical attack</b>
(1.5219 ± 0.0476) g / cm <sup>3</sup>

Table 2 - Specific surface area of corn leaf fibers.

<b>Corn leaf fibers before surface treatment with chemical attack</b>
0.9823 m <sup>2</sup> / g
<b>Corn leaf fibers after surface treatment with chemical attack</b>
16.2691 m <sup>2</sup> / g

The density is within the plant fibers parameters, similar to those used in composites. The chemical treatment by sodium hydroxide causes a greater roughness of the fiber, increasing the specific surface area and improving the mechanical adhesion between the fiber and the matrix.

Scanning Electron Microscopy allowed the morphological characterization of the corn leaf fibers. The micrographs in Figures 5 and 6 show the details of fibers from corn leaves before and after chemical treatment.

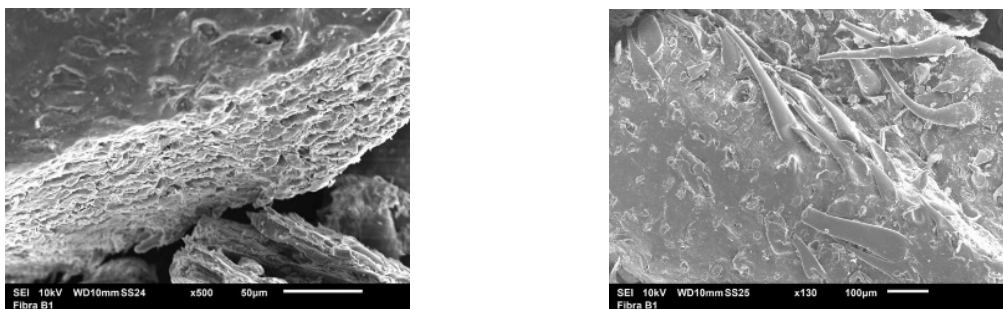


Figure 5 - corn leaf fiber before mercerization.

In the Micrographs of the fibers after chemical treatment (Figure 6), it is possible to observe that the cover or pulp adhered to the fiber was removed, exposing the fibers. This waste eliminated, provides better adhesion between the fiber and the matrix.

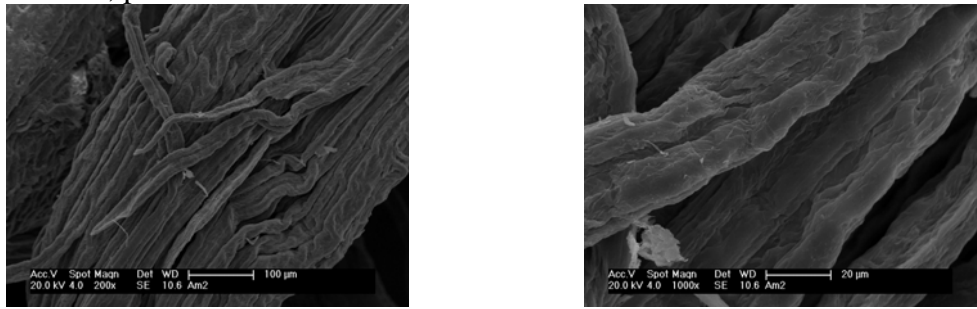


Figure 6 - corn leaf fiber after mercerization.

Figure 7 shows the thermogravimetric analysis of the corn leaf fibers, before and after chemical attack surface treatment .

The results show that before and after the chemical treatment, the starting temperature of oxidation of the corn leaf fibers is approximately 300 ° C.

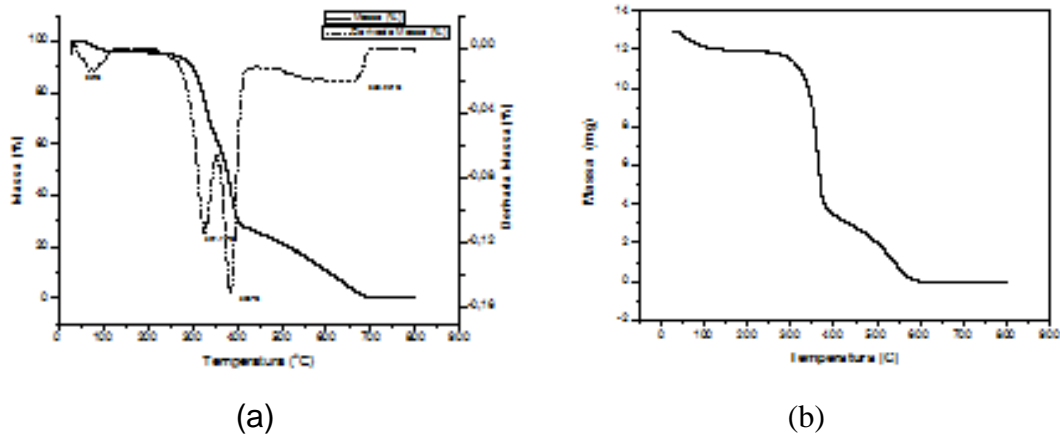


Figure 7 - Thermogravimetric analysis of the corn leaf fiber before (a) and after (b) treatment with surface etching.

In both figures 8 (a and b) the crystallinity of the fibers is evident, verifying an increase after the chemical treatment. there is an increase in crystallinity of the fibers after the modification with alkaline solution, therefore there is an increased adhesion between fiber / matrix in the composite formation [4].

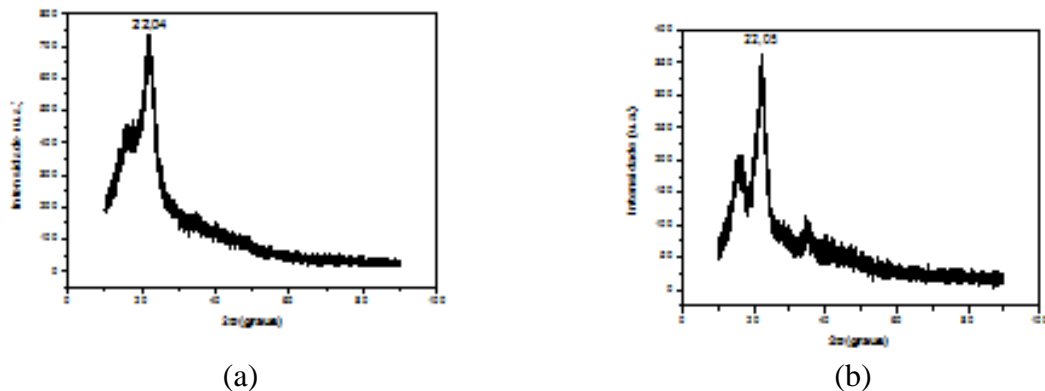


Figure 8 - X-ray diffractograms of the corn leaf fibers before (a) and after (b) treatment with surface etching.

The fibers of the corn leaves were characterized by Fourier Transform Infrared Spectroscopy analysis to identify natural cellulose fiber (-OH), hemicellulose (C + O) and lignin spectra (methoxyl-CH<sub>3</sub>) (Figure 9).

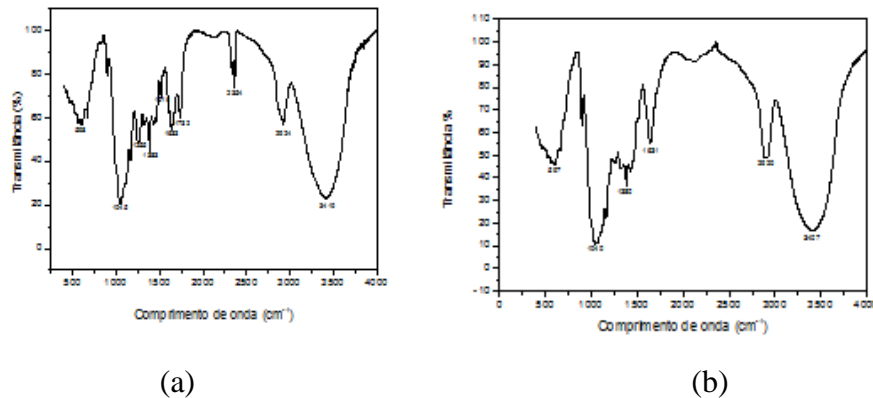


Figure 9 - infrared spectroscopy of the corn leaf fibers before (a) and after (b) treatment with surface etching.

Corn leaf fibers before and after the chemical process have similar characteristics, showing typical absorption bands of lignocellulosic material. Spectra of Curauá fibers, presented by Araújo, have strong similarities in absorption bands of the corn spectra. [3]

## Conclusion

Fibers from corn leaves were extracted and characterized, various extraction techniques were evaluated, obtaining preliminary results, that compared to other scientific studies in this area concluded that the corn leaf fibers have characteristics similar to other vegetable fibers already used for the reinforcement of polymeric composite materials.

The results obtained are promising, leading to a possibility of new technological uses for the corn leaf fibers.

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