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# Morphological, Chemical, and Thermal Characterization of Tobacco Stalk for Application in Composite Materials

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**ABSTRACT:** The main objective of this study is to analyze the use of tobacco stalk to develop new composite materials. For this purpose, tests and analyses were carried out to characterize the morphological, thermal, and chemical properties, as well as to obtain structural information about this plant waste. Morphology was analyzed by scanning electron microscopy (SEM). Thermogravimetry (TGA) was used to analyze mass loss and temperature degradation. Fourier-transform infrared spectroscopy (FTIR) was applied to determine the chemical composition. SEM showed the presence of filling components such as parenchyma and vascular elements of the xylem. FTIR showed the presence of elements such as absorbed water and primary and secondary aliphatic alcohols identified in cellulose, hemicelluloses, lignin, and carboxylic acids, among other substances. TGA showed cellulose degradation temperature between 250 and 350 °C. The test results indicate that tobacco stalks can be used to develop composite materials as fillers.

**KEYWORDS:** tobacco; tobacco stalk; composite materials; plant waste.

## 1. INTRODUCTION

Natural materials have been used to develop raw materials for over 3000 years. The Egyptians used plant-based materials such as clay and natural bamboo straw to build walls [1]. Currently, plant-based materials are applied to develop new raw materials through different manufacturing methods for a variety of products. Low density and cost are the main characteristics of these materials, which can exhibit high electrical resistance and acoustic insulation, in addition to being renewable and easily accessible [2–5].

The tobacco plant (*Nicotiana tabacum* L.) is an economically important agricultural product for several countries, including Brazil. According to the National Interstate Tobacco Industry Union [6], Brazil is the second-largest tobacco producer in the world and has been a leader in exports since 1993. Most of the production is concentrated in the South Region, which is responsible for almost all of the national production, with approximately 560 thousand tons in the 2021/22 crop [7].

The tobacco plant ranges from 0.8 to 2.5 m in height. Its production is mainly aimed at supplying high-quality leaves for the manufacture of cigarettes. However, harvesting tobacco leaves generates a residue, the stalks, which are normally burned for energy production or left in the field to be incorporated into the soil [8].

The end of the tobacco stalk life cycle is generally inappropriate [9]. The waste left in the field or burned can become a valuable resource. Tobacco stalk residue is a fibrous biomass consisting basically of cellulose, hemicelluloses, and lignin that has the potential to be used as a source of renewable energy in the manufacture of paper and xylose [8,10] and in the production of panels for furniture construction [8].

Materials are characterized by a set of properties [11]. Thus, this study aims to characterize the tobacco stalk using the following tests: morphology was analyzed by scanning electron microscopy (SEM), thermogravimetry (TGA) was applied to analyze mass loss and temperature degradation, and Fourier-transform infrared spectroscopy (FTIR) was employed to determine the chemical composition.

The tobacco plant was chosen because of the relevance of its cultivation in the state of Rio Grande do Sul, marked by significant production, mainly in the Rio Pardo Valley [12]. Considering the economic alternative to the work of tobacco growers, the tobacco stalk was characterized aiming to deepen the knowledge about its characteristics and its future use in the development of new materials from a composite that uses this plant waste as lignocellulosic reinforcement.

## 2. TOBACCO (*Nicotiana tabacum* L.)

*Nicotiana tabacum* L. is a solanaceous plant covered with thin hair, with an erect, robust, cylindrical stalk. The leaves are fixed, ovate or lanceolate, whole, sticky, with protrusions on the underside. Tobacco planting is divided into two phases: seedling production and cultivation in the field, with subsequent harvesting. All phases influence the plant composition, as well as the pests and diseases that may affect its performance and growth [13].

Harvest is determined by type – Burley, Virginia, and Oriental [6,7,13]. However, all types of tobacco present stalk as residue. According to Qin et. al.[9], the stalk corresponds to 20% of the plant and is its main residue. Also, it is estimated that the annual generation of tobacco stalks is approximately 3.2 million tons, which represents 40% of the world's total [14].

Regarding the representativeness of the stalk as waste, after the leaves are harvested, the stalks can be incorporated into the soil for subsequent planting, serving as an input, or can be burned for energy production. However, they point to the problem that the volume of this waste is significant, and the presence of nicotine in the stalks is a problem in solid waste disposal [8].

Shakhes et. al. [15] analyzed the dimensions of tobacco stalk fibers and their composition and concluded that they can be useful as substitutes for traditional raw materials in the pulp and paper industry because their fibers are short and present morphological properties similar to those of wood fibers. Indeed, tobacco stalks are a valuable and useful biological resource [9], but the methods commonly used to treat this waste are inadequate and cause environmental problems.

## 3. EXPERIMENTAL

This section of the study presents the materials and procedures used to prepare the tobacco stalk for the proposed characterization tests and detailed information about these tests.

### 3.1 Materials

To perform the aforementioned analyses, it was necessary to collect samples that could be representative and usable for characterization. Thus, stalk samples were obtained from the Tobacco Growers' Association of Brazil [7] in the municipality of Santa Cruz do Sul, state of Rio Grande do Sul. Waste from Virginia tobacco, also known as greenhouse tobacco, was used. After harvesting, the leaves of this variety are strung on sticks and oven-dried under controlled temperature. The curing process for this type of tobacco lasts 4-5 days and its color can vary between shades of yellow, orange, and brown [13].

### 3.2 Micronization of tobacco stalks

The tobacco stem samples received were initially characterized and analyzed by sectioning the material, which is a way of studying the substructure of most plant organs. Sectioning was performed manually using a blade [16]. The samples were sectioned into ~1 cm pieces (Figure 1) and then fixed in Formalin-Aceto-Alcohol (FAA) 50% solution[1] to preserve the botanical material. The material was stored in a glass container and a PVC sheet was placed between the container and the lid to prevent oxidation.

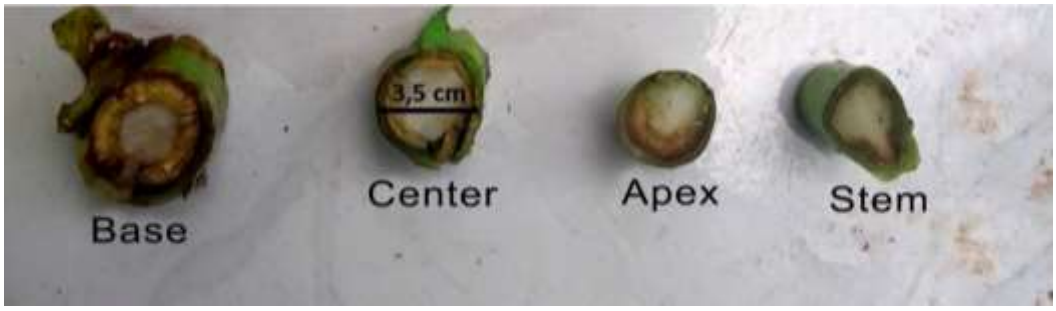


**Figure 1 - Sectioning of Virginia tobacco stalks.**

The material was prepared and the samples were characterized at the Plant Anatomy Laboratory of the Federal University of Rio Grande do Sul (UFRGS). The stalks were cross-sectioned using a blade. Plant tissue is a particularly suitable material for manual sectioning because it is structurally held together by its cell walls. Freehand sectioning, in addition to the speed of execution, has other advantages: it allows the observation of cells and tissues unaffected by fixation or, even when fixed, not influenced by shrinkage and extraction of components that inevitably follow dehydration and fixation in paraffin or plastic resin. It also enables a better mental reconstruction of the 3D structure [16].

O'Brien and McCully [16] report that, in many cases, sections are left unstained, as the natural color of the specimen is sufficient for observation. However, for some purposes, staining is required. For temporary slides, sections can be stained with toluidine blue O and/or periodic acid-Schiff

[1] Solution composed of formaldehyde, acetic acid, and 50% ethyl alcohol [22].

(PAS) and viewed in an aqueous medium. They recommend toluidine blue 0 for most structural studies. The use of stains can show cell structures, facilitating observation. Thus, the samples were prepared with sections of fresh botanical material stained with toluidine blue 0.

Sectioning was performed by holding the botanical material with the thumb and the first two fingers of the left hand. The material must be positioned so that the region of the first desired sections is close to the level of the thumb and the index and middle fingers. It is important not to hold the tissue or the blade too tightly and to thoroughly wet the blade, the tissue, and the thumb/fingers with water. In addition, it is crucial that the first section be performed quickly and smoothly to produce a new surface in the desired plane and that no more than 25% of the edge be used. The material from the first section is discarded and the remainder of the edge is then used for the following sections. Subsequent sections are performed slowly and intentionally with the blade located between the tip of the thumb and the top or side of the index finger [16].

O'Brien and McCuly [16] also recommend that the sections do not exceed 20 $mm^2$ in area and do not remain completely dry. They suggest removing the sections from the slide using a small spatula, floating them in a bowl of water, soaking them for at least 2 min, and removing possible debris using a camel hair brush. To mount the samples on the glass slides for subsequent microscopic analysis, it was necessary to place a drop of clean water (~5 mm in diameter) in the center of the slide and use a brush or spatula to transfer the section to the drop. The prepared samples were analyzed on a Leica DMR HC microscope coupled to an AxioCam ERC5s digital camera. The results of the characterization test are presented in Section 4.1.

Some of the tobacco stalk samples were prepared for SEM characterization, whereas some of them were ground using an agate pestle and mortar and then processed in a zirconia ball, planetary, centrifuge mill (Pulverizette, Fritsch) in 2 cycles of 50 min with an initial 20 min cycle (120 min) for FTIR analyses. These procedures are shown in Figure 2.

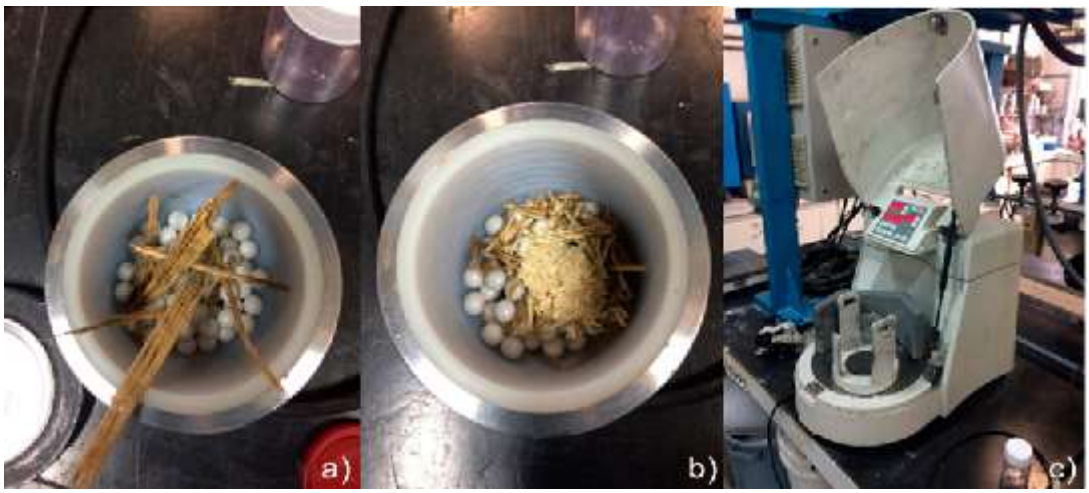


**Figure 2 - Sample grounding and milling: (a) tobacco stalks and zirconia balls in the vat; (b) part of the ground stalks; (c) milling equipment: zirconia ball, planetary, centrifuge mill (Pulverizette, Fritsch).**

Finally, TGA and SEM were performed on 10 cm pieces of dry plant material. The samples were placed in an oven (~100 $^{o}$C) to eliminate any humidity.

### 3.3 Characterization

SEM analyses were performed on a tabletop microscope (TM3000, Hitachi) at the Material Design and Selection Laboratory, Federal University of Rio Grande do Sul (LDSM–UFRGS).

Dried tobacco stalk samples were analyzed. The samples were sectioned, fixed in stubs, and observed at different magnifications: 100, 250, 500, 1000, 2000, and 2500x. The analyses showed aspects of the morphology of the tobacco stalk. Data compilation is described in Section 4.1.

FTIR characterization was performed at LACER–UFRGS on an IRAffinity-1 spectrophotometer (Shimadzu). This analysis enables the identification of functional groups in the organic structure of a sample [17]. In this method, the radiation contained in all wavelengths is divided into two beams: one of fixed length and the other of variable length. The variable distance between the two length paths results in a sequence of constructive and destructive interferences and, consequently, in intensity variations: an interferogram. The Fourier transform converts this interferogram into a spectral point [18].

The resulting infrared irradiation interacts with the sample molecules, originating the spectra. The spectra are materialized from a series of bands or peaks. It is the presence of these bands, which are characteristic of groups, that enables the acquisition of valid information to identify the structures [18].

According to Pavia et. al. [18], each bond category has its natural vibration frequency, and a small absorption range can

be defined for each bond type. They also point out that the infrared spectra identify molecules as the fingerprints identify human beings. An infrared spectrum can provide structural information about a molecule. The absorptions of each type of bond are, in general, found only in certain small regions of the vibrational spectrum. A small absorption range can be defined for each type of bond. Outside this range, absorptions are usually due to some other type of bond. Absorptions in the 3000 ±150 $cm^{-1}$ range are most often due to the presence of the C-H bond in the molecule [18], whereas absorptions in the 1715 ±100 $cm^{-1}$ range may be associated with the presence of the C=O bond (carbonyl group) in the molecule [18].

According to Jobim [17,19], TGA is relevant for the thermal characterization of natural materials since this property directly influences the final product to which the tobacco stalk could be applied.

In this technique, the mass variation of the product sample, which was previously dried, is determined as a function of temperature and/or time, enabling the evaluation of its thermal stability, as well as the maximum application temperature [17].

TGA was performed at the Chemical Engineering Department, Federal University of Rio Grande do Sul (DEQUI-UFRGS) on an SDT Q600 V20.9 Build 20 aiming to assess the mass loss between 100 and 440º C. The analyses were conducted at a heating rate of 10 °C/min under usual atmospheric flow (100 ml/min) from 21 to 700 °C.

## 4. RESULTS AND DISCUSSION

### 4.1 Morphology of the tobacco stalk

Histologically, the samples revealed that the tobacco stalk is composed basically of the dermal, fundamental, and vascular systems. The dermal system is composed of the epidermis; the fundamental system is formed by the cortical and medullary parenchyma, differentiated in the cortical region into mechanical tissue of the collenchyma type; the vascular system comprises the tracheal elements of the xylem and the sieve tube elements of the phloem, in addition to mechanical elements of the fiber and parenchyma types.

Figure 3 illustrates the morphology of the tobacco stalk, showing bundles of vascular elements of the xylem, a complex vascular tissue responsible for the transport of water and mineral salts and characterized by the presence of tracheal elements, parenchyma cells, and fibers.

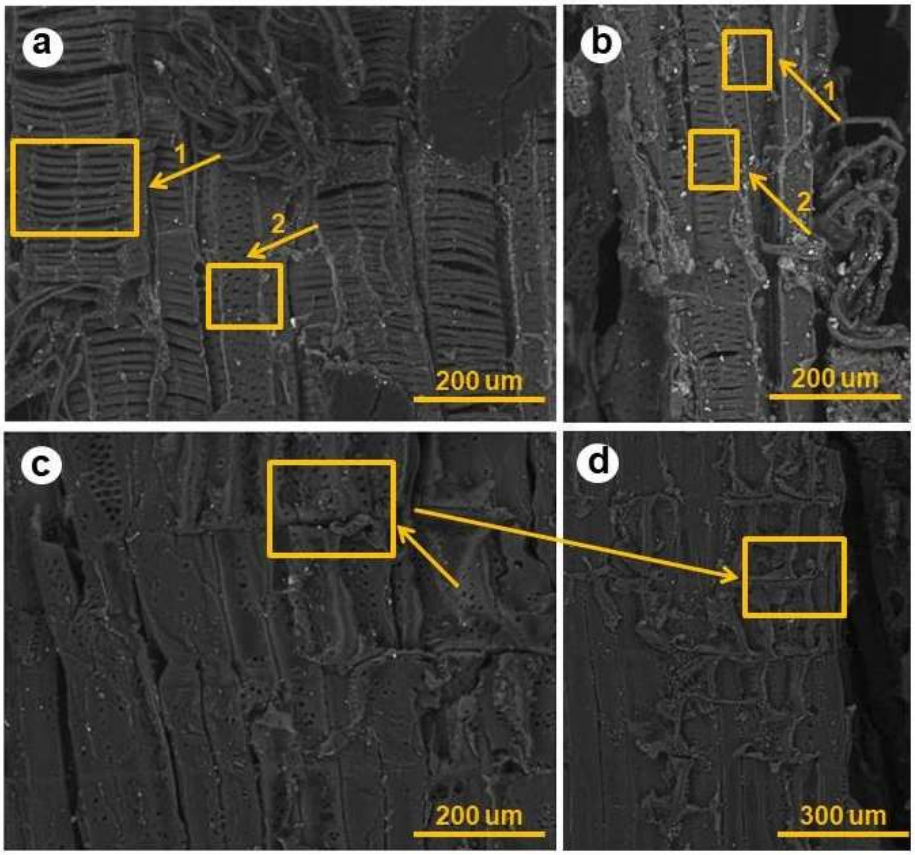


**Figure 3 – SEM image of a tobacco stalk sample: a) vessel elements with helical (arrow 1) and dotted (arrow 2) wall thickening; b) dotted (arrow 1) and scalariform (arrow 2) thickening; c) detail of parenchymal cells (arrow); d) parenchyma cells seen at greater magnification (arrow).**

The tracheal elements are the conducting cells of the xylem. The SEM images reveal tracheal elements called vessels, which are more frequent in angiosperms. The vessel elements have perforation plates in their structure. The figure also

shows parenchyma cells, responsible for activities such as photosynthesis, storage, and secretion, as well as for water flow and transport of substances in plants [20].

### 4.2 Chemical composition of the tobacco stem

Figure 4 shows the FTIR spectrum of the tobacco stalk. The band at 1732.21 cm $^{-1}$ corresponds to the carbonyl group (C=O). This group shows strong absorption in the 1880-11660 cm $^{-1}$ region. As the spectrum showed the presence of carbonyl, it was possible to identify the main types of the substance. The bands at 3340.87 and 2918.46 may correspond to OH compounds, which include absorbed water and primary and secondary aliphatic alcohols identified in cellulose, hemicelluloses, lignin, and carboxylic acids. The band at 1238.95 represents the ester group (C-O), a strong intensity band close to the 1300-1000 cm$^{-1}$ region. Double bonds or aromatic rings appear at the 1639.77 band since C=C shows a weak band close to 1650 cm$^{-1}$. Medium-to-strong absorptions in the 1600-1450 cm$^{-1}$ region suggest an aromatic ring. The band at 1504.01 can characterize aromatic rings (C-C). The band at 1420.18 cm$^{-1}$ may be associated with the C-C stretching of the aromatic ring. The band at 1372.30 cm$^{-1}$ may be related to the group of alkanes ($CH_3$ folding peak at ~1375 cm$^{-1}$). The band at 896.34 may be linked to the C-H vibration of lignin [17,18].

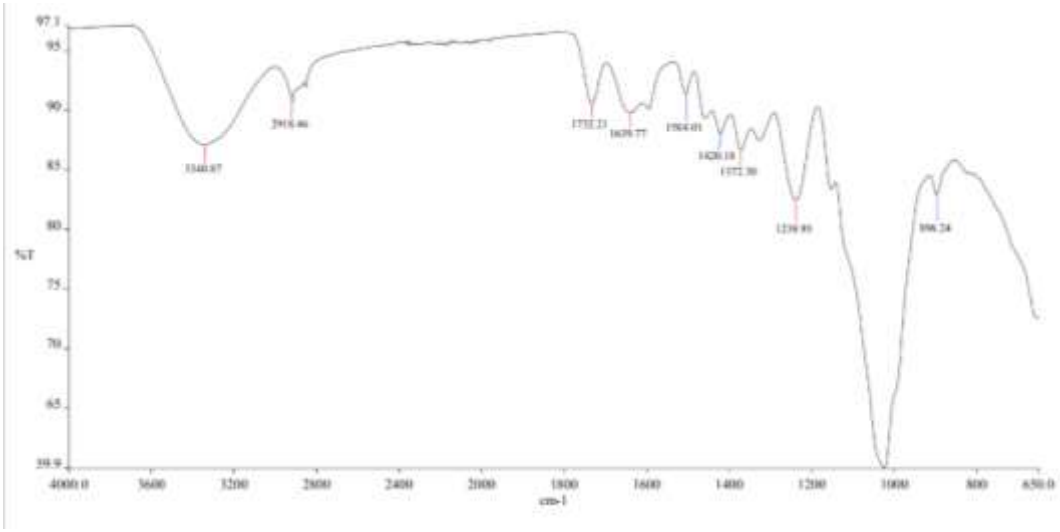


**Figure 4 – FTIR spectrum of the tobacco stalk.**

### 4.3Thermal properties of the plant compost

Figure 5, mainly in the green and blue lines, which represent the mass percentage and the mass percentage variation as a function of temperature in °C (weight % e deriv. weight %/Cº, respectively), shows that the stalk begins to lose mass (~10%) around 90 ºC, which may be associated with absorbed water, corroborating the analysis by Jobim [17]

It can also be observed that, from 90 to 250 ºC, the tobacco stalk sample loses about 20% of its mass, may be due to the decomposition of hemicelluloses and lignin. On the other hand, between 250 and 350 ºC, the mass loss can be related to the cellulose, which represents a loss of ~50%, whereas the following mass loss percentages are due to the thermal oxidation of the residues from the previous processes [17].

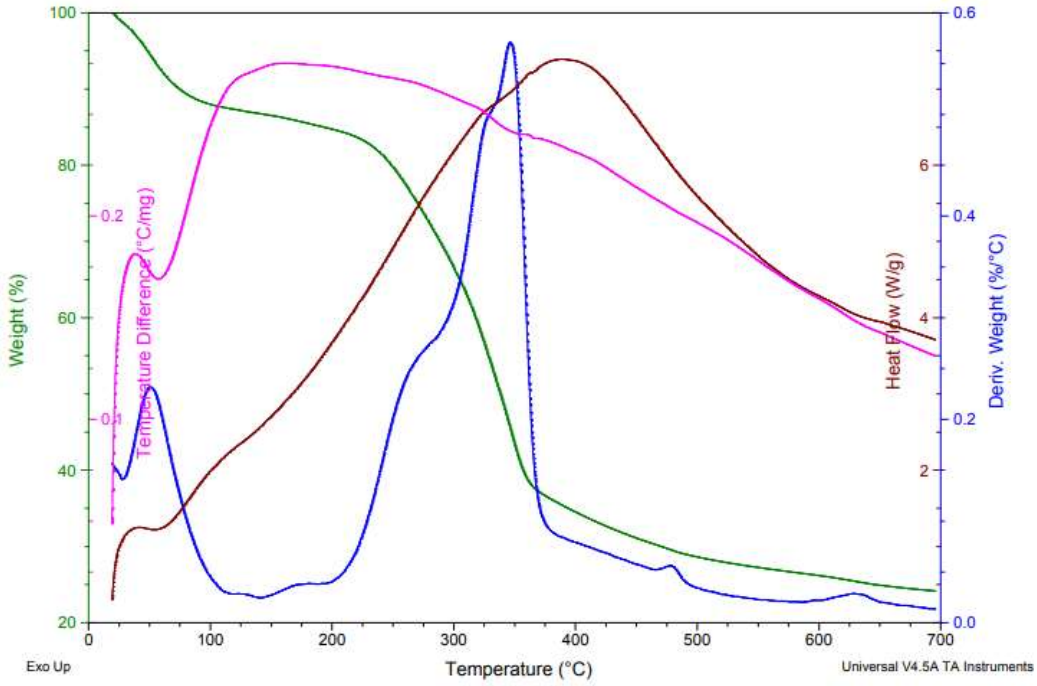


**Figure 5 - TGA results showing the relationship between temperature increase and weight loss of the tobacco stem. According to the graphic green line, from ~90 to 250 ºC, the tobacco stalk loses about 20% of its mass.**

## 5. CONCLUSIONS

The tobacco stem was characterized chemically, thermally, and morphologically through tests and analyses. FTIR showed the presence of compounds characteristic of polysaccharides, such as OH and C-O.

TGA showed that the tobacco stalk begins to lose mass at 90 °C. According to Fonseca (2012), plant fibers should not undergo procedures in which their mass loss reaches approximately 40%, because the material loses its chemical and physical integrity. Therefore, it is prudent that tobacco stalks not be processed at temperatures >300 °C.

SEM showed the presence of vascular components of the xylem that are characterized by having tracheal elements, parenchyma tissue, and fibers.

The tests and analyses carried out to determine the morphological, thermal, and chemical properties of the tobacco stalk show that this waste from the tobacco industry can be applied as a lignocellulosic filler in the form of particles in the manufacture of composite materials, mainly those with a polymer matrix.

A future study may move towards the development of a composite material made from tobacco waste with a polymeric matrix, in order to analyze its chemical, thermal, mechanical and morphological properties and propose the use of this material. Composites reinforced with a lignocellulosic filler are used in different areas and can be used as coating materials (construction components such as doors, windows, dividing walls), as furniture like chairs and tables and products for the automotive sector, such as interior finishing panels [21]. Still, it´s also possible to study the use of a composite made from a lignocellulosic filler to serve as raw material for new manufacturing technologies, such as 3D printing.

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