Use of musa paradisiaca fibers for the preparation and chemical, physical and mechanical characterization of a biodegradable composite material

C Martínez¹, W Ruiz¹, S Lesmes², D Laverde³, and A Pertuz¹

¹ Grupo de Investigación en Energía y Medio Ambiente, Universidad Industrial de Santander, Bucaramanga, Colombia
² Laboratorio de Espectroscopia Atómica y Molecular, Universidad Industrial de Santander, Bucaramanga, Colombia
³ Escuela de Ingeniería Química, Universidad Industrial de Santander, Bucaramanga, Colombia

E-mail: apertuzc@correo.uis.edu.co, sergio.lesmes@correo.uis.edu.co

Abstract. Composite materials with comparable properties and low environmental impact have been at the forefront of research. In the present work, the mechanical, physics and chemical properties that allow knowing the potential applications of Musa Paradisiaca fibers are evaluated, through an experimental process that starts from the selection of the binder and its fiber/binder ratio through tensile tests. The finally formed material is composed of a matrix of gelatin type C and glycerin and a reinforcement of banana fibers. The compound obtained was characterized by infrared spectroscopy by the Fourier transform, thermogravimetry, scanning electron microscopy and mechanical tensile and hardness tests. The results obtained from this study allowed confirming the presence of lignin and hemicellulose and evidencing the presence of good mechanical properties with a modulus of elasticity equivalent to 0.57 MPa, a percentage of elongation of 56%, an ultimate tensile strength of 3.5 MPa and a hardness of 82 Shore A, which allows us to consider a biodegradable composite material for applications in which soft gums and elastomers perform. The results obtained allow us to consider other alternatives for the use of Colombian banana fibers, in addition to their use as a biodegradable compound.

1. Introduction

In Colombia, Musa Paradisiaca is the most common banana crop, with consumption close to five million tons per year and which, in parallel to the harvest process, generates 95% of materials that are not used [1]. The remaining 5% is used as a fertilizer for the crop to decompose or as food for livestock, this excess waste without use adversely affects the environment. In search of a solution to this problem, the byproducts of the banana crop as a raw material have aroused interest in research that deals with the use of these, using them as an alternative animal feed [2].

However, there is an interest to expand its applications, for this, it is necessary to carry out a characterization that allows identifying the physical, chemical and structural properties that allow knowing the nature of the material and thus recognize its applications. Currently, there are many works devoted to the study of compounds made with banana fibers and cassava modified starch matrices [3], comparative studies of gelatin and nanocomposite films based on nanocellulose-modified starch for food packaging applications [4]. The group of researchers led by Zaman et al. [5] have presented a
comparison of the mechanical properties of polypropylene matrix compounds reinforced with banana fiber with and without ultraviolet radiation, demonstrating in this way that the mechanical properties of irradiated banana fibers and matrix-based compounds increase significantly compared to the untreated counterparts, which indicates the need to pretreat the fibers in order to obtain better mechanical properties.

For all the above, it is necessary to consider a study based on the use of banana fibers produced in Colombia and that allows, not only their use but to recognize their physicochemical characteristics aiming to obtain a product with added value. Thus, this paper presents the chemical, physical and mechanical characterization of the compound formed by binders and cellulose fiber obtained from the by-products of the harvest of the Musa Paradisiaca banana that allows recognizing its possible applications.

2. Materials and methods
To carry out the chemical, physical and mechanical characterization of the compound formed by binders and cellulose fiber obtained from the by-products of the plantain crop, the pre-treatment and selection of the fiber-binder with better properties was required.

2.1. Selection of raw material
The by-products selected as raw material are the leaves, the rachis and the pseudostem of the Musa Paradisiaca that were obtained after having collected the fruit, in the period of maturity of the harvest of a plantation of 18 months of age. The plantation is in Oiba, Santander, Colombia, at 1420 meters above sea level, has an average temperature of 20 °C and 88% relative humidity.

2.2. Pretreatment of fiber
Once the raw material is collected, it is washed with enough distilled water to eliminate external agents such as waxes, fats and soil residues. Later it was cut into pieces of approximately three centimeters in length and was crushed. The banana fibers obtained from the crushing are treated chemically with sodium hydroxide (97% NaOH, Merck) to eliminate the percentage of lignin and hemicellulose present in the fiber. Later they are cooked for half an hour at a temperature of 75 °C and 250 grams of NaOH per kilogram of banana fiber are added according to what was suggested by Sepúlveda et al. [6] The main function of the alkaline treatment is to act as a solvent, which helps to improve the surface roughness and wettability of the fibers, as well as the compatibility to form a composite material. After keeping at rest for a few minutes, the fiber is washed to remove excess NaOH. Next, in order to make the cellulose whitish, use sodium hypochlorite (NaClO 15%, Merck) in a concentration of 100 ml of NaClO per 2 kilograms of banana fiber, while stirring constantly until it starts to turn into a yellow color, which occurs after a period of 2 hours. Subsequently, the fiber is washed to remove the hypochlorite and finally subjected to 60 °C for 12 hours in a drying oven, the product obtained is preserved for later use.

2.3. Selection of the binder
In this phase, different types of binder were selected that function as a complement in the biodegradable part of the composite material as reported by Rodriguez and Orrego et al. [7] To carry out the selection, specimens standardized according to ASTM D3039 [8] with the following dimensions length 40 mm, width 2-3 mm and thickness 0.5 mm - 2 mm, which were adapted to make tensile tests in the MTS Bionix universal testing machine, the tests were carried out at room temperature (25 °C).

2.4. Characterization of the composite material
To carry out the chemical, physical and mechanical characterization of the composite material, mechanical characterization tests were carried out as tensile tests, for which the MTS Bionix universal testing machine was used to analyze different deformation modes of materials. The method used is an adaptation of the sample ASTM D 3039 [8] standards for the equipment. The Shor hardness test was
carried out according to the ASTM D2240 [9] standard for moderately hard rubber, thermoplastic elastomers, paper products and fibrous materials, using a Shore D durometer. The Shore hardness is a scale measuring the elastic hardness of the materials, determined from the elastic reaction of the material when an object is dropped on it. As for the morphology, it was evidenced by means of a scanning electron microscope SEM Quanta Feg 650.

For the chemical analysis of the composite material an infrared spectroscopy analysis by Fourier transform (FTIR) was performed on a Shimadzu IRTracer spectrophotometer. Finally, the physical analysis by thermogravimetry (TGA) was carried out with a thermogravimetric analyzer TGA 5500 TA Instruments, which uses a heating rate of 10 °C per minute up to 600 °C in a nitrogen-controlled atmosphere with a heat flow of 50 ml/min.

### 3. Results and discussion

The selection parameter for the binder of the composite material is the modulus of elasticity obtained through the tensile test, in which the behavior of each of the binders is compared to a tensile force exerted by the equipment.

**Figure 1.** Curve effort vs deformation for (a) different binders and (b) curve effort vs comparative deformation of fiber-gelatin proportions.

Figure 1(a) shows that the compounds of gelatin type C, cassava starch, glycerin and fiber show minimal entanglement at an approximate stress of 2.5 MPa, which implies a rigid behavior with a high modulus of elasticity (fragile material), contrary to the polyvinyl acetate (PVA) which indicates ease of strain at a lower tension force (ductile material), while the compound formed by gelatin type C, glycerin and fiber is distinguished by maintaining a greater proportionality between the stress and the strain, going from elastic to plastic behavior until it reaches its breaking point (ductile material). Taking into account the results obtained from the tensile test and the visual inspection based on the texture, roughness, dispersion and compaction of the fibers, it is inferred that the material formed by the type C gelatin and the fibers of the banana by-products represent a better possibility to be the ductile material of greater resistance to tension, with the ability to adopt specific shapes without breaking or changing its structure, which makes it attractive to be molded and extends its possibilities of use, as opposed to the fragile material formed by gelatin and starch that has a limited elasticity and suffers from permanent deformations when subjected to tension. Table 1 summarizes the modulus of elasticity obtained for each binder.

Based on the previous table, which ratifies the decision to use type C gelatin as a binder, considering that it is intended to find a material that has a greater range of uses and versatility, which makes fragile materials disposable. Once the binder was selected, the definition of the appropriate volumetric
proportions was continued, again based on the tensile test and the modulus of elasticity, this time establishing differences between the amounts that make up the compound. As can be seen in Figure 1 (b), compounds with 40%, 50% and 60% binder were put to the test, of which 4%, 5% and 6% glycerin respectively are added as a complement to the binder.

### Table 1. Modulus of elasticity for each binder.

| Binder          | Gelatin + Starch | PVA   | Gelatin type C |
|-----------------|------------------|-------|----------------|
| Elasticity module (MPa) | 4.196            | 0.002 | 0.416          |

This time the results show an obvious relationship between the percentage of binder and the tensile strength of the compound. As can be seen, the compound with 40% binder and 60% fiber reaches the breaking point with stress of approximately 2 MPa after having strain by 32%. On the other hand, the compound formed by 60% of binder and 40% of fiber shows a material that does not yield easily before the tension when reaching an elongation of only 10% with the same stress exerted to the previous compound. The compound made with equivalent fractions of binder and fiber has a more ductile behavior, has a higher tensile strength compared to the other compounds tested, which makes it a material with greater amplitude of applications. In this way, the characterization tests will be carried out from a biodegradable composite material with banana fibers with a 50%-50% fiber-binder ratio (type C gelatin and glycerin).

#### 3.1. Chemical characterization: Fourier-transform infrared spectroscopy analysis

FTIR spectra were taken to determine the vibration states and consequently the functional groups present in the banana fibers after the chemical treatment, as shown in Figure 2(a). Considering that natural fibers are composed mostly of cellulose, hemicellulose and lignin, the presence of these compounds was experimentally determined.

The aromatic vibratory plane C=C of the bands corresponding to lignin, which are at 2916 cm⁻¹; similarly, there are signs pertaining to C-O-C of glycosidic ether in 3311 cm⁻¹, 2849 cm⁻¹, 1317 cm⁻¹ and 1026 cm⁻¹, of polysaccharide components of cellulose, which is consistent with the results presented by Jiménez [10]. Also, traces are observed to stretch in the tension of the carbonyl bonds -C=O, because the band of 1597 cm⁻¹, corresponding to hemicellulose, is maintained. It was possible to determine that there is a reduction of lignin, but still, they have traces of this and hemicellulose in comparison to the results presented by Jiménez [10] for the banana fibers without treatment.

![Figure 2. FTIR spectrum (a) banana fibers (b) FTIR comparison of fibers and compound.](image-url)

Figure 2(b) shows the wavenumbers corresponding to the vibration states of cellulose, hemicellulose and lignin that were intensified with respect to the signals presented by the fiber with chemical treatment, due to the incorporation of the binders (gelatin type C + glycerin) which are a type of biologically based
elastomers [11], function as a natural polymer which in turn contains abundant hydrophilic groups in the chains, such as amino, hydroxyl and carboxyl. Due to the above, the formation of a large number of hydrogen bonds between the hydroxy groups of the fiber can be explained, glycerin has a large number of hydroxyl groups, which can alter the hydrogen bonds between the hydroxyl groups. Gelatin molecules, facilitating the mobility of these groups polarized in gelatin as reported by Nanying [11], which is why the interaction between the fibers and the binder becomes possible. Likewise, it should be noted that there are more carbons available for simple links than for double bonds, which is evident in the widening of the 1456 cm\(^{-1}\) band to 608 cm\(^{-1}\) and the absence of signals strong in the number of wave of the double bond, if not the presence of stretches of this in 1632 cm\(^{-1}\) and 1541 cm\(^{-1}\). On the other hand, the intensification of the signal of the hydrogen bonds present in 3296 cm\(^{-1}\) as well as the signal of the stretching of the C-H bond in 2916 cm\(^{-1}\) and 2849 cm\(^{-1}\).

3.2. Physical characterization: Thermogravimetric analysis

Figure 3 shows the weight loss of the composite material as a function of temperature. In the thermogravimetric analysis, a multistage decomposition is observed where three regions of weight loss are evidenced mainly.

![Figure 3. Weight percentage vs. temperature.](image)

In the first region there is a mass loss starting from 40 °C to 200 °C, which is due to the elimination of humidity from the sample, as well as the beginning of the decomposition of the fiber that begins with the hemicellulose to the 180 °C, with a weight loss of 19%; a second region where the main decomposition occurs with a decay between 200 °C to 355 °C, with a mass loss of 50%, caused by the thermal degradation of hemicellulose; Cellulose at 240 °C, which occurs in two stages: the first occurs by the breaking of bonds and the generation of free radicals, carboxyl and carbonyl groups, as well as carbon, causing the reduction of the length of the chain. And a second stage consisting in the depolymerization of cellulose by breaking glycosidic units and formation of levoglucosan, and lignin at 280 °C, main components of lignocellulosic fibers. Finally, the third region between 360 °C - 600 °C, with a mass loss of 13.4%, range corresponding to the degradation of lignin, some polysaccharides and inorganic substances, who are responsible for the continuity of the process degradative that occurs at temperatures above 550 °C [12].

The peak of degradation temperature was determined from the derivative of the thermogravimetric curve whose value is 340.35 °C and corresponds to the temperature at which the maximum decomposition rate was detected.

3.3. Mechanical characterization: tensile test, shore hardness D

The samples subjected to tension and tension are described in Table 2, in which the results are expressed for the composite material made with 50% binder and the remaining amount of banana fiber is
distinguished by having a modulus of elasticity equivalent to 0.574 MPa, an elongation percentage of 56.23% and a tensile strength of 3.521 MPa that can be compared with materials such as expanded polystyrene (EPS) that has a tensile strength between 0.1 MPa and 0.6 MPa.

It was evident in the test carried out on the sample that Shore D hardness is in a range between 29 and 32. The arithmetic mean of the 5 measurements is 30.6 which is low value in Shore D hardness that includes hard gums and thermoplastics, however, represents a relatively high value of 82 in Shore A hardness for soft rubbers, plastics and elastomers.

On the other hand, the SEM images of the composite material of the fractured sample subjected to tensile testing is presented in Figure 4, where the fracture of the fibers is observed due to the tensile test, in which the direction of the fibers caused is observed by the tension test, where the elongation is longitudinal and the maximum shear stresses act on planes orientated at 45° of the tension axis. These shear stresses are responsible for the plastic deformation process. Therefore, the clear behavior of a ductile material subjected to traction is observed where it will begin to fracture according to these planes, resulting in a rough surface and a ductile 45° edge.

![Figure 4. Micrography composite material.](image)

| Properties         | Value | Units |
|--------------------|-------|-------|
| Yield point        | 0.65  | MPa   |
| Elasticity module  | 0.57  | MPa   |
| Tensile strength   | 3.52  | MPa   |
| Breaking point     | 0.81  | MPa   |
| Resilience         | 0.37  | MPa   |
| Tenacity           | 136.78| MPa   |
| Ductility          | 537.23| %     |
| Hardness           | 82.00 | Shore A |
| Degradation temperature | 340.35 | °C |

Finally, it can be said that the obtained properties allow comparing the obtained composite material with other materials reinforced with natural fibers such as natural silk fiber with a gelatin matrix, this material has characteristics similar to the banana fiber compound, with a modulus of elasticity of 0.65 GPa, a tensile strength of 44.5 MPa, an elongation percentage of only 8.2% and a hardness of 96 Shore A. According to the above, the material formed by the fiber of Banana has a higher percentage of elongation, however, its other mechanical properties are low compared to those made by the referenced research.
4. Conclusions

It was possible to obtain a matrix composed of gelatin type C and cellulose fiber obtained from the waste of Musa Paradisiaca in a ratio of 1:1 in a matrix with a random arrangement of the fibers. The mechanical properties of the compound confirm the dependency ratio between the fiber and binder ratio and the modulus of elasticity. In turn, according to the Stress vs. Deformation curves obtained from the tensile test and the images generated in the SEM where the position of the fibers at 45° is evidenced giving evidence of the performance of the maximum internal shear stresses, it is deduced that the resulting compound behaves like a ductile material, with a tensile strength of 3.52 MPa and a percentage of elongation equivalent to 56.23% with the ability to deform without breaking which allows the molding of the compound in specific forms. The hardness test resulted in a hardness of 82 Shore A, which makes it possible to classify the material as an elastomer. The compound has good mechanical properties and can be compared with soft plastics which makes it attractive for applications that require fast, clean and biodegradable processing. On the other hand, the curve of percentage in weight vs temperature of the obtained from the thermogravimetric analysis the composite material suffers a constant loss of weight as the temperature increases, which makes it difficult to have exact information of the service temperature. On the other hand, the spectroscopy technique allows corroborating the chemical composition of the matrix. Evidence the typical C-C bonds of the FTIR spectra, confirming the presence of banana and gelatin fibers.

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