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Obtaining Composite Materials as an Alternative use of Pineapple Residual Biomass for the Generation of Polymers Reinforced with Natural Fibers

Mauricio Sierra-Sarmiento[a], Laura Marcela Barrera-González[a], Olga Marín-Mahecha[a], Jannet Ortiz-Aguilar[b], Maikel Suárez-Rivero[c], Deivis Suárez-Rivero*[a]

[a] Grupo de Investigación e Innovación Agroindustrial – GINNA, Fundación Universitaria Agraria de Colombia – UNIAGRARIA, Colombia
[b] Facultad de Ingeniería, sede Bogotá, Universidad Cooperativa de Colombia – UCC, Colombia
[c] Universidad Nacional Abierta y a Distancia – UNAD
deivissr2000@hotmail.com

One of the biggest problems in agricultural crops in general and, given its agronomic characteristics, in the pineapple crop, is the disposal of harvest residues. Currently, residues are removed and deposited in areas adjacent to the crop, becoming a focus of insect pests and diseases. Given the above context, the use of the afore mentioned foliar residues was evaluated as an alternative for obtaining composite materials from the plant's leaf fiber, thus generating added value to this residue. Initially, the leaves were subjected to three treatments to verify the effect on the tensile characteristics of the fibers. The treatments consisted of subjecting the leaves to fermentation at two temperatures (ambient and 30 °C) and in the presence of a NaOH solution [5%]. Subsequently, composite briquettes were made from pineapple fibers in proportions of 20 % and 30 % mixed with glycerol, guar gum, magnesium stearate and starch, evaluating the effect of the concentration of these fibers on the physico-mechanical properties of tension and elongation. Likewise, the effect of the use of a nanoclay in concentrations of 2.5 % and 5 % as reinforcement of the composite materials in a mixture with a fiber percentage of 20 % and 30 % was evaluated. In fermentation, significant differences ($P>0.5$) were found between the treatments, verifying that the best pretreatment was presented by the fermentation at room temperature with stress values of 373.33 ± 10.67 MPa, followed by the treatment at a temperature of 30 °C with 261.33 ± 23.09 MPa. On the other hand, when evaluating the tension and elongation, significant differences ($P>0.5$) were found between treatments, reporting values of 0.51 ± 0.03 MPa and 0.18 ± 0.0028 MPa for fiber concentrations of 20 % and 30 %, respectively. The effect of the use of nanoclay evidenced a higher strain in the treatment with 30 % fiber and 5 % nanoreinforcement with a value of 0.15 ± 0.0172 MPa.

1. Introduction

One of the great challenges currently facing the different production chains is the management of post-harvest solid waste and its insertion into the circular economy, thus generating higher value chains. Currently, the treatment of agricultural waste generated after the end of the pineapple crop production cycle is a major problem, since it is generally removed by four methods: the first method consists of abandoning the stubble in the field, which requires approximately 13 months, becoming a limitation for the producer since it is not possible to wait that period of time to carry out the new planting, in addition there is the possibility of the multiplication of insects such as the white fly affecting the environment (Salas, 2018); the second method, consists of performing chemical or physical burns (Lizarazo, et al. , 2020), thus generating high environmental damage in terms of loss of soil fertility and air pollution, causing damage to fauna, flora and people; the third method, consists of shredding the waste for application as herbicides, but one of the disadvantages of this action is the abuse given to herbicides thus causing a restriction in soil fertility; finally, the fourth method consists of burying the pineapple waste in pits, an action that not only causes loss of the fertile layer of the soil, but also its economic cost is high (Salas, 2018) , not only because of the characteristics of the fibers, but also because it is more than 60% of the

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total biomass produced in the plantation. In this order of ideas, the use of pineapple leaves to obtain composite materials is an alternative that plays a crucial role in the valorization of waste (Fontanelli, et al., 2015; Murcia, Ardila, Barrera, 2020).

2. Material and methods

The research was carried out with pineapple leaves (*Ananas comosus* (L.) Merr) from the "*Raíces*" farm in the municipality of Puerto López, Meta, Colombia. These were conditioned and processed in the laboratory of Natural Ingredients of the *Fundación Universitaria Agraria de Colombia* - UNIAGRARIA. As described by Suárez-Rivero, et al. (2016), this laboratory has the necessary equipment for the analysis of raw materials of biological origin, thus generating, from this space, added value to the productive chains.

2.1. Pretreatment of pineapple leaves and its influence on the physicochemical characteristics of the fibers obtained

The pineapple leaves were subjected to three pretreatments: alkaline, fermentation at 30°C and fermentation at room temperature, evaluating the physicochemical and physic-mechanical characteristics for each pretreatment, following the methodology proposed by Aguilar & Tapia (2023). Subsequently, the fibers obtained in the pretreatments were evaluated for dimensional characteristics, morphological analysis by optical microscopy and physicochemical analysis (Romero Granados, 2021).

For the dimensional analysis, the width, length, and thickness of 10 fibers chosen at random for each pretreatment were determined. On the other hand, for the morphological analysis, given the dimensions of the fiber obtained, an optical microscope with a coupled camera was used (Suárez-Rivero, et al., 2017). Likewise, for the chemical analysis, which was performed following the methodology proposed for vegetable fibers by Roni, Dian, Satriyo, (2014), who reflect that, to generate added value to these, at least their cellulose, hemicellulose and lignin content must be known; contents that can be calculated as shown in equations 1 to 3.

$$Cellulose\ (\%) = \frac{c-d}{a} * 100 \qquad [1]$$

$$Hemicellulose\ (\%) = \frac{b-c}{a} * 100 \qquad [2]$$

$$Lignin\ (\%) = \frac{d-e}{a} * 100 \qquad [3]$$

Where: (a) is the weight of the sample, (b) is the weight of the initial dry residue after boiling in water, (c) is the weight of the dry residue after the first digestion with 1N H_2SO_4, (d) is the weight of the dry residue after the second digestion with 1N H_2SO_4 and (e) corresponds to the weight of the final dry residue after being calcined in a muffle at a temperature of 600°C for four hours.

2.2. Analysis of the effect of pineapple leaves pretreatment on the physical-mechanical characteristics of the fibers obtained.

Analysis of the breaking strain: An analysis of the mechanical characteristics of the fibers was carried out in which the breaking strain was determined according to ASTM C1557-20 (2020). The tests were performed following the methodology presented by Romero Granados (2021). Thus, the nominal breaking strain was determined using equation 4, where (F) represents the force and (A) represents the cross-sectional area of the test specimen. The analysis was performed in triplicate.

$$Tension = \frac{F}{A} \qquad [4]$$

Determination of the modulus of elasticity of the fibers: To determine the stiffness of the material, the method established in Standard D3039/D3039M-08 (2014) was used. The tests were performed following the methodology presented by Cárdenas & Perdomo (2020). The value of Young's modulus or elastic modulus was determined with equation 5, where (L_0) represents the initial length, (ΔL) represents the increase in length or deformation of the specimen and (A) represents the cross-sectional area. The analysis was performed in triplicate.

$$Young's\ module = \frac{F * L_0}{A * \Delta L} \qquad [5]$$

2.3. Concentration of pineapple leaf fibers on the physic-mechanical characteristics of the obtained composite materials

After determining the best pretreatment by means of the physical-mechanical tests performed to the pineapple fibers, the effect of the concentration of pineapple fibers on the physical-mechanical characteristics in obtaining composite materials was established, the above considering previous studies (Alfaro, 2017) in which it is evidenced that the fiber concentration can significantly affect the mechanical characteristics of the composite materials. Following the methodology carried out by previous studies authors (Both, Leites, Tessaro, 2021), the obtaining of composite materials was carried out by mixing materials such as glycerin, water, guar gum, modified corn starch, magnesium stearate and pineapple leaf fiber.

For the analysis of the concentration of pineapple leaf fibers on the mechanical characteristics of tension and elongation of the composite materials, the fiber content was varied between 20% and 30% (Vercelheze, et al., 2012; Suárez-Rivero, et al., 2023) leaving constant the concentrations guar gum (1%), magnesium stearate (1.5%) and glycerin (10%); it is important to mention, that the amount of water varied depending on how the composition of the mixture was (Matsuda et al., 2013) and (Martins, Benelli, Tessaro, 2020). Similarly, it should be noted that a blank consisting only of starch was made, leaving constant concentrations of guar gum (1%), magnesium stearate (1.5%) and glycerin (10%) (Vercelheze, et al., 2012).

3. Results and Discussion

3.1. Effect of pretreatment of pineapple leaves on the physicochemical characteristics of the fibers obtained

It is important to mention that for each of the pretreatments, ten random samples were chosen, and their measurements were taken, taking the average for each pretreatment. The variability in the results is because during the process of enlistment and extraction of the fibers, these tend to fracture. Similarly, when comparing the results obtained by each method, it is observed that between pretreatment A and F-30 there is a small difference in the thickness, but in the width and length there is a significant difference. Likewise, a significant difference is observed between F-A and the other two pretreatments. Therefore, the best pretreatment in the dimensional characteristics of pineapple leaf fibers was F-A, because it has both thickness, width, and length with greater measure (Table 1).

Table 1. Dimensional characteristics of the fibers obtained in each pretreatment.

Pretreatments	Thickness (mm)	Width (mm)	Length (mm)
Alkaline (A)	0,029	0,140	170
Fermentation at 30°C (F-30)	0,025	0,078	199,2
Fermentation at room temperature (F-A)	0,371	1,100	386

Figure 1 describes the behavior of cellulose, hemicellulose, and lignin in the fiber structure of pineapple leaves, according to pretreatment. As can be observed for the percentage of cellulose, all pretreatments are significantly different with a probability P<0.05.

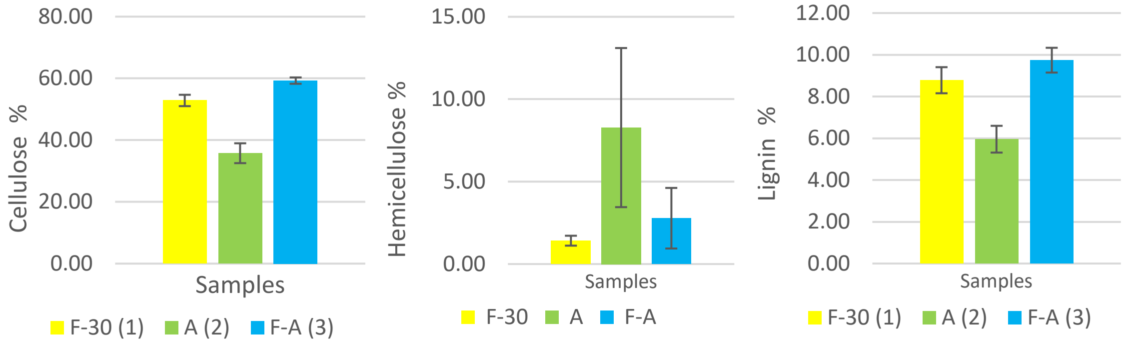

Figure 1: Percentage of cellulose, hemicellulose, and lignin in pineapple leaf fibers according to pretreatment. (The bars represent the error at 95% CI).

Likewise, for the percentage of hemicellulose the pretreatments F-30 and F-A are significantly different from pretreatment A, with a probability P<0.05, but when comparing F-A and F-30 no significant differences are observed between them. On the other hand, when analyzing the percentage of lignin, it is observed that F-30 and F-A do not differ from each other, but differ significantly with a probability P <0.05 with respect to pretreatment A, the latter presenting the lowest values.

3.2. Effect of pretreatment of pineapple leaves on the physic-mechanical characteristics of the fibers obtained

As can be seen in Table 2, the highest values are for pretreatment F-A with a range 373.33 ± 10.67 MPa, evidencing that pretreatment F-A presents a greater range of both stress and deflection compared to the other pretreatments analyzed. The differences between the stress means for all pretreatments are significantly different with a probability $P < 0.05$.

Table 2. Fiber breaking strain according to pretreatment.

Pretreatment	**F-30**	**A**	**F-A**
Breaking strain (MPa)	261 ± 23	36 ± 3	373.33 ± 10.67

When evaluating Young's Modulus, as can be seen in Table 3, the highest values were presented for pretreatment F-30, with a range 38338 ± 456 MPa, i.e., F-30 presents a greater stress range with a lower deviation value; on the contrary, it is observed that pretreatment A, as well as F-A present data with greater heterogeneity, which causes from the statistical point of view a nominal deformation. The differences between the means of Young's modulus for all pretreatments are significantly different with a probability $P < 0.05$.

Table 3. Young's modulus data of the fibers.

Pretreatment	**F-30**	**A**	**F-A**
Young's modulus (MPa)	38338 ± 456	3088 ± 2298	28759 ± 1490

3.3. Physic-mechanical characteristics of the composite materials obtained

According to the analysis presented above, the pretreatment by fermentation at room temperature (F-A) was chosen. In this sense, two mixtures (F-A1 and F-A2) were made with the fibers obtained, with the different concentrations mentioned in section 2.3, to establish the effect of the concentration of these fibers on the physical-mechanical characteristics of the briquettes obtained, compared with a mixture called control or blank. Thus, when evaluating the breaking strain, as can be seen in Figure 2, the highest values were found in the F-20 briquette, registering values of 0.51 ± 0.03 MPa, followed by F-30 0.18 ± 3.06 MPa. Thus, the differences between the stress means demonstrate that the F-20 briquette differs significantly from the blank and F-30, but the latter two do not have significant differences between them with a probability $P < 0.05$.

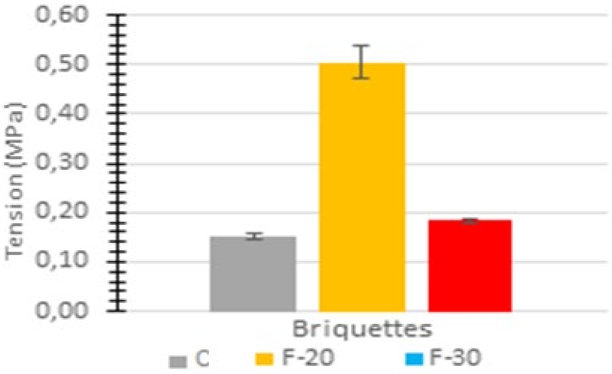

Figure 2: Tensile strength of briquettes varying the percentage of pineapple (Ananas comosus (L.) Merr) leaf fiber. (The bars represent the error at 95% CI).

On the other hand, the deformity analysis, as shown in Figure 3, evidences that the highest values are for the F-A1 mixture, coinciding with the highest strain values shown in Figure 2, presenting in this case a range 140.68 ± 1.78 MPa. The differences between the means of Young's modulus for all the briquettes are significantly different with a probability $P < 0.05$, where the F-A1 briquettes differ significantly with the rest of the briquettes analyzed, but there are no significant differences when comparing the behavior of this variable for the white and F-A2 briquettes with a probability $P < 0.05$.

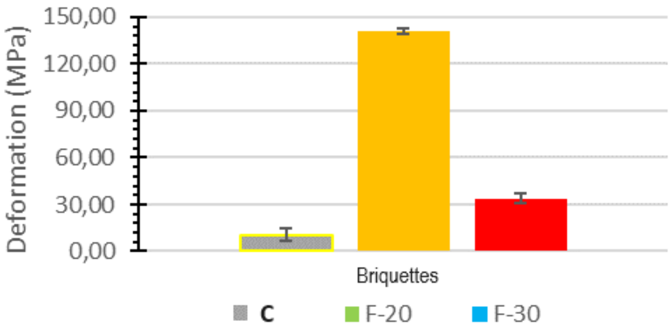

Figure 3: Young's modulus of the briquettes varying the percentage of pineapple leaf fiber (Ananas comosus (L.) Merr*).*

In tension and deformation tests, a correct adhesion of the matrix used in the manufacture of the briquettes, as well as the reinforcement used, has multiple factors that play against it, highlighting in the first place the humidity of the environment that penetrates the matrix before being melted, so it obtains a certain resistance to flow, which becomes one of the main disadvantages. Another of these can be associated with the irregularities of the mold, which leads to the appearance of pores in the briquettes that will become stress concentrators that affect the isotropy of the composite; finally, the method of inserting the fiber in the mold can cause the matrix not to achieve the ideal adhesion to the reinforcement, creating areas where the reinforcement can detach from the binder material more easily (Suárez-Rivero, et. al., 2023).

4. Conclusions

The low experimental stiffness presented in both tension and deformation tests, in the briquettes, under the formulations studied may be related to the presence of pores in the structure and the fiber; it could also be explained by the matrix-reinforcement adhesion ratio, which may not reach the optimum point; likewise, in this article a homogeneous distribution of the reinforcement along the matrix is assumed, which in practice is complex due to the manufacturing process used.

An increasing proportional relationship between the maximum tensile stress and the percentage of fiber is evidenced using the aligned arrangement of these, which reflects an increase of the stress as the amount of reinforcement increases; the behavior of the tensile modulus of elasticity presents a decreasing proportional tendency in the case of the long fiber, which means that the material has greater ductility when it has a lower percentage of reinforcement.

Nomenclature

A	Alkaline
F-A	Fermentation at room temperature
F-30	Fermentation at 30°C
F-A1	Fermentation at room temperature (16 g of starch / 4 g of natural fiber / 15 ml of H_2O)
F-A2	Fermentation at room temperature (14 g of starch / 6 g of natural fiber / 17 ml of H_2O)
C	Control (20 g of starch / 0 g of natural fiber / 15 ml of H_2O)

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