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Article

Potential of cocoa husk waste for the paper industry

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Abstract

The study evaluated the lignin and cellulose contents in the waste of three varieties of cocoa grown in the cantón of Francisco de Orellana (Ecuador)- Nacional, CCN-51, and Super Árbol- in order to explore their potential as an input for the production of kraft paper. The collection was carried out on farms of the Asopriabet association, and the chemical analyses were developed at the National Institute of Agricultural Research, applying standardized methodologies (TAPPI T-222 and Kürschner-Hoffer). The results showed that CCN-51 had the highest lignin content (45.13%), while Nacional showed the highest percentage of cellulose (28.57%). Nevertheless, the differences between varieties were not statistically significant. It was concluded that, despite this, the potential of this waste as an alternative raw material is supported, suggesting complementary studies that include crop variability, physical characterization of the pulp and production pilot tests to validate its industrial applicability.

Keywords:

agribusiness, biomass, byproducts, cultivars.

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Introduction

The use of agricultural byproducts has become highly relevant in the search for sustainable solutions for various industries, including paper production (Dey *et al*., 2021; Gómez-García *et al*., 2021). Among these byproducts, cocoa waste stands out as a potential source of cellulose, a component of great interest for the paper industry due to its ability to form resistant and cohesive fibers. Although this waste also contains lignin, this is usually removed during the pulping process, as its presence negatively affects the whiteness, durability, and quality of the paper (Gutiérrez-Macías *et al*., 2021; Jakob *et al*., 2022). Nonetheless, some authors mention that, in order to determine the true potential of agricultural waste in the production of kraft paper, it is necessary to consider other parameters in addition to the cellulose and lignin contents, such as the length and strength of the fibers, presence of ash, pulp yield, and the Kappa index, which indicates the degree of residual lignification (Megra *et al*., 2022; Hailemariam and Woldeyes, 2024).

Previous research has explored the use of agricultural waste for paper manufacturing, focusing on crops such as corn, sugarcane bagasse, and other byproducts rich in plant fiber (De Corato *et al*., 2018; Koul *et al*., 2022; Siqueira *et al*., 2022; Enawgaw *et al*., 2023; Plakantonaki *et al*., 2023). However, few studies have investigated the potential of cocoa waste despite its availability in producing countries such as Ecuador. Cabezas-Andrade *et al*. (2024) have documented that, based on the cocoa postharvest process, it was possible to obtain an amount of exosperm waste of around 252 kg ha^{-1} of the total exocarp weight and an amount of mucilage of around 930 L ha^{-1} of the total weight.

Some recent studies have begun to evaluate the chemical and morphological characteristics of residues such as cocoa husks and corn stalks, highlighting their contents of cellulose, hemicellulose, lignin, and ashes. For example, it has been found that the corn stalk has a higher content of cellulose (39%) and hemicellulose (42%), as well as a lower percentage of lignin (7.38%) compared to cocoa husks (Daud *et al*., 2014). In particular, the analysis of the chemical properties of the different varieties of cocoa, such as Nacional, CCN-51, and Super Árbol, in relation to their industrial potential, remains limited (García-Briones *et al*., 2021; Viteri, 2022; Cevallos, 2024).

Cocoa in Ecuador, as one of the largest producers in the world (Saravia-Matus *et al*., 2020; Villacis *et al*., 2022), generates large volumes of byproducts, which, in many cases, are not appropriately used, generating a significant environmental impact (Ramos-Ramos *et al*., 2020). The main objective of this study was to evaluate the lignin and cellulose contents in cocoa husks in three varieties, namely Nacional, CCN-51, and Super Árbol, as an alternative raw material for kraft paper. This analysis seeks to provide key information for the development of more sustainable and efficient industrial practices in the management of agricultural byproducts.

Materials and methods

Collection site

The cocoa husk collection was carried out in the cantón of Francisco de Orellana, belonging to the Province of Orellana, Ecuador, whose coordinates are -0.4625 north latitude, 76 984 167 west longitude. Located in the northwest of the Ecuadorian Amazon Region, this territory is part of Planning Zone 2, composed of the provinces of Pichincha, Napo, and Orellana. The cantón territory covers 7 047 km^2, with altitudes ranging from 100 to 720 m (GADM Francisco de Orellana, 2023).

Raw material sampling

Three samples of cocoa husks of 3 kg each were collected in triplicate from three farms in the cantón of Francisco de Orellana (Figure 1), belonging to the Asopriabet association, as part of the project to revalue Amazonian cocoa husks for the sustainable production of kraft paper.

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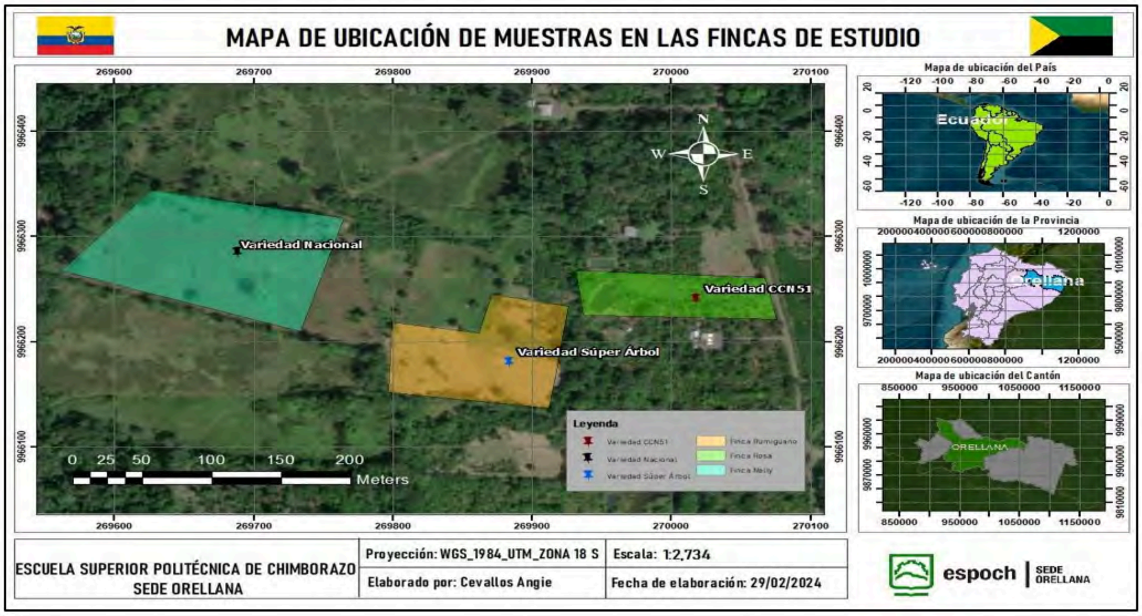
Figure 1. Location of the farms.



Biomass characterization

The chemical characterization of cocoa husk biomass was carried out at the National Institute of Agricultural Research (INIAP), for its acronym in Spanish at the Central Experimental Station of the Amazon. Cellulose, hemicellulose, and lignin contents were determined. Cellulose was quantified using the Kürschner-Hoffer method (Kürschner and Hoffer, 1931; Kapun *et al*., 2024) and lignin using the TAPPI T-222 method (TAPPI, 2002a). Sample preparation: 500 g of the sample was weighed and wrapped with newspaper. The sample was then subjected to a controlled heat treatment at 65 °C for 24 h or until a constant weight was reached. The sample was extracted and crushed with a mortar and pestle until adequate homogenization was obtained. The resulting fragments were stored in properly coded plastic bags for further analysis.

Determination of lignin content

In a volumetric flask, 665 ml of concentrated sulfuric acid was gradually poured into 300 ml of distilled water, followed by an adjustment with distilled water until a total volume of 1 000 ml was reached. One gram of the extract-free sample was weighed and placed in a container. Fifteen milliliters of 72% sulfuric acid were added while constantly stirring. The mixture was left to stand at room temperature (26 °C) for 2 h until it took on a dark tone. Subsequently, the mixture was transferred to the Dosi-fiber equipment, model DF-6, Selecta (Spain), together with 560 ml of water to obtain a solution of 4% H_2SO_4. This mixture was boiled for 4 h by a reflux process. Subsequently, the sample was separated by decantation, and the solid residue was washed with hot distilled water. Finally, it was dried in an oven at a temperature of 105 ±3 °C until it reached a constant weight. The entire procedure was performed in triplicate.

The following formula was applied to determine the percentage of lignin.

$$\%Lignin = \frac{Dry residue weight(g)}{Weight of original sample free of extract(g)} * 100$$

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Determination of cellulose

Initially, an extract-free sample, equivalent to 1 g, was weighed in the porous crucible and placed in the Dosi-fiber device. Twenty milliliters of ethanol and 5 ml of concentrated nitric acid were added to the sample. The mixture was boiled for 30 min using a reflux system. After this step, the solution was filtered. The resulting solid residue underwent a second digestion using 20 ml of ethanol and 5 ml of concentrated nitric acid, repeating the boiling process for 30 min under reflux conditions. The solution was decanted, and the solid residue underwent a third digestion with 100 ml of distilled water for one hour.

Subsequently, the solution was filtered, and washes were carried out with hot distilled water, followed by a wash with 100 ml of saturated solution of sodium acetate and finally, with an additional 500 ml of hot distilled water. The next step involved drying the residue in an oven at a temperature of 105 ±3 °C. Once the drying process was completed, the residue was cooled in a desiccator and weighed with an analytical balance.

The following formula was applied for its determination.

$$\%Celulose = \frac{Dryresidueweight(g)}{Weightoforiginalsamplefreeofextract(g)} * 100$$

Statistical analysis

The data on lignin and cellulose percentages were analyzed under a completely randomized design. The cocoa varieties were compared by means of an analysis of variance (Anova) with a significance level of $p \leq 0.05$. When significant differences were detected, Tukey's test was applied for mean separation, with a confidence level of 95% ($\propto = 0.05$). The statistical analysis was performed using RStudio software version 2024.09.01.

Results

The CNN-51 variety had the highest average lignin content (45.13%) and intermediate cellulose content (25.13%). On the other hand, the Super Árbol variety registered a slightly lower lignin content (42.88%) and the lowest cellulose value (20.77%). In contrast, the Nacional variety showed the lowest lignin content (38.97%), but the highest percentage of cellulose (28.57%) (Table 1).

Table 1. Average percentage of lignin and cellulose in the waste of three cocoa varieties.

Cocoa variety	Average lignin content (%)	Average cellulose content (%)
Súper Árbol	42.88	20.77
CCN-51	45.13	25.13
Nacional	38.97	28.57

When performing the statistical analyses, they did not show statistically significant differences between the percentages of lignin since the cocoa variety factor presented a *p*-value greater than 0.05 (0.1607). This result was the same for cellulose in the cocoa variety factor (Table 2).

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Table 2. Anova of the percentages of lignin and cellulose.

SV	SS	df	MS	F	*p*-value
Lignin					
Model	58.38	2	29.19	2.52	0.1607
Cocoa variety	58.38	2	29.19	2.52	0.1607
Error	69.59	6	11.59		
Total	127.93	8			
Cellulose					
Model	91.83	2	45.91	4.9	0.0547
Cocoa variety	91.83	2	45.91	4.9	0.0547
Error	56.21	6	9.37		
Total	148.03	8			

Discussion

The purpose of the study was to evaluate the chemical properties, specifically the lignin and cellulose contents, in the waste of the Nacional, CCN-51 and Super Árbol cocoa varieties, to explore its potential as an alternative raw material for kraft paper. Although numerical differences were observed in the average values of lignin and cellulose between the varieties, the experimental design used -based on replications within each variety without considering independent experimental units- did not allow us to establish conclusive statistical differences between them. In this sense, the variations reported must be interpreted descriptively, without necessarily attributing them to the effects of the variety.

According to Grzyb *et al*. (2020), factors such as cultivation practices, soil conditions, and climate can influence the lignin content in plant waste, which could be relevant in future studies with designs that consider these variables in a controlled manner. This behavior could be because this variety is highly hybrid and may respond differently to external factors, as suggested by other studies in hybrid tropical crops (Kumar and Kunhamu, 2022). In comparison, research has indicated that lower lignin content is favorable for the paper industry, as it facilitates chemical processing and improves paper quality in terms of flexibility and strength (Hawanis *et al*., 2024).

Although the Nacional variety presented a lower average percentage of lignin, this result cannot be considered statistically significant due to the limitations of the experimental design, which did not contemplate independent units per variety. Therefore, it is not possible to affirm real differences between the varieties evaluated. In kraft paper manufacturing, lignin removal is an important step in the process, as lignin negatively affects the quality and flexibility of the paper (Bajpai, 2015). In this sense, a lower lignin content in plant waste may facilitate the necessary chemical treatment but does not in itself imply a conclusive advantage without a more robust analysis.

Findings such as those by Romruen *et al*. (2022) highlight that agricultural byproducts with high lignin content may have specific applications, but their use in paper production will depend on the type of final product and the treatment applied. Although the lignin values in the Super Árbol variety showed slight variation between the samples analyzed, this apparent consistency cannot be interpreted as an indicator of compositional stability, since the experimental design was not aimed at evaluating the influence of environmental factors or the variability between crop units. The samples used by variety corresponded only to the replications necessary for laboratory analysis and do not represent a statistical assessment of chemical stability.

Studies by Rempelos *et al*. (2020); Hameed *et al*. (2020) have pointed out that certain agricultural varieties can maintain stable chemical compositions under controlled conditions; however, to establish claims about this type of cocoa waste, research with more complex experimental designs that include multiple sources of variation would be required. In the study, the Super Árbol variety

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presented the lowest average cellulose content compared to the other two varieties evaluated. Nevertheless, because the samples analyzed corresponded only to replications for technical analysis and not to independent experimental units, it is not possible to draw statistical conclusions about differences between varieties.

Therefore, any apparent trend in cellulose content should be interpreted with caution. Even so, studies such as that by Balea *et al*. (2020) have highlighted that a high cellulose content is an important factor for the strength, flexibility, and viability of paper in industrial applications. Although the Nacional variety presented the highest average of cellulose and numerical values that suggest less variability, the experimental design used does not allow us to establish differences between varieties. Therefore, from a statistical point of view, it is not possible to say that one variety is superior to another for use in the manufacture of kraft paper.

Studies such as that by Gil-Martín *et al*. (2022) have shown that some agricultural varieties can exhibit stable but insufficient chemical compositions for specific industrial applications. Nonetheless, in order to validate the applicability of cocoa waste as a raw material, it would be necessary to adopt an experimental approach that allows us to evaluate real differences between varieties and their response under different growing conditions. This suggests that, while this variety might not be ideal for kraft paper manufacturing, it could have alternative applications in less technically demanding products. Research has identified that hybrid varieties or varieties grown under varying conditions tend to show dispersion in their chemical contents, which may limit their predictability for certain applications (Mayakannan *et al*., 2023; Jagadeesan *et al*., 2023).

Conclusions

The study allowed us to explore the potential of cocoa waste as an alternative raw material in the manufacture of kraft paper, focusing on the chemical properties of lignin and cellulose in three varieties grown in the cantón of Francisco de Orellana, Ecuador. Although numerical differences were observed between the varieties analyzed, no statistically significant conclusive differences were established. Cocoa residues, both lignin and cellulose, presented chemical characteristics that position them as an alternative within sustainable use strategies; however, their effective application in the paper industry requires complementary studies with more robust designs that include multiple cultivation conditions and performance evaluation in specific industrial processes.

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