

Physicochemical Characterization Of Biochar Obtained From Coffee Husk: A Circular Economy Approach

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Abstract - This work presents the physical and chemical characterization of biochar obtained from the controlled calcination of coffee husks. Coffee husk, a residue from the pulping process in coffee production, was dried, pulverized, and sieved to obtain a fine powder. Thermal analysis (TGA-DTA-DSC) was performed under nitrogen and air atmospheres to determine the material's thermal decomposition and calcination temperatures. X-ray fluorescence (XRF) revealed the elemental composition, showing the presence of C, O, K, S, Ca, P, and Si. X-ray diffraction (XRD) analysis demonstrated significant structural changes due to calcination. The untreated coffee husk showed an amorphous structure with peaks corresponding to cellulose and hemicellulose. After calcination at 550°C, these organic phases disappeared, and new crystalline phases, including CaO, SiO₂, graphite, and Fe₂O₃, were identified. The formation of these phases suggests improved structural stability and potential benefits for soil applications. Decomposition stages were observed, with residue percentages of 7.6% in air and 25.8% in nitrogen at 1000°C. Based on these results, calcination at four temperatures (160°C, 330°C, 430°C, and 550°C) was selected, followed by analysis using infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Structural changes, including the disappearance of C=O bands and increased intensity of C-O bonds, indicated the potential to produce biochar at temperatures above 330°C. These findings suggest that coffee husk biochar could improve soil nutrient bioavailability, supporting its use in a circular economy framework.

Keywords: Biochar, Coffee husk, Calcination, Thermal analysis, XRF, XDR, FTIR, SEM, Circular economy.

1. Introduction

The coffee industry generates significant amounts of agroindustrial waste, with coffee husk being one of the main byproducts. In the Cauca region of Colombia, where coffee production is a key economic activity, the pulping process used to extract the coffee bean leaves behind large quantities of husk. Traditionally, this waste is either discarded or used in low-value applications, which poses both environmental and economic challenges. However, recent interest in sustainable practices and circular economy models has sparked research into alternative uses for coffee husk, including its transformation into biochar through controlled calcination processes.

Biochar is a carbon-rich material produced by pyrolysis or calcination of biomass under controlled conditions [1]. Due to its high surface area and porous structure, biochar has gained attention for applications in soil amendment, carbon sequestration, and environmental remediation [1,2]. The use of biochar from coffee husk not only provides a way to manage agricultural waste but also contributes to the development of sustainable agricultural practices by improving soil fertility and nutrient bioavailability [3].

This work aims to perform a detailed physicochemical characterization of biochar obtained from coffee husk calcination. Thermal analysis, including thermogravimetric (TGA), differential thermal (DTA), and differential scanning calorimetry (DSC) methods, was used to determine the thermal decomposition stages and identify optimal calcination temperatures. X-ray fluorescence (XRF) was employed to determine the elemental composition of the biochar, while Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were utilized to analyze the chemical structure and surface morphology of the calcined samples. By investigating the effects of different calcination temperatures on the properties of the biochar, this study explores its potential applications in soil enrichment and its relevance within the framework of the circular economy.

The results of this research are expected to provide insights into the valorization of coffee husk, turning a low-value waste material into a high-value product with potential agricultural and environmental benefits.

2. Results

The increasing global demand for sustainable agricultural practices requires the exploration of innovative solutions that align with circular economy principles [4]. One such approach is the conversion of agricultural waste into biochar, a carbon-rich material that can improve soil health and nutrient availability. In Colombia, where coffee production is a major agricultural activity, coffee husk—a byproduct obtained in the pulping process during coffee production in the Cauca region of Colombia—presents a significant waste management challenge [5]. This study explores the potential of transforming coffee husk into biochar through a controlled calcination process, thereby providing a sustainable solution to agricultural waste while enhancing soil health.

In this work, the physicochemical characterization of biochar derived from the controlled calcination of coffee husk is presented. The coffee husk was sun-dried for three days, ground into a fine powder, and sieved using a 300 μm mesh. The husk powder thermal stability was analyzed using simultaneous thermal analysis (TGA-DTA-DSC) under nitrogen and air atmospheres to determine the decomposition profile and appropriate calcination temperatures. The chemical composition was assessed via X-ray fluorescence (XRF).

The thermal analysis revealed distinct decomposition stages under air and nitrogen atmospheres. In air, the coffee husk decomposed in three stages, with significant weight loss occurring at temperatures corresponding to the oxidation of organic matter. Results shown in Figure 1 indicate that under nitrogen, the decomposition was less pronounced, with overlapping stages indicating a more complex thermal behavior. The residual mass after calcination at 1000°C was significantly higher in nitrogen, suggesting incomplete combustion. Exothermic peaks in the DTA and DSC thermograms indicated these decomposition stages. At 1000°C, the residual mass was 7.6% in air and 25.8% in nitrogen. Based on these findings, four calcination temperatures were selected: 160°C, 330°C, 430°C, and 550°C.

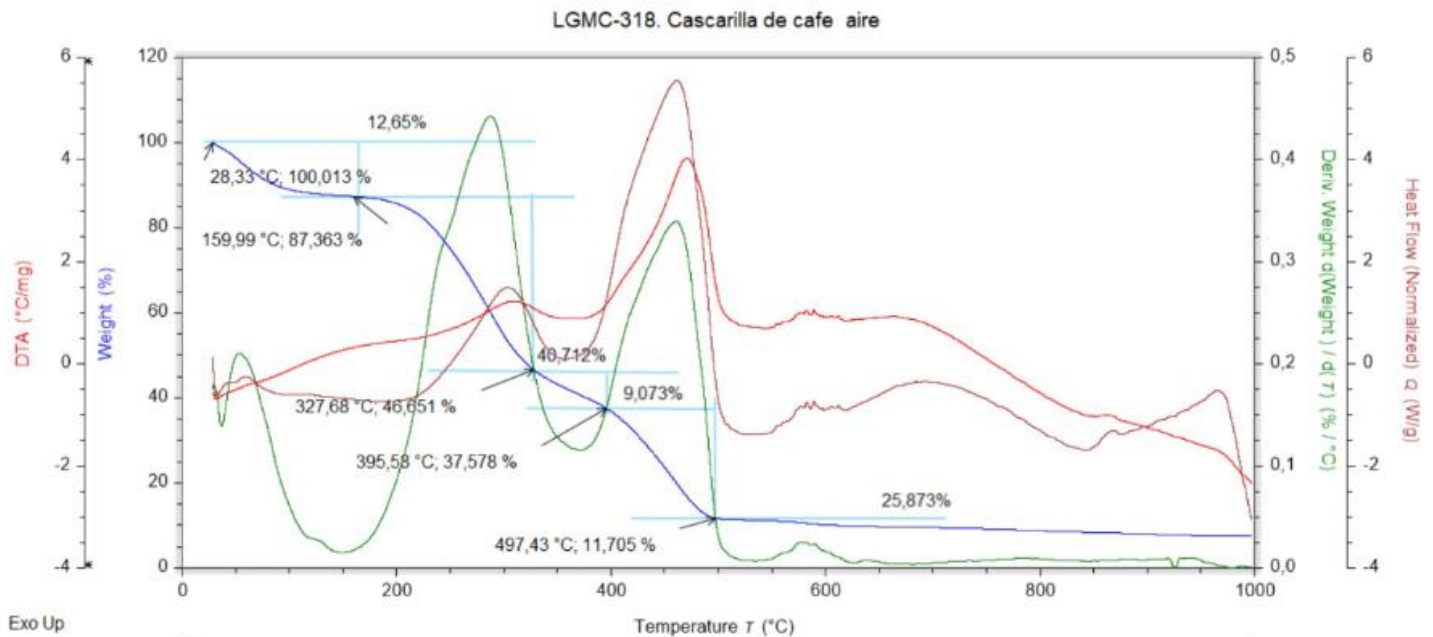


Figure 1: TGA-DTA-DSC Thermograms: Illustrating the thermal decomposition stages of coffee husk under air.

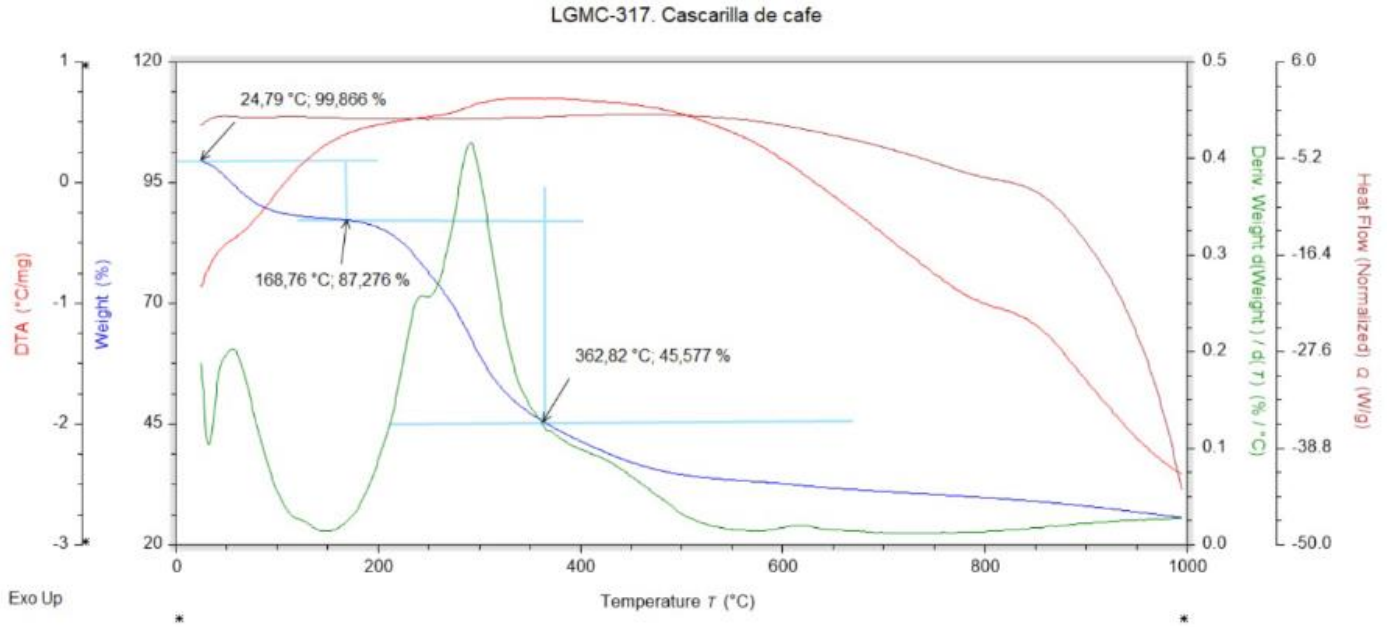


Figure 2: TGA-DTA-DSC Thermograms: Illustrating the thermal decomposition stages of coffee husk under nitrogen.

XRF results (shown in Table 1) identified key elements in the husk, including C, O, K, S, Ca, P, and Si. Infrared spectroscopy (FTIR) showed the disappearance of the C=O bond vibration band and an increase in the C-O stretching band intensity as the calcination temperature increased, suggesting the formation of biochar at temperatures above 330°C. Additionally, FTIR, shown in Figure 3, revealed the presence of CaO, Si-O, and P-O-P bonds, indicating that the biochar could enhance soil nutrient availability. Shows the results of X-ray fluorescence analysis (XRF) of a coffee husk sample. The results are presented in the form of oxides and in units of kilograms per kilogram (Kg/Kg), which is equivalent to percentage by weight.

Table 1: Oxide content and ignition losses

Oxide	kg/kg
Ignition losses at 1000°C for 1 hour	89.321
K ₂ O	6.71
S	1.10
CaO	0.98
P ₂ O ₅	0.87
SiO ₂	0.29
MgO	0.24
Cl	0.23
Al ₂ O ₃	0.11
Fe ₂ O ₃	0.09
SrO	0.04
MnO	0.02
Rb ₂ O	0.01
Na ₂ O	0.01

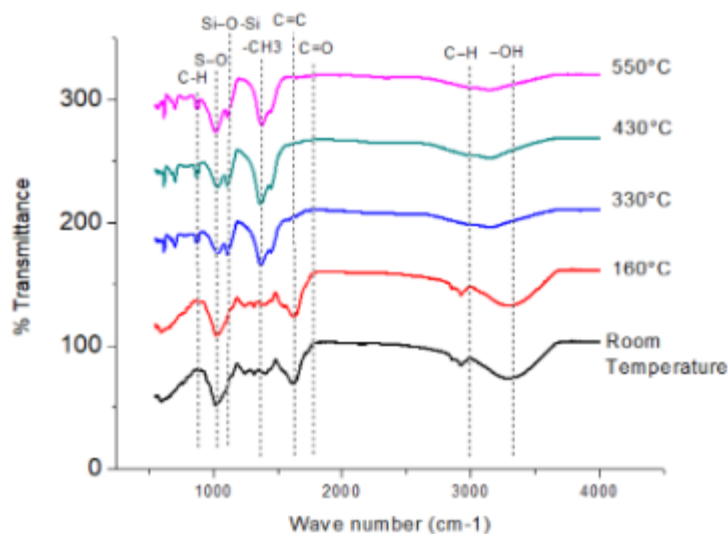


Figure 3: FTIR: pulverized coffee husk with temperature variation ambient temperature, 160°C, 330°C, 430°C, and 550°C.

SEM images are shown in Figure 4. It is revealed a porous structure, which is advantageous for soil aeration and water retention.

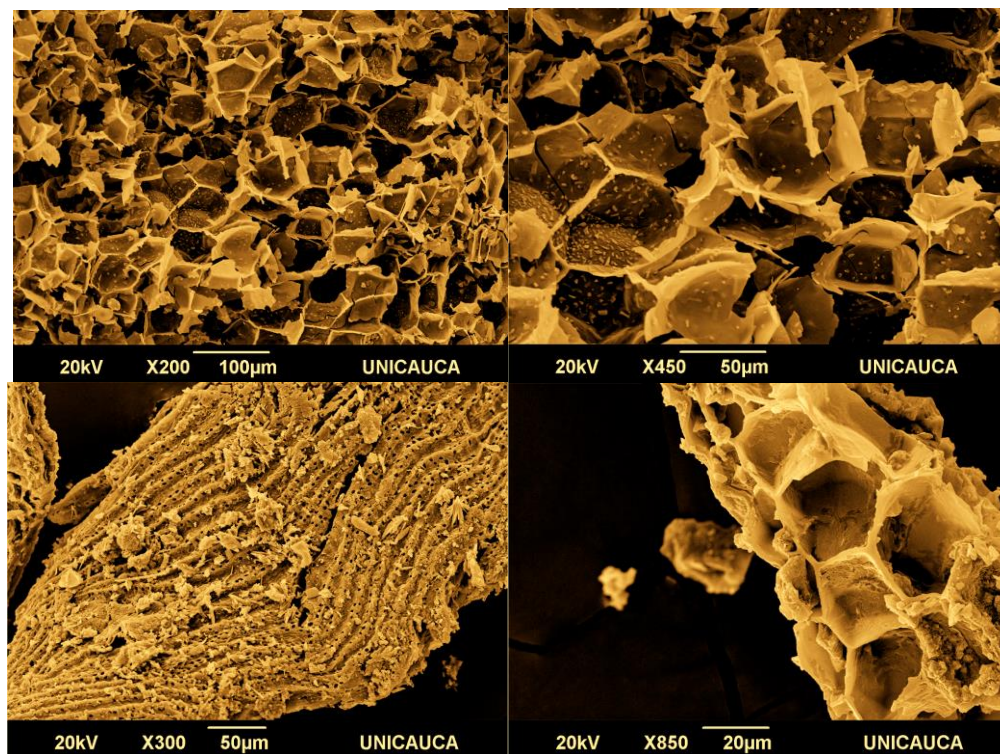


Figure 4: SEM Images: Depicting the morphological changes in biochar with varying calcination temperatures.

Finally, To further investigate the structural transformations during calcination, X-ray diffraction (XRD) analysis was performed on the untreated coffee husk and biochar obtained at 550°C. The diffractograms are presented in Figure 5, which illustrates the differences in crystalline phases before and after thermal treatment.

XRD of Untreated Coffee Husk: The untreated husk exhibited broad diffraction peaks, characteristic of an amorphous structure mainly composed of organic compounds like cellulose and hemicellulose. The primary peaks were observed at 2θ

$\approx 9^\circ$, corresponding to hemicellulose, and $2\theta \approx 22^\circ$ and 34° , associated with the crystalline regions of cellulose. These peaks confirm the dominance of biopolymeric structures in the raw material.

XRD of Biochar at 550°C : After calcination, the XRD pattern showed a significant reduction in amorphous organic content, with the disappearance of cellulose and hemicellulose peaks. Instead, new well-defined peaks emerged, corresponding to CaO (calcium oxide), SiO_2 (quartz), graphite, and Fe_2O_3 (hematite). The presence of P_2O_5 (phosphorus pentoxide) was also detected, which is relevant for soil fertility applications. These results suggest that the thermal treatment leads to the formation of stable inorganic phases that can contribute to the biochar's functionality as a soil amendment.

The formation of crystalline CaO is particularly important, as it can enhance soil pH and nutrient availability, while graphite-like structures indicate increased carbon stability, which is beneficial for long-term carbon sequestration. The comparison between untreated husk and biochar at 550°C is shown in Figure X, highlighting the transformation from an amorphous to a more crystalline structure.

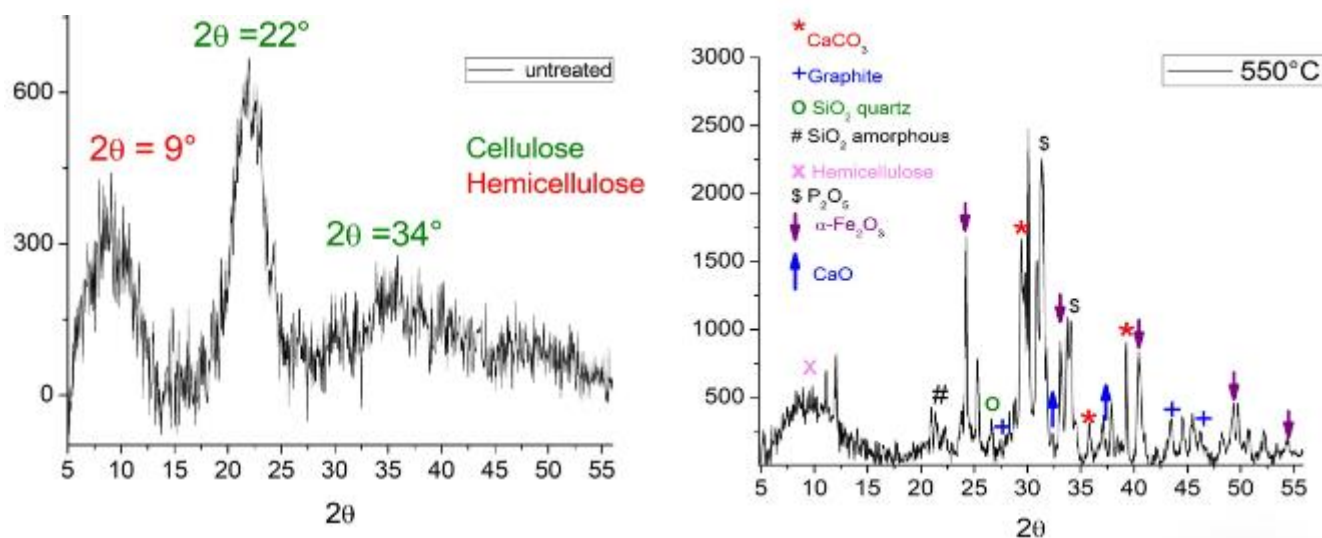


Figure 5: X-ray diffraction (XRD) patterns of untreated coffee husk (red) and biochar obtained at 550°C (blue). The untreated sample shows broad peaks corresponding to cellulose and hemicellulose, while the biochar exhibits crystalline phases associated with CaO, SiO_2 , graphite, and Fe_2O_3 .

This study highlights the potential of converting agricultural waste, such as coffee husk, into valuable biochar, contributing to sustainable agriculture within a circular economy framework and addressing global challenges related to food security and environmental sustainability. The biochar produced at higher calcination temperatures exhibited properties that could improve soil health, such as enhanced nutrient availability and water retention.

4. Conclusion

This study has demonstrated the potential of transforming coffee husk, an abundant agroindustrial waste, into biochar through a controlled calcination process. The thermal analysis revealed key decomposition stages, and the elemental composition analysis identified essential nutrients that could enhance soil health. The biochar produced at higher calcination temperatures (above 330°C) exhibited improved structural and chemical properties, including enhanced C-O bonding, which suggests its effectiveness in increasing nutrient bioavailability and water retention in soils. The findings support the use of coffee husk biochar in sustainable agricultural practices within the circular economy framework, providing both environmental benefits and economic value by repurposing waste into a useful product.

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