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Analysis of the thermodegradation of sugar cane residues for their revalorization in the manufacture of charcoal: Comparative study between varieties CP-72-2086, MEX-69-290 and sugar cane bagasse from a sugar mill

Análisis de la termodegradación de residuos de caña de azúcar para su revalorización en la fabricación de carbón vegetal: Estudio comparativo entre las variedades CP-72-2086, MEX-69-290 y bagazo de caña de azúcar procedente de un ingenio azucarero

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Abstract

In this study, the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of sugarcane varieties CP-72-2086, MEX-69-290, and industrial sugarcane bagasse (BICA) were compared to predict carbonization yield and hemicellulose/cellulose content. Results showed 50% yield temperatures of 400 °C (MEX-69-290), 409 °C (CP-72-2086), and 501.9 °C (BICA). DTG deconvolution revealed cellulose presence at 400-510 °C for CP-72-2086 and MEX-69-290, versus 325-410 °C for BICA. BET analysis of BICA carbons carbonized at 375 °C and 750 °C confirmed a temperature-dependent textural decline, with specific surface area decreasing by 51.3% (from 83.120 to 40.477 m^2 g^{-1}) and pore volume reducing by 47.95% (from 0.171 to 0.089 cm^3 g^{-1}). This behavior agrees with the TGA/DTG profiles of BICA, where cellulose is almost completely degraded around 410 °C, leading to a collapse of the porous structure at higher carbonization temperatures. In contrast, the field residues CP-72-2086 and MEX-69-290 retain a larger cellulose fraction at 410 °C, which helps preserve charcoal porosity and supports variety-specific carbonization strategies for optimized charcoal production.

Keywords: Industrial sugarcane bagasse, sugarcane varieties CP-72-2086 and MEX-69-290, carbonization, thermodegradation, BET analysis.

Resumen

En este estudio se compararon las curvas termogravimétricas (TG) y termogravimétricas derivadas (DTG) de las variedades CP-72-2086, MEX-69-290 y bagazo industrial de caña de azúcar (BICA) para predecir rendimiento de carbonización y contenido de hemicelulosa/celulosa. Los resultados mostraron temperaturas para 50% de rendimiento de 400 °C (MEX-69-290), 409 °C (CP-72-2086) y 501.9 °C (BICA). La deconvolución DTG reveló celulosa en 400-510 °C para CP-72-2086 y MEX-69-290, versus 325-410 °C en BICA. El análisis BET de los carbones de BICA carbonizados a 375 °C y 750 °C confirmó un deterioro textural dependiente de la temperatura, con una disminución del área superficial específica del 51.3% (de 83.120 a 40.477 m^2 g^{-1}) y una reducción del volumen de poro del 47.95% (de 0.171 a 0.089 cm^3 g^{-1}). Este comportamiento coincide con los perfiles TGA/DTG de BICA, donde la celulosa está casi completamente degradada alrededor de 410 °C, lo que conduce al colapso de la estructura porosa a temperaturas de carbonización más altas. En contraste, los residuos de campo CP-72-2086 y MEX-69-290 retienen una fracción mayor de celulosa a 410 °C, lo que ayuda a preservar la porosidad del carbón vegetal y respalda estrategias de carbonización específicas por variedad para una producción de carbón optimizada.

Palabras clave: Bagazo industrial de caña de azúcar, variedades de caña de azúcar CP-72-2086 y MEX-69-290, carbonización, termodegradación, análisis BET.

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1 Introduction

Mexico is one of the world's leading sugarcane producers, cultivating a diverse range of varieties adapted to the country's specific geographic, climatic, and agronomic conditions (Mexican Sugar Manual [MAM], 2024). During the 2022-2023 harvest, 111 sugarcane varieties were cultivated, resulting in an estimated production of approximately 46.5 million tons processed across 49 sugar mills. Among these, varieties CP-72-2086 and MEX-69-290 stand out due to their prevalence and distinctive agronomic characteristics. These traits contribute significantly to their adaptability and high yield performance across multiple production regions (CONADESUCA, 2016). Optimizing the utilization of these crops requires not only increasing sugar production but also developing sustainable strategies to valorize residues such as bagasse. Figure 1 presents the twelve most widely cultivated sugarcane varieties in Mexico.

The production of the CP-72-2086 and MEX-69-290 sugarcane varieties reached approximately 24,079,146.67 metric tons. Harvesting was split equally between mechanical and manual methods, with at least 5% of the total attributable to residual crop residues. This translates to an estimated 1,203,957.33 tons of residues (MAM, 2024, pp. 64-394). The CP-72-2086 variety is characterized by an erect growth habit with limited natural defoliation and an intermediate flowering cycle. It accounts for roughly 36% of Mexico's total cultivated sugarcane area, known for high biomass and sugar yields, underscoring its significance in the national sugar industry (Rea *et al*., 1994).

The MEX-69-290 variety can reach yields of up to 200 tons per hectare under optimal management and demonstrates adaptability across diverse terrains and environments (CONADESUCA, 2016). In a comparative study across twelve varieties, CP-72-2086 achieved 136 t ha^{-1}, second only to B-86-326 with 162 t ha^{-1}. However, CP-72-2086 produced a higher sugar yield (15.39 t ha^{-1}) than B-86-326 (11.37 t ha^{-1}) (Arreola-Enríquez *et al*., 2019). Additionally, CP-72-2086 showed superior salinity tolerance compared to varieties such as MEX-69-290 (Castañeda-Castro *et al*., 2014; Castañeda-Castro *et al*., 2021).

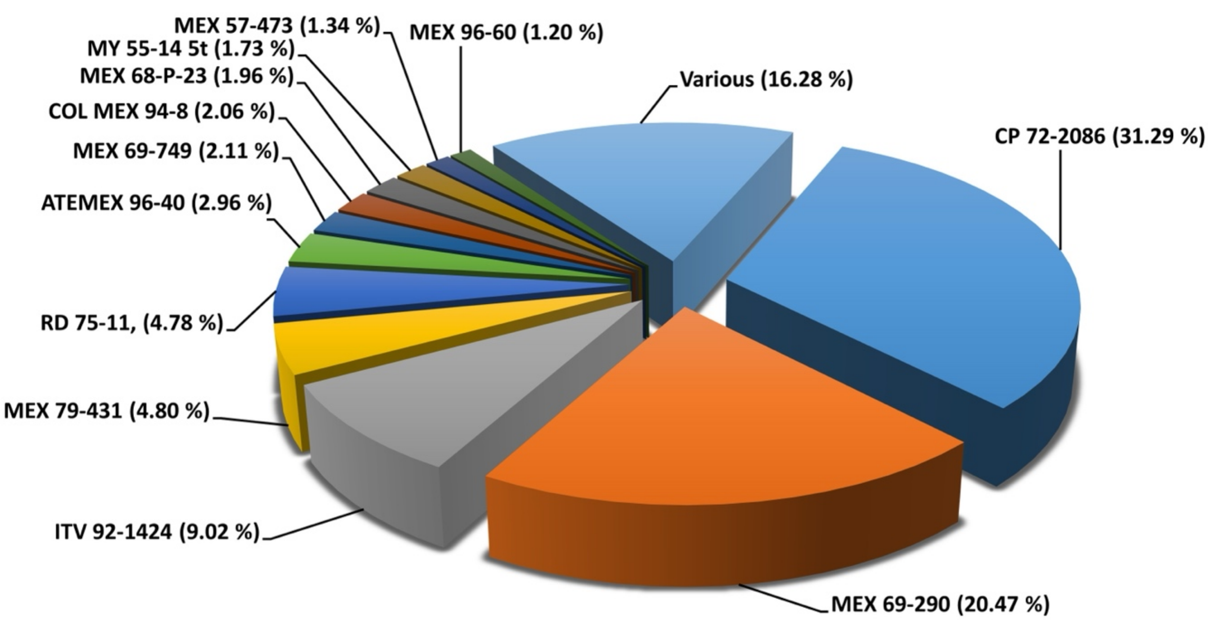

Figure 1. Use of sugarcane varieties in Mexico. Harvest 2022-2023 (MAM, 2024, pp. 64-394).

Sugarcane bagasse, an abundant by-product of the sugar industry, has traditionally been considered a raw material for energy generation or composting. However, controlled thermodegradation and carbonization of bagasse and derived lignocellulosic residues have growing industrial potential for obtaining charcoal with specific characteristics and uses (Skoczko and Gumiski, 2022; Arreola-Enríquez *et al*., 2019). Sugarcane bagasse contains on average: 38.4 to 41.3% cellulose, 23.2 to 31.3% hemicellulose, 14.9 to 25% lignin, 9.43 to 13.9% moisture, and 1.24 to 2.76% ash (Jacome *et al*., 2023, Skoczko and Gumiski, 2022).

Cellulose is a linear homopolysaccharide composed solely of glucose units, while hemicellulose is a heterogeneous branched polysaccharide made up of various sugars including xylose, mannose, galactose, arabinose, and glucose. Hemicellulose exists in two forms: water-soluble hemicellulose I, mainly xylans found in the primary cell wall, and water-insoluble hemicellulose II, primarily mannans and galactans present in the secondary cell wall (Peng *et al*., 2009).

Lignin in sugarcane is an essential component for the structure and stability of the material, with more resistant and stable thermal behavior in industrial waste such as bagasse, due to the prior extraction of juice that removes the less stable soluble lignin. This directly affects thermal reactivity and behavior in pyrolysis or combustion processes (Martínez *et al*., 2023).

Thermogravimetry (TGA) and differential thermogravimetry (DTG) are key analytical tools for characterizing the thermal behavior of lignocellulosic materials and predicting the content and quality of components such as cellulose, hemicellulose, and lignin, which are essential for determining the efficiency of carbonization and/or recovery processes, as well as in absorption and purification processes (Nunes *et al*., 2020, Parmar *et al*., 2024, Guevara *et al*. 2021, Almeida *et al*., 2019). The clear identification of the maximum degradation rates of each compound provides fundamental information to optimize industrial carbonization and activated carbon manufacturing processes, allowing the adjustment of temperature and time parameters to maximize the quality and yield of the carbon produced.

In the typical behavior of lignocellulosic biomass, three main thermal stages can be observed, which correspond to the degradation of hemicellulose, cellulose, and lignin. Hemicellulose decomposes at a relatively low temperature range, approximately between 200 and 350 °C, which is evident in the DTG curve as a defined peak towards lower temperatures. Subsequently, cellulose undergoes more pronounced thermal decomposition in the range of 320 to 400 °C, generating an intense and narrow DTG peak that reflects its homogeneous polymeric nature and relatively rapid degradation. Finally, lignin exhibits differentiated thermal behavior, degrading more gradually and extensively over a wide temperature range that can range from 200 to approximately 650 °C. This prolonged and complex process manifests itself in peaks of lower intensity and greater amplitude in the DTG curve, corresponding to the variety of chemical bonds present in its structure. Due to its structural diversity and superior thermal stability, lignin contributes to mass loss at higher temperatures compared to hemicellulose and cellulose (Díez, *et al*., 2020, Manals *et al*., 2011).

Several studies have evaluated the thermochemical potential of different lignocellulosic materials such as industrial sugarcane bagasse, sugarcane waste, coffee husk waste and tobacco waste, demonstrating that the behavior of the general thermodegradation curves is the same, but the specific thermal degradation properties depend largely on the type of material, its chemical composition and the processing conditions (Nunes *et al*., 2020; Manals *et al*., 2011; Parmar *et al*., 2024).

The study by Manals (2011) shows, based on TGA and DTG curves, that tobacco waste has the lowest content of volatile compounds, followed in order by coffee husks, sugarcane bagasse, and pine sawdust; which may explain the lower level of decomposition of tobacco waste and coffee husks compared to pine sawdust and sugarcane bagasse. In addition, the biomass that showed the highest degree of decomposition was sugarcane bagasse, which suggests a lower percentage of lignin and a higher percentage of cellulose and hemicellulose.

In the study conducted by Najafi *et al*. (2024), the thermal degradation of lignocellulose residues from the sugarcane variety CP-69-1062, including straw and bagasse, was meticulously analyzed using thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG). The findings revealed that hemicellulose exhibits two distinct degradation stages (Hemicellulose I and II), occurring within thermal ranges of approximately 150–275 °C and 275–310 °C, respectively. Cellulose displayed a predominant decomposition peak between 320 and 375 °C, while lignin underwent a slower and more protracted degradation between 400 and 490 °C, indicative of its higher thermal stability. Compared to straw, bagasse exhibited hemicellulose degradation peaks within similar temperature intervals; however, the principal cellulose degradation peak shifted slightly towards lower temperatures (320–360 °C), and lignin degraded within a narrower and lower temperature range (370–440 °C), suggesting reduced thermal stability of this component in bagasse. These results underscore significant compositional and thermal behavior differences between the two residues.

A review of the literature revealed a lack of studies directly comparing the specific degradation properties of the different commercial varieties cultivated in México with the residual bagasse processed in sugar mills. This study addresses this issue through a comparative analysis of the TGA and DTG curves of varieties CP-72-2086 and MEX-69-290, together with industrial bagasse from a sugar mill in Campeche, Mexico. The objective of this study is to evaluate the feasibility of recovering waste from these varieties of sugarcane for use in the production of charcoal for industrial applications.

2 Materials and methods

2.1 Materials used

Two sugarcane stalks were obtained from the varieties CP-72-2086 (CP) and MEX-69-290 (MEX), respectively. CP had a mass of 1.54 kg, a length of 2.10 meters, and an average diameter of 37.34 mm, with 23 canals. MEX had a mass of 2.64 kg, a length of 3.44 meters, and 28 canals. The geographical coordinates where they were located were: CP (Latitude 19° 29' 49.6536", Longitude -90° 32' 33.2982"), and MEX (Latitude 19° 30' 50.5", Longitude -90° 32' 46.4"); located within the experimental fields of the Postgraduate College, Campeche Campus. The sugarcane stalks CP-72-2086 and MEX-69-290 were fed into a manual cane chopper. Subsequently, 1 kg of chopped stalks was fed into a semi-industrial blender with 1 L of water to make smaller cuts of the chopped stalks. The blended mixture was left to stand for two days and then placed in a convection oven at 70°C for 48 hours to dry at room temperature. Water loss after grinding and drying in the sun and after being in a convection oven was 75.089% for CP, 64.246% for MEX, and 61.5% for BICA. The resulting particles were sieved in a U.S. 40 mesh sieve to ensure a particle size of less than 425 μm, which were then packaged in 100 mL COPROPACK^{MR} sterilized sample cups.

2.2 Experimental characterization techniques

2.2.1 Analysis BET

The porous texture of the materials was characterized by N_2 adsorption at -196.2 °C using a Micromeritics ASAP 2010 analyzer. Prior to analysis, samples were degassed at 80 °C for 72 h under vacuum (10^{-3} mmHg). Specific surface area was determined from the N_2 adsorption isotherms via the BET equation. Pore volume (V_{N_2}) was calculated by applying the Dubinin–Radushkevich (DR) equation to the N_2 adsorption isotherms. Microporous surface area was derived from the equation: S_{mic} (m^2 g^{-1}) = 2000 V_{N_2} (cm^3 g^{-1}) / L_0 (nm), where L_0 denotes the average micropore width (Pacheco-Catalán *et al.*, 2011).

2.2.2 Thermogravimetric analysis

A Perkin Elmer thermogravimetric analyzer, model TGA-8000, was used at a temperature range of 50 °C to 750 °C and a heating rate of 10 °C min^{-1} in a nitrogen atmosphere (20 mL min^{-1}) and 30 mL min^{-1} for purge gas flow. Alumina crucibles approximately 7.2 mm in diameter and 8.6 mm in height (250 μL) were used to contain the sample, whose mass was in the order of 5 mg. Baseline correction was performed automatically using the equipment software, which uses a fourth-order polynomial fit in an empty crucible. The tests were performed in triplicate for the samples of varieties CP-72-2086 and MEX-69-290. For industrial sugarcane bagasse (BICA), two replicates were considered sufficient to ensure repeatability, so a third test was not necessary. For each variety, graphs were prepared using Origin^{MR} software based on the data obtained with the thermogravimetric analyzer, and an interpolation curve was constructed using the cubic B-spline method. From the TGA curve, the DTG curve (derivative of the TGA) was obtained using Origin^{MR} software, facilitating the precise identification of temperatures at which decomposition processes or significant thermal changes occur. Subsequently, single multi-peak deconvolution was performed on the experimental curves. The fitting was carried out using Gaussian functions, which assume symmetrical bell-shaped profiles for each peak. Baseline correction was applied beforehand to isolate the peaks of interest, and the number and initial positions of the peaks were manually defined within the analysis range. Deconvolution parameters included Gaussian peak functions (n = 4 peaks) with Levenberg-Marquardt optimization and convergence criteria of $R^2 \geq 0.80$ and reduced $\chi^2 < 0.01$, to achieve an adequate correspondence between the experimental data and the sum of the adjusted peaks, corresponding, in ascending order, to the degradation stages of hemicellulose I, hemicellulose II, cellulose and lignin, in accordance with previous reports on the thermal decomposition of lignocellulosic biomass. (Díez, *et al.*, 2020; Manals *et al.*, 2011; Nunes *et al.*, 2020; Parmar *et al.*, 2024).

3 Results and discussion

The thermal decomposition profile of industrial sugarcane bagasse (BICA) is presented in Figure 2, together with its derivative (DTG) and the deconvolution of the DTG, which is essential for interpretation. The solid black line represents the

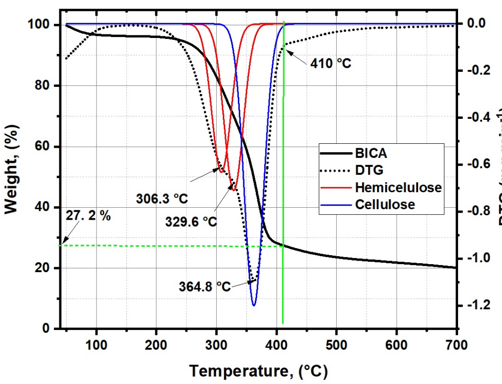

Figure 2. TGA, DTG and deconvolution curves of BICA.

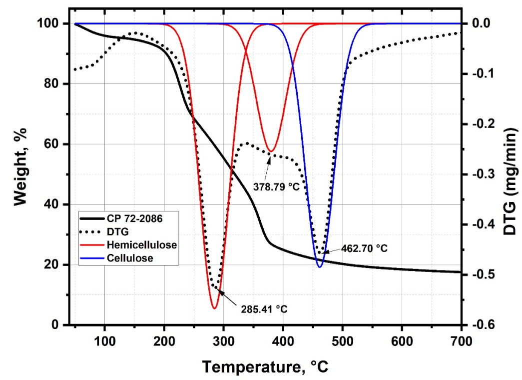

Figure 3. TGA, DTG and deconvolution curves of CP 72-2086.

thermal degradation of BICA, while dotted line represents the DTG of BICA. Inflection points on the TGA curve are indicated at 306.3 °C, 329.6 °C, and 364.8 °C, corresponding to the points of maximum degradation rate for hemicelluloses I and II, as well as cellulose, respectively, as has been evidenced in other lignocellulose materials (Solís-Fuentes *et al*., 2012; Varhegyi *et al*., 1989). Unlike previous reports by Solís-Fuentes *et al*. (2012), no concave region was observed above 550 °C, which has been attributed to lignin decomposition. Instead, lignin exhibited a nearly horizontal behavior, consistent with the findings of Varhegyi *et al*. (1989) and represented by the blue curve in the range of 425–700 °C. The temperature of 410 °C was selected as the carbonization reference point based on the DTG deconvolution analysis of BICA (Figure 2), which shows the complete degradation of hemicelluloses I and II, as well as cellulose, at this temperature. A vertical green bar at approximately 410 °C on the TGA curve marks the beginning of the lignin degradation shoulder, where the residual mass stabilizes at ~27%, primarily representing the lignin content that contributes to carbon formation. Degradation curves for other lignocellulosic materials, from which carbon yield can be deduced, confirm that the result obtained for BICA falls within the estimated carbon yield range for this type of material, which is between 20 and 35% (Diez *et al*., 2020, Manals *et al*., 2011). This selection optimizes carbon yield by removing volatiles and preserving thermally stable lignin.

Figure 3 shows the degradation curve of the CP-72-2086 sugarcane variety with the points identified at 285.4 °C, 378.7 °C and 462.7 °C, which represent the points of maximum degradation rate of hemicelluloses I and II, as well as cellulose, respectively (Solís-Fuentes *et al*., 2012; Varhegyi *et al*., 1989). This can be validated by means of the deconvolution carried out and where the red colored curves are shown, which coincide in the points already indicated. Like BICA, there is no reference in this TGA curve, as well as in the DTG deconvolution that indicates the presence of lignin in a significant way, however, its presence can be validated through the horizontal behavior that starts at the maximum point at 565 °C, which is the temperature reported by Solís-Fuentes *et al*. (2012). The same figure shows a blue curve, whose behavior is horizontal between 425 °C and 700 °C, which is the range where lignin is found and its degradation.

Figure 4 shows the TGA and DTG curves of the MEX-69-290 sugarcane residue. At the beginning of the test, the sample exhibits a slight mass loss associated with moisture removal, followed by two well-defined degradation events whose DTG maxima occur at 222.1 °C and 290.9 °C, attributed to the first and second hemicelluloses, respectively. A third, sharper DTG peak is observed at 355.9 °C, corresponding to the maximum degradation rate of cellulose, after which the TGA curve continues to decline more gradually up to about 400 °C, where the remaining mass is close to 28%. Beyond this point, the curve shows a progressive decrease in slope up to 700 °C, indicating the slow and extended degradation of lignin and the formation of the final carbonaceous residue.

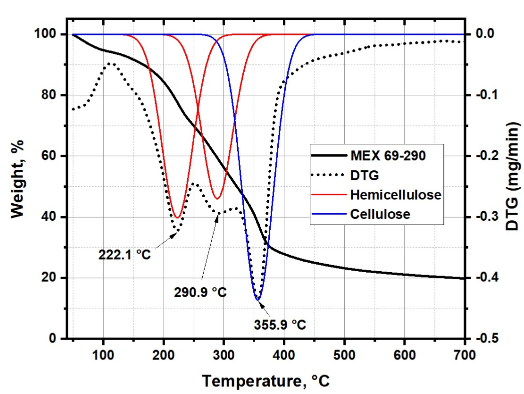

Figure 4. TGA, DTG and deconvolution curves of MEX 69-290.

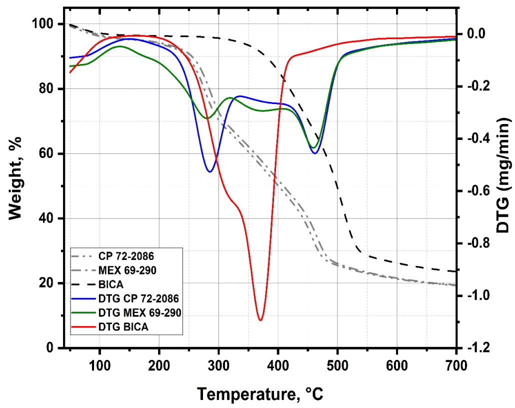

Figure 5. Comparison of TGA and DTG curves.

Figure 5 shows a plot of the TGA and DTG curves of the sugarcane varieties CP-72-2086, MEX-69-290 and BICA. A vertical green line at 375 °C is also shown. When comparing the performances of each of the varieties, it can be noted that they would practically have a performance of approximately 76% if they were to be carbonized in an inert atmosphere; almost totally eliminating hemicelluloses I and 50% of hemicelluloses II for MEX-69-290 and CP-72-2086. At that temperature, cellulose would be present in both varieties without beginning its degradation; in the case of BICA, at 375 °C hemicelluloses I and II and 50% of the cellulose would already be degraded. In all three cases, lignin would be present without any degradation.

In addition to the thermogravimetric analysis used, it is important to consider thermal kinetics studies, which provide additional parameters for understanding the degradation of hemicellulose, cellulose, and lignin. Recent research has applied advanced isoconversional methods to determine activation energy and pre-exponential factors, enabling robust kinetic modeling of the pyrolysis of bagasse and its varieties (Bahú et al., 2022; Najafi *et al.*, 2024). These techniques confirm that the thermal degradation of hemicellulose occurs at lower temperatures with lower activation energy, while cellulose and lignin require higher temperatures and energies, consistent with the present interpretation of the TGA and DTG curves.

According to all the results obtained, the residues of varieties CP-72-2086 and MEX-69-290 showed similar thermal profiles in terms of cellulose retention when carbonized at temperatures around 410 °C, maintaining this polysaccharide and thus potentially improving the quality of the resulting charcoal by preserving a more resistant carbon structure. In contrast, industrial sugarcane bagasse (BICA) lost significant cellulose at that temperature, which could result in charcoal with different physical properties, possibly with lower mechanical strength or different

Table 1. BET surface area characteristics of BICA carbons carbonized at 375 °C and 750 °C.

Parameter	BICA-375 °C	BICA-750 °C
Surface area (m^2 g^{-1})	83.120	40.477
Pore volume (cm^3 g^{-1})	0.171	0.089
Pore diameter (nm)	3.666	3.903

porosity. This different behavior can be explained by variations in the intrinsic chemical and structural composition of the fibers depending on the variety, as well as by the processing to which industrial bagasse is subjected, which could affect the arrangement and thermal stability of its lignocellulose components.

Carbonization of industrial sugarcane bagasse (BICA) was performed at 375 °C and 750 °C, with the samples characterized via BET analysis. Both carbons exhibited similar pore sizes, with average diameters of 3.666 nm and 3.903 nm, respectively. However, the specific surface area decreased by 51.3%, from 83.120 m^2 g^{-1} to 40.477 m^2 g^{-1}, and the pore volume reduced by 47.95%, from 0.171 cm^3 g^{-1} to 0.089 cm^3 g^{-1} as the carbonization temperature increased. This decline is attributed to the progressive degradation of cellulose and lignin, which correlates with the TGA/DTG profiles of BICA, where cellulose is nearly absent at 410 °C, thereby impacting the porous structure of the resulting charcoal.

These findings are relevant to the sugar and bioprocessing industries, as they highlight the importance of selecting or segmenting waste according to variety for thermal recovery processes. For example, CP-72-2086 and MEX-69-290 residues could be preferable for applications requiring specific coal characteristics, such as high strength or controlled porosity, while industrial bagasse could be used in other applications with lower structural requirements or combined with other materials to compensate for cellulose losses.

The results are consistent with previous reports on the thermal stability of lignin and the differential degradation of hemicellulose and cellulose in lignocellulose biomass (Najafi *et al.*, 2024; Martínez *et al.*, 2023; Varhegyi *et al.*, 1989). Furthermore, the significant presence of lignin, which degrades at higher temperatures, suggests that carbonization could take advantage of this phase to generate carbonaceous residues with good thermal and chemical stability, useful in the manufacture of activated carbons for adsorption and energy storage (Mubarak *et al.*, 2024).

Conclusions

The thermal analysis curves (TGA and DTG) clearly identified the degradation stages of the main components of bagasse, such as initial moisture, hemicellulose, cellulose, and lignin. It was observed that mass loss occurs in distinct stages, showing maximum degradation peaks related to hemicellulose and cellulose, while lignin exhibits more gradual and persistent degradation at high temperatures. These thermal profiles allow the behavior of bagasse during carbonization processes to be predicted, with potential application in the manufacture of activated carbons or derived materials. In addition, the study highlights the importance of the particular characteristics of each variety for the optimization of industrial processes based on bagasse. In addition, the study highlights the importance of the particular characteristics of each variety for the optimization of industrial processes based on bagasse.

The BET analysis of BICA carbons carbonized at 375 °C and 750 °C revealed a significant temperature-dependent decline in textural properties, with specific surface area decreasing by 51.3% (from 83.120 to 40.477 m^2 g^{-1}) and pore volume reducing by 47.95% (from 0.171 to 0.089 cc g^{-1}), while maintaining similar pore diameters (~3.7-3.9 nm). This confirms the TGA/DTG findings, demonstrating that near-complete cellulose loss at 410 °C leads to collapse of the porous structure at higher carbonization temperatures, limiting the quality of the resulting charcoal for applications requiring high surface area. These results validate variety-specific carbonization strategies, positioning residues from the CP-72-2086 and MEX-69-290 varieties as superior raw materials due to their better retention of cellulose-derived porosity at carbonization temperatures around 375-410 °C as compared to industrial BICA.

In summary, this work contributes to understanding how the thermal and textural properties of sugarcane bagasse vary according to variety and carbonization temperature, which is key for their efficient utilization in the manufacture of carbons and other industrially relevant products.

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