

Near-infrared spectroscopy for predicting the carbonization temperature of charcoal derived from Amazonian wood wastes

Espectroscopia no infravermelho próximo para predição da temperatura de carbonização do carvão vegetal derivado de resíduos de madeira da Amazônia

Michael Douglas Roque Lima^{1,2*} , Ana Beatriz Martins Soares¹ , Joabel Raabe¹ , Paulo Fernando Trugilho² ,
Thiago de Paula Protásio² , Paulo Ricardo Gherardi Hein² 

ABSTRACT

The precise monitoring of the maximum carbonization temperature is vital for quality control of charcoal produced from Amazonian wastes. However, no viable or efficient technologies exist for classifying charcoal derived from wood wastes generated under sustainable forest management plans (SFMPs) based on their final carbonization temperature. In this study, the accuracy of multivariate models calibrated with near-infrared (NIR) spectral data was assessed for estimating the final carbonization temperature of charcoals from wood waste in Amazonia. Furthermore, residual samples of the Amazonian woods—angelim-vermelho (*Dinizia excelsa*), timborana (*Pseudopiptadenia suaveolens*), pequiara (*Caryocar* sp.), inharé (*Brosimum gaudichaudii*), tapiririca (*Tapirira guianensis*), and Marupá-amarelo (*Simaba guianensis*)—were carbonized in the laboratory at four different final temperatures (400, 500, 600, and 700 °C). Moreover, the spectral data from the radial and transverse surfaces of the charcoals were analyzed using principal component analysis (PCA) and partial least squares discriminant analysis (PLS-DA). Although PCA effectively grouped the samples into different temperature ranges, it could not fully differentiate them due to spectral overlap. In contrast, PLS-DA demonstrated superior performance, accurately classifying over 89% of the charcoals. The carbonization temperature of charcoal from Amazonian wood wastes was effectively predicted through benchtop NIR combined with multivariate models. For broader applicability under operational conditions, future studies should assess the performance of portable NIR devices for in-field classification and monitoring.

Index terms: Residual biomass; slow pyrolysis; NIRS; charcoal quality.

RESUMO

O monitoramento preciso da temperatura máxima de carbonização é vital para o controle de qualidade do carvão vegetal produzido a partir de resíduos amazônicos. No entanto, não existem tecnologias viáveis ou eficientes para classificar o carvão vegetal derivado de resíduos de madeira gerados sob planos de manejo florestal sustentável (PFMS) com base em sua temperatura final de carbonização. Neste estudo, a precisão de modelos multivariados calibrados com dados espectrais do infravermelho próximo (NIR) foi avaliada para estimar a temperatura final de carbonização de carvões de resíduos de madeira na Amazônia. Além disso, amostras residuais das madeiras amazônicas — angelim-vermelho (*Dinizia excelsa*), timborana (*Pseudopiptadenia suaveolens*), pequiara (*Caryocar* sp.), inharé (*Brosimum gaudichaudii*), tapiririca (*Tapirira guianensis*), e marupá-amarelo (*Simaba guianensis*) — foram carbonizadas em laboratório em quatro diferentes temperaturas finais (400, 500, 600 e 700 °C). Os dados espectrais das superfícies radial e transversal dos carvões foram analisados por meio de análise de componentes principais (PCA) e análise discriminante por mínimos quadrados parciais (PLS-DA). Embora a PCA tenha agrupado as amostras em diferentes faixas de temperatura de forma eficaz, não foi possível diferenciá-las completamente devido à sobreposição espectral. Em contraste, a PLS-DA demonstrou desempenho superior, classificando com precisão mais de 89% dos carvões. A temperatura de carbonização do carvão vegetal proveniente de resíduos de madeira da Amazônia foi prevista de forma acurada por meio de NIR de bancada combinado com modelos multivariados. Para uma aplicabilidade mais ampla em condições operacionais, estudos futuros devem avaliar o desempenho de dispositivos NIR portáteis para classificação e monitoramento em campo.

Termos para indexação: Biomassa residual; pirólise lenta; NIRS; qualidade do carvão vegetal.

Agricultural Sciences

Ciênc. Agrotec., 49:e007725, 2025
<http://dx.doi.org/10.1590/1413-7054202549007725>

Editor: Renato Paiva

¹Universidade Estadual da Região Tocantina do Maranhão/ UEMASUL, Centro de Ciências Agrárias, Departamento de Ciência Florestal, Imperatriz, MA, Brasil

²Universidade Federal de Lavras/UFLA, Departamento de Ciências Florestais/ DCF, Lavras, MG, Brasil

*Corresponding author: michael.lima@uemasul.edu.br

Received in April 3, 2025 and approved in September 16, 2025

Introduction

Charcoal, an essential renewable biofuel, is extensively used in industries as a bioreducing agent in pig iron and steel production (Rodrigues & Braghini Junior, 2019a). Notably, the steel and metallurgical sectors benefit significantly from charcoal's high energy density and reducing properties. In 2024, Brazil ranked 9th among global steel producers and contributed approximately 33.74 million tons of crude steel (World Steel Association, 2025). In 2023, Brazil led Latin America's steel production, accounting for 54.9% of the regional output (Instituto Aço Brasil, 2023).

In 2022, the steel sector produced 7.8 million tons of pig iron from charcoal, representing 24.1% of total pig iron production

(32.4 million tons) (Sindicato da Indústria do Ferro no Estado de Minas Gerais, SINDIFER, 2023). Rising environmental concerns and regulatory pressure necessitate cleaner production methods with lower pollution levels (Santos & Hatakeyama, 2012). Consequently, researchers and industries are seeking economically viable alternatives that align with sustainability objectives while ensuring efficiency.

In 2023, Brazil produced approximately 6.7 million tons of charcoal, with only 0.05 million tons originating from native forest sources, while the remaining 6.65 million tons were sourced from energy plantations, predominantly composed of species from the *Eucalyptus* genus (Indústria Brasileira de Árvores, IBÁ, 2024). Notably, *Eucalyptus* wood remains the primary raw material for charcoal production in Brazil (Rodrigues & Braghini Junior, 2019b). Hence, wood waste has emerged as a sustainable and efficient alternative for charcoal production.

As defined by Normative Instruction No. 5 (December 11, 2006) (Brasil, 2006), wood wastes include branches, buttresses, and trunk remnants from harvested trees, which can be repurposed for firewood and energy production. Recent research demonstrates that derived charcoal exhibits favorable industrial qualities, which in certain cases surpass those of eucalyptus charcoal (Lima et al., 2020b; Lima et al., 2022a).

The energetic valorization of Amazonian wood wastes aligns with Brazil's National Solid Waste Policy (Law No. 12,305 of August 2, 2010) (Brasil, 2010), which prioritizes waste reduction, reuse, and recycling. Additionally, it supports the United Nations' 2030 Agenda for Sustainable Development Goals (SDGs), particularly SDG 7 (Affordable and Clean Energy), SDG 11 (Sustainable Cities and Communities), and SDG 13 (Climate Action) (United Nations, 2015). Therefore, utilizing waste for charcoal production represents a sustainable and clean practice, particularly for meeting the requirements of the Brazilian industrial sector.

Notably, the quality control of the final product is one of the major challenges with using waste for charcoal production. Hence, it is essential to monitor various parameters throughout the carbonization process, particularly the final temperature, which directly influences the physical and chemical properties of charcoal (Lima et al., 2022b; Santos et al., 2025). Although conventional assessment methods, including physical-chemical laboratory analyses, are accurate, they are destructive, time-consuming, costly, and hinder rapid decision-making on an industrial scale (Picancio et al., 2018; Silva et al., 2018a). These limitations delay the production process, increase operational costs, and hinder the implementation of real-time control strategies.

The classification of charcoal based on final carbonization temperature holds practical implications beyond post-production assessment. Although the temperature cannot be altered after carbonization, determining whether the material was produced

within the optimal range enables quality control and traceability and facilitates more accurate product classification. Generally, charcoal produced outside ideal temperature thresholds presents inadequate and inconsistent properties, adversely affecting both industrial performance and market acceptance. Therefore, post-production classification allows producers to segregate batches by quality, convey product specifications to buyers, and redirect lower-quality material to less demanding applications. Furthermore, rapid NIR-based classification models serve as a feedback tool, enabling producers to monitor outcomes and adjust carbonization parameters in subsequent cycles, thereby enhancing consistency and efficiency in production.

Interestingly, near-infrared (NIR) spectroscopy emerges as a promising alternative in this context to overcome these limitations. Following model calibration, it constitutes an advanced, fast, non-destructive, and low-cost analytical method that facilitates the analysis of chemical and physical properties through the interaction of light with matter (Nisgoski et al., 2016).

According to previous literature, multivariate models calibrated with NIR spectral data can predict important wood properties, including basic density (Medeiros et al., 2023), moisture (Amaral et al., 2020; Santos et al., 2021; Tham, Inagaki, & Tsuchikawa, 2018), extractives, and lignin content (Estopa et al., 2017; He & Hu, 2013; Silva et al., 2013). In the case of charcoal, NIR spectroscopy can estimate relevant parameters, including chemical composition (volatile matter and fixed carbon content) and gravimetric yield, and even differentiate samples based on the final carbonization temperature (Costa, Trugilho & Hein, 2018; Nisgoski et al., 2020; Ramalho et al., 2019). Costa, Trugilho and Hein (2018) demonstrated that NIR can distinguish charcoal by its final carbonization temperature, which is essential for quality control and process optimization. Therefore, implementing NIR spectroscopy represents a significant improvement over conventional methods, facilitating rapid monitoring, greater production efficiency, and cost reduction, while also contributing to more sustainable practices in the steel industry.

This study presents, to the best of our knowledge, the first instance of applying NIR spectroscopy with multivariate models to categorize charcoal derived from Amazonian residual wood based on the final carbonization temperature. Previous studies have primarily focused on charcoal produced from *Eucalyptus* wood (Costa, Trugilho & Hein, 2018) or on sawmill wastes from *Spirostachys africana*, *Terminalia* sp., and *Colophospermum mopane* in Mozambique (Nisgoski et al., 2020). However, greater anatomical (Perdigão et al., 2020) and chemical (Lima et al., 2020a) diversity observed in Amazonian species significantly influences both the carbonization process and the resulting spectral responses. Therefore, developing models specifically adapted to these native wastes is a crucial step toward expanding the applicability of NIR technology to more heterogeneous and complex biomass scenarios.

This study is relevant as it addresses the lack of rapid and non-destructive methods for estimating the final carbonization temperature of charcoal produced from heterogeneous Amazonian wood wastes. We hypothesize that multivariate classification models based on near-infrared (NIR) spectral data can accurately distinguish charcoal samples by their final carbonization temperature, even when derived from heterogeneous wood wastes. Therefore, in this study, the accuracy of multivariate models calibrated with NIR was assessed for estimating the final carbonization temperature of charcoals from wood waste in Amazonia.

Material and Methods

Origin of wood wastes

Amazonian wood wastes were sourced from the waste storage yard of a charcoal production unit at Rio Capim Farm, Paragominas Town, Pará State, Brazil (coordinates: 3° 30' and 3° 45'S – 48° 30' and 48° 45 'W; altitude: ~ 87 m). This unit belongs to the Keilla Group and operates under a license issued by the Pará State Secretariat for the Environment and Sustainability. The storage yard consists of residual wood from buttress roots, trunk remnants, and branches. In this study, we specifically analyzed branch waste from trees logged under a Sustainable Forest Management Plan (SFMP).

Sample collection and botanical identification

Based on their availability and frequent use in charcoal production, six distinct Amazonian tropical wood species were selected for this study from a broader group of twelve species reported by Lima et al. (2022b). Following the classification proposed by Csanady, Magoss and Tolvaj (2015), which defines low (0.300–0.500 g/cm³), medium (0.500–0.750 g/cm³), and high (0.750–1.000 g/cm³) density classes, these species were grouped according to their basic wood density. Table 1 presents the details of the selected species. Although this study builds upon previous research conducted during a doctoral thesis, it must be noted that all data presented herein are original and have not been previously published.

The wood in the waste piles in the storage yard was identified using a botanical identifier. The wood pieces were relocated using a Volvo L90F loader and subsequently sent to the preparation stage. At this stage, the material was sectioned into six wood pieces per species, each measuring 60 cm in length. A chainsaw operator sectioned each wood piece to extract a 200 mm-thick wood disc. Subsequently, these discs were designated for anatomical identification at the xylotheque of Embrapa Eastern Amazon (P. A. Costa – Embrapa/PA/Brazil), laboratory carbonization, and spectral analyses. A total of 72 test specimens (40 × 40 × 30 mm) were produced per species from each 200 mm-thick disc.

Laboratory carbonization process

Laboratory carbonization was performed using an electric muffle furnace (model Q318S25T, Quimis, São Paulo, Brazil), equipped with a carbonization capsule connected to a water-cooled condenser, which is coupled to a condensable gas collection flask. The carbonization process involved 72 wood samples per species, subjected to four final carbonization temperatures (400, 500, 600, and 700 °C). In particular, this temperature range was chosen to simulate the final temperature ramp reached by traditional “hot tail” kilns in the Brazilian Amazon (Lima et al., 2022a). Notably, 18 samples per wood type were used at each temperature.

The test specimens were oven-dried before carbonization under forced-air circulation at 105 ± 2 °C for 24 hours. The carbonization process followed a standardized protocol following the same methodology adopted by Protásio et al. (2021). The process began at an initial temperature of 100 °C, with a heating rate of 1.67 °C per minute, and a residence time of 60 minutes.

Near-infrared spectral data collection

The spectral readings of 432 laboratory-produced charcoal samples were acquired using diffuse reflectance mode. Here, the analysis was performed using a Bruker spectrometer (model MPA, Optik GmbH, Ettlingen, Germany), equipped with an integrating sphere and optical fiber.

Table 1: Tropical woods selected for the study.

Scientific name	Family	Common name	WD (g/cm ³)*
<i>Dinizia excelsa</i> Ducke	Fabaceae	Angelim-vermelho	0.927 ± 0.010
<i>Pseudopiptadenia suaveolens</i> (Miq.) J.W.Grimes	Fabaceae	Timborana	0.790 ± 0.006
<i>Caryocar</i> sp.	Caryocaraceae	Pequiarana	0.701 ± 0.015
<i>Brosimum gaudichaudii</i> Trécul	Moraceae	Inharé	0.599 ± 0.029
<i>Tapirira guianensis</i> Aubl.	Anacardiaceae	Tapiririca	0.397 ± 0.016
<i>Simaba guianensis</i> (Aubl.) Engl.	Simaroubaceae	Marupá-amarelo	0.354 ± 0.029

WD = wood density. *Source: (Lima et al., 2022b).

Furthermore, spectral data were acquired using OPUS software (version 7.5), covering a radiation range of 12500–3500 cm^{-1} , with a resolution of 8 cm^{-1} . Notably, the spectra collected within the range of 9000–4000 cm^{-1} were used for calibration purposes, as the remaining range contained static noise and lacked relevant information (Costa, Trugilho & Hein, 2018). As proposed by Costa et al. (2019), Lima et al. (2025), and Nisgoski et al. (2021), the charcoal samples were conditioned in a room with a controlled temperature of approximately 20 °C and relative humidity of around 65%, and the spectral readings were conducted directly on their transverse and radial surfaces.

Multivariate statistical analysis

Principal component analysis (PCA) and partial least squares discriminant analysis (PLS-DA) were performed using Chemoface software, version 1.61 (Nunes et al., 2012). Notably, data dependency and spectral similarity were assessed through PCA, with model validation performed using the cross-validation method (leave-one-out). The number of latent variables was determined based on the lowest root mean square error of cross-validation (RMSECV) or prediction (RMSEP) and the highest coefficient of determination for cross-validation (R^2_{cv}) or prediction (R^2_p).

The classification models for the final carbonization temperatures were developed using PLS-DA. Based on the PLS regression (PLS-R) approach, PLS-DA correlates two variable blocks: X (independent variables) and Y (dependent variables). The independent variables (X) consisted of the NIR spectral matrix, while the dependent variables (Y) corresponded to the temperature classes (400, 500, 600, and 700 °C) assigned for each type of charcoal sample. The models were adjusted using spectral data treated with the first and second derivatives and untreated data. Meanwhile, outliers were identified through graphical residual analysis.

In practice, the charcoal samples were grouped into four different classes based on their final carbonization temperatures (400, 500, 600, and 700 °C). The PLS-DA models were validated using both cross-validation and independent validation methods. Finally, a confusion matrix was generated, which describes the percentages of correct and incorrect classifications following PLS-DA model testing (Costa et al., 2019; Costa, Trugilho & Hein, 2018; Ramalho, Andrade, & Hein, 2018).

The models were fitted using both raw and mathematically treated spectral data. Herein, the data were pre-processed using first- (1d) and second-order derivatives (2d), based on a second-order polynomial with a 15-point window on each side, thereby improving visualization and reducing spectral noise. These pre-treatments are commonly applied in spectral studies involving charcoal discrimination (Lima et al., 2025; Nisgoski et al., 2015; Vieira et al., 2020). The first- and second-order derivatives were computed using the Savitzky–Golay algorithm (15-point filter and second-order polynomial) (Savitzky & Golay, 1964), enabling numerical derivation of a vector while incorporating a

smoothing step. It is noteworthy that derivative transformations effectively minimize additive and multiplicative effects in spectral data and correct baseline offsets (Muniz et al., 2016). Specifically, the first derivative removes the baseline, whereas the second-order derivative eliminates both the baseline and linear trends (Rinnan et al., 2009).

Results and Discussion

Average of NIR spectra

Figure 1 presents the spectral curves of the mean data obtained from the radial and transverse surfaces of charcoal produced from tropical woods at different final carbonization temperatures. It is noted that the curve shifts upward as the final carbonization temperature increases between 400 and 600 °C, resulting in higher NIR absorbance. However, at 700 °C, spectral curves significantly overlap with those at 400 °C and 500 °C on both sample surfaces (Figure 1). The spectral curves of the radial surface varied within the NIR absorbance range from 0.6 to 1.4 for charcoal produced at 400 and 500 °C (Figures 1A and 1C), while the curve of the transverse surface ranged from 0.8 to 1.6 (Figures 1B and 1D). This variation amplitude decreased as the carbonization temperature increased to 600 °C and 700 °C (Figures 1E, 1F, 1G, and 1H). Notably, a distinct absorption band was observed in the 5500–5000 cm^{-1} area (highlighted in red lines in plots of Figures 1A and 1B) in the charcoals produced at 400 °C, regardless of the analyzed surface.

The NIR spectra measured between 9000 and 4000 cm^{-1} on charcoal samples from both radial and transverse surfaces revealed variations linked to thermal degradation at different carbonization temperatures. Moreover, as reported by Costa, Trugilho and Hein (2018), higher final carbonization temperatures resulted in lower NIR absorbance.

The absorption bands observed in the 5000–5500 cm^{-1} range correspond to specific molecular vibrations associated with functional groups in the charred material. Typically, these bands correspond to overtones or combination bands of fundamental vibrations, particularly related to C–H stretching modes, which can be influenced by the degree of carbonization and the presence of various functional groups (Low et al., 1990). Although the bands at 5000 and 5500 cm^{-1} provide valuable insights into the vibrational characteristics of char, these spectral features may vary depending on the type of precursor material and processing conditions, resulting in differences in spectral profiles across different charcoal samples.

Notably, the average spectral curves facilitated a clear visual distinction among charcoals produced at 400 °C, 500 °C, and 600 °C. However, the spectra of charcoals produced at 700 °C partially deviate from this trend, demonstrating significant overlap with those from lower temperatures. This behavior was consistent across all species and both spectral acquisition surfaces (Figure 2).

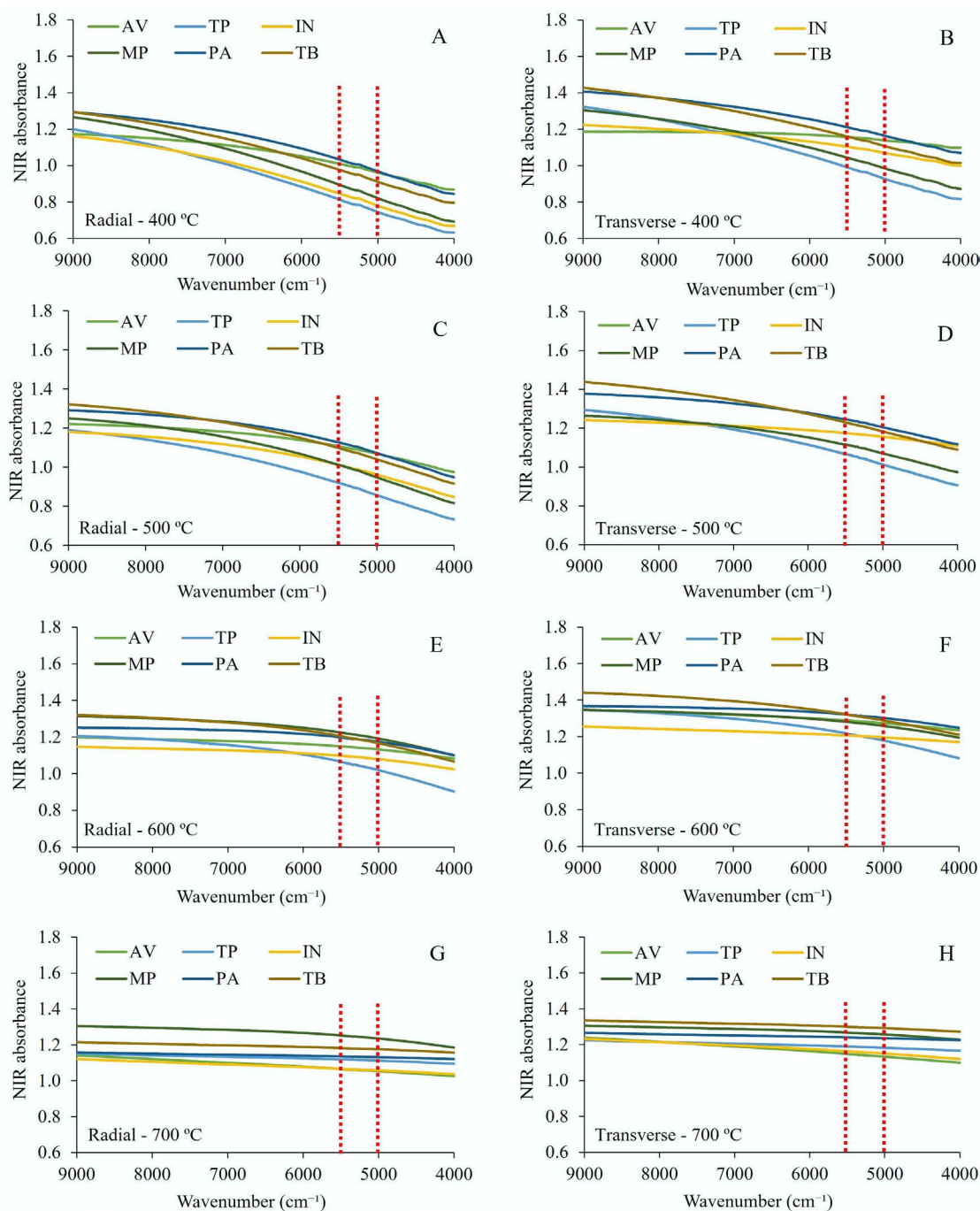


Figure 1: Mean spectra collected from charcoal samples of tropical woods on the radial and transverse surfaces at temperatures of 400 °C (A – radial; B – transverse), 500 °C (C – radial; D – transverse), 600 °C (E – radial; F – transverse), and 700 °C (G – radial; H – transverse). AV = *D. excelsa*; TB = *P. suaveolens*, PA = *Caryocar* sp., IN = *B. gaudichaudii*, TP = *T. guianensis*, MP = *S. guianensis*.

The clear spectral separation observed among charcoal samples produced at 400 °C, 500 °C, and 600 °C emphasizes the predominant role of carbonization temperature in shaping the NIR spectral characteristics of charcoal. This pattern was observed despite the inherent variability in

density, chemical composition, fixed carbon, and ash content among the Amazonian species analyzed. This finding indicates that thermal degradation processes exert a significantly stronger influence on spectral characteristics than species-specific properties.

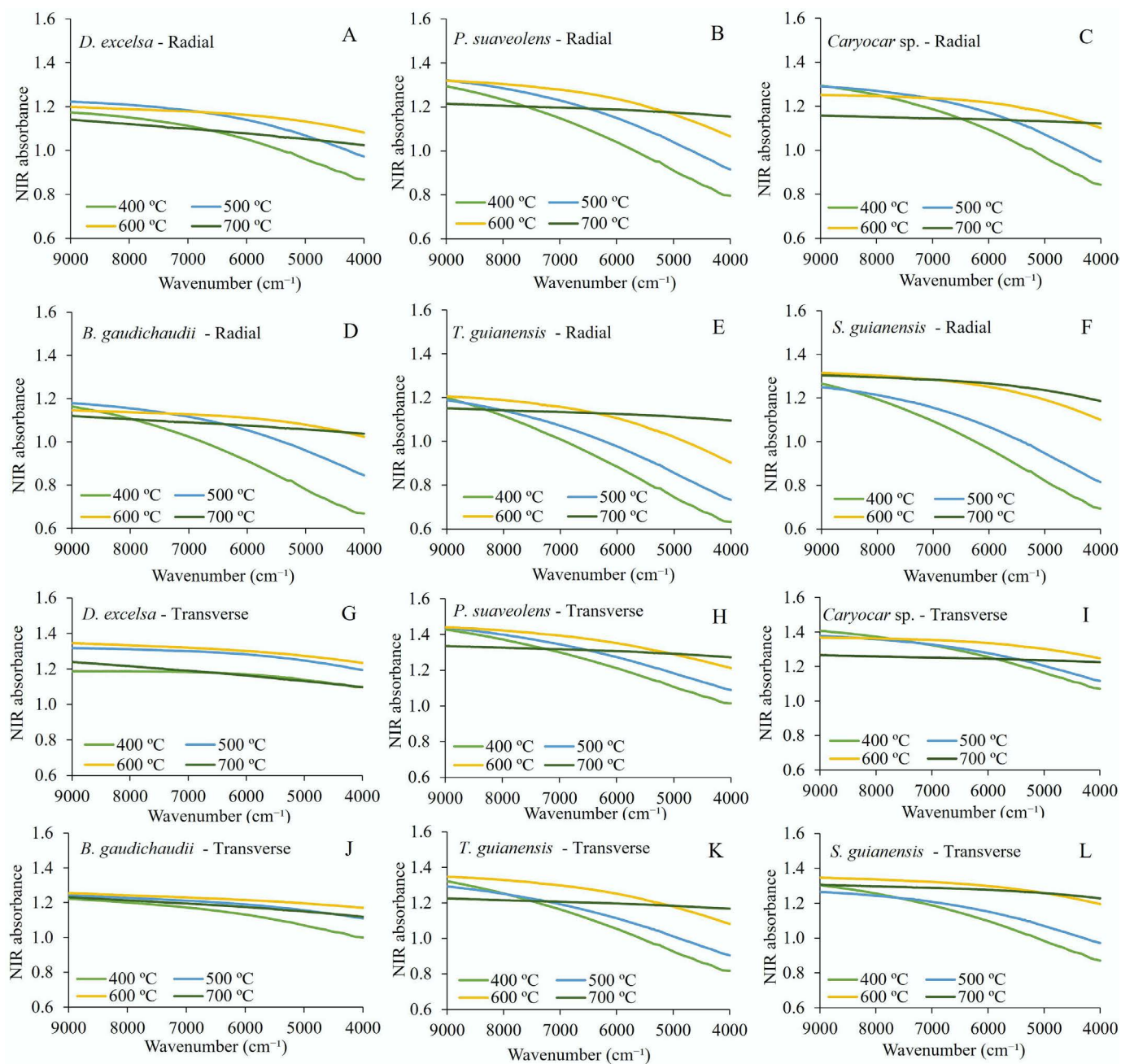


Figure 2: Mean spectra collected from charcoal samples produced at different final temperatures from the tropical woods *D. excelsa* (A – Radial; G – Transverse), *P. suaveolens* (B – Radial; H – Transverse), *Caryocar* sp. (C – Radial; I – Transverse), *B. gaudichaudii* (D – Radial; J – Transverse), *T. guianensis* (E – Radial; K – Transverse), and *S. guianensis* (F – Radial; L – Transverse).

This effect is directly linked to the thermal decomposition behavior of wood constituents during carbonization. The degradation of cellulose, hemicelluloses, and lignin occurs across different temperature ranges, with hemicelluloses breaking down between 173 and 690 °C, cellulose between 250 and 395 °C, and lignin between 170 and 835 °C (Apaydin Varol & Mutlu, 2023). Notably, lignin is the most thermally stable

molecular component of wood due to its complex chemical structure and the types of bonds present in its macromolecule.

Trugilho and Silva (2001) evaluated charcoals from *Hymenaea courbaril* (Jatobá) wood produced at different temperatures of 300–900 °C and found that the fixed carbon and ash content increase with increasing carbonization temperature, while the volatile material content decreases. Additionally,

similar trends were reported by Padilla et al. (2019) and Silva et al. (2018b), who found that elevated final carbonization temperatures result in higher fixed carbon content in charcoal. While fixed carbon was not quantified in this study, this well-documented pattern likely explains the increased spectral uniformity and the overlap observed in the average curves of the samples carbonized at 700 °C, as depicted in Figure 2.

The overlap of data points at 700 °C results from the higher homogeneity of charcoal produced at high temperatures, making sample differentiation using NIR spectroscopy more challenging (Ramalho et al., 2017). As reported in the study by Kim and Hanna (2006) on charcoals derived from *Quercus variabilis*, this can be attributed to cell wall thinning and volumetric shrinkage, resulting in a more uniform structure that resembles the chemical composition of graphite. Moreover, this behavior is related to the structural homogenization of charcoal at extremely high temperatures, which reduces spectral contrast due to the advanced degradation of organic compounds.

This spectral behavior may be attributed to the stronger interaction between infrared radiation and the molecular constituents of the charcoal at lower temperatures, where cell wall components remain largely undegraded (Ramalho et al., 2017). The degradation and rearrangement of carbon structures may lead to a converging chemical profile at higher temperatures, diminishing the variability detected by NIR spectroscopy.

The absorption bands identified between 5000 and 5500 cm^{-1} are attributed to the structural and chemical changes that occur due to the decomposition and transformation of organic components like lignin, cellulose, and hemicelluloses into carbonized structures. Thus, these bands reflect the progressive degradation of polymers from the original biomass during the carbonization process (Muñiz et al., 2013).

The overlap of the spectral curve across different temperatures limits the visual evaluation of spectral signatures, as factors such as noise, compression, and metamerism impact their accuracy (Fu et al., 2024). Therefore, the spectral temperature ranges were distinguished more effectively by performing multivariate statistical analysis on the spectral data. This approach enables more robust interpretation by revealing subtle patterns and similarities across the spectral dataset (Nascimento et al., 2017).

Principal component analysis

The principal component analysis (PCA) scores revealed the spectral variability of the radial and transverse surfaces of charcoal samples produced at 400, 500, 600, and 700 °C. As shown in Figure 3A, the accumulated variance of the untreated spectral data for the radial surfaces from PC1 (99.72%) and PC2 (0.27%) totaled 99.99%. Similarly, the raw spectral data also explained 99.99% of the variability in the transverse surfaces (Figure 3B). Notably, a greater dispersion of points was observed on the radial surface, whereas there was an overlap between the

evaluated temperature levels on the transverse surface. The data processed with the first derivative (1d) markedly enhanced the spectral distinction of the charcoal samples, both on the radial surface (Figure 3C) and the transverse surface (Figure 3D). In contrast, the scores from the spectral data processed with the second derivative (2d) (Figures 3E and 3F) showed no significant improvement over 1d.

The greater overlap of scores on the transverse surface reflects the larger number of anatomical structures exposed and the increased signal variability caused by wood porosity. Here, the high vessel density and large pore diameters serve as scattering centers, amplifying spectral noise and affecting data consistency (Muñiz et al., 2013). Additionally, thermal degradation of polymers during carbonization promotes the formation of micropores and macropores within the charcoal matrix (Gmach et al., 2023), along with cracks and fissures, which are intrinsic characteristics of charcoal.

Nevertheless, NIR demonstrates sufficient sensitivity to distinguish charcoal samples by carbonization temperature, despite PCA being an initial exploratory technique. Although the radial surface data treated with the first derivative (1d) produced clearer groupings of charcoals produced at different temperatures, some overlap of points is still noticeable, particularly at 700 °C (Figure 3C). This overlap reflects the statistical limitations, like collinearity of NIR spectra (Monteiro et al., 2010). Furthermore, it increases the chemical homogeneity of charcoal at higher temperatures, where different species tend to converge in composition due to advanced thermal degradation. Therefore, complementary techniques such as PLS-DA become essential to achieve more precise discrimination and reduce classification errors between temperature groups.

Cross-validation of charcoal classification models

Considering the final carbonization temperatures and the two surfaces from which the spectra were obtained, the PLS-DA models for charcoal classification demonstrated accuracy levels ranging from 53.09% (transverse surface data treated with 2d) to 89.59% (radial surface data with no mathematical treatment) (Table 2). By contrast, the PLS-DA model calibrated with spectral data from the transverse surface treated with 2d achieved an accuracy of 74.64%.

The prediction of the final carbonization temperature is crucial to the steel industry, as it influences the quality indices of charcoal, including apparent relative density and fixed carbon content (Santos et al., 2025), as well as gravimetric yield in charcoal and specific consumption of firewood (Lima et al., 2022a). As reported by Andrade et al. (2012) and Ramalho et al. (2017), wood specimens carbonized at high final temperatures produce homogeneous charcoal due to the higher concentration of carbon in their composition. This is attributed to the extensive degradation of wood polymers at higher temperatures, increasing the fixed carbon content (Santos et al., 2025).

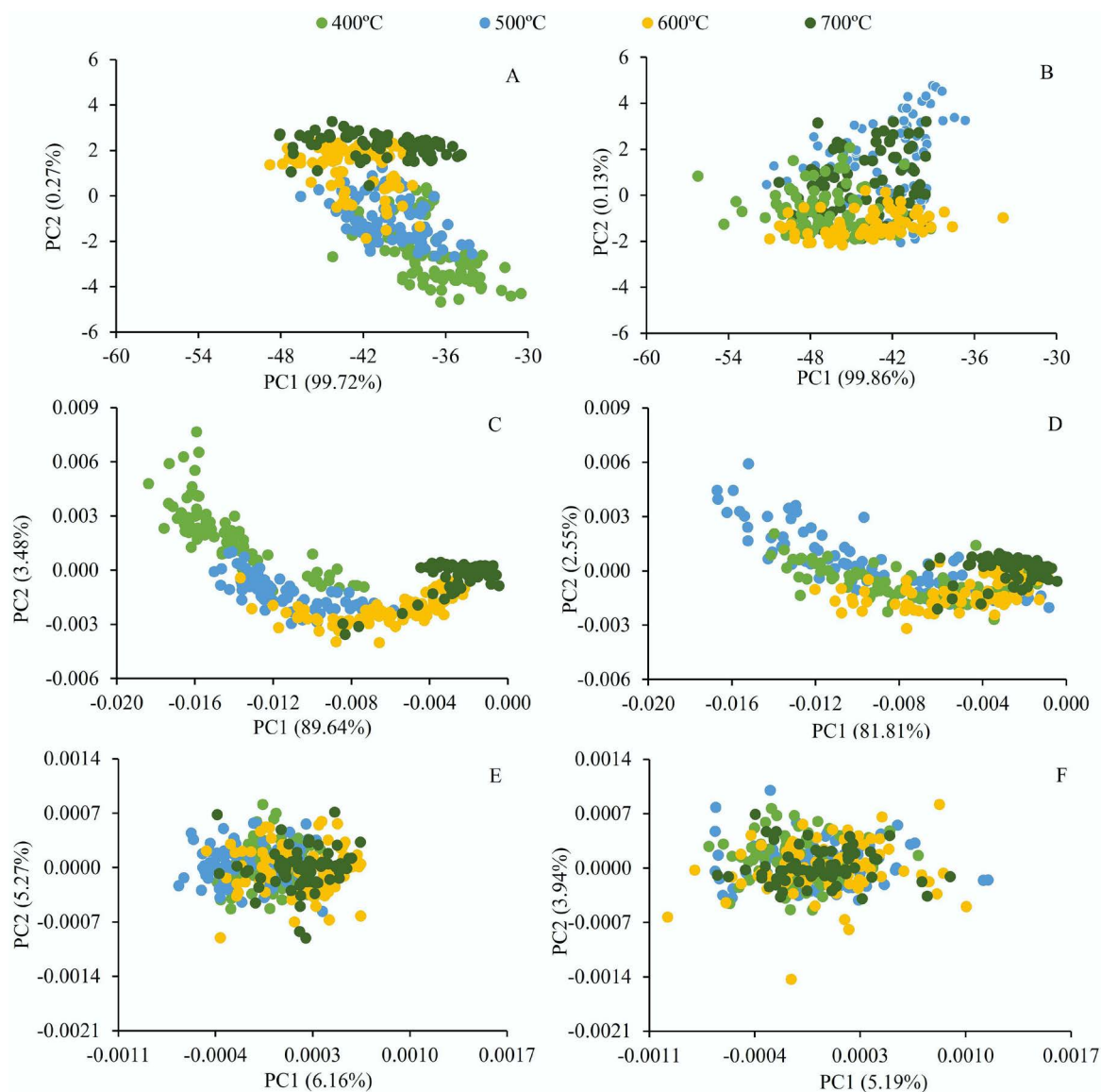


Figure 3: Principal component analysis (PCA) scores of spectral data obtained from the radial (A - raw spectra; C - spectra processed with the first derivative; and E - spectra processed with the second derivative) and transverse (B - raw spectra; D - spectra processed with the first derivative; and F - spectra processed with the second derivative) surfaces of laboratory-produced charcoal samples at four different carbonization temperatures.

As higher temperatures enhance the fixed carbon content, the charcoal received by the steel industry must be of higher quality, consequently yielding higher added value. Thus, the precise control of temperature can improve the commercialization value of charcoal, whether measured by mass or volume. Although charcoal produced at higher temperatures contains higher fixed carbon content, the carbonization yield is lower, making it necessary for producers to increase their marketing prices to account for the higher fixed carbon content.

The confusion matrix based on untreated radial surface spectral data of charcoal correctly classified 297 samples by temperature,

corresponding to an accuracy rate of 89.59% (Table 3). Notably, prediction accuracy values exceeding 85% were reported for all temperatures, with 96.47% and 91.78% recorded for 400 and 700 °C, respectively. The PLS-DA model made the highest number of classification errors between 500 and 600 °C.

According to our findings, the optimal temperature range for carbonization is between 400 and 500 °C. Although higher temperatures increase the apparent density of charcoal, they also result in significant losses in gravimetric yield (Lima et al., 2022a). Additionally, high-temperature carbonization prolongs the cycle, decreasing productivity and increasing gas emissions.

Table 2: Summary of charcoal sample classifications using PLS-DA analysis and cross-validation method, considering the surface of spectrum acquisition and the mathematical pre-treatment used.

Temperature (°C)	Radial (10 LV)						Transverse (10 LV)					
	NT		1d		2d		NT		1d		2d	
	n	%	n	%	n	%	n	%	n	%	n	%
400	82	96.47	81	95.29	80	94.12	75	86.21	79	90.80	69	79.31
500	74	85.06	75	86.21	49	56.32	63	73.26	61	70.93	43	50.00
600	74	85.06	73	83.91	39	44.83	49	57.65	49	57.65	34	40.00
700	67	91.78	64	87.67	33	45.21	57	79.17	57	79.17	31	43.06
Average	--	89.59	--	88.27	--	60.12	--	74.07	--	74.64	--	53.09

LV: Latent Variable, NT: No treatment, 1d: first derivative, 2d: second derivative, and n: number of charcoal samples correctly classified by the PLS-DA model. The highest accuracy values for both surfaces are highlighted in bold.

Table 3: Classification of charcoal samples by final carbonization temperature using PLS-DA with cross-validation and a test set of spectral data from the untreated radial surface.

Temperature (°C)	NIR predicted classification (10 LV)				Classification	
	400	500	600	700	n	%
400	82	2	1		82	96.47
500	2	74	11		74	85.06
600		8	74	5	74	85.06
700			6	67	67	91.78
Overall rating					297	89.59

LV: Latent Variable.

Due to the increased degradation of wood polymers and the higher fixed carbon content, high temperatures make charcoal more homogeneous. The radial surface, which is less porous and has longitudinally cut vessels, provides a more uniform interaction with NIR radiation. This results in less spectral noise and scattering compared to the more irregular and porous transverse surface. Moreover, this explains the higher classification accuracy observed for the radial surface (ranging from 85.06% at 500 and 600 °C to 96.47% at 400 °C), suggesting more reliable spectral patterns for predicting carbonization temperature.

Notably, the PLS-DA model exhibited the highest number of classification errors between 500 and 600 °C, which is attributed to the increased spectral similarity of samples carbonized at these intermediate temperatures. As the chemical changes occurring between 500 and 600 °C may be more subtle compared to those at lower or higher temperatures, this spectral overlap reduces the discriminative power of the model, making it more difficult for the algorithm to distinguish between these classes.

The vessels are cut longitudinally on the radial surface, resulting in a more uniform interaction of NIR light with cell walls and reducing spectral noise. In contrast, pores exposed transversely create depth variations that intensify light scattering and make readings less stable. Thus, the higher structural

homogeneity of the radial surface enhances more consistent absorption and reflection of NIR radiation, which explains the superior performance of PLS-DA models on this surface.

Additionally, Costa, Trugilho and Hein (2018) utilized the PLS-DA technique to classify eucalyptus charcoal samples carbonized at final temperatures of 400°C, 500°C, 600°C, and 700°C. In that study, over 97.5% of the samples were correctly classified, a result that surpasses the validation achieved in this study. However, NIR spectroscopy combined with the PLS-DA technique provides satisfactory results for the temperature classes analyzed. In this context, the present study underscores the potential of multivariate analysis to enhance the accuracy of charcoal classification based on different carbonization temperatures. Thus, combining NIR and PLS-DA is a robust approach for industrial and research applications, where precise characterization of this material is essential.

The temperature control in the carbonization process is essential to ensuring the quality and homogeneity of charcoal characteristics, which is both relevant and necessary for industrial applications (Santos et al., 2025). Although standardized test specimens with uniform dimensions were used to minimize the variability in carbonization behavior and spectral response in this study, Amazonian wood wastes often

exhibit high variability in species, dimensions, and anatomical orientations under industrial conditions. Such heterogeneity influences the thermal degradation patterns and the surface characteristics of the resulting charcoal, potentially impacting the spectral signatures captured by NIR. Therefore, future applications of NIR technologies for charcoal classification should consider such variability to enhance model robustness and predictive reliability in operational settings.

This study addresses the challenge of classifying charcoal from Amazonian Forest wastes based on the final carbonization temperature, thereby advancing the application of near-infrared (NIR) spectroscopy combined with multivariate models. Although a previous study has explored the use of NIR to classify charcoal based on density classes (Lima et al., 2025), few studies have focused on distinguishing charcoal based on thermal processing conditions, particularly in the context of biomass derived from native tropical species. Our results demonstrate that NIR spectra, despite exhibiting overlapping regions, contain sufficient information to differentiate charcoal according to temperature ranges when combined with multivariate models. This helps bridge both scientific and practical gaps by providing a rapid and non-destructive approach to assessing the carbonization process. Such an approach is vital for quality control and traceability, particularly in contexts with high variability in biomass origin and processing conditions, such as the Brazilian Amazonia.

Implications and limitations of the study

As demonstrated in this study, near-infrared (NIR) spectroscopy combined with multivariate classification models is a promising tool to accurately estimate the final carbonization temperature of charcoal produced from heterogeneous Amazonian wood residues. The ability to classify charcoal according to carbonization temperature has significant practical implications for industrial quality control and process optimization. By enabling rapid, non-destructive, and low-cost monitoring, NIR technology allows producers to ensure product consistency, reduce waste, and increase overall process efficiency, while aligning with sustainability goals through optimized resource use and minimized environmental impacts.

From an economic perspective, implementing NIR-based models reduces operational costs by reducing dependence on conventional laboratory analyses, which are time-consuming and expensive. Faster decision-making facilitates timely adjustments to carbonization parameters, potentially resulting in higher yield and improved charcoal quality. Additionally, enhanced quality control increases the commercial value of the product, as buyers gain greater confidence in product specifications and traceability. Moreover, redirecting the lower-quality charcoal to less demanding applications reduces financial losses associated with off-spec material disposal.

Although a detailed economic analysis is beyond the scope of this study, preliminary assessments indicate that adopting portable NIR devices can be economically viable for medium to large producers. Hence, future research should further explore the economic impacts and scalability of this technology.

Although these results are promising, some limitations deserve attention. As the developed models are specific to the selected Amazonian species and the examined temperature range, their applicability to other species or different carbonization conditions requires further validation. Furthermore, variability in wood properties and processing conditions can influence spectral responses and, consequently, model robustness.

Although NIR spectroscopy enables rapid evaluations, initial model calibration requires a representative and well-characterized sample set, potentially involving significant upfront investment. To comprehensively understand the benefits and challenges of implementing NIR monitoring in the charcoal industry, future studies should expand the database to include a wider variety of species, wood conditions, and carbonization parameters, while also integrating economic and life-cycle analyses.

Conclusions

Wood identification could not be achieved through visual analysis of the spectral signature. However, spectral data from the radial and transverse surfaces, processed with the first derivative, effectively grouped the charcoal samples via PCA. Notably, the PLS-DA models calibrated with raw data from the radial surface successfully classified over 89% of the charcoal samples according to the final carbonization temperature. Additionally, future studies should investigate the potential of portable NIR devices for in situ, rapid, and non-destructive charcoal analysis.

Author contribution

Conceptual idea: Lima, M.D.R.L.; Protásio, T.P.; Hein, P.R.G., Methodology design: Lima, M.D.R.L.; Protásio, T.P.; Hein, P.R.G., Data collection: Lima, M.D.R.L., Data analysis and interpretation: Lima, M.D.R.L.; Soares, A.B.M.; Raabe, J.; Trugilho, P.F.T.; Protásio, T.P.; Hein, P.R.G., Writing and editing: Lima, M.D.R.L.; Soares, A.B.M.; Raabe, J.; Trugilho, P.F.T.; Protásio, T.P.; Hein, P.R.G.

Acknowledgements

This work was supported by the Brazilian National Council for Scientific and Technological Development (CNPq: grants no. 406593/2021-3, 306793/2019-9, 406053/2022-7, 304165/2022-0, 350017/2023-9, and 309620/2020-1), the Coordination for the Improvement of Higher Education Personnel (CAPES: Finance

Code 001), the Minas Gerais State Research Support Foundation (FAPEMIG: APQ-00742-23 and APQ-05431-24), Banco da Amazônia (financial support contract n°. 2018/193), Keilla Group, Multiuser Laboratory of Biomaterials and Biomass Energy of the Federal University of Lavras (UFLA, Brazil), and Laboratory of Wood Anatomy and Technology of the State University of the Tocantina Region of Maranhão (UEMASUL, Brazil).

Data Availability Statement

Data available upon request to authors.

References

- Amaral, E. A. et al. (2020). Estimation of moisture in wood chips by near infrared spectroscopy. *Maderas. Ciencia y Tecnología*, 22(3):291-302.
- Andrade, C. R. et al. (2012). Near infrared spectroscopy for estimating eucalyptus charcoal properties. *Journal of Near Infrared Spectroscopy*, 20(6):657-666.
- Apaydin Varol, E., & Mutlu, Ü. (2023). TGA-FTIR analysis of biomass samples based on the thermal decomposition behavior of hemicellulose, cellulose, and lignin. *Energies*, 16(9):3674.
- Brasil. (2010). Lei N° 12.305, de 2 de Agosto de 2010. Institui a Política Nacional de Resíduos Sólidos; altera a Lei no 9.605, de 12 de fevereiro de 1998; e dá outras providências. *Diário Oficial*: seção 1, Brasília, DF, N° 147 Available in: <https://www.planalto.gov.br/ccivil_03/_ato2007-2010/2010/lei/l12305.htm>.
- Brasil. Ministério do Meio Ambiente. (2006). *Instrução Normativa N° 5, de 11 de Dezembro de 2006*. Dispõe sobre procedimentos técnicos para elaboração, apresentação, execução e avaliação técnica de Planos de Manejo Florestal Sustentável-PMFS nas florestas primitivas e suas formas de sucessão na Amazônia Legal, e dá outras providências. Available in: <<https://www.legisweb.com.br/legislacao/?id=76720>>.
- Costa, A. C. P. R. et al. (2019). Classification of commercial charcoal for domestic use by near infrared spectroscopy. *Biomass and Bioenergy*, 127:105280.
- Costa, L. R., Trugilho, P. F., & Hein, P. R. G. (2018). Evaluation and classification of eucalypt charcoal quality by near infrared spectroscopy. *Biomass and Bioenergy*, 112:85-92.
- Csanady, E., Magoss, E., & Tolvaj, L. (2015). *Quality of machined wood surfaces*. Springer International Publishing.
- Estopa, R. A. et al. (2017). NIR spectroscopic models for phenotyping wood traits in breeding programs of *Eucalyptus benthamii*. *Cerne*, 23(3):367-375.
- Fu, Q. et al. (2024). Limitations of data-driven spectral reconstruction: Optics-aware analysis and mitigation. *ArXiv*. Available in: <<https://arxiv.org/abs/2401.03835>>.
- Gmach, F. et al. (2023). Anatomy and identification of charcoal marketed in the region of Papanduva, Santa Catarina, Southern Brazil. *FLORESTA*, 53(3):361-370.
- He, W., & Hu, H. (2013). Rapid prediction of different wood species extractives and lignin content using near infrared spectroscopy. *Journal of Wood Chemistry and Technology*, 33(1):52-64.
- Indústria Brasileira de Árvores - IBÁ. (2024). *IBÁ annual report 2024*. IBÁ. Available in: <<https://iba.org/wp-content/uploads/2025/05/relatorio2024.pdf>>.
- Instituto Aço Brasil. (2023). *Indústria do aço em números*. Available in: <https://www.acobrasil.org.br/site/wp-content/uploads/2023/08/MBA_Edicao-2023_AcoBrasil.pdf>.
- Kim, N.-H., & Hanna, R. B. (2006). Morphological characteristics of *Quercus variabilis* charcoal prepared at different temperatures. *Wood Science and Technology*, 40:392-401.
- Lima, M. D. R. et al. (2022a). Clarifying the carbonization temperature effects on the production and apparent density of charcoal derived from Amazonia wood wastes. *Journal of Analytical and Applied Pyrolysis*, 166:105636.
- Lima, M. D. R. et al. (2022b). Efficiency of near-infrared spectroscopy in classifying Amazonian wood wastes for bioenergy generation. *Biomass and Bioenergy*, 166:106617.
- Lima, M. D. R. et al. (2025). Multivariate models based on spectral data for the classification of charcoals produced at different final carbonization temperatures. *Cerne*, 31:e-103530.
- Lima, M. D. R. et al. (2020a). Logging wastes from sustainable forest management as alternative fuels for thermochemical conversion systems in Brazilian Amazon. *Biomass and Bioenergy*, 140:105660.
- Lima, M. D. R. et al. (2020b). Charcoal of logging wastes from sustainable forest management for industrial and domestic uses in the Brazilian Amazonia. *Biomass and Bioenergy*, 142:105804.
- Low, M. J. D. et al. (1990). Unusual bands in the infrared spectra of some chars. *Spectroscopy Letters*, 23(4):527-531.
- Medeiros, D. T. et al. (2023). Prediction of the basic density of tropical woods by near-infrared spectroscopy. *Cerne*, 29:e103262.
- Monteiro, T. C. et al. (2010). Use of near infrared spectroscopy to distinguish carbonization processes and charcoal sources. *Cerne*, 16(3):381-390.
- Muniz, G. I. B. et al. (2016). Wood and charcoal identification of five species from the miscellaneous group known in Brazil as "angelim" by near-ir and wood anatomy. *Maderas: Ciencia y Tecnología*, 18(3):505-522.
- Muñiz, G. I. B. et al. (2013). SEM and NIR characterization of four forest species charcoal. *Wood Science and Technology*, 47(4):815-823.

- Nascimento, C. C. et al. (2017). Estimation of the basic density of wood *Eschweilera odora* (Poepp.) Miers by near infrared spectroscopy. *Ciência da Madeira*, 8(1):42-53.
- Nisgoski, S. et al. (2021). Near-infrared spectroscopy for discrimination of charcoal from *Eucalyptus* and native Cerrado species-contribution to a database for forestry supervision. *Forest Science*, 67(4):419-432.
- Nisgoski, S. et al. (2016). Use of NIR for trees discrimination in different fertilizations based on wood and bark spectra. *Ciência da Madeira*, 7(2):100-110.
- Nisgoski, S. et al. (2015). NIR and anatomy of wood and charcoal from Moraceae and Euphorbiaceae species. *Ciência da Madeira*, 6(3):183-190.
- Nisgoski, S. et al. (2020). Charcoal anatomy and NIR spectra of *Spirostachys africana*, *Terminalia* sp. and *Colophospermum mopane* in different carbonization process. *SN Applied Sciences*, 2:908.
- Nunes, C. A. et al. (2012). Chemoface: A novel free user-friendly interface for chemometrics. *Journal of the Brazilian Chemical Society*, 23(11):2003-2010.
- Padilla, E. R. D. et al. (2019). Eucalyptus bark charcoal: The influence of carbonization temperature in thermal behavior. *Materials Research*, 22:e20190371.
- Perdigão, C. R. V. et al. (2020). Forestry control in the Brazilian Amazon I: Wood and charcoal anatomy of three endangered species. *IAWA Journal*, 41(4):490-509.
- Picancio, A. C. S. et al. (2018). Controle do processo de produção de carvão vegetal para siderurgia. *Caderno de Administração*, 12(1):1-17.
- Protásio, T. P. et al. (2021). Charcoal productivity and quality parameters for reliable classification of *Eucalyptus* clones from Brazilian energy forests. *Renewable Energy*, 164:34-45.
- Ramalho, F. M. G., Andrade, J. M., & Hein, P. R. G. (2018). Rapid discrimination of wood species from native forest and plantations using near infrared spectroscopy. *Forest Systems*, 27(2):e008.
- Ramalho, F. M. G. et al. (2017). Potential of near-infrared spectroscopy for distinguishing charcoal produced from planted and native wood for energy purpose. *Energy & Fuels*, 31(2):1593-1599.
- Ramalho, F. M. G. et al. (2019). Influence of particles size on NIR spectroscopic estimations of charcoal properties. *Floresta e Ambiente*, 26:e20180397.
- Rinnan, Å. et al. (2009). Review of the most common pre-processing techniques for near-infrared spectra. *Trends in Analytical Chemistry*, 28(10):1201-1222.
- Rodrigues, T., & Braghini Junior, A. (2019a). Charcoal: A discussion on carbonization kilns. *Journal of Analytical and Applied Pyrolysis*, 143:104670.
- Rodrigues, T., & Braghini Junior, A. (2019b). Technological prospecting in the production of charcoal: A patent study. *Renewable and Sustainable Energy Reviews*, 111:170-183.
- Santos, L. M. et al. (2021). Estimating wood moisture by near infrared spectroscopy: Testing acquisition methods and wood surfaces qualities. *Wood Material Science & Engineering*, 16(5):336-343.
- Santos, P. L. et al. (2025). Exploring the effects of slow pyrolysis temperature and species on the quality of charcoal from Amazonian woody wastes. *Renewable Energy*, 240:122257.
- Santos, S. F. O. M., & Hatakeyama, K. (2012). Processo sustentável de produção de carvão vegetal quanto aos aspectos: ambiental, econômico, social e cultural. *Produção*, 22(2):309-321.
- Savitzky, A., & Golay, M. J. E. (1964). Smoothing and differentiation of data by simplified least squares procedures. *Analytical Chemistry*, 36(8):1627-1639.
- Silva, A. R. et al. (2013). Assessment of total phenols and extractives of mahogany wood by near infrared spectroscopy (NIRS). *Holzforschung*, 67(1):1-8.
- Silva, R. C. et al. (2018a). Influence of the final carbonization temperature on the characteristics of tropical species charcoal. *Pesquisa Florestal Brasileira*, 38:e201801573.
- Silva, R. C. et al. (2018b). Influence of the final carbonization temperature on the characteristics of tropical species charcoal. *Pesquisa Florestal Brasileira*, 38:e201801573.
- Sindicato da Indústria do Ferro no Estado de Minas Gerais - SINDIFER. (2023). *Pig iron production in Minas Gerais and Brazil. Statistical yearbook reference: 2022*. SINDIFER. Available in: <<https://sindifer.com.br/sndfr/anuario-estatistico/>>.
- Tham, V. T. H., Inagaki, T., & Tsuchikawa, S. (2018). A novel combined application of capacitive method and near-infrared spectroscopy for predicting the density and moisture content of solid wood. *Wood Science and Technology*, 52(1):115-129.
- Trugilho, P. F., & Silva, D. A. (2001). Influence of final carbonization temperature in the physical and chemical characteristics of the jatobá (*Himenea courbaril* L.) charcoal. *Scientia Agraria*, 2(1):45-53.
- United Nations. (2015). *General Assembly Resolution A/RES/70/1. Transforming our world: The 2030 Agenda for Sustainable Development*. Available in: <https://www.un.org/en/development/desa/population/migration/generalassembly/docs/globalcompact/A_RES_70_1_E.pdf>.
- Vieira, H. C. et al. (2020). Near-infrared spectroscopy for the distinction of wood and charcoal from Fabaceae species: comparison of ANN, KNN AND SVM models. *Forest Systems*, 29(3):e020.
- World Steel Association. (2025). *December 2024 crude steel production and 2024 global crude steel production totals*. Available in: <<https://worldsteel.org/media/press-releases/2025/december-2024-crude-steel-production-and-2024-global-totals/>>.