



# Prediction of the Energy Properties of Charcoal Obtained from *Eucalyptus* and *Corymbia* Biomass Using Portable and Benchtop NIR Spectrometers

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## Abstract

Monitoring charcoal quality is essential for the industry. Near-infrared (NIR) spectroscopy enables fast and accurate predictions of key properties. This study evaluated the use of benchtop and portable NIR sensors to predict charcoal characteristics from woody biomass of 15 commercial clones (11 *Eucalyptus* and 4 *Corymbia*). Two trees per clone were sampled at six stem positions, generating 30 composite wood samples. After carbonization and grinding, spectral data were collected, totaling 600 spectra per sensor. Partial least squares regression was used to develop models for gravimetric yield (GY), apparent relative density (ARD), fines content (FC), volatile matter content (VMC), ash content (AC), and fixed carbon content (FCC). For *Eucalyptus* clones, the benchtop sensor outperformed the portable one for GY ( $R^2p=0.74$ ; RPD=2.02), ARD ( $R^2p=0.87$ ; RPD=2.82), VMC ( $R^2p=0.72$ ; RPD=1.92), AC ( $R^2p=0.72$ ; RPD=1.92), and FCC ( $R^2p=0.63$ ; RPD=1.64). The portable sensor was better only for FC ( $R^2p=0.64$ ; RPD=1.60). Similarly, for *Corymbia* clones, the benchtop sensor performed better for GY ( $R^2p=0.79$ ; RPD=2.15), ARD ( $R^2p=0.87$ ; RPD=2.77), FC ( $R^2p=0.69$ ; RPD=1.73), and AC ( $R^2p=0.61$ ; RPD=1.62). The portable sensor showed better results for FCC ( $R^2p=0.61$ ; RPD=1.48) and VMC ( $R^2p=0.64$ ; RPD=1.40). Overall, benchtop and portable NIR spectrometers showed similar performance in estimating charcoal parameters.

**Keywords** Immediate chemical analysis · Bioenergy · Forest biomass · Multivariate statistics · Wood pyrolysis

## Introduction

Brazil stands out on the world stage for being the largest producer and consumer of charcoal. According to the IBÁ report [1], Brazilian production was 6.7 million tons in 2022, almost entirely destined to supply the domestic market, emphasizing the steel and iron and steel sectors. The report also points out that of this total, 1.12 million hectares of planted forests were destined for the production of charcoal, mainly with the use of clones of the *Eucalyptus* genus [1–3], in addition to the use of clones of the *Corymbia* genus [4, 5], indicating advances in clonal silviculture practiced by forestry companies and a reduction in the use of native woods from deforestation.

As it is a versatile energy material, charcoal offers various applications. In the steel industry, it is used as a source of energy and a bioreducing agent, in addition to supporting a load of iron ore [6]. This material, in turn, is used as a raw

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material for pig iron production, and its destination is steel production [7]. Therefore, charcoal improves the quality of pig iron and steel due to the absence of sulfur and lower ash content when compared to coal, making the process environmentally cleaner [8].

To increase the quality of charcoal, carbonization plants use the strategy of selecting wood with desirable characteristics for energy purposes and controlling the pyrolysis process to improve gravimetric yield and conversion efficiency [9]. Therefore, at the end of the wood carbonization process, it is necessary to monitor charcoal's chemical, physical, and energetic characteristics, such as apparent relative density and immediate chemistry, since homogeneous materials are desirable to maintain the quality of products from the steel sector.

Variables such as gravimetric yield, apparent relative density, fines content, volatile matter content, ash content, and fixed carbon content are indicators of charcoal quality, depending on the parameters of the carbonization process and the classification of the raw material [2, 10]. In other words, this information makes it possible to optimize final carbonization temperatures [6], furnace loading adjustments to improve carbonization efficiency [8], the particle size of the biomass used for charcoal production [11], and heating rates [12]. However, the production dynamics of charcoal in industrial settings are highly intense and often demand speed and accuracy in the results. This, in turn, is not always achievable through laboratory procedures which, although precise, are time-consuming, costly, and require high implementation and maintenance investments.

A possible alternative to optimize the charcoal characterization process is the NIR technique, which is fast and reliable [13, 14]. In the forestry sector, several studies have shown that this technique can contribute to the monitoring and classifying of raw lignocellulosic material and can also help characterize its products, such as charcoal [15, 16].

For example, Ramalho et al. [17] developed predictive models to classify charcoal produced from native and planted wood and analyze the influence of the final carbonization temperature on the differentiation. The researchers' results were satisfactory, so the percentage of correct classifications ranged from 66 to 100%. Abreu Neto et al. [18] applied NIR spectroscopy to estimate the mechanical properties of charcoal. The authors found suitable parameters for the model to estimate the friability of charcoal, with  $R^2$  of 0.91 for dynamic hardness with a final carbonization temperature of 750 °C. Therefore, the studies reported in the literature addressing the use of NIR spectroscopy in charcoal used the benchtop instrument.

As observed, although scarce, the available studies in literature using NIR spectroscopy on charcoal have provided relevant information regarding the quantitative and qualitative analyses of this material. However, these studies

were conducted using benchtop equipment and, as far as we know, no studies have been carried out on the characterization and classification of charcoal from different sources using portable NIR spectrometers. This represents a gap in scientific knowledge, given that benchtop instruments are calibrated and developed to provide higher resolution and more detailed spectral information, which makes them more expensive. In addition, benchtop devices are more sensitive and require specific operating conditions, which may hinder their use in the field, unlike portable NIR devices [19, 20], which can make the charcoal characterization process more dynamic, optimizing decision-making in the control of the production process.

The portable NIR instrument thus presents itself as a promising alternative, as it offers, in addition to the functionalities provided by benchtop equipment, advantages such as reduced size, lower commercial cost, and the ability to evaluate the desired parameter at the point of use [21–23]. Despite the narrower spectral range of the portable NIR instrument (11.000–6.000  $\text{cm}^{-1}$ ) compared to most benchtop instruments (12.500–3.500  $\text{cm}^{-1}$ ), the spectra resulting from the interaction between the emitted light beam and the analyzed material provide the necessary information to enable the construction of multivariate statistical models [24, 25].

Therefore, this study proposes, as an innovative approach to address scientific gaps, the analysis of *Eucalyptus* and *Corymbia* charcoal parameters using portable NIR spectroscopy to assess charcoal quality. Furthermore, no studies were found that independently use portable NIR spectrometers for the characterization of charcoal derived from *Corymbia* clones. Thus, the hypotheses of this work are as follows: (i) portable NIR spectrometers provide accuracy comparable to benchtop instruments in evaluating charcoal properties; and (ii) both portable and benchtop instruments can accurately estimate the chemical, physical, and energetic properties of charcoal produced from *Eucalyptus* and *Corymbia* clones.

In this context, this study aimed to predict the gravimetric yield, apparent density, fines content, and immediate chemical composition of *Eucalyptus* and *Corymbia* charcoal using benchtop and portable NIR spectrometers. Once the predictive potential of these instruments for such parameters is confirmed, their use could be recommended to the charcoal production industry for quality control of the manufactured material.

## Material and Methods

### Obtainment and Preparation of Vegetal Material

Fifteen commercial clones (11 from the genus *Eucalyptus* and 4 from the genus *Corymbia*) were collected, planted at a spacing of 3 × 3 m and 84 months of age, from plantations

located in the city of Itamarandiba, Minas Gerais (latitude 17° 44' 45" S; longitude 42° 45' 11" W; and altitude 1000 m). Then, the stems of each sampling unit were sectioned to obtain discs in six different longitudinal positions (0, DAP, 25, 50, 75, and 100%), obtaining a composite sample for each clone. Posteriorly, the material was sent for qualitative analyses and carbonization.

**Wood Basic Density**

Samples were collected at 1.30 m above ground level (DBH), and the resulting average of the basic density of two opposite wedges passing through the disc pith with a thickness of 2.5 cm was calculated, following the ABNT NBR 11941 standard [26].

**Wood Carbonization**

After obtaining samples from each clone, the wood was sectioned and dried in an oven at 103 ± 2 °C for 72 h until constant mass. Carbonization was performed in an electric muffle furnace, with approximately 400 g of wood in each cycle, and the sample from each clone was carbonized individually.

The wood samples were inserted into a metal container with nominal dimensions of 0.3 m in length, 0.12 m in diameter, and a volume of 0.003 m<sup>3</sup>, and the container was subsequently fixed to the muffle. The heating system was manually controlled, with an average heating rate of 4.17 °C/min, as shown in Table 1 [27]. The initial and final temperatures were set at 150 and 450 °C, respectively, remaining stabilized at the latter for 60 min, totaling 7 h and 30 min of carbonization. Subsequently, the samples were cooled by natural convection for 16 h.

After each carbonization cycle, the gravimetric yield of charcoal was determined by the ratio between the mass of dry charcoal produced and the mass of dry wood, as shown by Eq. 1:

$$GYC = \frac{mdc}{mdw} \times 100 \tag{1}$$

**Table 1** Heating rate used in the carbonization of genotypes of *Eucalyptus* and *Corymbia*

Steps	Temperature (°C)	Heating rate (°C/min)
1	150	-
2	200	3.33
3	250	4.17
4	350	3.89
5	400	4.44
6	450	5.00

where *GYC* is the gravimetric yield of charcoal (%), *mdc* is the mass of dry charcoal (kg), and *mdw* is the mass of dry wood (kg).

**Characteristics of Charcoal**

The preparation of the samples to determine the immediate chemical composition (volatile materials content, ash, and fixed carbon) and the collection of the spectra were similar. The samples were crushed manually and then classified with 0.42 mm and 0.25 mm sieves. The material analyzed was retained on the sieve with 0.25 mm [28]. The immediate chemical composition of the charcoal was determined according to the ABNT NBR 8112 standard [29]. To determine the volatile material content of the charcoal, the samples were deposited in covered porcelain crucibles and subjected to a muffle furnace stabilized at 950 °C, with 2 and 3 min of acclimatization at the entrance and edge of the muffle, respectively, and 6 min inside the muffle, totaling 11 min of exposure.

The ash content was obtained after the complete combustion of the charcoal after the material was exposed to a temperature of 600 °C for 6 h. The fixed carbon content was obtained by difference, subtracting the volatile material and ash contents from 100. The apparent relative density of charcoal was determined by the hydrostatic method, in which the samples were immersed in mercury, as reported by Vital [30]. The test was performed on charcoal samples with 5% moisture, with seven replicates for each carbonization, so that the average apparent relative density was obtained by the arithmetic average.

The determination of friability was performed with approximately 20 g of charcoal samples, which were subjected to a friabilometer for 14 min at a speed of 35.5 RPM, following the protocol established by the Technological Center of Minas Gerais (CETEC), as mentioned by Oliveira et al. [31]. Subsequently, the charcoal was sieved through 9.5 mm, and the mass loss was calculated.

**Description of the Chemical, Physical, and Energetic Properties of Charcoal**

The charcoal from *Eucalyptus* and *Corymbia* clones was subjected to physical and energy laboratory analyses following the standards and methodologies presented and the results obtained can be seen in Table 2.

**Sample Preparation for Spectra Collection**

Ground charcoal samples for each clone (positions 0, DBH, 25, 50, 75, and 100%) were classified using 0.42 mm and 0.25 mm sieves, using the material retained on the 0.25 mm. Afterward, the samples were kept in a room with

**Table 2** Chemical, physical, and energetic characteristics of charcoal from clones of *Eucalyptus* and *Corymbia*

*CL	GY (%)	ARD (kg/m <sup>3</sup> )	FC (%)	VMC (% db)	AC (% db)	FCC (% db)
C1	33.37 ± 0.79	408.96 ± 8.31	5.60 ± 1.10	22.81 ± 0.15	1.33 ± 0.05	75.86 ± 0.11
C2	36.17 ± 0.39	424.07 ± 4.59	8.57 ± 0.40	23.90 ± 0.23	0.51 ± 0.09	75.60 ± 0.28
C3	34.56 ± 0.15	386.61 ± 4.69	7.92 ± 0.53	21.86 ± 0.87	1.54 ± 0.17	76.60 ± 0.71
C4	33.68 ± 0.30	455.59 ± 14.11	7.55 ± 0.63	22.38 ± 1.69	1.29 ± 0.02	76.34 ± 1.71
E1	33.40 ± 0.94	362.61 ± 4.55	7.57 ± 0.41	21.99 ± 0.83	1.52 ± 0.11	76.49 ± 0.94
E2	34.33 ± 1.43	408.49 ± 13.19	8.80 ± 0.56	21.97 ± 1.56	0.72 ± 0.13	77.32 ± 1.43
E3	33.84 ± 0.92	301.22 ± 13.50	12.54 ± 0.91	23.96 ± 0.93	0.61 ± 0.02	75.43 ± 0.92
E4	35.12 ± 0.19	287.77 ± 10.11	9.90 ± 1.41	24.37 ± 1.42	0.77 ± 0.33	74.86 ± 1.09
E5	34.88 ± 0.52	332.54 ± 3.13	7.77 ± 0.38	23.16 ± 0.58	0.61 ± 0.16	76.23 ± 0.52
E6	35.07 ± 0.02	345.62 ± 47.92	8.85 ± 0.34	22.79 ± 0.61	0.91 ± 0.06	76.29 ± 0.57
E7	34.34 ± 0.03	222.59 ± 6.11	6.51 ± 1.16	23.70 ± 0.28	0.62 ± 0.06	75.68 ± 0.35
E8	34.64 ± 1.13	339.76 ± 11.86	6.79 ± 1.28	22.57 ± 0.22	0.87 ± 0.28	76.57 ± 0.32
E9	35.06 ± 0.45	312.95 ± 2.84	8.40 ± 0.56	21.33 ± 0.43	1.01 ± 0.04	77.66 ± 0.44
E10	34.97 ± 0.05	362.71 ± 7.87	8.21 ± 0.80	23.16 ± 1.24	0.71 ± 0.14	76.13 ± 1.37
E11	34.74 ± 0.42	288.64 ± 34.72	7.98 ± 0.93	24.54 ± 0.42	0.85 ± 0.06	74.61 ± 0.43

\*CL Clone, C *Corymbia*, E *Eucalyptus*, GY gravimetric yield, ARD apparent relative density, FC fines content, VMC volatile material content, AC ash content, FCC fixed carbon content, db dry basis

a temperature of 20 °C and relative humidity of around 65% until they reached an equilibrium moisture of approximately 6% (dry basis).

After adequate sample conditioning, spectral analyses were performed using benchtop and portable NIR spectrometers. In the benchtop equipment, measurements were performed with samples deposited in specific cuvettes compatible with the spectrometer sensor. In the portable equipment, acquisitions were made directly on the material deposited in translucent containers (Petri dishes).

## Spectra Acquisition

The spectra in the NIR region were obtained with a portable and benchtop instrument, according to the procedures described by Medeiros et al. [20]. The benchtop instrument used for the analysis was a Fourier transform NIR spectrometer, model MPA, manufactured by Bruker Optik GmbH (Ettlingen, Germany), operating with the OPUS software version 7.0. The spectra were obtained through diffuse reflection in the integration sphere (12.500–3.500 cm<sup>-1</sup>), with a resolution of 8 cm<sup>-1</sup>, resulting in 1.300 spectral variables. Sixteen scans were performed for each spectrum collected, and the averages of these scans were calculated and compared with the standard to generate the specimen's absorption spectrum. Background compensation was performed every 10 min during the spectral acquisition, and the light emission from the MPA window was shielded. The 9.000–4.000 cm<sup>-1</sup> spectral range was used for the calculations after applying a band selection method to eliminate possible noise.

The portable instrument used was the MicroNIR On-site (Viavi Solutions Inc., CA, USA), connected to a computer to store the spectral data collected in the Spectral Solutions program (Viavi Solutions Inc., CA, USA). The acquisition range was 11.000–6.000 cm<sup>-1</sup> with a resolution of 5.6 nm and 125 spectral variables. Each spectra results from an average of 16 scans in diffuse reflectance mode. Twenty readings were performed per sample so that the sample was homogenized in the interval between each reading, totaling 600 spectra. The NIR spectral signatures were associated with the laboratory analysis data, and multivariate models were developed.

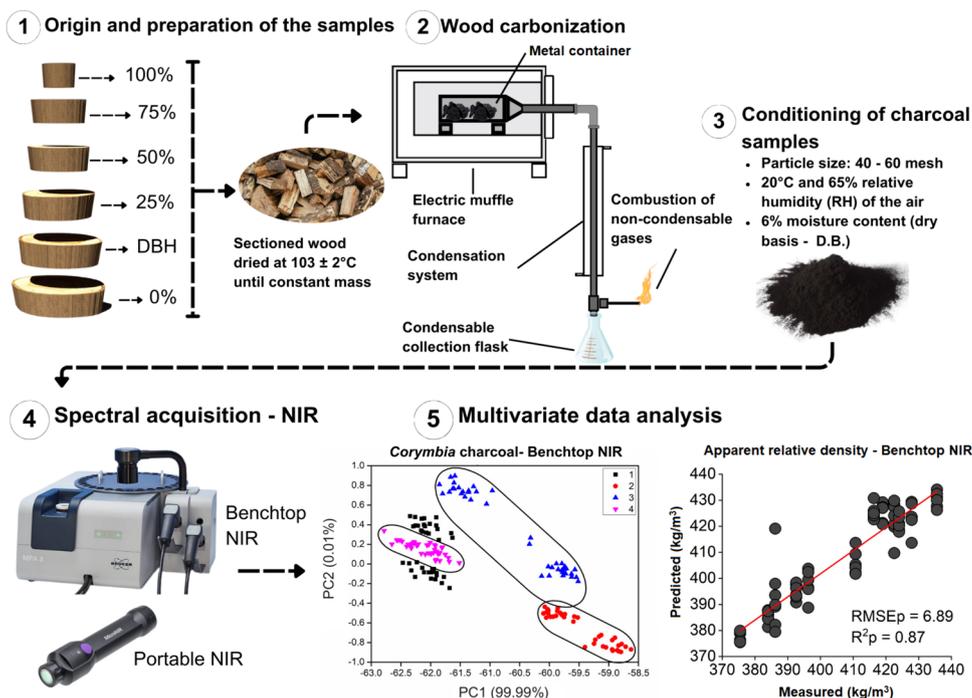
## Multivariate Data Analysis

Principal component analysis (PCA) and partial least squares regression (PLS-R) were fitted based on NIR spectra (Fig. 1).

Chemoface software version 1.63 was used for multivariate data analysis. PCA was applied to evaluate the clustering of charcoal data from *Eucalyptus* and *Corymbia* clones within each genus.

The PLS-R models were adjusted using wood spectra to predict gravimetric yield, apparent relative density, fines content, volatile matter content, ash content, and fixed carbon content. Cross-validation and independent validation (through a set of tests) were used to validate the models. The number of latent variables was defined based on minimizing the standard error and maximizing the coefficient of determination of the validation, resulting in the selection of 8 to 11 latent variables in the adjusted models. The *leave-one-out* method was applied for cross-validation. The data set was

**Fig. 1** Experimental scheme for data collection and processing using NIR spectroscopy



divided into calibration and validation sets for independent validations, using 66.6% of the data for model calibration and the remaining 33.3% for validation, according to the data selection process proposed by Medeiros et al. [20].

The original spectra and those mathematically treated with first derivative (13-point filter and second-order polynomial), normalization, and standard normal variation (SNV) were used to improve the mathematical parameters of the models. The statistical parameters used to select the best prediction models were the calibration coefficient of determination ( $R^2c$ ), mean square error of calibration (RMSEC), cross-validation coefficient of determination ( $R^2cv$ ), root mean square error of cross-validation (RMSEcv), cross-validation deviation performance ratio (RPDcv), root mean square error of prediction (RMSEP), and prediction deviation performance ratio (RPD).

## Results and Discussion

### NIR Spectral Signatures

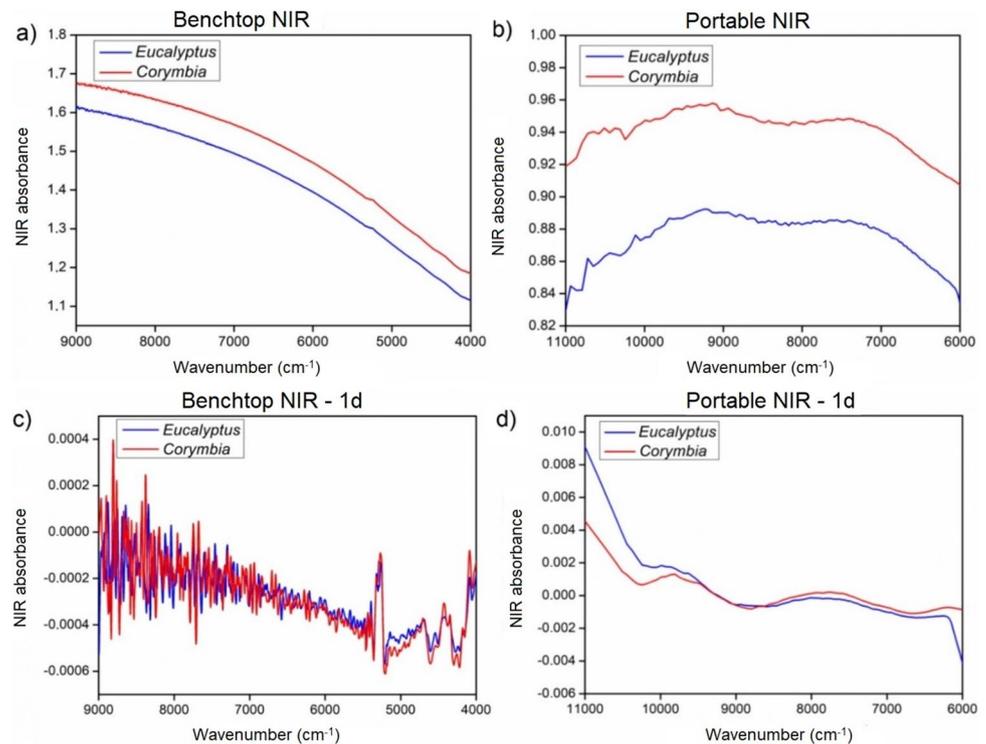
Figure 2 shows the original and first derivative (1d)-treated average spectral signatures for the charcoal dataset of *Eucalyptus* and *Corymbia* clones. The spectra were treated with the first derivative (Fig. 2c and d) for better visualization and identification of the spectral peaks. Thus, similarity was observed between the spectral behavior of the two genera, so the differences found correspond to the intensity of the peaks.

Absorption bands are more intense at specific wavenumbers [6]. These wavenumbers vary according to the chemical components present in the material, whether wood or charcoal. In this study, it was possible to observe that charcoal provided lower NIR absorbance intensity, resulting in greater homogeneity of the spectra and reduction of informative peaks. Similar results were found in the study by Abreu Neto et al. [18] when analyzing the spectral behavior of *Eucalyptus* charcoal using a final carbonization temperature of 450 °C.

After the wood pyrolysis stage, a significant fraction of the structural components are thermally degraded, resulting in more homogeneous charcoal with a higher concentration of carbon in its composition. The study by Ramalho et al. [17] demonstrated that the final carbonization temperature directly influences the spectral behavior of charcoal since the higher the final carbonization temperature, the smaller the absorption bands in the NIR. This fact can be associated with the thermochemical conversion of the main components of wood (cellulose, hemicelluloses, lignin, and extractives), which occurs in different temperature ranges [32, 33]. The final carbonization temperature adopted in this study was 450 °C, resulting in the thermal degradation of a large part of the carbohydrates in the wood.

Figure 2a and c correspond to the benchtop NIR spectra (untreated and treated with first derivative, respectively). The spectra in Fig. 2a showed homogeneous behavior, so that it was not possible to identify peaks related to the organic chemical components of charcoal. However, after treating the spectra with the first derivative (Fig. 2c), it was

**Fig. 2** Average spectral signatures of *Eucalyptus* and *Corymbia* charcoal obtained with benchtop (a untreated spectra; c treated with first derivative) and portable (b untreated spectra; d treated with first derivative) NIR instruments



possible to observe peaks between 7.200 and 5.300  $\text{cm}^{-1}$  more clearly, which can be associated with the vibrational energy of OH stretching [34]. The peak around 9.800  $\text{cm}^{-1}$ , evidenced by Fig. 2b and d (portable NIR spectra), can be related to the second overtone of vibrations of C-H and N-H bonds [35].

Despite the greater homogeneity of the spectral signatures of charcoal of both types, it was possible to observe that the absorbance bands presented interaction in certain absorption bands. Furthermore, although the spectral range of the benchtop and portable NIR presented different intervals, it was possible to identify characteristic peaks of organic chemical constituents in both instruments, which allowed the exploration of various information through appropriate multivariate statistical analyses.

### Principal Component Analysis (PCA)

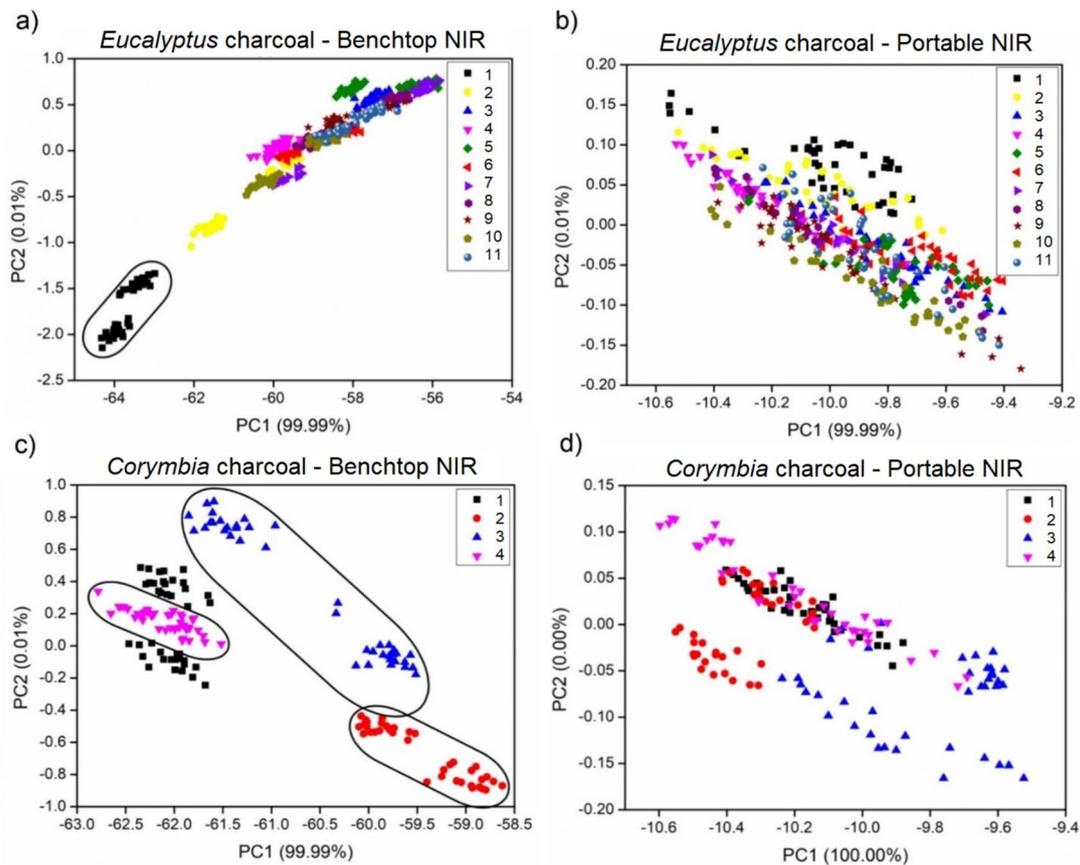
The results indicated variations among charcoals from clones of the same genus since many samples did not overlap (Fig. 3). The benchtop NIR instrument was able to group 1 charcoal from *Eucalyptus* clone (Fig. 3a) and 3 charcoals from *Corymbia* (Fig. 3c). In Fig. 3a and c, a subtle separation of the groups was observed for the same clone in *Eucalyptus* and *Corymbia* (highlighted in black). Several explanations can be attributed to this behavior, one of which is related to the method of sampling wood from the base to the top for charcoal production, indicating greater sensitivity of the benchtop NIR, whose spectral

range is 12.500–3.500  $\text{cm}^{-1}$ , compared to portable NIR (9.000–4.000  $\text{cm}^{-1}$ ), as reported by other studies using NIR for the classification of other wood products [20].

Another aspect is that the differences in the structural molecular composition in the base-top direction of the woods that originated the charcoal are probably more discrepant in relation to the other clones evaluated. These differences, at the time of carbonization, may have generated different chemical structures and/or variations in the amount of compounds, promoting variations in the spectral response in NIR [6, 17].

The untreated and first derivative-treated spectra allowed the formation of linear combinations corresponding to the original variables of the charcoal samples of *Eucalyptus* and *Corymbia* clones. The sum of the principal components (PC1 + PC2) explained 100% of the variability of the raw spectral data of *Eucalyptus* and *Corymbia* charcoal with both instruments.

The greater tendency for charcoal grouping from *Corymbia* clones in relation to *Eucalyptus* clones may be related to the apparent relative density (ARD) values (see Table 1). The ARD values of charcoals from *Corymbia* clones are higher than those of *Eucalyptus* clones, resulting in a greater quantity of carbonized woody material in the same volume. Thus, it is likely that a greater amount of organic chemical constituents is present in charcoals from this genus, which made it possible to group almost all these clones using benchtop NIR.



**Fig. 3** Principal component analysis (PCA) of treated and untreated spectral data obtained from charcoal of *Eucalyptus* and *Corymbia* clones with benchtop (a and c) and portable (b and d) NIR instruments

Regarding the portable NIR instrument, it was not possible to identify the formation of charcoal groups within each genus (*Eucalyptus* and *Corymbia*) based on their spectral signatures. The hypothesis raised to justify this behavior involves the smaller spectral range of the portable NIR compared to the benchtop one, which may have influenced the smaller amount of information in the spectra available for the multivariate analyses. It is worth noting that the portable NIR instrument was not effective specifically for this analysis, which does not prevent its effectiveness for other materials and/or different objectives.

As common aspects for both instruments, it is essential to highlight that a large part of the structural and chemical components of the wood are thermally degraded during the carbonization process, which makes the spectra more homogeneous, with a reduced amount of informative bands and peaks [17]. In addition, the release of water from the wood in the form of vapor during the drying phase of carbonization is another factor that can influence the grouping process of the materials. According to Wang et al. [14], the NIR absorbance is intensified by the greater amount of water since the vibrational energy of the hydroxyl groups is

intensified through electromagnetic radiation in regions of higher moisture content. Therefore, the information available in the spectral signatures is reduced after the carbonization of the wood, which may have made it challenging to group similar materials.

### Partial Least Squares Regression (PLS-R)

Table 3 shows the PLS-R models used to estimate the chemical, physical, and energetic characteristics of charcoal from *Eucalyptus* and *Corymbia* clones with benchtop and portable NIR instruments. Figures 4 (*Eucalyptus* clones) and 5 (*Corymbia* clones) represent the best calibration and independent validation models. The NIR spectra were treated mathematically (first derivative, normalization, and SNV), and the best models were selected, including those without spectra treatment.

Gravimetric yield (GY) is a fundamental parameter for optimizing charcoal production, as it is directly related to the efficiency of the thermochemical conversion of wood into charcoal [2]. Characteristics such as process efficiency, economic impact, sustainability, quality of the

**Table 3** Statistical parameters of PLS-R models for predicting the chemical, physical, and energetic characteristics of charcoal from clones of *Eucalyptus* and *Corymbia*

Genus	Characteristics	Instrument	Treatment	R <sup>2</sup> <sub>cv</sub>	RMSE <sub>cv</sub>	R <sup>2</sup> <sub>p</sub>	RMSE <sub>p</sub>	RPD
				Cross-validation	Independent validation			
<i>Eucalyptus</i>	GY (%)	Benchtop	-	0.67	0.259	0.74	0.213	2.02
		Portable	-	0.65	0.243	0.65	0.244	1.70
	ARD (kg/m <sup>3</sup> )	Benchtop	-	0.82	10.855	0.87	9.160	2.82
		Portable	-	0.78	11.633	0.78	11.385	2.12
	FC (%)	Benchtop	-	0.56	0.476	0.60	0.448	1.60
		Portable	-	0.61	0.523	0.64	0.495	1.60
	VMC (%)	Benchtop	-	0.71	0.159	0.72	0.154	1.92
		Portable	-	0.71	0.203	0.70	0.223	2.03
	AC (%)	Benchtop	*SNV	0.69	0.094	0.72	0.088	1.92
		Portable	SNV	0.68	0.103	0.65	0.107	1.76
	FCC (%)	Benchtop	-	0.63	0.155	0.63	0.155	1.64
		Portable	-	0.66	0.218	0.63	0.225	1.71
<i>Corymbia</i>	GY (%)	Benchtop	-	0.74	0.187	0.79	0.165	2.15
		Portable	-	0.61	0.222	0.58	0.232	1.60
	ARD (kg/m <sup>3</sup> )	Benchtop	-	0.82	8.122	0.87	6.893	2.77
		Portable	-	0.67	12.033	0.62	14.615	2.45
	FC (%)	Benchtop	-	0.72	0.613	0.69	0.645	1.73
		Portable	-	0.65	0.662	0.63	0.692	1.68
	VMC (%)	Benchtop	-	0.53	0.215	0.54	0.214	1.47
		Portable	-	0.50	0.336	0.64	0.371	1.40
	AC (%)	Benchtop	SNV	0.60	0.050	0.61	0.048	1.62
		Portable	SNV	0.57	0.058	0.57	0.067	1.52
	FCC (%)	Benchtop	-	0.54	0.227	0.58	0.251	1.33
		Portable	-	0.55	0.348	0.61	0.374	1.48

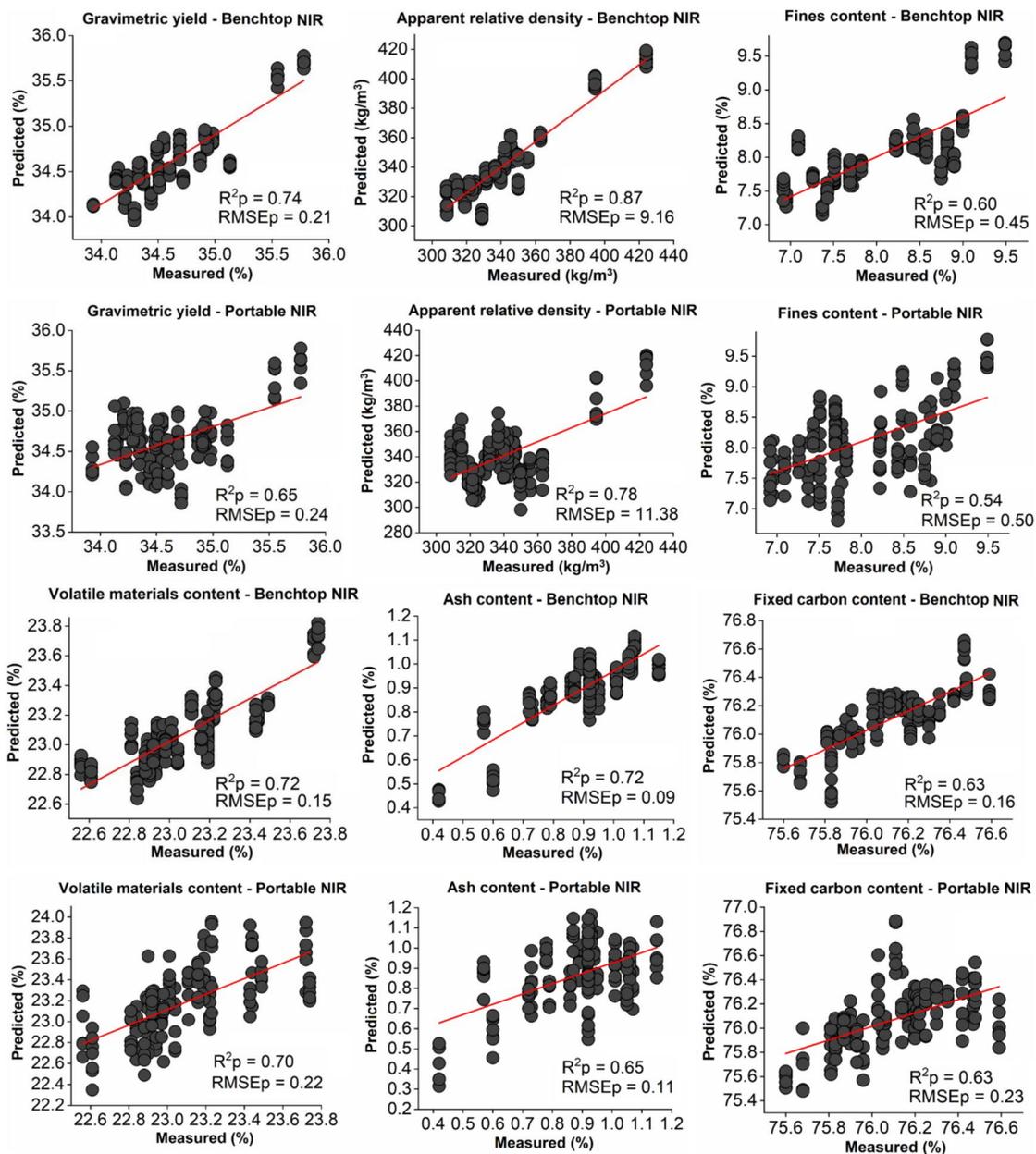
\*SNV standard normal variation. R<sup>2</sup><sub>cv</sub> coefficient of determination for cross-validation. RMSE<sub>cv</sub> mean squared error for cross-validation. R<sup>2</sup><sub>p</sub> coefficient of determination for independent validation. RMSE<sub>p</sub> mean squared error for independent validation. RPD performance deviation ratio for independent validation

final product, and process control are directly influenced by the GY of the wood carbonization process [36]. Thus, maximizing this parameter is desirable to ensure the efficiency and profitability of charcoal production units. The best models for predicting GY presented an R<sup>2</sup><sub>p</sub> of 0.74 and an RPD of 2.02 (benchtop NIR), an R<sup>2</sup><sub>p</sub> of 0.65, and an RPD of 1.70 (portable NIR) for charcoal from *Eucalyptus* clones with spectra without mathematical treatment.

The best models for charcoal from *Corymbia* clones presented R<sup>2</sup><sub>p</sub> of 0.79 and RPD of 2.15 (benchtop NIR) and R<sup>2</sup><sub>p</sub> of 0.58 and RPD of 1.60 (portable NIR) with the spectra without mathematical treatment. The study by Costa et al. [6], when evaluating the quality of *Eucalyptus* charcoal by NIR spectroscopy, found superior results for GY about this study, with R<sup>2</sup><sub>cv</sub> values of 0.85 and RPD of 2.57 for the original spectral data and varied final carbonization temperatures (400, 500, 600, and 700 °C). Despite this, the results found in our study were satisfactory, except for charcoal from *Corymbia* clones using portable NIR.

The apparent relative density (ARD) directly affects the industrial processes that use charcoal as a source of raw material. The higher the apparent density, the higher the relative density. The mechanical resistance and the amount of charcoal that can be stored in a smaller space are greater, facilitating the storage and transportation of these materials [37]. Furthermore, the higher ARD improves the quality of the charcoal because it generally presents a greater amount of fixed carbon, resulting in a greater amount of energy released during the burning process and optimizing the production of pig iron and steel [8]. Thus, the best models for predicting ARD presented R<sup>2</sup><sub>p</sub> of 0.87 and RPD of 2.82 (benchtop NIR) and R<sup>2</sup><sub>p</sub> of 0.78 and RPD of 2.12 (portable NIR) for charcoal from *Eucalyptus* clones with spectra without mathematical treatment.

The best models for charcoal from *Corymbia* clones presented R<sup>2</sup><sub>p</sub> of 0.87 and RPD of 2.77 (benchtop NIR) and R<sup>2</sup><sub>p</sub> of 0.62 and RPD of 2.45 (portable NIR) with spectra without mathematical treatment. Abreu Neto et al. [18], when analyzing the hardness and apparent density of charcoal from



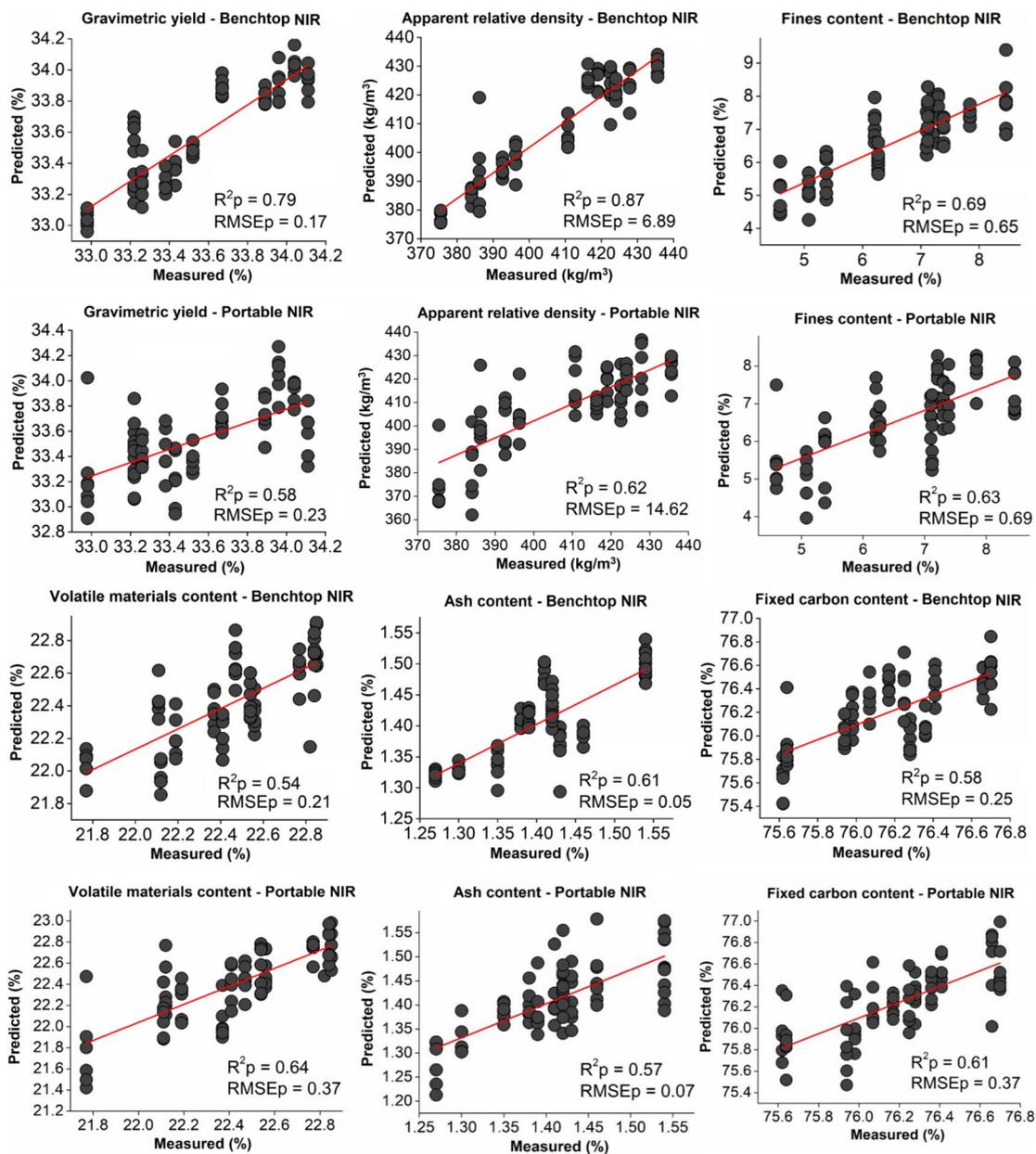
**Fig. 4** PLS-R validation graphs for predicting charcoal characteristics from *Eucalyptus* clones using benchtop and portable NIR instruments

clones of *Eucalyptus* sp. and *Corymbia citriodora* together, did not find satisfactory values for apparent density ( $R^2_{cv}$  of 0.49 and RPD of 1.40) with original spectral data and a final carbonization temperature of 450 °C. These results reinforce the promising results found in our study for the models of this variable.

In the wood carbonization process, charcoal is desirable to be less friable to reduce the amount of fine particles generated during handling and transportation to the steel companies, which can increase the energy released in the form of heat [5]. In addition, charcoal’s low fine content (FC) can increase the permeability of the charge bed in the

blast furnace, positively influencing the efficiency of the steelmaking process [38, 39]. Therefore, the best models for predicting the FC presented  $R^2_p$  of 0.60 and RPD of 1.60 (benchtop NIR) and  $R^2_p$  of 0.64 and RPD of 1.60 (portable NIR) for charcoal from *Eucalyptus* clones with spectra without mathematical treatment.

For charcoal from *Corymbia* clones, the best models presented  $R^2_p$  of 0.69 and RPD of 1.73 (benchtop NIR) and  $R^2_p$  of 0.63 and RPD of 1.68 (portable NIR) with the spectra without mathematical treatment. Although no study registered in the literature has developed statistical models to predict this parameter based on the NIR spectra, the statistics



**Fig. 5** PLS-R validation graphs for predicting charcoal characteristics from *Corymbia* clones with benchtop and portable NIR instruments

found in this study were considered satisfactory for both the genus and the instruments tested. This result was considered one of the differentials of this study due to the novelty of the information, which may be of interest to charcoal-producing units, given the relevance of this parameter within the charcoal production and transportation chain.

The volatile material content (VMC) is another fundamental parameter in charcoal production. The literature recommends that charcoal should present VMC between 22 and 25% by mass for steelmaking use. Levels above this range result in lower fixed carbon contents, directly affecting charcoal's energy availability [40, 41]. Thus, the best

models for predicting TMV presented  $R^2_p$  of 0.72 and RPD of 1.92 (benchtop NIR) and  $R^2_p$  of 0.70 and RPD of 2.03 (portable NIR) for charcoal from *Eucalyptus* clones with spectra without mathematical treatment.

The best models for charcoal from *Corymbia* clones presented  $R^2_p$  of 0.54 and RPD of 1.47 (benchtop NIR) and  $R^2_p$  of 0.64 and RPD of 1.40 (portable NIR) with spectra without mathematical treatment. Andrade et al. [42] analyzed the properties of charcoal from *Eucalyptus* using NIR spectroscopy. They found superior results in relation to those found in this study, with  $R^2_p$  values of 0.91 and RPD of 2.94 with spectra treated with first derivative and final carbonization

temperatures of 350, 450, 550, and 900 °C. However, the results found in our study were satisfactory, except for the charcoal from *Corymbia* clones using benchtop NIR.

Lower ash content (AC) is desirable for charcoal since the higher the ash content, the lower the calorific value [43]. Thus, selecting clones with lower AC can be an essential factor in charcoal production since it can corroborate the increase in energy per volume of charcoal [44]. Thus, the best models for predicting AC presented  $R^2_p$  of 0.72 and RPD of 1.92 (benchtop NIR) and  $R^2_p$  of 0.65 and RPD of 1.76 (portable NIR) for charcoal from *Eucalyptus* clones with spectra treated with SNV.

For charcoal from *Corymbia* clones, the best models presented  $R^2_p$  of 0.61 and RPD of 1.62 (benchtop NIR) and  $R^2_p$  of 0.57 and RPD of 1.52 (portable NIR) with spectra treated with SNV. The results found in this study for AC were acceptable, although the model did not present a high correlation. However, Ramalho et al. [19], when evaluating the influence of particle size on the estimates of the properties of charcoal from *Eucalyptus* sp., did not find satisfactory results for this parameter, with  $R^2_c$  values of 0.38 and RMSEcv of 0.058 for fine charcoal powder, with spectra without mathematical treatment and a final carbonization temperature of 450 °C. The authors highlighted that the difficulty in finding satisfactory results for this parameter may be associated with the composition of the ash since these materials are inorganic, and NIR spectroscopy is recommended for organic materials.

The fixed carbon content (FCC) directly influences charcoal's efficiency, quality, and sustainability since the higher the FCC, the greater the energy efficiency and durability of the charcoal due to the increased combustion process time [45]. From a sustainability point of view, charcoals with a higher FCC tend to emit fewer polluting gases during combustion because burning is more efficient, releasing fewer substances such as carbon monoxide (CO), carbon dioxide (CO<sub>2</sub>), and other volatile compounds [46]. Thus, the best models for predicting the FCC presented  $R^2_p$  of 0.63 and RPD of 1.64 (benchtop NIR) and  $R^2_p$  of 0.63 and RPD of 1.71 (portable NIR) for charcoal from *Eucalyptus* clones with spectra without mathematical treatment.

For charcoal from *Corymbia* clones, the best models presented  $R^2_p$  of 0.58 and RPD of 1.33 (benchtop NIR) and  $R^2_p$  of 0.61 and RPD of 1.48 (portable NIR) with the spectra without mathematical treatment. Similar results were found in Ramalho et al. [19]. The authors found the FCC values of  $R^2_{cv}$  of 0.60 and RMSEcv of 1.402 for charcoals retained in the 0.25-mm screen. Spectra were treated with normalization, and the final carbonization temperature was 450 °C.

In the general context, it was observed that the models of charcoals from *Eucalyptus* clones presented superior results to those of *Corymbia*. Despite the same carbonization conditions of both genera, the structural chemical composition

becomes more homogeneous due to the thermodegradation of a significant fraction of the components. Differences concerning the statistical parameters of the models were observed. A possible explanation may be associated with the number of clones used in constructing the models. The models estimated that the genus *Eucalyptus* used eleven different clones, while only four were used for the genus *Corymbia*. According to Zhu et al. [25], the greater the variability between the components and the number of samples available, the better the statistical parameters of the models.

Regarding the portable NIR instrument, it was observed that some parameters were higher and others lower than the benchtop one. The same trend was observed when comparing the results of other studies reported in the literature. As previously stated, studies on charcoal using NIR spectroscopy are scarce, and no study has been carried out using portable NIR instruments. Despite this, most of the models generated were classified as adjusted and would be suitable for use. It should be noted that this study has some limitations regarding the environmental conditions in which it was carried out.

The samples evaluated were controlled at the laboratory level, presenting significant differences in relation to the charcoal-producing units. Another important aspect is related to the particle size of charcoal. This study used ground and sieved charcoal (0.42 mm and 0.25 mm), presenting significant differences in relation to the charcoal commercialized by the charcoal-producing industries. Therefore, future studies are necessary to fill these gaps in scientific knowledge.

## Conclusion

The benchtop and portable NIR instruments showed promising results in almost all the chemical, physical, and energetic characteristics of the charcoal of both genera analyzed. Thus, these instruments can be valuable tools for charcoal-producing units since the evaluation of charcoal quality can be optimized, resulting in time and operating cost savings.

The PCA allowed the distinction of one clone of *Eucalyptus* and three clones of *Corymbia* using the benchtop NIR. With the portable NIR instrument, no charcoal clone was grouped within each genus through this analysis. Following the pre-established parameters to evaluate the models' predictive capacity for both instruments, it was observed that the models presented variation in precision, with the best model presenting an  $R^2_p$  of 0.87.

The models obtained for charcoal from *Eucalyptus* clones showed similar results with both instruments, which may favor the portable NIR due to its lower cost and easier operation. However, for *Corymbia* charcoals, the portable

NIR models showed poorer performance compared to the benchtop.

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**Data Availability** The datasets supporting the findings of this study are included within the manuscript. Additional data analyzed during this research are available from the corresponding author upon reasonable request.

## Declarations

**Ethical Approval** Not applicable.

**Consent to Participate** The authors' participation was authorized during the construction of the study.

**Consent for Publication** All authors agree with the publication of the article in this journal.

**Conflict of Interest** The authors declare no competing interests.

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