Research Article

Charcoal anatomy and NIR spectra of *Spirostachys africana*, *Terminalia* sp. and *Colophospermum mopane* in different carbonization process

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Abstract

In Mozambique, charcoal processes can result in several disturbances in natural forests. In the present study, we evaluated the effect of four carbonization methodologies on the anatomical characteristics and near infrared spectra of *Spirostachys africana*, *Terminalia* sp. and *Colophospermum mopane*, to contribute with information for an identification database for charcoal control. The carbonization was done in an electric muffle furnace with different final temperature (400/450 °C) and total time (40 min/2 h, 4 h, 6 h). The quantitative changes in anatomy of charcoal samples was not directly related to carbonization programs with respect to particular features of each species. Species are best distinguished for the NIR spectra in the region from 4000 to 5000 cm⁻¹. In the analysis by species, charcoal produced at 450 °C—4 h were more distinct in comparison to other programs that resulted in mixed samples. Final temperature influenced more than total process time in species distinction and the use of NIR is reinforced as a potential tool to discriminate species submitted to different carbonization processes. These traits can be applied in species discrimination based on databases of wood anatomical descriptions and charcoal NIR spectra.

Keywords Nature forest · Wood anatomy · Carbonization · Spectroscopy · Africa

1 Introduction

Charcoal production in Africa mainly depends on natural forests [1]. In urban centers of Mozambique, charcoal is the major energy source for cooking, and growing demand is responsible for high wood extraction rates from miombo and mopane woodlands. The process can contribute with changes in ecosystem and woodland degradation [2], due to people who produce charcoal to increase income, which normally is against current Mozambican law [3].

Planting fast-growing wood species on degraded sites and the improvement of carbonization techniques can

be a promising alternative to reduce the pressure on the remaining natural forest [1, 4]. Integrated land use management is necessary in order to ensure sufficient regeneration of woodland species valuable for the livelihood and the ecological value of the region [5].

The carbonization conditions affect charcoal quality [6] and suitability for different purposes, such as energy, soil improvement for farming and absorbance of pollutants [7]. Afonso et al. [8] analyzed charcoal properties of nine species with high commercial value in Mozambique and their results indicated similar quality comparing with that

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made from *Eucalyptus* species. Another alternative for producing charcoal is from biomass waste [9].

The anatomical features of wood remain largely intact after carbonization, so even though some changes are observed in parenchyma, vessels and fibers [10], identification can still be accomplished based on species anatomical characteristics, principally comparing with a database [11]. Also, application of near infrared spectroscopy (NIR) has been evaluated. Studies indicate the possibility of distinguishing carbonized material from native and planted forests [12, 13], some carbonization characteristics [14, 15] and also species discrimination [16–18]. Final results must be compared, and so, a database is necessary in species distinction by anatomy and spectroscopic techniques.

In light of the foregoing, this study anatomically characterizes charcoal from *Spirostachys africana*, *Terminalia sp.* and *Colophospermum mopane* produced with different carbonization processes. The main goal is to evaluate the influence of carbonization parameters on charcoal anatomy by optical and scanning electron microscopy, and near infrared spectroscopy, to provide information to a database for charcoal discrimination and control.

2 Materials and methods

Samples were obtained randomly from sawmills in Mozambique (Table 1). Boards without details of tree age or trunk position (heartwood, transition or sapwood; basal or apical portion), were divided and three samples of 25 × 25 × 50 mm (transverse, longitudinal radial and longitudinal tangential sections) from each species were carbonized by each process. The wood was collected based on popular name in sawmills, after anatomical structure was analyzed and identification was confirmed in Inside Wood [19] online key for wood identification. Wood and charcoal evaluation was performed in the same samples.

2.1 Carbonization process

The samples, covered with aluminum foil, were carbonized in an electric muffle furnace with four different parameters: P1, final temperature of 400 °C and total time 40 min

[22]; P2, with 450 °C—2 h [23]; P3, with 450 °C—4 h [24]; P4, with 450 °C—6 h [25]. The final temperature was chosen by following the temperature traditionally employed by local people for charcoal production. Samples were cooled at the furnace, then first evaluated at near infrared. P1, with different time and temperature, will have lower degradation in comparison to others, and so its anatomical and chemical information are important for the database.

2.2 Anatomical characterization

For anatomical analysis, charcoal was hand broken following the three surfaces of the wood and the morphological characteristics were analyzed with a Zeiss Discovery v12 stereo microscope. The description was done in accordance with the recommendations of the IAWA Committee [26]. Images with details of charcoal surfaces were obtained by scanning electron microscopy (SEM) with direct observation of material on a Hitachi TM-1000 tabletop microscope. Quantitative anatomical features data were obtained based on the average of 25 measurements of vessel and ray frequency, tangential diameter of vessels, height and width of rays.

Statistical analysis compared charcoal produced with different carbonization programs. When normal distribution of data was verified, mean values were compared by the Scott-Knott test at 5% of probability, while the Kruskal–Wallis test at 5% was applied otherwise.

2.3 Near-infrared spectroscopy

Infrared analyses were performed, in a room with temperature of 23 ± 2 °C and relative humidity of 60%, with a Bruker Tensor 37 spectrometer equipped with an integrating sphere and operating in reflectance mode with resolution of 4 cm⁻¹ and a spectral range of 10,000–4000 cm⁻¹. The charcoal samples were placed on top of the integrating sphere and three spectra were obtained from each surface (transverse, longitudinal radial and longitudinal tangential sections), resulting in a total of 9 separate spectra for each sample and 27 for each species. Spectra from each surface were averaged and analysis was performed with three spectra per sample, and nine per species in

Table 1 Nomenclature of studied species. Source: Ministério da Agricultura [20], Coates-Palgrave [21]

Scientific name—family	Commercial name	Commercial class ^a
<i>Spirostachys africana</i> Sond.—Euphorbiaceae	Sandalo	Precious
<i>Terminalia sp.</i> —Combretaceae	Messinge	Second
<i>Colophospermum mopane</i> (J. Kirk ex Benth.) J. Léonard—Fabaceae Caesalpinioideae	Mopane	Fourth

^aAttributed for wood commerce based on rarity, commercial and scientific value, utility, resistance and quality

each carbonization process. Also, the influence of surface on species distinction was evaluated, applying: (1) mean spectra by sample; (2) mean only of transverse sections; (3) mean only of radial and tangential longitudinal sections.

The Unscrambler X chemometric program (version 10.1) was used to analyze the data. Exploratory modeling was done by visual analysis of the score and loading graphs obtained by principal component analysis (PCA) to verify possible differences/grouping in charcoal in function of carbonization process or species. PCA was performed based on NIPALS algorithm, cross validation method random with 3 segments. To verify the results, a Linear Discriminant Analysis was done with PCA scores from each species and program parameters, based on linear distance, projecting 2 components assuming equal prior probabilities. After confusion matrix was evaluated. Second derivative of Savitzky-Golay (polynomial order = 2, smoothing point = 21) was applied to raw data.

3 Results

3.1 Charcoal anatomy

3.1.1 *Spirostachys africana* (Fig. 1)

Growth rings: few distinct, demarcated by thick-walled fibers. **Vessels:** diffuse-porous; solitary and in radial multiples of 2-4; simple perforation plate; intervessel pits alternate; presence of gums and other deposits in heartwood vessels. **Axial parenchyma:** diffuse and in some regions in narrow bands, up to three cells wide. **Rays:** uniseriate; heterogeneous, body cells procumbent with one row of upright or square marginal cells; not storied. **Fibers:** thick-walled, non-septate. **Mineral inclusions:** prismatic crystals in ray and axial parenchyma cells.

Fig. 1 SEM images of *S. africana* charcoal: transverse (a, e, i, l, m), longitudinal tangential (b, c, f, g, j, n, o) and longitudinal radial (d, h, k, p) sections. (AP) axial parenchyma, (G) gum in vessels, (R)

uniseriate rays, (IP) alternate intervessel pits, (PP) simple perforation plate, (C) crystals, (GR) growth rings. Scale bar = 100 µm

3.1.2 *Terminalia* sp. (Fig. 2)

Growth rings: distinct, demarcated by thick-walled fibers. **Vessels:** diffuse porous; solitary and in radial multiples of 2–3; simple perforation plate; intervessel pits alternate. **Axial parenchyma:** unilateral, vasicentric and confluent. **Rays:** homogeneous, multiseriate (mostly 2 to 3-seriate, uniseriate also present, not storied). **Fibers:** thin to thick-walled, non-septate. **Mineral inclusions:** crystals in axial parenchyma cells.

3.1.3 *Colophospermum mopane* (Fig. 3)

Growth rings: distinct, demarcated by thick-walled fibers. **Vessels:** diffuse-porous; solitary and in radial multiples of 2–3; simple perforation plate; intervessel pits alternate, vestured; presence of gums and other deposits in heart-wood vessels. **Axial parenchyma:** aliform and confluent.

Rays: homogenous, all ray cells procumbent, 1 to 3-seriate; not storied. **Fibers:** thick-walled, non-septate. **Mineral inclusions:** prismatic crystals in axial and radial parenchyma.

The low temperature and time of carbonization applied in P1 did not produce charcoal from wood of this species.

3.1.4 Quantitative characteristics

Mean values of anatomical characteristics of the three species showed different tendency after carbonization process (Table 2).

1. Vessels

Tangential diameter of vessels, in *S. africana* and *Terminalia* sp., were influenced by temperature and time of carbonization forming two groups: P1 and P2; and P3 and P4. For *C. mopane*, the highest temperature produced charcoal

Fig. 2 SEM images of *Terminalia* sp. charcoal: transverse (a, d, e, i, m), longitudinal tangential (b, c, f, g, j, k, n, o) and longitudinal radial (h, l, p) sections. (GR) growth rings, (R) rays, (IP) alternate

intervessel pits, (AP) axial parenchyma, (V) vessel, (PP) simple perforation plate. Scale bar = 100 μ m

Fig. 3 SEM images of *Colophospermum mopane* charcoal: transverse (a, b, e, f, i, j) and longitudinal tangential (c, d, g, h, k, l, m) sections. (V) vessels, (C) crystals, (R) multiseriate rays, (IP) alternate intervessel pits, (G) gum in vessels. Scale bar = 100 μ m

with larger vessel diameter. Variation in vessel tangential diameter from P1 to P4 showed decrease of approximately 11% in *S. africana* and 10% in *Terminalia* sp.

Evaluating vessel frequency, in *S. africana* charcoal, highest value occurred in P3 and lowest in P1, while the other carbonization programs were similar. In *Terminalia* sp., P4, produced higher vessel frequency, probably associated with contraction of fiber cells, parameter not evaluated in the present work. For *C. mopane*, the highest temperature produced charcoal with lower vessel frequency. Variation in vessel frequency from P1 to P4 showed increases of approximately 13% in *S. africana* and 42% in *Terminalia* sp., while a decrease of 28% in *C. mopane* was verified from P2 to P4.

2. Rays

Ray height, in *S. africana* and *Terminalia* sp., was influenced by the carbonization program, indicating the formation of two groups: P1 and P2; and P3 and P4. For *C. mopane*, no influence of time and carbonization temperature was verified in ray height. The variations in ray height from the lowest temperature and/or time to the highest were a decrease of 26% in *S. africana*, and increases of approximately 10% in *Terminalia* sp. and 14% in *C. mopane* (P2–P4).

In ray width, increases were associated to the carbonization systems in *S. africana*. *Terminalia* sp. presented the opposite behavior, with decrease of cell dimensions. For *C. mopane*, the systems had no influence. The variations in ray width from P1 to P4 were an increase of 61% in *S. africana* and a decrease of 37% in *Terminalia* sp.

For ray frequency, in *S. africana*, it decreased with higher temperature and longer carbonization time. In contrast, *Terminalia* sp. was not influenced. For *C. mopane*, P3 produced the highest values. A decrease of approximately 31% in *S. africana* and increase of 9% in *Terminalia* sp. occurred in charcoal produced from lowest time and temperature (P1) to the highest parameters (P4), and a decrease of 7% in *C. mopane* occurred from the lowest time (P2) to the highest (P4).

3.2 Near-infrared spectroscopy

Raw spectra presented some noise and were not suitable to distinguish species and programs. Hence, second-derivative pretreatment was applied. The mean second-derivative NIR spectra of each species with each program indicated the wavenumbers with greatest distinction between them, from 4000 to 5000 cm^{-1} (Fig. 4). Some regions showed different behavior, principally for *S. africana* carbonized at 400 °C for 40 min.

Table 2 Mean value and (standard deviation) of anatomical characteristics in function of species and carbonization program

Species	Program code			
	P1 400 °C—40 min	P2 450 °C—2 h	P3 450 °C—4 h	P4 450 °C—6 h
Vessel tangential diameter (μm)				
<i>Spirostachys africana</i>	54.16 a (8.0)	54.21 a (7.7)	48.48 b (10.7)	48.12 b (11.6)
<i>Terminalia</i> sp.	100.00 a (14.5)	100.43 a (19.3)	90.84 b (10.9)	89.49 b (14.3)
<i>Colophospermum mopane</i>	No charcoal	68.08 b (11.2)	70.21 b (11.9)	81.51 a (16.7)
Vessels per mm ²				
<i>Spirostachys africana</i>	19.03 c (5.9)	22.37 b (4.5)	27.87 a (4.9)	21.57 b (5.0)
<i>Terminalia</i> sp.	17.03 b (4.0)	17.93 b (5.2)	15.87 b (3.4)	24.17 a (4.7)
<i>Colophospermum mopane</i>	No charcoal	50.27 a (15.6)	53.57 a (15.38)	36.00 b (12.9)
Ray height (μm)				
<i>Spirostachys africana</i>	190.04 a (62.9)	192.07 a (75.5)	152.28 b (53.2)	140.81 b (54.46)
<i>Terminalia</i> sp.	187.35 b (31.3)	196.82 b (51.6)	220.67 a (41.6)	206.05 a (37.3)
<i>Colophospermum mopane</i>	No charcoal	189.90 a (38.6)	182.83 a (51.2)	216.40 a (79.3)
Ray width (μm)				
<i>Spirostachys africana</i>	8.78 c (1.9)	8.96 c (1.7)	11.34 b (3.4)	14.12 a (4.54)
<i>Terminalia</i> sp.	40.09 a (6.1)	34.11 b (7.3)	32.73 b (6.5)	25.17 c (6.1)
<i>Colophospermum mopane</i>	No charcoal	18.21 a (4.1)	20.79 a (4.6)	18.28 a (6.8)
Rays per mm				
<i>Spirostachys africana</i>	18.87 a (2.9)	15.50 b (2.6)	15.97 b (2.5)	13.07 c (2.5)
<i>Terminalia</i> sp.	8.17 a (1.6)	8.07 a (1.8)	8.10 a (2.0)	8.90 a (1.9)
<i>Colophospermum mopane</i>	No charcoal	8.90 ab (2.0)	9.93 a (2.1)	8.27 b (2.2)

Same letters in the row indicate no difference between programs for each characteristic at 5% probability by the Scott-Knott or Kruskal–Wallis test

The analysis of NIR spectra based on each species (Fig. 5) revealed differences in function of its anatomical and chemical characteristics. Material carbonized at 450 °C—4 h showed a good distinction in all species. In *S. africana* (Fig. 5a) and *Terminalia* sp. (Figure 5c), samples carbonized at 400 °C are distinct from other programs, and in *C. mopane* (Fig. 5e), all carbonization programs are distinct. As expect, material carbonized at P1 (400 °C—40 min), with less degradation in each species, could be distinguished.

When the analysis was carried out with all species and programs (Fig. 6; Table 3), the same behavior was observed. Evaluating all material (Fig. 6a), samples from P3

carbonization program exhibited good distinction from all other programs, and *S. africana* from 400 °C to 40 min are also distinct. Bands at 4688–4630 cm⁻¹; 4480–4457 cm⁻¹; 4413 cm⁻¹; 4322 cm⁻¹; 4123 cm⁻¹; 4075 cm⁻¹ presented more influence in species and program discrimination (Fig. 6b). In this analysis, *S. africana* and *Terminalia* sp. carbonized at 400 °C—40 min were influenced by intrinsic characteristics of material. Generally lower changes in chemistry and anatomical degradation were expect as a function of temperature and time. *C. mopane* did not form charcoal.

Evaluating the discrimination of species individually by program (Fig. 7), the results indicated that with lower

Fig. 4 Second-derivative NIR spectra of the three species in different carbonization processes

time and temperature, P1 (Fig. 7a) and P2 (Fig. 7b), *S. africana* could be easily identified, while with other programs, samples are mixed with others, i.e. symbols from all species are grouped, indicating the influence of species degradation.

Evaluating the influence of anatomical section were spectra was obtained (Fig. 8; Table 4), in all cases, material carbonized with P3 and *S. africana* from P1, showed adequate discrimination from other programs. In the case of species, longitudinal sections showed better distinction of all species and programs.

4 Discussion

In Mozambique, species classified by Ministério da Agricultura [20] from precious to third class (including two of the species studied, *S. africana* and *Terminalia* sp.) are not allowed to be utilized for energy but are frequently applied in commerce. Standard techniques produce reliable quality control and the identification techniques, wood anatomy and NIR spectroscopy, require a database. During the practical application of inspections, variations in carbonization programs can interfere with the data.

Colophospermum mopane charcoal is not produced when the high density wood is heated at 400 °C for 40 min due to chemical changes in the cell wall during the carbonization process [6, 7]. At the lower time and temperature of P1 treatment, degradation is not expected. For NIR spectra, final temperature is constant and total process time varies.

4.1 Charcoal anatomy

1. Vessels

Variations in vessel diameter were different among the species, resulting from degradation of cell wall chemical substances, and can be influenced by rays, as observed by Kwon et al. [27], who reported vessel shrinkage in tangential direction higher than in radial direction at carbonization temperatures varying from 350 to 500 °C in *Quercus variabilis* samples. In other Mozambican species, Nisgoski et al. [18] verified the influence of species intrinsic characteristics in changes in vessel diameter.

Other authors described the influence of species in changes in vessel dimension, in relation to fiber wall thickness and axial parenchyma type [16, 22]. Evaluating carbonization processes with diverse heating rates and temperature from 350 to 650 °C, Nisgoski et al. [28] related no influence of temperature on vessel diameter, but Gonçalves et al. [22], evaluating Cerrado species in Brazil, concluded that vessel dimensions was the character most affected in wood by carbonization. One possibility is the alteration in the form of cells, which can become more angular instead of circular in charcoal [10].

As expected, modifications in vessel frequency were different in the species, since final carbonization temperature can also directly influence porosity [29]. In a review of factors affecting mechanics of carbonized wood, Assis et al. [6] reported that total porosity increases directly with temperature until 600 °C, and even up to 1000 °C under slow heating rates due to the release of volatile matter and formation of cracks and voids. There is a linear relation

Fig. 5 PCA scores of second-derivative NIR spectra in the region 4000–5000 cm⁻¹ for each species in different carbonization processes (**a**, **c**, **e**) and respective plots of mean spectra (**b**, **d**, **f**)

with temperature increase and total micropore volume and specific surface area of charcoal [30].

There is an opposite relation between modifications in vessel diameter and their frequency. Generally, if carbonization parameters cause vessels to contract, their

frequency increases, as observed in this study, but it is also related to species intrinsic characteristics. Gonçalves et al. [22], observed no significant changes in vessel frequency and Stange et al. [31] verified increased vessel frequencies of 92% in *Eugenia pyriformis* and 51% in *Myrcia retorta*.

Fig. 6 PCA scores with second-derivative NIR spectra (a) and loading graph (b) of all species and carbonization programs in the region 4000–5000 cm⁻¹

The impact of species is closely related with the vessels' performance, as can be noted in previous studies and as described by Gonçalves and Scheel-Ybert [11]. Nisgoski et al. [18] verified no linear relation from vessel frequency and different parameters of carbonization, being the intrinsic characteristics of species more important in this analysis.

2. Rays

Results related to changes in ray height are associated with species characteristics, as also reported by Nisgoski et al. [28] for *Ocotea porosa* in carbonization programs with final temperature varying from 350 to 650 °C, and

in carbonization process with temperature of 450 °C by Nisgoski et al. [32] and Stange et al. [31]. In other Mozambican species, in the same carbonization parameters of this study, Nisgoski et al. [18] described decrease in ray height in *Sterculia quinqueloba* from 60 to 37% (P2–P4) and in other species verified increase in P1 and decrease in P2.

Ray structure and cell number in width influence the results of the carbonization process. Kim and Hanna [33] verified, in oak samples, that in uniseriate rays little distinction of cells occurred, and morphological characteristics remained largely intact, while in multiseriate rays, retraction cracks and cell wall disintegration were observed. Also, thickness of fiber cell wall can also affect the ray width. The literature reports modifications as well as no

Table 3 Confusion matrix based on PCA scores, with second-derivative NIR spectra of all species and carbonization programs in the region 4000–5000 cm^{-1}

Species/programs	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>
	P1		P2			P3			P4		
<i>S. africana</i> —P1	7	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P1	0	4	4	0	0	0	0	0	2	1	0
<i>S. africana</i> —P2	0	5	5	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P2	0	0	0	9	0	0	0	0	0	0	0
<i>C. mopane</i> —P2	0	0	0	0	6	0	0	0	0	3	2
<i>S. africana</i> —P3	0	0	0	0	0	5	2	3	0	0	0
<i>Terminalia</i> sp.—P3	0	0	0	0	0	0	7	2	0	0	0
<i>C. mopane</i> —P3	0	0	0	0	0	4	0	4	0	0	0
<i>S. africana</i> —P4	0	0	0	0	0	0	0	0	6	0	2
<i>Terminalia</i> sp.—P4	0	0	0	0	3	0	0	0	0	5	0
<i>C. mopane</i> —P4	2	0	0	0	0	0	0	0	1	0	5

Fig. 7 - PCA with second-derivative NIR spectra of all species in each carbonization program

Fig. 8 PCA with second-derivative NIR spectra of all species and carbonization program: (a) mean spectra by sample; (b) only mean of transverse sections spectra; (c) only mean of radial and tangential longitudinal sections spectra

statistical variations in charcoal, depending on the species [16, 22, 34]. The divergences can be attributed to chemical changes in wood during carbonization. McBeath et al. [35] described a clear relationship between production temperature, aromaticity and aromatic condensation in biochar in function of progressive dehydration, decarbonylation and decarboxylation reactions. During pyrolysis, temperature can change the chemical nature and increase the aromatic character of organic composites [36].

Ruptures in charcoal can be the result of regions with concentration of tension related to the high impermeability of heartwood [6]. They were observed in rays in some Mozambican species [37], in *Eucalyptus* species [38], Brazilian Cerrado species [39] and also in *Araucaria* species [34].

Modifications in ray frequency can be influenced by fiber cell wall thickness and axial parenchyma distribution. Assis et al. [6] cited that from 280 to 500 °C, mass loss and decrease in mechanical properties of carbonized material occurs, and that wood's basic density is positively correlated with charcoal density. The main component that acts in the formation of charcoal is lignin, since it is technically more stable than the carbohydrates of wood. The literature reports, in some carbonization procedures

with temperature of 450 °C, an increase in ray frequency, decrease and no statistical changes [16, 31, 32]. In the same carbonization parameters of this study, Nisgoski et al. [18] related increase of 88% (*Sterculia quinqueloba*—P2) and a reduction of 16% (*Bobgunnia madagascariensis*—P1) in ray frequency.

4.2 Near-infrared spectroscopy

In general, bands that can be applied for species characterization are related to cellulose, hemicelluloses and lignin. In wood and its products, bands at 4686 cm^{-1} are related to lignin, extractives and acetyl groups in hemicelluloses; that at wavenumber of 4608 cm^{-1} is associated with cellulose and xylans; at 4435 cm^{-1} all cell wall components are present; the region from 4404 to 4405 cm^{-1} is attributed to cellulose and hemicelluloses; cellulose is also present in bands at 4365 cm^{-1} , 4392 cm^{-1} and 4235 cm^{-1} ; the region from 4288 to 4296 cm^{-1} is related to hemicelluloses; and the peak at 4195 cm^{-1} indicates lignin and that at 4063 cm^{-1} is attributed to carbohydrates [40].

The influence of anatomical and chemical characteristics of each species (Fig. 5) in NIR spectra resulted in a

Table 4 Confusion matrix based on PCA scores, with mean second-derivative NIR spectra by sample in the region 4000–5000 cm⁻¹

Species/programs	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>S. africana</i>	<i>S. mopane</i>	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>
P1	P2	P3	P4	P3	P4	P3	P4	P3	P4	P3	P4	P3	P4
Mean of all spectra by sample													
<i>S. africana</i> —P1	3	0	0	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P1	0	2	0	0	0	0	0	0	0	0	0	0	0
<i>S. africana</i> —P2	0	1	2	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P2	0	0	0	3	0	0	0	0	0	0	0	0	0
<i>C. mopane</i> —P2	0	0	0	0	3	0	0	0	0	0	0	1	0
<i>S. africana</i> —P3	0	0	0	0	0	3	0	1	0	0	0	0	0
<i>Terminalia</i> sp.—P3	0	0	0	0	0	0	3	0	0	0	0	0	0
<i>C. mopane</i> —P3	0	0	0	0	0	0	0	2	0	0	0	0	0
<i>S. africana</i> —P4	0	0	0	0	0	0	0	0	0	0	3	0	0
<i>Terminalia</i> sp.—P4	0	0	0	0	0	0	0	0	0	0	0	2	0
<i>C. mopane</i> —P4	0	0	0	0	0	0	0	0	0	0	0	0	3
Mean of transverse sections spectra by sample													
<i>S. africana</i> —P1	3	0	0	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P1	0	2	0	0	0	0	0	0	0	0	1	0	0
<i>S. africana</i> —P2	0	1	2	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P2	0	0	0	3	0	0	0	0	0	0	0	0	0
<i>C. mopane</i> —P2	0	0	0	0	2	0	0	0	0	0	0	1	0
<i>S. africana</i> —P3	0	0	0	0	0	3	0	1	0	0	0	0	0
<i>Terminalia</i> sp.—P3	0	0	0	0	0	0	3	1	0	0	0	0	0
<i>C. mopane</i> —P3	0	0	0	0	0	0	0	1	0	0	0	0	0
<i>S. africana</i> —P4	0	0	0	0	0	0	0	0	1	0	1	0	0
<i>Terminalia</i> sp.—P4	0	0	0	0	1	0	0	0	0	0	0	2	0
<i>C. mopane</i> —P4	0	0	0	0	0	0	0	0	0	0	1	0	3
Mean of longitudinal sections spectra by sample													
<i>S. africana</i> —P1	3	0	0	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P1	0	3	0	0	0	0	0	0	0	0	0	0	0
<i>S. africana</i> —P2	0	0	2	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P2	0	0	0	3	0	0	0	0	0	0	0	0	0
<i>C. mopane</i> —P2	0	0	0	0	3	0	0	0	0	0	0	0	0
<i>S. africana</i> —P3	0	0	0	0	0	3	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P3	0	0	0	0	0	0	3	0	0	0	0	0	0
<i>C. mopane</i> —P3	0	0	0	0	0	0	0	1	0	0	0	0	0
<i>S. africana</i> —P4	0	0	0	0	0	0	0	0	0	0	0	0	0
<i>Terminalia</i> sp.—P4	0	0	0	0	0	0	0	0	0	0	0	0	0
<i>C. mopane</i> —P4	0	0	0	0	0	0	0	0	0	0	0	0	0

Table 4 (continued)

Species/programs	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>	<i>S. africana</i>	<i>Terminalia</i> sp.	<i>C. mopane</i>
	P1		P2			P3			P4		
<i>Terminalia</i> sp.—P4	0	0	0	0	0	0	0	0	0	3	0
<i>C. mopane</i> —P4	0	0	0	0	0	0	0	0	0	0	3

diverse range of degradation parameters [16]. Kwon et al. [41] commented that chemical and physical characteristics of wood components in cell walls can be easily changed by increasing carbonization temperature. Also, Davrieux et al. [12] separated charcoal samples of *Tabebuia serratifolia* from *Eucalyptus grandis* by applying PCA and discriminant factorial analysis with NIR spectra in raw form.

Evaluating all samples, species and carbonization process (Fig. 6) material in P3 (450 °C—4 h) resulted in different behavior, also samples from *S. africana* at P1 (400 °C—40 min). This is in accordance with the chemical reactions [35] that occur during carbonization that can change or increase the organic compounds [36].

In this study, longitudinal sections showed better distinction of all species and programs. On the other hand, some studies have reported that the anatomical surface does not influence species discrimination by PCA [16, 17].

Monteiro et al. [13] informed that NIR spectroscopy was unable to distinguish eucalyptus species from 20 Cerrado species carbonized from ground material; although it separated the industrial and laboratorial carbonization process. The authors concluded that PCA with first derivative spectra of charcoal samples is a promising technique to evaluate the quality of charcoal. Likewise, Ramalho et al. [14] detected that lower carbonization temperature led to greater distinction of charcoal from planted and native forests. They commented that PCA with first-derivative NIR spectra was able to differentiate charcoal into two major groups (native and planted) when charcoal was produced at 300 °C, but this was not possible at 500 and 700 °C.

Costa et al. [15] studied *Eucalyptus* charcoal carbonized from 400 to 700 °C and found an adequate discrimination of samples produced at 700 °C by PCA applying first-derivative NIR spectra. They verified that these spectra varied in accordance with the final carbonization temperature, i.e., the higher the final temperature, the lower the absorbance bands in the spectra. This behavior was probably associated with the existence of differences in the chemical constitution of the charcoal specimens at different temperatures.

5 Conclusion

Quantitative anatomical characteristics of charcoal presented no linear relation to carbonization programs as the result of species uniqueness. The qualitative structure remained unchanged, and thus these traits can be applied in species discrimination based on databases of wood anatomical descriptions.

In near-infrared spectra, the region from 4000 to 5000 cm⁻¹ showed the best distinction between species and carbonization programs. When the study was done by

species, the P3 carbonization program (450 °C—4 h) presented the best distinction of samples. When evaluation was based on carbonization program, *S. africana* was easily identified at P1 (400 °C—40 min) and P2 (450 °C—2 h). Anatomical surfaces did not show a strong influence for species discrimination based on NIR spectra, but distinction of samples carbonized at P3 (450 °C—4 h) was verified. Near-infrared spectroscopy can be applied to discriminate species in charcoal samples produced in different carbonization processes. Parameter of final temperature influenced the data more than total process time.

The results of this study can be applied in species discrimination based on databases of charcoal anatomical descriptions and NIR spectra, and when applied in forest control, can contribute to an adequate use of wood from natural forest in Mozambique, by directing its application.

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Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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