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DISCRIMINATION OF WOOD AND CHARCOAL FROM SIX CAATINGA SPECIES BY NEAR-INFRARED SPECTROSCOPY

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ABSTRACT

Correct identification of species in wood and charcoal commerce is important, and rapid and nondestructive evaluation based on near-infrared techniques can be a good alternative. Four trees from *Combretum leprosum*, *Croton argyrophylloides*, *Jatropha mutabilis*, *Luetzelburgia auriculata*, *Mimosa tenuiflora* and *Poincianella bracteosa*, were cut in a natural forest in the municipality of Coremas, Paraíba state and stem discs with thickness of about 80 mm were taken at 0, 25, 50, 75 and 100% of commercial height. For charcoal production, each sample was wrapped in aluminum foil and carbonized in a muffle furnace, with a final temperature of 450 °C and a heating rate of 1,66 °C min^{1}. Spectra were collected directly from sample surface. The best pretreatment was second derivative, while the best classification method was PCA-LDA, and the analysis of full spectra (4000-10000 cm^{-1}) was indicated. In classification, there was no difference between surfaces where spectra was collected, so in practice, in commercial control for example, the information can be obtained from any surface. For rapid analysis for purposes of control of forest practices or illegal commerce, spectra collected directly from wood and charcoal can be applied to distinguish these six Caatinga species.

Keywords: Classification method, species distinction, spectroscopy, wood identification, wood commerce.

INTRODUCTION

The Caatinga is a biome that covers an area of about 844453 km^2 (11% of Brazilian territory), involving the states of Alagoas, Bahia, Ceará, Maranhão, Pernambuco, Paraíba, Rio Grande do Norte, Piauí, Sergipe and the north of Minas Gerais (MMA 2016). It contains 932 described plant species, 318 of them endemic that only occur in this biome. There is great amplitude in vegetation, from shrubs to seasonal forests (Seyffarth 2012). The family Euphorbiaceae has the highest richness with 34 species, while at the genus level the richest is *Croton*, comprising 11 species. Climate changes also can influence diversity of species because only some have strong potential to survive varied conditions (Santos *et al.* 2014).

The main use categories are wood for construction and medicinal plants (Santos *et al.* 2008, Cartaxo

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et al. 2010, Trentin *et al.* 2011). Also seeds present potential for oil production (Silva *et al*. 2014) and the species diversity is threatened by other non-wood uses (Cavalcanti *et al*. 2015). The use of species for wood and firewood depends on availability and some material properties, like density and moisture content, and also finality: domestic or industrial (Ramos *et al.* 2008a, Ramos *et al.* 2008b). Although species richness is not influenced by seasonality, the volume of wood varies significantly between the dry and rainy seasons, and the local vegetation suffers great extraction pressure from this use (Ramos and Albuquerque 2012).

Density and calorific value of *Combretum leprosum*, *Croton argyrophylloides*, *Jatropha mutabilis, Luetzelburgia auriculata*, *Mimosa tenuiflora* and *Poincianella bracteosa* are in studies of Machado-Neto *et al.* (2015), and the principal knowledge about uses of Caatinga species can be observed in Barbosa (2015). Species distribution and occurrence are influenced by local desertification and human actions are producing changes in the environment (Souza *et al.* 2015). Correct knowledge of species is important for adequate final use in industry and also for conservation.

The Caatinga presents potential for sustainable uses of plant resources that can be decisive for development of the region and country. The economic use of the biome's biodiversity mainly involves the cosmetic, chemical and food industries. The biome is marked by widespread deforestation. The most recent estimate is that 46% of the total area has been cleared of native vegetation, principally for firewood, pastures and agriculture (MMA 2016). In Paraiba state, the last evaluation available, based on data from 2012, shows 41% of illegal deforestation and the offer in commerce of 371262 st (55,8%) of illegal timber (Ndagijimana *et al.* 2015). Also in 2012, 915 ton of charcoal were produced (IBGE 2015) and a total of 12754 ha were registered as active Plan of Sustainable Management (APNE 2015). It is not available in literature detailed data from species and charcoal production.

Because of this degradation, correct identification of species in wood and charcoal commerce is important, and rapid and nondestructive evaluation based on near-infrared (NIR) techniques can be a good alternative. Near-infrared spectroscopy is being used in research and monitoring in different industries for detection of chemical, physical and mechanical properties of lignocellulosic materials, pulp and paper, wood modification, degradation and classification (Tsuchikawa and Kobori 2015).

Near-infrared studies for species identification include discrimination of species similar to *Swietenia macrophylla* (Braga *et al.* 2011, Pastore *et al.* 2011), samples from different geographic origins (Sandak *et al.* 2011, Nisgoski *et al*. 2016) and samples influenced by hybridization (Meder *et al.* 2014) and granulometry (Nisgoski *et al.* 2015a). For carbonized material, near-infrared spectrometry has been used to distinguish species (Davrieux *et al*. 2010) and type of carbonization process (Monteiro *et al.* 2010), including with solid samples (Nisgoski *et al.* 2015b, Muñiz *et al.* 2013, Muñiz *et al.* 2016). Literature does not report studies with NIR identification and Caatinga species, and in wood and charcoal discrimination, a wide database from different species present in Brazilian biomes is necessary, with exotic or native species, listed or not on endangered directory.

The objective of this study was to evaluate the potential of NIR spectroscopy to discriminate wood and charcoal from six Caatinga species, based on solid samples, and to evaluate the influence of sample surface, pretreatment and classification method. Another goal is to contribute to control of illegal logging improving the database of NIR spectra of solid samples of wood and charcoal for a future application in commerce control.

MATERIAL AND METHODS

The wood samples of the species *Combretum leprosum* Mart. – Combretaceae (mufumbo), *Croton argyrophylloides* Müll. Arg. – Euphorbiaceae (marmeleiro), *Jatropha mutabilis* (Pohl.) Baill – Euphorbiaceae (pião), *Luetzelburgia auriculata* (Allemão) Ducke – Fabaceae (pau serrote), *Mimosa tenuiflora* (Willd.) Poir. – Fabacae (jurema) and *Poincianella bracteosa* (Tul.) L.P. Queiroz – Fabaceae (catingueira) came from Paraíba state, from the municipality of Coremas (07º00'52" S and 37º56'45"

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W) at 266 m of altitude and As climate based on Köppen, with dry summers and temperature of coldest month near 23 °C (Alvares *et al.* 2013). Material was selected based on its availability in number of samples to increase the NIR database of wood and charcoal and its wood present potential for energy production (Barbosa 2015).

Four trees of each species were cut in a natural forest and stem discs with thickness of about 25 mm were taken at 0, 25, 50, 75 and 100% of commercial height. Samples were air dried and remained in a climatic chamber at temperature of 20 ± 3 °C and relative humidity of 65 ± 1%.

For charcoal production, each sample was wrapped in aluminum foil and carbonized in a muffle furnace, with a final temperature of 450 °C and a heating rate of 1,66 °C min^{-1}. The carbonized material remained at the final temperature for two hours. Aluminum foil was removed only for near infrared measurement.

Infrared analyses were performed with a Bruker Tensor 37 spectrometer (Bruker Optics, Ettlingen, Germany) equipped with an integrating sphere and operating in reflectance mode; 64 scans were averaged with resolution of 4 cm^{-1} and a spectral range of 10000–4000 cm^{-1}. In a room with temperature of 23 ± 2 °C and relative humidity of 60%, the wood and charcoal samples were placed on top of the integrating sphere and three spectra were obtained from the transverse surface and five from radial/tangential surface, resulting in a total of 8 separate spectra for each disc, or 40 per tree and 160 per species.

Spectral analysis was done with full spectra (4000-10000 cm^{-1}) and without wavenumbers associated with water bands, which can produce noise and interfere in results, from 4000-5000 cm^{-1} and 5500-6000 cm^{-1}, that are the more informative bands in wood and charcoal of the six Caatinga species. For wood, the surface measurements presented different spectral characteristics, so we divided the analysis into three groups: with mean of all surface spectra in each disc, only transverse surface spectra, and only radial/tangential surface (lateral) spectra. One spectrum represents the information in an area of approximately 4 mm^2. For analysis, we used spectra from 3 trees (15 samples) of each species for calibration and one other tree (5 samples) for classification testing.

The Unscrambler X chemometric program (version 10.1, from CAMO Software AS) was used to analyze the data. Exploratory modeling was done by analyzing the score and loading graphs obtained by principal component analysis (PCA) to verify possible differences in wood and charcoal. Pretreatment of second derivative of Savitzy-Golay (polynomial order = 2, smoothing point = 3) and multiplicative scatter correction (MSC) were applied to raw data. For MSC, the mean of the calibration set was used as the reference for the test set. Classification methods of SIMCA and PCA-LDA were also performed. In the SIMCA models, the first two PCs were used. LDA was calculated based on constant weight, through the quadratic method assuming equal prior probabilities and using PCA scores projected for four components. Spectral analysis was based on ASTM E1655-05 (ASTM 2000).

RESULTS AND DISCUSSION

NIR spectra of Caatinga species are similar and Figure 1 illustrates the data from *Poincianella bracteosa* wood and charcoal. To evaluate bands with more influence in Caatinga species discrimination, a comparison between mean spectra of wood and charcoal was done with second derivative data, which eliminates baseline influence. Informative wavenumbers are correlated with the presence of polysaccharides, lipids and protein, which are related to cell structure: region from 5938-5919 cm^{-1} are associated to aromatic rings of lignin; bands around 5878-5734 cm^{-1} and 4440-4378 cm^{-1} are related to all cell wall compounds and phenol content; region from 5605-5573 cm^{-1} and 4050-4000 cm^{-1} are correlated to cellulose and lignin content; the range 4240-4212 cm^{-1} is associated with cellulose; bands near 4281 cm^{-1} and the region between 4540-4445 cm^{-1} are attributed to the aromatic ring of lignin; bands in 4840-4654 cm^{-1} are related to all components of cell wall in wood and extractives; peaks at 4686 cm^{-1} corresponds to acetyl groups, lignin and extractives; while that at 4014 cm^{-1} is associated with C-H and C-C stretching and cellulose in wood and wood products (Schwanninger *et al.* 2011).

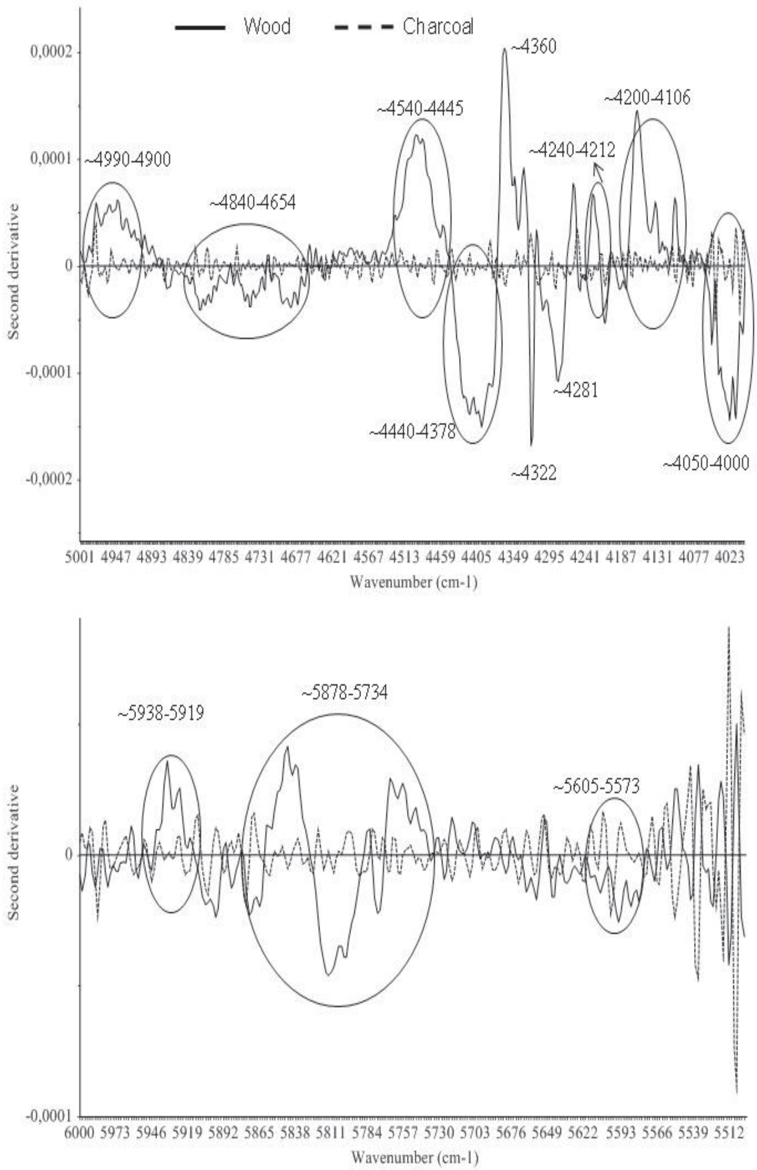

Figure 1. Second derivative spectra of wood and charcoal from *Poincianella bracteosa*.

In wood samples, *Poincianella bracteosa* presented a peak with stronger intensity at 4322 cm^{-1}, and all species presented a peak at 4360 cm^{-1} related to cellulose. In other bands, wood and charcoal presented inverse behavior. In charcoal, *Mimosa tenuiflora* presented in some regions peaks with higher intensity, revealing the differences in chemical composition and degradation. In the same carbonization process, degrees of degradation and species were found to be related (Muñiz *et al.* 2013, Muñiz *et al.* 2016), and if its chemical constituents are isolated or combined in wood cells, the behavior is different (Popescu *et al.* 2011).

In the PCA analysis of mean spectra of all surfaces, i.e. transverse and radial/tangential surface

(Figure 2), second derivative of wood differentiated all species and formed visually a group with the Fabaceae family (*Luetzelburgia auriculata, Mimosa tenuiflora* and *Poincianella bracteosa*). Based on visual discrimination of samples, in the analysis of full spectra wavenumber (4000-10000 cm^{-1}), the second derivative presented better distribution of samples in PCA graphs of wood and in charcoal the best distribution only present a tendency of separation of *Mimosa tenuifolia* and *Combretum leprosum* from other species. In region from 4000-5000 cm^{-1}, MSC pretreatment was more efficient for wood, showing the best distinction of samples from *Jatropha mutabilis* and the similarity of other species in these wavenumbers, while for charcoal, second derivative was best and present the distinction of *Mimosa tenuifolia* and *Combretum leprosum* and showed the tendency of two groups of similarity: i) *Jatropha mutabilis* and *Poincianella bracteosa;* ii) *Croton argyrophylloides* and *Luetzelburgia auriculata.* In region from 5500-6000 cm^{-1}, second derivative presented better results for wood, with separation of all species and some sobreposition of samples from *Poincianella bracteosa* and *Mimosa tenuifolia*, while for charcoal MSC was best also with the separation of all species.

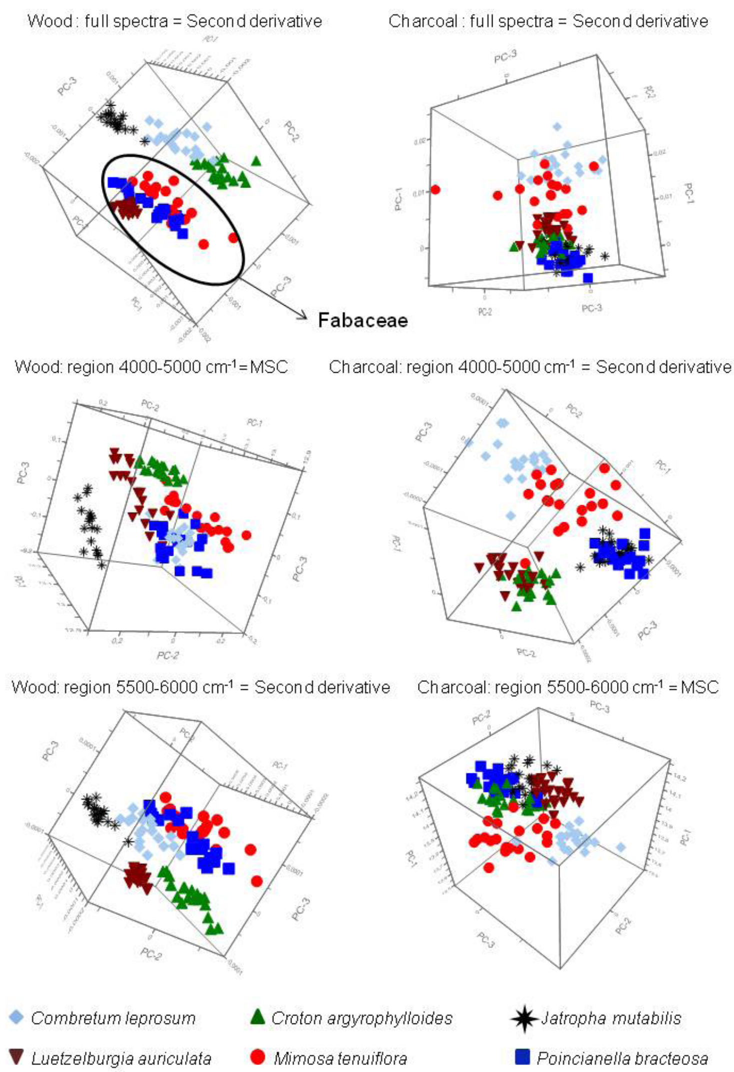

Figure 2. Best results of PCA from wood and charcoal with mean spectra from all surfaces in different wavenumbers.

When the analysis of wood was performed to evaluate surface influence on species discrimination, i.e. with spectra from transverse and from radial/tangential surface separated (Figure 3), for full spectra second derivative presented better results showing the similarity of species grouping the samples from Fabaceae family (*Luetzelburgia auriculata, Mimosa tenuiflora* and *Poincianella bracteosa*) in both surfaces. Radial/tangential surface presented a better distinction of other species (*Combretum leprosum, Jatropha mutabilis, Croton argyrophylloides*), with individual samples more grouped.

In the region from 4000-5000 cm^{-1}, also better results based on visual distribution were observed with second derivative. In transverse section, *Poincianella bracteosa* and *Jatropha mutabilis* are more distinct from other species. On the other hand, in radial/tangential surface only *Jatropha mutabilis* is easily identified.

For the region from 5500-6000 cm^{-1}, MSC was more efficient in both surfaces, and radial/tangential showed better distribution for a visual separation of *Jatropha mutabilis, Poincianella bracteosa, Combretum leprosum* and *Mimosa tenuiflora.*

Though in all evaluated regions some species can be discriminated, the distinction of six studied species present the best results with full spectra and in radial/tangential surface.

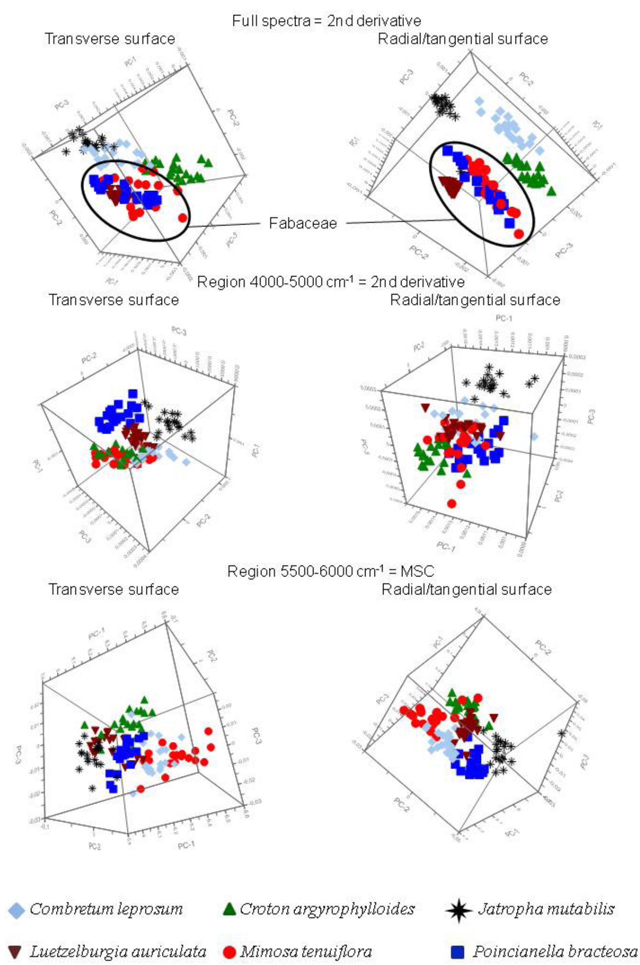

Figure 3. Best results of PCA from wood in transverse and radial/tangential surfaces in different wavenumbers.

Different pretreatments and spectral regions presented adequate results, in function of material. For wood (mean of all surfaces), second derivative and full spectra (4000-10000 cm^{-1}) were better

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for discrimination. For charcoal, second derivative and wavenumber between 4000-5000 cm^{-1} present better distribution. When the analysis was based on wood surface, better response was from radial/tangential surface and second derivative of full spectra.

The analysis with second derivative has also been reported to be efficient in other studies for species discrimination based on solid samples of wood and charcoal (Horikawa *et al.* 2015, Nisgoski *et al.* 2015b, Hwang *et al.* 2016, Muñiz *et al.* 2016). When the analysis is done with solid samples, a larger number of samples are necessary to establish a characteristic spectra or informative bands for each species, because features of surface (anatomical characteristic as vessels and ray dimension; cut and/or sand), shape and particle size (powder granulometry) can influence species discrimination by NIR (Hein *et al*. 2010, Braga *et al*. 2011, Nisgoski *et al.* 2015a).

The NIR absorbance values present variations among species, and for their discrimination, some regions can have more influence based on anatomical and chemical characteristics. Species similar to mahogany were distinct in a spectral range from 4249-6100 cm^{-1} (Pastore *et al.* 2011), 4000-6200 cm^{-1} showed potential in discriminating six provenances of *Criptomeria japonica* planted in southern Brazil (Nisgoski *et al.* 2016), and wood and charcoal samples from the miscellaneous "angelim" group in Brazil were discriminated in two regions, from 6200 to 5500 cm^{-1} plus from 5000 to 4000 cm^{-1} (Muñiz *et al.* 2016). In this study, the bands in regions from 4000-5000 cm^{-1} and 5500-6000 cm^{-1} present more contrast between wood and charcoal. For wood, full spectra showed more information for discriminating six Caatinga species, and for charcoal bands in regions from 4000-5000 cm^{-1}, supporting the influence of individual species.

Classification of test samples by SIMCA and PCA-LDA were done based on the original spectra and pretreatments using second derivative and multiplicative scatter correction (MSC). We also compared full spectra and region between 4000-5000 cm^{-1} and 5500-6000 cm^{-1}. For wood, surface where spectra were acquired was also evaluated. To observe the potential of NIR spectroscopy to discriminate Caatinga species and the influence of surface and regions of spectra analyzed, we summarize the correct classifications in Table 1. In SIMCA, there was overlapping classification, resulting in some samples not being uniquely classified. In fact, some individual samples may have been classified to two or more species.

Table 1. Total correct classification (%) of wood and charcoal samples of six Caatinga species.

Material	Spectra	SIMCA (%)			PCA-LDA (%)		
		Raw	2nd derivative	MSC	Raw	2nd derivative	MSC
Wood	4000-10000 cm^{-1}	0	93	0	67	100	80
	4000-5000 cm^{-1}	0	0	7	87	57	50
	5500-6000 cm^{-1}	23	7	37	100	47	17
Charcoal	4000-10000 cm^{-1}	3	0	3	67	57	43
	4000-5000 cm^{-1}	0	0	0	43	60	17
	5500-6000 cm^{-1}	0	0	0	67	57	17
Wood Transverse surface	4000-10000 cm^{-1}	0	0	3	37	100	83
	4000-5000 cm^{-1}	7	0	7	80	33	50
	5500-6000 cm^{-1}	13	0	23	93	53	57
Wood Radial/Tang surface	4000-10000 cm^{-1}	0	13	3	33	100	17
	4000-5000 cm^{-1}	0	0	0	53	53	57
	5500-6000 cm^{-1}	3	0	7	3	10	17

For wood, mean spectra of transverse and radial/tangential surface, SIMCA classification was efficient with second derivative and full spectra (4000-10000 cm^{-1}), for all species except *Croton argyrophylloides*. In PCA-LDA, also second derivative and full spectra were adequate for all species. When the analysis was performed with distinction of regions (4000-5000 cm^{-1} and 5500-6000 cm^{-1}), raw data presented better results. When spectra of transverse surface of wood were analyzed, SIMCA classification was not efficient for all species and bands. PCA-LDA presented better results with second derivative and full spectra for all species. Also when observing regions tested, raw data showed adequate results. In analysis of radial/tangential surface of wood, SIMCA classification was not efficient for all species. For PCA-LDA, second derivative and full spectra were efficient in classification. When the region between 5500-6000 cm^{-1} was evaluated, raw data presented better results.

For charcoal, no species were correctly classified by SIMCA. In PCA-LDA, samples of C*ombretum leprosum* were correctly classified with second derivative and MSC in full spectra and bands, *Croton argyrophylloides* presented correct classification with raw data and bands between 5500-6000 cm^{-1}, *Jatropha mutabilis* was adequately classified with raw data and full spectra, and *Mimosa tenuiflora* was discriminated with MSC pretreatment and the region from 5500-6000 cm^{-1}. For SIMCA, misclassifications occurred for all species, meaning that the analysis returned more than one species for each spectrum. This can be the result of the reflection of spectra, if is predominant more cell or lume wall. One single spectrum represents one area of approximately 4 mm^2, and its reflection depends on cell dimensions, cell wall width and type of cell which is characteristic of each species. For example, anatomical dimensions, obtained by França (2015) for the samples of this study, showed that *Mimosa tenuiflora* present vessels with tangential diameter of 120 µm and frequency of 15/mm^2 and *Jatropha mutabilis* present tangential diameter of vessel equal 57 µm and frequency of 5/mm^2. The reflection was different in function of the number and dimensions of vessels and because of the point where the laser fall, if it is the cell wall or cell lumen. In PCA graphics with best result, it is possible to observe that this species do not overlap.

In anatomical analysis, transverse surface bring more information about cell type and distribution, but chemical composition can be very similar. Also, surface irregularities can influence results (Brunner *et al.* 1996) when samples are only sawn, as here. In charcoal, there is different degradation of species in the same carbonization process (Muñiz *et al.* 2013, Nisgoski *et al.* 2015b).

Other studies have presented adequate classification by SIMCA for red oak and white oak wood (Adedipe *et al.* 2008), and thermally modified wood of spruce, beech and ash (Bächle *et al.* 2012). In turn, PCA-LDA has shown potential to discriminate wood and charcoal solid samples of "angelim" species (Muñiz *et al.* 2016).

Data pretreatment presented adequate results in wood sample analysis and varied for each material. Second derivate is in general applied with better discrimination of species (Sandak *et al.* 2011, Zhang *et al.* 2014, Muñiz *et al.* 2016, Nisgoski *et al*. 2016), but in charcoal MSC also can be applied (Muñiz *et al.* 2016).

In the classification analysis, for wood SIMCA was 93% efficient and PCA-LDA was 100%, with full spectra and second derivative. The surface where the spectra were obtained did not influence the total results. Transverse and radial/tangential surface presented 100% correct classification results for PCA-LDA with full spectra and second derivative. For charcoal, the best results for classification method were for PCA-LDA and raw data with full spectra (67%).

CONCLUSIONS

In NIR infrared analysis of wood from Caatinga species, the results were influenced by pretreatment, classification method and spectral region analyzed. The best pretreatment was second derivative, the best classification method was PCA-LDA, and the analysis of full spectra (4000-10000 cm^{-1}) was indicated.

The distribution of species from wood spectra (all surfaces, transverse and radial/tangential) showed distinction of the Fabaceae family. In external classification, there was no difference between surfaces where spectra was collected, so in practice, in commercial control for example, the information can be obtained from any surface.

For charcoal, PCA-LDA resulted in 67% correct classification for full spectra with raw data, but to determine other characteristics, like anatomical features, more detailed analysis is necessary.

For rapid analysis in control of forest practices of illegal commerce, spectra collected directly from wood and charcoal can be applied to distinguish the six Caatinga species analyzed here. Efforts must be done to improve de database from NIR spectra of wood and charcoal of Brazilian species, transfer it from laboratory equipment to a hand-held one, and also training government agents in this technique.

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