CANONICAL CORRELATION ANALYSIS OF THE CHARACTERISTICS OF CHARCOAL FROM *Qualea parviflora* Mart.

Thiago de Paula Protásio[1], Rosalvo Maciel Guimarães Neto[2], João de Deus Pereira de Santana[2], José Benedito Guimarães Júnior[2], Paulo Fernando Trugilho[1]

*Autor para correspondência: depaulaprotasio@gmail.com

ABSTRACT: *This study aimed to examine the relationships between the characteristics of charcoal from Qualea parviflora Mart. using canonical correlation analysis. Five trees were analyzed in such way that 5-cm thick discs were removed from each tree at the base, DBH (1.30 m), middle and top sections. The wood was carbonized in a muffle furnace at a heating rate of 1.67 °C min-1. A canonical correlation analysis was conducted to investigate the relationships between the group formed by fixed carbon, volatile matter, ash, elemental carbon, hydrogen, nitrogen, sulfur and oxygen levels and a second group formed by the gravimetric yield, higher heating value and relative bulk density of the charcoal. A tendency was noted for high levels of fixed carbon and elemental carbon to be associated to low levels of volatile matter, ash and oxygen and to low gravimetric yield. Fixed carbon and elemental carbon levels had a positive relation to higher heating value and to relative bulk density, whereas volatile matter, ash and oxygen levels had a negative relation to such characteristics. The higher the gravimetric yield from carbonization, the higher the volatile matter, ash and oxygen levels will be in the resulting charcoal.*

Keywords: charcoal, chemical composition, heating value, multivariate analysis

ANÁLISE DE CORRELAÇÕES CANÔNICAS DAS CARACTERÍSTICAS DO CARVÃO VEGETAL DE *Qualea parviflora* Mart.

RESUMO: Neste trabalho, objetivou-se utilizar a análise de correlação canônica para avaliar as relações existentes entre as características do carvão vegetal de *Qualea parviflora* Mart. Foram avaliadas cinco árvores das quais foram retirados discos de 5 cm de espessura na base, DAP (1,30 m), meio e topo. A madeira foi carbonizada em uma mufla, considerando a taxa de aquecimento de 1,67 ºC min^{-1}. Foi realizada a análise de correlação canônica entre o grupo formado pelos teores de carbono fixo, materiais voláteis, cinzas, carbono elementar, hidrogênio, nitrogênio, enxofre e oxigênio com um segundo grupo formado pelo rendimento gravimétrico, poder calorífico superior e a densidade relativa aparente do carvão vegetal. Observou-se uma tendência de elevados teores de carbono fixo e carbono elementar associados ao carvão vegetal de baixos teores de materiais voláteis, cinzas e oxigênio e de baixo rendimento gravimétrico. Os teores de carbono fixo e de carbono elementar possuem relação positiva com o poder calorífico superior do carvão vegetal e com a densidade relativa aparente. Já, os teores de materiais voláteis, cinzas e oxigênio possuem relação negativa com essas características. Quanto maior o rendimento gravimétrico da carbonização, maiores serão os teores de materiais voláteis, cinzas e oxigênio presentes no carvão vegetal.

Palavras-chave: carvão vegetal, composição química, poder calorífico, análise multivariada

1 INTRODUCTION

Species *Qualea parviflora* Mart. has high basic density (690 kg m^{-3}) and provides high dry matter yield of wood (524 kg ha^{-1}) (VALE et al., 2002). Popularly known as Pau-terra-de-folha-miúda or simply Pau-terra, the species belongs to family Vochysiaceae and is most commonly found in the transition zone between the Cerrado and Caatinga biomes. *Qualea parviflora* Mart. naturally occurs in Bahia, Minas Gerais, Mato Grosso do Sul, Piauí and São Paulo states, in cerrado and campos cerrados savannas (LORENZI, 2002).

Around 35% to 50% of the charcoal consumed by Brazilian steel companies is sourced from residues from management and exploration of natural forests, particularly of the Cerrado biome, since homogeneous forest plantations have proved unable to meet the entire market demand (ASSOCIAÇÃO BRASILEIRA DE PRODUTORES DE FLORESTAS PLANTADAS - ABRAF, 2012; BRASIL, 2011; CALAIS, 2009). Therefore, research studies become increasingly critical not only to evaluate the quality of charcoal from native species with high potential for use in energy and steel companies, as is the case of *Qualea parviflora* Mart. (PROTÁSIO et al., 2011a; VALE et

[1] Universidade Federal de Lavras - Lavras, Minas Gerais, Brasil.

[2] Universidade Federal do Piauí - Bom Jesus, Piauí, Brasil.

al., 2002), but also to investigate the relationships or correlations between the characteristics of interest.

In order to evaluate charcoal intended for use in energy and/or steel production, it is necessary to quantify characteristics such as carbonization yield, heating value, bulk density and immediate chemical composition, and learning the relations between such characteristics can be very useful for controlling the pyrolysis process, optimizing charcoal quality and ensuring genetic improvement of the species of interest.

With that in mind, multivariate techniques such as canonical correlation are a viable alternative not only for analysis and interpretation of data in studies exploring wood and charcoal quality, but also for identifying quality coefficients and/or indices (TRUGILHO et al., 1997a).

Canonical correlation analysis enables verifying the existing linear correlations between two groups or sets of variables (X and Y) and is intended to maximize the correlations between such variables. The analysis consists of obtaining a pair of latent variables, known as canonical statistical variables, that are linear combinations of the variables of two vectors (X and Y), and the information contained in the parameters should be focused on the correlation between these new variables. Each pair of canonical statistical variables is termed canonical function (ABREU; VETTER, 1978; CRUZ; REGAZZI, 1994; FERREIRA, 2008; HAIR JÚNIOR et al., 2005, 2009; MINGOTI, 2005).

The above technique is used in exploratory studies, as in some situations researchers may have a large set of variables and yet be interested only in studying a few linear combinations of the variables within such set (TRUGILHO et al., 1997b, 2003).

Ferreira (2008) argued that knowledge of the interrelation between distinct variable vectors can allow researchers to identify possible dependence structures between the different sets of variables, which would otherwise not be easily identifiable by simple linear correlation analysis. And yet canonical correlation analysis is the multivariate statistical technique least explored by users (MINGOTI, 2005).

Given the above, the aim of this study was to use canonical correlation analysis to evaluate the relationships between the characteristics of charcoal from *Qualea parviflora* Mart.

2 MATERIAL AND METHODS

2.1 Carbonization procedure and analysis of the resulting charcoal

The wood material was obtained from a Cerrado remnant sitting between the municipalities of Bom Jesus and Currais, in Piauí state. Five trees were randomly sampled, and from each tree 5-cm thick discs were removed from the base, DBH (1.30 m), middle and top sections, with a total of five replicates being run per lengthwise position, noting that 5.38 m was defined as the average total height. The average DBH outside bark was 51.0 cm, against 40.4 cm inside bark. For purposes of sampling, height was defined as including only tree trunks.

The wood material was carbonized in an electric muffle furnace at a heating rate of 1.67 °C min-1. The initial temperature was 100 °C and the final temperature was 450 °C, remaining stable for 30 minutes (ASSIS et al., 2012; NEVES et al., 2011; TRUGILHO et al., 1997a, 1997b, 2005). The total carbonization time was four hours. In each trial around 800 gr. of wood was used, which had been previously oven dried at 103 ± 2 ºC. After each carbonization procedure, the gravimetric charcoal yield was computed.

The relative bulk density of the resulting charcoal was determined by the hydrostatic method, considering water as the medium and as described in NBR 11941 (ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS - ABNT, 2003).

The charcoal was subjected to immediate chemical analysis for quantification of fixed carbon, volatile matter and ash levels, according to the guidelines set out in standard NBR 8112 (ABNT, 1983).

For the quantification of higher heating value, an IKA C-200 digital calorimeter was used following standard NBR 8633 (ABNT, 1984).

For the elemental analysis, the samples were crushed and sieved, and the fraction retained by a 270-mesh sieve was used, as described by Paula et al. (2011) and Protásio et al. (2011b). The quantification of carbon, hydrogen, nitrogen and sulfur levels, relative to dry charcoal weight, was performed using a universal Elementar analyzer (model Vario Micro Cube). This analyzer uses helium as carrier gas and oxygen as ignition gas. The 2-mg samples were placed in tin capsules and thoroughly incinerated at 1,200 °C. Oxygen content was obtained by the difference (Eq. 1), following the guidelines provided by Bech et al. (2009).

$$O = 100 - C - H - N - S - Cz \quad (1)$$

where: O is oxygen content (%), C is carbon content (%), H is hydrogen content (%), N is nitrogen content (%), S is sulfur content (%) and Cz is ash content (%).

2.2 Canonical correlation analysis

A canonical correlation analysis was performed to establish correlations between the group formed by fixed carbon, volatile matter, ash, elemental carbon, hydrogen, nitrogen, sulfur and oxygen levels and a second group formed by the gravimetric yield, higher heating value and relative bulk density of the charcoal from *Qualea parviflora* Mart. The first group represents the dependent variables (Y) whereas the second group represents the independent variables (X).

It was possible to determine three canonical functions, or three pairs of canonical statistical variables. Estimations were made of the canonical loadings, that is, the correlations between the original variables and their respective canonical statistical variables, the canonical cross loadings which represent the correlation between an original variable of a given group and the canonical statistical variable of the opposite group, and also the canonical weights (similar to beta coefficients of a multiple regression).

To correct any possible effects of the lengthwise sampling positions on the characteristics of the charcoal being evaluated, the canonical correlations were computed based on the residuals of variance analyzes performed for each variable.

Preliminarily to the canonical correlation analysis, simple linear correlations were also estimated between the variables so as to test for multicollinearity between variables from different groups.

The Hotelling's multivariate test (approximation of the F-distribution) was used to test the significance of the canonical roots jointly. Also, the generalized Shapiro-Wilk test was used to test for multivariate normality in the data used, since the significance test of canonical correlation is only valid when vectors X and Y are multivariate normal (FERREIRA, 2008; JOHNSON; WICHERN, 1992; MINGOTI, 2005).

The amount of explained variance, that is, the percentage of variance in the dependent canonical statistical variable that may be explained by the independent canonical statistical variable, and vice versa, was determined by squaring the canonical correlation (canonical R^2).

The amount of explained variance between the observed dependent and independent variables and their respective canonical statistical variables was determined by squaring the canonical loadings. To calculate the amount of variance explained by the canonical statistical variable, an arithmetic average was taken of the canonical loadings squared. The same procedure was used for the canonical cross loadings in order to estimate the shared variance between the observed dependent or independent variable and the opposite canonical statistical variable.

The redundancy index was also computed, it being the average squared canonical loading times the canonical R^2. The redundancy index expresses the amount of variance in a canonical statistical variable (dependent or independent) explained by the opposite canonical statistical variable in a canonical function. A high redundancy index entails a high canonical correlation and a high degree of shared variance explained by the canonical statistical variable. The redundancy index should be considered in trying to overcome the bias and uncertainty inherent in the use of canonical roots (squared canonical correlations) as a measure of shared variance (HAIR JÚNIOR et al., 2009).

The statistical analysis procedures used were grounded in studies performed by Cruz and Regazzi (1994), Ferreira (2008), Hair Júnior et al. (2005, 2009), Mingoti (2005), Protásio et al. (2012) and Trugilho et al. (1997b, 2003).

All analysis procedures were performed using software R version 2.11.0 (R DEVELOPMENT CORE TEAM, 2008), packages CCA (GONZÁLEZ; DÉJEAN, 2009), CCP (MENZEL, 2009) and yacca (BUTTS, 2009).

3 RESULTS AND DISCUSSION

Table 1 provides values of canonical correlations, canonical R^2 and Hotelling's multivariate significance test with approximation of the F-distribution.

Table 1 *- Canonical correlations and Hotelling's multivariate test.*

Tabela 1 - Correlações canônicas e teste multivariado de Hotelling.

Function	Canonical correlation	Canonical R^2	DF_1	DF_2	aF	p-value
1	0.9865	0.9732	24	20	11.60	$3.23{,}10^{-7}$
2	0.8912	0.7942	14	26	3.38	$3.52{,}10^{-3}$
3	0.7848	0.6159	6	32	2.85	$2.44{,}10^{-2}$

DF_1: degrees of freedom for number of observations; DF_2: degrees of freedom for error; aF: approximate F statistic.

Canonical functions 1 and 2 were found to be significant and have a high canonical R^2. Results reveal that 97.32% and 79.42% of the variance between the groups of charcoal characteristics is explained by canonical functions 1 and 2 respectively. This is indicative of interdependence between the groups of variables and can be of great help in modeling, in understanding the pyrolysis process and in ensuring the genetic improvement of the species for bioenergy applications, particularly in the steel industry.

Figure 1 provides scores of significant canonical functions by Hotelling's test. A significant interdependence was found between the variables of each group, that is, the characteristics of charcoal encompassed by groups X and Y not only are not independent but have a high correlation.

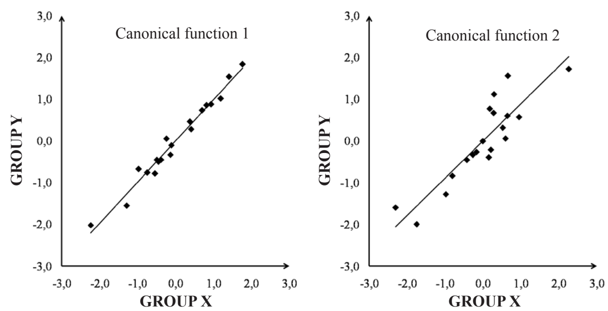

***Figure 1** - Scores of significant canonical functions by Hotelling's test.*

Figura 1 - Escores das funções canônicas significativas pelo Teste de Hotelling.

Table 2 provides canonical weights, canonical loadings and canonical cross loadings for canonical function 1.

An analysis and interpretation of canonical weights entails examining the signal and its magnitude. Variables with relatively higher canonical weights contribute more toward the statistical variables and vice versa. As regards the dependent group, it was noted that fixed carbon, volatile matter and ash levels have the highest canonical weights. And as for the independent group, gravimetric charcoal yield can be considered the most important variable. It should be noted, however, that canonical weights are subject to high variability (instability) and are sensitive to multicollinearity (HAIR JÚNIOR et al., 2009).

In the dependent group, the highest canonical loadings, in module, relate to fixed carbon and volatile matter levels. These variables can thus be considered the most important in the dependent canonical variable set. Hydrogen and sulfur levels had statistically negligible canonical loadings. This probably occurred because the low levels of these chemical components in charcoal from *Qualea parviflora* are indicative of their low correlations with other parameters being evaluated.

***Table 2** - Canonical weights, canonical loadings and canonical cross loadings of canonical function 1.*

Tabela 2 - Pesos canônicos, cargas canônicas e cargas canônicas cruzadas da função canônica 1.

Variables	Canonical weights	Canonical loadings	Canonical cross loadings
Dependent (Y)			
Fixed carbon	55.80	-0.935114	-0.922498
Volatile matter	56.21	0.902173	0.890001
Ash	49.10	0.190477	0.187907
Elemental carbon	-8.19	-0.401506	-0.396089
Hydrogen	-7.44	-0.044895	-0.044289
Oxygen	-8.17	0.360293	0.355432
Nitrogen	-12.11	-0.197468	-0.194804
Sulfur	-7.55	0.029620	0.029220
Independent (X)			
Gravimetric yield	$-3.98,10^{-1}$	0.723043	0.713288
Relative bulk density	$-2.11,10^{-4}$	-0.254627	-0.251192
Higher heating value	$-8.01,10^{-3}$	-0.975417	-0.962257

In the dependent group, the highest canonical loadings, in module, relate to fixed carbon and volatile matter levels. These variables can thus be considered the most important in the dependent canonical variable set. Hydrogen and sulfur levels had statistically negligible canonical loadings. This probably occurred because the low levels of these chemical components in charcoal from *Qualea parviflora* are indicative of their low correlations with other parameters being evaluated.

In the independent canonical variables group, only relative bulk density had low canonical loadings. The same trend was observed for canonical cross loadings.

An analysis of the canonical loadings revealed a tendency for high fixed carbon levels to be associated to low volatile matter, ash and oxygen levels and to high elemental carbon level. The existing relationships between fixed carbon and nitrogen levels were not sufficiently explained by the canonical correlation analysis, and this could be attributed to the low canonical loading observed for nitrogen level.

Again in the independent group, a positive correlation was found between relative bulk density and higher heating value. Gravimetric charcoal yield, in its turn, had a negative correlation with relative bulk density and higher heating value.

Vale et al. (2001) found no correlation between gravimetric charcoal yield and bulk density using simple linear correlation analysis, which reinforces the importance of using canonical correlation analysis for understanding existing correlations between charcoal characteristics.

Trugilho and Silva (2001) found no positive correlation between relative bulk density and higher heating value in charcoal from Jatobá, unlike the findings in this study. These authors found a positive linear correlation between charcoal yield and bulk density. However, similarly to the findings from the canonical correlation analysis, Trugilho and Silva (2001) found a negative correlation between gravimetric charcoal yield and higher heating value.

An analysis of the canonical cross loadings revealed that higher fixed carbon and elemental carbon levels correlate positively with higher heating value and relative bulk density but negatively with gravimetric yield.

It was further noted that the higher the gravimetric charcoal yield the higher the volatile matter, ash and oxygen levels in charcoal will be. Likewise, the higher the volatile matter, ash and oxygen levels, the lower the values of relative bulk density and higher heating value will be and vice versa.

Therefore, when evaluating charcoal for use in the steel industry, a high fixed carbon content is desirable on account of the higher percentage of reducing carbon that can be used in the chemical reactions. Additionally, the higher the fixed carbon content and the lower the volatile matter content, the greater the higher heating value will be for the resulting charcoal, as has been found both in this study and in studies performed by Reis et al. (2012) and by Trugilho and Silva (2001), and the greater the thermal stability of the fuel and its residence time in the furnace will be for thorough burning (BRAND, 2010; PROTÁSIO et al., 2013; VALE et al., 2001).

Also, it is important to consider the influence maximum carbonization temperature has on charcoal characteristics, such as immediate chemical composition and yields evaluated (TRUGILHO; SILVA, 2001; VALENTE et al., 1993). In this study, however, the *Qualea parviflora* charcoal samples were obtained under the same condition of pyrolysis, that is, the observed correlations allow understanding the typical interrelations between the characteristics of this bioreductant without the effect of temperature.

Determining the ash content is critical when evaluating the bioenergetic potential and use potential of charcoal in the steel industry, inasmuch as high ash contents contribute to reducing the heating value, given that minerals do not participate in the combustion process. This statement corroborates the results found both in this work and in studies performed by Brand (2010), Paula et al. (2011), Protásio et al. (2011b) and Reis et al. (2012). Additionally, high ash contents in charcoal may cause impurities to accumulate in the center of solidified metal pieces, leading to variations in the physical, chemical and mechanical properties of the resulting pig iron or iron alloys.

With respect to the influence of oxygen content plus minerals on the heating value of biomass fuels, Protásio et al. (2011b) reported a negative correlation between such variables, which supports the findings for *Qualea parviflora* charcoal. The relationships found between levels of fixed carbon, volatile matter, higher heating value and gravimetric yield are consistent with results found in literature (TRUGILHO; SILVA, 2001; VALE et al., 2001).

Table 3 provides canonical weights, canonical loadings and canonical cross loadings for canonical function 2. In the dependent group, elemental chemical components had the highest canonical weights, whereas in the independent group, gravimetric yield had the highest canonical weights.

In the independent group, relative bulk density had the highest canonical loading in module, whereas in the dependent group, nitrogen content can be considered the most important variable. Analysis results for canonical function 2 revealed a tendency for nitrogen content to increase with decreasing relative bulk density and vice versa.

Redundancy measures of shared variance for canonical functions 1 and 2 are provided in Table 4.

In canonical function 2, the variables relative bulk density and nitrogen content had the highest percentage of explained variance within and between groups.

Results demonstrate that fixed carbon, volatile matter, higher heating value, gravimetric yield and relative bulk density are the most significant variables in the canonical correlation analysis.

Figure 2 provides redundancy indices for significant canonical functions according to Hotelling's test.

Table 3 - *Canonical weights, canonical loadings and canonical cross loadings of canonical function 2*
Tabela 3 - Pesos canônicos, cargas canônicas e cargas canônicas cruzadas da função canônica 2.

Variables	Canonical weights	Canonical loadings	Canonical cross loadings
Dependent (Y)			
Fixed carbon	-137.60	-0.049739	-0.044327
Volatile matter	-137.58	-0.006639	-0.005917
Ash	6.24	0.366677	0.326785
Elemental carbon	143.74	0.291253	0.259567
Hydrogen	142.76	0.200911	0.179053
Oxygen	143.77	-0.403456	-0.359563
Nitrogen	154.66	0.466239	0.415516
Sulfur	145.29	0.192849	0.171868
Independent (X)			
Gravimetric yield	$-3.19{,}10^{-1}$	-0.272979	-0.243280
Relative bulk density	$-3.14{,}10^{-2}$	-0.940697	-0.838355
Higher heating value	$-6.18{,}10^{-6}$	0.091299	0.081366

Table 4 - *Redundancy measures of shared variance for canonical functions 1 and 2.*
Tabela 4 - Medidas de redundância de variância compartilhada para as funções canônicas 1 e 2.

Variables	**Canonical function 1**		**Canonical function 2**	
Dependent (Y)	**SCL**	**SCCL**	**SCL**	**SCCL**
Fixed carbon	0.8744	0.8510	0.0025	0.0020
Volatile matter	0.8139	0.7921	0.000044	0.000035
Ash	0.0363	0.0353	0.1345	0.1068
Elemental carbon	0.1612	0.1569	0.0848	0.0674
Hydrogen	0.0020	0.0020	0.0404	0.0321
Oxygen	0.1298	0.1263	0.1628	0.1293
Nitrogen	0.0390	0.0380	0.2174	0.1727
Sulfur	0.0009	0.0009	0.0372	0.0295
MEAN	0.2572	0.2503	0.0849	0.0675
Independent (X)	**SCL**	**SCCL**	**SCL**	**SCCL**
Gravimetric yield	0.5228	0.5088	0.0745	0.0592
Relative density	0.0648	0.0631	0.8849	0.7028
Higher heating value	0.9514	0.9259	0.0083	0.0066
MEAN	0.5130	0.4993	0.3226	0.2562

SCL: squared canonical loadings; SCCL: squared canonical cross loadings.

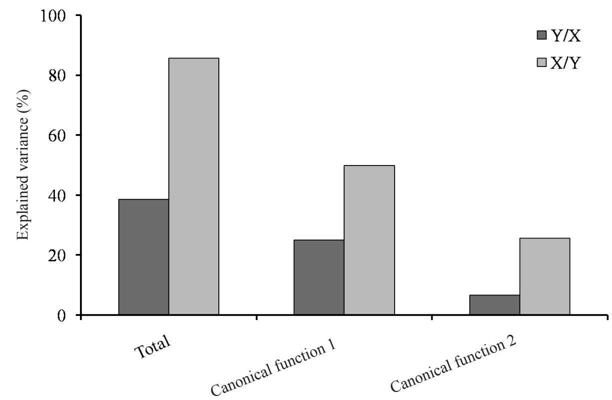

Figure 2 - *Redundancy indices for canonical functions.*

Figura 2 - Índices de redundância para as funções canônicas.

In canonical function 1, 50% of variance in the dependent variables was explained by the independent canonical statistical variable. The dependent canonical statistical variable (Y), on the other hand, explained around 25% of variance in the independent variables (X). In canonical function 2, the independent group explained around 26% of variance in the dependent variables.

4 CONCLUSIONS

The canonical correlation analysis proved effective in determining the existing relationships between the characteristics of charcoal from *Qualea parviflora* Mart., revealing an interdependence between the groups of interest.

A tendency was noted for high fixed carbon and elemental carbon contents to be associated to charcoal with low volatile matter, ash and oxygen contents and with low gravimetric yield.

Fixed carbon and elemental carbon levels correlated positively with higher heating value of charcoal and with relative bulk density. Volatile matter, ash and oxygen levels, on the other hand, correlated negatively with such characteristics.

The higher the gravimetric yield from carbonization, the higher the volatile matter, ash and oxygen contents present in the resulting charcoal.

The authors hope these results will be valuable in modeling, in understanding the pyrolysis process and in improving the bioenergetic qualities of charcoal from *Qualea parviflora* Mart.

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