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CHARACTERIZATION AND STATISTICAL CORRELATION BETWEEN CHARCOAL'S PHYSICAL AND MECHANICAL PROPERTIES OF *Eucalyptus* AND *Corymbia* CLONES

CARACTERIZAÇÃO E CORRELAÇÃO ESTATÍSTICA ENTRE AS PROPRIEDADES FÍSICAS E MECÂNICAS DO CARVÃO DE CLONES DE *Eucalyptus* e *Corymbia*

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ABSTRACT

The study aimed to analyze the physical and mechanical properties of charcoal from eucalypt clones by principal component analysis and demonstrate the relationships between these properties, in order to assess which charcoal property should aimed in the process to obtain a higher quality product. In this way, was cut eight clones of *Eucalyptus* and two of *Corymbia*, collecting three trees per clone and five disk in different heights. The disks were transformed into test samples, totaling an average of 75 samples per clones, which were carbonized under specific conditions for analysis of apparent density, compressive strength parallel to grain and linear and volumetric degradation due to high temperature. It is noteworthy that the data were weighted by disk and per tree, to an average closer to reality. For correlations, was used multivariate analysis of principal components. Herein, it is found that the apparent density of charcoal acts as the focal point of the other properties studied, and observed that as the higher the density, higher will be the compressive strength parallel to grain, the elastic modulus and the gravimetric yield.
Keywords: energetic characteristics; principal components analysis; Eucalypt.

RESUMO

O estudo teve como objetivo analisar as propriedades físicas e mecânicas do carvão de clones de eucalipto por análise de componentes principais e demonstrar as correlações entre essas propriedades, a fim de avaliar qual propriedade de carvão deve ser almejada no processo para obter um produto de alta qualidade. Desta forma, foram cortados oito clones de *Eucalyptu*s e dois de *Corymbia*, coletando três árvores por clone e cinco discos em diferentes alturas. Os discos foram transformados em amostras para ensaio, totalizando uma média de 75 amostras por clones, que foram carbonizados sob condições específicas para a análise da densidade aparente, compressão paralela às fibras e degradação linear e volumétrica devido à alta temperatura. Vale ressaltar que os dados foram ponderados pelo disco e por árvore, para a uma média mais próxima da realidade. Para as correlações, foi utilizada a análise multivariada de componentes principais.

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Nisto, verificou-se a ação da densidade aparente do carvão como o ponto focal das outras propriedades estudadas e observou-se que quanto maior a densidade, maior será a força de compressão paralela à grã, o módulo de elasticidade e o rendimento gravimétrico.
Palavras-chave: caracterização energética; análise dos componentes principais; Eucalipto.

INTRODUCTION

Nearly 53% of the wood produced in the world has some energetic purpose (FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS, 2014). In Brazil, this one can be considered as the fourth primary source of energy, being that in the production of charcoal, the country is world leader in production and consume, using it mainly in steel making, as bio reducer, and in the residential sector (BRITO, 2007). Emphasizing that about 35% of the firewood produced in the country, nearly 27 million of tons per year, is transformed into charcoal seeking, among other, the energy supply of industries, corresponding to 4.1% of the energy utilized by the sector (EMPRESA DE PESQUISA ENERGÉTICA, 2014).

According to Heimman, Dresch and Almeida (2015), Brazil is the largest producer and/or charcoal consumer in the world, exclusively on the use of this input on a large scale, such as bio-reducer in the steel industry.

The establishment of forests to the production of charcoal involves selection of superior genetic material and adoption of silvicultural techniques, allying productivity of the forests to the desired quality of the wood for energetic purpose (OLIVEIRA et al., 2010). Trugilho et al. (2005) and Trugilho and Silva (2001) had shown that the utilization of wood for energy production, in spite of not being restrictive, depends on some internal characteristics of the wood, as for example, the basic density and its variation inside the timber. Works inherent to correlation between chemical and physical properties of wood with chemical and physical properties of the charcoal are not uncommon to be found (TRUGILHO; LIMA; MORI, 2003; PROTÁSIO et al., 2012; 2013; COUTO et al., 2013), however, it is questioned how the properties of the charcoal interact among themselves.

For steel uses, where the bio-reducer has also functions supporting the iron ore load inside the furnace, it is essential that the charcoal present the greatest possible strength and stiffness, highlighting the practical and scientific importance of the study of the mechanical properties.

By the exposed, the present subject referees to the physical and mechanical analysis of the produced charcoal from ten clones of *Eucalyptus* and *Corymbia*, in the comprehension of its properties referring to apparent density, mechanical resistance, gravimetric yield and linear and volumetric degradations, with the analysis of principal components, seeking to demonstrate the relationship existent between this properties, in order to assess which charcoal property should be worked in the process to the acquisition of a product with more suitable characteristics to the steel making use.

MATERIALS AND EXPERIMENTAL METHODS

Material of study and characterization of the area

They were collected eight *Eucalyptus* clones and two *Corymbia* clones (Table 1), with approximately six years old, in the municipalities of Capelinha (Cap) and Itamandiba (Ita), State of Minas Gerais – Brazil, in the geographic coordinates 17º 41’ 38”S and 42º 31’ 07”O, and 1.070 m of altitude. It’s important to say that the weather, soil correction, spacing and other silvicultural treatments were homogeneous for all the worked clones.

To each clone studied, were selected 3 (three) trees, in a total of 30 trees. After the cut, it was removed discs from the logs, with 15 cm of thickness, at six different heights, to mention 0%, diameter at 1,30 m (DBH), 25%, 50%, 75% and 100% of the trunk. The last diameter limit was 6 cm with bark.

TABLE 1: Clones of *Eucalyptus* and *Corymbia*: origin, identification, age and collection site.
TABELA 1: Clones de *Eucalyptus* e *Corymbia*: origem, identificação, idade e local de coleta.

Clone	Clone (n.)	Age (years)	Collection site (site/city)
Corymbia toreliana x *Corymbia citriodora*	06	5	São Bento (Ita)
Corymbia citriodora x *Corymbia toreliana*	43	5	São Bento (Ita)
Eucalyptus urophylla (4)	182	6	São Caetano (Cap)
Eucalyptus urophylla (5)	220	6	Cruz Grande (Ita)
Eucalyptus urophylla (6)	224	6	Cruz Grande (Ita)
Eucalyptus urophylla x *Eucalyptus grandis*	344	6	Cruz Grande (Ita)
Eucalyptus grandis x *Eucalyptus urophylla*	1528	6	Cruz Grande (Ita)
(*Eucalyptus camaldulensis* x *Eucalyptus grandis*)x *Eucalyptus urophylla*	2034	6	São Bento (Ita)
Eucalyptus urophylla x *Eucalyptus pellita*	2475	6	São Bento (Ita)
Eucalyptus urophylla Flores	-	5	São Bento (Ita)

Preparation of the samples

From the discs test specimens were produced with 2 cm x 2 cm x 4 cm, corresponding to tangential, radial and axial direction respectively. The number of test specimens taken from the disc ranged according to the diameter of the wood along the height of trees, trunks generating an average of 25 test specimens per tree or 75 test specimens per clone.

The test specimens from the wood were kept in air forced circulation hothouse (100º C) until reach 0% of humidity, because it is known that moisture content can affect the charcoal yield and its properties. The test specimens from the wood were identified and determined the linear (tangential, radial e axial) dimensions (digital caliper; 0.01 mm resolution; ± 0.005 mm precision) as described in ABNT NBR 7190 (1997), to evaluate thermal degradation after the carbonization process.

Posteriorly, the same test specimens were remarked with copy pencil, seeking to assure the identification and the position of the measures with the caliper after the carbonization process. The samples of wood were transferred to steel trays and placed in horizontal direction by the enchase in metallic rods. The pyrolysis of wood was carried out in an electric laboratory oven. The initial temperature was 100 ºC, and the final temperature was 400 ºC, remaining stable in the last 60 minutes which an average heating rate of 1 ºC min^{-1}. These experiment conditions were used according to Trugilho and Silva (2001) and adapted according to the equipment limitations. The muffle furnace was held closed until the return to the ambient temperature, when the test specimens were removed and analyzed.

We determined gravimetric yield in dry base of the charcoal (Eq. 1) and linear/volumetric degradation of the test specimens of charcoal (Eq. 2). The volumetric degradation is the sum of the linear degradations.

$$RGC = \frac{Mc}{Mm} \, x \, 100\% \quad (1)$$

$$Dl = \frac{Lm - Lc}{Lm} \, x \, 100\% \quad (2)$$

where: * (1) RGC = gravimetric Yield (%); MC = dry charcoal mass (g) and MM = dry wood mass (g); (2) Dl= Linear degradation; Lm = wood's size; Lc = charcoal's size.

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

To the mechanical assays of resistance to parallel compression to the charcoal fibers, it was used a universal testing machine, model EMIC DL-300. Due to the absence of a specific standard that could direct the works of mechanical assay in charcoal, extra samples were carbonized to drive assay seeking to comprehend the charcoal behavior and obtain preliminary results to the confection of a script that could provide the necessary results on a consistent way.

Thereby, it was determined the load speed in 0.2 $mmmin^{-1}$ and it was inserted a collapse detector when the abrupt loss of 20% of the material resistance, indicating the rupture of the test specimen and the not existing possibility of its structural arrange, ending the assay. It was obtained the values of the resistance to compression and the module of elasticity, where this last one was calculated by means of a secant line from the graphic of the force exerted by the material deformation at the points between 60Kgf and 120Kgf.

Emphasizing that, after the obtainment of inherent data to the studied properties of the wood and the charcoal, they were weighted considering the position in the disc area and height of it, aiming to provide a greater accuracy to the mean values.

The values of physical and mechanical properties of the charcoal were submitted to the analysis of principal components (PCA), allowing to evaluate the influence of the physical and mechanical properties among themselves and the similarity of the individuals by one graphic dispersion.

All analysis procedures were performed using software R version 2.11.0 (R DEVELOPMENT CORE TEAM, 2011), package agricolae (MENDIBURU, 2013).

RESULTS AND DISCUSSION

As for the apparent density, it was verified that the clones *Corymbia toreliana* x *Corymbia citriodora* and *Corymbia citriodora* x *Corymbia toreliana* presented the greatest values among the studied species, 0.470 gcm^{-3} and 0.480 gcm^{-3}, respectively; while *Eucalyptus urophylla* (6) presented the minors values, 0.300 gcm^{-3} (Table 2).

For Vieira (2009) and Andrade and Machado (2004), the apparent density is one important query for the utilization of charcoal for steel making purpose, considering that it reflects directly in the utilization of the volume of the high-oven, to example that, the lower apparent density of the charcoal, the lower it will be the useful volume to the production of pig iron.

As for the thermal degradation due to de action of the temperature, it is noticed a behavior similar to the contraction of wood when there is water loss. The tangential plane contracts more than the radial plane that, which following, contracts more than the axial plane, to mention the averages of 19%, 15%, and 11% respectively, to the studied species. At this point it is worth to emphasize that the heating was made resembling inside the muffle furnace to all of the test specimens, as well as the same were arranged randomly, what allow us to discard a significant influence of the carbonization process and the equipment in the obtainment of this result. It is important to mention that the pyrolysis is a thermal degradation phenomenon, as well the volumetric and linear dimension was taken in dried test specimens so, in this way, was not expected the resemblance to the wood shrinkage behavior. It is known that the wood shrinkage by the exchange of molecules of water with the ambient occurs, mainly, due to the approximation of the microfibrils of cellulose. Analyzing more minutely it is noticed that the relative proportion of latewood per unit length in the specimens used is greater in the tangential plane than in the radial plane, which help understand, among other factors, the highest values of the first mentioned plan. Thereby, it is assumed also that there is a bigger proportion of wall cell to be degraded and, consequently, a bigger loss of area. This is in agreement with theory by Durlo and Machiori (1992) to explain the difference in shrinkage between the longitudinal planes of the wood.

The gravimetric yield of the worked clones stayed about 35%, where *Eucalyptus grandis* x *Eucalyptus urophylla* and *Eucalyptus urophylla* x *Eucalyptus pellita* presented the minors values of yield, about 30%, while the hybrids of the genus *Corymbia* presented the major values of yield, about 40%. Pereira et al. (2013) found gravimetric yield of 35% for different Eucalyptus clones with maximum temperature of 450 ºC and speed of carbonization of 1.67 ºC min^{-1}. This authors report that when goal is a high yield into charcoal the quality of lignin (low S/G ratio) is more relevant than the lignin contents. Oliveira et al. (1982), when worked with the carbonization of *Eucalyptus grandis* under maximum temperature of 450 ºC, found

TABLE 2: General characterization of the charcoal produced of studied species.
TABELA 2: Caracterização geral do carvão produzido das espécies estudadas.

Species	R_{c0}(MPa)	MOE (MPa)	Degradation of the test specimens due to the action of the temperature (%)				GYC (%)	AD (g cm^{-3})
			Tg	Rd	Ax	Vol		
Corymbia toreliana x *Corymbia citriodora*	15.58^{a}	696^{a}	21.09^{ac}	17.55^{ab}	9.20^{a}	59.33^{ab}	39.27^{ab}	0.47^{a}
Corymbia citriodora x *Corymbia toreliana*	15.69^{a}	736^{a}	24.08^{ab}	19.83^{a}	10.90^{a}	55.24^{b}	43.70^{a}	0.48^{a}
Eucalyptus urophylla (4)	12.89^{ab}	415^{bc}	17.40^{cde}	13.06^{bc}	9.64^{a}	64.43^{a}	33.00^{b}	0.34^{bc}
Eucalyptus urophylla (5)	11.06^{bc}	535^{b}	16.13^{def}	16.66^{abc}	12.79^{a}	34.26^{c}	37.71^{ab}	0.34^{bc}
Eucalyptus urophylla (6)	10.44^{bcd}	381^{c}	17.22^{cde}	11.90^{c}	9.78^{a}	65.66^{a}	32.29^{b}	0.30^{c}
Eucalyptus urophylla x *Eucalyptus grandis*	11.37^{bc}	399^{bc}	19.55^{bcd}	15.96^{abc}	10.88^{a}	38.80^{c}	35.06^{b}	0.31^{c}
Eucalyptus grandis x *Eucalyptus urophylla*	10.86^{bc}	436^{bc}	15.52^{e}	12.76^{bc}	10.35^{a}	39.00^{c}	32.36^{b}	0.33^{bc}
(*Eucalyptus camaldulensis* x *Eucalyptus grandis*) x *Eucalyptus urophylla*	8.28^{cd}	316^{c}	17.14^{def}	15.04^{abc}	12.03^{a}	34.51^{c}	35.65^{ab}	0.35^{bc}
Eucalyptus urophylla x *Eucalyptus pellita*	7.32^{d}	463^{bc}	13.06^{f}	14.17^{bc}	11.93^{a}	63.00^{a}	32.84^{b}	0.31^{c}
Eucalyptus urophylla Flores	13.62^{abc}	534^{b}	21.56^{ab}	14.24^{bc}	10.23^{a}	40.61^{c}	37.66^{ab}	0.39^{b}

Where:Tg= Tangential, Rd = Radial, Ax= Axial and Vol= volumetric; R_{c0} = Resistance to compression parallel fibers; MOE = Module of elasticity; GYC = gravimetric yield in coal; AD = apparent density. Means followed by the same letter do not differ at 5% of significance, by Tukey test.

yield of 32.89%, while Vella et al. (1989), under same maxim temperature and speed of carbonization of 0.57 ºC min^{-1} obtained yield of about 33% to *Eucalyptus tereticornis*. However, Santiago and Andrade (2010) obtained yield of 27%, when carbonized under temperature of 400 ºC specimens of *Eucalyptus urophylla* of seven years old. This way it is verified that the specimens worked had values of gravimetric yield resembling or superior to that found in the literature.

The resistance and the module of elasticity to the compression parallel to the charcoal fibers were, in average, of 12 MPa and 490 MPa, respectively. Vieira (2009), when worked with two clones of *Eucalyptus* carbonized at 450 ºC, found values of 95 MPa and 142 MPa of resistance to the compression parallel to the fibers, and module of elasticity of 475 MPa and 571 MPa. However, the same author, when worked with maxim temperature of carbonization at 300 ºC, verified that the values of resistance to the compression decrease significantly, to cite 7 MPa and 8 MPa, approaching of the results obtained in this work.

Such differences can be elucidated by the work of Blankehorn, Kline and Beal (1973) that, when worked with the resistance to the compression parallel of the fibers and module of elasticity of the charcoal produced in different temperatures, verified one decrease behavior of the resistance until approximately 500 ºC, when this tendency is inverted. The phenomenon occurrence can be explained by the instability of the beams of graphite formed between 300 ºC and 500 ºC, as well as by the expressive loss of the hydrogen, crossing the beams of graphite with material and by the orientation of the mass of fibrils between the band from 500 ºC to 900 ºC (BLANKENHORN; JENKINS; KLINE, 1972).

This way, considering the possibility of the utilization of the worked material as source of energy and bio reducer to steel making, it is interesting the study referent to the increase of the maxim temperature of carbonization to the increase of resistance of the produced charcoal, seen the overhead the charcoal suffers when inserted into the blast furnaces.

It can be observed in Table 3, the explained and accumulated variances in each principal component, as well as its respective graphic representation (Figure 1) in function of the principal components. Emphasizing that the importance of analyzing both the graphic and the table is the acquired notion of the quantity of necessary components to explain the majority of the data variance. In this case, utilizing three principal components it is possible to explain 91% of the original data variance, wherethe first two (PC1 and PC2) are responsible for 81%.

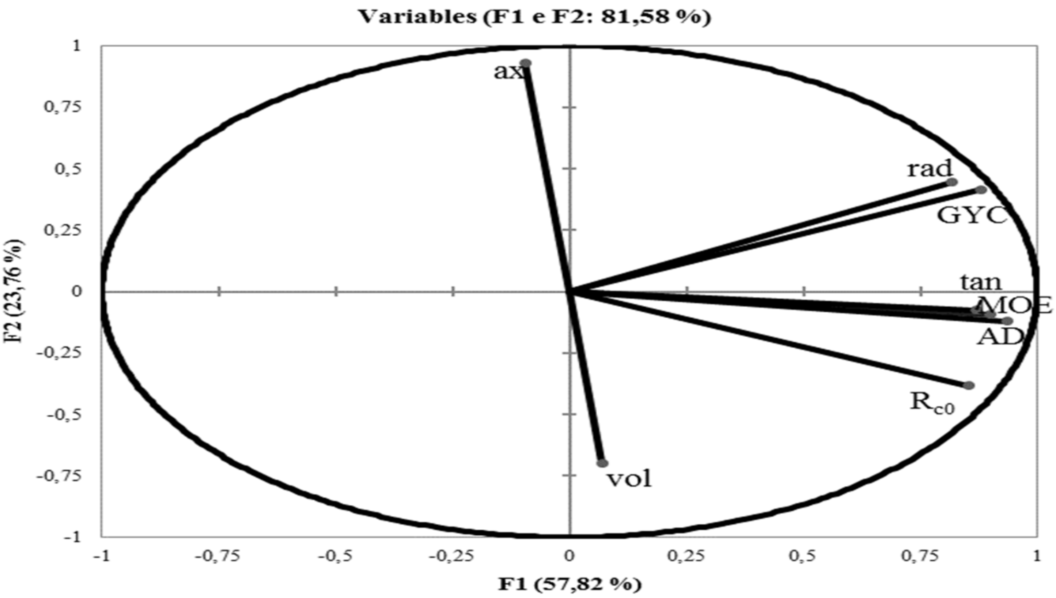
TABLE 3: Explained and accumulated variances in each of the major components for the ten clones evaluated.
TABELA 3: Variâncias explicadas e acumuladas em cada um dos componentes principais para os dez clones avaliados.

Principal Components	Explained variance (%)	Accumulated variance (%)
PC 1	57.82	57.82
PC 2	23.76	81.58
PC 3	9.77	91.34
PC 4	3.99	95.34
PC 5	1.93	97.26
PC 6	1.78	99.05
PC 7	0.70	99.75
PC 8	0.25	100.00

TABLE 4: Auto vectors normalized of the three first principal components and the contribution of each variable.
TABELA 4: Vetores Autonormalizados dos três primeiros componentes principais e a contribuição de cada variavél.

Variable	Auto vectors normalized					
	PC 1	FC (%)	PC 2	FC (%)	PC 3	FC (%)
Resistance to the parallel compression to the charcoal fibers	0.85*	15.7*	-0.38	7.7	-0.23	6.5
Module of elasticity	0.90*	17.5*	-0.09	0.5	0.21	5.5
Tangential linear degradation	0.87*	16.3*	-0.08	0.3	-0.32	12.9
Radial linear degradation	0.82*	14.4*	0.45	10.4	0.19	4.5
Axial linear degradation	-0.10	0.2	0.93*	45.7*	0.29	10.4
Degradation in volume	0.07	0.1	-0.70	25.6	0.68*	59.0*
Gravimetric yield	0.88*	16.8*	0.41	9.0	0.09	1.1
Apparent density	0.93*	18.9*	-0.12	0.8	0.03	0.1

Where: * Significant correlation at 5%; PC = Principal Components; FC = Factor Contribution



Where: R_{c0}= resistance to the parallel compression to the charcoal fibers, MOE = module of elasticity. tan = tangential linear degradation, rad = Radial linear degradation, ax = Axial linear degradation, GYC = Gravimetric yield, AD= apparent density

FIGURE 1: Graphic analysis of the properties of the studied clones charcoal by its principal components.

FIGURA 1: Análise gráfica das propriedades do carvão dos clones estudados pelos seus principais componentes.

In the principal component 1, the variables density, modulus of elasticity, compression strength, charcoal yield and degradation tangential / radial explained 57% of the total variation.

As for the auto vectors normalized (Table 4) of the three first principal components, the higher coefficients, above 0.9; refers to the resistance to the compression parallel to the fibers, to the module of elasticity, to the radial and tangential linear degradation, to the gravimetric yield and the apparent density to the first component, demonstrating a strong correlation among themselves. The second and third components, on the other hand, present high values for axial linear degradation and degradation in volume respectively, not having strong correlation between the other properties.

As for its relations of influence, it can be stated that, the apparent density presented as a convergence point of the major part of the analyzed properties (Figure 2), to cite: radial and tangential linear degradation, resistance to the parallel compression of the charcoal fibers, gravimetric yield and module of elasticity (MOE), emphasizing that the line of this last one almost overlapped to the apparent density, what infers in one strong positive correlation.

In parallel, it is verified a positive relation between the module of elasticity and the radial linear degradation, as well as between the resistance to the compression and the tangential linear degradation. It is worth to cite that the variable until now described are found well represented in the graphic plane due to its approximation of the unitary circle, contrary to the axial and in volume linear degradation, which are distant when compared with the others. However, it is possible to infer a negative relation between the axial linear degradation and the resistance to the compression / tangential linear degradation.

CONCLUSION

To the studied clones, the resistance to the parallel compression to the fibers, modulus of elasticity and gravimetric yield is significantly correlated with charcoal apparent density.

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