CHEMICAL AND ENERGETIC CHARACTERIZATION OF *Eucalyptus grandis × Eucalyptus urophylla* CLONES SUBJECT TO WIND DAMAGE

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ABSTRACT – Wind damages are common in forest plantations and the use of this wood can minimize losses. The objective was to evaluate the chemical composition and the energetic potential of wood and charcoal from trees subject to wind damage. Eight (A to H) two-years-old *Eucalyptus grandis × Eucalyptus urophylla* clones were selected in a region where wind damage is frequent. The basic density, calorific value, chemical composition of wood and calorific value, immediate chemistry and gravimetric yield of charcoal were determined for all clones. Materials with high lignin content and low S/G ratio had higher gravimetric yield. The energy density of wood and charcoal showed high relationship with the basic and apparent relative density, respectively. All materials showed potential for bioenergy, but the clone E stood out with higher gravimetric yield and energy density.

Keywords: Calorific value; Charcoal; Lignin.

CARACTERIZAÇÃO QUÍMICA E ENERGÉTICA DE CLONES DE Eucalyptus grandis × Eucalyptus urophylla SUJEITOS A DANOS PELOS VENTOS

RESUMO – Danos por ventos são frequentes em plantações florestais, e a utilização desta madeira pode minimizar os prejuízos causados. O objetivo foi avaliar a composição química e o potencial energético da madeira e carvão de árvores sujeitas a danos pelos ventos. Oito clones de **Eucalyptus grandis × Eucalyptus urophylla** *com idade de dois anos foram selecionados em região onde danos por ventos ocorrem com alta frequência. A densidade básica, poder calorífico, composição química da madeira e poder calorífico, química imediata e rendimento gravimétrico do carvão foram determinados para todos os clones. O rendimento gravimétrico de materiais com alto teor de lignina e baixa relação S/G foi maior. A densidade energética da madeira e carvão apresentou maior relação com a densidade básica e aparente, respectivamente. Todos os materiais apresentaram potencial para bioenergia, mas o clone E se destacou com alto rendimento gravimétrico e densidade energética.*

Palavras-Chave: Carvão Vegetal; Lignina; Poder calorífico.

1. INTRODUCTION

The winds are characterized by the air movement from areas with high to low pressures (Moore et al., 2013; Kramer et al., 2014; Hale et al., 2015). Wind damage in forests are reported since 1940 (Mitchell et al., 2012) in many regions of the world (Lagergren et al., 2012; Moore et al., 2013; Kramer et al., 2014).

Wind damages occur mainly from 24 to 36 months old in eucalyptus plants and it can bend or break the trees (CENIBRA, 2014). The first causes loss of apical dominance and reduced the productivity. In the second, it is necessary to harvest broken material and plant a new forest. In both cases, losses are considerable and threaten eucalyptus plantations.

Trees broken by winds are mainly young ones with smaller diameter, low density and poor quality fibers (Veenin et al., 2005; Ramírez et al., 2009), hindering its use in pulp (Severo et al., 2013; Pirralho et al., 2014) and lumber (Luna et al., 2013) production. Therefore, these materials are used mainly for energy purposes (Guerra et al., 2014).

The aim of this study was to characterize the chemical and energetic potential of wood and charcoal from eucalyptus clones subject to wind damage aiming to find a better use for these materials.

2. MATERIAL AND METHODS

2.1. Biological Material

Three trees per each of eight (A to H) two-years-old *Eucalyptus grandis* × *Eucalyptus urophylla* different clones were collected in the region of Belo Oriente, Minas Gerais state, Brazil (42º22'30" S and 19º15'00" O). This age and region were chosen because they have high incidence of wind damage.

Disks were withdrawn at 1.3 meters above ground level, from each felled tree, the basic density, chemical and energetic properties of these materials were analyzed.

2.2. Wood chemical characterization

One disk from each tree were milled with a Standard Wiley knife mill with a 2 mm screen. This material was sieved with a 40-60 mesh sieve and the retained fraction was used to determine the total extractives according to ASTM D-1105-94 (ASTM, 1994); besides the insoluble lignin (Gomide and Demuner, 1986); soluble lignin (Goldschimid, 1971) and Siringil/Guaiacil (S/G) ratio (Lin and Dence, 1992). The total lignin was obtained with the sum of soluble and insoluble lignin. Finally, the holocellulose content was determined by subtracting these components from 100.

The same sample was used for elemental analysis. The carbon, hydrogen and nitrogen content, based in wood dry mass, were quantified with a universal analyzer Vario Microcube model. The oxygen content was obtained by subtracting the carbon, hydrogen and nitrogen from 100.

2.3. Physical and energetic characterization of wood and charcoal

The wood basic density was determined according to NBR 11941 (ABNT, 2003), the gross calorific value according to NBR 8633 (ABNT, 1984) and the wood energy density by the product of these two parameters.

The wood was carbonized at 1.67°C/min heating rate, until 450°C and 30 min residence time in electric furnace at atmospheric pressure and controlled presence of oxygen. The ash, volatile matter and fixed carbon were evaluated according to ABNT NBR 8112 (ABNT, 1983): the gross calorific value according to ABNT NBR 8633 (ABNT, 1983) and apparent relative density according to ABNT NBR 9165 (ABNT, 1985). The charcoal energy density was determined by the product of the apparent relative density and gross calorific value. The elemental analysis of charcoal was performed similarly to that of the timber.

2.4. Statistical analysis

The variance homogeneity (Bartlett's test at 5% significance) and normality test were performed (Shapiro-Wilk test at 5% significance). Means of treatments were compared with the Scott-Knott test at 5% probability.

3. RESULTS

3.1. Wood chemical characterization

The extractives, ash, soluble, insoluble and total lignin, holocellulose and the S/G ratio of the eight different clones evaluated were determined to characterize the wood (Table 1).

Table 1 – Chemical composition of eight different two-years-old *Eucalyptus grandis* × *Eucalyptus urophylla* clones (Cl.).
Tabela 1 – *Composição química de oito diferentes clones de **Eucalyptus grandis** × **Eucalyptus urophylla** (Cl.) com dois anos de idade.*

Cl.	Ext. (%)	Ash (%)	Sol. Lig (%)	Ins. Lig (%)	Tot. Lig. (%)	S/G	Hol. (%)
A	$2.54^{3.7}$ c	$0.425^{4.7}$ a	$4.4^{2.1}$ a	$25.2^{2.3}$ a	$29.6^{2.7}$ a	$2.99^{1.8}$ a	$67.44^{2.5}$ b
B	$0.73^{1.6}$ a	$0.370^{5.3}$ a	$4.5^{3.4}$ a	$24.5^{2.4}$ a	$29.0^{2.6}$ a	$3.93^{2.2}$ c	$69.90^{2.7}$ c
C	$2.25^{3.6}$ c	$0.450^{5.9}$ a	$4.1^{3.9}$ a	$26.4^{2.9}$ b	$30.5^{3.2}$ b	$3.42^{3.3}$ b	$66.8^{3.2}$ b
D	$2.95^{3.1}$ d	$0.385^{5.7}$ a	$4.7^{4.1}$ a	$26.0^{3.7}$ b	$30.7^{3.7}$ b	$3.11^{2.7}$ a	$65.97^{3.3}$ a
E	$1.23^{3.9}$ b	$0.405^{6.1}$ a	$4.3^{4.2}$ a	$26.8^{4.2}$ b	$31.1^{4.1}$ b	$3.11^{4.3}$ a	$67.27^{3.9}$ b
F	$2.95^{4.2}$ d	$0.420^{5.3}$ a	$4.7^{2.9}$ a	$26.3^{3.5}$ b	$31.0^{3.3}$ b	$2.97^{2.9}$ a	$65.63^{3.3}$ a
G	$2.71^{2.8}$ c	$0.390^{6.3}$ a	$4.2^{3.2}$ a	$26.4^{3.8}$ b	$30.6^{4.0}$ b	$3.31^{2.9}$ b	$66.30^{4.0}$ a
H	$2.17^{3.2}$ c	$0.405^{5.9}$ a	$4.1^{3.5}$ a	$27.8^{4.7}$ c	$31.9^{4.3}$ c	$2.98^{3.5}$ a	$65.53^{4.3}$ a

Cl.	Carbon (%)	Oxygen (%)	Hydrogen (%)	Nitrogen (%)
A	$50.78^{4.8}$ a	$42.58^{4.6}$ a	$5.88^{5.2}$ a	$0.02^{5.2}$ a
B	$50.55^{4.1}$ a	$42.67^{4.3}$ a	$5.78^{3.8}$ a	$0.06^{5.5}$ a
C	$50.66^{4.0}$ a	$42.80^{3.4}$ a	$5.85^{4.2}$ a	$0.03^{4.1}$ a
D	$50.49^{3.9}$ a	$43.02^{4.1}$ a	$5.76^{4.6}$ a	$0.03^{4.2}$ a
E	$50.90^{4.5}$ a	$42.35^{2.8}$ a	$5.90^{4.2}$ a	$0.05^{4.4}$ a
F	$50.77^{4.6}$ a	$42.63^{3.7}$ a	$5.89^{4.7}$ a	$0.08^{4.8}$ a
G	$50.43^{3.7}$ a	$42.95^{4.1}$ a	$5.85^{3.6}$ a	$0.03^{4.7}$ a
H	$50.30^{4.6}$ a	$43.28^{3.5}$ a	$5.82^{4.2}$ a	$0.07^{4.2}$ a

Ext.= Extractives; Sol. Lig.= soluble lignin; Ins. Lig= Insoluble lignin; Tot. Lig.= Total lignin; S/G= syringyl/guaiacyl ratio in the lignin; Hol.= holocellulose. Means followed by the same letter does not differ by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation.
Ext.= Extrativos; Sol. Lig.= lignina solúvel; Ins. Lig= lignina insolúvel; Tot. Lig.= lignina total; S/G= relação siringil/guaiacil na lignina; Hol.= holocelulose. Médias seguidas pela mesma letra não diferem pelo teste de Scott-Knott a 5%. Valores em sobrescrito representam o coeficiente de variação.

3.2. Physical and energetic characterization of wood and charcoal

The wood of *Eucalyptus grandis* × *Eucalyptus urophylla* clones showed higher basic density, while the charcoal produced presented high calorific value and energy density (Table 2).

The gravimetric yield in charcoal production ranged from 30.60 to 33.84%, the fixed carbon from 17.99 to 23.28%, volatile matter from 17.99 to 23.28% and ash from 0.600 to 0.685% (Table 3).

Table 2 – Basic density and apparent relative density (g.cm⁻³), gross calorific value (MJ.g⁻¹) and energy density (MJ.cm⁻³) of the wood and charcoal from eight *Eucalyptus grandis* × *Eucalyptus urophylla* clones.
Tabela 2 – *Densidade básica e densidade relativa aparente (g.cm⁻³), poder calorífico superior (MJ.g⁻¹) e densidade energética (MJ.cm⁻³) da madeira e do carvão vegetal de oito clones de **Eucalyptus grandis** × **Eucalyptus urophylla**.*

Clone	Wood		
	Basic density	Gross calorific value	Energy density
A	$0.412^{3.1}$ b	$19.13^{4.2}$ a	$7.92^{3.7}$ c
B	$0.331^{4.5}$ a	$19.95^{4.6}$ b	$6.61^{3.8}$ a
C	$0.372^{4.0}$ a	$19.80^{4.7}$ b	$7.31^{3.4}$ b
D	$0.423^{3.6}$ c	$19.92^{3.5}$ b	$8.44^{4.1}$ d
E	$0.421^{4.1}$ c	$19.80^{3.6}$ b	$8.31^{3.3}$ d
F	$0.370^{3.5}$ a	$19.79^{4.1}$ b	$7.29^{3.7}$ b
G	$0.370^{3.9}$ a	$19.02^{3.8}$ a	$7.09^{4.2}$ b
H	$0.383^{3.1}$ a	$19.96^{4.1}$ b	$7.65^{3.9}$ b

Clone	Charcoal		
	Apparent relative density	Gross calorific value	Energy density
A	$0.305^{2.5}$ c	$29.68^{4.3}$ a	$9.01^{3.8}$ e
B	$0.235^{3.1}$ a	$29.89^{3.7}$ a	$7.12^{3.2}$ b
C	$0.241^{3.4}$ a	$29.29^{4.8}$ a	$7.15^{4.2}$ b
D	$0.296^{3.6}$ c	$29.29^{5.1}$ a	$8.61^{4.2}$ d
E	$0.308^{3.4}$ c	$29.30^{4.5}$ a	$9.08^{3.7}$ e
F	$0.227^{3.5}$ a	$29.30^{3.8}$ a	$6.68^{3.6}$ a
G	$0.258^{3.1}$ a	$29.31^{4.3}$ a	$7.51^{3.3}$ b
H	$0.271^{2.8}$ b	$29.31^{4.1}$ a	$7.94^{3.4}$ c

Means followed by the same letter does not differ by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation.
Médias seguidas pela mesma letra não diferem pelo teste de Scott-Knott a 5%. Valores em sobrescrito representam o coeficiente de variação.

Table 3 – Gravimetric yield (Grav), fixed carbon (FC), volatile matter (VM), ash (%) and elemental composition from charcoal of eight *Eucalyptus grandis* × *Eucalyptus urophylla* clones.

Tabela 3 – *Rendimento gravimétrico (Grav), carbono fixo (FC), materiais voláteis (VM), cinzas (%) e composição elementar do carvão de oito clones de **Eucalyptus grandis** × **Eucalyptus urophylla**.*

Clone	Grav (%)	FC (%)	VM (%)	Ash (%)
A	$32.29^{4.5}$ b	$76.22^{2.5}$ a	$23.28^{3.5}$ c	$0.600^{5.5}$ a
B	$30.96^{4.3}$ a	$79.86^{3.6}$ b	$19.49^{3.7}$ b	$0.625^{6.2}$ a
C	$31.73^{3.2}$ b	$81.15^{3.3}$ c	$18.40^{2.4}$ a	$0.685^{6.4}$ a
D	$31.13^{4.1}$ a	$79.69^{4.1}$ b	$19.61^{2.6}$ b	$0.660^{5.2}$ a
E	$33.42^{2.5}$ c	$76.35^{2.7}$ a	$23.00^{3.2}$ c	$0.645^{5.6}$ a
F	$33.47^{3.3}$ c	$78.63^{2.8}$ b	$20.72^{3.6}$ b	$0.655^{6.2}$ a
G	$30.60^{2.7}$ a	$81.36^{3.4}$ c	$17.99^{3.6}$ a	$0.645^{5.6}$ a
H	$33.84^{3.6}$ c	$77.16^{3.2}$ a	$22.14^{2.9}$ c	$0.670^{5.2}$ a

Clone	Carbon (%)	Oxygen (%)	Hydrogen (%)	Nitrogen (%)
A	$82.38^{4.5}$c	$11.75^{4.8}$d	$3.28^{4.3}$a	$0.13^{5.6}$a
B	$80.08^{3.9}$b	$14.10^{3.6}$c	$3.20^{4.6}$a	$0.12^{6.1}$a
C	$81.43^{4.2}$c	$13.44^{4.2}$c	$3.20^{4.8}$a	$0.09^{5.3}$a
D	$80.91^{4.1}$b	$14.19^{4.4}$c	$3.29^{4.1}$a	$0.11^{5.8}$a
E	$78.01^{3.7}$a	$16.08^{3.9}$b	$3.15^{3.8}$a	$0.16^{6.4}$a
F	$77.41^{3.8}$a	$18.01^{4.6}$a	$3.37^{3.6}$a	$0.07^{5.9}$a
G	$77.68^{4.5}$a	$17.57^{4.1}$a	$3.32^{4.0}$a	$0.07^{6.0}$a
H	$77.68^{4.2}$a	$17.31^{3.4}$a	$3.31^{4.2}$a	$0.06^{6.4}$a

Means followed by the same letter does not differ by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation.

Médias seguidas pela mesma letra não diferem pelo teste de Scott-Knott a 5%. Valores em sobrescrito representam o coeficiente de variação.

4. DISCUSSION

4.1. Wood chemical characterization

The clone A showed a lower extractive content, while the clones D and F had the highest (Table 1). The extractive content of the eight clones evaluated was lower than those reported for seven-years-old *Eucalyptus grandis* × *Eucalyptus urophylla*, *Eucalyptus urophylla* and *Eucalyptus paniculata*, 3.41 and 9.12% (Arantes et al., 2011; Zanuncio et al., 2014). The proportion of extractives in the xylem is higher at the heartwood (Adamapoulos et al., 2005), the process that turns sapwood into heartwood is incipient in two-years-old trees (Sousa et al., 2013; Gominho et al., 2015), resulting in woods with low extractives content. For energy use, some extractive classes, such as those soluble in dichloromethane have high resistance to thermal degradation (Mészáros et al., 2007), which increases charcoal gravimetric yield, gross calorific value and volatile matter (Zanuncio et al., 2014). All materials showed similar ash quantity, which is very resistant, represent impurities and hinder the use of wood for energy (Bustamante-García et al., 2013).

The soluble lignin content of the materials was similar in all the eight clones, therefore, total lignin content followed the trend of insoluble lignin, with higher values for clone H and lower for A and B clones (Table 1). The lignin is important because of its high carbon content (Fengel and Wegener, 1984) and resistance to high temperatures (Varfolomeev et al., 2015), what makes its presence desirable for energy production. The lignin quality also influences the energy use (Pereira et al., 2013), because wood with high S/G ratio, as that of clone C, have structure with fewer linkages between carbons, and therefore, lower resistance to thermal degradation (Prasad et al., 2015).

The clones with lower lignin content showed higher holocellulose quantity, as reported for the clone B. Holocellulose has high oxygen content (Sjöströn, 1981) and poor resistance in high temperatures (Moreno and Font, 2015), reducing its calorific value and gravimetric yield (Liu and Han, 2015) and being unwanted in wood for energy production.

All wood materials showed similar elemental composition (Table 1). Materials with high carbon and low oxygen content are most desirable in the wood for energy production, because they increase wood calorific value and the gravimetric yield of carbonization (Soares et al., 2014). High nitrogen content is unwanted due to its pollution potential,

generating toxic oxides during charcoal combustion that can induce acid rain and soil acidification (Demirbas, 2004).

4.2. Physical and energetic characterization of wood and charcoal

The charcoal from clones with higher wood basic density had higher apparent relative density with Pearson correlation coefficient of 0.891 between these variables (Table 2). This trend was also reported for *E. grandis* × *E. urophylla* and *E. urophylla* with three, four, five and seven years old (Castro et al., 2013). The charcoal with apparent relative density has better mechanical properties and lower fine production and therefore desirable for energy production (Antal and Mok, 1990).

The gross calorific value showed low variation in the wood and charcoal. This parameter is related with wood chemistry, being directly proportional to the lignin (Pereira et al., 2013) and extractives content (Zanuncio et al., 2014), and inversely proportional to that of holocellulose (Liu and Han, 2015). The low variation in wood chemistry resulted in low variation of the calorific value of wood and charcoal.

Carbonization reduced the basic density and increased the gross calorific value of all clones (Table 2), being the second effect with highest proportion, and therefore, the charcoal had higher energy density than the wood in most of the clones selected. This trend was observed for *Eucalyptus grandis* × *Eucalyptus urophylla* with three, five and seven years old (Soares et al., 2014) and for native wood of *Luehea divaricata, Casearia sylvestris, Guazuma ulmifolia* and *Rapanea ferruginea* (Costa et al., 2014). The low variation of the gross calorific value of wood and charcoal among clones resulted in high relationship of energy density with wood basic density and charcoal relative apparent density, with Pearson correlation coefficient of 0.973 and 0.998, respectively.

The gravimetric yield of the clones E, F and H were higher (Table 3), with Pearson's correlation coefficient of 0.6358 and -0.6424 with the total lignin content and S/G ratio. The gravimetric yield is the main quantitative parameter for charcoal production (Rousset et al., 2011) showing how the quantity and quality of lignin in the wood are important for carbonization.

Clones C and G showed higher fixed carbon and low volatile matter (Table 3). High fixed carbon contents result in slow burning of the material and better mechanical properties of charcoal, facilitating its use in steelmaking. On the other hand, a high volatile matter content is important to the calorific value and charcoal reactivity (Antal and Mok, 1990; Demirbas, 2001; Rousset et al., 2011).

The ash content increases after carbonization in all materials (Table 3). This occurred because the minerals in the wood resist to high temperatures and, therefore, do not degrade during carbonization. Thus, the increase in ash percentage was due to the thermal degradation of other constituents (Moreno and Font, 2015). In the furnace for steel production, minerals present in charcoal may adversely affect the mechanical properties of steel, which makes them undesirable in this process (Oliveira et al., 1982).

The charcoal elemental composition varied with the genetic material, unlike the wood elemental composition, showing that the wood behavior at high temperatures can be complex. There was an increase in the carbon content and a decrease in oxygen and hydrogen content in for all materials. Carbon present in greater proportion in wood components with high resistance to thermal degradation, such as lignin, whereas the oxygen and hydrogen are present in in greater proportion in the holocellulose, which has low resistance to high temperatures.

5. CONCLUSIONS

The clones E, F and H had higher gravimetric yield, while the A, D and E higher energy density in the wood and charcoal. The gravimetric yield was correlated with the lignin content and S/G ratio, while the energy density had a higher relation to density. All clones showed potential for energy generation, especially the clone E, making this an important alternative to use wind broken trees.

6. ACKNOWLEDGMENT

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