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Anatomy of Charcoal and Carbonization Effect under *Eucalyptus Fibers' Dimensions*

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Abstract

The anatomy of wood and charcoal was analyzed by scanning electron microscopy in three radial positions of E. urophylla and E. Grandis in different diameter classes at six years. The carbonization process was given in the muffle with an average rate of 0.5 °C increment per minute, using initial temperature of 150 °C and maximum of 450 °C. It was observed that the pore diameter presented higher values in the samples obtained in the internal position. The diameter classes of the trees almost did not influence the characteristics of the fibers. In the external position (close to the shell) fibers with higher values of cell wall thickness and smaller values of lumen diameter and width were identified. The fiber lumen diameter was not affected by the wood carbonization process, whereas the cell wall thickness and fiber width were reduced.

Keywords: thermal wood degradation, scanning electron microscopy, pores and fibers of charcoal.

1. INTRODUCTION AND OBJECTIVES

Charcoal is widely utilized in Brazil because of its good qualities as fuel and in the reduction of iron ore. In the steel sector, there are some specific requirements regarding the final quality of charcoal, which is affected by wood and species characteristics from its origin.

The search for the quality of charcoal promotes chemical and anatomical studies of wood, considering that the knowledge of the raw material promotes the convenient use of the commercial qualities of the final product. During pyrolysis, the wood undergoes modifications in its coloration, as well as changes in the physical and mechanical characteristics that generate a product with greater dimensional stability, lower hygroscopicity and greater resistance against fungi (Moura & Brito, 2011; Moura et al., 2012).

The influence of tree diameter especially over wood drying process and its liability to produce anatomic deformation of the material are well known. Wood may crack under temperature influence and also modify fiber structure and collapse vessels (Rémond et al., 2007), besides other effects in the porous structure. The chemical composition also undergoes modifications such as dehydration and degradation of cellulose and hemicelluloses, in addition to mass loss, which tends to be higher with increasing temperature (Esteves & Pereira, 2009; Poubel et al., 2013). Although there is a lot of knowledge available on wood anatomy variation in radial and longitudinal direction, little is known about the variation effect of wood anatomy structure under high temperatures and even less regarding anatomy and structure of charcoal.

Anatomic description of carbonized wood vessels is a powerful tool which can be used in different field of studies,

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such as, botany, ecology, archeology and paleontology. Besides, it enables taxonomic identification, which can be employed for conservation purposes, to provide a path to anthacological studies in the tropics and also providing paleoenvironmental and paleoclimatic studies (Gonçalves et al., 2008). Furthermore, it can be applied to identify charcoal origin, whether came from planted or native forest, and also may contribute to wood inspection and control of forest trade (Gonçalves et al., 2012).

Scanning electron microscope (SEM) is one of the techniques used for anatomy studies in wood and charcoal. It allows the observation and analysis of microstructural characteristics of solid objects, which can quickly provide information on the morphology and identification of elements of wood or charcoal samples. Besides, SEM provides micrographs with a large depth of field, which allows a three-dimensional appearance of the structure specimen and also high-resolution images, generally lower than 1 ηm (Nagatani et al., 1987).

Few types of researches used SEM to characterize fiber dimensions of both wood and charcoal, especially regarding the modification of cell elements after carbonization from a transversal section of the trunk considering radial variations of these features.

After reviewing these studies, the aim of this work was to analyze the charcoal anatomy in the radial direction of transversal sections of the trunks, in which samples were collected in three tree diameter classes of *Eucalyptus* plantation. Also, the carbonization influence under fibers dimensions was verified applying SEM.

2. MATERIALS AND METHODS

It was collected six-year-old trees of *Eucalyptus urophylla* and *Eucalyptus grandis* clones planted in 3 × 2 m spacing, located at ArcelorMittal Forests in Martinho Campos, MG, Brazil. At the sample site, it was used a 10 × 10 trees plot where the diameter at breast height (DBH) of all 100 trees was measured at DBH regarding the classification in three distinct tree diameter classes.

It was chosen three tree diameter classes ranging from 3.1 to 9.5 cm of diameter (in which class mean was 8.1 cm), from 9.7 to 12.7 cm of diameter (in which class mean was 11.4 cm) and from 12.8 to 17.0 cm of diameter (in which class mean was 14.2 cm). Then, disc samples were collected from one representative tree at each tree diameter class.

Discs were obtained at DBH 2.5-cm thick, in which three wood samples were collected at three regions, in the external position (near the bark), intermediate zone (between bark and pith) and in the inner position (near the pith), being 2.0-mm thick each sample using a parallel double circular saw. Samples' measurements ensured to analyze cell wall thickness, fiber lumen diameter, and fiber width.

Double samples were collected, one to produce charcoals and the other to analyze wood. The carbonization process was given at muffle with an average rate of 0.5 °C increment per minute, using initial temperature of 150 °C and maximum temperature of 450 °C, remaining stable for 30 min. The total time of carbonization was 10 hours.

With wood and charcoal samples from the three distinct positions of the disc, they were mounted on an aluminum stub with tapes. Wood samples were coated with gold using sputter coater (Sputtering – Bal-Tec) and then observed in the SEM LEO EVO 40 XVP microscope.

It was measured 20 cell elements (fibers) for each sample of wood and charcoal at 2000× magnification. For charcoal, individual measurement of the fiber wall was not possible (Figure 1). As reported by Cutter et al. (1980), after a certain carbonization temperature, the walls of the fibers merge, making it impossible to see the boundary between the cells. Pereira et al. (2016) also observed such homogenization of the layers of pyrolyzed *Eucalyptus* cell walls at 450 °C.

Thus, fiber measurement was performed based on the methodology used by Abreu Neto (2015), in which three neighboring fibers were considered (Figure 1). The measurement of the width 1 (W1) and the fiber flame diameter (FLD) was used to determine the fiber width (LW) and the single cell wall thickness (CWT), with the aid of Equations 1 and 2 (Figure 1).

$$CWT = \frac{W1 - FLD}{4} \quad (1)$$

$$CWT = FLD + 2 \times CWT \quad (2)$$

Descriptive statistics was used for vessel diameter and frequency analysis. Mean values of the vessel diameter, wood fiber dimensions and charcoal were compared adopting a completely randomized design arranged in a factorial scheme of two factors (diametric class × radial position) and 20 replicates. With a significant interaction in the analysis, the Tukey test was performed at a level of 5% significance.

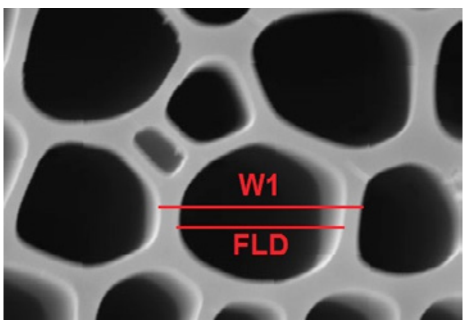

Figure 1. Homogenization of the fiber wall and measurement of width 1 (W1) and the fiber flame diameter (FLD) lumen observed at the SEM.

3. RESULTS AND DISCUSSION

3.1. Charcoal porosity

Mean values of pore diameter in the charcoal samples of *E. grandis* W. Hill ex Maiden × *E. urophylla* S. T. Blake clones were described in Table 1. Performing Anova enabled us to identify the interaction between the factors (tree diameter class × radial position) and the diameter of pores. These results indicate a dependence between factors and that we should proceed to the evaluation of one effect within another.

It was observed a reduction, considering pore diameter in the charcoal samples, from the external region for the tree diameter class of 11.4 and 8.1 cm, the lowest result was also verified in the intermediate zone of the diametrical class of 11.4 cm. Concerning the influence of the diametric class, it was observed that, in the external zone, the highest diametrical class (14.1 cm) had the largest pore diameter.

Diffuse porosity was observed in the samples of the inner region, with solids and multiple pores dispersed. The samples from the intermediate and external regions presented a diagonal arrangement (Figure 2).

Table 1. Mean values of diameter of pore in the charcoal samples of *Eucalyptus grandis* W. Hill ex Maiden × *Eucalyptus urophylla* S. T. Blake clones.

Tree diameter class (cm)	External (μm)	Intermediate (μm)	Inner (μm)
14.1	109.534 Aa	110.094 Aa	115.623 Aa
11.4	86.237 Bb	87.603 Bb	123.595 Aa
8.1	88.387 Bb	101.307 Aab	113.715 Aa

Means followed by the same lowercase letters in columns or by the same uppercase letters in lines do not differ of 5% probability by Tukey test.

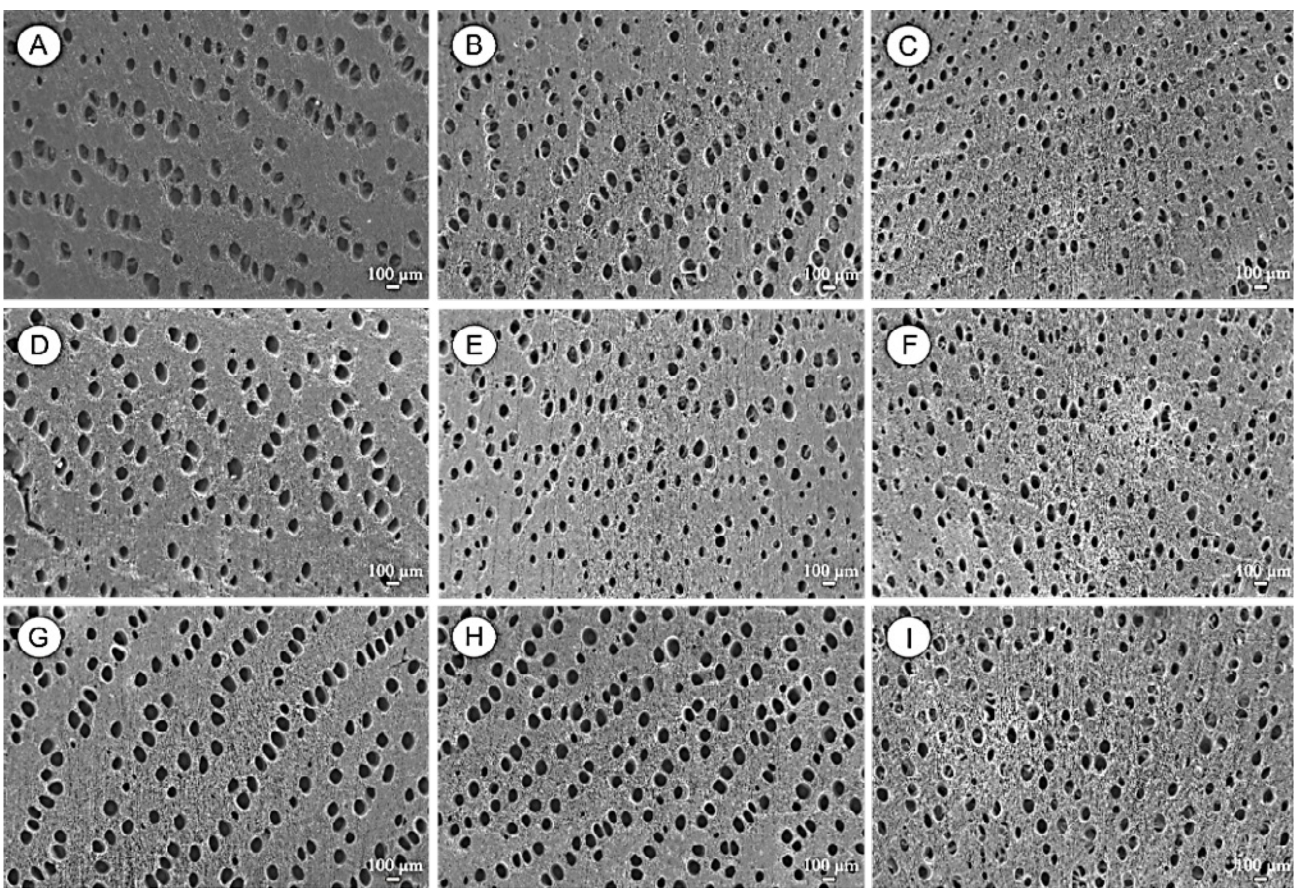

Figure 2. Scanning electron microscope micrography in the three sampled regions.

(a) external, (b) intermediate, and (c) inner tree diameter class of 14.2 cm; (d) external, (e) intermediate, and (f) inner tree diameter class of 11.4 cm; and (g) external, (h) intermediate, and (i) inner tree diameter class of 8.1 cm.

3.2. Fibers dimensions of charcoal

Performing Anova enabled us to identify that the interaction between the factors (tree diameter class × radial position) for cell wall thickness, fiber lumen diameter, and charcoal fiber width was significant. These results indicate that there is a dependence between factors and that we should proceed to the evaluation of one effect within another. In Table 2, mean values for charcoal fiber dimensions in the three different radial positions and within each tree diameter class were described in the analysis.

Tree diameter class did not significantly influence the cell wall thickness of the fiber in the external region of the trunk. But, in the intermediate zone and inner position, statistical differences occurred between tree diameter classes for all radial sampled positions, except for the cell wall thickness of the fiber in the inner position of the disc.

The fiber cell wall thickness had higher values in the intermediate zone, differing statistically only from the inner position, for the tree diameter class of 14.2 cm. For the one of 11.4 cm, the highest value was in the external position, which differed statistically from the other radial sampled regions. For the tree diameter class of 8.1 cm, the highest value was in the intermediate zone, but there was no statistical difference between the radial sampled areas. In general, the increment trend is evidenced by the wall thickness of the fiber considering the pith to bark direction, corroborating the variation commonly found in the wood fibers of the *Eucalyptus spp.*, in which some authors have found an increase in it (Evangelista et al., 2010; Lima et al., 2011; Ramos et al., 2011; Sette et al., 2009; Silva et al., 2007).

Table 2. Mean values and multiple comparison analysis test for fiber cell wall thickness, fiber lumen diameter, and fiber width in the charcoal of *Eucalyptus grandis* W. Hill ex Maiden × *Eucalyptus urophylla* S. T. Blake clones.

Fiber cell wall thickness (µm)			
Tree diameter class (cm)	Sampled region		
	External	Intermediate	Inner
14.2	1.019 ABa	1.190 Aa	0.965 Ba
11.4	1.114 Aa	0.885 Bb	0.894 Ba
8.1	1.168 Aa	1.173 Aa	0.992 Aa
Fiber lumen diameter (µm)			
Tree diameter class (cm)	Sampled region		
	External	Intermediate	Inner
14.2	7.249 Ba	9.628 Aa	8.401 ABb
11.4	6.917 Ba	7.735 Bb	9.333 Aab
8.1	8.165 Ba	9.070 ABab	10.477 Aa
Fiber width (µm)			
Tree diameter class (cm)	Sampled region		
	External	Intermediate	Inner
14.2	9.288 Ba	12.008 Aa	10.332 Bb
11.4	9.146 Ba	9.505 ABb	11.121 Aab
8.1	10.501 Ba	11.415 ABa	12.461 Aa

Means followed by the same lowercase letters in columns or by the same uppercase letters in lines do not differ of 5% probability by Tukey test.

3.3. Carbonization effect over fibers dimensions of Eucaliptus wood and charcoal

In Table 3 it can be observed the mean values of fiber cell wall thickness, fiber lumen diameter and fiber width from wood and charcoal of *Eucalyptus* for both the tree diameter class and the radial sampled regions.

Wood carbonization process significantly reduced a wall thickness of the fiber in all tree diameter classes. Fiber cell wall degradation ranged from 41% to 66%, with a mean value of 47%. Abreu Neto (2015) verified a reduction of 75% in cell wall thickness of the wood fiber of a hybrid *Eucalyptus urophylla* at six years of age for carbonized wood up to 450 °C, which was a higher value than that found in this work. Besides, Cutter et al. (1980) analyzed the cell wall thickness of Southern pine charred at temperatures of 400 °C and 800 °C and they found a reduction of the doubled cell wall thicknesses ranging from 37% to 51%.

Fiber cell wall is structurally constituted by three primary compounds: cellulose, hemicellulose and lignin. When pyrolysis process is led up to the temperature of 450 °C, it may occur total or partial thermal decomposition of the cellulose and hemicellulose (Yang et al., 2007), causing a significant reduction in cell wall thickness of charcoal fiber. In Figure 3, the cross-sections of the wood and the charcoal fibers obtained in inner, intermediate and external regions can be visualized.

Table 3. Mean values and comparison test of fiber cell wall thickness, fiber lumen diameter and fiber width from wood and charcoal of *Eucalyptus grandis* W. Hill ex Maiden × *Eucalyptus urophylla* S. T. Blake clones.

Sampled region	Tree diameter class					
	14.2 cm		11.4 cm		8.1 cm	
	Wood	Charcoal	Wood	Charcoal	Wood	Charcoal
Fiber cell wall thickness (µm)						
External	2.028 a	1.019 b	1.947 a	1.114 b	2.429 a	1.167 b
Intermediate	2.095 a	1.190 b	2.626 a	0.885 b	1.808 a	1.172 b
Inner	1.727 a	0.965 b	1.756 a	0.894 b	1.698 a	0.992 b
Fiber lumen diameter (µm)						
External	5.745 b	7.249 a	9.164 a	6.917 b	9.093 a	8.165 a
Intermediate	9.679 a	9.627 a	6.253 b	7.735 a	10.063 a	9.070 a
Inner	7.997 a	8.401 a	9.769 a	9.335 a	9.578 a	10.477 a
Fiber width (µm)						
External	9.801 a	9.288 a	13.059 a	9.146 b	13.952 a	10.501 b
Intermediate	13.869 a	12.008 b	11.504 a	9.505 b	13.680 a	11.415 b
Inner	11.450 a	10.332 a	13.282 a	11.121 b	13.816 a	12.461 b

Means followed by the same lowercase letters in lines and the tree diameter class do not differ of 5% probability by Tukey test.

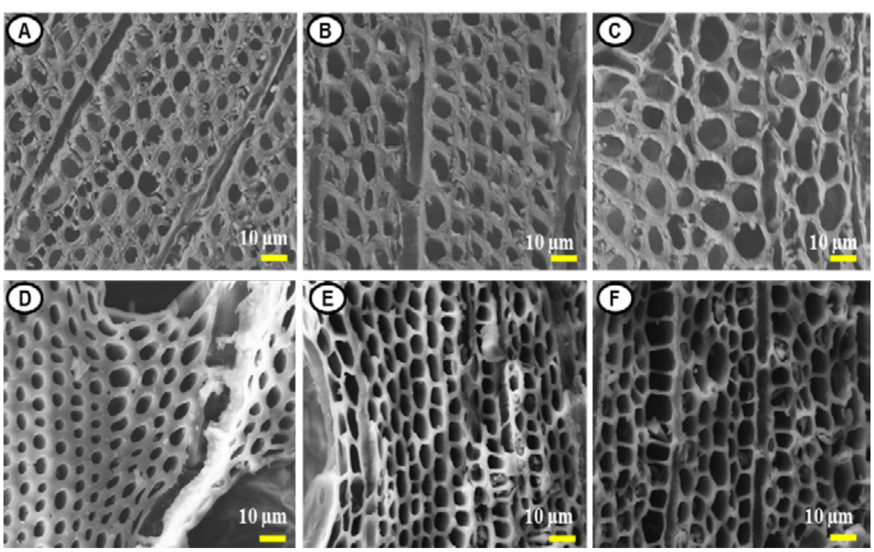

Figure 3. Scanning electron microscope micrography in the three sampled regions of wood and charcoal.

(a) external region of wood; (b) intermediate zone of wood; (c) inner region of wood; (d) external region of charcoal; (e) intermediate zone of charcoal; (f) inner region of charcoal.

In general, when analyzing the diameter of the fiber lumen, it is verified that there was little variation of its values between wood and charcoal fibers, corroborating the results of Abreu Neto (2015), who observed a non-linear trend when he analyzed this feature. Cutter et al. (1980) found a close reduction of 8% in the fiber lumen diameter in Southern Pine wood carbonized at 600 °C. For the external position, there was a significant difference only within the tree diameter classes of 14.2 cm and 11.4 cm, but for the tree diameter class of 14.2 cm, the wood had a lower value than the charcoal, whereas in the tree diameter class of 11.4 cm occurred the opposite. The intermediate zone had a significant difference only in the tree diameter class of 11.4 cm, in which the charcoal had higher value than the wood. In the inner region, there was no significant difference in all tree diameter classes. The increase in the fiber lumen diameter may be associated with a severe thermal degradation of the chemical constituents of the wood, causing the reduction of the cell wall thickness.

The fiber width reduced significantly when carbonized, except for the intermediate zone for the class of 14.2 cm, which did not present statistical difference between wood and charcoal. The reduction of fiber width ranged from 5% to 25%, mainly as an effect of the decrease in cell wall thickness. Abreu Neto (2015) found reductions of 40% of fiber width at the maximum temperature of 450 °C, and he also observed a reduction trend in the fiber width with an increase of pyrolysis temperature. Cutter et al. (1980) found a reduction of 23% in the diameter of the wood trachyte in carbonized samples at 600 °C.

Among the characteristics of the fibers studied to verify the effect of carbonization on the physical structure of the fiber, the cell wall thickness was the one that presented the greatest variations in its dimensions, caused by the thermal degradation of the chemical constituents present in the cell wall of fibers.

4. CONCLUSIONS

Pore diameter was higher in the samples obtained in the inner position of the wood. In the smallest diametric class, there were lower values of pore diameter in external zone.

Within the three tree diameter classes, fiber cell wall thickness increased regarding the pith to bark direction, and also reducing fiber width and fiber lumen diameter.

Wood carbonization process altered the structural elements of the fiber, and the fiber cell wall was the most affected compound, once there was a reduction within the three tree diameter classes and in the three radial sampled regions.

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