



# Effect of thermal treatment on fiber morphology in wood pyrolysis

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Received: 25 April 2020 / Accepted: 21 October 2020 / Published online: 8 November 2020  
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## Abstract

Pyrolysis temperature can alter wood cell anatomical components. However, temperature effects applied to fibers during the pyrolysis process are not very clear. Thus, the aim of this study was to evaluate the influence of thermal treatment on the quality of fiber walls of wood in the pyrolysis process. For this, ten trees of *Eucalyptus urophylla* were cut, five of each hybrid's clones, VM4 and MN463, both 6 years old. Specimens of 0.02 × 0.02 × 0.02 m were prepared for treatment performed at four different temperatures: 100, 250, 350 and 450 °C. Fiber width (FW) and fiber lumen diameter (LD) were measured by scanning electron microscopy, and fiber wall thickness (WT) was calculated as a function of these dimensions. FW decreased approximately 40% with treatment at 450 °C; this trend was verified for both clones analyzed. It was possible to estimate a reduction of 8% in LD every 100 °C of temperature increase. LD of wood was larger than charcoal. LD showed no linear tendency for the thermal treatments analyzed. WT of wood was higher for VM4 clone compared to MN463. The temperature of 100 °C did not imply a large WT change. However, both genetic materials showed tendency to a decrease in the thickness of fiber walls with increasing temperature. The temperature of 350 °C reduced WT by approximately 45% and 64% for VM4 and MN463, respectively. WT of *Eucalyptus urophylla* of charcoal reduced by approximately 76%, compared to original thickness. Wood fiber wall thickness was four times greater than wall thickness of carbonized material at 450 °C.

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## Introduction

Pyrolysis is biomass conversion by heat with controlled oxygen and results in the production of charcoal, gasses, and other subproducts (Sanchez-Silva et al. 2012). During pyrolysis, the temperature can decompose the three main components of wood, hemicellulose, cellulose and lignin, and alter the morphology of cell elements (Giudicianni et al. 2013). Slow pyrolysis is used to increase carbon concentration in solid products in order to increase the calorific value and fixed carbon content (Trugilho and Silva 2001; Jouhara et al. 2018). The properties of wood, such as density, mechanical strength, chemical and anatomical composition, are irreversibly modified by action of temperature, including the fiber as a tubular structure.

Wood fiber structure has been studied, both in natura and carbonized (McGinnes et al. 1971; Schaffer 1973; Xu et al. 2017; Arantes et al. 2020). Studies on tubular mechanical structure modeling have also been conducted to understand the mechanical behavior of materials with isotropic behavior, such as steel (Masikh et al. 2014; Vullo 2014; Juskiewicz and Nowak 2015). This modeling considers, of course, the lamellar structure of the wood fibers with their cellulosic microfibrils arranged helically. According to Bodig and Jayne (1982), the modeling incurs some assumptions, such as considering that the fiber has no punctuation and ignoring that it is tapered at the ends. This implies some misinterpretations, depending on whether the intention is to model the behavior of wood for the flow of liquids in the first case or for structural purposes in the second.

According to Kwasniakova et al. (1996), small changes in temperature can lead to changes in the tensile stress–strain curve of wood. For *Eucalyptus saligna* and *Corymbia citriodora* wood, Menezes et al. (2019) observed that temperatures below 180 °C improved the modulus of elasticity in compression parallel to the fiber and modulus of rupture of wood. However, wood mechanical properties decreased at 180 °C. The authors concluded that high temperature reduced the mechanical properties of both species studied. Schaffer (1973), studying wood submitted to thermal treatments at temperatures close to 160 °C, observed lignin melts and begins to solidify causing variations in mechanical properties. His work reports that properties such as tensile strength, which did not change at temperatures below 170 °C, drastically decreased above this value.

Different wood species can show different thermal degradation behavior. Even in the same species, different anatomical components and compositions can produce distinct charcoal (Gonçalves et al. 2012). Results of changes can also be volumetric shrinkage and unequal mass losses (Kwon et al. 2009) with generation of different products at each temperature due to different thermal resistances (Yang et al. 2007). Between 180 and 280 °C, the initial phase of pyrolysis called torrefaction occurs with reduction in both mass and resistance. Poletto et al. (2012) observed greater mass loss in wood with higher extractives content, while Yang et al. (2007) found different behavior for hemicellulose, cellulose and lignin when exposed to heat.

According to Schaffer (1973), above 200 °C lignin loses mass and solidifies, cellulose softens and depolymerizes. Reactions at this stage are endothermic and

release volatile compounds. Pyrolysis of hemicelluloses occurs between 220 and 315 °C (Yang et al. 2007), with reduction in apparent density and lower volumetric shrinkages (Poubel et al. 2013). Between 240 and 350 °C, the cellulose begins to degrade, losing much of its mass, which almost completes at temperatures close to 450 °C (Yang et al. 2007). Lignin, the most important compound for charcoal formation, begins to degrade around 150 °C, but slowly and not expressively, reducing mass until material reaches temperatures around 300 °C (Yang et al. 2007). Meincken and du Plessis (2013) observed that temperatures above 250 °C negatively affected most of the wood properties, such as cell wall density and decreased the volume of the material. The authors suggested that these changes would affect the macroscopic and mechanical properties of treated wood, such as bending strength or elasticity.

Xu et al. (2017) consider the temperature of 300 °C as key in the transformation of wood into charcoal. The authors observed cellulose, hemicellulose and lignin were no longer identifiable above 325 °C using confocal Raman microscope. The composition of wood cell walls becomes homogeneous, and it was not possible to observe the boundary between wood cellular walls above this temperature. Yang et al. (2007) observed marked degradation of cellulose mainly at temperatures between 315 and 400 °C. Kwon et al. (2009) studied the crystalline structure of cellulose using X-ray diffraction. Above this temperature, they observed a drastic change in the cellulosic structure when layers of the cell wall are no longer visible, being transformed into an amorphous structure.

It is known that slow pyrolysis causes wood volumetric degradation, with damage to the microfibrillar orientation of the original cell wall during the process of transformation into charcoal (McGinnes et al. 1971). Anatomical components are altered with visible decrease in thickness of fiber wall (Cutter et al. 1980; Pereira et al. 2016; Arantes et al. 2020). The wood cell wall is replaced by a smooth amorphous wall structure (McGinnes et al. 1971).

It is also known that the thickness of the fiber wall is directly correlated with the strength and stiffness of wood (Chalk 1983), tensile strength of paper sheets (Pulkkinen et al. 2008) and strength and stiffness of the composite (Bouafif et al. 2009). In several studies, it has been shown that the fiber has an influence on charcoal properties, such as density, parallel compression of fibers, strength modulus, crushing strength modulus at dynamic flexion and gravimetric yield, improvement of energy properties (Esteves and Pereira 2009; Abreu Neto et al. 2018, 2020; Veiga et al. 2018).

However, the effects of temperature applied to fibers during the pyrolysis process are not very clear. To seek subsidies for understanding the mechanical behavior of pyrolyzed wood, the objective of this study was to evaluate the influence of temperature between 100 and 450 °C on the morphology of *Eucalyptus urophylla* wood fiber.

## Materials and methods

### Collection and preparation of material

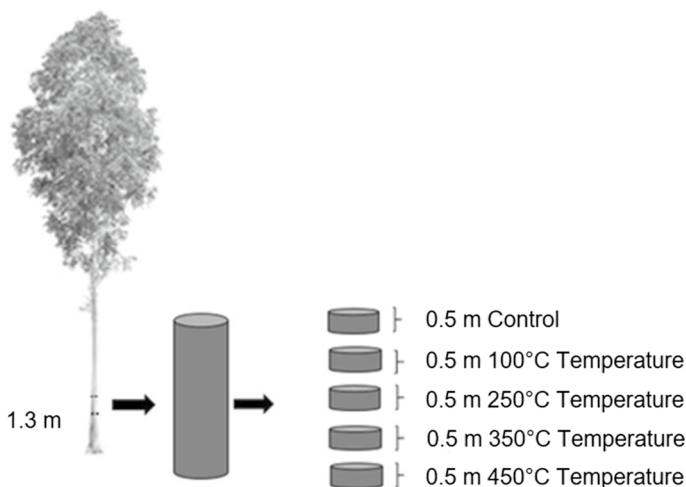
Five trees of VM4 clone and five of MN463, both of *Eucalyptus urophylla* hybrids, were obtained from *Vallourec Florestal Ltda* company, located in the region of Paraopeba, MG, Brazil. The selected trees with a mean diameter breast height of 16 cm, were planted with  $3 \times 2.5$  m spacing and cut at 6 years of age. Logs were cut from each stem and sliced into five 5-cm-thick discs, as shown in Fig. 1.

Discs were air dried in a covered shed, then cut into transverse blocks (from one bark to the other, passing through the pith) and then cut again to reach the dimensions of approximately  $0.02 \times 0.02 \times 0.02$  m. An intermediate specimen (between bark and pith) was used for further analysis.

A part of the wood specimens was separated and did not receive heat treatment, comprising the Control treatment. Control treatment specimens remained at room temperature (*in natura*), i.e., 20 °C, while the others were prepared for thermal treatment.

### Thermal treatment

Four thermal treatments were applied to the wood samples (Table 1). The samples of Treatment 1 were submitted to a temperature of 100 °C, in a drying oven until constant mass. Samples of Treatments 2, 3 and 4 were taken to a muffle furnace, at an initial temperature of 100 °C and heating rate of  $1.67$  °C  $\text{min}^{-1}$ . At this



**Fig. 1** Material collection and sample separation for thermal treatments

**Table 1** Heat treatment parameters analyzed

Treatment	Initial temperature (°C)	Final temperature (°C)	Heating rate (°C min <sup>-1</sup> )	Process total time (h)
1	100	100	–	3.0
2		250	1.67	2.5
3		350		3.5
4		450		4.5

rate of heating, the temperature increases approximately 100 °C per hour. Different final pyrolysis temperatures and total process times were used to increase the variation between treatments, as shown in Table 1.

### Scanning electron microscope (SEM) analysis

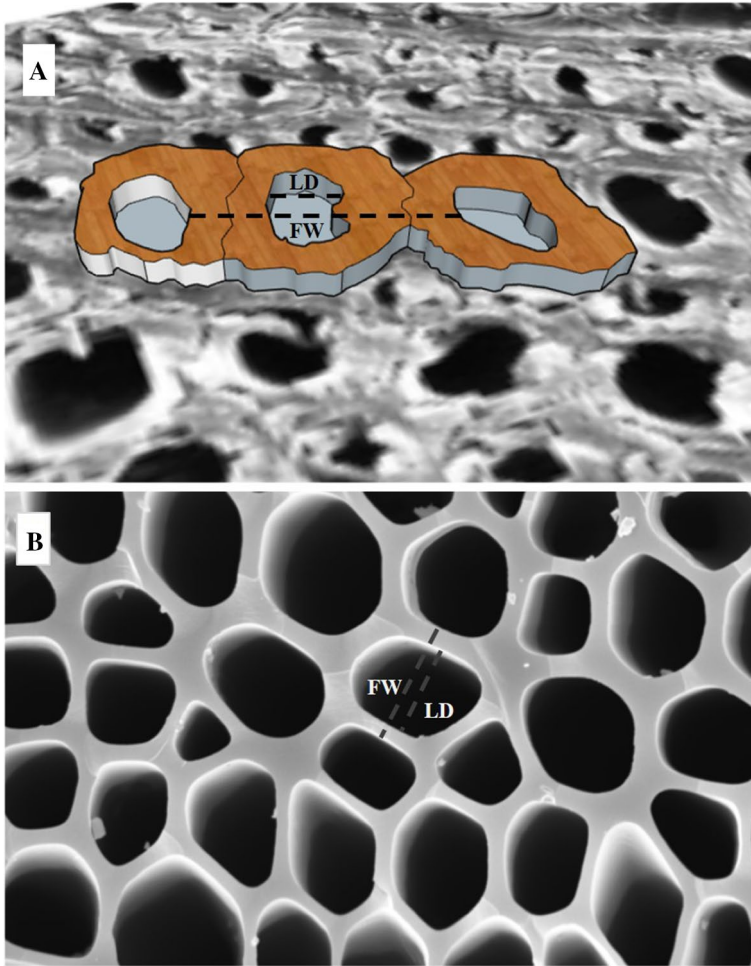
Prior to scanning electron microscope (SEM) analysis, wood and charcoal specimens were cut into 5×5×5 mm and the transversal face was smoothed in a Leica sliding microtome, model Jung SM2000. This equipment, used originally to remove thin pieces of wood for observation on microscope, was used with a methodology adapted to smooth the material surface. In this study, the thin part of wood removed from the microtome was excluded, and the smoothed surface of the block was used for SEM observation.

Charcoal specimens were easily prepared, since its rigid structure and dry surface provided clean cuts with good depth, allowing for better observation of fibers. On the other hand, wood preparation proved to be more difficult. Nevertheless, the thin slice of wood (originally used for observation under the optical microscope) has presented adequate thickness and an almost transparent appearance, which was considered ideal. The wood block that was observed by SEM had a lower quality surface, with a rough and wrinkled appearance, taking several slides to reach an ideal observation depth.

Charcoal samples were oven-dried at 70 °C for 1 h and kept in a container with silica gel; the Control sample was not oven-dried but added to others into the silica container. This procedure was necessary to reduce moisture content for the next phase of preparation. Sputtering-Bal-Tec evaporator was used to cover all sample surfaces with gold, necessary to allow observation under SEM.

SEM was used for measuring the fiber width and lumen diameter of twenty fibers per sample. Fiber wall thickness was calculated as a function of fiber width and fiber diameter. Images were visualized, captured and measured in LEO EVO 40 XVP equipment. Measurements were taken with a magnification of 2000 times.

It was not possible to observe the individual fiber wall boundary of all samples. Charcoal samples show an amorphous structure, without differentiation between the cell wall and the neighbor cells, caused by the large shrinkage of wood in the conversion to charcoal (McGinnes et al. 1971). Thus, for the measurement of wood (Fig. 2a) and charcoal (Fig. 2b), it was necessary to adapt a



**Fig. 2** Measurement of fiber characteristics observed in transversal surface of *Eucalyptus urophylla* wood (a) and charcoal (b) using scanning electron microscope (SEM), with fiber width (FW) and fiber lumen diameter (LD)

measurement method, since the method for evaluating charcoal fibers is not yet standardized. Two points were required (Fig. 2): FW measures fiber width ( $\mu\text{m}$ ) and LD measures fiber lumen diameter ( $\mu\text{m}$ ). The thickness of the fiber wall (WT) was determined using Eq. 1.

$$\text{WT} = \frac{\text{FW} - \text{LD}}{4} \quad (1)$$

WT: fiber wall thickness ( $\mu\text{m}$ ), FW: fiber width ( $\mu\text{m}$ ), LD: fiber lumen diameter ( $\mu\text{m}$ ).

## Statistical analysis

Fiber width, fiber lumen diameter and fiber wall thickness of *Eucalyptus urophylla* clones as a function of temperature: 100, 250, 350 and 450 °C, for MN463 and VM4, were analyzed using analyses of variance (ANOVA) at 5% probability. The influence of temperature on the variables was analyzed by linear regression technique.

## Results and discussion

The dimensional variations in *Eucalyptus urophylla* clone fibers observed by SEM are presented in Fig. 3.

FW and WT results showed statistical significance at 5% probability of analysis of variance (ANOVA). Thus, the influence of temperature on dimension variables was analyzed using the linear regression technique. LD did not present significant values by analysis of variance (Fig. 3).

### Fiber width (FW)

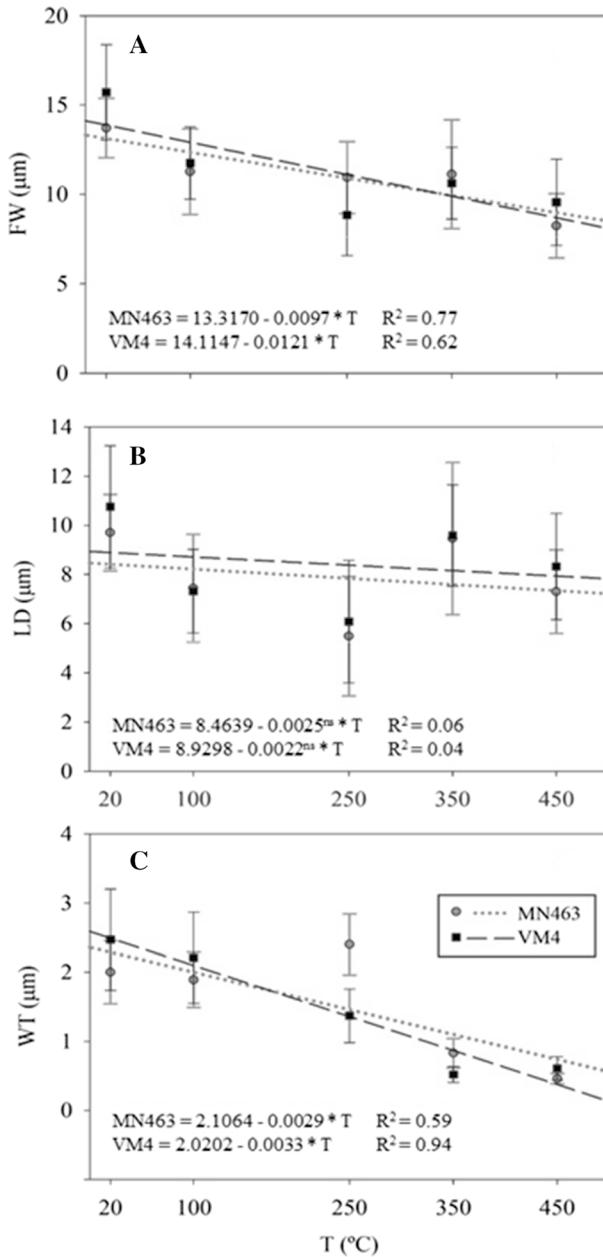
Wood fiber width (FW) decreased with increasing the temperature in both *E. urophylla* clones analyzed (Fig. 3a). Wood (Control treatment) has the highest FW averages, 14 µm for MN463 and 16 µm for VM4. After heat treatment at 450 °C, FW reduced by approximately 39.5% of its original width, from 14 to 8 µm for MN463 clone, and 16 µm to 9 µm for VM4 clone (Fig. 3a).

From the data of width variation, it was possible to observe that the increase in 100 °C caused an 8% decrease in FW of clone MN463 and 9.4% in clone VM4 (Fig. 3a). Coefficient of determination of MN463 was  $R^2=0.77$  and of VM4 was  $R^2=0.62$ . These results indicate the extent to which the model can explain variation of FW as a function of temperature. It can be inferred that the shrinkage in fiber width is approximately 40% for clone MN463 and VM4, compared to Control treatment with charcoal produced at 450 °C.

These fiber width results of wood are similar to those found by Monteiro et al. (2017). Cutter et al. (1980) observed a 23% reduction in tracheid width of Southern pine wood carbonized at 600 °C, a magnitude similar to that found in this study.

### Fiber lumen diameter (LD)

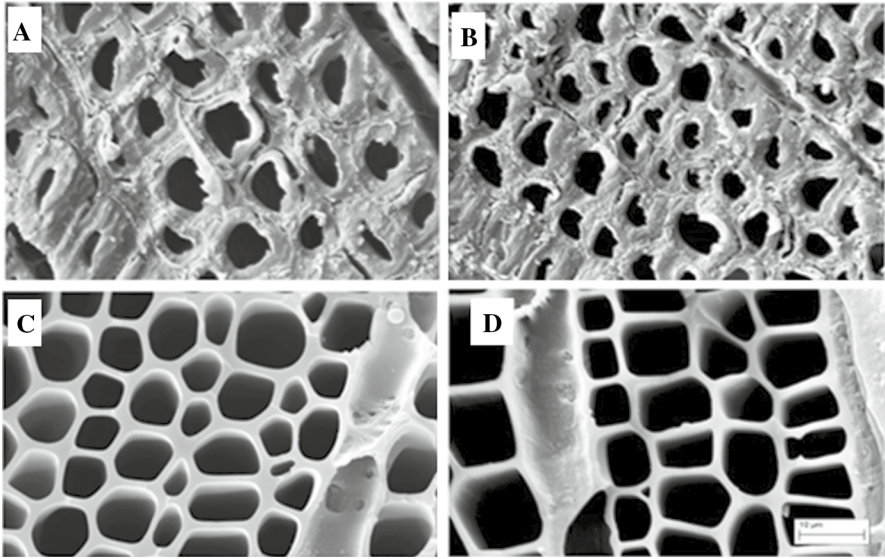
Fiber lumen diameter (LD) of *Eucalyptus urophylla* clones, MN463 and VM4, observed by SEM did not show a linear trend under effect of analyzed temperatures (Fig. 3b). It is possible to observe that the temperature causes changes in the lumen shape (Fig. 4) and deformations in the entire fiber (Fig. 5). However, LD



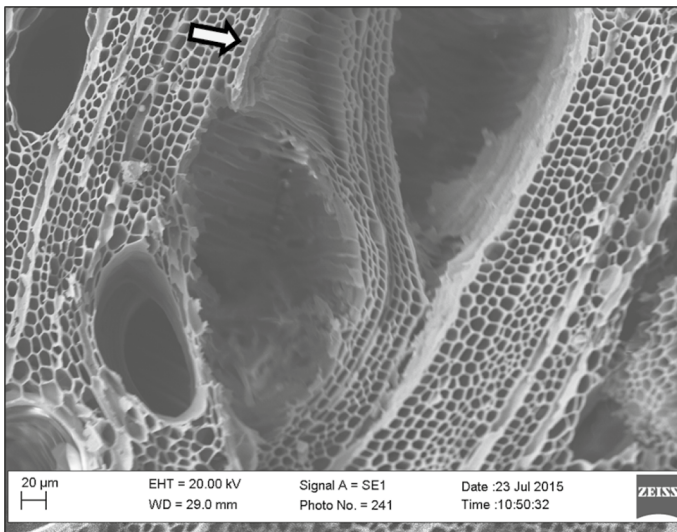
**Fig. 3** Mean trend line of **a** fiber width (FW), **b** fiber lumen diameter (LD) and **c** fiber wall thickness (WT) as a function of temperature ( $T$ ) for MN463 and VM4, *Eucalyptus urophylla* clones

behaves irregularly under the influence of temperature and did not present significant values by analysis of variance (ANOVA) at 5% probability.





**Fig. 4** Scanning electron microscope images of the wood in natura and pyrolyzed of MN463 and VM4 *Eucalyptus urophylla* clones in different thermal treatments. **a** Control, **b** treatment at 100 °C, **c** treatment at 350 °C, **d** treatment at 450 °C



**Fig. 5** Arrow highlights the cell wall detachment due to pyrolysis temperature of VM4 *Eucalyptus urophylla* clone produced at 350 °C observed by SEM

Results found in the literature corroborate those observed in this study. González et al. (2014) found similar values and observed that the structural characteristics of the wood were preserved after carbonization. Evangelista et al. (2010), in a study with *E.*

*urophylla* wood, observed average values of fiber lumen diameters close to those found in this study for Control sample (not thermally treated). Cutter et al. (1980) observed an irregular variation in LD caused by carbonization temperature. The authors found an increase in diameter of tracheids at temperatures close to 300 °C and 350 °C. However, at temperatures close to 600 °C, a reduction in LD of 8% in radial direction was observed.

### Fiber wall thickness (WT)

Heat treatment reduces fiber wall thickness (WT). It is possible to observe a reduction in WT above 250 °C, probably due to the thermal degradation of fiber components; this reduction increases with increasing temperature to 450 °C (Fig. 3c). From data obtained and fitted equation, a temperature of 350 °C caused a reduction in fiber wall thickness of approximately 45% and 64% for MN463 and VM4 clones, respectively (Fig. 3c). It can be estimated using the same model that the reduction of WT of clone MN463 will be greater than 70% with coefficient of determination of 0.94.

Original wood WT was four times greater than WT of carbonized material at 450 °C (Fig. 4). The MN463 clone reduced by 77%, while VM4 clone reduced by 75%. Both genetic materials, VM4 and MN463, showed the tendency of reduced WT with increasing temperature (Fig. 3c). This reduction could indicate degradation of cellulose microfibrils and of cellulose crystalline structure. These components are broken at above 350 °C, making it difficult to observe individual layers of fiber wall (Fig. 4).

Thermal degradation breaks the chemical bonds of its components, causing depolymerization of cellulose and a quick volatilization of chemical components. Retraction of fibers can cause internal stresses in wood at the microscopic level, such as cracks (Fig. 5). These phenomena contribute to the alteration of mechanical properties of original wood, such as a decrease in mechanical resistance of charcoal, in addition to an accelerated loss of mass (Poncsak et al. 2006).

Some results found in the literature corroborate the findings in this study (Monteiro et al. 2017; Chen et al. 2018). Xu et al. (2017) observed a 50% reduction in fiber wall thickness of treated *Quercus* wood at 300 °C; with an increase in temperature to 450 °C, the reduction reaches 62%. The authors considered 300 °C as key temperature, above which material becomes homogeneous, it being no longer possible to distinguish secondary cell wall or cell wall from wood fiber. At this point, carbonized wood began to appear as a form of graphitic carbon, as shown in Fig. 4. According to Cutter et al. (1980), fusion of fiber wall layers depends on the carbonization temperature and heating rate. They verified the disappearance of individual wall layer in tracheids of Southern pine samples heated to 350 °C, which caused a reduction in the thickness of tracheid double walls between 66 and 80%.

### Fiber as a tubular structure

Models of mechanical behavior of tubular structures help to understand wood strength and stiffness, which tend to behave as isotropic material, considering the lamellar structure of wood fibers with their cellulosic microfibrils, helically arranged

and assuming that the fibers are free of punctuation and are not tapered at the ends (Bodig and Jayne 1982).

In the present study, carried out with carbonized wood, it is more important to understand the influence of this tubular structure on the mechanical strength and stiffness, properties generally well correlated (Castro et al. 2016; Andrade et al. 2018; Abreu Neto et al. 2018; Veiga et al. 2018). Comparing mechanical strength of a fiber to longitudinal strength ( $\sigma_L$ ) of a thin-walled cylinder,  $\sigma_L = p (\pi/4) d^2$ , where 'P' is the internal pressure (stress), and 'd' is the inner diameter of the cylinder (Vullo 2014). If cross-sectional area that resists this force  $\times \pi \times d \times t$ , where 't' is wall thickness, the resistive force  $= \sigma \times \pi \times d \times t$ . Thus, it can be inferred that as fiber thickness decreases, resistance also decreases. In the present case, it is possible to calculate the resistance reduction from fiber diameter and thickness values, considering only 'd' and 't' as variables.

The influence of the carbonized fiber thickness on the mechanical behavior of charcoal is still a subject that deserves more careful investigation, since no publications on that subject were identified which support the interpretation of the present results. However, it is possible to assume that charcoal preserves some properties of the wood from which it originated and, in other functions, behaves like a derived material, mainly in terms of its porous tubular structure.

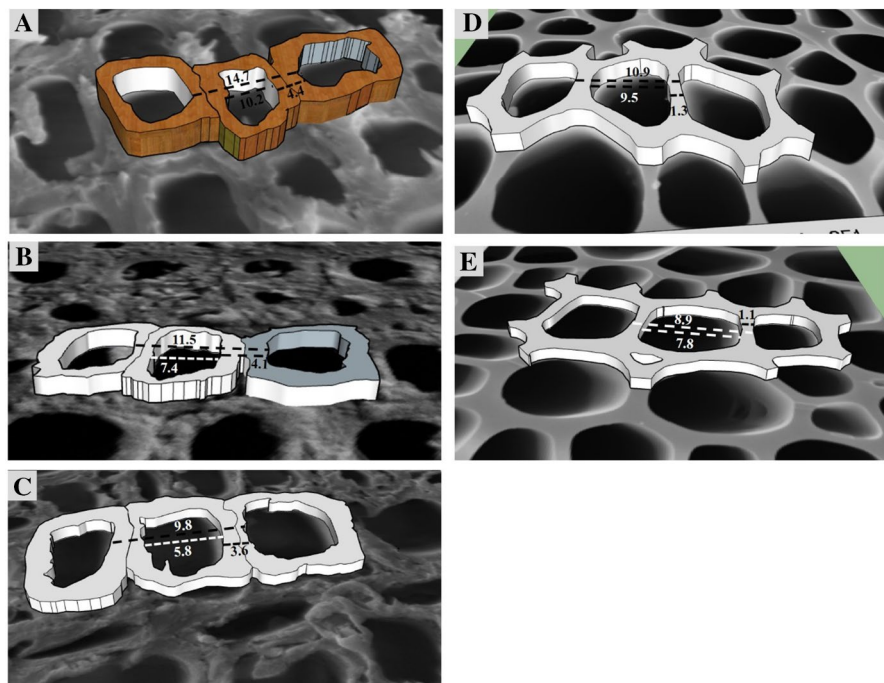
In several studies, it has been shown that the thickness of the fiber wall is directly correlated with the tensile strength of the paper sheet (Pulkkinen et al. 2008), strength and stiffness of wood (Chalk 1983), and strength and stiffness of the composite (Bouafif et al. 2009). However, the relationship between the properties of wood and the properties of charcoal has been the subject of some investigations that show, in general, a positive, albeit moderate, dependence between the two materials for mechanical strength and rigidity (Zhao et al. 2013; Veiga et al. 2018; Abreu Neto et al. 2020).

Fiber wall thickness decreased with increased pyrolysis temperature (Fig. 6). With these results, it can be inferred that the stiffness and compressive strength of charcoal also decrease with the increase in temperature. This deduction is supported by Veiga et al. (2018) and Poncsak et al. (2006). According to Bodig and Jayne (1982), an individual fiber or a segment of the cell wall are important examples of small systems of interest. However, they argue that most often a system of interest is an aggregate of basic elements.

The importance of the results found here consists in evaluation of mass loss caused by cell wall thinning. This thinning corresponds to the reduction in gravimetric yield as the carbonization temperature increases. According to the data presented in Fig. 3, an 8% reduction in volume of charcoal fiber can be estimated at each 100 °C increase in the carbonization temperature.

## Conclusion

Thermal treatment modifies the wood fiber morphology structure. Above 350 °C, it becomes amorphous and homogeneous, and it is no longer possible to observe the individual layers of fiber wall. The temperature of 450 °C reduced the fiber



**Fig. 6** Illustrative scheme of measuring *Eucalyptus urophylla* fiber characteristics with mean values of WF, LD for the Control treatments (a) and at temperatures of 100 °C (b), 250 °C (c), 350 °C (d), 450 °C (e)

wall width by approximately 40%, for both analyzed *Eucalyptus urophylla* hybrids, MN463 and VM4. A decrease of 8% in fiber wall width with an increase of each 100 °C in temperature was estimated. Lumen diameter did not present a linear trend for analyzed thermal treatments.

Fiber wall thickness of wood was higher for VM4 clone compared to MN463. Both genetic materials showed a tendency of reduced thickness of fiber wall with increasing temperature. Temperature of 350 °C reduced fiber wall thickness by approximately 45% and 64% for VM4 and MN463, respectively.

Fiber wall thickness of *Eucalyptus urophylla* charcoal reduced by approximately 76%, compared to original thickness. Wood fiber wall thickness was four times greater than wall thickness of carbonized material at 450 °C.

**Acknowledgements** The authors express their special gratitude, in memoriam, to Dr. Hélder Bolognani Andrade, Research and Development Manager, Vallourec Florestal Ltda, participant of this project, who for many years represented an important partnership in the researches of Wood Science and Technology team at Federal University of Lavras. The authors thank the Wood Science and Technology Graduation Program (DCF/UFLA, Brazil) for all the support for this study.

**Funding** This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior—(CAPES) and partly by Vallourec Florestal Ltda.

**Availability of data and materials** The material and database are available.

## Compliance with ethical standards

**Conflict of interest** We have no conflicts of interest to declare.

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