ANTÔNIO JOSÉ VINHA ZANUNCIO

AVALIAÇÃO DE CLONES DE EUCALIPTO SUJEITOS A DANOS PELOS VENTOS E UTILIZAÇÃO DE SUA MADEIRA NA INDÚSTRIA FLORESTAL

Tese apresentada à Universidade Federal de Viçosa, como parte das exigências do Programa de Pós-Graduação em Ciência Florestal, para obtenção do título de Doctor Scientiae.

VIÇOSA
MINAS GERAIS – BRASIL
2016

Ficha catalográfica preparada pela Biblioteca Central da Universidade Federal de Viçosa - Câmpus Viçosa

T

Zanuncio, Antonio José Vinha, 1987-
Z34a Avaliação de clones de eucalipto sujeitos a danos pelos
2016 ventos e utilização de sua madeira na indústria florestal / Antonio José Vinha Zanuncio. - Viçosa, MG, 2016.
vi, 67f. : il. ; 29 cm.

Orientador : Jorge Luiz Colodette.
Tese (doutorado) - Universidade Federal de Viçosa.
Inclui bibliografia.

1. Eucalipto. 2. Ventos. 3. Madeira - Propriedades mecânicas. 4. Anatomia vegetal. 5. Parede celular vegetal - Ultraestrutura . I. Universidade Federal de Viçosa. Departamento de Engenharia Florestal. Programa de Pós-graduação em Ciência Florestal. II. Título.

CDD 22. ed. 634.94212

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APROVADA: 29 de Janeiro de 2016.

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AGRADECIMENTOS

A Deus, por tudo

Aos meus pais, Teresinha e José, pela ajuda, apoio e por estarem sempre presentes nestes anos.

A minha noiva Amélia, pelo amor, carinho e palavras de incentivo.

As minhas irmãs, Virginia e Andressa, sempre presentes na minha vida.

Aos meus sobrinhos Cecília, Eduardo e Bernardo pelas alegrias que sempre me proporcionam ao vê-los.

Ao professor Jorge Luiz Colodette pela orientação, incentivo, ensinamentos e oportunidade de crescimento pessoal e profissional.

Aos amigos do laboratório de Celulose e papel; Propriedades da Madeira e Painéis e Energia da Madeira e ao Departamento de Engenharia Florestal pela ajuda, conselhos e disponibilidade em ajudar sempre.

À professora Angélica de Cassia Oliveira Carneiro pela disposição, ajuda e incentivo durante a realização do trabalho.

A Celulose Nipo-Brasileira S.A, principalmente ao Fernando Palha Leite e Everton de Souza, por fornecer o material do trabalho e pelo e apoio no desenvolvimento do projeto.

A Universidade Federal de Lavras, na figura do professor Paulo Fernando Trugilho e José Tacísio Lima; Escola Superior de Agricultura "Luiz de Queiroz na figura do professor Mario Tomazello Filho e Universidad del Bío-Bío na figura do professor William Gacitua pela ajuda em diversos testes realizados nesta tese e pelo suporte na interpretação dos resultados.

À Universidade Federal de Viçosa e o Departamento de Engenharia Florestal pela oportunidade.

Ao Conselho Nacional de Desenvolvimento Científico e Tecnológico, CAPES, pela bolsa de doutorado concedida.

OBRIGADA!

SUMÁRIO

RESUMO

ZANUNCIO, Antônio José Vinha, D.Sc., Universidade Federal de Viçosa, janeiro de 2016. **Avaliação de clones de eucalipto sujeitos a danos pelos ventos e utilização de sua madeira na indústria florestal.** Orientador: Jorge Luiz Colodette. Coorientadores: Angélica de Cássia Oliveira Carneiro e Fernando José Borges Gomes.

Danos por ventos são frequentes em plantios florestais do gênero Eucalyptus, entretanto, poucos estudos abordam esta temática. Desse modo, o objetivo desta tese foi avaliar características da madeira que estão associadas a resistência das árvore à ação dos ventos e propor possíveis usos para a madeira danificada pelo vento. No capítulo um, uma nova metodologia para avaliação da resistência das árvores contra a ação dos ventos foi testada, características da madeira foram levantadas e essas características foram relacionadas com os dados históricos de danos por ventos. A nova metodologia apresentou alta relação com os dados históricos de danos por ventos. Clones com maior densidade básica, fração parede das fibras, módulo de elasticidade das fibras e lamela média, dureza das fibras, módulo de ruptura, tensão de crescimento e baixo ângulo microfibrilar apresentaram maior resistência à quebra por ventos. O capítulo dois estudou a utilização da madeira quebrada por ventos para a fabricação de polpa celulósica e papel. A proporção de madeira de dois anos que pode ser utilizada variou com o parâmetro e nível de refino. Entretanto, polpa produzida com 5% de madeira de dois anos e 95% daquela de sete anos apresentou a mesma qualidade que aquela com 100% de madeira desta última idade. O capitulo três avaliou a utilização de árvores tombadas por ventos na fabricação de madeira sólida. Os clones apresentaram boa resistência mecânica, entretanto com grandes variações em suas propriedades físicas. A madeira dos materiais com densidade mais uniforme no sentido radial e baixo coeficiente de anisotropia são mais propício para utilização em estruturas e movelaria. O capitulo quatro avaliou o potencial da madeira quebrada pelo vento para geração de energia. O poder calorífico dos materiais pouco variou, entretanto aqueles com maior densidade básica tiveram maior densidade energética, tornando-os mais propícios para geração de energia por queima direta ou produção de carvão vegetal.

ABSTRACT

ZANUNCIO, Antônio José Vinha, D.Sc., Universidade Federal de Viçosa, January, 2016. **Evaluation of eucalyptus clones subject to wind damage and use of its wood in the forest industry.** Adviser: Jorge Luiz Colodette. Co-advisers: Angélica de Cássia Oliveira Carneiro and Fernando José Borges Gomes.

Wind damage is common in eucalyptus forest plantations; however few studies addressed this issue. Thus, the aim of this thesis was to evaluate the wood characteristics that make trees resistant to wind action, and propose possible uses for timber felled by the winds. In chapter one, a new methodology to evaluate tree resistance against wind action was tested. Besides, the wood characteristics were evaluated and related to historical data of wind damage. The new methodology showed high correlation with historical data from damage by wind. Clones with high basic density, cell wall fraction and modulus of elasticity of the fiber and the middle lamella, hardness of the fibers, modulus of rupture; growth stress and low microfibril angle were more resistant to wind damage. In chapter two, the use of broken trees by wind to manufacture of pulp and paper was studied. The proportion of two-years-old wood which can be used varied with the parameter and refining level, but pulp produced with 95% of seven-year-old wood and 5% of two-years-old wood has similar quality to those with 100% of seven-year-old wood. Chapter three evaluated the use of wind felled trees for solid wood manufacturing. The clones showed good mechanical properties, however, with high variation between their physical properties. Materials with uniform density in the radial direction and low coefficient of anisotropy are better for structures and furniture industry. The chapter four evaluated the potential of wind broken trees for power generation. The calorific value of the materials was similar and those with high density resulted in higher energy density, making them better for power generation, both for direct burning and charcoal production.

1 INTRODUÇÃO GERAL

1.1 O gênero Eucalyptus

O gênero Eucalyptus pertence à família Myrtaceae e inclui aproximadamente 700 espécies, com grande maioria originada da Austrália, onde constitui o gênero dominante da flora, algumas espécies também podem ser encontradas na Nova Guiné e Indonésia (BOSCARDIN, 2009).

A boa adaptação do gênero Eucalyptus ao Brasil aliado aos investimentos em pesquisa e treinamento de mão de obra fizeram das florestas plantadas desde gênero as mais produtivas do mundo, ampliando a sua utilização. Atualmente a madeira deste gênero é utilizada na produção de polpa celulósica e papel, energia, painéis, estruturas, movelaria e outros (LONGUE JUNIOR e COLODETTE, 2013).

A área de florestas plantadas no Brasil abrangia aproximadamente 7,7 milhões de hectares em 2014, sendo que deste total, 5,5 milhões de hectares eram do gênero Eucalyptus, concentrados principalmente nos estados de Minas Gerais, São Paulo, Mato Grosso do Sul, Bahia, Rio Grande do Sul e Espirito Santo (IBA, 2015) (Tabela 1).

Tabela 1 – Área plantada com espécies do gênero Eucalyptus em diferentes estados (IBA, 2015).

Estado	Área ocupada por árvores de eucalipto em 2014 (ha)
Minas Gerais	1.400.232
São Paulo	976.186
Mato Grosso de Sul	803.699
Bahia	630.808
Rio Grande do sul	309.125
Espirito Santo	228.781
Total	5.558.653

Entre as principais espécies cultivadas no Brasil, destacam o Eucalyptus grandis, Eucalyptus urophylla, Eucalyptus camaldulensis e outras (IBA, 2015). Cada espécie possui características diferentes, desse modo, a escolha do material a ser utilizado deve levar em consideração as exigências edáfoclimáticas e o uso final da madeira (BOTREL et al., 2010; TRUGILHO et al., 2015).

1.2 O hibrido Eucalyptus grandis × Eucalyptus urophylla

A utilização de híbridos foi um dos fatores importantes para o sucesso das florestas plantadas no Brasil. A utilização destes materiais é atrativa por possuírem em uma única planta, características presentes em espécies distintas.

O híbrido Eucalyptus grandis × Eucalyptus urophylla é o mais importante para o sucesso das florestas plantadas no Brasil. Ele se destaca na produção de madeira por apresentar rápido crescimento, com ciclo de corte entre seis e sete anos (BASSA et al., 2007; CASTRO et al., 2013). Este híbrido, amplamente utilizado na indústria de polpa celulósica e papel, se mostraram altamente adaptado a esta indústria devido a sua densidade básica, composição química e qualidade das fibras, apresentando altos rendimentos e alta qualidade na polpa produzida (QUEIROZ et al., 2004; BARBOSA et al., 2014).

1.3 Danos pelos ventos

O vento é o deslocamento do ar atmosférico, sendo influenciado por fatores como altitude, latitude, longitude, radiação solar e umidade (MITCHELL, 2012). A direção do vento parte da área de maior pressão atmosférica para áreas de menor pressão e quanto maior a diferença entre as pressões dessas áreas, maior será sua velocidade.

O vento é um fenômeno que causa distúrbios consideráveis em florestas naturais e plantadas, sendo registrados danos desde 1940 (MOORE et al., 2013). Atualmente, os danos pelos ventos em florestas ocorrem no mundo inteiro (MOORE et al., 2013; LAGERGREN et al., 2012; MITCHELL, 2012). Em 1999, o volume de árvores quebradas pelo vento correspondeu a 175 milhões de metros cúbicos na Europa (BOMERSHEIM, 2000).

A passagem dos ventos facilita a circulação de ar na copa, facilitando a transpiração e a troca de oxigênio e dióxido de carbono com a atmosfera (MITCHELL, 2012). Entretanto, segundo mesmo autor, a ação dos ventos causa um esforço de flexão na árvore, resultando na produção de lenho de reação para melhorar a resistência à flexão. Este lenho possui propriedades indesejadas na fabricação de polpa celulósica e no uso como madeira sólida, diminuindo o valor de mercado da madeira.

O relevo altera a velocidade dos ventos e por isso pode maximizar ou minimizar os danos dos ventos nas plantações florestais. Os ventos aumentam de velocidade quando atingem as encostas. Nas regiões de vale, a canalização dos ventos pode aumentar a sua velocidade e com isso causar danos às plantações florestais.

No Brasil, os danos por ventos em plantações de eucalipto são registrados principalmente no oeste e no norte de Minas Gerais, sul da Bahia, Espírito Santo, vale do Paranaíba e oeste de São Paulo (ROSADO, 2006). Nos últimos anos, os danos pela ação dos ventos em plantações florestais na região do Rio Doce têm causado prejuízos econômicos (CENIBRA, 2014) (Figura 1). Atualmente, os danos causados pela ação dos ventos vêm sendo monitorados desde 1998. Com base nesses dados, o ano de 2010 foi marcado pela maior incidência de quebras de árvores pela ação do vento, com mais de 2500 hectares quebrados (CENIBRA, 2014).

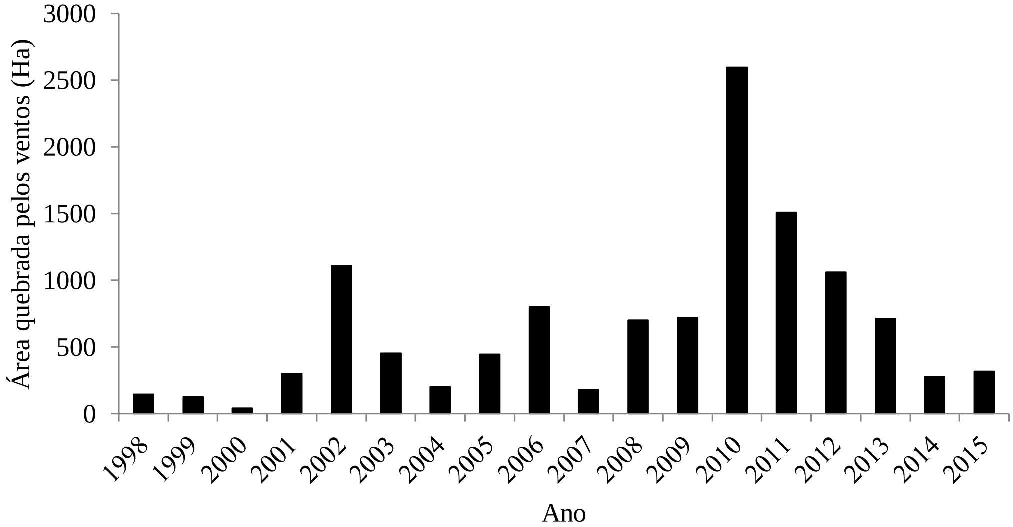

Figura 1- Área em hectares quebrada pelos ventos na região baixa da empresa Cenibra.

Estes danos ocorrem principalmente entre 24 e 36 meses após o plantio, neste período, o estágio de crescimento (relação copa/tronco) deixa as árvores mais vulneráveis aos efeitos maléficos dos ventos (ROSADO, 2006). A ação dos ventos pode quebrar ou entortar a árvore (ATAIDE et al. 2015). No primeiro caso, a quebra pode ocorrer em altura variável, além do prejuízo econômico, este dano pode resultar em aumento das chances de erosão do solo descoberto. No segundo caso, a árvore perde a sua dominância apical, gerando brotações ao longo do fuste com o passar dos anos, estas brotações resultarão em árvores sem aspecto retilíneo.

Os danos pelos ventos afetam negativamente toda a cadeia produtiva das florestas plantadas. Quando ocorre a quebra, a colheita deste material é mais onerosa e demanda maior tempo devido ao menor diâmetro de suas toras, após a retirada desta madeira, um novo plantio precisa ser implantado precocemente, por fim, a madeira com dois anos de idade possui baixo diâmetro e densidade básica (BRAZ et al., 2014), o que dificulta a sua utilização. No caso em que há entortamento da árvore, o povoamento perderá parte da sua produtividade e como as árvores apresentam tortuosidade, a colheita também será mais demorada e onerosa.

A suscetibilidade dos clones contra os danos pelos ventos pode variar conforme o material genético (Braz et al., 2014; Ferreira et al., 2010), justificando estudos sobre a qualidade da madeira e parâmetros dendrométricos das árvores a fim de minimizar os danos por ventos.

1.4 Madeira de eucalipto para produção de polpa celulósica e papel

Atualmente, a madeira é a principal fonte de matéria prima para a produção de polpa celulósica e papel no mundo. Quanto ao tipo da polpa celulósica produzida, se divide em dois grupos, a fibra curta, proveniente das folhosas e a fibra longa, proveniente das coníferas. O primeiro grupo é recomendado para fabricação de papeis de impressão e tissue, enquanto o segundo grupo é recomendado principalmente para produção de papeis para embalagem (BASSA et al., 2007; MOKFIENSKI et al., 2008).

O setor de produção de polpa celulósica e papel atravessa excelente momento no Brasil. Em 2013, foram consumidos aproximadamente 56 milhões de metros cúbicos de madeira para este fim. O que resultou na produção de 16,4 milhões de toneladas de polpa celulósica e 10,4 de papel, transformando o Brasil no quarto maior produtor de polpa celulósica e o nono maior produtor de papel no mundo (IBA, 2015).

A maioria da polpa celulósica produzida no Brasil é exportada para a Europa, Estados Unidos e Ásia, onde é utilizada principalmente como matéria prima para a produção de papéis tissue e para impressão e escrita (IBA, 2015). A maioria do papel exportado pelo Brasil tem como destino os demais países da América do Sul, com aproximadamente 50% do total, a América do Norte, Europa e Ásia também aparecem como destinos do papel produzido no Brasil.

1.5 Madeira de eucalipto para produção energética

No Brasil, visando à geração de energia, a madeira do gênero Eucalyptus é utilizada através da sua queima direta ou através da produção de carvão vegetal (LONGUE JUNIOR e COLODETTE, 2013), sendo interessante que ela possua baixa umidade (ZANUNCIO et al., 2013a; ZANUNCIO et al., 2013b), alto teor de extrativos

(ZANUNCIO et al., 2014), alto teor de lignina e baixa proporção siringil/guaiacil da lignina (PEREIRA et al., 2013).

Em 2014, foram consumidos 50 milhões de metros cúbicos de madeira para produção de energia no Brasil (FAO, 2015). A maioria deste material foi consumida através da queima direta e atende a população de baixo poder aquisitivo, mostrando a importância social deste produto.

O carvão vegetal é utilizado principalmente como termorredutor na produção de ferro-gusa, ferro-ligas e aço. Em 2013, 215 indústrias utilizaram carvão vegetal neste processo, sendo que 80% se encontravam no estado de Minas Gerais (IBA, 2015).

O setor de carvão vegetal enfrenta grandes desafios no Brasil, em 2013, foram produzidas 5,3 milhões de toneladas de carvão vegetal, entretanto, apenas 81% eram provenientes de florestas plantadas, criando uma pressão sobre os remanescentes florestais nativos (IBA, 2015).

1.6 Madeira de eucalipto como produto sólido

A utilização da madeira como produto sólido abrange a madeira roliça, falquejada ou serrada, além da utilização dos painéis (LONGUE JUNIOR e COLODETTE, 2013). Este segmento de utilização da madeira depende do setor de construção civil e movelaria, que consumiu 7,99 milhões e exportou 1,24 milhão de metros cúbicos em 2014 (IBÁ, 2015).

A madeira utilizada para estes fins deve possuir alta resistência mecânica, mostrando alto módulo de ruptura, módulo de elasticidade, resistência à compressão e resistência a tração (MÜLLER et al., 2014). Entre as propriedades físicas, é necessário que a madeira tenha alta estabilidade dimensional (LOPES et al., 2011), representado pela razão entre o inchamento tangencial e radial, chamado de coeficiente de anisotropia.

Sendo assim o presente trabalho teve como objetivo avaliar a relação entre as características da madeira e os danos pelos ventos, além de propor possíveis utilizações para a madeira tombada pelo vento.

Assim, esta tese foi estruturada em quatro artigos, conforme apresentados a seguir:

- **Artigo I**: Characterization of Eucalyptus grandis × Eucalyptus urophylla clones subject to wind damage.

- **Artigo II**: Pulp manufacturing with wood of Eucalyptus grandis × Eucalyptus urophylla trees broken by wind.

- **Artigo III**: Anatomical, ultrastructural, physical and mechanical properties of two-year-old Eucalyptus clones subject to wind damage for solid wood production.

Artigo IV: Chemical and energetic characterization of Eucalyptus grandis × Eucalyptus urophylla clones subject to wind damage.

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2 CAPÍTULO 1: Characterization of Eucalyptus clones subject to wind damage

Abstract

Currently, eucalyptus wood is the main source of raw material for pulp production in South America, but obstacles, such as damage caused by wind actions, can reduce its productivity by breaking or bending trees. This work aimed to test a new methodology to assess the resistance of trees to wind damage and determine the characteristics that increase clone resistance to winds. Tree resistance to breakage, basic density, ultrastructure, anatomy, mechanical properties, growth stress and chemical composition of wood have been evaluated in seven two-years-old Eucalyptus grandis × Eucalyptus urophylla clones, collected from a region with a high incidence of wind damage. The Pearson correlation coefficient between the tree resistance to breakage and the ratio between the area damaged by the winds and the total area planted was -0.8392, showing the efficiency of the methodology adopted. Trees with a high basic density, cell wall fraction, modulus of elasticity of the middle lamella and fibers, fiber hardness, modulus of rupture, growth stress and low microfibril angle and height and width of the rays showed greater resistance to wind damage. Therefore, the selection of clones with these features may reduce the incidence of damage by winds in Eucalyptus plantations.

Key words: basic density; cell wall fraction; cell wall ultrastructure; fiber; wood.

2.1 Introduction

The forestry segment represents 6% of the gross national product in Brazil (IBA, 2014). Eucalyptus wood is the main raw material in this industry and can be used to produce pulp (Gomes et al., 2014; Pirralho et al., 2014), energy (Guerra et al., 2014; Zanuncio et al., 2013), panels (Bal and Bektaş, 2014), and lumber (Ananias et al., 2014). Despite the favorable outlook, environmental factors such as wind damages, can limit or disable the Eucalyptus wood production in the field.

Wind is a phenomenon that causes disturbances in natural and planted forests, with damage recorded since 1940 (Mitchell, 2012) and reports worldwide (Allen et al., 2012; Moore et al., 2013; Lagergren et al., 2012; Kramer et al., 2014). In Brazil, the damage caused by wind in the forest plantations of the Eucalyptus spp. occurs mainly 24 and 36 months after planting, and depending on the material and intensity of the winds, this damage can exceed 20% of the planted area (Cenibra, 2014).

The winds can bend or break trees (Mitchell, 2012). In the first case, bending results in the loss of apical dominance (Panshin and De Zeew, 1980), reducing wood production and quality (Boschetti et al., 2015). In the second case, breaking of trees affects the entire supply chain, as harvesting trees with smaller diameters reduces the efficiency of this operation and increases costs (Spinelli et al., 2009; Hiesl et al., 2015), in addition, a new cultivation needs to be prepared.

Studies on wind damage in forests focus on the effect of weather conditions (Kramer et al., 2014; Moore et al., 2013; Hale et al., 2015), and those of the anatomy, physical, mechanical properties of wood and ultrastructure of the cell wall, are primarily directed toward the using and identification of plants (Donaldson, 2008; McLean et al., 2010; Niklas and Spatz, 2012; Uetimane and Ali, 2011). Thus, the objective of this study was to propose a new methodology for assessing the resistance of trees to breakage, evaluating the properties of Eucalyptus wood, and relating them to tree resistance against wind damage.

2.2 Methodology

2.2.1 Biological material

Five two-year-old Eucalyptus grandis × Eucalyptus urophylla (B, C, D, E, F) and two two-year-old Eucalyptus grandis clones (A, G) from Belo Oriente, Minas Gerais State, Brazil, 42º22'30" “South longitude” and 19º15'00" “West latitude” were selected. This region and Eucalyptus age was chosen because they have a high incidence of wind damage.

Four trees per clone were cut and two discs were removed at 1.3 meters height to evaluate the anatomy, basic density and ultrastructure of the wood and a three-meter log was removed above these discs for characterization of the wood’s mechanical properties. Resistance to breakage and growth stress in trees was conducted in four other trees, per clone.

2.2.2 Tree Resistance Test

A rope was tied at 85% of the total height of the tree to be tested to evaluate the force required to breach in the field. A pulley was attached 12 meters away in a rope between two nearby trees. The rope tied in the tree to be tested passed through this pulley and another pulley was coupled with a dynamometer to measure the force necessary to break the tree. At the end of the rope, a motor was used to pull the tree (Figure 1), according to Braz et al. (2014a; 2014b).

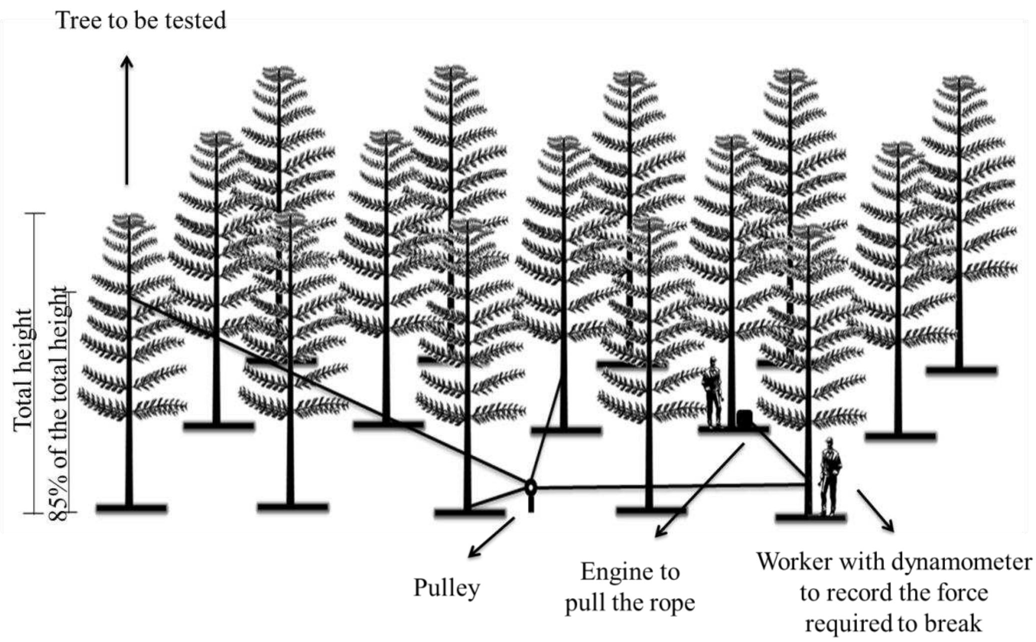

Figure 1: Representation of test for resistance of the trees to breakage.

The data of the area broken by the wind and the total planted area were cataloged for each clone. The force to break the tree and the ratio between the damaged area and the total planted area, per clone, was analyzed by the Pearson correlation coefficient, to assess the quality of the tree resistance test.

2.2.3 Basic density characterization of wood

The basic density of wood was determined from the ratio of the wood dry mass and the wood green volume in one of the 5 cm discs removed from the tree, at a height of 1.3 meters, according to standard NBR 11941 (Associação Brasileira de Normas Técnicas - ABNT, 2003).

2.2.4 Anatomical characterization

A sample was obtained from the intermediate position, from pith to bark, from one of the 5 cm discs, for anatomical characterization. Histological sections were made (Johansen, 1940) and the macerated material was prepared (Franklin, 1945). The microscopic description of the wood was done according to the International Association of Wood Anatomists - IAWA (1989). The fiber cell wall thickness was obtained by the difference between the width of the fiber and the lumen diameter, divided by two. The cell wall fraction was calculated according to the equation 1:

$$C.W.F. = \frac{(2 \times C.W.T)\times 100}{F.W.} \qquad (1)$$

Where, C.W.T. = cell wall thickness (µm); F.W. = fiber width (µm); C.W.F. = Cell wall fraction (%).

2.2.5 Measurement of microfibril angle

The microfibril angle of the S2 layer was determined in the sample used for anatomical characterization. After saturation, the blocks were cut into 10 µm thick sections with a microtome, in the tangential plane, and were macerated with hydrogen peroxide solution and glacial acetic acid in a 2:1 ratio at 55°C for 24 hours. Next, the fibers were washed in distilled water and temporary slides were prepared to measure the microfibril angle.

The measurement of microfibril angle was performed by polarized light microscopy (Leney, 1981), using an Olympus BX 51 microscope adapted with a rotary stage, graduated from 0° to 360°, connected to the image analysis program, Image Pro-plus.

2.2.6 Nanoindentation

A sample was removed from the opposite position to that used for anatomical characterization. A 3 × 3 × 3 mm specimen was made from this sample and embedded in epoxy resin solution to determine the modulus of elasticity and hardness of the S2 layer of the fiber and the middle lamella. The nanoindentation was performed using the TriboIndenter Hysitron TI-900®. The maximum applied load was 100 µN for 60 seconds, with discharge performed in 20 µN/s. The elastic modulus was determined according to the equation 2:

$$MOE = (1 - vm^2) \times \left(\frac{1}{Er} - \frac{1 - vi^2}{Ei}\right)^{-1} \qquad (2)$$

Where: MOE = modulus of elasticity (GPa); According instructions from the device manufacturer, vi = 0.07; vm = 0.35 and Ei = 1140 GPA. The reduced Modulus (Er) was obtained from the load-displacement curve from the initial slope of the unloading wherein the elastic response was generated (Muñoz et al., 2012).

The hardness was determined by the maximum load supported by the specimen divided by the contact area, according to the equation 3:

$$H = \frac{Pmax}{A} \quad (3)$$

Where: Pmax = maximum load of indenter penetration; A = Projected contact areas at maximum load.

2.2.7 Mechanical characterization and growth stresses

A central plank was removed from the three-meter log with a saw blade and samples were made for evaluation of the wood's mechanical properties. The compression parallel to the grain, modulus of elasticity (MOE), and modulus of rupture (MOR) were determined according to ASTM (American Society for Testing and Materials – ASTM- 1997).

The longitudinal displacement was obtained with the CIRAD's sensor. This method consisted in install two nails, separated by 45 mm, in the longitudinal direction of the debarked wood (Dassot et al., 2015). These nails were connected to a sensor, to record the longitudinal displacement, and a hole was made between these two nails. The longitudinal displacement was recorded and the growth stresses calculated according to the equation 4:

$$G.S. = \frac{LD \times MOE}{45} \quad (4)$$

Where: GS = growth stresses; LD = longitudinal displacement (mm); MOE = modulus of elasticity (Mpa).

2.2.8 Wood chemical characterization

One disk from each tree was 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 (American Society for Testing and Materials, 1994); besides the soluble lignin (Gomide and Demuner, 1986); insoluble lignin (Goldschimid, 1971) and lignin's syringyl/guaiacyl (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.

2.2.9 Statistical analysis

The variance homogeneity (Bartlett's test at 5% significance) and normality test were performed (Shapiro-Wilk test at 5% significance). The means of the treatment were compared with the Scott-Knott test at 5% probability. The Pearson correlation coefficient between the wood properties and the ratio between the damaged and the total area planted, per clone, was generated to assess the characteristics that best related to clone resistance against wind damage.

2.3 Results and discussion

The area of wind damage per clone represented 11.7; 1.49; 0.1; 1.17; 5.9; 9.9; 15.1 and 1.82 of the total planted area for clones A to G, respectively. The force required to break the trees varied between 16.2 and 45.6 kgf. Clones B, C, and D had a smaller area affected by winds and a higher force required to break its trees, resulting in a correlation coefficient between these variables of - 0.8392 (Figure 2). This value shows that the high force needed to break results in lower damage and this technique can be used to evaluate the Eucalyptus clones resistance to wind damage.

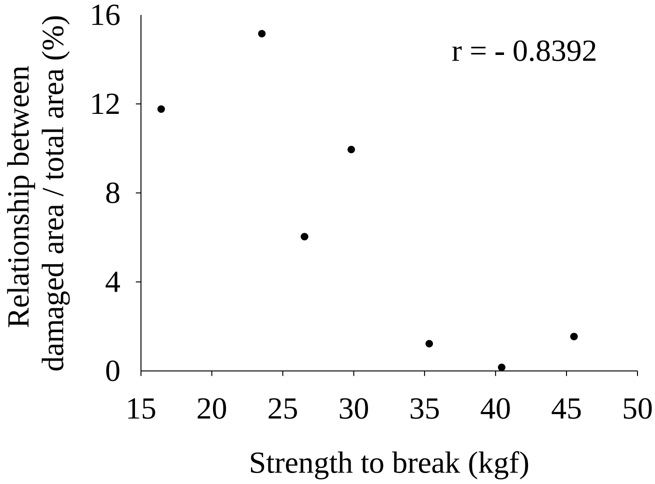

Figure 2: Relationship between the force needed to break the trees and the ratio of damaged area by the winds and total planted area.

The basic density, microfibril angle, and the ratio between these two parameters showed relationship with wind damage (Table 1). A higher basic density resulted in higher wood material per unit volume and greater resistance to breakage (Niklas and Spatz, 2010; Niklas and Spatz, 2012). In addition, a smaller microfibril angle provided a better arrangement of cellulose chains, and therefore, a higher mechanical resistance (Donaldson, 2008; Cortini et al., 2014). These two features can be summarized in just one, as the ratio between the basic density and microfibril angle (McLean et al., 2010; Hein et al., 2013), parameter that showed a better relationship with resistance against winds.

Table 1: Basic density, microfibril angle and the ratio between these parameters in the seven Eucalyptus grandis x Eucalyptus urophylla clones.

Clone	**Basic density (g/cm^3)**	**Microfibril angle (°)**	**Bd/Ma (g/cm^3/°)**
A	$0.372^{(4.0)}$ a	$10.40^{(8.2)}$ b	$0.035^{(8.5)}$ a
B	$0.423^{(3.6)}$ c	$9.16^{(7.2)}$ a	$0.0457^{(9.2)}$ c
C	$0.421^{(4.1)}$ c	$9.21^{(9.8)}$ a	$0.0460^{(8.3)}$ c
D	$0.383^{(3.1)}$ a	$10.11^{(8.5)}$ b	$0.0372^{(9.7)}$ a
E	$0.412^{(3.1)}$ b	$10.45^{(6.2)}$ b	$0.0398^{(10.4)}$ c
F	$0.370^{(3.9)}$ a	$9.56^{(7.5)}$ a	$0.0389^{(9.8)}$ b
G	$0.370^{(3.5)}$ a	$10.88^{(6.6)}$ c	$0.0343^{(9.3)}$ a
***r**	-0.8475	0.7089	-0.869

Bd/Ma = ratio between basic density and microfibril angle. Means followed by same letter vertically by parameter does not differ by the Scott-Knott test at 5% probability. Values in superscript represent the coefficient of variation. r* = Pearson correlation coefficient between the variable and the ratio between wind damaged area and the total area planted.

Trees with high basic density and low microfibril angle showed greater resistance, and therefore, should be recommended for areas with high incidence of wind damage.

The ratio between the basic density and microfibril angle (Den/MFA) has shown the best relationship with the area damaged by the wind (%), but for practical purposes, it is possible to use only the basic density to evaluate the resistance of clones against wind, because its determination is quicker and easier than the microfibril angle.

All the evaluated anatomical parameters varied between the clones (Table 2). Among the fiber classification parameters, the lumen diameter, cell wall thickness, and cell wall fraction had the highest coefficient of variation. In the evaluation of the histological sections, the highest values of the coefficient of variation were found for the height and width of the rays, demonstrating the anatomical constituents with higher variation in wood.

Table 2: Anatomical analysis of seven Eucalyptus grandis x Eucalyptus urophylla clones

Cl.	F.L. (mm)	F.W. (µm)	L.D. (µm)	C.W.T. (µm)	C.W.F. (%)
A	$0.929^{(14.6)}$ c	$20.05^{(12.1)}$ e	$12.06^{(16.4)}$ c	$3.98^{(17.1)}$ c	$39.71^{(17.2)}$ b
B	$0.873^{(14.6)}$ b	$18.02^{(12.4)}$ d	$10.18^{(16.8)}$ b	$3.92^{(17.2)}$ c	$43.65^{(16.6)}$ c
C	$0.971^{(15.2)}$ d	$18.49^{(13.3)}$ d	$9.77^{(17.3)}$ b	$4.36^{(16.8)}$ d	$47.26^{(15.2)}$) d
D	$0.904^{(13.7)}$ b	$16.64^{(14.5)}$ b	$9.17^{(15.9)}$ a	$3.73^{(16.5)}$ b	$45.07^{(15.2)}$ c
E	$0.794^{(12.8)}$ a	$15.67^{(13.4)}$ a	$8.9^{(16.4)}$ a	$3.38^{(17.3)}$ a	$43.24^{(16.2)}$ c
F	$0.831^{(13.9)}$ a	$18.76^{(14.3)}$ d	$11.93^{(16.4)}$ c	$3.41^{(16.4)}$ a	$36.63^{(16.2)}$ a
G	$0.822^{(14.7)}$ a	$17.62^{(13.2)}$ c	$10.47^{(16.6)}$ b	$3.57^{(17.3)}$ b	$40.96^{(16.3)}$ b
*r	-0.4637	0.3001	0.6128	-0.4817	-0.7826

	Vessel diameter (µm)	Freq. (pores/mm^2)	Ray height (µm)	Ray width (µm)
A	$100.1^{(12.4)}$ b	$10.4^{(10.8)}$ b	$217.8^{(18.3)}$ b	$8.48^{(13.5)}$ d
B	$113.23^{(13.5)}$ b	$8.83^{(12.1)}$ a	$226.3^{(16.9)}$ b	$6.28^{(15.4)}$ a
C	$90.0^{(13.6)}$ a	$10.1^{(11.5)}$ b	$239.3^{(18.5)}$ b	$6.84^{(17.9)}$ b
D	$77.5^{(14.2)}$ a	$14.2^{(11.5)}$ d	$199.3^{(17.4)}$ a	$7.39^{(16.3)}$ c
E	$106.6^{(13.2)}$ b	$13.9^{(11.8)}$ d	$207.3^{(17.8)}$ a	$5.80^{(17.5)}$ a
F	$91.64^{(13.1)}$ a	$12.1^{(11.7)}$ c	$226.3^{(17.2)}$ b	$8.53^{(17.2)}$d
G	$109.6^{(14.5)}$ b	$11.1^{(12.5)}$ c	$306.4^{(18.1)}$ c	$7.64^{(16.8)}$ c
*r	0.3768	-0.006	0.5724	0.5869

CL.= Eucalyptus grandis x Eucalyptus urophylla clone; F.L. = Fiber length; F.W.= fiber width; L.D.= Lumen diameter; C.W.T. = Cell wall thickness; C.W.F.= Cell Wall Fraction; Freq.= Vessel Frequency. Means followed by the same letter vertically by parameter does not differ by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation. r* = Pearson correlation coefficient between the variable and the ratio between wind damaged area and the total area planted.

Materials with a lower lumen diameter and higher cell wall thickness corresponded to clones that had smaller damaged area by winds (%). The fibers have structural function in hardwoods and their morphology influenced the mechanical properties of wood (Uetimane and Ali, 2011; Slater and Ennos, 2013). The lumen diameter and cell wall thickness had a different influence on the mechanical properties of wood (Kollmann and Côté, 1968). These two parameters can be related to one variable, the cell wall fraction, an anatomical variable which had the best relationship with the area damaged by winds, having a Pearson's correlation coefficient of - 0.7826.

The vessels conduct water in plants and do not have a structural function. This explains the lack of relationship between this structure and wind damage. The ray cells have thin walls, resulting in low mechanical resistance (Panshin and De Zeew, 1980), and for this reason, they offer little resistance to wind. This was evidenced by the Pearson correlation coefficient between the height and width of the rays, of 0.5724 and 0.5869, respectively, with the area damaged by winds.

Thus, between the anatomical characteristics of the wood, the cell wall fraction showed a better relationship with the resistance of trees against winds and should be considered when selecting clones for areas with high incidence of wind damage.

The fibers showed higher values for modulus of elasticity, while the middle lamella had higher hardness values (Table 3). The higher cell wall fraction and lower microfibril angle resulted in a higher modulus of elasticity of the fiber (Gindl et al., 2004; Borrega and Gibson, 2015), increasing its resistance against winds. A smaller microfibril angle resulted in a better arrangement of these structures (McLean et al., 2010; Hein et al., 2013), increasing the fiber resistance per unit area, and thus, its hardness (Li et al., 2014). The middle lamella connects the adjacent cells (Panshin and De Zeew, 1980), being important in the tree structure, thus the modulus of elasticity of this structure showed a relationship with resistance to wind damage. Finally, there was no relationship between the hardness of the middle lamella and the resistance against winds.

Table 3: Modulus of elasticity (MOE) and Hardness (H) of the fibers and middle lamella obtained by nanoindentation in seven Eucalyptus grandis x E. urophylla clones

Clone	Moe of S2 layer (Gpa)	Hardness of S2 layer (Gpa)	Moe of middle lamella (Gpa)	Hardness of middle lamella (Gpa)
A	$12.6^{(7.1)}$ b	$0.269^{(7.3)}$ a	$7.95^{(6.9)}$ b	$0.298^{(7.0)}$ b
B	$14.1^{(7.6)}$ b	$0.278^{(7.1)}$ b	$8.47^{(7.5)}$ b	$0.302^{(7.3)}$ b
C	$15.5^{(6.2)}$ c	$0.289^{(7.0)}$ c	$9.97^{(7.2)}$ c	$0.335^{(6.9)}$ c
D	$13.2^{(7.1)}$ b	$0.302^{(6.8)}$ c	$9.61^{(6.9)}$ c	$0.322^{(7.2)}$ c
E	$13.6^{(6.3)}$ b	$0.278^{(6.6)}$ b	$7.15^{(6.8)}$ a	$0.274^{(7.2)}$ a
F	$11.0^{(6.8)}$ a	$0.256^{(6.9)}$ a	$6.12^{(7.6)}$ a	$0.301^{(7.3)}$ b
G	$11.6^{(7.0)}$ a	$0.266^{(7.3)}$ a	$7.13^{(7.2)}$ a	$0.299^{(6.8)}$ b
***r**	-0.8148	-0.7861	-0.7559	-0.5161

Means followed by the same letter vertically by parameter does not differ by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation. r* = Pearson correlation coefficient between the variable and the ratio between wind damaged area and the total area planted

The mechanical properties of wood and the growth stress of trees showed a relationship with the area of wind damage (%) (Table 4).

Table 4: Compression parallel to grain (C.P.G.), modulus of rupture (MOR), modulus of elasticity (MOE), Longitudinal displacement (L.D.) and growth stress (G.S.) of the seven clones E. grandis x E. urophylla

Clone	C.P.G. (Mpa)	MOR (Mpa)	MOE (Mpa)	L.D. (mm)	G.S.(Mpa)
A	$32.9^{(10.2)}$ b	$50.70^{(11.3)}$ b	$5423^{(10.2)}$ b	$0.099^{(26.5)}$ a	$11.79^{(26.7)}$ a
B	$34.1^{(10.1)}$ b	$62.76^{(10.5)}$ c	$5643^{(11.3)}$ b	$0.138^{(22.4)}$ b	$17.22^{(23.6)}$ c
C	$40.7^{(9.5)}$ d	$74.63^{(11.2)}$ d	$7038^{(12.4)}$ d	$0.156^{(23.5)}$ c	$24.45^{(22.5)}$ d
D	$32.8^{(10.9)}$ b	$59.33^{(9.5)}$ c	$5482^{(13.1)}$ b	$0.116^{(25.7)}$ a	$14.15^{(27.4)}$ b
E	$36.1^{(11.2)}$ c	$70.22^{(10.4)}$ d	$6235^{(9.3)}$ c	$0.125^{(28.9)}$ b	$17.4^{(28.3)}$ c
F	$35.6^{(10.3)}$ c	$55.31^{(10.7)}$ b	$5994^{(10.5)}$ c	$0.141^{(22.4)}$ b	$18.3^{(24.1)}$ c
G	$33.9^{(10.5)}$ b	$51.78^{(9.4)}$ b	$4527^{(11.2)}$ a	$0.102^{(27.8)}$ a	$10.11^{(22.1)}$ a
***r**	-0.4021	-0.7590	-0.6525	-0.6099	-0.6252

Means followed by the same letter vertically by parameter does not differ by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation. r* = Pearson correlation coefficient between the variable and the ratio between wind damaged area and the total area planted.

The modulus of rupture showed a better relationship with the area damaged by the winds per clone (%), followed by the modulus of elasticity and compression parallel to grain, respectively. The modulus of rupture had a relationship with the cell wall fraction and basic density (Dixon et al., 2015; Longui et al., 2014), parameters that also showed high correlation with the area damaged by the winds per clone (%). This showed how wind resistance was the result of a set of wood characteristics.

The Pearson correlation coefficient between the growth stress and the area damaged by winds (%) was - 0.6252. The growth stresses resulting from the internal forces that keep the trees standing (Archer, 1989; Jullien et al., 2013), being important in areas with wind damage. However, a high growth stress increases the incidence of defects such as cracks and warping, reducing the wood value (Chauhan and Walker, 2011; Solorzano et al., 2012). Thus, plants with high growth stresses are suitable for planting in areas with high wind damage, but this may compromise wood use for sawmill.

The wood chemical characterization is presented in table 5. There was no relationship between the wood chemical composition and area damaged by the winds per clone (%). The wood chemical composition has a structural role in the wood (Fengel and Wegener, 1984). However the variation of other factors, such as anatomy, basic density and microfibril angle minimizes the effect of the wood chemical composition against wind damage.

Table 5: Chemical composition of two-years-old Eucalyptus grandis × Eucalyptus urophylla clones (Cl.)

Cl.	Ext. (%)	Ash (%)	Sol. Lig. (%)	Ins. Lig (%)	Tot. Lig. (%)	S/G	Hol. (%)
A	$2.25^{3.6}$ 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
B	$2.95^{3.1}$ 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
C	$1.23^{3.9}$ 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
D	$2.17^{3.2}$ 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
E	$2.54^{3.7}$ 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
F	$2.71^{2.8}$ 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
G	$2.95^{4.2}$ 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
***r**	0,3137	0,3119	0,1181	-0,2968	-0,2921	0,2980	-0,1547

Cl.	Carbon (%)	Oxygen (%)	Hydrogen (%)	Nitrogen (%)
A	$50.66^{4.0}$ a	$42.80^{3.4}$ a	$5.85^{4.2}$ a	$0.03^{14.1}$ a
B	$50.49^{3.9}$ a	$43.02^{4.1}$ a	$5.76^{4.6}$ a	$0.03^{14.2}$ a
C	$50.90^{4.5}$ a	$42.35^{2.8}$ a	$5.90^{4.2}$ a	$0.05^{14.4}$ a
D	$50.30^{4.6}$ a	$43.28^{3.5}$ a	$5.82^{4.2}$ a	$0.07^{14.2}$ a
E	$50.78^{4.8}$ a	$42.58^{4.6}$ a	$5.88^{5.2}$ a	$0.02^{15.2}$ a
F	$50.43^{3.7}$ a	$42.95^{4.1}$ a	$5.85^{3.6}$ a	$0.03^{14.7}$ a
G	$50.77^{4.6}$ a	$42.63^{3.7}$ a	$5.89^{4.7}$ a	$0.08^{14.8}$ a
***r**	0,1742	-0,1475	0,3634	0,1169

Means followed by the same letter vertically by parameter does not differ by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation. r* = Pearson correlation coefficient between the variable and the ratio between wind damaged area and the total area planted.

The smallest area (%) of the clones B, C, and D damaged by winds is due to the greater cell wall fraction of these materials. This results in the higher basic density and better mechanical properties of fiber (Vincent et al., 2014) and wood (Slater and Ennos, 2013) and, with a smaller microfibril angle, improves the resistance of the wood to breakage and tree resistance against wind. On account of these characteristics, these materials are more suited for areas with a high incidence of wind damage.

2.4 Conclusion

The methodology used is adequate to evaluate the resistance of Eucalyptus clones to winds, with the clones B, C, and D showing lower area damage (%) and a higher force to break its trees. The resistance against wind damage resulted from several factors inherent to the wood quality. Higher basic density, cell wall fraction, modulus of elasticity of the middle lamella and fibers, fiber hardness, modulus of rupture, growth stress and lower microfibril angle and height and width of the rays show eucalyptus trees with better resistance to breakage, and therefore, are suitable for areas with a high incidence of wind damage.

Acknowledgements

To “Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq)”, “Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES)” and “Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG)” for financial support. Celulose Nipo-Brasileira S.A for the support and supply of biological materials. Global Edico Services corrected and proofread the English of this manuscript.

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3 CAPÍTULO 2: Pulp manufacturing with wood of Eucalyptus grandis × Eucalyptus urophylla trees broken by wind

Abstract

Forest plantations may be damaged by winds, especially 24 months after planting and the reduced quality of the wood fibers hinders cellulosic pulp production. The objective was to evaluate the use of these materials for pulp manufacturing in mixture with seven-year-old wood. The pulp of two Eucalyptus grandis × Eucalyptus urophylla clones were produced, bleached, and refined with 100, 95, 85, 75, and 0% of seven-year-old wood, related to cutting age. Wood from two-year-old trees, the age at which most trees are damaged by wind was used to complete each treatment. A 5 cm thick disk was taken from a 1.3-m height for each tree, for anatomical and ultrastructural characterization. The seven-year-old wood showed a lower frequency of vessels and fibers with a higher length, cell wall fraction, modulus of elasticity and hardness, and a lower microfibril angle. The refining of pulps decreased the opacity and specific volume, increased air resistance and improved the mechanical properties. The addition of two-year-old wood in pulp production reduced the mechanical properties and opacity, and increased the air resistance of the paper. The proportion of the two-year-old wood that can be used in pulp production varied with the clone, parameter, and refining level. However, the pulp produced with 5% of two-year-old wood and 95% of seven-year-old wood was similar to that produced with 100% of seven-year-old wood. Therefore, 5% of two-year-old wood can be used for pulp production without loss in quality.

Keywords: Fiber, nanoindentation, opacity, tear index, tensile index

3.1 Introduction

In Brazil, the planted forests of Eucalyptus produce an average of 39 m^3/ha/year (IBA, 2015) in a cutting cycle of seven years. These results are because of climate conditions and investment in research. The wood from these crops are used for multiple purposes, such as, production of panels (Bal and Bektaş, 2014), energy (Rousset et al., 2011; Zanuncio et al., 2013), lumber (Ananias et al., 2014) and cellulose pulp (Muguet et al., 2013; Gomes et al., 2014). This generates jobs and taxes for the Brazilian economy, but the damage by winds may limit the performance of this segment.

Wind damage results from air displacement from a low to a high pressure area (Mitchell, 2012; Moore et al., 2013). In Brazil, these damages are common in Eucalyptus plantations, mainly between 24 and 36 months after planting, reaching up to 20% of the planted area (Cenibra, 2014).

The losses by the winds reach the entire production chain. When the wind bend the trees, they remain in the field to complete its cycle, however the timber will present greater amount of reaction wood, hindering the production of pulp (Boschetti et al., 2015). When the trees are broken by the wind, the wood harvest with a smaller diameter increases the cost of this operation (Hiesl et al., 2015; Spinelli et al., 2009) and a new cultivation must be started early. Trees broken by the wind have poorer quality fibers because of early wood age (Ramírez et al., 2009), hindering its industrial use for pulp and paper manufacturing (Pirralho et al., 2014; Severo et al., 2013). Thus, this wood is considered for lower value use, such as power generation (Guerra et al., 2014).

The use of wood broken by wind in pulp and paper production can reduce the financial loss caused by the wind damage. The objective of this study was to characterize the anatomy and ultrastructure of the wood and assess the pulp quality from two Eucalyptus grandis × Eucalyptus urophylla clones made with mixtures of two and seven-year-old woods.

3.2 Methodology

3.2.1 Biological Material

Two Eucalyptus grandis × Eucalyptus urophylla clones with seven and two-year-old wood were selected. The first age refers to the cutting time and the second to the highest wind damage. Three trees per clone and age were harvested. A disk of 5 cm was removed from a height of 1.3 m, for anatomical and ultrastructural characterization. Finally, one meter logs were taken at 0, 25, 50, 75 and 100% of the commercial height, for pulp production.

3.2.2 Anatomical characterization

A wood sample (1.5 x 1.5 x 1.5 cm) was removed from intermediate position from pith to bark in the disk, at a height of 1.3 m. The slides (Johansen, 1940) and the macerated material (Franklin, 1945) were prepared. The microscopic description of the wood was done according to the International Association of Wood Anatomists (IAWA, 1989). The cell wall thickness and the cell wall fraction of the fibers were calculated with the equations:

$$\mathrm{C.W.T} = \frac{F.W. - L.D.}{2}$$

$$C.W.F. = \frac{(2 \times C.W.T) \times 100}{F.W.}$$

Where, C.W.T. = cell wall thickness (µm); F.W.= fiber width (µm); L.D.= Lumen diameter; C.W.F. = Cell wall fraction (%).

3.2.3 Microfibril angle measurement

The microfibril angle of the S2 layer was determined using the same sample used for anatomical characterization. After saturation, the sample was cut with a microtome in the tangential plane, in 10 µm thick sections. These were macerated with hydrogen peroxide solution and glacial acetic acid in the ratio 2:1 at 55°C for 24 hours. Next, the fibers were washed in distilled water and temporary slides were prepared to measure the microfibril angle.

The measurement of the microfibril angle was performed by polarized light microscopy (Leney, 1981), using an Olympus BX 51 microscope, adapted with a rotary stage, graduated from 0° to 360°, and connected to the image analysis program, Image Pro-plus. The size was increased 200 times and 20 fibers were analyzed per wood sample.

3.2.4 Nanoindentation

A sample (1.5 × 1.5 × 1.5 cm) was removed from the opposite position to that used for anatomical characterization. From this sample, a 3 × 3 × 3 mm specimen was made and embedded in epoxy resin solution to determine the modulus of elasticity and hardness of the S2 layer of the fiber and the middle lamella. The nanoindentation was performed in a TriboIndenter Hysitron TI-900®. The maximum applied load was 100 µN for 60 seconds, with discharge performed in 20 µN/s. The modulus of elasticity was determined according to the equation:

$$MOE = (1 - vm^2) \times \left(\frac{1}{Er} - \frac{1 - vi^2}{Ei}\right)^{-1}$$

Where: MOE = modulus of elasticity (GPa); according to device manufacturer instructions, vi = 0.07; vm = 0.35, and Ei = 1140 GPA. The reduced modulus (Er) was obtained from the load-displacement curve, from the initial slope of unloading, wherein, the elastic response was generated (Muñoz et al., 2012).

The hardness was determined by the maximum load supported by the specimen divided by the contact area, according to the equation:

$$H = \frac{Pmax}{A}$$

Where: H= hardness (GPa); Pmax = maximum load of nanoindenter penetration; and A = Projected contact areas at maximum load.

3.2.5 Manufacturing and characterization of the pulp

Wood pulping was done to obtain pulp with a kappa number 18 ± 0.5. The pulping was carried out using 600g of dry wood, 25.3% sulfidity, liquor-to-wood ratio of 4: 1, cooking temperature of 170 ° C and residence time of 60 minutes. The effective alkali and yield are shown in Table 1.

Table 1: Effective Alkali, total yield, screened yield and reject yield of pulping

Treatments	Clone A				
	T1	T2	T3	T4	T5
Effective alkali (%)	17.0	16.7	16.7	16.6	16.0
Screened yield (%)	50.4	50.2	50.3	50.0	49.6
Reject yield (%)	0.03	0.02	0.02	0.03	0.03
Total yield (%)	50.4	50.2	50.3	50.0	49.6
Treatments	Clone B				
	T1	T2	T3	T4	T5
Effective alkali (%)	15.7	15.4	15.4	15.3	14.7
Screened yield (%)	50.7	50.3	50.1	50.5	49.8
Reject yield (%)	0.02	0.03	0.04	0.03	0.03
Total yield (%)	50.4	50.2	50.3	50.0	49.6

Pulps were made with 100% seven years wood (T1); pulp made from 95% (T2); 85% (T3) and 75% (T4) seven years old wood and 100% two years wood (T5).

The bleaching was done to obtain pulp with brightness 90% ISO ± 1. The pulps were bleached by sequence OD(EP)D. The oxygen delignification (O stage) was run at 10% consistency, 100 °C, 60 min, 700 kPa pressure 20 kg NaOH/odt pulp, and 20 kg O_2/odt pulp. The first chlorine dioxide stage (D) was carried out at 90°C for 120 minutes, 10% consistency, end pH between 2.5 to 3.0 and kappa factor of 0.23. The hydrogen peroxide stage was carried out at 80°C for 120°C and at 10% consistency. The second dioxide stages (D) was carried out 80°C for 120°C, at 10% consistency and end pH between 4.5 to 5.0.

Samples were refined to 0, 500, 1500, and 3000 revolutions in the PFI mill. The pulping was carried out with seven-year-old wood in proportion of 100, 95, 85, 75, and 0%, the supplement of each treatment utilized two-year-old wood.

The paper produced with pulp from different mixtures of two and seven-year-old wood was analyzed according to the "Technical Association of Pulp and Paper Industry- TAPPI" (Table 2).

Table 2. Physical, mechanical and optical characterization of sheets produced.

Test	**Standard**
Refining in PFI mill	TAPPI T248 sp-08
Paper sheets for tests	TAPPI T205 sp-06
Schopper Riegler degree - ºSR	TAPPI T423 cm-07
Brightness	TAPPI 452 om-08
Opacity	TAPPI 1214 sp-07
Resistance to air passage	TAPPI T460 om-02
Tear Resistance	TAPPI T414 om-04
Tensile index and stretch	TAPPI T494 om-06
Stretch	TAPPI T494 om-06

3.2.6 Statistical analysis

The variance homogeneity (Bartlett's test at 5% significance) and normality were performed (Shapiro-Wilk test at 5% significance). The means obtained in the anatomical characterization were analyzed by t-test at 5% probability and those obtained in the pulp characterization were subjected to Scott-Knott at 5% probability.

3.3 Results and discussion

The wood anatomical composition and mechanical properties of the S2 cell wall layer and middle lamella varied between clones and ages of the same clone (Table 3).

Table 3. Anatomical and ultrastructural characterization of Eucalyptus grandis x Eucalyptus urophylla clones with two and seven years old

Cl.	Age	F.L. (mm)	F.W. (µm)	L.D. (µm)	C.W.T. (µm)	C.W.F. (%)
A	2	$0.822^{14.7}$ a	$17.62^{13.2}$a	$10.47^{16.6}$ b	$3.57^{17.3}$ a	$40.96^{16.3}$ a
	7	$1.01^{17.5}$ b	$16.97^{13.2}$a	$7.45^{16.1}$a	$4.73^{17.5}$b	$55.83^{15.5}$ b
B	2	$0.831^{13.9}$ a	$18.76^{14.3}$a	$11.93^{16.4}$ b	$3.41^{16.4}$ a	$36.63^{16.2}$ a
	7	$1.03^{13.8}$b	$18.41^{12.3}$a	$9.63^{17.4}$a	$4.18^{15.9}$ b	$46.25^{11.4}$ b
Cl.	**Age**	**Diam. (µm)**	**Freq. (pores/mm^2)**	**Alt. (µm)**	**Larg. (µm)**	**Microfibril angle (°)**
A	2	$109.6^{14.5}$ a	$11.1^{12.5}$ a	$306.4^{18.1}$a	$7.64^{16.8}$ a	$10.88^{6.6}$ b
	7	$112.5^{15.1}$ a	$9.5^{12.1}$ b	$315.6^{17.5}$ a	$7.12^{17.5}$ a	$9.35^{6.7}$ a
B	2	$91.64^{13.1}$ a	$12.1^{11.7}$ a	$226.3^{17.2}$ a	$8.53^{17.2}$ a	$9.56^{7.5}$ b
	7	$110.6^{13.6}$b	$9.5^{11.4}$ b	$235.5^{17.4}$a	$8.12^{16.5}$ a	$9.02^{6.2}$ a
Cl.	**Age**	**Moe of S2 layer (Gpa)**	**Hardness of S2 layer (Gpa)**		**Moe of m.l. (Gpa)**	**Hard. of m.l. (Gpa)**
A	2	$11.0^{6.9}$ a	$0.256^{6.9}$ a		$6.12^{7.7}$ a	$0.301^{7.3}$ a
	7	$16.5^{7.9}$ b	$0.310^{7.1}$ b		$6.28^{7.3}$ a	$0.306^{7.6}$ a
B	2	$11.6^{7.0}$ a	$0.266^{7.3}$ a		$7.13^{7.2}$ a	$0.299^{6.9}$ a
	7	$15.9^{7.2}$ b	$0.305^{6.5}$ b		$7.22^{7.1}$ a	$0.313^{7.9}$ a

CL. = Eucalyptus grandis × Eucalyptus urophylla clone; F.L.=Fiber length; F.W.=Fiber width; L.D.=Lumen diameter; C.W.T.= Cell wall thickness; C.F.R.=Cell Wall Fraction; Ves. Diam.= Vesssel diameter; Freq.=Vessel Frequency; Moe of m.l.= Moe of middle lamella; Hard. of m.l.= Hardness of middle lamella. Means followed by the same letter vertically per parameter does not differ by the t-test at 5% probability. Values in superscript represent the coefficient of variation.

Two-year-old wood fibers had a higher microfibril angle and smaller length and cell wall fraction in both clones. This can be explained by the disorganized production of cells during the beginning of the cambial activity, resulting in weaker fibers. The organization of the cambial activity results in fibers with a smaller microfibril angle (Donaldson et al., 2018; Lima et al., 2014) and greater length and cell wall fraction, as the age increases (Panshin and Zeeuw, 1980). This agrees with a greater cell wall fraction with increasing age of Eucalyptus grandis × Eucalyptus urophylla and Eucalyptus globulus (Quilho et al., 2006; Ramírez et al., 2009) and reduction of microfibril angle in Eucalyptus grandis (Lima et al., 2014).

The pore frequency decreased in 14.41 and 21.48% with increasing age in the A and B clones, respectively. The highest growth rate and concentration of auxin in the trees during the first years changed the cambial activity and increased the pore frequency (Panshin and Zeeuw, 1980; Nugroho et al., 2012; Leal et al, 2003). The pores are important during pulping (Pirralho et al., 2014), because they facilitated the penetration of reagents into the wood. Finally, the height and width of the rays were similar in the two and seven-year-old wood of both clones.

A lower microfibril angle results in better arrangement of these structures, increasing the mechanical resistance per unit area, and thus, the hardness (Li et al., 2014). And with a higher cell wall fraction, increase modulus of elasticity of the fiber S2 layer (Gindl et al., 2004; Muñoz et al., 2012). The fibers are the principal constituents of pulp and paper and therefore, they have to present good mechanical properties (Pirralho et al., 2014). Finally, increasing age did not affect the mechanical properties of the middle lamella.

Clone B had a higher gain in the Schopper Riegler (°SR) degree during refining, and the same was found per clone with an increase in the proportion of wood with two years old (Figure 1). This showed that smaller fibers with lower cell wall fractions have a better fit between them, reducing the empty spaces and increasing resistance to the water passage, and thus, increasing the Schopper Riegler degree (Severo et al., 2013), as reported for Corymbia citriodora, Pinus contorta and Pinus sylvestris (Sable et al., 2012; Severo et al., 2013).

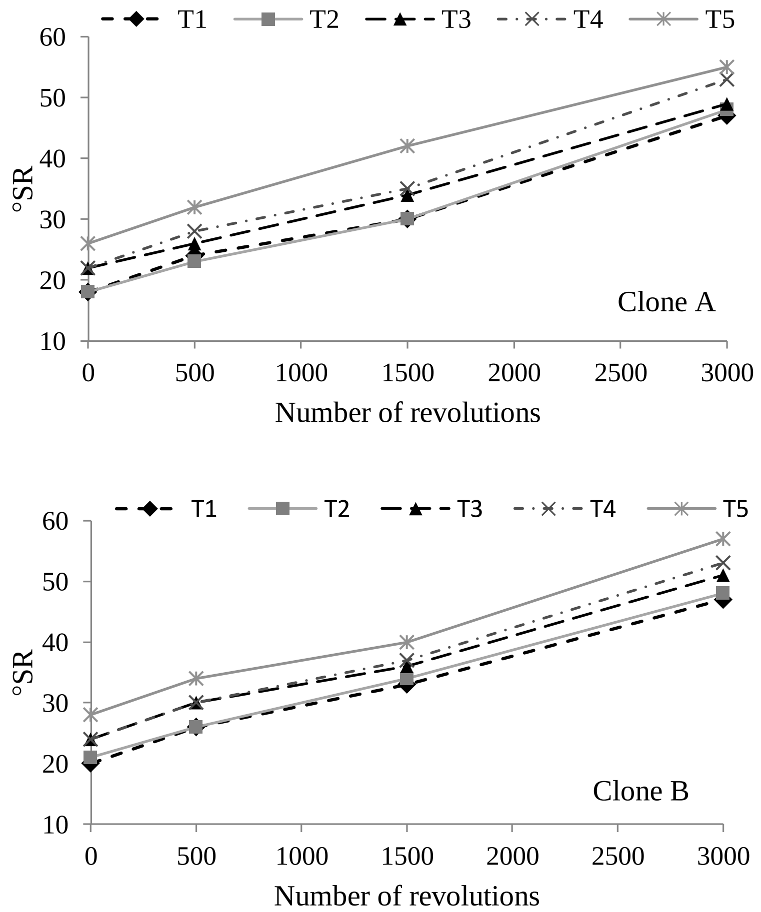

Figure 1: Schopper Riegler (°SR) degree with different number of revolutions during refining in pulps made from wood with two and seven years old; Pulps were made with 100% of seven years old wood (T1); pulp made from 95% (T2); 85% (T3) and 75% (T4) of seven years old wood and 100% of two years old wood (T5).

The specific volume decreased with refining and utilization of two-year-old wood (Table 4). The lower cell-wall fraction of wood fibers with two-year-old facilitated the arrangement between them, and thereby, reduced the specific volume. This also explained the reduction in the specific volume with intensity refining, because the collapse of cells through this technique improved the arrangement of the fibers and reduced this parameter (Biermann, 1996), especially in the initial refining levels. The addition of 25% of two-year-old wood in the manufacture of cellulose pulp did not reduce the specific volume of the pulp compared with that produced with 100% seven-year-old wood. However, treatment with 100% two-year-old wood showed lower values for this parameter.

The refining and utilization of two-year-old wood for pulp production increased the air resistance (Table 4). Refining induced a collapse of the fibers, improving their arrangement in the paper sheet by reducing the empty spaces and increasing air resistance. Wood fibers with two-year-old have a lower cell wall fraction and the worst mechanical properties, and therefore, are more fragile (Gindl et al., 2004; Muñoz et al., 2012), resulting in large amount of fines that fill the voids of the paper and hinder the air passage (Santos and Sansígolo, 2007; Pirralho et al., 2014). However, the addition of 5% of two-year-old wood in the paper production showed similar values to those of pulps produced with 100% seven-year-old wood.

Table 4: Specific volume and air resistance with different levels of revolution during the refining of wood pulps made with two and seven years old of Eucalyptus grandis × E. urophylla wood

Clone	Treatment	Specific volume (cm^3/g)			
		Revolutions during the refining			
		0	500	1500	3000
A	T1	2.15$^{4.2}$Aa	1.64$^{3.8}$ Ba	1.35$^{3.6}$ Ca	1.16$^{2.4}$ Da
	T2	2.12$^{4.1}$ Aa	1.65$^{4.1}$ Ba	1.37$^{2.6}$ Ca	1.19$^{2.3}$ Da
	T3	2.12$^{3.9}$ Aa	1.69$^{3.9}$ Ba	1.32$^{2.5}$ Ca	1.18$^{2.1}$ Da
	T4	2.14$^{4.0}$ Aa	1.68$^{3.9}$ Ba	1.29$^{3.1}$ Ca	1.16$^{2.7}$ Da
	T5	1.96$^{3.9}$ Ab	1.49$^{4.1}$ Bb	1.20$^{3.3}$ Cb	1.10$^{2.2}$ Db
B	T1	2.12$^{4.1}$ Aa	1.58$^{4.1}$ Ba	1.30$^{2.9}$ Ca	1.15$^{2.3}$ Da
	T2	2.10$^{4.5}$ Aa	1.54$^{3.9}$ Ba	1.36$^{2.8}$ Ca	1.13$^{2.3}$ Da
	T3	2.13$^{3.6}$ Aa	1.57$^{3.6}$ Ba	1.35$^{2.8}$ Ca	1.15$^{2.2}$ Da
	T4	2.08$^{4.2}$ Aa	1.58$^{4.1}$ Ba	1.32$^{2.6}$ Ca	1.14$^{2.5}$ Da
	T5	1.94$^{4.0}$ Ab	1.42$^{3.8}$ Bb	1.22$^{2.7}$ Cb	1.14$^{2.6}$ Da
Clone	Treatment	Air resistance ($s/100\ cm^3$)			
		Revolutions during the refining			
		0	500	1500	3000
A	T1	0.95$^{10.3}$ Aa	2.26$^{11.5}$ Ba	6.76$^{12.6}$ Ca	46.9$^{9.8}$ Da
	T2	1.13$^{11.2}$ Aa	2.33$^{11.2}$ Ba	6.55$^{12.4}$ Ca	48.9$^{10.0}$ Da
	T3	3.26$^{12.4}$ Ab	11.9$^{11.8}$ Bb	26.9$^{11.1}$ Cb	59.3$^{9.6}$ Db
	T4	4.11$^{11.6}$ Ac	13.9$^{11.3}$ Bb	42.9$^{10.8}$ Cc	93.6$^{10.6}$ Dc
	T5	7.6$^{10.8}$Ad	18.7$^{12.5}$ Bc	60.6$^{11.6}$ Cd	186.9$^{10.2}$ Dd
B	T1	1.13$^{12.6}$Aa	3.45$^{13.5}$ Ba	8.56$^{13.4}$ Ca	53.6$^{11.5}$ Da
	T2	1.29$^{12.9}$Aa	3.59$^{14.5}$ Ba	8.88$^{11.1}$ Ca	54.2$^{10.5}$ Da
	T3	4.69$^{12.4}$ Ab	14.5$^{12.1}$ Bb	36.8$^{10.2}$ Cb	65.9$^{10.3}$ Db
	T4	5.45$^{12.5}$ Ab	18.9$^{11.2}$ Bc	56.1$^{10.7}$ Cc	100.9$^{11.5}$ Dc
	T5	8.13$^{12.1}$ Ac	26.8$^{12.4}$ Bd	89.7$^{11.3}$ Cd	225.6$^{10.6}$ Dd

Pulps were made with 100% seven years wood (T1); pulp made from 95% (T2); 85% (T3) and 75% (T4) seven years old wood and 100% two years wood (T5). Means followed by the same capital letter per line and lower case letter per column did not differ between them by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation.

The tear index decreased with the use of two-year-old wood and increased with the refining process (Table 5). Refining increases the contact surface and intensifies the connections between the fibers, but it damages the fiber and reduces its resistance (Aracri and Vidal, 2012; Biermann, 1996). Gains in tear index were higher up to 1500 revolutions because of the high number of connections between fibers with low damage in their structure, but with 3000 revolutions, such damages were more intense and reduced the gain in tear index. The lower gain in the tear index with 3000 revolutions of refining was found with 100% two-year-old wood because of the presence of fibers with poorer mechanical properties (Muñoz et al., 2012), which broke during the paper production process, explaining the reduced tear resistance.

The addition of two-year-old wood resulted in paper more fragile to tear. However, proportions up to 5% of this wood did not decrease the tear index, suggesting that this wood proportion of wind felled trees can be used in pulp production.

The addition of two years old wood in the pulp production decreased the tensile index while refining increased its values (Table 5). The tensile index depends on the number of inter-fiber connections (Sixta, 2006; Gorski et al., 2012). The highest average length and the lowest production of fines in cellulosic pulp from seven-year-old wood guaranteed greater connectivity between the fibers and a higher tensile index (Fu et al., 2015). Refining also increases the inter-fiber bonds (Biermann, 1996), resulting in a higher tensile index.

Pulp without and with 500 revolutions of refining allowed the addition of 15% of two-year-old wood in its production without affecting the tensile index compared to pulp produced from seven-year-old wood. However, in more severe refining conditions, only pulp produced with up to 5% two-year-old wood showed a similar tensile index to that produced with 100% seven-year-old wood.

The stretch (%) of the paper followed a similar trend in relationship to the tensile index, decreasing with the addition of two-year-old wood and increasing with refining (Table 5). The stretch depends on fibers length and low fine production, allowing a greater number of connections between the fibers and a greater stretch of the paper (Sable et al., 2012; Severo et al., 2012). The increase in inter-fiber connections, propitiated by refining, besides the quality of the fibers, increased the stretch in both clones evaluated, with higher gain up to 1500 revolutions. The addition of up to 15% two-year-old wood, age with higher wind damage, did not reduce the pulp stretch.

Table 5: Tear Index (mN.m² / g), tensile index (Nm / g) and stretch (%) at different levels of revolution during the refining in pulps made with two and seven years old wood of Eucalyptus grandis × E. urophylla

Clone	Treatment	Tear index (mN.m² / g)			
		Revolutions during the refining			
		0	500	1500	3000
A	T1	5.01 $^{8.9}$Aa	6.64$^{7.2}$ Ba	10.35 $^{4.6}$Ca	12.56 $^{4.1}$ Da
	T2	5.15 $^{9.4}$Aa	6.53 $^{6.9}$ Ba	10.09 $^{4.9}$ Ca	12.35 $^{4.2}$ Da
	T3	4.63 $^{10.1}$Ab	5.95 $^{6.7}$ Ba	9.78 $^{4.9}$Cb	11.15 $^{4.3}$ Db
	T4	4.31 $^{9.1}$Ab	5.35 $^{6.6}$ Bb	9.36 $^{4.8}$Cb	10.63 $^{4.0}$ Db
	T5	3.42 $^{9.4}$ Ac	4.85 $^{7.1}$ Bb	7.75 $^{4.7}$Cc	8.12 $^{4.4}$ Dc
B	T1	4.56 $^{8.4}$ Aa	5.95 $^{6.8}$ Ba	9.85$^{4.8}$Ca	11.51 $^{3.9}$ Da
	T2	4.43 $^{7.9}$ Aa	5.92 $^{7.1}$ Ba	9.77$^{4.7}$Ca	11.22 $^{4.1}$ Da
	T3	4.15 $^{7.1}$ Ab	5.29 $^{6.2}$ Bb	8.12$^{4.8}$ Cb	9.30 $^{4.2}$ Db
	T4	4.00 $^{7.3}$ Ab	5.11 $^{6.7}$ Bb	7.91$^{4.5}$ Cb	9.63 $^{3.7}$ Db
	T5	3.52 $^{8.0}$ Ac	4.52 $^{6.2}$ Bc	6.43$^{4.5}$ Cc	7.45 $^{4.3}$ Dc
Clone	Treatment	Tensile index (Nm / g)			
		Revolutions during the refining			
		0	500	1500	3000
A	T1	25.6 $^{6.4}$ Aa	44.5$^{5.7}$ Ba	60.4 $^{5.3}$ Ca	77.5$^{4.2}$ Da
	T2	25.1 $^{5.9}$Aab	45.1 $^{5.6}$ Ba	59.7 $^{5.8}$Ca	77.8$^{4.6}$ Da
	T3	25.2 $^{5.6}$ Aab	44.2 $^{5.3}$ Ba	56.6 $^{4.9}$Cb	74.1$^{4.2}$ Db
	T4	24.0 $^{5.9}$ Ab	40.2 $^{5.4}$ Bb	54.1 $^{5.2}$Cc	72.5$^{4.7}$ Dc
	T5	21.5 $^{6.1}$Ac	36.5 $^{6.4}$ Bc	53.3$^{5.3}$Cc	66.6$^{4.1}$ Dd
B	T1	25.8 $^{6.2}$Aa	45.5 $^{5.7}$ Ba	65.2 $^{4.9}$ Ca	80.4 $^{5.1}$ Da
	T2	25.6 $^{6.5}$Aa	45.3$^{6.3}$ Ba	65.8 $^{5.2}$ Ca	79.6 $^{4.7}$ Da
	T3	25.3 $^{5.9}$Aa	42.6 $^{6.1}$ Bb	61.6 $^{5.4}$ Cb	76.0 $^{4.7}$ Db
	T4	24.8 $^{6.0}$ Aa	40.5 $^{6.1}$ Bc	58.8 $^{5.2}$ Cc	74.8 $^{4.8}$ Dc
	T5	22.3 $^{6.1}$Ab	39.6 $^{6.2}$ Bc	56.3$^{5.6}$ Cd	73.2 $^{4.9}$ Dd
Clone	Treatment	Stretch (%)			
		Revolutions during the refining			
		0	500	1500	3000
A	T1	1.98 $^{8.2}$Aa	2.43$^{7.5}$Ba	3.78$^{6.9}$ Ca	4.11$^{6.3}$Da
	T2	1.88 $^{7.4}$Aa	2.33$^{7.1}$Ba	3.60$^{6.7}$ Ca	4.00$^{7.1}$Da
	T3	1.85 $^{7.8}$Aa	2.28$^{6.9}$ Ba	3.69$^{6.8}$ Ca	4.05$^{6.8}$ Da
	T4	1.56 $^{7.5}$Ab	2.22$^{7.3}$ Bb	3.23$^{6.5}$ Cb	3.77$^{6.1}$ Db
	T5	1.34 $^{7.4}$Ac	2.00$^{6.8}$ Bc	2.88$^{6.4}$ Cc	3.25$^{7.2}$ Dc
B	T1	1.88 $^{8.0}$Aa	2.38$^{7.4}$ Ba	3.24$^{6.5}$ Ca	3.79 $^{6.8}$Da
	T2	1.76 $^{7.5}$ Aa	2.22$^{7.6}$ Ba	3.09$^{6.8}$ Ca	3.60$^{5.8}$ Da
	T3	1.76 $^{6.8}$Aa	2.28$^{7.5}$ Ba	3.25 $^{6.6}$ Ca	3.52$^{6.8}$ Da
	T4	1.51 $^{7.1}$ Ab	2.05$^{6.5}$ Bb	3.00 $^{6.2}$ Cb	3.22$^{6.4}$ Db
	T5	1.28 $^{6.9}$Ac	1.90$^{6.3}$ Bb	2.66 $^{6.5}$ Cc	3.10$^{6.7}$ Db

Pulps were made with 100% seven years wood (T1); pulp made from 95% (T2); 85% (T3) and 75% (T4) seven years old wood and 100% two years wood (T5). Means followed by the same capital letter per line and lower case letter per column did not differ between them by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation.

The refining and addition of two-year-old wood in pulp production reduced its opacity (Table 6). The opacity is related to the ability of light in penetrating the paper (Sixta, 2006). A higher value imply in lower passage of visible light. The fiber from seven-year-old wood had a greater cell wall fraction and mechanical properties, providing greater resistance to collapse and resulting in paper with higher void volume. Thus, the transition of light between these void spaces and the fiber cell wall causes them to scatter, preventing its passage through the paper and increasing the opacity (Biermann, 1996; Anjos et al., 2014). The reverse occurred with refining, where the fibers collapsed, reducing the paper opacity.

Table 6. Opacity (%) at different levels of revolution during the refining in pulps made with two and seven years old wood of Eucalyptus grandis × E. urophylla

Clone	Treatment	Revolutions during the refining			
		0	500	1500	3000
A	T1	$84.8^{1.32}$ Aa	$80.7^{1.27}$ Ba	$77.5^{1.45}$ Ca	$72.3^{1.49}$ Da
	T2	$84.2^{1.35}$ Ab	$80.2^{1.53}$ Ba	$77.8^{1.23}$ Ca	$72.7^{1.39}$ Da
	T3	$84.8^{1.13}$ Ab	$80.4^{1.13}$ Ba	$77.4^{1.28}$ Ca	$72.8^{1.75}$ Da
	T4	$84.3^{1.02}$ Ab	$81.3^{1.18}$ Ba	$78.5^{1.45}$ Ca	$73.6^{1.24}$ Db
	T5	$82.6^{1.89}$ Ac	$78.7^{1.10}$ Bb	$75.8^{1.54}$ Cb	$70.3^{1.58}$ Dc
B	T1	$80.5^{1.77}$ Aa	$78.3^{1.35}$ Ba	$75.4^{1.48}$ Ca	$72.1^{1.89}$ Da
	T2	$80.6^{1.74}$ Aa	$78.6^{1.43}$ Ba	$76.3^{1.24}$ Ca	$72.8^{1.11}$ Da
	T3	$81.0^{2.11}$ Aa	$78.1^{1.25}$ Ba	$76.2^{1.45}$ Ca	$70.1^{1.32}$ Db
	T4	$78.1^{1.56}$ Ab	$77.6^{1.36}$ Bb	$76.1^{1.33}$ Ca	$70.6^{1.27}$ Db
	T5	$78.4^{1.34}$ Ab	$75.8^{1.67}$ Ac	$74.3^{1.65}$ Cb	$70.4^{1.89}$ Db

Pulps were made with 100% seven years wood (T1); pulp made from 95% (T2); 85% (T3) and 75% (T4)of seven years old wood and 100% two years old wood (T5). Means followed by the same capital letter per line and lower case letter per column did not differ between them by the Scott-Knott test at 5%. Values in superscript represent the coefficient of variation.

Refining improved mechanical properties and reduced the paper opacity. Thus, it is necessary to achieve an optimal point to obtain acceptable values for these parameters, because high mechanical properties and opacity values are desired for printing and writing paper (Biermann, 1996; Anjos et al., 2014; Severo et al, 2013).

The proportion of wood broken by wind that can be used in the pulp production varied according the parameters, the genetic material, and refining intensity. However, its use up to 5% did not change the pulp properties. Thus, this proportion is suggested when using wood broken by wind in pulp production, without quality loss.

3.4 Conclusion

The wood from two-year-old Eucalyptus plants showed a higher frequency of vessels and fibers with lower length, lower cell wall fraction, modulus of elasticity and hardness, and a higher microfibril angle. This wood can be used in pulp production at 5%, when mixed with seven-year-old wood without loss of quality. The use of two-year-old wood, above this percentage, reduced the tensile index, tear index and stretch, and increased the opacity and air resistance of the paper. Refining increased the tear index, tensile index, air resistance and stretch, and decreased opacity, thus, because of its importance, this step should be considered before using wood broken by winds for manufacture of pulp.

Acknowledgements

To "Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq)", "Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES)" and "Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG)" for financial support. Celulose Nipo-Brasileira S.A for the support and supply of biological materials. Global Edico Services corrected and proofread the English of this manuscript.

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4 CAPÍTULO 3: Anatomical, ultrastructural, physical and mechanical properties of two-year-old Eucalyptus clones subject to wind damage for solid wood production

Abstract

Wind damages may require early harvest of Eucalyptus wood, these damages occur mainly 24 months after planting, being necessary to create uses for this wood. This study aims to characterize and propose uses of two-year-old Eucalyptus wood. Four Eucalyptus grandis × Eucalyptus urophylla and two Eucalyptus grandis clones have been selected and their anatomical, ultrastructural, physical and mechanical characteristics evaluated. Clone A showed more robust fibers with better microfibril arrangement, resulting in better mechanical properties, and therefore, a better performance for structural use. Clone F showed a low variation of basic density in the radial direction, facilitating its machinability, and with the Clone B, showed a lower anisotropic factor, and therefore, is recommended for locations with high variations of humidity. The heterogeneity of the characteristics of the evaluated clones confirms the need for further studies, to choose those most adequate to each use.

Key words: fiber, modulus rupture, nanoindentation, x-ray densitometry, wood

4.1 Introduction

Wood is a heterogeneous material, therefore, its use requires specific care. The anatomical and ultrastructural characteristics reflect the physical and mechanical behavior of wood (Muñoz et al., 2012; Hein et al., 2013; Longui et al., 2014), and therefore, its use depends on a complete survey of these features.

Natural phenomena, such as wind damage, can induce wood harvest when the trees are young. This damage occurs mainly 24 and 36 months after planting in , and depending on the material and intensity of the winds, this damage can exceed 20% of the planted area (Cenibra, 2014).

The lack of alternatives for this material leads to its use for energy (Guerra et al., 2014). The low diameter is a limitation for use of two-years-old eucalyptus trees for solid wood production (Murara Junior et al., 2005). However, the trees broken by wind need to be removed from planted forests, thus, its possible get this wood for low price, allowing their use in the production of small objects (Vieira et al., 2010) and in the furniture industry (Lopes et al., 2011).

Using two-year-old wood depends on a complete study of their anatomy, ultrastructure, physics and mechanics. Therefore, the aim of this study was to characterize two-year-old Eucalyptus clones and suggest uses for this material.

4.2 Methodology

4.2.1 Biological Material

Three two-year-old trees were selected from each of the four Eucalyptus grandis × Eucalyptus urophylla (A, D, E, F) and two Eucalyptus grandis (B, C) clones. The trees were collected in Belo Oriente, Minas Gerais State, Brazil, 42º22'30" "South longitude" and 19º15'00" "West latitude". This region and Eucalyptus age was chosen because they have a high incidence of wind damage.

Three 5 cm thickness disks were removed at 1.3 m above the ground level to determine the density, anatomy, and ultrastructure of its wood. A three-meter log was removed from just above this position, and central plank was obtained to make the samples for the mechanical characterization and evaluation of the dimensional and volumetric variation of the wood.

4.2.2 Anatomical characterization

A sample was obtained from an intermediate position from pith to bark, in one of the 5 cm disks that was removed from 1.3 m above the ground level. Histological slides (Johansen, 1940) and macerated materials were prepared (Franklin, 1945). The length and width of the fiber, lumen diameter, diameter and frequency of the vessels, and height and width of the rays were measured. The cell wall thickness of the fiber was obtained by the difference between the width of the fiber and the lumen diameter, divided by two. The cell wall fraction was calculated using the equation:

$$C.W.F. = \frac{(2 \times C.W.T) \times 100}{F.W.} \qquad (1)$$

Where, C.W.T. = cell wall thickness (µm); F.W. = fiber width (µm); C.W.F. = Cell wall fraction (%).

4.2.3 Microfibril angle measurement

The microfibril angle of the S2 layer was measured in the specimens used in the anatomical characterization. After saturation, the wood blocks were cut with a microtome in the tangential plane in 10 µm thick sections and macerated with hydrogen peroxide solution and glacial acetic acid in the ratio 2:1 at 55°C for 24 hours to prepare temporary slides.

The measurement of the microfibril angle was performed by polarized light microscopy (Leney 1981), with an Olympus BX51 microscope adapted with a rotary stage, graduated from 0° to 360°, connected to the image analysis program Image Pro-plus.

4.2.4 Nanoindentation

A sample was removed in the opposite position to that used for anatomical characterization. A 3 × 3 × 3 mm specimen was made from this sample and embedded in epoxy resin solution to determine the modulus of elasticity and hardness of the S2 layer and the middle lamella. The nanoindentation was performed in a TriboIndenter Hysitron TI-900®. The maximum applied load was 100 µN for 60 seconds, with discharge performed in 20 µN/s. The modulus of elasticity of the fiber was determined according to the equation:

$$MOE = (1 - vm^2) \times \left(\frac{1}{Er} - \frac{1-vi^2}{Ei}\right)^{-1} \quad (2)$$

Where: MOE = modulus of elasticity (GPa); According instructions from the device manufacturer, vi = 0.07; vm = 0.35 and Ei = 1140 GPA. The reduced Modulus (Er) was obtained from the load-displacement curve from the initial slope of the unloading wherein the elastic response was generated (Muñoz et al., 2012).

The hardness was determined by the maximum load supported by the specimen divided by the contact area, according to the equation 3:

$$H = \frac{Pmax}{A} \quad (3)$$

Where: Pmax = maximum load of indenter penetration; A = Projected contact areas at maximum load.

4.2.5 Characterization of the physical properties

The basic density of the wood was determined by the ratio between the dry mass and green volume of wood in one of the 5 cm disks removed from 1.3 m above the ground level, according to NBR1194 (Associação Brasileira de Normas Técnicas - ABNT, 2003).

The wood samples were subjected to x-ray densitometry, to determine their apparent density variation in the radial direction. Diametral samples were obtained in one of the disks removed at 1.3 m above the ground. These sections were conditioned at 23°C and 50% relative humidity. The analysis was performed using the TRQ-01XTree-Ring Analyzer equipment.

Thirty samples (2 × 2 × 4 cm) per clone were saturated with water and the volume and dimensions were recorded, the volume of the sample was obtained by immersion in liquid, and the radial and tangential dimensions were measured with a caliper. Next, the samples were dried at 103°C and the volume and dimensions were recorded again. The volumetric swelling of the wood was determined using the equation:

$$VS\ (\%) = \frac{(Vs - Vd) \times 100}{Vd}$$

Where: VS(%) = volumetric swelling; Vs = volume of saturated wood; and Vd = volume of dry wood.

The radial swelling was determined using the equation:

$$RS\ (\%) = \frac{(RLs - RLd) \times 100}{RLd}$$

Where: RS(%) = radial swelling; RLs = radial length of the saturated wood; and RLd = radial length of dry wood.

The tangential swelling was calculated according to the equation:

$$TS\ (\%) = \frac{(TLs - TLd) \times 100}{TLD}$$

Where: TS (%) = tangential swelling; TLs = tangential length of saturated wood; and TLd = tangential length of dry wood.

Finally, the anisotropic factor was determined by the ratio between the tangential and radial swellings.

The dry wood mass was obtained from thirty samples (2 × 2 × 4 cm), dried at 103°C and placed in a climatic chamber at 23°C and 50% relative humidity for 15 days. The equilibrium moisture content was calculated using the equation:

$$EMC\ (\%) = \frac{(WM - DM) \times 100}{DM}$$

Where: EMC = equilibrium moisture content; WM = wet mass; DM = dry mass.

4.2.6 Mechanical characterization

The samples were conditioned at 23°C and 50% relative humidity to stabilize their mass. The compression parallel to the grain was determined from the samples with 2 x 2 x 4 cm, and the modulus of elasticity (MOE) and rupture (MOR) from samples with 2 x 2 x 30 cm, in a procedure adapted from the American Society for Testing and Materials (ASTM-1997). Thirty samples were used per clone.

4.2.7 Statistical analysis

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

4.3 Results and discussion

The anatomical parameters evaluated varied among the clones (Table 1). The height and width of the rays showed higher coefficients of variation in the classification of histological sections of Eucalyptus wood. The lumen diameter, cell wall thickness and cell wall fraction showed higher values for this parameter in the classification of fibers, indicating constituents with higher wood variation. All parameters in the evaluation of the ultrastructure of the wood had coefficient of variation below 10%.

Table 1: Anatomical and ultrastructural characterization of two-year-old Eucalyptus grandis × Eucalyptus urophylla clones

Cl.	F.L. (mm)	F.W. (µm)	L.D. (µm)	C.W.T. (µm)	C.W.F. (%)
A	0.971$^{(15.2)}$c	18.5$^{(13.3)}$b	9.8$^{(17.3)}$b	4.36$^{(16.8)}$c	47.3$^{(15.2)}$ c
B	0.822$^{(14.7)}$a	17.6$^{(13.2)}$b	10.4$^{(16.4)}$c	3.57$^{(17.3)}$a	40.9$^{(16.3)}$ a
C	0.929$^{(14.6)}$c	20.1$^{(12.1)}$c	12.1$^{(16.4)}$d	3.98$^{(17.1)}$b	39.7$^{(17.2)}$ a
D	0.873$^{(14.6)}$b	18.0$^{(12.4)}$b	10.2$^{(16.8)}$c	3.92$^{(17.2)}$b	43.7$^{(16.2)}$ b
E	0.880$^{(15.7)}$b	17.9$^{(13.7)}$b	10.2$^{(16.9)}$c	3.80$^{(17.7)}$b	42.5$^{(17.5)}$ b
F	0.794$^{(12.8)}$a	15.7$^{(13.4)}$a	8.9$^{(16.4)}$a	3.38$^{(17.3)}$a	43.2$^{(16.2)}$ b

	Ves. Diam. (µm)	Freq. (pores/mm^2)	Ray height (µm)	Ray width (µm)
A	90.1$^{(13.6)}$a	10.1$^{(11.5)}$b	239.3$^{(18.5)}$a	6.84$^{(17.9)}$b
B	109.6$^{(14.5)}$b	11.1$^{(12.5)}$b	306.4$^{(18.1)}$c	7.64$^{(16.8)}$c
C	100.1$^{(12.1)}$a	10.4$^{(10.8)}$b	217.8$^{(18.3)}$a	8.48$^{(13.5)}$d
D	113.2$^{(13.5)}$b	8.8$^{(12.1)}$a	226.3$^{(17.2)}$a	6.28$^{(15.4)}$a
E	112.5$^{(14.3)}$b	10.7$^{(12.8)}$b	278.2$^{(18.2)}$b	6.13$^{(16.2)}$a
F	106.6$^{(13.2)}$b	13.9$^{(11.8)}$c	207.3$^{(17.8)}$a	5.80$^{(17.5)}$a

CL.	Microfibril angle (°)	Moe of S2 layer (Gpa)	Hardness of S2 layer (Gpa)	Moe of m.l. (Gpa)	Hard. of m.l. (Gpa)
A	9.21$^{(9.8)}$a	15.5$^{(6.2)}$ d	0.289$^{(7.02)}$ b	9.97$^{(7.23)}$ c	0.335$^{(6.97)}$ c
B	10.88$^{(6.6)}$d	11.6$^{(7.0)}$ a	0.266$^{(7.34)}$ a	7.13$^{(7.23)}$ a	0.299$^{(6.89)}$ b
C	10.4$^{(8.2)}$c	12.6$^{(7.1)}$ b	0.269$^{(7.32)}$ a	7.95$^{(6.96)}$ b	0.298$^{(7.07)}$ b
D	9.16$^{(7.2)}$a	14.1$^{(7.6)}$ c	0.278$^{(7.19)}$ b	8.47$^{(7.55)}$ b	0.302$^{(7.34)}$ b
E	9.8$^{(7.3)}$b	13.2$^{(7.2)}$ b	0.262$^{(7.12)}$ a	8.67$^{(7.18)}$ b	0.309$^{(7.04)}$b
F	10.45$^{(6.2)}$c	13.6$^{(6.4)}$ b	0.278$^{(6.63)}$ b	7.15$^{(6.81)}$ a	0.274$^{(7.21)}$ a

CL. = Eucalyptus grandis × Eucalyptus urophylla clone; F.L.=Fiber length; F.W.= fiber width; L.D.=Lumen diameter; C.W.T.= Cell wall thickness; C.F.R.=Cell Wall Fraction; Ves. Diam.= Vesssel diameter; Freq.=Vessel Frequency; Moe of m.l.= Moe of middle lamella; Hard. of m.l.= Hardness of middle lamella. Means followed by the same letter vertically per parameter does not differ by the Scott-Knott test at 5% probability. Values in superscript represent the coefficient of variation.

Clone A had a greater cell wall thickness and a smaller lumen diameter, with a reverse tendency for Clone C. Thus, Clone A showed the largest cell wall fraction, while Clone C the lowest. The fibers are the main components of hardwood (Panshin and De Zeew, 1980), and therefore, a high cell wall fraction ensures better mechanical properties to the timber, as reported Eucalyptus propinqua wood (Longui et al., 2014).

The frequency and the average diameter of the pores, which vary between clones, are hollow structures that increase the wood permeability (Panshin and De Zeew, 1980). Thus, a higher frequency and diameter of these structures result in a good response to the drying (Shahverdi et al., 2012) and preservative treatments (Taghiyari, 2012). The ray cells are fragile because of their thin cell walls (Gricar and Eler, 2015), thus, materials with a higher height and width of the rays may have inferior mechanical properties, limiting their use for structural purposes.

Clones A and D had the lowest microfibril angle values. The arrangement of the cellulose chains in the cell wall was fundamental to its strength (Donaldson, 2008; Hein et al., 2013). Combining the fact that Clone A showed larger cell wall fraction, the fibers of this material showed higher hardness and modulus of elasticity. The opposite happened for clone B, which, due to its high microfibril angle and low cell wall fraction, showed the lowest values for modulus of elasticity (MOE) and hardness of fibers.

The physical behavior of wood differed between the Eucalyptus clones, but with less variability for equilibrium moisture and basic density (Table 2).

Table 2: Basic density, equilibrium moisture content, volumetric swelling, radial swelling, tangential swelling and anisotropic factor in six two-year-old Eucalyptus grandis × Eucalyptus urophylla clones

Clone	Basic density (g/cm^3)	Equi. mois. cont. (%)	Vol. swell. (%)
A	$0.421^{(4.2)}$d	$11.23^{(2.1)}$ a	$18.24^{(7.3)}$ b
B	$0.370^{(3.6)}$a	$10.58^{(2.5)}$ b	$16.43^{(7.2)}$ a
C	$0.372^{(4.0)}$a	$10.55^{(1.9)}$ b	$16.62^{(6.3)}$ a
D	$0.423^{(3.7)}$d	$11.32^{(1.6)}$ a	$20.99^{(7.3)}$ c
E	$0.387^{(3.2)}$b	$10.41^{(1.8)}$ b	$16.53^{(6.5)}$ a
F	$0.412^{(3.1)}$c	$10.56^{(2.1)}$ b	$16.91^{(6.7)}$ a
	Rad. swell. (%)	Tang. swell. (%)	Anisotropic factor
A	$5.89^{(7.7)}$b	$10.23^{(7.9)}$c	$1.76^{(7.5)}$b
B	$5.24^{(7.4)}$a	$8.86^{(8.3)}$a	$1.66^{(7.6)}$a
C	$5.68^{(6.9)}$b	$9.78^{(7.8)}$b	$1.77^{(7.2)}$b
D	$6.21^{(7.6)}$a	$11.54^{(8.3)}$d	$1.81^{(7.4)}$c
E	$5.12^{(6.1)}$a	$9.21^{(8.9)}$a	$1.74^{(7.4)}$b
F	$6.28^{(6.2)}$c	$9.67^{(8.9)}$b	$1.62^{(7.9)}$a

Equi. mois. cont,=Equilibrium moisture content; Vol. swell.=Volumetric swelling; Rad. swell.=Radial swelling; Tang. swell.= Tangencial swell. Means followed by the same letter vertically per parameter does not differ by the Scott-Knott test at 5% probability. Values in superscript represent the coefficient of variation.

The basic density, equilibrium moisture content, volumetric, radial, and tangential swelling were higher for Clone D and lower for Clone B. Materials with a higher basic density have a higher mass per unit volume, resulting in a higher adsorption of humidity and increasing the equilibrium moisture content (Pérez-PEÑA et al., 2011) and linear and volumetric swelling (Schulgasser and Witztum, 2015; Rouco and Muñoz, 2015).

All materials had a lower radial swelling than tangential swelling. The reason for this is still debated, but this may be because of the orientation of the ray cells, which provided their microfibrils in the radial direction, offering greater resistance to compression (Kollmann and Côté, 1968; Glass and Zelinka, 2010). This was also reported for Corymbia citriodora, Eucalyptus grandis, Eucalyptus saligna and Pinus elliottii (Pelozzi et al., 2012; Menezes et al., 2014).

The anisotropic factor did not show any relationship with the basic density of wood, even being the ratio of the tangential and radial swelling. The highest value for the anisotropic factor in clone D indicates that its wood has restricted use in places with high humidity variation. However, treatments such as acetylation (Himmel and Mai, 2015; Xie et al., 2013) and heat treatment (Korkut, 2012; Zanuncio et al., 2014b) can reduce the variation in the wood dimensions and allow its use in such places.

The X-ray densitometry showed that even clones with similar average density, may have different density patterns along the radial direction (Figure 1).

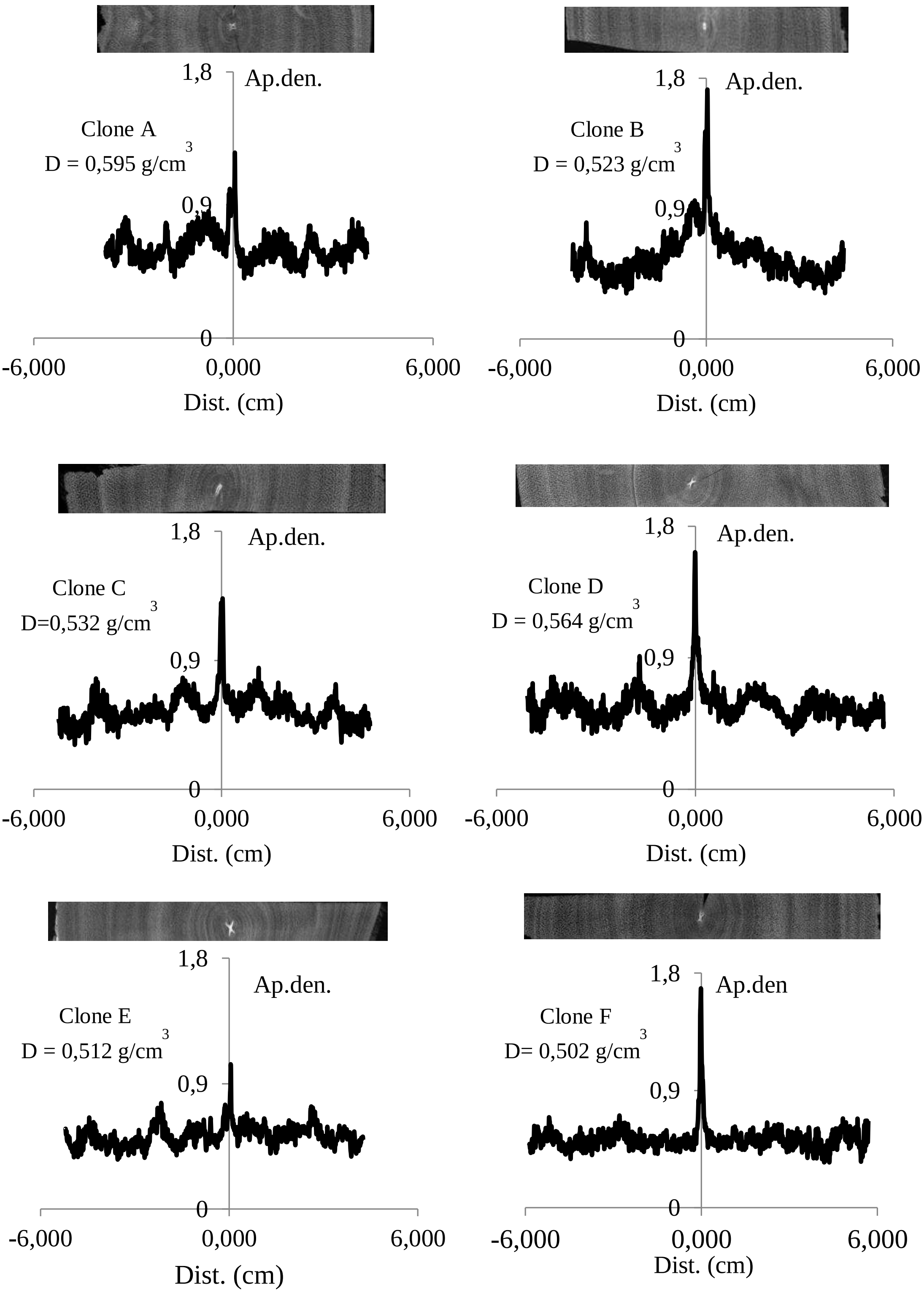

Figure 1: X-ray densitometry of the most representative samples per clone. Dist = distance from pith (cm.); Ap. den. = Apparent density (g/cm^3); D = average apparent density of the sample.

The apparent density of the samples was higher in the pith region corresponding to the parenchymal cell deposits, such as crystals and starch granules, which interacted strongly with the x-rays, resulting in high apparent density (Panshin and De Zeew, 1980; Belini et al., 2011). The region corresponding to these parenchyma cells was not accounted for the average apparent density, because it was not considered as wood.

Sample F showed less variation in the apparent density from pith to bark, while Clone A showed the highest variation. The density has relation with drying (Zanuncio et al., 2013b; Zanuncio et al., 2015) and wood machinability (Moura et al., 2011). Thus, materials with a homogeneous density along the radial direction have a more uniform behavior, facilitating its use.

The mechanical properties varied between clones, with the highest values for the modulus of elasticity and the least for the compression parallel to the grain (Table 3).

Table 3: Modulus of rupture (MOR) and elasticity (MOE) and compression parallel to the grain in six two-year-old Eucalyptus grandis × Eucalyptus urophylla clones

Clones	MOR (Mpa)	MOE (Mpa)	Comp. par. (Mpa)
A	$74.6^{(11.2)}$d	$7038^{(12.4)}$d	$40.8^{(9.5)}$c
B	$51.8^{(9.4)}$a	$4527^{(11.2)}$a	$33.9^{(10.5)}$a
C	$50.7^{(11.3)}$a	$5423^{(10.2)}$b	$32.9^{(10.2)}$a
D	$62.8^{(10.5)}$c	$5643^{(11.3)}$b	$34.1^{(10.1)}$a
E	$57.6^{(10.1)}$b	$5247^{(10.5)}$b	$32.7^{(9.5)}$a
F	$70.2^{(10.4)}$d	$6236^{(9.3)}$c	$36.1^{(11.2)}$b

Comp. par.= Compression parallel to the grain; Means followed by the same letter vertically per parameter does not differ by the Scott-Knott test at 5% probability. Values in superscript represent the coefficient of variation.

Clone A had a higher mechanical strength, suggesting it is more suitable for structural use and furniture manufacture subjected to mechanical stress, such as bookcases and chairs (Lopes et al., 2011).

Materials with greater cell wall fraction showed higher basic density, reducing the dimensional stability, and with the lower mocrofibril angle, improved the mechanical properties of the fibers and consequently the wood as a whole. This demonstrated how the anatomical and ultrastructural characteristics affected the physical and mechanical characteristics of the wood, and consequently, its use.

4.4 Conclusions

The evaluated parameters varied among the Eucalyptus clones. The materials with greater cell wall fraction resulted in higher basic density, and consequently, higher dimensional instability. Those with the lower microfibril angle and basic density indicated better mechanical properties. Clone A showed a low microfibril angle, high cell wall fraction, and better mechanical properties for fibers and wood and was suitable for structural use. Clones B and F showed a low anisotropic factor and were suitable for use in locations with high humidity variations. The heterogeneity of the material revealed the importance of a comprehensive study of each clone, to define the best use of its wood.

Acknowledgments

To Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Fundação de Amparo a Pesquisa do Estado de Minas Gerais (FAPEMIG) for financial support. Global Edico Services corrected and proofread the English of this manuscript. Celulose Nipo-Brasileira S.A to provide the biological material.

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5 CAPÍTULO 4: Chemical and energetic characterization of Eucalyptus clones subject to wind damage

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. Six two-years-old Eucalyptus grandis × Eucalyptus urophylla and two Eucalyptus grandis clones were selected in a region where wind damage is frequent. The basic density, calorific value, chemical composition of wood and calorific value, apparent relative density, immediate chemistry and gravimetric yield of charcoal were determined for all clones. Wood with high lignin content and low S/G ratio had higher gravimetric yield. The energetic 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, being the most suitable for this purpose.
Keywords: Charcoal; calorific value; gravimetric yield; lignin.

5.1 Introduction

The winds are characterized by the air movement from areas with high to low pressures (Kramer et al., 2014; Moore et al., 2013; Hale et al., 2015). Wind damage in forests are reported since 1940 (Mitchell, 2012) in many regions of the world (Moore et al., 2013; Lagergren et al., 2012; 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. 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, resulting in small diameter, low density and poor quality fibers (Ramirez et al., 2009; Veenin et al., 2005), hindering its use in pulp (Severo et al., 2013) 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.

5.2 Material and methods

5.2.1 Biological Material

Three trees per each of six (A, B, D, E, G, H) two-years-old Eucalyptus grandis × Eucalyptus urophylla and two Eucalyptus grandis (C, F) 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, and the basic density, chemical and energetic properties of these materials were analyzed.

5.2.2 Wood chemical characterization

One disk from each tree was 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 (American Society for Testing and Materials, 1994); besides the soluble lignin (Gomide and Demuner, 1986); insoluble lignin (Goldschimid, 1971) and lignin's syringyl/guaiacyl (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.

5.2.3 Physical and energetic characterization of wood and charcoal

The wood basic density was determined according to NBR 11941 (Associação Brasileira de Normas Técnicas- ABNT, 2003), the gross calorific value according to NBR 8633 (ABNT, 1983) and the wood energetic 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 wood.

5.2.4 Statistical analysis

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

5.3 Results and discussion

The extractives, ash, soluble, insoluble and total lignin, holocellulose, lignin S/G ratio and elemental composition were determined to characterize the wood chemical composition (Table 1).

Table 1: Chemical composition of eight two-years-old Eucalyptus grandis × Eucalyptus urophylla clones (Cl.)

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	$0.02^{15.2}$ a
B	$50.55^{4.1}$ a	$42.67^{4.3}$ a	$5.78^{3.8}$ a	$0.06^{15.5}$ a
C	$50.66^{4.0}$ a	$42.80^{3.4}$ a	$5.85^{4.2}$ a	$0.03^{14.1}$ a
D	$50.49^{3.9}$ a	$43.02^{4.1}$ a	$5.76^{4.6}$ a	$0.03^{14.2}$ a
E	$50.90^{4.5}$ a	$42.35^{2.8}$ a	$5.90^{4.2}$ a	$0.05^{14.4}$ a
F	$50.77^{4.6}$ a	$42.63^{3.7}$ a	$5.89^{4.7}$ a	$0.08^{14.8}$ a
G	$50.43^{3.7}$ a	$42.95^{4.1}$ a	$5.85^{3.6}$ a	$0.03^{14.7}$ a
H	$50.30^{4.6}$ a	$43.28^{3.5}$ a	$5.82^{4.2}$ a	$0.07^{14.2}$ a

Cl.= Eucalyptus grandis × Eucalyptus urophylla clone; 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.

The clone B showed lower extractives content, while the D and F had the highest. 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 represent impurities and hinder the use of wood for energy (Bustamante-García et al., 2013).

The soluble lignin content of the materials were 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. 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 B, 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 holocelulose 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 carbonization yield (Liu and Han, 2015) and being unwanted in wood for energy production.

All wood materials showed similar elemental composition. 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).

5.3.1 Physical and energetic characterization

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).

Table 2: Density (g/cm^3), gross calorific value (MJ/g) and energy density (MJ/cm^3) of the wood and charcoal from eight Eucalyptus grandis × Eucalyptus urophylla clones

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
	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.

The charcoal from clones with higher wood basic density had higher apparent relative density with Pearson correlation coefficient of 0.891 between these variables. 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; Pereira 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 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, being the second effect with highest proportion, and therefore, the charcoal had higher energetic 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 between energetic density and wood basic density and charcoal relative apparent density, with Pearson correlation coefficient of 0.973 and 0.998, respectively.

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 3: Gravimetric yield (Grav), fixed carbon (F.C.), volatile matter (V.M.), ash (%) and elemental composition from charcoal of eight Eucalyptus grandis × Eucalyptus urophylla clones

Clone	Grav. (%)	F.C. (%)	V.M. (%)	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.

The gravimetric yield of the clones E, F and H were higher 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. 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. 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 degradation of other constituents, especially holocellulose (Moreno and Font, 2015). In the furnace for steel production, minerals presents 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 nitrogen are present in in greater proportion in the holocellulose, which has low resistance to high temperatures.

5.4 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.

Acknowledgment

To “Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq)” and “ Fundação de Amparo a Pesquisa do Estado de Minas Gerais (FAPEMIG)” for financial support. Celulose Nipo-Brasileira S.A. provided biological material for this study.

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6 CONCLUSÕES GERAIS

A técnica proposta para avaliação dos clones contra a ação dos ventos se mostrou satisfatória, com índice de correlação de Pearson de -0.8392 entre a força para quebra da árvore e a razão entre a área danificada pelo vento e a área total plantada, evidenciando que quanto maior a força necessária para quebra da árvore, menor será a área quebrada pelo vento para aquele material. A melhoria na qualidade da madeira dos clones de Eucalyptus é uma importante alternativa para reduzir os danos pelo vento. Entre as características da madeira avaliadas, a densidade básica merece destaque, devido a sua rápida determinação, baixo custo para aplicação do teste e por apresentar correlação de Pearson de -0,8475 com área danificada pelo vento em porcentagem, evidenciando que quanto maior a densidade básica do material, menor será a área quebrada pelo vento. Outras características também foram importantes na seleção de clones resistentes à ação dos ventos, visto que materiais com alta fração parede, módulo de elasticidade das fibras e lamela média, dureza das fibras, modulo de ruptura, tensão de crescimento e baixo ângulo microfibrilar foram encontrados nos clones com menor área danificada pelos ventos (%). Desse modo, tais características devem ser visadas em clones de eucalipto que serão plantados em áreas com alta incidência de danos pelos ventos.

Visando a utilização da madeira quebrada pelo vento para a fabricação de polpa celulósica. A proporção de madeira de dois anos, correspondente a idade com maior índice de quebra pelo vento, que pode ser utilizada na fabricação de polpa celulósica, variou com o clone, parâmetro e nível de refino. No entanto, a polpa produzida com 5% de madeira de dois anos e 95% daquela de sete anos foi semelhante aquela com 100% de madeira desta ultima idade em todos os parâmetros avaliados. Por isto, pode-se utilizar 5% de madeira de dois anos sem perda da qualidade do produto. A razão para a queda na qualidade da polpa se deve principalmente as características anatômicas, visto que a madeira de dois anos apresentou fibras com menor fração parede, comprimento, módulo de elasticidade, dureza e maior ângulo microfibrilar.

Para a utilização da madeira quebrada pelo vento para madeira sólida. Os clones avaliados apresentaram características distintas, confirmando a necessidade de estudos por clone para uma melhor utilização dos mesmos. Os clones com fibras mais robustas e melhor arranjo microfibrilar resultaram em melhores propriedades mecânicas e, por isto, melhor desempenho para uso estrutural. Os clones com variação da densidade no sentido radial apresentam comportamento mais uniforme, facilitando sua trabalhabilidade. Por fim, clones com menor coeficiente de anisotropia são recomendados para locais com alta variação de umidade.

Para utilização na geração de energia, todos os materiais apresentaram potencial para bioenergia, tanto a partir da queima direta quanto através da fabricação de carvão. Madeiras com alto teor de lignina e baixa relação S/G apresentaram rendimento gravimétrico maior, visto que esta estrutura possui alta resistência a altas temperaturas, prevalecendo no carvão produzido a 450 °C. Enquanto que uma alta densidade básica da madeira resultou em alta densidade energética da madeira e carvão, visto que houve pouca variação do poder calorífico dos materiais. Evidenciando como as propriedades físicas e a composição química da madeira são importantes na sua utilização energética.