CHEMICAL CHARACTERISTICS AND KRAFT PULPING OF TENSION WOOD FROM *Eucalyptus globulus* LABILL[1]

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ABSTRACT – Tension (TW) and opposite wood (OW) of *Eucalyptus globulus* trees were analyzed for its chemical characteristics and Kraft pulp production. Lignin content was 16% lower and contained 32% more syringyl units in TW than in OW. The increase in syringyl units favoured the formation of β-O-4 bonds that was also higher in TW than in OW (84% *vs.* 64%, respectively). The effect of these wood features was evaluated in the production of Kraft pulps from both types of wood. At kappa number 16, Kraft pulps obtained from TW demanded less active alkali in delignification and presented slightly higher or similar pulp yield than pulps made with OW. Fiber length, coarseness and intrinsic viscosity were also higher in tension than in opposite pulps. When pulps where refined to 30°SR, TW pulps needed 18% more revolutions in the PFI mill to achieve the same beating degree than OW pulps. Strength properties (tensile, tear and burst indexes) were slightly higher or similar in tension as compared with opposite wood pulps. After an $OD_0(EO)D_1$ bleaching sequence, both pulps achieved up to 89% ISO brightness. Bleached pulps from TW presented higher viscosity and low amount of hexenuronic acids than pulps from OW. Results showed that TW presented high xylans and low lignin content that caused a decrease in alkali consumption, increase pulp strength properties and similar bleaching performance as compared with pulps from OW.

Keywords: *Eucalyptus globulus*, Reaction wood, Kraft pulping, Xylans and Lignin structure.

CARACTERÍSTICAS QUÍMICAS E POLPAÇÃO KRAFT DE MADEIRA DE TRAÇÃO DE Eucalyptus globulus LABILL

RESUMO – *Madeira de tração e oposta de árvores de* ***Eucalyptus globulus*** *foram analisadas quanto a suas características químicas e produção de polpa Kraft. A caracterização química da madeira de tração (TW) de* ***Eucalyptus globulus*** *Labill. mostrou um conteúdo similar de celulose, alto conteúdo de xilanas e baixo conteúdo de lignina quando comparada com a madeira oposta (OW) de uma mesma árvore. O conteúdo de lignina foi 16% menor e contém 32% mais unidades siringila em TW que em OW. O aumento das unidades siringila favoreceu a formação de ligações β-O-4 que também foram mais altas em TW que em OW (84%* vs. *64%, respectivamente). O efeito destas características foi avaliado na produção de polpas Kraft de ambos tipos de madeira. A um número kappa de 16, as polpas Kraft obtidas de TW demandaram menos álcali ativo na deslignificação e apresentaram rendimento de polpa levemente maior ou similar que as polpas obtidas com OW. O comprimento das fibras, o* coarseness *e a viscosidade também foram maiores em polpas de madeira de tração que oposta. Quando as polpas foram refinadas a 30°SR, as polpas TW necessitaram 18% mais revoluções no refinador PFI para atingir o mesmo grau de refino que as polpas OW. As propriedades de resistência mecânica (índices de tração, rasgo e estouro) foram levemente maiores ou similares em polpas de madeira de tração quando comparadas com polpas de madeira oposta. Depois de uma sequência de*

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branqueamento $OD_0(EO)D_1$, ambas polpas alcançaram até 89% de alvura ISO. As polpas branqueadas de TW apresentaram viscosidade mais alta e menor conteúdo de ácidos hexenurônicos que as polpas de OW. Os resultados mostraram que TW apresenta alto teor de xilanas e menor teor de lignina que ocasionam uma diminuição no consumo de álcali, aumento das propriedades de resistência mecânica e comportamento similar no branqueamento em comparação com polpas de OW.

Palavras-chave: ***Eucalyptus globulus****, Madeira de reação, Polpação Kraft, Xilanas e Estrutura da lignina.*

1. INTRODUCTION

Eucalyptus species are particularly relevant to the forest industry in different parts of the world with *E. globulus* the most widely planted in regions with Mediterranean climate such as Australia, Chile, Spain and Portugal (PAMPOLINA et al., 2002). The fiber of *E. globulus* presents suitable characteristics for printing and writing papers with good strength and optical properties (KIBBLEWHITE et al., 2000).

E. globulus trees could present the formation of strongly stressed wood in the upper side of branches, in leaning stems or even in well-growth erected trees (WASHUSEN 2002; WASHUSEN et al., 2003; CLAIR et al., 2006a; AGUAYO et al., 2010). The stressed wood, known as tension wood in hardwoods, tend to restore the verticality of the stem and is formed when the tree is growing under determined geographical or climatic conditions that could affect the natural trend of the tree to upright growth (BOYD, 1977; KUBLER, 1988; BAMBER, 2001; WASHUSEN, 2002). Tension wood is characterized by the development of an extra cell wall layer, the gelatinous layer (G-layer), which replaces the S2 or S3 layer in the cell wall and is made up of crystalline cellulose with a low microfibrilar angle (BABA et al., 1996; RUELLE et al., 2007; HENRIKSSON et al., 2009).

Trees that presented a high amount of tension wood or deformation in its trunk are considered inadequate for use as timber or structural material due to shrinkage and formation of fissures during drying and mechanical processing causing yield losses of lumber (CARVALHO et al., 2010; CLAIR; THIBAUT, 2001; CLAIR et al., 2006b) but could be a suitable raw material for chemical transformation of wood such as pulp for paper or cellulosic bioethanol. This work presents comparative results for the chemical characterization and Kraft pulping feasibility of tension and opposite wood of *E. globulus* growing in southern Chile.

2. MATERIALS AND METHODS

2.1 Wood sampling

Forty *E. globulus* trees were randomly selected and cut down from a commercial stand located in the Biobío Province, southern Chile. Trees growing at the edge of the plantation were not included in the sampling. All trees were well grown and erect without any evident trunk deformation, and with the diameter at breast height (DBH) between 20 and 25 cm. Logs of 35-40 cm were cut above 1.0 m height from each tree's bottom. All the logs were visually examined and ten with evident tension wood formation, determined by pith asymmetry in the longitudinal axis of the log, were selected. Wood discs with 4-5 cm thickness were cut from each log. In the discs, the wood was separated into tension (TW) and opposite wood (OW). Wood chips from each separated region, TW and OW, were hand-made. A fraction of the wood chips was milled in a knife mill and sieved to 45/60 mesh for chemical characterization.

2.2 Chemical characterization of wood

Milled wood (2 g) was extracted in a Soxhlet apparatus with ethanol/toluene for 8 h, followed by extraction with 95% ethanol for another 8 h. Extractive free milled wood was used for subsequent chemical analysis. Samples were characterized for glucans and lignin content by the acid hydrolysis with 72% H2SO4 following the procedure detailed by Mendonça et al. (2008). The carbohydrates present in hemicelluloses were quantified by acid methanolysis followed by gas chromatography analysis according to Sundberg et al. (1996). Thioacidolysis was performed on 20 mg of extractive-free milled wood in 10 mL of reagent according to the method published by Rolando et al. (1992) and using gas chromatography conditions detailed by Lu and Ralph (1997). This technique allows the quantification of the amount of syringyl and guaiacyl units bonded by β-O-4 linkages and also the frequency of β-O-4 linkages in the lignin. All analyses described in this section were performed in triplicate for each sample.

2.3 Kraft pulping and ECF bleaching

Wood chips from tension and opposite wood regions were submitted to Kraft pulping in a rotary digester equipped with 4 independent 1.5-L vessels (Regmed, Brazil). Each vessel was loaded with 50 g of wood chips (dry basis) and 200 mL of cooking liquor with active alkali (AA) varying from 17% to 22%, and 30% sulfidity (both calculated on dry wood basis and expressed as NaOH equivalents). Reactor heating rate was 2.1°C/min, cooking temperature was 165°C and the H-factor used was 800. Produced pulps were screened through a 0.2 mm slot screen and the screened pulp yield was determined. Kappa number and pulp brightness were measured according to TAPPI Standards T236 om-99 and T218 sp-02, respectively. Pulp viscosity was measured according to ISO 5351:2004 standard. Mean fiber length, fiber width, kink index, coarseness and fines were determined in Fiber Tester equipment (Lorentzen and Wettre, Sweden). Approximately 3000 fibers were measured by the equipment for the biometric characteristics determination. Samples were preparaed according to TAPPI Standard T271 om02. Hexenuronic acids (HexA) content in Kraft pulps was quantified by TAPPI Standard T282 pm-07. Pulps from tension and opposite wood of E. globulus produced at kappa number 16±1 were refined in a PFI mill and handsheets prepared according to TAPPI Standard T248 sp-00 and T 220 sp-01, respectively. Strength properties were measured according to Tappi Standards: T494 om-92 (tensile index), T414 om-98 (tear index) and T403 om-97 (burst index).

Kraft pulps from tension and opposite wood of *E. globulus* with kappa number 16±1 were bleached using an elemental chlorine free (ECF) sequence, $OD_0(EO)D_1$. Oxygen delignification was performed in a 1-L Parr reactor (Parr Instrument Co., USA) loaded with 10 g pulp (dry basis) impregnated with 0.5% $MgSO_4$ and 2% NaOH (both on pulp basis). Pulp consistency was adjusted to 10% with distilled water; the reactor was sealed and pressurized with 100 psi O_2. Reaction was performed at 100°C/60 min. D_0 and D_1 steps were performed in polyethylene bags at 10% pulp consistency using ClO_2 solution with active chlorine concentration adjusted according to the kappa factor (GELLERSTEDT et al., 2009). The bag was sealed and the reaction carried out at 60°C/40 min (D_0 step) and 70°C/150 min (D_1 step). After D_0 and before D_1, pulp was submitted to alkaline extraction in presence of oxygen (EO step). This step was performed in the 1-L Parr reactor at 10% pulp consistency with 2% NaOH, 60 psi O_2 and 60°C/60 min. Pulp was washed in water, centrifuged and used for the D_1 bleaching step. After each bleaching step, kappa number, brightness, intrinsic viscosity and HexA content in the pulps were determined following the procedures described previously.

3. RESULTS

3.1 Chemical composition of tension and opposite wood of *E. globulus*

Ten trees of *E. globulus* that presented tension wood formation were selected from 40 trees sampled in a commercial plantation. The diameter at breast heigth of the trees varied from 18.1 to 25.2 cm. Detailed chemical composition of tension and opposite wood of *E. globulus* is shown in Table 1. The main differences observed were in the amount of hemicelluloses and xylans, which were 9% and 34% higher in tension than in opposite wood, respectively; and in lignin content, which was 16% lower in tension than in opposite wood. Structurally, tension wood lignin contains 32% more syringyl units (3158 μmol/g lignin) than opposite wood (2379 μmol/g lignin). The increase in syringyl units favoured the formation of aryl-ether linkages and the amount of β-O-4 bonds was also higher in tension than in opposite wood (84% *vs.* 64%, respectively).

3.2 Kraft pulping

Kraft pulping of tension and opposite wood of *E. globulus* was performed at H-factor 800 and active alkali (AA) concentrations varying from 17% to 22%. Due to the low lignin content and the high amount of aryl-ether linkages, tension wood was easier delignified than opposite wood, and pulps with similar kappa number were obtained with less active alkali (Figure 1A). Bleachable-grade Kraft pulps with kappa number 16±1 were obtained with 20% AA for tension wood and with 22% AA for opposite wood. Pulp yield was higher in pulps from tension wood with kappa number higher than 16 (Figure 1B). This is mainly owing to the low amount of rejects in pulps from tension wood that varied from 7.2 to 0.9% as compared with the amount of rejects from pulps from opposite wood (from 24.6 to 1.7%).

Table 1 – Chemical composition of tension and opposite wood of *E. globulus**.
***Tabela 1** – Composição química da madeira de tração e oposta de **E. globulus**.*

Component	Opposite wood	Tension wood	p-value
Cellulose (%)	48.0^{a}	47.2^{a}	0.2002
Hemicelluloses (%)	22.1^{b}	24.1^{a}	<0.0001
Arabinans (%)	0.3^{a}	0.3^{a}	0.0004
Mannans (%)	1.2^{a}	1.3^{a}	0.2486
Xylans (%)	10.0^{b}	13.4^{a}	<0.0001
Glucans (%)	5.1^{a}	4.2^{b}	0.0004
Galactans (%)	1.2^{a}	0.9^{b}	0.0014
Rhamnnans (%)	0.9^{a}	0.7^{b}	0.0026
Uronic groups (%)	3.3^{a}	3.3^{a}	0.8974
Lignin (%)	25.9^{a}	21.7^{b}	<0.0001
G-units (μmol/g lignin)	640^{b}	805^{a}	0.0064
S-units (μmol/g lignin)	2379^{b}	3158^{a}	<0.0001
β-O-4 linkages (%)	63.7^{b}	83.7^{a}	<0.0001
Extractives (%)	3.0^{a}	2.7^{a}	0.3319

* Values with different letter within a row differ significantly at $p < 0.05$ (t-Test).

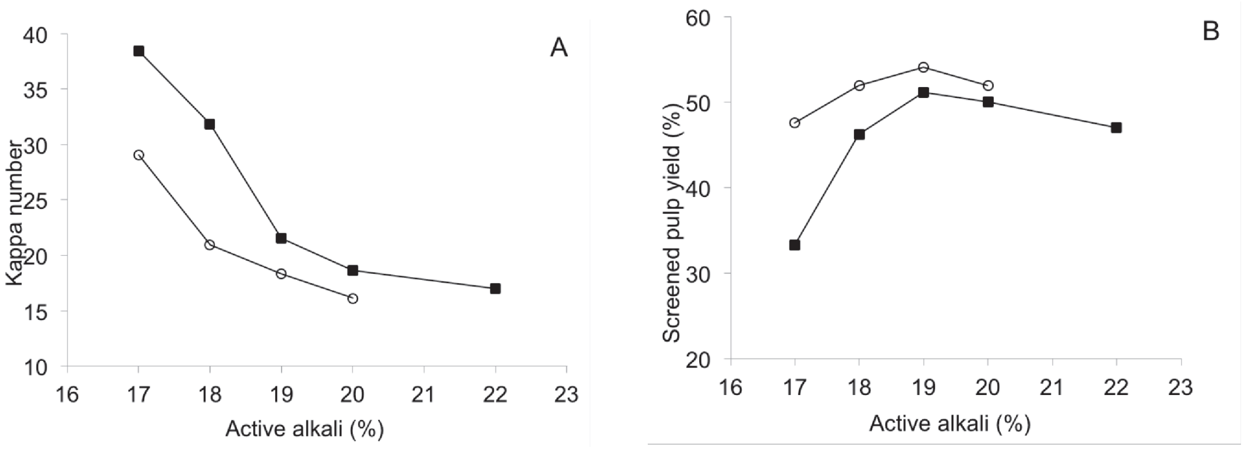

Figure 1 – (A) Kappa number and (B) screened pulp yield variation with different active alkali in cooking liquor (30% sulfidity in all cases). (-■-) Tension and (-○-) opposite wood of *E. globulus*.
***Figura 1** – Variação do (A) número kappa e (B) rendimento de polpa depurada com diferente álcali ativo no licor de cozimento (30% de sulfidez em todos os casos). (-■-) Madeira de tração e (-○-) oposta de **E. globulus**.*

However, at kappa number 16, pulp yield of both samples was around 51% (but in tension wood it was achieved with less AA, as showed in Fig. 1A).

3.3 Fiber properties

Fiber characteristics and pulp strength properties (Table 2) were determined in the unbleached Kraft pulps (UKP) of tension and opposite wood of *E. globulus* produced with kappa number 16. Fiber length was higher in pulp from tension than in pulp from opposite wood with values of 0.75 mm and 0.83 mm, respectively. Fiber width is rather similar in both pulps (approximately 18 μm). Pulps from tension wood presented lower number of vessels than pulps from opposite wood (185 *vs.* 209, respectively). Fiber coarseness (weight of the fiber divided by its length) was 11% higher in pulps from tension wood. The kink index, a local deformation on the fiber that can be a weak point and the amount of fines in pulps of both types of wood was similar. The intrinsic viscosity

Table 2 – Characteristics of unbleached Kraft pulps (kappa 16) of tension and opposite wood of *E. globulus*.
Tabela 2 – *Características das polpas Kraft não branqueadas (kappa 16) da madeira de tração e oposta de* ***E. globulus***.

Characteristic	Opposite wood	Tension wood	Variation* (%)
Fiber length (mm)	0.75^a	0.83^b	10.7
Fiber width (µm)	18.2^a	18.8^a	3.3
Vessels per 100,000 fibres	209^a	185^a	-11.5
Coarseness (mg/100 m)	7.0^a	7.8^a	11.4
Kink index	1.41^a	1.37^a	-2.8
Fines content (%)	3.5^a	3.4^a	-2.9
Intrinsic viscosity (cm^3/g)	821^a	932^b	13.5
PFI revolutions (rpm)**	3300^a	3900^b	18
Tensile index (N.m/g)**	92^a	100^a	8.7
Tear index (mN.m^2/g)**	9.6^a	10.4^a	8.3
Burst index (kPa.m^2/g)**	6.8^a	7.4^a	8.8

* Increase or decrease of the characteristic in pulp from tension wood as compared with pulp from opposite wood. Values with different letter within a line differ significantly at $p < 0.05$ (t-Test). ** Measured at 30°SR.

of the tension wood pulp is 13.5% higher than in pulp from opposite wood, indicating that cellulose was degraded at less extent during cooking.

The unbleached pulps produced at kappa 16 were refined in a PFI mill to determine their strength properties. Pulps from tension wood need more PFI revolutions to achieve the same beating degree than pulps from opposite wood (Table 2). Both, fiber anatomy and composition have influence in the pulp refining and its properties. Despite pulps from tension wood demanded more energy to refine, their strength properties were similar or slightly higher than pulps from opposite wood. The strength properties were favored by the higher fiber length and viscosity of the pulps from tension wood. The contribution of the residual xylans to the fiber strength properties is more controversial and varied information is found in the literature on its importance (LINDSTRÖM et al., 1992; SPIEGELBERG et al., 1966; MOLIN et al., 2002; SILVA et al., 2011).

3.4 Pulp bleaching

ECF bleaching of pulps from tension and opposite wood was performed using an $OD_0(EO)D_1$ sequence. The main parameters evaluated after each of the different stages were kappa number, brightness, viscosity and HexA amount (Figure 2). Unbleached Kraft pulps (UKP) produced at kappa number 16 presented the following characteristics for opposite and tension wood, respectively: 37 *vs.* 39% ISO brightness, 821 *vs.* 932 cm^3/g intrinsic viscosity and 58 *vs.* 52 mmol HexA/kg pulp.

Kappa reduction and brightness gain presented a similar behaviour in both types of pulps during bleaching (Figs. 2A and 2B, respectively). Fully bleached pulps presented 89% ISO brightness and kappa number below 2. Significant differences in bleaching of pulps from tension and opposite wood were found for viscosity and HexA amount. During bleaching, viscosity decreased 24% for opposite wood pulps and 21% for tension wood pulps. The high viscosity of pulp from tension wood was maintained during bleaching and at the end of the process the viscosity of the pulp from tension wood was 18% higher than for pulp from opposite wood (737 *vs.* 623 cm^3/g, respectively). The HexA amount did not decreased with O_2 delignification since HexA does not react with oxygen in alkaline medium (SHACKFORD et al., 2000). However, after the two ClO_2 bleaching steps, the HexA content was lower than 15 mmol/kg pulp that is in the range of values used obtained for eucalyptus pulps (COLODETTE et al., 2008; CADENA et al., 2010).

4. DISCUSSION

Tension wood is commonly associated with branches or leaning stems but could also be present in well-growth erected trees, which make difficult its identification in commercial stands planted for cellulosic pulp production. Ten from forty trees of 8-year old *E. globulus* were randomly sampled in a commercial plantation of a forest company and presented the formation of tension wood. The reaction wood was observed in the first 2 m from the bottom of the tree

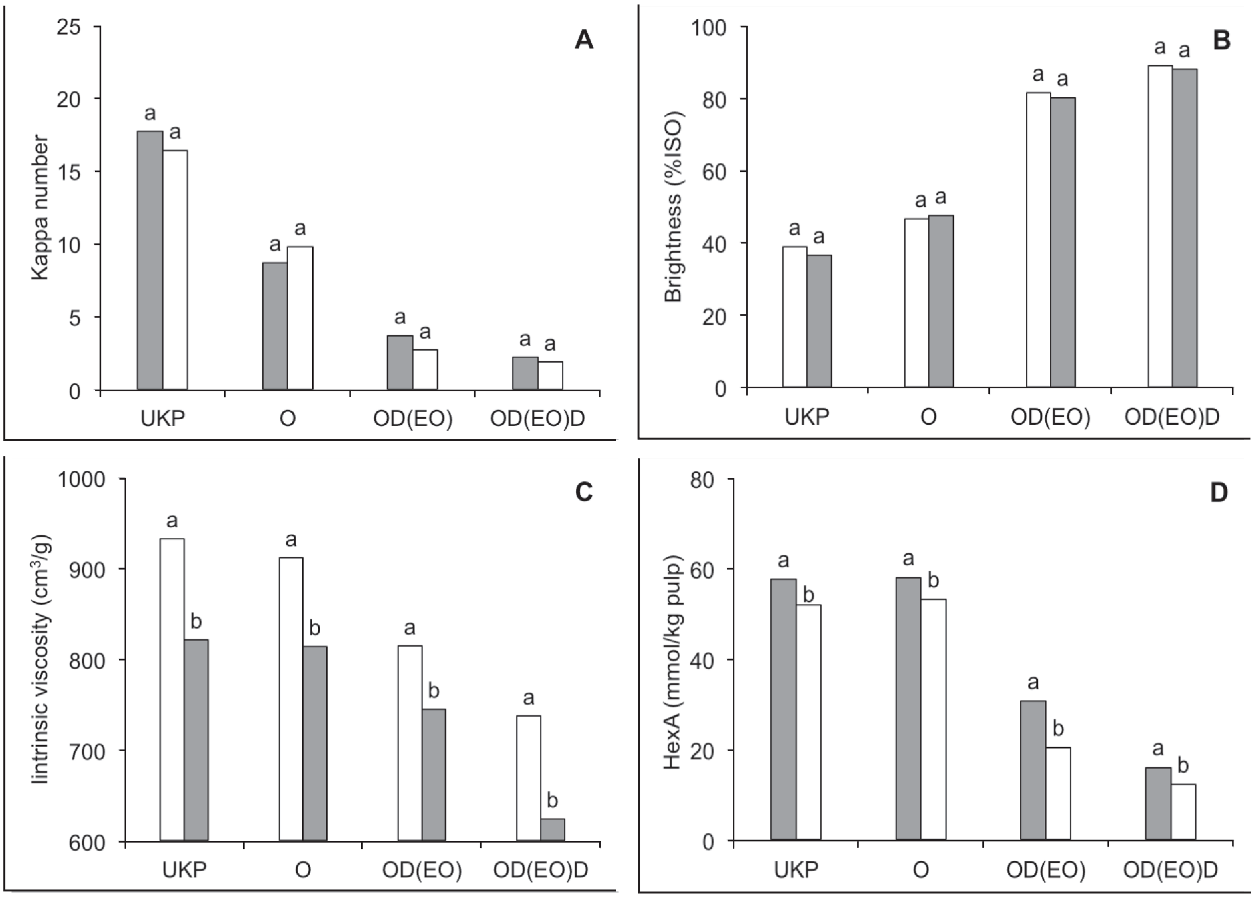

Figure 2 – ECF bleaching of Kraft pulps from opposite (gray bars) and tension (white bars) wood of *E. globulus*. (A) Kappa number, (B) brightness, (C) intrinsic viscosity and (D) hexenuronic acids. Different letter for a given property differ significantly at $p < 0.05$ (t-Test). UKP: unbleached Kraft pulp, O: delignification with oxygen, D: chlorine dioxide bleaching, EO: alkaline extraction in presence of oxygen.

***Figura 2** – Branqueamento ECF de polpas Kraft de madeira oposta (barras cinzas) e de tração (barras brancas) de **E. globulus**. (A) Número kappa, (B) alvura, (C) viscosidade intrínseca e (D) ácidos hexenurônicos. Diferentes letras para uma dada propriedade representa uma diferença significativa a $p < 0.05$ (Teste t). UKP: polpa Kraft não branqueada, O: deslignificação com oxigênio, D: branqueamento com dióxido de cloro, EO: extração alcalina em presença de oxigênio.*

and was determined as an asymmetry of the pith in the longitudinal axis of the log. Chemical characterization showed differences mainly in the content of xylans and lignin, and no significant variation in the cellulose content. Aguayo et al. (2010) showed that the distribution of enriched carbohydrate cell walls in tension wood is not homogeneous throughout all the regions, being located only in disperses zones of xylem in an amount that was not enough to be reflected in differences between the chemical composition of tension and opposite wood. The amount of other carbohydrates present in the hemicelluloses and the extractives were rather similar or presented small differences between both types of wood. Lignin of tension wood also presented a higher amount of syringyl units and β-O-4 linkages than opposite wood. The increase of syringyl units in lignin and its positive correlation with the β-O-4 linkages formation was previously reported by several authors (JOSELEAU et al., 2004; PILATE et al., 2004; RALPH et al., 2004; GUERRA et al., 2008). The low lignification of tension wood cells and the high amount of xylans, allow the tension wood fibers act like “springs” to restore the verticality of the tree.

Despite there were no previous antecedents on the pulping performance of tension wood in hardwood species; some characteristics of the wood and lignin composition supported the results obtained. Pinto et al. (2005) evaluated the effect of structural wood components of different hardwoods species (including *E. globulus*) in Kraft pulping and reported that the ease degradation and dissolution of lignin in cooking liquor was determined by differences in the proportion of syringyl and guaiacyl units and the degree of condensation. Mansfield and Weineisen (2007) studied the wood fiber quality and Kraft pulping of *Populus tremuloides* clones and found that those trees with high syringyl to guaiacyl ratio (determined by thioacidolysis) account for improved ease of pulping and pulping yield. These results were supported by the published by Guerra et al. (2008) that evaluated the chemical composition and pulping of different *E. globulus* genotypes. Significant variations were observed in the pulp yield and specific consumption of wood to produce pulps with similar kappa numbers. The data reported provides evidence of the influence of lignin features in the pulping response of different eucalyptus clones. Significant correlations were observed between pulp yield and specific consumption of wood, and the content of β-O-4 linkages.

The fiber characteristics are in accordance to those previously reported for the species. Jorge et al. (2000) reported that the fiber length in *E. globulus* wood growing in Portugal varied from 0.7 to 1.4 mm, while Muneri and Raymond (2001) reported fiber length for *E. globulus* growing in the south of Australia varying from 0.56 to 0.75 mm. High fiber length, coarseness, thicker cell walls, number of fibres per gram of pulp and pulp viscosity can increase the energy need to refine the pulp (PATT et al., 2006; GOMIDE et al., 2005). On the other hand, the residual xylans on the cellulose fibres improve the beatability of pulps and less energy is need in refining (BHADURI et al., 1995; SILVA et al., 2011). The high viscosity is probably due to a less damage during pulping and preservation of the fiber length in tension fibers. These characteristics contributed to the slightly higher fiber properties of fibers from tension wood.

Genomics studies of tension wood formation have determined the existence of differentially expressed genes during tension wood formation some of them related with lignin biosynthesis (PILATE et al., 2004). With this knowledge and the impact of tension wood in pulp production, future studies for genetic improvement of the species could be performed and directed toward the generation of genotypes with features typical of tension wood but maintaining the adequate stem form and volumetric production of wood.

5. CONCLUSION

Results obtained showed that tension wood presented chemical differences in the content of xylans and lignin content and structure, that caused a decrease in alkali consumption, increase pulp strength properties and caused similar bleaching performance as compared with pulps from opposite wood. However, if the tension wood is not abundant in a eucalyptus plantation, the benefits observed could be negligible when processing large volumes of wood.

6. ACKNOWLEDGEMENTS

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