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Selection criteria of *Eucalyptus globulus* Labill. for production of chemithermomechanical pulps (CTMP)

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Abstract

Utilization of rapidly growing trees, such as eucalypts, for high-yield mechanical pulps is limited by low brightness owing to high contents of alkali and neutral extractives. Wood supply problems have developed in many areas of the world and new sources of high-yield pulp are needed. Ten *Eucalyptus globulus* trees were selected from two plantation sites to evaluate suitability as raw material for high-quality and high-yield pulp. Chemithermomechanical pulp (CTMP) was prepared from tree chips pretreated with sodium sulfite prior to refining. Characteristics of the CTMP were correlated with chemical and physical properties of the wood. There was a linear relationship between the content of alcohol-benzene extractives in wood and CTMP brightness. Klason lignin content in wood was inversely correlated with pulp sheet density, which is an important characteristic affecting the physical properties of pulp. The content of alkali extractives were inversely correlated with pulp yields. Color reversion was tested by exposing CTMP sheets to heat and light. Heat-induced yellowing of CTMP was of a low level and satisfied requirements for printing paper. The rate of yellowing was inversely associated with extractives and can be reduced by antioxidants.

Keywords: brightness; chemithermomechanical pulp (CTMP); color reversion; *Eucalyptus globulus*; heat-induced discoloration; light-induced discoloration.

Introduction

Numerous eucalypt plantations have been established in tropical and subtropical regions. *Eucalyptus globulus* is an extremely fast-growing tree in hot areas with high rainfall, and it has a typical rotation period of 8–10 years. This species is well suited to kraft pulping because of its low lignin and high cellulose content. Plantations of *E. globulus* are therefore expected to be a satisfactory solution to wood supply problems in large areas of the world. However, more efficient utilization of eucalyptus wood is necessary as a raw material for high-quality and high-yield mechanical pulp (Hillis 1991).

Eucalyptus trees are known to have variable characteristics depending on growing conditions and genetic variation (Zobel 1981). Moreover, the chemical characteristics of *E. globulus* vary widely among individual trees, and through this the properties of mechanical pulps are more affected than chemical pulps. To grow plantations of *E. globulus* for chemithermomechanical pulp (CTMP), several individual trees should be selected from forest stands as seed sources. Wood sampling by means of increment borer facilitates the analysis of a large amount of trees, as felling is not necessary. Therefore, correlations between characteristics of CTMP and the chemical characteristics of wood will be helpful for selection without having to fell individual trees.

The requirements of CTMP for printing paper are high. Good physical properties and brightness stability after exposure to light and heat are necessary (Janson and Forssakahl 1989; Leary 1994). The yellowing is caused by the oxidation of lignins and phenols in extractives that are distributed in the cell walls of pulp fibers (Johnson 1989). CTMPs contained nearly the same amount of these compounds as wood; thus, the yellowing tendency of CTMP and wood is also similar. For CTMP production, wood chips are treated. Sulfite solutions at various pH levels have been used for this purpose in the course of which lignins are sulfonated. The subsequent swelling decreases damage during refining. Additionally, the sulfonation of the lignin is believed to be advantageous in maintaining higher brightness and in increasing interfiber bonding in the pulp.

In this study, ten *E. globulus* trees were selected for CTMP preparation on a laboratory scale. The physical and optical characteristics of the CTMP were then assessed and correlated with the chemical properties of the wood source. The yellowing of the CTMP by exposure to heat and light was measured.

Experimental methods

Materials

Ten *E. globulus* trees grown under the same silviculture conditions (9.5 years old, trunks at 7–18 cm diameter at breast height) were selected from two plantation sites (A: Manjimup, B: Albany) in Western Australia. The climates of the areas were moderate, with an annual maximum temperature of 20°C and a minimum of 10°C. The mean annual rainfall was 1069 mm at site A and 850 mm at site B. Wood chips from the trees were prepared with a laboratory chipper. An average chip was 25–30 mm long, 15–20 mm wide and 2–3 mm thick.

CTMP production

Eucalyptus wood chips were impregnated with 5% sodium sulfite solution (pH 9.8) at 70°C for 1 h in a pretreatment step prior to refining. The chips were preheated for 5 min at 135°C and were then defibrated in a laboratory Asplund type D defibrator as the first refining. A second refining was performed with a PFI mill. The pulp consistency was 20% and the clearance was 0.5 mm. Refined pulp was screened with a laboratory flat screen with an 8-cut plate.

Characterization

Chemical characteristics of wood and physical characteristics of CTMP were analyzed according to the following Tappi standards (alcohol-benzene extraction; Tappi T 204 cm-07, 1% sodium hydroxide extraction; Tappi T 212 om-07, physical testing of pulp hand sheets; T 220 sp-06, brightness; T 452, other optical properties; Tappi T 1214 sp-07).

Sulfonic acid group content of CTMP

Sulfonic acid groups formed by sulfonation in lignin molecules during the impregnation and refining of pulp fibers were measured by a conductometric method proposed by Katz et al. (1984).

Color reversion

Before light irradiation and heat treatment, CTMPs were washed with 1% HCl solution and deionized water to avoid the effect of metal ion on yellowing. Hand sheets from CTMPs were irradiated with light of 365 nm from 10 min to 60 min at 20°C in a quarts vessel with inlet and outlet cock. A mercury-tungsten light source was applied. The size of the quarts vessel was 15 mm in height, 65 mm in diameter and 50 mm×50 mm in paper size. Air was supplied continuously to the vessel and the brightness of the sheets before and after treatment was measured. The photo-yellowing index ΔBL60 was calculated using the equation: $\Delta BL60 = 100(B0\text{-}B60)/B0$, where $B0$ is the brightness of CTMP before UV irradiation and B60 is the brightness of CTMP after UV irradiation.

Decreasing rate of CTMP sheets brightness were calculated by using the following equation before and after 24 h heating at 105°C in 0% humidity: $\Delta BH24 = 100(B0\text{-}BH24)/B0$, where $B0$ is the brightness of CTMP before heating and BH24 is the brightness of CTMP after 24 h heating.

Results and discussion

Physical characteristics of CTMP and relationship to wood characteristics

Physical and chemical characteristics of 10 *E. globulus* trees from two plantation sites are presented in Table 1. Each tree had low lignin content and high alkali extractives content, which was similar to many other tropical tree species. In general, there were no substantial differences in chemical characteristics, other than lignin content (Student's t-test, $P<0.05$), between the two sites. CTMP produced from the 10 *E. globulus* trees was of high quality (tensile index and brightness) with high yields of over 85%, as shown in Table 2. Generally, mechanical pulps produced from tropical hardwood species have lower brightness than those from hardwoods from temperate zones, because of their high content of alkali and neutral extractives (Higgins et al. 1977). However, the results of this study demonstrate that high brightness CTMP can be produced from *E. globulus* by impregnation with sodium sulfite. In the first refining stage, reddish

Table 1 Physical and chemical properties of *E. globulus* trees used in the study.

Tree no.	Density (kg/m³)	Extractives (%)	Alkali extractives (%)	Holocellulose (%)	α-Cellulose (%)	Hemicellulose (%)	Lignin (%)
A1	495	2.0	17.3	85.2	44.8	40.3	18.0
A2	527	1.9	16.4	85.0	46.8	38.2	18.7
A3	562	3.2	17.6	86.7	46.7	40.1	17.7
A4	547	3.7	16.0	86.4	44.9	41.4	17.5
A5	563	3.6	16.2	86.2	45.1	40.9	16.1
B1	582	3.8	16.7	86.7	47.2	39.4	17.0
B2	509	6.8	18.2	85.1	45.1	39.9	16.4
B3	532	3.5	16.0	87.7	48.3	39.4	15.4
B4	502	2.8	16.4	86.9	46.7	40.2	16.7
B5	529	5.4	17.7	85.3	46.3	39.8	16.3

[a]Pretreated with 5% Na_2SO_3 at pH 9.9, 70°C for 1 h.

Table 2 Physical properties of CTMP of *E. globulus*.

Tree no.	Yield (%)	Lignin (%)	Density (g/cm³)	Tensile index (Nm/g)	Brightness ISO (%)	$SO3^-$ (mM/kg)
A1	86.6	17.0	0.328	4.970	65.2	19.3
A2	88.2	18.0	0.308	3.940	65.9	11.2
A3	86.3	16.1	0.325	2.400	64.9	24.3
A4	88.8	17.0	0.319	3.040	64.9	10.1
A5	87.9	15.3	0.323	3.880	63.8	7.4
B1	87.3	16.1	0.350	5.000	60.3	4.6
B2	84.5	15.2	0.368	5.630	55.9	4.0
B3	90.1	15.4	0.367	6.650	65.9	9.3
B4	88.4	15.9	0.339	4.780	66.5	15.3
B5	85.7	14.3	0.343	6.230	59.8	8.8

materials were dissolved from the pulp fibers by the sodium sulfite solution, and a high brightness pulp was obtained after washing with water. Sodium sulfite is a sulfonation reagent (Aziz 1995). Some other types of chromophores may also react with sulfite and may have reductively altered their chemical structure. The possibility of this effect is supported by the results illustrated in Figure 1, CTMP with small amounts of sulfonic acid groups, under 10 mM kg^{-1}, had lower brightness values.

Large variations in pulp quality were found among individual trees at each plantation site, whereas there was no significant difference in physical characteristics of CTMP, other than tensile index (Student's t-test, $P<0.01$), between the two plantation sites. Pulp yield and brightness level varied over a broad range at site B, and the tensile index was different for CTMP prepared from each tree at site A. These characteristics of CTMP were correlated with the chemical characteristics of eucalyptus wood. Wood extractives contents were highly correlated with CTMP brightness ($R=-0.903$, $P<0.01$), as shown in Figure 2a. As mentioned above, some phenols in extractives were dissolved at pre-impregnation with sodium sulfite. Remaining extractives in CTMP may consist mainly of fats and terpenoids. Some of these substances are helpful as antioxidants. Therefore, we hypothesize that the extractives prevented the oxidation of lignin during refining, as the pulp was heated while exposed to air, and thus maintained the high brightness of the pulp.

This correlation provides a basis for selecting individual trees as pollen donors to establish plantations that can supply commercial amounts of raw materials for high-brightness CTMP. The lignin content of eucalypt wood was found to have no correlation with CTMP brightness (Figure 2b). Figure 3a shows a high correlation ($R=-0.937$, $P<0.01$) between pulp yield and alkali extractives. As mentioned above, some fraction of the alkali extractives was dissolved in the first refining stage as a dark red colored substance. For this reason, the decrease in pulp yield was found to depend on the alkali extractives content of the wood. From the data of Tables 1 and 2, it can be deduced that a small fraction of the lignin was eluted from the wood in one or both of the impregnation process steps and the subsequent refining. Consequently, no correlation was found between pulp yield and wood lignin content (Figure 3b). This retention

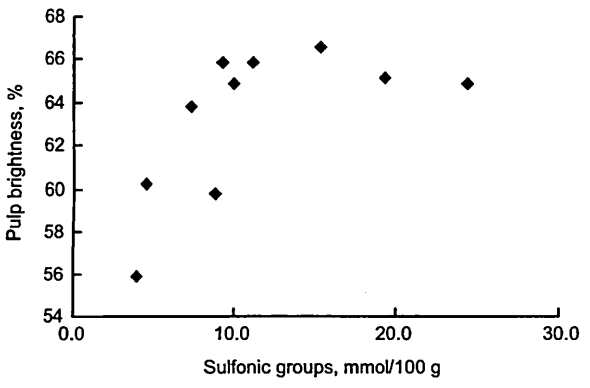

Figure 1 Relationship between brightness and content of sulfonic group of eucalyptus CTMPs.

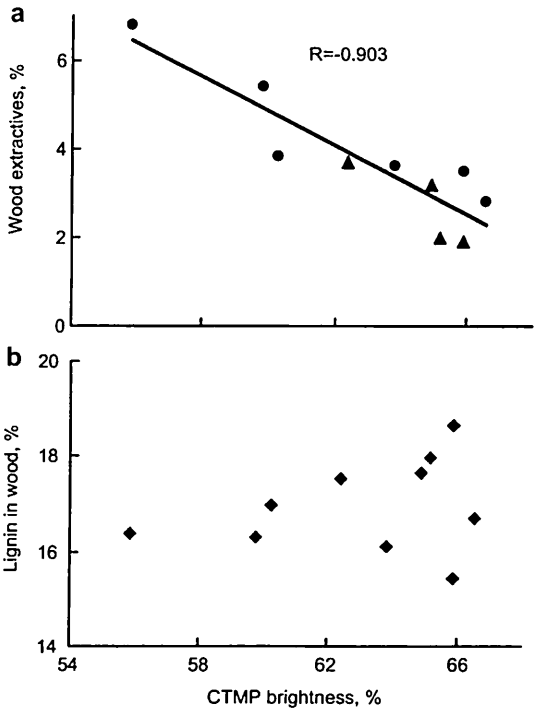

Figure 2 Relationship between CTMP brightness and contents of extractives (a) and lignins in eucalyptus wood (b), respectively.

of lignin in the pulp fiber renders possible the CTMP production with high yield.

The lignin content of wood samples also correlated with sheet density of CTMP ($R=-0.746$, $P<0.05$), which affects many physical characteristics of pulp sheets (see Figure 4). In the course of refining wood chips treated with sulfite solutions, fibers are separated between the middle lamellae and mechanical damage of pulp fibers are minimized. Therefore, the surfaces of the CTMP fibers were covered with middle lamella, which has high lignin content. Lignin impedes interfiber bonding in the pulp sheets by hydrophobicity. It is well known that interfiber bonding is strongly related to sheet density. Fur-

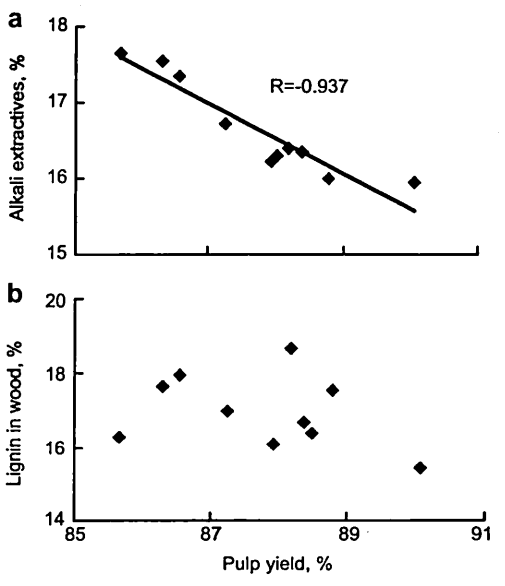

Figure 3 Relationship between alkali-extractives and pulp yield (a), and between wood lignin and pulp yield (b).

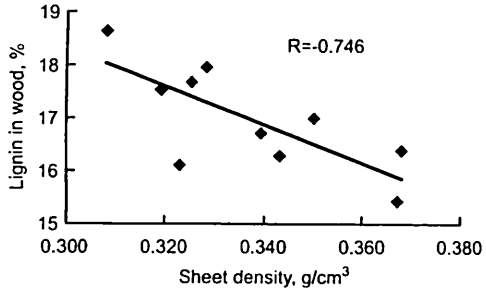

Figure 4 Relationship between wood lignin and sheet density.

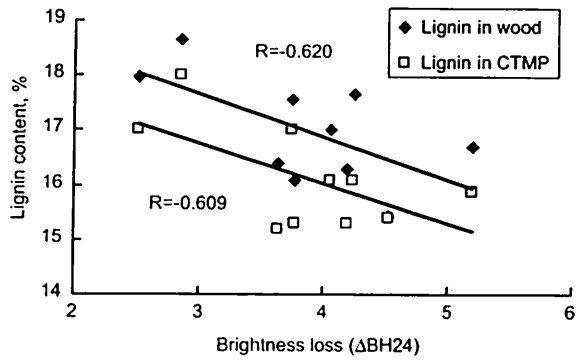

Figure 5 Relationship between wood lignin and heat-induced yellowing.

thermore, lignin in the fiber inhibits delamination of the fiber cell wall and decreases the swelling. As a result, the flexibility of fiber is low and the interfiber bonding in the pulp sheet is diminished. CTMPs obtained from wood with high lignin content will be of low quality.

Optical characteristics of CTMP and relationship to wood characteristics

Unbleached or bleached mechanical pulps undergo a loss in brightness as a result of exposure to light or heat. This is a serious problem and is a major impediment to the wider use of these pulps in various grades of printing paper. Despite intensive research, so far no broadly applicable, economically feasible method has been found for increasing brightness stability of CTMPs comparable to that of bleached chemical pulp.

It is generally believed that lignin reactions are the main cause of brightness instability in such pulps. Both the light-induced and heat-induced oxidation of lignin was studied using appropriate lignin model compounds (Gierer and Lin 1972; Gellerstedt and Pettersson 1977; Gellerstedt et al. 1983; Castellan et al. 1990). These studies revealed that phenoxyl radicals are formed and subsequently transformed into quinones by the action of oxygen and water. Therefore, in the case of light-induced oxidation, wood with low lignin content, such as eucalyptus wood, is expected to produce high-yield pulp with good brightness stability. Results of color reversion of CTMP by heating at 105°C in a dry oven for 24 h are listed in Table 3. Color stabilities of pulps are indicated as $\Delta BH24$ by calculation from $B0$ and $BH24$.

Our ultimate goal is to produce CTMP with 70% brightness without bleaching and to utilize unbleached CTMP from *E. globulus* for the production of printing paper. For this reason, unbleached CTMP was examined for color reversion by heat treatment. $\Delta BH24$ of CTMP produced from trees at site A were slightly lower than at site B. The brightness loss ($\Delta BH24$) of the CTMP were correlated with lignin content of the wood and CTMP, as shown in Figure 5. There was an inverse correlation of $\Delta BH24$ with lignin content of the wood ($R=-0.620$, $P<0.1$) and CTMP ($R=-0.609$, $P<0.1$). From this result, it can be interpreted that sulfonated lignin in *E. globulus* CTMP has low tendency to heat-induced color reversion.

In the sulfonation experiments of wood with neutral sulfite solution, it was found that sulfonic groups are introduced to aliphatic unsaturated groups, as well as stilbene structures of lignin (Gellerstedt 1976). Thus, sulfonation decreased the number of chromophores in lignins which are stabilized against auto-oxidation which occurs during heat-induced color reversion. This is the reason why the $\Delta BH24$ values are low for all CTMPs measured in this study.

High resistance to light discoloration is important for printing paper of higher quality. The results of the light-induced color reversion of CTMP by irradiation of UV at 365 nm are listed in Table 3. Most CTMPs were sensitive to light irradiation, and the brightness of CTMP sheets decreased. The stability of the pulp brightness ($\Delta BL60$) to UV irradiation was inversely correlated with extractives ($R=-0.774$, $P<0.01$), as illustrated in Figure 6a. In the case of light-induced color reversion of CTMP, the aging stability of pulp can be explained by antioxidant activity of wood extractives. It is well known that extractives of eucalypt trees contain large amounts of resin and polyphenols, such as tannin (Bland 1985; Pereira 1988; Ona

Table 3 Heat-induced and light-induced yellowing of CTMP.

Tree no.	ISO brightness (%)			ISO brightness (%) after UV irradiation				
	Control	Heat 24 h	$\Delta BH24$*	0 min	10 min	30 min	60 min	$\Delta BL60$**
A1	65.16	63.52	2.52	65.16	60.54	56.23	52.09	20.02
A2	65.86	63.98	2.85	65.86	61.48	56.58	52.76	19.89
A3	64.87	62.12	4.24	64.87	59.63	56.07	52.11	19.67
A4	62.37	60.04	3.74	62.37	58.15	54.84	50.81	18.53
A5	63.80	61.39	3.78	63.80	59.14	55.23	51.46	19.34
B1	60.28	57.83	4.06	60.28	55.51	51.99	48.42	19.67
B2	55.87	53.84	3.63	55.87	52.67	48.78	46.24	17.24
B3	65.85	64.87	4.53	67.85	63.34	58.52	54.57	19.57
B4	66.54	65.08	5.20	68.54	61.64	57.57	53.20	22.38
B5	59.80	57.29	4.20	59.80	55.62	52.08	49.05	17.98

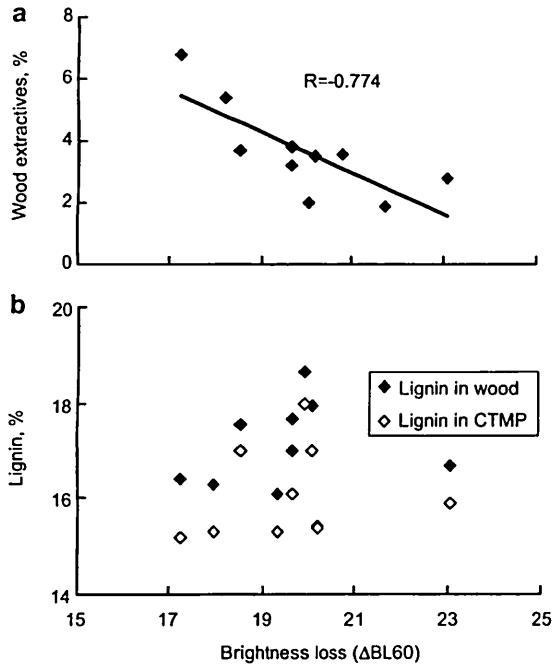

Figure 6 Relationship between wood extractives and light-induced yellowing (a), and between wood lignin and light-induced yellowing (b).

et al. 1997; Gutiérrez et al. 1999; Speranza et al. 2002). These substances act as antioxidants by scavenging oxygen radical (Ariga and Hamano 1990; daSilva et al. 1991; Aruoma et al. 1993; Rice-Evans et al. 1996; Hagerman et al. 1998). Wallis and Wearne (1999) have demonstrated that wood extractives are difficult to dissolve by pulping under acidic or neutral conditions. In this study, we treated wood chips with sodium sulfite solution at pH 9.8 under mild conditions. It is likely that most extractives of eucalypt wood remained in CTMP with the exception of some low molecular phenols.

To evaluate the antioxidant effects, we attempted to measure the extractives content of CTMP, but this was unsuccessful owing to contamination of soluble lignin in extractives during alcohol-benzene extraction of CTMP. For example, the CTMP extractives value from A1 is 3.1 (wood extractives is 2.0) and that from A2 is 2.5 (wood extractives value is 1.9). Some lignin molecules were subjected to hydrolysis and sulfonation during the impregnation process and subsequent refining. As a result, the molecular weight of lignin decreased, and it became soluble in the alcohol-benzene solution. Lignin content in the wood and CTMP was expected to correlate with the light-yellowing index ($\Delta BL60$). However, as shown in Figure 6b, there was no correlation ($R=0.090$ for wood and $R=0.196$ for CTMP).

Many researchers have tried to utilize antioxidants or photostabilizers to control photo-yellowing of printing paper (Nolan 1945; Reineck and Lewis 1945; Cole et al. 1987; Johnson 1989; Li and Ragauskas 2000). However, the chemicals were too expensive in industrial processing. In addition, chromophores of lignins in pulp were modified to inhibit photo-yellowing. Chemical modification would be commercially preferable in view of the high costs of chemicals. From these results, the content of extractives is an important selection criterion for breeding eucalypt trees for the production of high-brightness CTMP, because extractives have antioxidant properties.

Conclusion

Physical and optical characteristics of CTMP produced from *E. globulus* differed greatly among individual trees. The brightness of some unbleached CTMP samples was high (over 60% of ISO brightness) and was highly stable even after exposure to heat. The heat yellowing performances of CTMP correlated with lignin content of the wood source. Wood extractives content was correlated to CTMP brightness and to photostability. Lignin content of wood exhibited correlation to CTMP sheet density. Alkali extractives of wood were bleached in the pulping process and decreased pulp yield. These correlations provide criteria for the selection of pollen donors. The chemical mechanisms of photo-yellowing of CTMPs, as well as the role of extractives in this process, still need to be investigated.

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