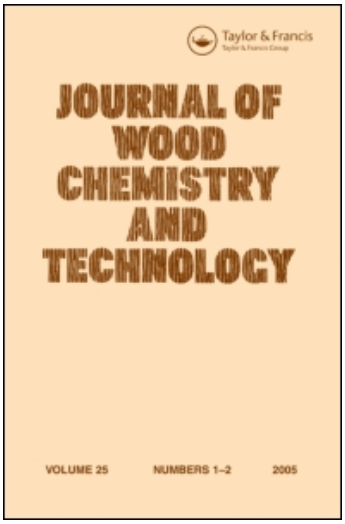

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Bio-Chemimechanical Pulps from *Eucalyptus grandis*: Strength Properties, Bleaching, and Brightness Stability

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Bio-Chemimechanical Pulps from *Eucalyptus grandis*: Strength Properties, Bleaching, and Brightness Stability

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Abstract: *Eucalyptus grandis* wood chips were treated with the white-rot fungus *Ceriporiopsis subvermispora* in a 100-L bioreactor for 15 days. The treatment was characteristic of a selective biodelignification (7.6 ± 0.2% and 0.3 ± 0.2% of lignin and glucan losses, respectively) with concomitant extractive removal (17.7 ± 0.2%). Biotreated samples and non-inoculated controls were pre-cooked in alkaline sulfite and post-refined in a Jokro mill. The biotreated pulps fibrillated more rapidly and contained lower amounts of rejects than the control. To achieve a freeness of 400 mL, the control pulp required 125 min of beating, whereas the biopulp required

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only 95 min, a reduction of 24%. Unbleached biopulps had better strength properties than control pulps because higher tensile indexes were obtained for the entire range of tear indexes. Bleaching with 8% hydrogen peroxide increased the brightness of these pulps by 17 points. At low peroxide loads, the brightness increase for biopulps was lower than for the control pulps. Still, the bleachability of both pulps was similar for peroxide loads higher than 2%. After a two-stage H_2O_2-bleaching sequence, final brightnesses for the control and biopulps were $59.7 \pm 0.8\%$ and $60.5 \pm 0.4\%$, respectively. Brightness stability of the bleached control and bio-CMP pulps to photo and thermal aging were very similar.

Keywords: *Eucalyptus grandis*, *Ceriporiopsis subvermispora*, biopulping, bleaching, yellowing

INTRODUCTION

Mechanical pulping produces pulps with superior optical properties and high yields. However, high electrical energy consumption and low pulp strength are drawbacks of this process. Some improvements, such as chemimechanical (CMP) and chemithermomechanical (CTMP) pulping have been implemented in mechanical pulping to produce stronger fibers.[1] Biopulping is a more recent variation in which wood chips are treated with a ligninolytic fungus prior to mechanical pulping.[2] Most biopulping research has been focused on refiner mechanical pulping (RMP). The treatment of hardwoods or softwoods with selected white-rot fungi provides at least 30% energy savings during RMP and significant pulp-strength improvements.[3–5] Thermomechanical (TMP), CTMP, and chemical pulping of biotreated wood samples have received attention only in the last few years.[2,6–9] Considering that TMP and CTMP are well-established RMP improvements, evaluation of the response of biotreated wood samples to these processes is desirable.

Bio-TMP and bio-CTMP have been evaluated for pulp production from the softwoods, loblolly pine, and lodgepole pine.[2] In these cases, energy savings during refining were similar to the ones observed in the RMP process. However, the pulps' strength properties depended on the fungal strain used in the biotreatment and the pulping process. In general, pulp strength properties were diminished in bio-TMP and increased in bio-CTMP. A recent study evaluating bio-TMP of the hardwood *Eucalyptus grandis* presented encouraging results concerning both energy savings and pulp-strength improvements.[6] Data compiled on biopulping clearly demonstrate that the wood and the fungal species as well as pre- and post-refining conditions affect the overall process, and optimization for each case is necessary.

Another key aspect is that biopulps are darker than control pulps.[2,3,6,10,11] In a pioneering study on the bleachability and brightness stability of biopulps, Sykes[11] reported that although bio-RMP aspen pulps

have a lower initial brightness, high brightness levels could be attained during bleaching using only slightly higher hydrogen peroxide dosages. Control RMP pulps required 2% H_2O_2 to increase brightness from 62% to 80%, whereas 3% H_2O_2 was necessary to improve bio-RMP pulps from 50% to 76%. Brightness stability for the aspen biopulps was only slightly lower than for the control pulps.

In this article we report the results of a study in which biotreated *Eucalyptus grandis* was used to produce alkaline sulfite-CMP pulps. Strength properties, bleachability, and brightness stability of the bio-CMP pulps were determined.

MATERIALS AND METHODS

Wood Preparation

Eucalyptus grandis wood chips (2.5 × 1.5 × 0.2 cm) were supplied by a Brazilian pulp mill. The wood chips were air-dried to a moisture content of 10% and stored. Prior to biotreatment, the wood chips were immersed in water for 16 h after which the water was drained. The moist chips were sterilized (121°C, 30 min) and cooled. After sterilization, the moisture content of the wood chips was 55%. Wood weight loss due to soaking and sterilization was 1.2%. This value was subtracted from the total weight losses measured after the biotreatments. The chemical composition of these wood chips (determined by acid hydrolysis) was 49 ± 2% glucan, 16.3 ± 0.8% polyoses, 27.3 ± 0.2% total lignin, and 2.8 ± 0.1% ethanol-soluble extractives.

Fungus, Inoculum Preparation, and Wood Biodegradation

Cultures of the white-rot fungus *Ceriporiopsis subvermispora* SS3 were grown on 20 mL of solid medium ($24\,g \cdot L^{-1}$ of potato-dextrose broth, $7\,g \cdot L^{-1}$ of yeast extract and $20\,g \cdot L^{-1}$ of agar) for 10 days at 27°C. After fungal growth, the plates were stored at 4°C. In 2-L Erlenmeyer flasks, 200 mL of sterilized liquid medium composed of potato-dextrose broth ($24\,g \cdot L^{-1}$) and yeast extract ($7\,g \cdot L^{-1}$) were inoculated with 20 discs (8 mm in diameter) of *C. subvermispora*-precultured solid medium. These liquid cultures were maintained unshaken for 10 days at 27°C. The grown mycelium mat was filtered and washed with 300 mL of sterilized water. Washed mycelia obtained from several cultures were blended with 100 mL of sterilized water in 3 cycles of 15 s. Blended mycelia were used to inoculate the wood chips contained in 2-L bioreactors. Seven bioreactors were loaded with 100 g of sterilized wood chips and a volume of fungal suspension corresponding to 500 mg of mycelia/kg of wood (both on a dry basis). The bioreactors were closed, shaken by hand and stored at 27°C for 30 days.

Humidified air passing through a 0.2-μm membrane was supplied to the bioreactors throughout biodegradation (2.3 L of air/h). Colonized wood chips were used as inoculum seeds for the scale-up biodegradation experiments. In an aseptic room, 14 kg of sterilized wood chips were mixed with 700 g of precolonized wood chips (amount of inoculum seeds for 5% w/w) and added to a 100-L stainless-steel bioreactor (approximately 45 cm diameter × 65 cm height). During bioreactor loading, 12 cylindrical containers (5-cm diameter × 9-cm height) made of 0.84 mm steel screen were filled with 25 g of sterilized wood chips and inserted at three pre-defined heights of the bioreactor (bottom, center, and near the bioreactor's top). The bioreactor was closed and stored at 27°C for 15 days. Humidified air passing through a 0.2-μm membrane was supplied to the bioreactor throughout the biodegradation (60 L of air/h).

After biotreatment, the bioreactor was opened and the wood chips were washed with water to remove the superficial mycelium. The bulk of the biotreated wood chips were used in the pulping experiments. The wood chips contained in the cylindrical containers were also washed. These decayed wood chips were dried in the air, their moisture was determined, and the calculated initial and final dry weights were used to determine weight losses. Component loss calculations were based on the weight loss values and the chemical compositions of the decayed wood chips recovered from these cylindrical containers.

Chemimechanical Pulping of the Wood Samples

Chemimechanical pulping of untreated and biotreated eucalyptus wood chips were performed in two steps. The first was a mild cook of wood chips (150 g od) in alkaline sulfite liquor (900 mL; 10% Na_2SO_3 and 5% NaOH, od wood basis) at 121°C (1 atm) for 2 h. The second step was fiberization of the cooked wood chips and refining of the fibers in a Jokro mill (Regmed, Brazil). Each pot of the Jokro mill was filled with 25 g od of cooked wood chips and 250 mL of fresh cooking liquor. The material was fiberized/refined for periods varying from 30 to 135 min to obtain pulps with freenesses in the range of 800 to 200 mL. After refining, the pulps were washed with water and screened in a 0.15 mm slot screen. The screened pulp was centrifuged, giving a pulp consistency of approximately 30%. A sample of this pulp was used for moisture determination. Rejects retained on the 0.15-mm screen were dried at 105°C to constant weight. Total yield was calculated as the sum of accepted pulp plus the dry weight of the rejects. Freenesses of the screened pulps were determined by TAPPI standard T 227 om-85. Standard deviations for the measurements of freeness were lower than 20 mL. Yield, reject, and freeness data are presented in Table 1.

Handsheets with a basis weight of 150 g cm^{-2} were prepared with CMP pulps obtained from both untreated and 15-day biotreated wood chips

Table 1. Yield, rejects, and freeness of refined CMP pulps from untreated and biotreated *Eucalyptus grandis* chips

	Untreated *E. grandis*			Biotread *E. grandis*		
Beating time (min)	Total yield (%)	Rejects (%)	Freeness (mL)	Total yield (%)	Rejects (%)	Freeness (mL)
30	86.8	33.6	708	84.9	30.4	702
60	84.3	10.0	638	83.2	7.4	622
90	83.4	2.8	504	79.5	1.2	424
120	80.8	0	432	77.6	0	323
135	78.3	0	323	76.0	0	253

following TAPPI Standard T 205 sp-95. Handsheet strength properties were evaluated using TAPPI standard methods: tensile index (T 494 om-01), tear index (T 414 om-98) and burst index (T 403 om-02). Standard deviations for the measurements of strength properties were within the values reported in TAPPI standards (5%, 4.2%, and 22% for tensile, tear and burst indexes, respectively).

Chemical Analysis of Wood and Pulp Samples

Undecayed and biotreated eucalyptus wood chips were milled in a knife mill to pass through a 0.5-mm screen. Approximately 1.5 g of milled sample (in duplicate) was extracted with 95% ethanol for 6 h in a Soxhlet apparatus. The amount of ethanol-soluble extractives was determined on the basis of the dry weight of extracted and unextracted milled samples.

Ethanol-extracted wood samples or unscreened CMP pulps (also milled to 0.5 mm screen but not extracted) were hydrolyzed with 72% (w/w) sulfuric acid at 30°C for 1 h (300 mg of wood and 3 mL of acid) as previously described by Ferraz et al.[12] The acid was diluted to a final concentration of 3% (w/w) with the addition of 79 mL of water, and the mixture was autoclaved at 121°C for 1 h. The residual material was cooled and filtered through a porous glass filter number 3. Solids were dried to constant weight at 105°C and measured as insoluble lignin. To calculate the soluble lignin concentration in the aqueous fraction the absorbance at 205 nm was measured and the value of $105\,L \cdot g^{-1} \cdot cm^{-1}$ was used as the absorptivity of soluble lignin.[13] The concentrations of monomeric sugars in the soluble fraction were determined by HPLC using a BIORAD HPX-87H column at 45°C, eluted at 0.6 mL/min with 5 mM of sulfuric acid.[12] The values reported are averages of duplicate hydrolyses. The deviations from the average values were lower than 3%.

Bleaching of Chemimechanical Pulps

Bleaching experiments were carried out with CMP pulps refined for 120-min as described before. Prior to bleaching, the pulps were pretreated with diethylenetriaminepentaacetic acid (DTPA) as described by Robert and Daneault.[14] Pulps suspensions at 0.3% consistency and pH 6.0–6.5 were shaken for 30 min at 60°C with DTPA (0.4 g of DTPA/100 g of pulp on dry basis). Treated pulps were filtered, washed, and dried at room temperature. The treatment with DTPA was repeated until the filtrate presented no absorption from 380 to 700 nm.

Bleaching conditions (peroxide load, consistency, temperature and time) were defined following a 2^4 experimental design.[15] The level adjusted for each variable is shown in Table 2. In a typical bleaching experiment, 3 g of pulp (dry basis) was treated with bleach liquor containing hydrogen peroxide, magnesium sulfate (0.05 g/100 g of pulp), and sufficient 6 M NaOH to adjust the pH to 11. Bleaching reactions were performed in sealed polyethylene bags immersed in a water bath at the desired temperatures (Table 2). After bleaching, the pH of the pulp suspension was adjusted to 6.0 with 6 M HCl, and the pulp was filtered and thoroughly washed with distilled water.

Brightness Determination, Photo-, and Thermo-Aging Tests

Pulp handsheets were prepared according to TAPPI standard T218sp-97. Handsheet brightness was measured with Photovolt equipment (model 577). After brightness measurement, handsheets were exposed to UV/visible light for photo aging.[16] Light exposure of the handsheets was performed inside a vented chamber equipped with a 250 W mercury lamp (Philips) from which the glass capsule was removed. Four handsheets were simultaneously exposed to light at a 10-cm distance from the lamp. Brightness of photo-aged handsheets was monitored for 7 h at one-hour time intervals. Thermal aging of another set of handsheets was determined following TAPPI standard T453 pm-85.

RESULTS AND DISCUSSION

Biodegradation of *Eucalyptus grandis* by *Ceriporiopsis subvermispora*

E. grandis wood chips were treated for 15 days with *C. subvermispora* in a 100-L bioreactor. Wood colonization was homogeneous as indicated by formation of a mycelium net on the wood chips contained at the bottom, in the center, and near the top of the bioreactor. Weight losses of the wood

Table 2. Brightness levels and variable effects on bleaching of CMP pulps prepared from untreated and biotreated *Eucalyptus grandis* wood chips based on the 2^4 experimental design

	Variables and levels				Brightness (% ISO)	
Test no.	H_2O_2 charge (% on dry basis)	Consistency (% on dry basis)	Temperature (°C)	Time (h)	Control pulp	Biopulp
1	0	5	40	0.5	36.3	36.4
2	4	5	40	0.5	44.1	41.5
3	0	10	40	0.5	36.7	35.2
4	4	10	40	0.5	41.8	51.5
5	0	5	60	0.5	38.9	39.9
6	4	5	60	0.5	45.8	47.6
7	0	10	60	0.5	41.5	33.9
8	4	10	60	0.5	47.1	52.5
9	0	5	40	3	35.1	36.8
10	4	5	40	3	44.7	45.3
11	0	10	40	3	35.8	33.3
12	4	10	40	3	47.8	48.1
13	0	5	60	3	35.9	32.6
14	4	5	60	3	50.7	51.0
15	0	10	60	3	39.8	34.0
16	4	10	60	3	52.5	54.0
Effect of the variables[a]						
Average					42.1	42.7
H_2O_2 charge					13.6	12.7
Consistency					1.4	2.4
Temperature					2.2	3.1
Time					−0.4	−1.4
H_2O_2 charge × Consistency					3.7	2.8
H_2O_2 charge × Temperature					2.5	1.6
H_2O_2 charge × Time					1.7	2.7
Consistency × Temperature					−0.6	0.3
Consistency × Time					−0.5	−1.4
Temperature × Time					−0.1	−1.1
H_2O_2 charge × Consistency × Temperature					−0.6	−1.6
H_2O_2 charge × Consistency × Time					−1.7	−0.8
H_2O_2 charge × Temperature × Time					1.3	2.2
Consistency × Temperature × Time					1.9	0.9
H_2O_2 charge × Consistency × Temperature × Time					−0.5	0.4

[a]Based on *t*-test, only the values higher than 3.5 are significant at the 95% confidence level. Brightness values obtained for three replicates at the central point (2% H_2O_2, 7.5% consistency, 50°C and 1.75 h of reaction time) were: 42.2%, 44.0%, and 44.6% for control pulps and 47.3%, 46.4%, and 48.2% for biopulps.

chips sampled in these areas of the bioreactor were 1.6 ± 0.1%, 1.4 ± 0.2%, and 1.4 ± 0.2% for the bottom, center, and top, respectively. Wood colonization was also characterized by brightening of the wood chips' surfaces. Average values for weight and component losses were: weight, 1.5 ± 0.2%; glucan, 0.3 ± 0.2%; polyoses, 0.9 ± 0.2%; lignin, 7.6 ± 0.2%; and ethanol-soluble extractives, 17.7 ± 0.2%. This pattern of selective biodelignification and extensive degradation of wood extractives by *C. subvermispora* confirms previous reports.[5,8,17]

Chemimechanical Pulping of Untreated and Biotreated *E. grandis*

Chemimechanical pulping of untreated and 15-day biotreated *E. grandis* wood chips was performed in two steps: a mild cooking in alkaline sulfite liquor followed by fiberization and refining of the cooked wood chips in a Jokro mill. Total pulp yield, amount of rejected pulp, and freeness pulp levels are presented in Table 1. Total pulp yield varied from 87% to 76% as a function of the beating time. The biopulps presented lower total pulp yield than the control pulps. On the other hand, the biopulps fibrillated more rapidly and contained lower amounts of rejects. Rapid fibrillation of the biopulps could represent increases in the process throughput or energy savings during the refining step, because a reduced beating time is necessary to achieve a desired freeness level. For example, to achieve 400 mL of freeness, the control pulp required 125 min of beating, whereas the biopulp required only 95 min (24% reduction). Akhtar et al.,[2] using loblolly pine treated with *C. subvermispora*, also reported that the refining steps were enhanced during production of alkaline hydrogen peroxide- and alkaline sodium sulfite-CTMP pulps, resulting in energy savings and pulp-strength improvements during the processes.

Strength properties of pulps prepared from 15-day biotreated samples are shown in Figure 1. Biopulp tensile indexes increased significantly (+24.3% and +6.2% at 630 mL and 430 mL freeness, respectively), whereas tear strength improved only for pulps with freeness values below 500 mL (+25% at 320 mL of freeness). Burst indexes were similar for both pulps. Tensile versus tear graphs (Figure 1D) clearly indicated that the biopulps presented better strength properties than the control pulps, because higher tensile indexes were obtained for the entire range of tear indexes.

In a recent study, Scott et al.[6] evaluated a two-stage TMP process for biotreated *E. grandis*. In this case, the energy savings for producing pulps with 400 mL of freeness was 17%, and bio-TMP pulps presented doubled tensile and tear indexes when compared to control pulps. This improved strength of biopulps allows the preparation of pulp blends for tissue paper production using 80% bio-TMP and only 20% bleached kraft pulp instead of the typical 50%–50% used with conventional TMP pulps. In the case of CMP pulps of *E. grandis*, increases in the strength properties were not as

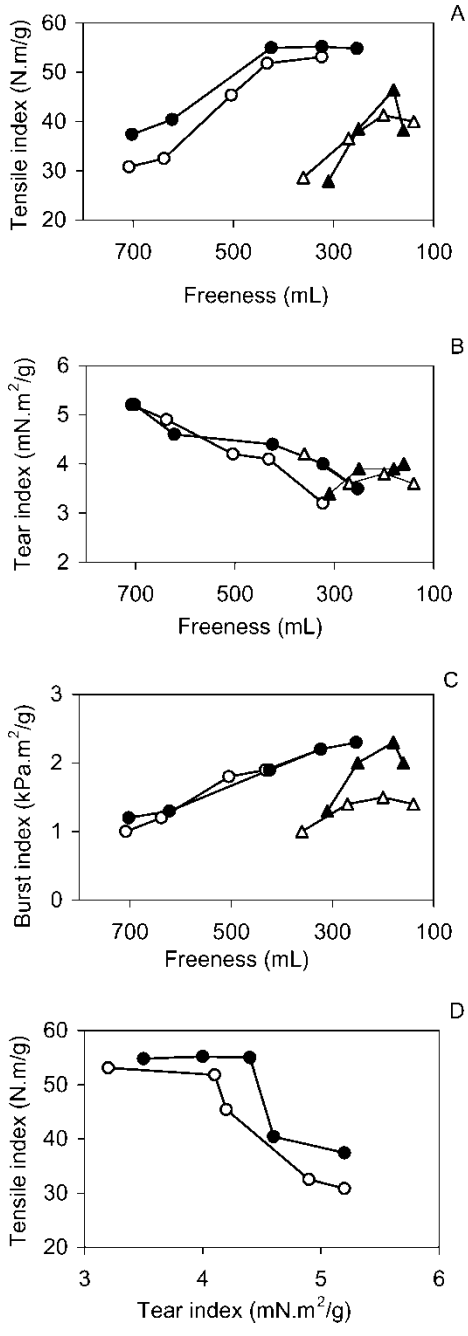

Figure 1. (A)–(D) Strength properties of CMP pulps prepared from untreated (open symbols) and biotreated (filled symbols) *Eucalyptus grandis* chips. Circles are unbleached pulps. Triangles are hydrogen peroxide-bleached pulps.

significant as observed for TMP. For comparison purposes, bio-CMP pulps with 400 mL of freeness presented tensile and tear indexes improved by 6% and 13%, respectively (Figure 1).

Bleachability and Brightness Stability of CMP Pulps

Unbleached biopulps were slightly darker than the control pulps (initial brightness values of 22.7% and 23.7%, respectively). Initial brightness values of such pulps were very low in comparison to commercially available unbleached alkaline sulfite-CTMP pulps from *E. grandis* (56% ISO for pulps from a Brazilian pulp mill). Inefficient washing of the laboratory-prepared pulps as well as autoclaving of the wood chips could contribute to the initial low brightness levels.[11] Treatment with DTPA and alkaline washing without hydrogen peroxide increased the initial pulp brightness to 36.7% and 35.2% for the control and biopulps, respectively. The two-step pulping process also produces pulp darkening because mechanical refining is preceded by a long period of impregnation in autoclave. Under industrial conditions, impregnation and refining are very quick due to the use of efficient disk refiners. Still, laboratory-prepared control and biopulps are suitable for comparison purposes because complete control of the process variables can be assured in their preparation.

The bleachability of the pulps was evaluated according to a standard 2^4-experimental design. The variables evaluated were peroxide load, consistency, temperature, and bleaching time (Table 2). Analysis of variance performed on the data from Table 2 indicated that, under the conditions used in this work, only the peroxide load significantly affected the final brightness of both the control and biopulps.

To further evaluate the effect of the peroxide load on pulp brightness, additional bleaching experiments were conducted with increasingly peroxide dosages. Although both the control and biopulps had low initial brightness values, a single bleaching step with 8% hydrogen peroxide was enough to increase the brightness values by 17 points (Figure 2). At low peroxide load, the brightness increase for biopulps was lower than for the control pulps. Bleaching with 1% H_2O_2 increased the brightness of the control pulps 8 points whereas the biopulps gained only 4 points. On the other hand, the bleachability of both pulps was similar for peroxide loads higher than 2%, reaching maximal brightness values of 52% ISO with 8% H_2O_2. Higher peroxide levels had a negligible effect on final pulp brightness. One-stage-bleached pulps were washed and submitted to a second peroxide bleaching step with 8% H_2O_2, where the maximal brightness achieved was $59.7 \pm 0.8\%$ and $60.5 \pm 0.4\%$ for control and biopulps, respectively. Mechanical pulps of *E. grandis* wood are usually difficult to bleach to high brightness levels. For example, bleaching of industrially prepared TMP pulps (using a similar optimization procedure) showed that

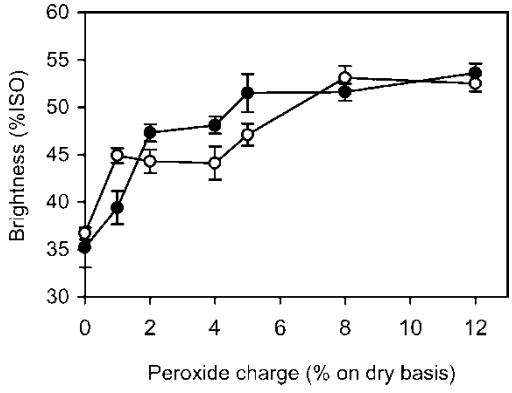

Figure 2. Bleaching response of CMP pulps prepared from untreated (open circles) and biotreated (filled circles) *Eucalyptus grandis* wood chips.

hydrogen peroxide loads of 8% were necessary to increase initial brightness values from 59% to 75–80%. Similar pulps prepared from aspen wood gained 15 points in brightness, reaching 75–80% with a load of only 3% hydrogen peroxide.[11]

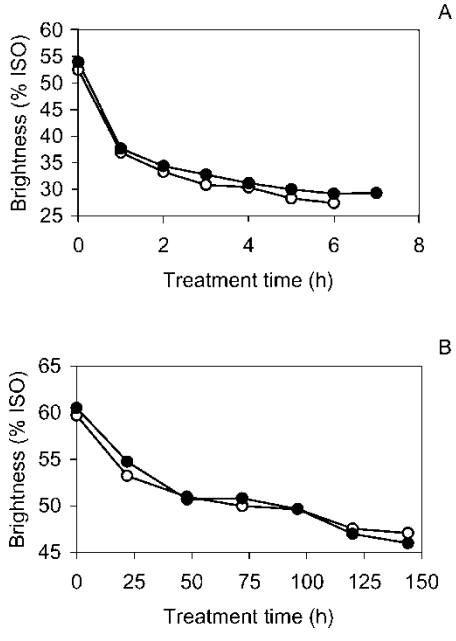

Figure 3. Photo- (A) and thermal-reversion (B) of brightness in H_2O_2-bleached CMP pulps prepared from untreated (open circles) and biotreated (filled circles) *Eucalyptus grandis* wood chips.

After two-stage bleaching, the strength properties of the control and biopulps were almost the same whereas burst indexes were slightly higher in biopulps (Figure 1).

The brightness stability of bleached CMP and bio-CMP pulps was determined by submitting samples of different initial brightness levels to accelerated thermal- and photo-aging tests.[11] Figure 3 illustrates accelerated photo and thermal brightness reversions versus time. On exposing the pulp handsheets for one hour to light, the brightness values rapidly decreased, stabilizing after approximately 4 h (Figure 3A). Thermal reversion of brightness for these pulps was slower but also very substantial during the first day (Figure 3B). Figure 4 shows the brightness reversion (brightness points lost) plotted against initial brightness levels of several of the control and biopulps exposed to photo- and thermal- aging tests. This graphic method has been reported as an appropriate manner for comparing the brightness stability of different pulps regardless of the initial brightness.[11] The data in

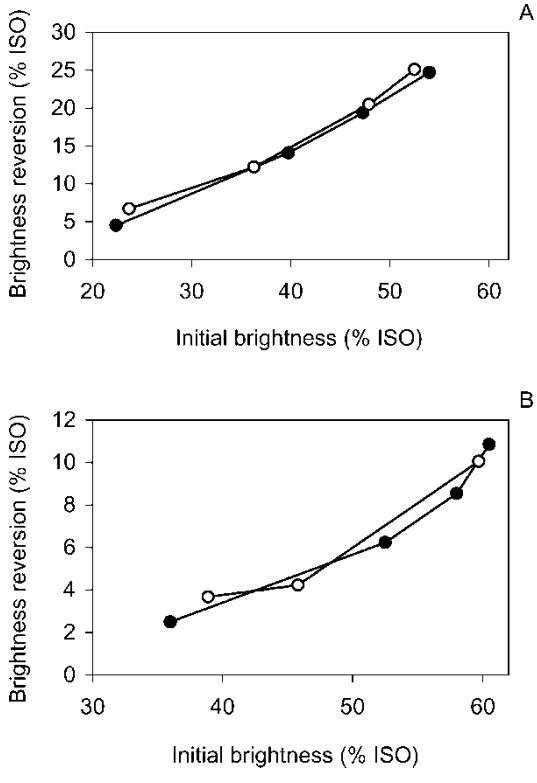

Figure 4. Brightness reversion as a function of initial brightness levels in H_2O_2-bleached CMP pulps prepared from untreated (open circles) and biotreated (filled circles) *Eucalyptus grandis* chips. Photo- (A) and thermal-reversion (B).

Figure 4 indicates that the brightness stability for bleached control and bio-CMP pulps to photo and thermal aging was very similar.

CONCLUSIONS

Biotreatment of *E. grandis* chips with *C. subvermispora* prior to CMP reduced the beating time required to prepare pulps of a desired freeness level. For pulps with 400 mL of freeness, beating time was reduced by 24%. Strength properties of unbleached biopulps were improved in comparison to control pulps. Bio-CMP pulps were slightly darker than control-CMP pulps. However, for hydrogen peroxide dosages higher than 2%, the bleachability of both pulps was very similar, where the maximal brightness achieved after one bleaching stage was 52%. An additional bleaching step improved pulp brightness to approximately 60%. The brightness stability of the bleached bio-CMP and control-CMP pulps to photo and thermal reversion was almost identical.

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