High-brightness CMP from Eucalyptus globulus *using a nitric acid pretreatment*

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ABSTRACT

A high-brightness, high-yield chemimechanical pulp was obtained from Eucalyptus globulus *using low-environmental-impact chemical reagents. The pulping chemicals were nitric acid and sodium hydroxide, and the bleaching chemical was hydrogen peroxide. Chips were impregnated for 24 h in nitric acid, cooked under variable conditions, washed, impregnated with soda for 24 h, cooked again, rewashed, defibrated, refined, screened, and finally bleached under variable conditions. Under the optimal pulping conditions identified in this study, pulp strength was not especially high (tensile strength 2.04 km, tear strength 3.9 mN·m²/g), but the ease of bleaching and final pulp brightness were impressive enough (light-scattering coefficient 49.3 m²/kg, brightness 81.3% Elrepho) to warrant further research.*

RECENT EFFORTS TO DEVELOP new high-yield pulping processes are largely driven by a desire to conserve fiber resources and reduce pollution. Much of the current research in this field is focusing on sulfur-free chemimechanical pulping (CMP) processes.

The nitric acid pulping process is receiving renewed attention because it offers a potentially low-environmental-impact alternative to conventional processes. Pulping with nitric acid has been studied since the beginning of the last century, with the first known study of nitric acid's action on wood made by Braconnot in 1833 *(1)*. Subsequent development of the nitric acid process has been slow, although it was advanced by research in the early 1960s *(2, 3)*. Research from that era also showed that the effluent from this process contains fewer pollutants than the widely used sulfur-based processes *(2, 4)*. Recent works suggest that effluent from the nitric acid process could be used as a fertilizer by mixing precipitated nitrolignin with the effluent from the soda extraction *(5–11)*. However, further research will be needed to verify these claims. Another reason for the renewed interest in nitric acid as a pulping chemical is its low cost and the availability of new acid-resistant construction materials *(12, 13)*. Finally, the process is inherently attractive because of its low cooking temperatures and short cooking times. Handling risks are reduced by using dilute solutions of acid and alkali.

This paper examines a chemimechanical pulping method for *Eucalyptus globulus* using nitric acid and sodium hydroxide in the cooking stages and hydrogen peroxide in the bleaching stage. *Eucalyptus globulus*, the best known of the eucalypts, is a very fast-growing hardwood species with white wood and a pale yellowish-brown duramen. These trees grow to a height of 40–55 m and produce more wood per hectare than the natural forest. Moreover, the wood is not the only useful part of the tree. The leaves are used in medicine; the flowers are a source of honey; and the trees are widely used in landscaping to recuperate eroded soils *(14–19)*.

EXPERIMENTAL

All experiments were carried out using 14-year-old *E. globulus* wood measuring 30–35 cm in diameter. The wood was chipped and classified by size, with most of the work done using chips between 0.5 in. and 0.75 in. (12.7 mm and 19.1 mm). Figure 1 is a diagram representing the experimental pulping process.

Chip impregnation

Chips were impregnated for 24 hours in nitric acid at dosages ranging from zero to 7% based on the ovendry (o.d.) weight of the wood chips. The chips were then cooked, washed, and impregnated again for 24 hours in sodium hydroxide at dosages ranging from zero to 7% based on chip dry weight. A total of 41 trials was carried out under a range of conditions.

Pulping

Pulping was carried out in small plastic flasks using a 3.5:1 liquor-to-wood ratio under various conditions of temperature and time for both the acid and the soda treatments. Heat was applied by means of a controlled-temperature water bath. After the nitric acid and soda treatments, the cooked chips were washed with warm water and defibrated in a vessel with rotating blades and toothed bed plate at a consistency of 20%. The defibrated chips were then refined in a disc refiner (Sprout–Waldron, disc pattern D2A 507) at 8% consistency. Finally, the refined pulps were screened on a plate with 0.15-mm slots.

Bleaching

Bleaching was carried out on 10-g pulp samples in plastic bags. Again, heat was applied in a controlled-temperature water bath. Before bleaching, the pulp was treated with 0.4%

DTPA (diethylenetriamine pentaacetic acid) at room temperature for 30 min and 5% consistency. The pulp was bleached using various quantities of hydrogen peroxide in one or two steps. The bleaching liquor contained 0.15% magnesium sulfate, 4% sodium silicate (40° Bé), and enough sodium hydroxide to achieve pH 11. Bleaching was carried out at 10% consistency at 60°C, 70°C, or 90°C. Bleaching time was 120 min in each step. Bleached pulps were washed first with SO_2 water and then with warm water (50°C).

RESULTS AND DISCUSSION

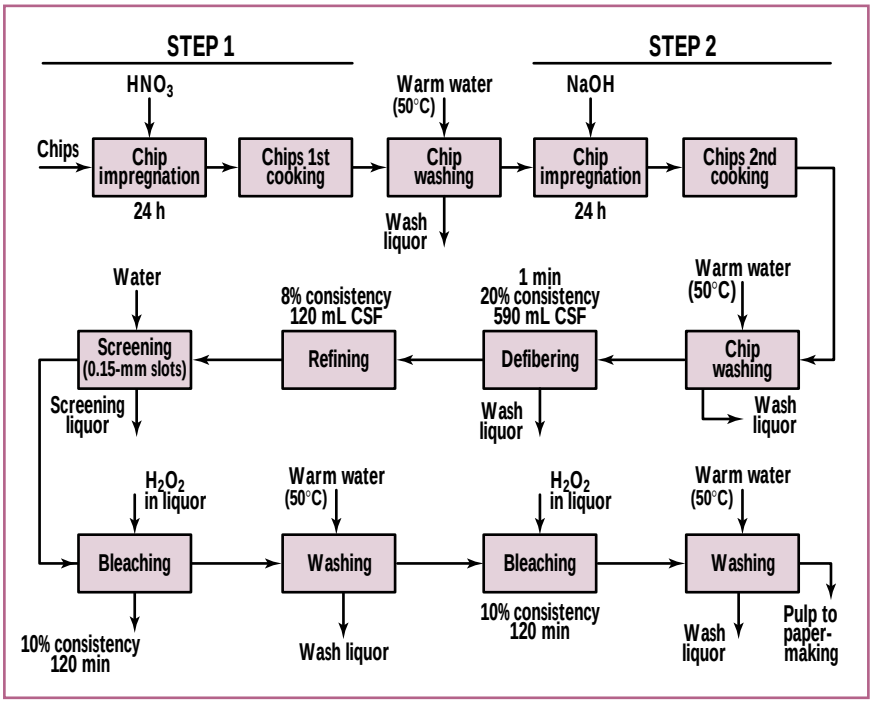
Nitric acid action on carbohydrates

Pulp yields were higher than 84%, with an average of about 93% after the two cooking stages. Pulp yield decreased slightly with increasing concentration of nitric acid. The yield loss is due to the presence of nitrogen compounds (e.g., NO_2 and HNO_2) in the dilute nitric acid solution *(20, 21)*. Such compounds have oxidation and nitration action on carbohydrates, producing nitrogen compounds and causing esterification. The functional groups attacked by oxidation are the aldehyde and hydroxyl groups, forming aldehyde, ketone, and carboxylic groups *(22–24)*. In strong acid conditions, as in the NO_2–H_2O system, some cellulose and hemicellulose is expected to be hydrolyzed *(25)*. This results in the rupture of cellulose B-0,4 glucosidic linkages *(26, 27)*, which contributes to depolymerization of the carbohydrates during the pretreatment stage and to a loss in pulp viscosity when acid concentration is increased *(28–33)*. The pentosan chemical reactions also are influenced by the equilibrium in the nitric acid solution, which depends on concentration, temperature, and the presence of other substances *(24)*.

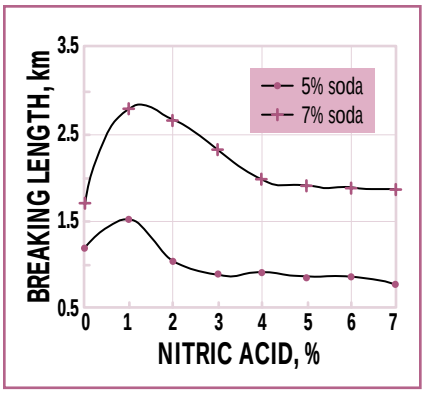
Nitric acid action on lignin

The reduction of pulp yield in the presence of nitric acid is attributed only to the acid hydrolysis of lignin linkages and not to the by-product nitrous acid dissociation *(26)*. Very complex reactions are expected between the NO_2–H_2O system and lignin *(34)*. Some nitration can take place in the presence of nitrous acid, and the nitrous group can then be oxidized to form nitro groups. Moreover, some nitration reactions can occur because of the formation of nitronium ion in the system *(34)*, forming nitrolignin as a product *(35)*. The nitrolignin formation mechanism consists of two irreversible first-order reactions. The formed nitrolignin is attacked by the nitric acid, forming acid-soluble compounds *(24, 35)*. Another reaction is oxidation, but as already stated, lignin is slow to nitrate, since it is very susceptible to oxidation, even in dilute nitric acid *(25, 36)*, so its interaction with the nitric acid is always an oxinitration reaction *(37)*.

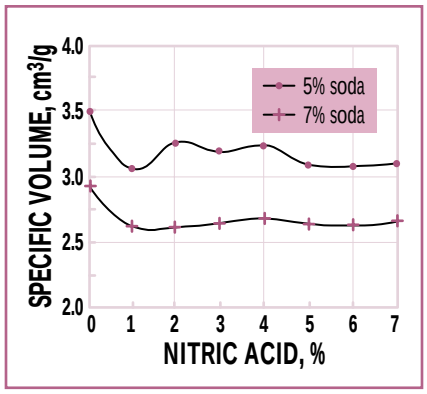
Phenolic groups can be oxidized by the nitric acid to form quinonoid



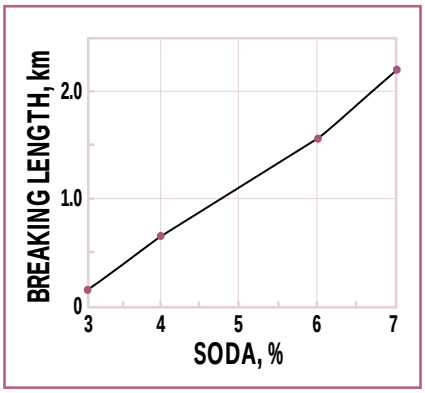
1. Experimental pulping process



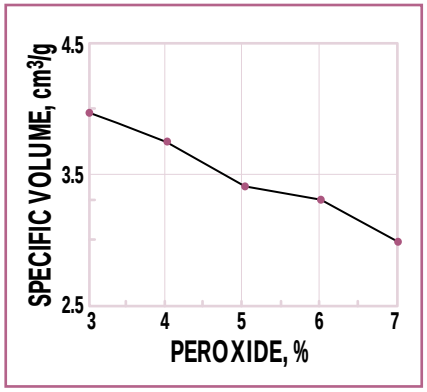
2. Effect of nitric acid dosage (Step 1) on breaking length at two levels of soda addition (Step 2). Pulps were cooked for 60 min at 80°C in each step.



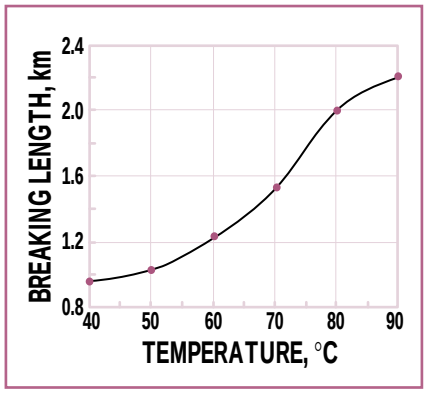
3. Effect of nitric acid dosage (Step 1) on specific volume at two levels of soda addition (Step 2). Pulps were cooked for 60 min at 80°C in each step.



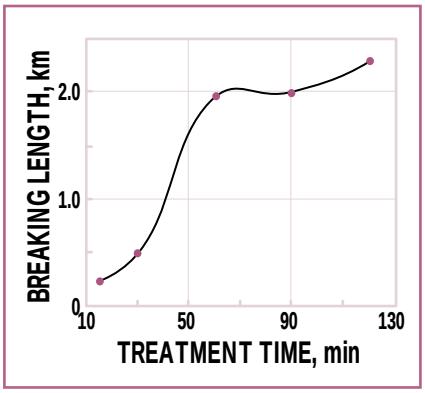
4. Effect of soda concentration (Step 2) on breaking length for chips treated with 1% nitric acid (Step 1). Pulps were cooked for 60 min at 80°C in each step.



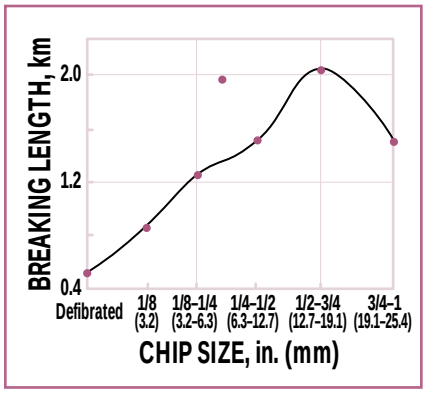
5. Effect of soda concentration (Step 2) on specific volume for pulps treated with 1% nitric acid (Step 1). Pulps were cooked for 60 min at 80°C in each step.



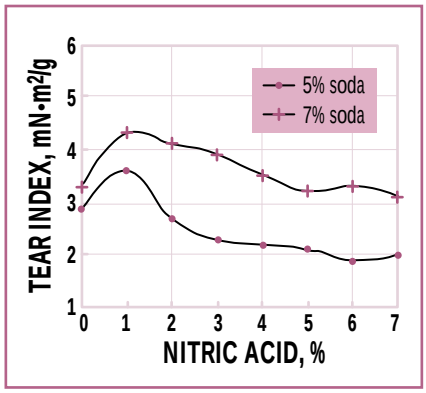
6. Effect of cooking temperature on breaking length. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min in each step.



7. Effect of cooking time on breaking length. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked at 80°C in each step.



8. Effect of chip size on breaking length. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min at 80°C in each step.



9. Effect of nitric acid dosage (Step 1) on tear index at two levels of soda addition (Step 2). Pulps were cooked for 60 min at 80°C in each step.

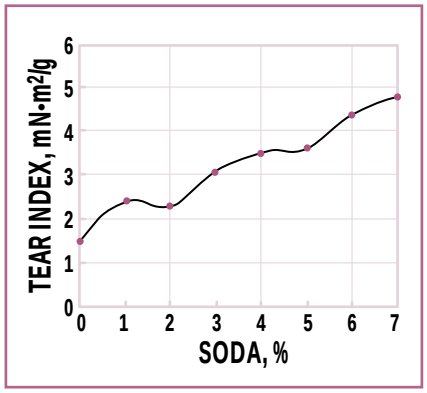
products and subsequently converted to carboxylic acids by opening the rings *(34)*. Also, displacement reactions, condensation reactions, and the rupture of ether alkyl–aryl linkages can occur in lateral chains *(34)*. The lignin chemical reactions are influenced by the chemical equilibrium that prevails in the nitric acid solution, which depends on concentration, temperature, and the presence of other substances *(24)*. Free phenols, which are present in both softwood lignin and hardwood lignin, react swiftly under mild conditions, whereas the completely etherified compound requires harsher conditions to react *(38)*. It is significant that only one of the completely etherified compounds, similar to hardwood lignin, shows an appreciable degree of demethylation *(38)* that is parallel to delignification *(39)*. This suggests that the ethyl ether lignin linkages for hardwood can be broken faster than those for softwood *(38)*. It has been reported that hardwoods can be pulped faster than softwoods with nitric and nitrous acid mixtures *(40)*.

Soda action on carbohydrates

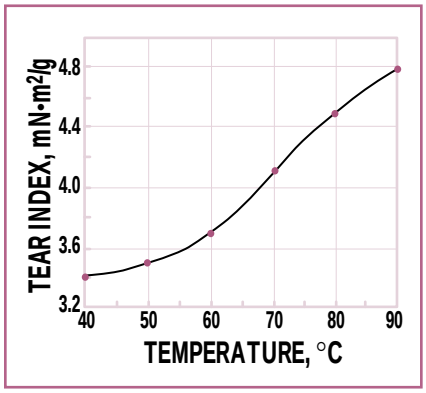
Pulp yield decreased with increasing soda content (over the range zero to 7%) in the second step, probably because high base concentrations produce pulps with a low glucomannan and xylan content *(41)*. It has also been reported that soda concentrations in the range 0.8–19 g/L—which corresponds to the range used in the present work—have a bigger influence on the speed of pentosan extraction than on delignification *(42)*, which explains the considerable yield reduction that was observed.

Soda action on lignin

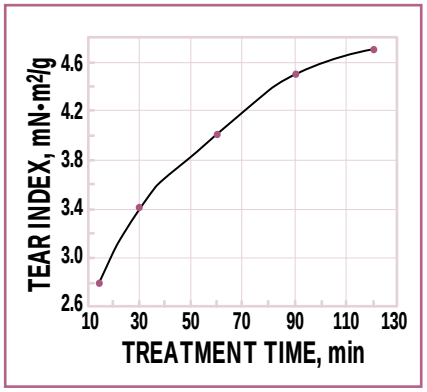
While nitric acid contributes to pulp-yield reduction through pentosan extraction, nitric acid also favors delignification and the formation of alkali-soluble lignin during the pretreatment stage *(29, 42)*. Furthermore, many of the lignin degradation products that are not soluble in the nitric acid liquor, such as phenol or quinone, are alkali soluble *(43)*. These degradation products undergo subsequent degradation, especially the nitro groups, resulting



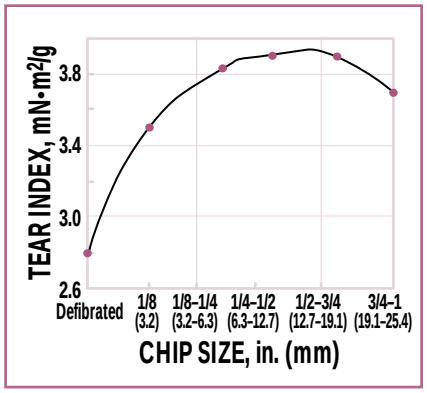
10. Effect of soda concentration (Step 2) on tear index for chips treated with 1% nitric acid (Step 1). Pulps were cooked for 60 min at 80°C in each step.



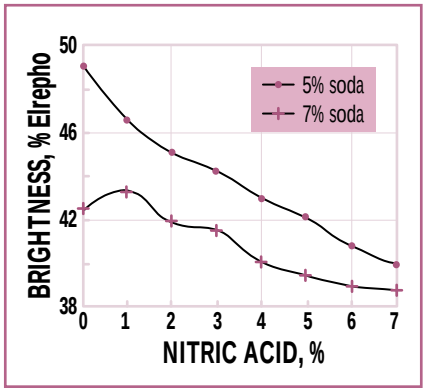
11. Effect of cooking temperature on tear index. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min in each step.



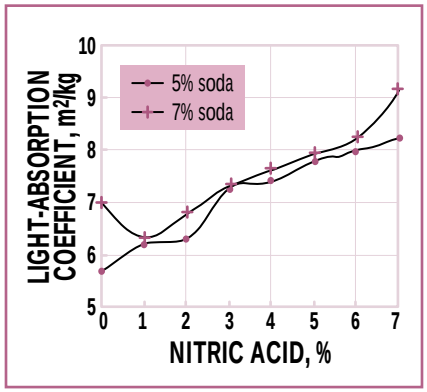
12. Effect of cooking time on tear index. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked at 80°C in each step.



13. Effect of chip size on tear index. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min at 80°C in each step.



14. Effect of nitric acid dosage (Step 1) on pulp brightness at two levels of soda addition (Step 2). Pulps were cooked for 60 min at 80°C in each step.



15. Effect of nitric acid dosage (Step 1) on light-absorption coefficient at two levels of soda addition (Step 2). Pulps were cooked for 60 min at 80°C in each step.

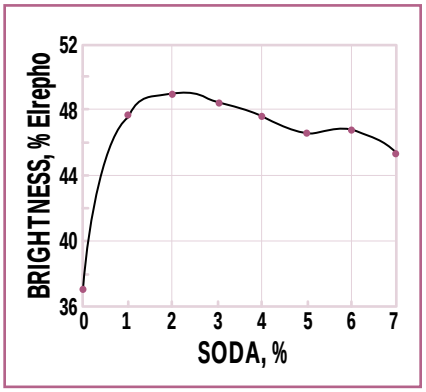
in the possible formation of hydroxinitroquinone configurations and xylan and xylose hydrolyzation *(22)*. The presence of nitro groups in the phenyl nuclei and in the carboxyl groups increases the rupture of ether linkages and the dissociation of alkali-soluble lignin, which serves to counteract condensation reactions *(27)*.

Tensile strength

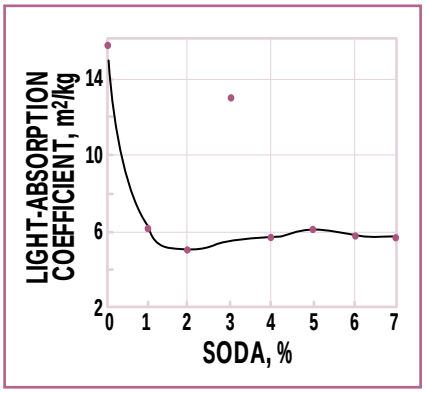
Figure 2 shows tensile strength over the range of nitric acid applied in the first cooking stage (Step 1) for pulps treated with either 5% or 7% soda in the second cooking stage (Step 2). The maximum tensile strength at 1% nitric acid corresponds to the maximum reduction in pulp specific volume, as seen in Fig. 3. It is probable that lignin nitration occurs and that this increases with decreasing nitric acid concentration *(24)*. Pulp specific volume increases when the nitric acid quantity is increased from 1%, and tensile strength drops steadily up to 4% nitric acid. At higher levels of nitric acid, tensile strength declines only slightly.

When nitric acid in Step 1 was held at 1% and soda in Step 2 was varied from zero to 7%, tensile strength increased continuously with increasing soda content, as seen in Fig. 4. This is due to delignification and the formation of alkali-soluble lignin during the pretreatment stage *(24, 42, 43)* as well as to the hydrolyzation of xylans to xylose. Pulp specific volume decreased with increasing soda content, as seen in Fig. 5, indicating an increment in the fiber-consolidation capacity.

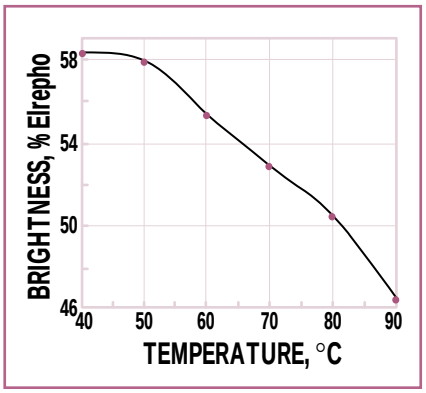
When nitric acid in Step 1 was held at 1% and soda in Step 2 was held at 7%, tensile strength increased with increasing pulping temperature (40°C–90°C in both stages), as seen in Fig. 6. Others have reported that dissolution of lignin and carbohydrates increases with treatment temperature *(31, 39)*. Temperatures between 52°C and 92°C exert a higher influence on the pentosan extraction rate than on the delignification rate *(42)*. It has also been reported that in dilute acid solutions at temperatures under 100°C, lignin



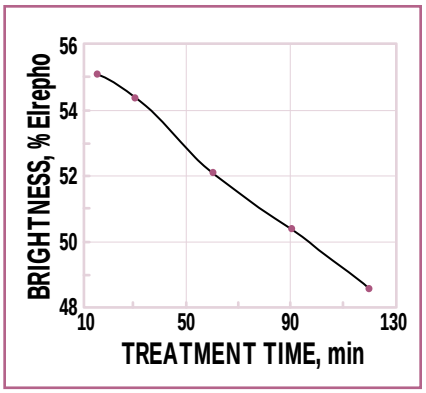
16. Effect of soda concentration (Step 2) on pulp brightness for chips treated with 1% nitric acid (Step 1). Pulps were cooked for 60 min at 80°C in each step.



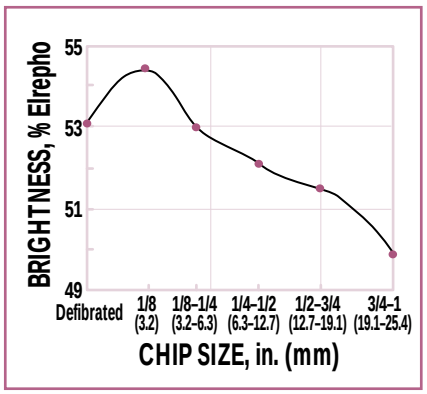
17. Effect of soda concentration (Step 2) on light-absorption coefficient for chips treated with 1% nitric acid (Step 1). Pulps were cooked for 60 min at 80°C in each step.



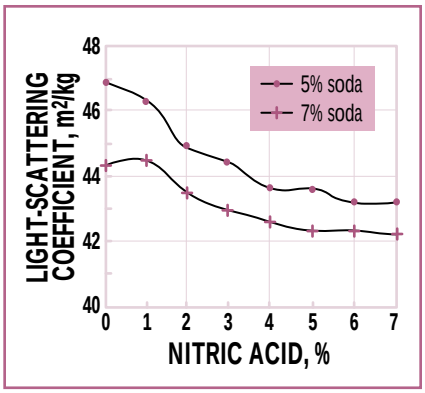
18. Effect of cooking temperature on pulp brightness. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min in each step.



19. Effect of cooking time on pulp brightness. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked at 80°C in each step.



20. Effect of chip size on pulp brightness. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min at 80°C in each step.



21. Effect of nitric acid dosage (Step 1) on light-scattering coefficient at two levels of soda addition (Step 2). Pulps were cooked for 60 min at 80°C in each step.

is nitrated to form nitrolignin, which is immediately attacked by the acid to form acid-soluble compounds *(35)*. This probably reduces the specific volume and increases fiber conformability.

With nitric acid at 1%, soda at 7%, and temperature at 80°C in both pulping steps, tensile strength increased with extended treatment time from 15 min to 120 min, as seen in Fig. 7. It has been reported that pulp delignification in the presence of nitric acid is improved by prolonging the pretreatment time *(26)*, and carbohydrate depolymerization increases if the temperature is increased. At low temperature, the time influence is unimportant *(31)*. Lignin demethylation also increases if the pretreatment time is prolonged *(39)*.

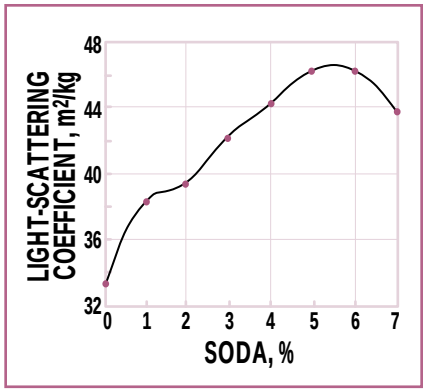
On the other hand, pulp tensile strength declines for chips larger than 0.75 in. (19.1 mm) or smaller than 0.5 in. (12.7 mm), as seen in Fig. 8. The smaller chips suffer too much mechanical damage, while the larger chips resist impregnation by the pulping chemicals.

Tear strength

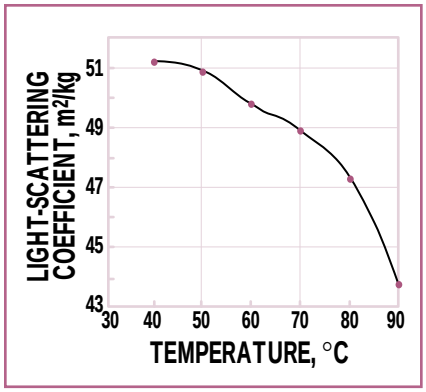
Treatment conditions during Steps 1 and 2 had similar effects on tear strength. Figure 9 shows that 1% nitric acid again produced the highest level of tear strength for pulps treated with 5% or 7% soda in Step 2 (compare with Fig. 2). Similarly, Fig. 10 shows that tear strength of pulps treated with 1% nitric acid increased with increasing soda content in Step 2 (compare with Fig. 4). Tear strength also increased with increasing temperature and treatment time, as seen in Figs. 11 and 12, respectively (compare with Figs. 6 and 7, respectively). The highest tear strength value was obtained with chips between 0.25 in. (6.3 mm) and 0.75 in. (19.1 mm), as seen in Fig. 13 (compare with Fig. 8).

Brightness

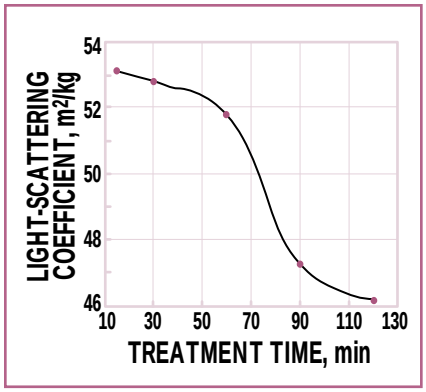
Pulp brightness decreased with increasing nitric acid in Step 1 for



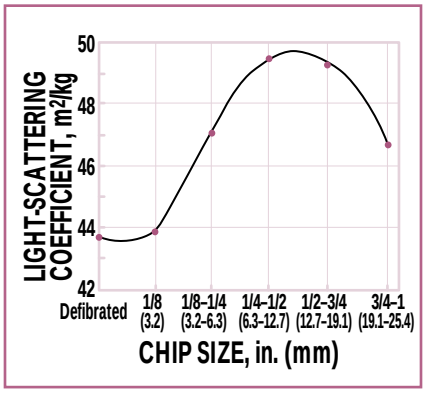
22. Effect of soda concentration (Step 2) on light-scattering coefficient for chips treated with 1% nitric acid (Step 1). Pulps were cooked for 60 min at 80°C in each step.



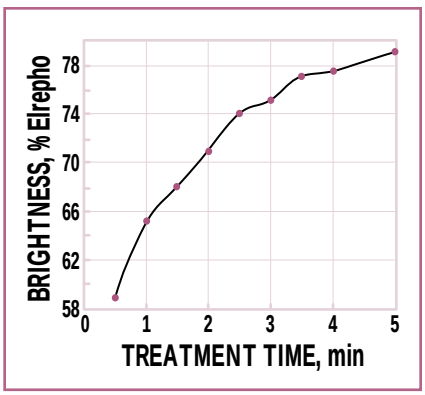
23. Effect of cooking temperature on light-scattering coefficient. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min in each step.



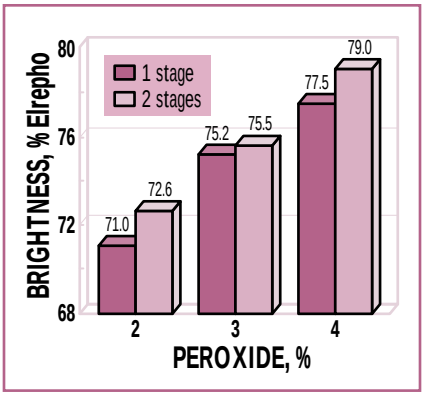
24. Effect of cooking time on light-scattering coefficient. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked at 80°C in each step.



25. Effect of chip size on light-scattering coefficient. Pulps were treated with 1% nitric acid (Step 1) and 7% soda (Step 2) and cooked for 60 min at 80°C in each step.



26. Effect of hydrogen peroxide charge on pulp brightness. Pulps were bleached for 120 min at 70°C in a single stage.

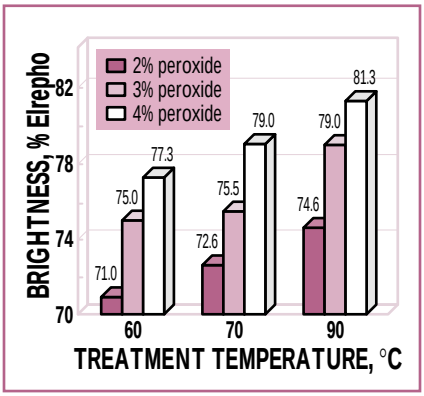


27. Effect of hydrogen peroxide charge on pulp brightness after one or two bleaching stages. Pulps were bleached for 120 min at 70°C in each stage. Peroxide charge for the two-stage sequences was split evenly between both stages.

pulps treated with 5% or 7% soda in Step 2, as seen in Fig. 14. Brightness was much higher when a smaller quantity of soda (5%) was used, although the gap in brightness narrowed at higher dosages of nitric acid in Step 1. However, for the pulps treated with 7% soda in Step 2, brightness increased slightly for the pulp treated with 1% nitric acid in Step 1. This behavior is mirrored by the effect of nitric acid on pulp light-absorption coefficient, as seen in Fig. 15. It has been pointed out that lignin is very susceptible to oxidation, even if it is treated with dilute nitric acid *(25, 36)*. On the other hand, the phenolic groups can be oxidized to quinonoid groups by the nitric acid, and some condensation reactions can occur *(34)*, leading to the pulp darkening.

Figure 16 shows that the addition of 1–3% soda in Step 2 substan-

28. Effect of bleaching temperature on pulp brightness for two-stage bleaching at three levels of peroxide charge. Pulps were bleached for 120 min at 70°C in each stage, and peroxide charges were split evenly between both stages.



Sample no.	HYDROGEN PEROXIDE, wt.% on o.d. pulp 1st step[a]	2nd step[a]	Pulp brightness, % Elrepho[b]	Temperature, °C
1	0.5	…	59.0	70
2	1.0	…	65.0	70
3	1.5	…	68.0	70
4	2.0	…	71.0	70
5	2.5	…	74.0	70
6	3.0	…	75.2	70
7	3.5	…	77.0	70
8	4.0	…	77.5	70
9	5.0	…	79.1	70
10	1.0	1.0	72.6	70
11	1.5	1.5	75.5	70
12	2.0	2.0	79.0	70
13	1.0	1.0	74.6	90
14	1.0	1.0	71.0	60
15	1.5	1.5	79.0	90
16	1.5	1.5	75.0	60
17	2.0	2.0	81.3	90
18	2.0	2.0	77.3	60

[a]Bleaching time, 120 min
[b]Unbleached pulp brightness, 48.6% Elrepho

I. Pulp bleaching conditions using peroxide in one or two steps

tially improved pulp brightness, with higher levels of soda reducing pulp brightness slightly. This behavior is explained by the trend in pulp light-absorption coefficient tendency illustrated in Fig. 17. It has been reported that nitric acid favors delignification and the formation of alkali-soluble lignin during the pretreatment stage *(29, 42)*, which explains the initial brightness increment. However, the presence of nitro groups in the phenyl nuclei and in carboxyl groups can lead to condensation reactions. This would explain the brightness loss with excessive addition of soda.

Increasing the temperature over the range 40°C–90°C during the two cooking steps caused a great brightness loss, as seen in Fig. 18. This is probably because higher temperatures promote condensation reactions. A similar behavior is observed when the treatment time is prolonged, as seen in Fig. 19. The most suitable chip size, from a brightness point of view, is 1/8 in. (3.18 mm), while larger sizes and defibrated chips before the pretreatment stage present a lower unbleached brightness, as seen in Fig. 20.

Light-scattering coefficient

Light-scattering coefficient decreased with increasing nitric acid content during Step 1 for pulps treated with either 5% or 7% soda in Step 2, as seen in Fig. 21. On the other hand, for pulp treated with 1% nitric acid during Step 1, light-scattering coefficient increased with increasing soda quantity in Step 2 up to 5%, after which this property declined, as seen in Fig. 22. Light-scattering coefficient dropped sharply with increasing temperature or time of treatment, as seen in Figs. 23 and 24, respectively. The influence of chip size on the light-scattering coefficient is very irregular, as seen in Fig. 25. Smaller chips are easier to impregnate with the pretreatment reagents than larger chips. However, larger chips suffer less mechanical damage during chipping. In both cases, the fines content in the refined pulp is low, resulting in a low light-scattering coefficient.

Peroxide bleaching

The use of nitric acid in the first cooking step and soda in the second step caused a general decline in pulp brightness, as seen in Figs. 14 and 16, respectively. However, the resultant pulps showed a very good response to hydrogen peroxide bleaching, gaining more than 10 brightness points with the addition of 0.5% peroxide and more than 30 points with 5% peroxide. Figure 26 shows the results for a brownstock pulp of 48.6% Elrepho, which achieved a brightness of 79.1% Elrepho after a 120-min treatment at 70°C. When pulp bleaching is carried out in two steps, the final pulp brightness is improved, as seen in Fig. 27. Brightness also increased with increasing bleaching temperature, as seen in Fig. 28. At 90°C, brightness reached 79% Elrepho with 3% peroxide (1.5% in each bleaching step) and 81.3% with 4% hydrogen peroxide (2% in each step).

Table I shows the pulp bleaching conditions and the final pulp brightness. Table II compares the

	Present work	CSIRO (44)	South China University (45)	Tasmania (46)
Wood species	*E. globulus*	*E. regnans*	*E. leilin* 1	*E. globulus*
Pulping process	HNO_3 + NaOH	Cold NaOH	NaOH + Na_2SO_3	Cold NaOH
Freeness, mL CSF	120	238	115	150
Tensile strength, km	2.04	5.6	3.2	4.4
Tear index, mN·m²/g	3.9	7.1	2.5	3.8
Light-scattering coefficient, m²/kg	49.2	45.7	…	44.9
Unbleached brightness, % Elrepho	48.6	56.2	49.0	50.0
Peroxide bleaching	2 steps	1 step	1 step	1 step
Bleached brightness, % Elrepho	81.3[a]	77.6[b]	65.3[c]	67.2[d]

[a]4% peroxide (2% in each stage), 90°C, 4 h (2 h in each stage)
[b]3% peroxide, 60°C, 1 h
[c]4% peroxide, 60°C, 1 h
[d]2% peroxide, 65°C, 1.5 h

II. Comparison of chemimechanical eucalypt pulps

KEYWORDS
Bibliographies, bleaching, brightness, chemimechanical pulps, cold soda pulping, eucalyptus globulus, *hydrogen peroxide, nitric acid, peroxide bleaching, pretreatment, pulp properties, pulping, sodium hydroxide.*

pulp characteristics of several eucalypt pulps, including the pulps obtained in these laboratory experiments. The pulp obtained with the proposed process is weaker, especially in tensile strength, but bleached pulp brightness was high enough to warrant further studies. Regarding the repeatability of the results presented here, the average variation in pulp characteristics was 11.6%.

CONCLUSIONS

The optimal pulping conditions for the proposed eucalyptus chemimechanical pulping process are 1% nitric acid during the first cooking step and 7% soda in the second step with both cooking steps carried out for 60 min at 80°C.

Pulps obtained in this process showed very good response to the hydrogen peroxide bleaching, gaining more than 10 brightness points with a 0.5% peroxide addition and more than 30 points with 5% peroxide.

Pulp brightness after two-step bleaching (90°C and 120 min in each step) was 79% with 3% peroxide and 81.3% with 4% peroxide. TJ

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