CHEMIMECHANICAL PULPING OF *EUCALYPTUS GRANDIS*

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ABSTRACT

Eucayptus is currently one of the main fibrous raw materials used in the pulp and paper industry in given parts of the world. The objective of the present paper is to optimize the chemimechanical pulping conditions for *Eucalyptus grandis,* evaluate the pulp quality obtained, and draw conclusions regarding its potential use. The raw material used was *Eucalyptus grandis* industrial chips obtained at a Celulosa Argentina mill in Puerto Piray, Misiones, Argentina. For all the experiments, the chemical stage was carried out in a stainless steel digester with a liquor recirculation system. The mechanical stage was carried out in an 8-in. atmospheric disk refiner. Sodium sulfite and sodium hydroxide were added as chemical reactives. The central composite experimental design used involved five levels for the two variables studied (2^2 factorial design + star + central point). Three repetitions of the central point were carried out to check for errors. The variables studied were: initial amount of sodium sulfite in the wood (0.9 to 3.5% oven-dry wood) and reaction temperature (96 to 124 C). Times until maximum temperature and time at maximum temperature were, respectively, 20 and 90 minutes. A constant level of sodium hydroxide was maintained in all the experiments (1.5% oven-dry wood). Pulp evaluation was carried out using the usual characterization techniques. Chemical and physical evaluations, including optical testing, were, for the most part, done in accordance with TAPPI procedures.

The results obtained indicate that the central point of the design used in our research (110 C and 2.5% oven-dry wood sulfite), appears to represent the optimal conditions for the variables studied for the chemimechanical pulping of *Eucalyptus grandis.* The pulps obtained could be used as furnish in printing and writing paper grades. The positive correlation between sulfonate concentration and water retention value (WRV) suggests that by increasing fiber wall swelling, the number of sites accessible to sulfonation is increased. The tensile index correlates positively with the degree of sulfonation and with the water retention value of the pulps. It decreases according to the fraction retained in a 30-mesh screen (due to the presence of numerous shives) and increases according to the fraction of fines passing through a 270-mesh screen.

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INTRODUCTION

The use of *Eucalyptus* in cellulosic pulps was initially developed 50 years ago in Australia, where there are vast natural forests of that species. Work on developing *Eucalyptus* pulps was begun in Argentina in 1947 by Celulosa Argentina S.A. at its Capitan Bermúdez mill. Since then, growth in the number of users of *Eucalyptus* has stimulated significant forestation in that country (Ordoñez and Zilli 1971). *Eucalyptus* is currently one of the main fibrous raw materials used in the pulp and paper industry.

Eucalyptus is part of the group of woods known as hardwoods. The anatomical, physical, and chemical characteristics of the various species have been widely studied, mainly in Australia but also in other countries such as Spain, Portugal, Argentina, and Brazil. In the latter countries, the main species planted are *E. regnans, E. globulus, E. saligna,* and *E. grandis.* The species most cultivated in Misiones (Argentina) is *Eucalyptus* grandis, which is why it was chosen for this study.

Table 1 presents a summary of the most important chemical characteristics of *Eucalyptus* found in the literature. They are, in order of importance: age, cellulose, holocellulose (holoc.), insoluble lignin, soluble lignin, alcohol-benzene extractives (a.b.E), cold water extractives (c.w.E), hot water extractives (h.w.E), 1% NaOH solubles, ashes, and pentosans.

Although the characteristics studied by various authors vary significantly, the levels of the main chemical components (cellulose, hemicellulose, lignin) are similar. Differences appear in the minor chemical components, due to numerous factors, such as: the age of the trees, climate, soil type, the presence of tension wood, etc. While age is not indicated in most of the references, it is possible that the Argentina trees studied (mainly thinnings) were younger than those of South Africa.

Table 2 shows the physical and anatomical characteristics of *Eucalyptus grandis* as found in the literature. As indicated in Table 2, *Eucalyptus grandis* is a comparatively high density wood. Its fiber length is typical of an average hardwood (0.9 to 1.0 mm), and the fiber length/fiber diameter ratio is approximately 66. Its flexibility coefficient (fiber diameter/lumen width) is 0.46. These values suggest that the species' rigid fibers will produce pulps with high opacity but low resistance.

The general objective of the present research work was to optimize the chemimechanical pulping conditions for *Eucalyptus grandis* (from Argentina's northeastern region) by evaluating the pulp quality obtained, and subsequently to come to conclusions regarding its potential use.

Because of the selective actions of NaOH (action on the carbohydrates) and Na_2SO_3 (action on the lignin), particularly on hardwoods, the optimum conditions for chemical treatments using both reactives appear to be quite different (Area and Valade 1992a, b). Several authors have postulated the benefits of impregnation over a long period at room temperature. The response of white birch (*Betula papyrifera* Marsh.) to a process consisting of a 5-h impregnation of the chips at 50 C, using 1.5% NaOH and 2.5% Na_2SO_3, followed by a 5-min heating to 128 C and subsequent pressurized refining was evaluated in other research work (Area and Valade 1993, 1994). This process was referred to as "modified CTMP." Time and pretreatment temperature were the two main variables of the chemical stage. Treatment temperature was found to be the most significant parameter in the range studied (22 to 78 C).

No advantages were found for carrying out impregnation at room temperature. Moreover, better results were obtained working with temperatures of approximately 80 C. The present study was aimed at verifying if *Eucalyptus* reacts in the same way when chemimechanical pulping is carried out. A temperature range between 96 and 124 C was examined, the spe-

TABLE 1. *Chemical characteristics of* Eucalyptus grandis.

Origin	Age (years)	Cellulose (%)	Holocel. (%)	Insoluble lignin (%)	Soluble lignin (%)	a.b.E (%)	c.w.E (%)	h.w.E (%)	1% NaOH soluble (%)	Ashes (%)	Pentosans (%)
Castelar [1]	—	—	—	24	—	—	2.0	4.0	18	—	—
Concordia [1]	—	—	—	23.0	—	—	—	3.1	16.8	—	—
South Africa [2]	18–24	40.8–49.9	—	21.37–26.32	—	2.00–5.40	—	0.33–2.74	9.04–16.96	—	9.87–13.28
Misiones [3]	8–11	42.8–46.5	66.3–68.7	24.1–26.7	3.3–3.5	1.24–2.47	—	1.30–1.61	—	0.2	—

[1] Ordoñez and Zilli 1971; [2] duPlooy 1980; [3] Nuñez 1988.

cific objective being to verify if physical properties continue to improve above 80 C.

Other authors found that birch pulps displayed a mild degree of sulfonation (15 to 25 mmol/kg), which greatly modified the physical properties (Area and Valade 1993). The degree of carboxylation and total ions, measured using a conductimetric technique, did not, however, affect the properties studied.

It was then decided to study the effect of various low levels of sulfite on wood in chemimechanical pulping. The normal sulfite level used being 2.5%, the range in the experimental design was set between 0.9 and 3.5% oven-dry wood.

This study also involved analyzing the response of the various parameters with respect to the independent variables selected (treatment temperature and charge of sodium sulfite on wood). Response surfaces relating independent variables to the pulp evaluation parameters (chemical and physical properties) were obtained. Correlations were subsequently found relating the influence of alkaline swelling and sulfonation action on the other properties.

EXPERIMENTAL

The raw material used was industrial *Eucalyptus grandis* chips from a Celulosa Argentina mill in Puerto Piray, Misiones, Argentina. The chips were classified according to thickness, the mean being 3.5 mm. Knots and wood containing kino pockets were discarded. One kilogram of wood was prepared for each operation. The chemical stage was carried out in a stainless steel digester with a liquor recirculation system and electronic temperature control.

To determine the pulping strategy, it was decided to use a central composite experimental design (Barker 1985) consisting of five levels for the two variables studied (2^2 factorial design + star + central point). Three repetitions of the central point were done in order to check for possible errors. A diagram of the experimental design is shown in the Appendix (Fig. A1).

The variables studied were the initial

TABLE 2. *Physical and anatomical characteristics of* Eucalyptus grandis.

Origin	Age (years)	Density (kg/m^3)	Fiber length (mm)	Fiber diameter (μm)	Wall thickness (μm)	Vessel diameter (μm)	Vessels/ mm
South Africa [1]	18–24	344–670	0.89–1.36	13.7–17.0	3.2–6.6	—	2.0
Australia [2]	—	444	0.90	14.8	2.8	117	—
Florida [3]	14	345–393	—	—	—	—	—
Misiones [4]	8–11	400–570	—	—	—	—	—

[1] duPlooy 1980; [2] Higgins et al. 1973; [3] Wang et al. 1984; [4] Nuñez 1988.

amounts of sodium sulfite on wood (0.9 to 3.5% oven-dry wood) and treatment temperature (96 to 124 C). Concentrated solutions of Na_2SO_3 and NaOH were used to prepare the pulping liquor. The sulfite charge was set by the experimental design. A constant sodium hydroxide level was maintained in all the experiments (1.5%). The liquor was heated to 80 C before the chips were added. Time to maximum temperature was set according to previous experiments, taking into consideration the thermal inertia of the system. Times to and at maximum temperature were 20 and 90 min, respectively. To quantify the residuals, spent liquor samples were taken after the chemical stage. The chips were weighed and fed directly into the refiner.

The mechanical stage was carried out in an 8-in. atmospheric disk refiner equipped with a 5-hp motor, using a bar-and-groove disk design, without dams. To regulate inlet consistency, water temperature was maintained at 60 C. After defiberization and refining of the chips, the pulp was collected in a fabric bag installed inside a semi-industrial centrifugal tank to avoid losing fines.

Pulp evaluation was carried out using the usual characterization techniques. Chemical and physical evaluations, including optical testing, were done, for the most part, according to TAPPI procedures. The following were also determined: freeness, using the SCAN Schopper Riegler method; water retention value (WRV), using TAPPI UM 256; and sulfonates and carboxylates in the pulp, using conductimetry. (Katz et al. 1984).

All statistical analyses were performed using special computer software. The results were statistically evaluated to a 95% confidence level. The response surfaces of each of the dependent variables as a function of the independent variables were obtained. Correlations among responses were also calculated in order to verify the effect of the degree of sulfonation, carboxylation, total ions, and alkaline swelling on the physical and mechanical properties.

RESULTS

Table 3 shows the spent liquor residuals, the chemical and physical properties of the pulp, and the levels of the variables in each experiment. Figures 1 to 7 show responses of some of the dependent variables.

RESULTS AND DISCUSSION

As can be seen in Table 3, the final liquor pH remained alkaline, while sulfite consumption varied. Minimum consumption (30%) occurred at the lowest temperature (96 C), while the greatest consumption occurred at the lowest initial sulfite levels, implying that consumption due to sulfonation is low.

The degree of sulfonation obtained was within the low limit of the so-called "low level chip sulfonation" for coniferous species (quoted at 50 mmol/kg) (Atack 1987), but can be considered high for hardwood species. As a rule, this sulfonating action produces little improvement in pulp resistance and brightness. The low carboxylate level may indicate dissolution of some hemicelluloses, since esters and lactones are the groups susceptible to carboxylation.

Freeness levels, measured using the SCAN Schopper Riegler method (the corresponding range in CSF is 100 to 250), appeared appro-

TABLE 3. *Results of the experiments.*

Experiment number	1	2	3	4	5	6	7	8	9	10	11
Sulfite (% oven-dry wood)	1.5	3.5	1.5	3.5	1.1	3.9	2.5	2.5	2.5	2.5	2.5
Coded variable and level	−1	+1	−1	+1	−1.41	+1.41	0	0	0	0	0
Temperature (°C)	100	100	120	120	110	110	96	124	110	110	110
Coded variable and level	−1	−1	+1	+1	0	0	−1.41	+1.41	0	0	0
Final liquor pH	8.8	8.4	8.0	7.6	8.9	7.8	8.9	7.5	8.3	8.3	8.2
Consumed $SO_3^=$ (% initial)	44	34	68	44	75	32	30	62	38	37	44
Sulfonates (mmol/kg)	25	32	29	43	25	45	34	36	27	29	34
Carboxylates (mmol/kg)	101	100	99	115	99	109	91	113	89	89	101
Total ions (mmol/kg)	126	132	128	158	124	154	125	149	116	118	135
Pulp consistency (%)	7.5	8.0	6.5	7.0	6.5	7.0	6.5	7.5	4.5	5.0	6.0
° Schopper Riegler	67	67	58	74	68	47	62	60	45	45	47
Drainage time (sec)	15	18	8	28	14	6	12	9	6	6	7
Energy (MJ/kg)	7.1	7.3	7.5	8.1	7.4	6.5	6.8	7.2	6.4	6.0	6.2
R30 (%)	11	6	12	5	13	12	13	15	14	19	16
P150 (%)	46	67	58	63	52	40	44	37	33	32	47
P270(%)	15	31	18	37	8	22	16	1	4	6	19
Tensile index (Nm/g)	12	19	11	19	13	19	14	14	10	11	15
Burst index ($kPam^2/g$)	0.24	0.35	0.26	0.32	0.26	0.63	0.30	0.49	0.25	0.28	0.20
Tear index (Nm^2/g)	6.3	5.7	6.2	6.5	6.2	7.6	5.4	5.4	5.4	6.7	6.9
Bulk (cm^3/g)	3.1	2.7	3.1	2.6	2.8	2.8	2.9	2.6	3.2	3.1	3.3
Brightness (% ISO)	43	44	43	39	48	50	41	41	49	50	46
WRV (%)	128	136	126	144	126	135	138	134	126	132	132
Extractives (% oven-dry wood)	0.13	0.07	0.14	0.12	0.13	0.11	0.10	0.12	0.10	0.10	0.13

priate for printing and writing paper grade furnish. Bulk values were in the medium-to-high range (2.6 to 3.3 cm^3/g). Tensile strength and explosion values were poor when compared with the references cited for *Eucalyptus* CMP (Marton et al. 1979; Croon and Farinha 1985), and tear strength was particularly high. It should be noted that the soda level used was far below the level currently used. In hardwood pulping, it is a well-known fact that resistance improves with an increased soda charge (Prusas et al. 1957; Higgins et al. 1978). Brightness levels obtained were lower than those quoted for *E. regnans* (Higgins et al. 1978). All resistance and brightness levels were higher, however, than those found for Brazilian *Eucalyptus saligna* (Pinho and Assumpçao 1983).

The low content of extractives in the pulps suggests that they were almost completely eliminated during the various pulp processing stages.

Microscopic observation of the pulps indicated large quantities of shives, especially in the fraction retained in the 30-mesh screen. There was a wide variation in the quantity of fines. This is typical of atmospheric refining (Heikkurinen et al. 1993) and is a result of the disk design used. The fine bar-and-groove pattern is well adapted to refining, but contributes to extensive fiber damage when applied to chip defiberization. The strong correlation between the fractions retained in the 30- and 270-mesh screens shows that large quantities of fines are generated when a stronger mechanical action is applied to lower the content of shives.

The amount of residual sulfite in the spent liquor increased significantly according to initial sulfite levels. Residual sulfite decreased according to the pulping temperature (Fig. 1), most likely due to secondary decomposition reactions with the sodium thiosulfate generated. Residuals exhibited a significant positive correlation ($R^2 = 0.90$) with the degree of sulfonation. This apparently contradictory result can be explained by an increase in sulfonation and consumed sulfite due to an increase in the initial sulfite charge.

The degree of sulfonation depends linearly on the initial quantity of sulfite ($R^2 = 0.67$, Table A1, Appendix) as shown in Fig. 2. The sulfonate content in the range of sulfite concentrations studied (0.9 to 3.5% oven-dry wood) was 25 to 45 mmol/kg. The temperature range tested (between 96 and 124 C) did not produce significant variations in the degree of sulfonation. The variation coefficients among duplicated experiments were satisfactory. Differences found in the central points were probably due to operational problems.

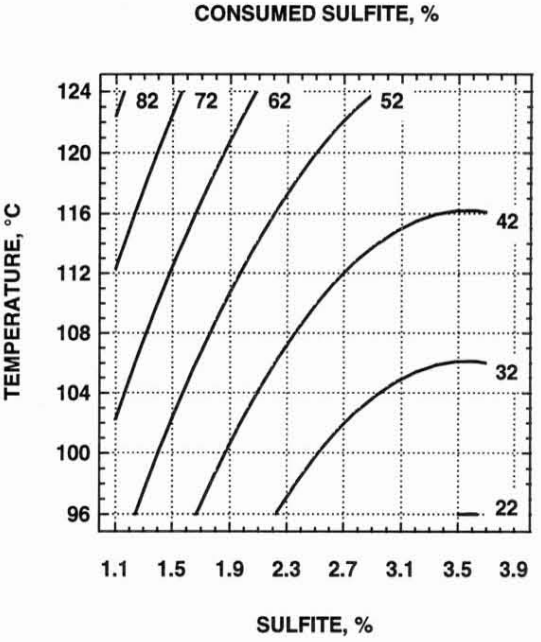

FIG. 1. Contours lines of consumed sulfite as a function of original amount of sulfite and temperature.

Pulp swelling, measured by water retention value, also increased linearly in relation to the initial amount of sulfite applied (Fig. 3). The positive correlation between sulfonate concentration and water retention value (WRV) suggests that the more swelling there is in the fibrous walls, the more sites are susceptible to sulfonation (guaiacyl units) in the outer layers of the cell walls and in the middle lamella (Beatson 1986; Beatson et al. 1985). At the same time, lignin sulfonation rendered the pulp more hydrophilic, thus increasing its water retention capacity.

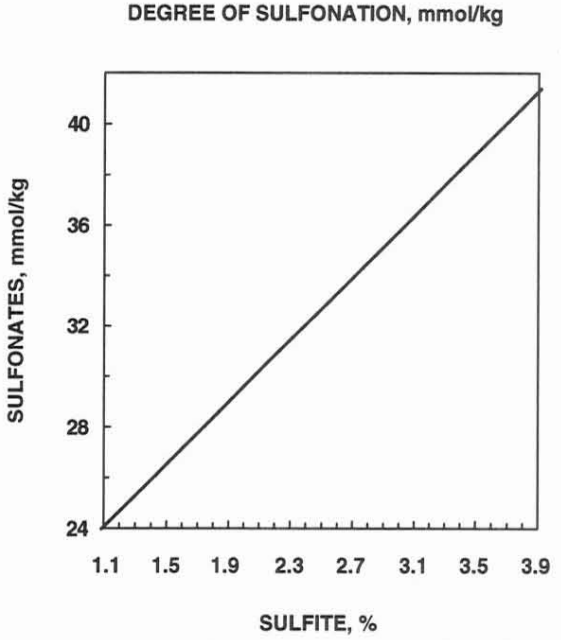

FIG. 2. Degree of sulfonation shown as a linear function of original amount of sulfite and quantity of sulfonates generated where temperature had no influence.

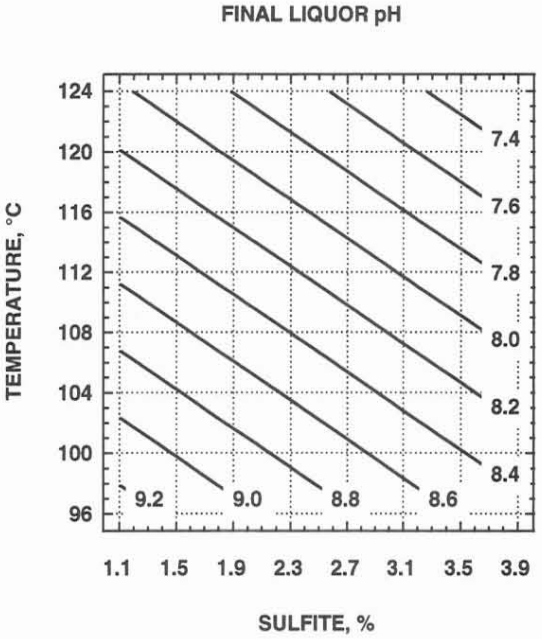

FIG. 4. Contours lines of final liquor pH as a function of original amount of sulfite and temperature.

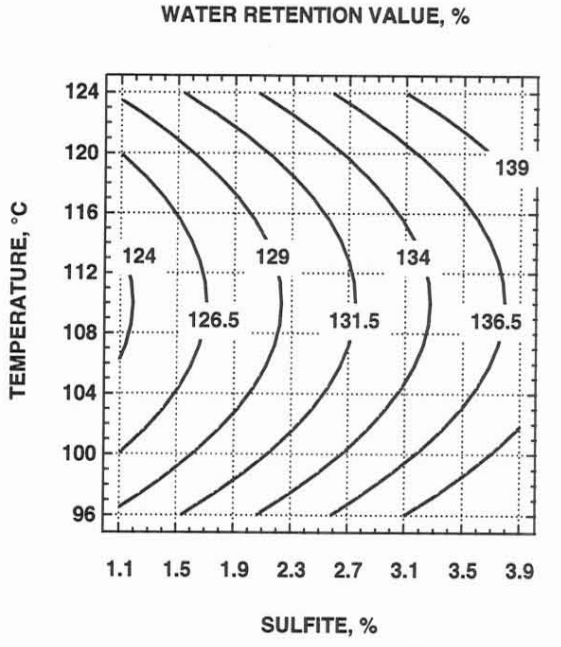

FIG. 3. Contours lines of water retention value as a function of original amount of sulfite and temperature.

An increase in the initial sulfite charge and the reaction temperature resulted in a reduction of the final liquor pH, the reaction temperature being the more important factor (Fig. 4). This suggests that a greater consumption of soda occurs when both variables are at higher levels, as a result of the combination of alkaline swelling (measured by WRV), hemicellulose ester saponification (Heikkurinen et al. 1993) and secondary dissolution reactions of extractives. The negative correlation between the final liquor pH and the degree of sulfonation of pulp lignin indicates that the latter increases under the same conditions in which soda is consumed. The final liquor pH is on line with the degree of carboxylation.

Energy consumption varied significantly depending on the chemical pretreatment of the chips (Tables A1 and A2, Appendix). Figure 5 shows that under the conditions used, energy consumption was lowest with 2.5% sulfite on wood and at 110 C, and higher percentages of sulfite resulted in higher energy consumption.

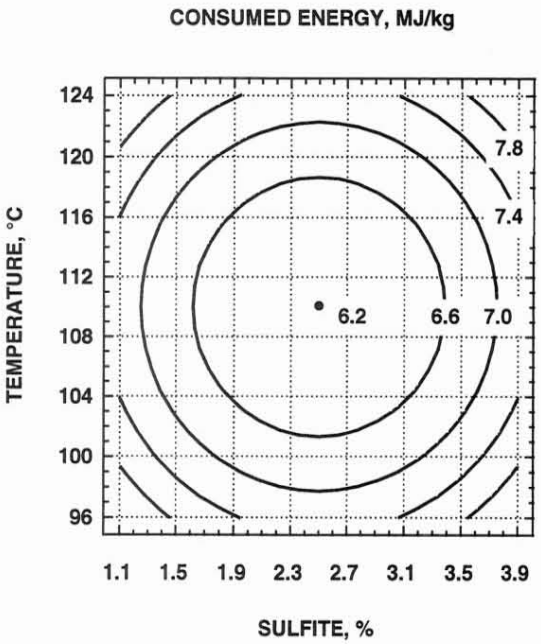

FIG. 5. Contours lines of consumed energy as a function of original amount of sulfite and temperature where the energy consumed is at minimum at 2.5% sulfite and 110 C.

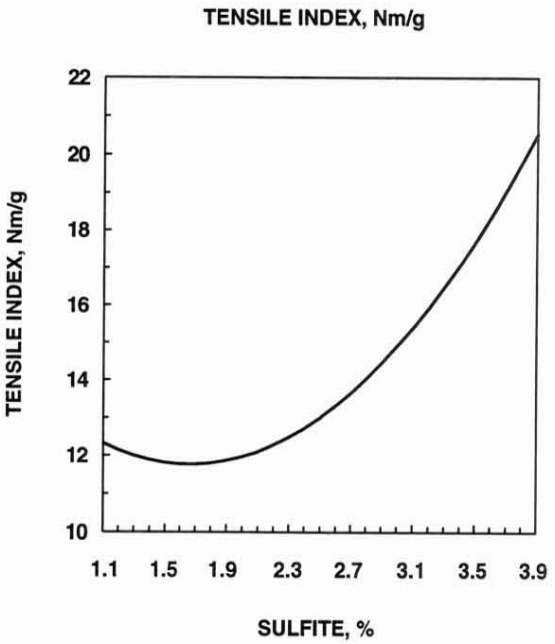

FIG. 6. Response of tensile index as a function of original amount of sulfite. The temperature did not affect that property. A minimum of 1.8% sulfite is required.

These findings coincide with those obtained in a previous study (Area and Valade 1993) and are consistent with other data reported (Bengtsson and Simonson 1990). This could be because fiber separation occurs within the weakest part of the wood. When the amount of sulfite added is high in relation to the amount of sodium hydroxide, the middle lamella is the preferred site for sulfonation, resulting in a softened lignin encapsulating the separated fibers. This could explain the large quantity of energy required for refining. When an NaOH charge prevails, fiber separation occurs in the weakened, alkaline-swelled cell walls.

Energy consumption increased linearly with an increase in pulp consistency (R^2 = 0.50), resulting in a decrease in shives (retained in the 30-mesh screen) and an increase in the fraction of fines (passed through the 270-mesh screen). It also produced a significant increase in sheet density and improved tensile strength by increasing fiber bonding. Conversely, an increase in energy consumption would be harmful to tear strength (presumably due to fiber cutting).

Tensile index increased quadratically with sulfite charge. This might be explained by the fact that there is a plateau until 2% sulfite is reached (Fig. 6). Tensile index correlated positively with the degree of sulfonation (R^2 = 0.62), that is, in relation with the initial sulfite charge. The correlation of the tensile index with the water retention value is proof of the effect of alkaline swelling on fiber conformability. This property also decreased according to the fraction retained in the 30-mesh screen (numerous shives), and increased in relation to the fraction of fines passing through the 270-mesh screen.

Bulk correlated with typical variables of the mechanical stage, such as: mean pulp consistency, R^2 = 0.44; consumed energy, R^2 = 0.48; freeness, R^2 = 0.48; and drainage time, R^2 = 0.40, as did the fibrous composition of the pulp (fractions of Bauer McNett classification). Water retention value also showed some depen-

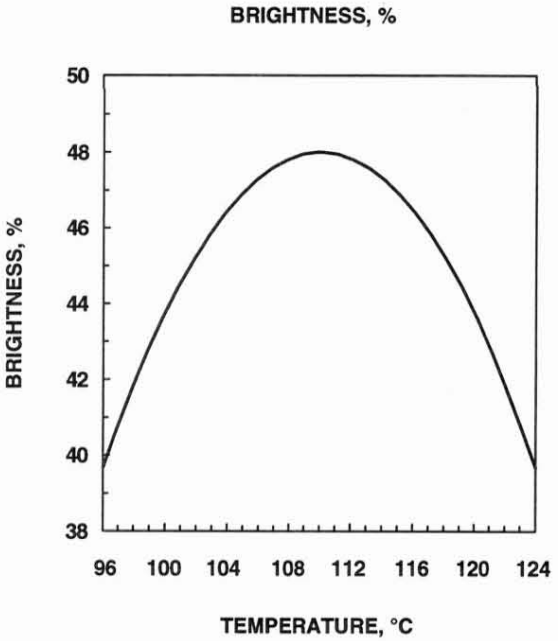

FIG. 7. Response of the brightness as a function of temperature. The amount of original sulfite did not affect the property. The brightness shows a maximum of 48 at 110 C.

dency on the fibrous composition of the pulp, due to greater specific surfaces in the fraction of fines passing through the 270-mesh screen.

In the slightly alkaline sulfite system used in this study, brightness varied in relation to the square of the temperature, reaching a maximum near the central point (48 points of brightness at 110 C) as shown in Fig. 7. Within the range studied, no variation in brightness in relation to sulfite charges was observed, preventing alkaline darkening.

Reaction temperatures between 105 and 110 C resulted in lower energy consumption and improved brightness. Although physical resistance increased with sulfite charge, energy consumption was lowest at 2.5% oven-dry wood. The central point of the design used in this study (110 C and 2.5% oven-dry wood sulfite) seems to represent the optimum conditions for the chemimechanical pulping of *Eucalyptus grandis*. At these levels, sulfite consumption was 50% of the initial amount, and the final liquor pH value was approximately 8.3.

The present "soda-sulfite" system with low charges of alkali behaved in a different way than the "soda" process conventionally used for hardwoods. In the presence of sulfite, lengthy treatments at room temperature are not necessary to prevent brightness loss due to alkaline darkening. This had already been proven in a previous study (Area and Valade 1993).

CONCLUSIONS

The central point of the experimental design used (110 C and 2.5% oven-dry wood sulfite), appears to represent the optimum conditions for chemimechanical pulping of *Eucalyptus grandis,* the pulps obtained being appropriate as furnish for printing and writing paper grades.

The positive correlation between sulfonate concentration and water retention value (WRV) suggests that by increasing fiber wall swelling, the number of sites accessible to sulfonation is increased.

The tensile index correlates positively with the degree of sulfonation and with the water retention value of the pulps. It decreases according to the fraction retained in the 30-mesh screen (due to the presence of numerous shives) and increases according to the fraction of fines passing through the 270-mesh screen.

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APPENDIX

The experimental design strategy is shown in Fig. A1.

Table A1 presents the results of the experimental design analysis, including the correlation coefficients (R^2) and levels of significance (P) obtained for each regression equation relating independent linear and quadratic variables with the properties studied.

The coefficients of the equations obtained for each parameter are presented in Table A2. The equations are valid for the independent variables expressed in an orthogonal form (−1, 0, 1, etc.). The equations are presented in the form:

$$\text{Property} = C + a(\text{sulfite}) + b(\text{temperature}) + c(\text{sulfite})^2 + d(\text{temperature})^2.$$

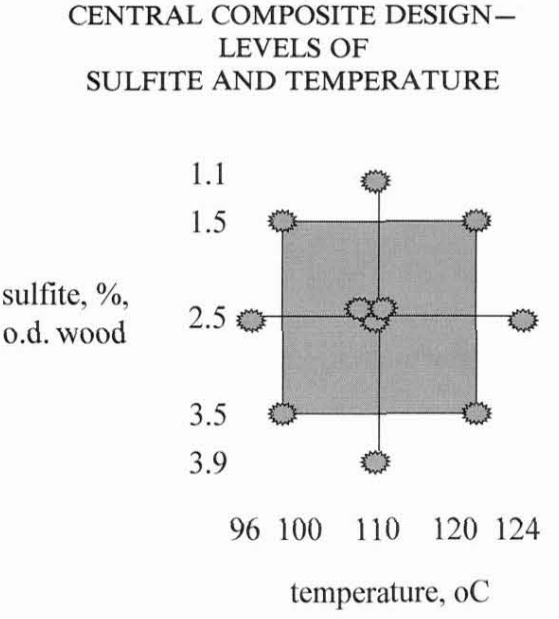

FIG. A1. Experimental design strategy showing the 5 levels of sulfite and the 5 levels of temperature used including the 3 replicates at the center location for a total of 11 trials.

TABLE A1. *Statistical parameters of the regression equations.*

Property	R^2	$P_{sulfite}$	$P_{temperature}$	$P_{sulf.}^2$	$P_{temp.}^2$
Energy	0.58	—	—	0.0303	0.0248
Sulfonates	0.67	0.002	—	—	—
Final liquor pH	0.95	0.0002	0.000	—	—
Consumed $SO_3^=$	0.90	0.0006	0.0018	0.0465	—
Tensile index	0.77	0.018	—	0.0432	—
Brightness	0.73	—	—	—	0.0008
WRV	0.73	0.034	—	—	0.0573

TABLE A2. *Parameters of the regression equations in function of the independent variables (orthogonal form): Property = C + (sulfite) + b(temperature) + c(sulfite)^2 + d(temperature)^2.*

Properties	C	a	b	c	d
Energy	6.2	—	—	0.51	0.53
Sulfonates	32.63	6.16	—	—	—
Final liquor pH	8.25	−0.29	−0.45	—	—
Consumed $SO_3^=$	42.12	−11.85	5.59	—	—
Tensile index	13.0	2.94	—	1.75	—
Brightness	48.0	—	—	—	−4.25
WRV	130.29	4.84	—	—	2.97