NSSC PROCESS OPTIMIZATION: PULPING, PULPS AND SPENT LIQUORS

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ABSTRACT

The objective of this work was to obtain the best physical properties of NSSC pulps using low charges of chemical products, and to determine the operating conditions at which spent liquors containing the lowest ratio of inorganic/organic solids can be obtained (improving or maintaining the pulp quality). Hybrid poplar (Populus deltoides) was used as raw material. The experimental design did examine four variables at five levels (Central Composite Design CCD= 29 runs, five central points). Minimum and maximum limits of the independent variables were established using mill conditions as set point values. Variables studied were: cooking time (10 to 30 min), cooking temperature (154 to 186ºC), sodium sulfite (4 to 12% o.d.) and sodium carbonate charges on wood (0 to 3% o.d.). Each property was evaluated by comparison with the mill reference data (yield: 80%, 260 CSF, tensile index: 38 N.m/g). Cooking conditions for highest mechanical properties were: temperature of 186ºC, cooking time of 30 min, 11.6% of Na_2SO_4 and 3.05% of Na_2CO_3 on o.d. wood. These conditions implied important pulp brightness and yield reductions. Optimum conditions minimizing the inorganic/organic solids ratio were: 26 min, 177ºC, 6.0% Na_2SO_3, 2.54% Na_2CO_3. Spent liquors organic solids compositions were also determined. Time, temperature, and sulfite charge showed the greatest effects on organic material dissolution. Carbonate charge acts through interactions. Pilot plant trials were done to verify the laboratory results and the found equations. Four pulps were manufactured, using Spanish poplar as raw material. On these pulps, physical testing on 60 g/m^2 handsheets and typical corrugated board tests on 120 g/m^2 handsheets (CMT, RCT, STFI) were completed.

KEYWORDS

Populus deltoides - semichemical pulps - neutral sulfite pulping - pulp properties - experimental design - physical properties - board properties - spent liquors properties

INTRODUCTION

Among "traditional" pulping processes, the neutral sulfite semichemical process (NSSC) is surviving, maintaining its relative importance. Modern chemimechanical processes (CMP and CTMP) were developed from its chemical basis (1). Even when NSSC process is generally considered only for the production of hardwood corrugating medium, current trend is to use it as a partial kraft pulp replacement in linerboard and bag grades (1 - 4). In addition, bleached NSSC pulp is used to produce printing and writing papers, business forms, reply cards and tissue paper (5). It was also suggested that NSSC is more flexible than Kraft process. It presents higher yields for similar resistance. At the same yield, NSSC pulps are easily bleached and they require less energy to be refined (2).

In neutral sulfite pulping a major drawback needs to be resolved. In many cases, NSSC spent liquors are sent directly to effluents. Despite great efforts from its beginning, spent liquors chemicals recovery is still to be addressed. Difficulties can be attributed in part to their properties, particularly their high inorganic to organic ratio. The reduction of inorganic solids in spent liquors is then a must.

References of NSSC spent liquors characterizations are limited. In general, spent liquors have final pH varying from 6.5 to 8.5 and contain10-13 g/l of sodium acetate and 8 to 22% of total solids. Their calorific capacity is rather low (6.3-12.7 GJ/t) (6). They contain hemicelluloses partially degraded having DP of 115-131 (7), being their viscosity proportional to the polysaccharide content (8).

Other work on eucalyptus NSSC spent liquor presents an ash content of 41.6% of total solids (9). They can only be evaporated to 50% because of their high viscosity (10). They are more corrosive than Kraft spent liquors, even after neutralization (11). The most used recovery system is the cross recovery with Kraft spent liquors (1). Some mills have fluidized bed combustion systems (12). Sulfur dioxide can be recovered from spent liquor by acidification, and recycled to the white liquor (13).

The objective of this work is to obtain the best physical properties of NSSC pulps, using low chemical charges in order to reduce inorganic solids in spent liquors, and to determine the operating conditions where spent liquors containing the lowest ratio of inorganic/organic solids can be obtained. These operating conditions had to be such that pulp quality is maintained or improved.

Minimum and maximum variable limits were based on conditions in “Productos Pulpa Moldeada” mill (Cipolleti, Rio Negro, Argentina). The capacity of the mill is 20,000 t/y of bleached and semibleached pulp for thin boards and crude pulp for high quality corrugating medium. Its technology was provided partially by Sunds Defibrator (Sweden).

EXPERIMENTAL

Laboratory trials

Hybrid poplar (Populus deltoides) from the delta of Paraná River in Argentina was used as raw material. It was received as industrial chips from Celulosa Argentina S.A., Zárate mill.

The experimental design selected to accomplish the study of NSSC process analyzed four variables at five levels (design type CCD= 29 runs, including 5 central points for experimental error detection). The full experimental design (real and transformed variables) can be retrieved in the full version of this work (14).

Variables studied and process conditions were:

- Cooking time (time at maximum temperature): 10 to 30 minutes.
- Temperature: 154 to 186ºC.
- Sodium sulfite charge (% $SO_4^{=}$): 4 to 12% o.d.
- Sodium carbonate charge (% $CO_3^{=}$): 0 to 3% o.d.

Pulping methodology

- **Pre-chemical stage**: In order to reproduce the effect of the compression screw in the mill, a light mechanical stage was accomplished before the chemical one.
- **Chemical stage**: It was carried out in a 4-lt. stainless steel digester, having a vacuum connection and two heating systems. Time at maximal temperature was calculated on line with a software attachment, using the H factor at time intervals of 0.5 min (15). The activation energy used was 32 000 cal/mole. It was calculated from values obtained for NSSC cooks at long and short times (16). The liquor to wood ratio was 6.5:1. Yields were initially obtained by weighting a known quantity of fibrous material packed in a small - 100-mesh - stainless steel basket before and after cooking. The basket was located in the center of the digester. As duplicates of yield values showed great dispersions, organic materials balances were made in spent liquors to recalculate them (17).
- **Defibration stage**: The defibration stage to 590 CSF was executed in a laboratory single disk refiner (Bauer, 8", 5 HP). The disks design reproduces those of the mill's first stage refining. Dilution water temperature was maintained at 60ºC. The energy consumption in the defibration stages (before and after the chemical stage) were recorded and added.
- **Refining stage**: The final refining stage was accomplished using a Valley beater. Final freeness as well as pulp yields corresponded to Productos Pulpa Moldeada mill conditions for thin bleached boards. The time required to reach 260 CSF was recorded as an indication of refining energy.

Evaluated parameters

- **Pulps at 590 ml CSF**: yield, total energy consumption, Somerville shives, initial CSF, time to reach 260 CSF.
- **Pulps at 260 ml CSF**: Bauer MacNett fiber classification, physical tests on 60 g/m^2 handsheets (tensile, burst and tear strength, bulk, and brightness), all using TAPPI standards.
- **Spent liquors**: ash, lignosulfonates, and xylans concentrations, acetic and formic acid concentrations and sugar contents. Xylans were taking as representing hemicelluloses. Materials and methods were presented in a previous work (17).

The statistical analysis of data (confidence interval of 95%) and the optimization study, were executed using an adequate software.

Selection of the best runs

The optimization study was performed using the matrix of equations for each property. The obtained values were evaluated by comparison with "Productos Pulpa Moldeada" mill settings (yield of 80%, 260 CSF, tensile index of 38 N.m/g). Yield value has been considered superior to 83%, since yields obtained as described above involve only the lost of material in the chemical stage.

Eight runs were selected as the best ones. In all of them the temperature was 178ºC. Sulfonic and carboxylic acid contents in pulps were determined using conductimetry (18). The results of the eight pulps selected were statistically analyzed as a 3 variables factorial design.

Pilot plant trials were done to verify laboratory results and derived equations. Four pulps were manufactured using a 20-liter digester and a 12" Sprout Waldron refiner at the Instituto de Investigaciones Agrarias (INIA), Madrid, Spain. Spanish poplar was the raw material. On these pulps, physical tests on 60 g/m^2 handsheets and typical corrugated board tests on 120 g/m^2 handsheets (RCT, CMT, STFI) were performed using TAPPI standards.

RESULTS AND DISCUSSION

The chemical composition of the raw material (Populus deltoides), as well as all tables of results can be found in the full versions of this paper (14, 19).

Yield, energy, and physical properties are plotted in function of the most significant parameters for each property in **Figures 1** to **6**.

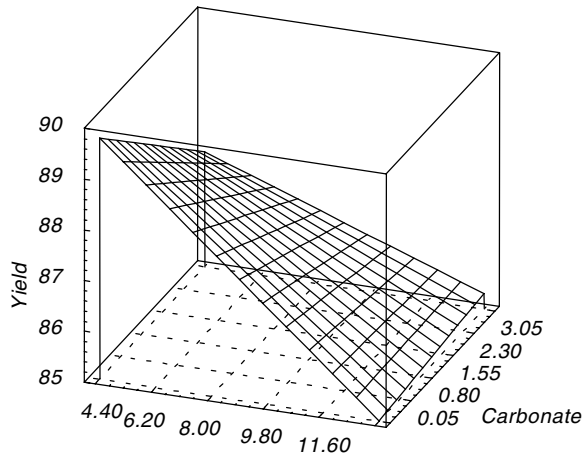


Figure 1. Variation of yield with sulfite and carbonate charges.

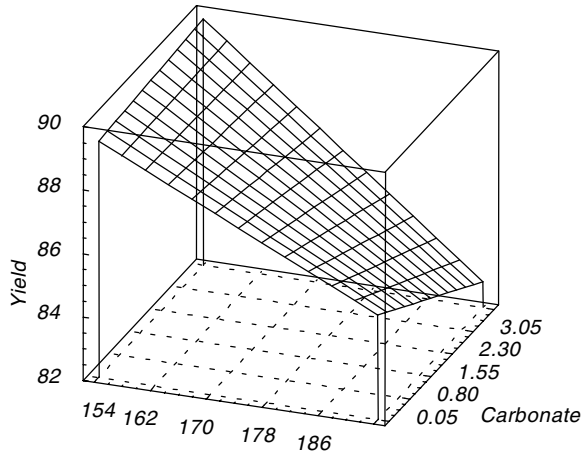


Figure 2. Variation of yield with temperature and carbonate charge.

The derived equations show a very good adjustment with experimental data. Temperature exhibits the greater influence on yield and physical properties. Sulfite charge is the most important parameter for energy. Bauer MacNett pulps fractions show a low correlation with

physical properties, excluding R30 mesh fraction that possesses a correlation coefficient of R =0.7 with burst.

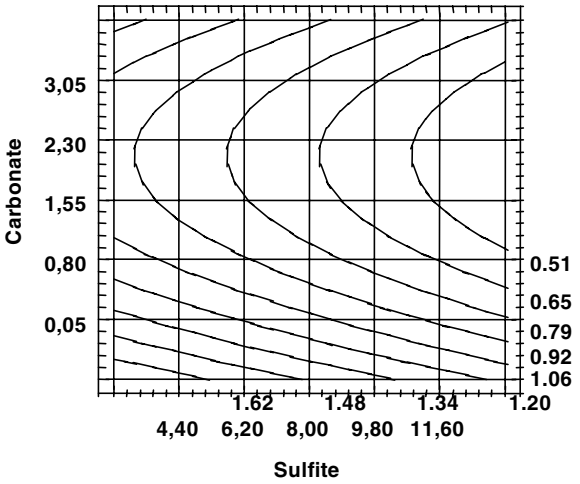


Figure 3. Energy variation with sulfite charge and carbonate charge.

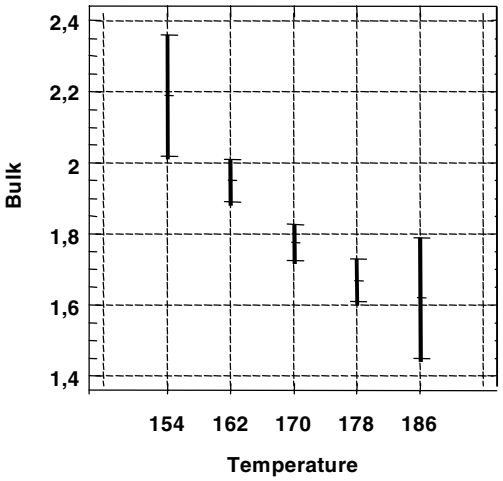


Figure 4. Bulk vs. temperature.

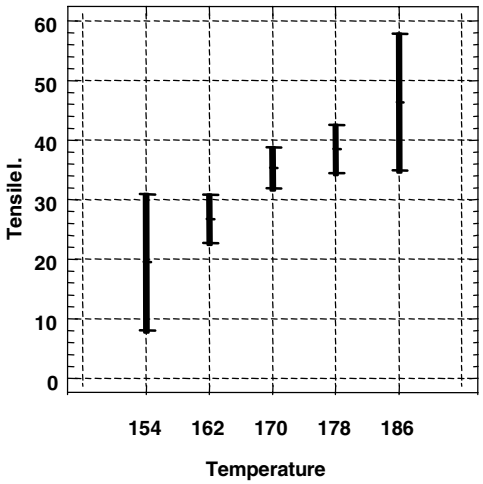


Figure 5. Tensile index vs. temperature.

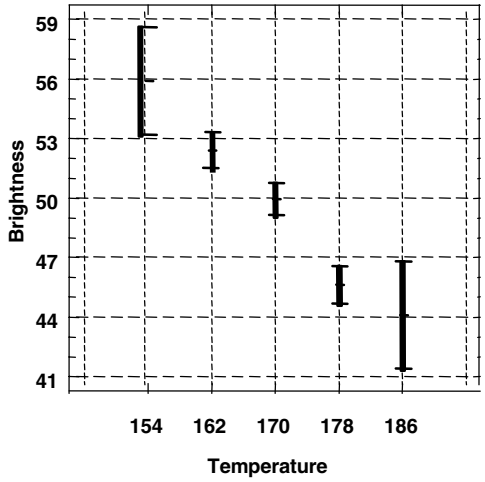


Figure 6. Brightness vs. temperature.

Yield

The different yields obtained illustrate the wide range of conditions of our experiments, in spite of small chemical charges used. Yields from 79.9 to 93.9% represent only the chemical stage. As water-soluble extractives were eliminated during the pre-chemical stage, global yields are about 3% lower.

The four variables have a linear influence on yield. Time, sodium sulfite charge, and temperature interactions with sodium carbonate have significant effects. At high sulfite charges or at low temperatures, the carbonate amount does not affect yield (Figures 1 and 2). The Tukey Test of variance homogeneity (20) indicates that yields obtained at 154 and 162ºC behave as a homogeneous group. The same reasoning was valid at 178 and 186ºC.

Yield reduction produces an improvement on mechanical properties, and a decrease of refining energy consumption (time to reach 265 CSF), bulk, and brightness.

Defibration and refining energy

Low defibration energy was necessary because of treating mechanically destructured lignocellulosic material. The energy consumption was very dependent on pulping conditions. All studied variables reduce defibration energy consumption. The obtained pulps present the typical behavior of chemically pretreated high yield pulps. Sodium sulfite charge was the most important factor in wood softening by lignin sulfonation. Temperature effect was also considerable, as a consequence of an increase in reaction rate. Amount of sodium carbonate affects energy due to the softening of the hemicellulosic material (21). Energy consumption seems minimal toward 2.2% of sodium carbonate charge (Figure 3). The required refining time in the Valley beater evolves in the same form that defibration energy in the refiner, but it does not depend on cooking time.

As yield does, the defibration energy influences only certain pulp properties, but this fact may be explained by the large experimental errors. The refining energy (time to reach 260 CSF) presents a strong correlation with physical properties.

Physical properties

Freeness values obtained after the defibration stages are typical for NSSC process (590 CSF). Somerville shives contents seem rather high.

Refined pulps properties were similar to those of industrial process (reference values). Pulp physical properties increase with yield lost, while bulk and brightness decrease. Temperature has strong influence on mechanical properties, followed in importance by sodium sulfite charge. Properties evolution seems to stabilize at maximum temperature levels (Figures 4 and 5).

Independent variables values to obtain highest mechanical properties were located (by the equation matrix resolution), toward the maximum levels of the experimental plan (temperature of 186ºC, cooking time of 30 minutes, 11.6% of sodium sulfite and 3.05% of sodium carbonate).

Brightness levels were generally high. The brightness was negatively influenced by temperature, reaction time and sodium carbonate charge, but positively affected by sodium sulfite amount. Strong pulping conditions affect initial brightness. The decrease of brightness with cooking temperature is illustrated in Figure 6.

Sulfonic and carboxylic acids content in pulps

Mean effects for of three studied variables (time, sulfite, and carbonate charges) are shown in the full version of the work (14). **Figures 7** and **8** illustrate the interaction influences on pulps sulfonic acids content.

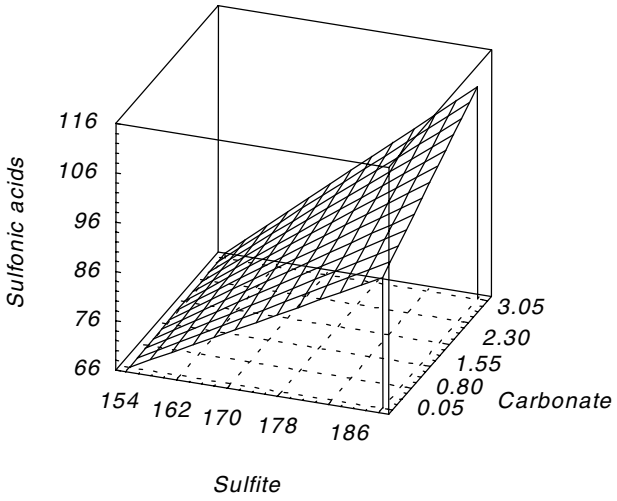


Figure 7. Sulfonic acids content in pulps (Sulfite-Carbonate interaction) .

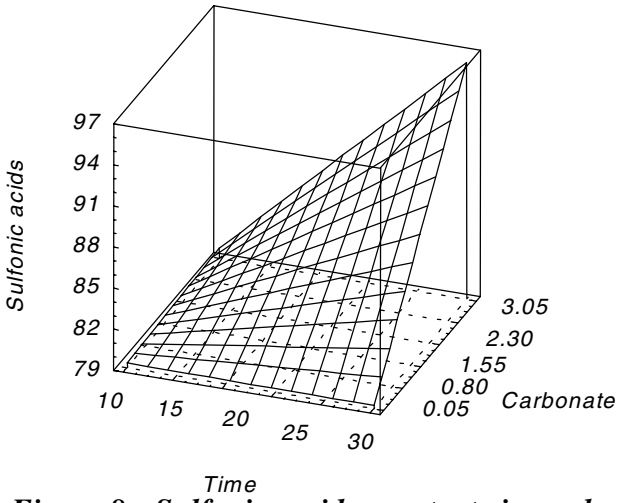


Figure 8. Sulfonic acids content in pulps (Time-Carbonate interaction).

Sulfite charge on wood presents the greatest influence on acid groups. Figure 7 and 8 show how carbonate affects sulfonation. Carbonate charge does not influence this response at low levels of the other factors, but it produces a great increase of sulfonation at their high levels.

Figures 9 to **11** show the variation in organic materials dissolution at different pulping conditions (mean of all values for each point). **Figure 12** shows changes of some spent liquor properties in function of cooking temperatures.

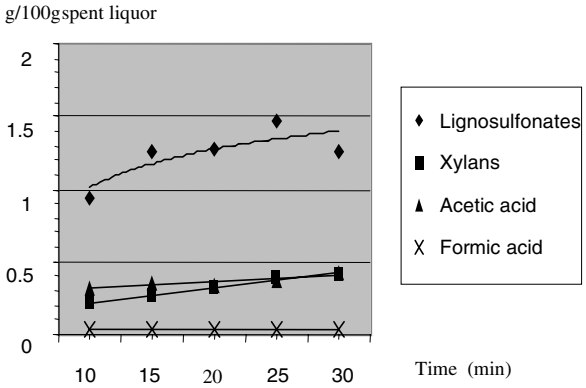


Figure 9. Organic components evolution in NSSC spent liquors vs. cooking time.

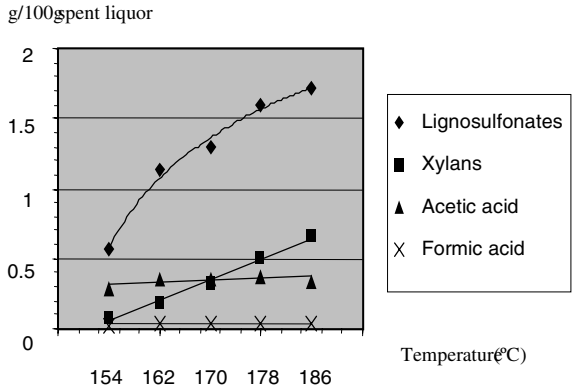


Figure 10. Organic components in NSSC spent liquors in function of temperature.

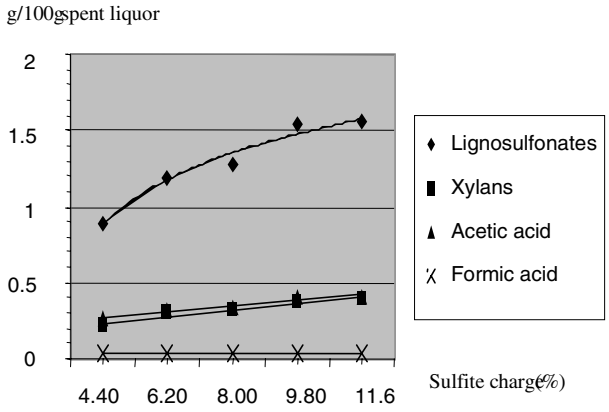


Figure 11. Organic components in spent liquors vs. sulfite charge.

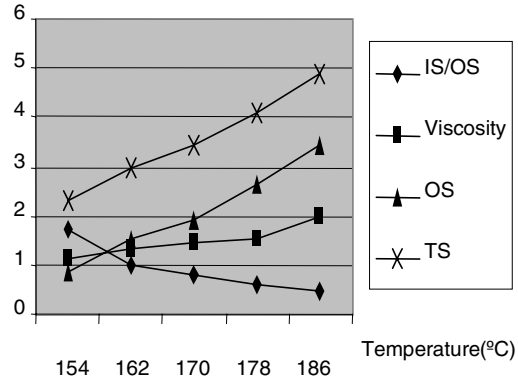


Figure 12. IS/OS ratio, TS, OS and viscosity vs. cooking temperatures.

Figure 9 shows that organic acids are present in spent liquors since the early stages of NSSC cooking. While xylan concentration in spent liquor increases linearly during cooking, lignosulfonates concentration seems to stabilize at the end of the cooking.

Acid generation present similar pattern with temperature increase (Figure 10). Xylan dissolution at 154°C is almost zero, however it is important at 186°C. Lignosulfonates presence in spent liquors is high at 154ºC, and it seems to increase linearly toward higher ones.

While organic acids and xylans content in spent liquors present little modification with sulfite charge, changes in lignosulfonates concentration is high, showing an intermediate level between its variation with time and temperature (Figure 11).
The total solids increase with temperature is parallel to organic solids content in spent liquors (Figure 12). Their strong correlation can be also detected. Inorganic to organic solids ratio evolution is inversely proportional to viscosity increase.

The regression coefficients of the equations relating the properties of spent liquors with the variables studied show that adjustments of all the equations are very good.

NSSC spent liquors characteristics results are presented in **Figures 13** to **16**.

Temperature and sulfite charges are the variables that show the greatest effect on consumed sulfite. Their influence is positive. Carbonate-Time interaction can be seen in Figure 13. At short cooking times, carbonate does not affect consumed sulfite, but its effect is strongly positive at long cooking times.

Inorganic solids and organic solids ratio has a minimum (optimum) value at 25 minutes and 181°C (Figure 14). Time and temperature variables are the most significant ones, and act lowering the ratio. Sulfite charge increases IS/OS, while carbonate charge does not influence this ratio.

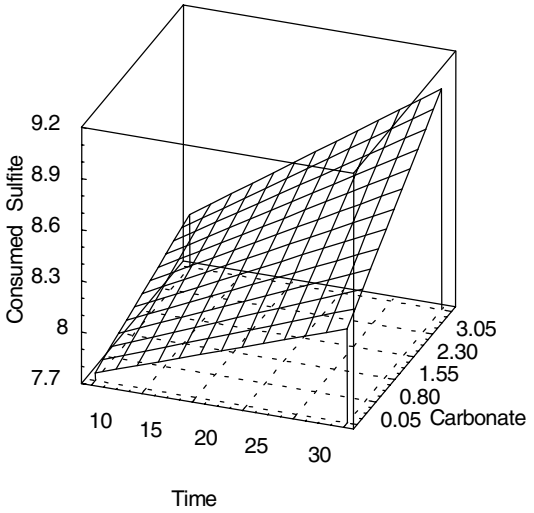


Figure 13. Consumed sulfite vs. cooking time and carbonate charge on wood (sulfite charge: 8%, temperature: 170ºC).

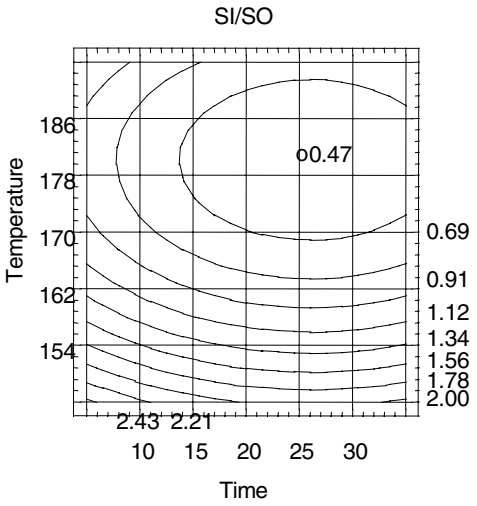


Figure 14. IS/OS ratio vs. cooking time and temperature (sulfite charge: 8% carbonate charge: 1.55%).

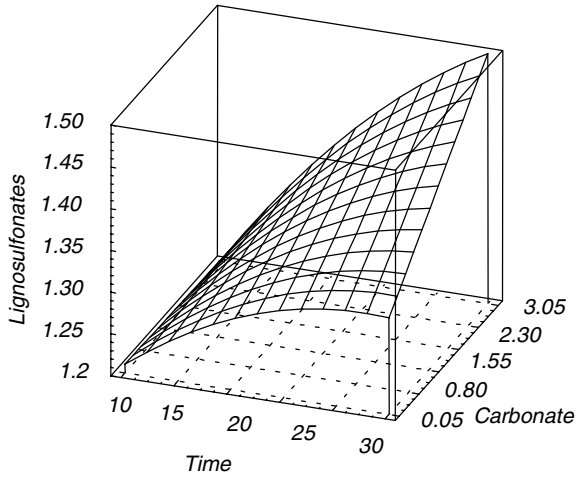


Figure 15. Lignosulfonates content in S. L. vs. time and carbonate charge. (sulfite charge: 8%, temperature: 170ºC).

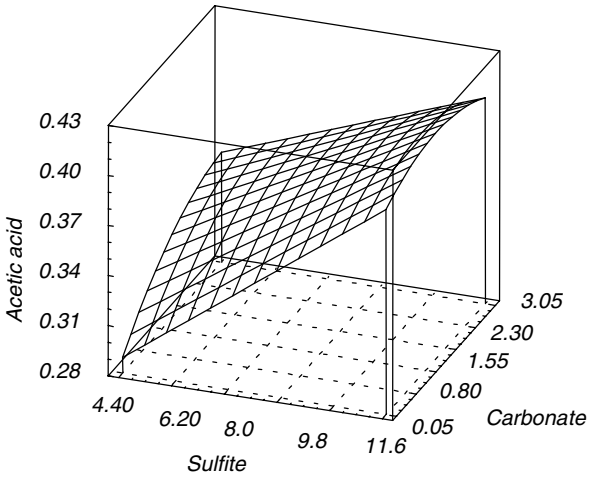


Figure 16. Acetic acid in spent liquors vs. sulfite and carbonate charges. (time: 20 min, temperature: 170ºC).

Dissolved lignosulfonates increase at higher temperatures and sulfite charges. The quadratic effect of time and its interaction with carbonate charge on wood is shown in Figure 15. While delignification reaches a limit value at short cooking times, it progresses continuously at longer times.

Temperature and sulfite charge shows the greatest influence on acetic acid generation during the cooking. Again, carbonate charge acts through interactions, this time with sulfite charge (Figure 16). While carbonate charge on wood exhibit an important effect on acetic acid formation, it does not evidence any effect at higher sulfite charges.

Chemical composition of several pulps was determined. Results show the selectivity of the process. Lignin dissolution proceeds at a higher rate than hemicelluloses solubilization in the range studied. However, NSSC is applicable only to the production of high-yield pulps, as the selectivity is very good at the beginning of the delignification.

Selection of best Runs

The resolution of the matrix of derived equations presented conditions near point 12 as the optimum, based on Productos Pulpa Moldeada reference. Optimum conditions minimizing the inorganic/organic solids ratio were: 26 min, 177ºC, 6.0% Na_2SO_3, 2.54% Na_2CO_3 (named "optimum"). Best conditions maximizing physical properties were: 30 min, 186ºC, 11.6% Na_2SO_3, 3.05% Na_2CO_3 (named "Alfa"). The points selected to verify the scaling up variations were: 25 min, 178ºC, 6.2% Na_2SO_3, 0.8% Na_2CO_3 (called "point 4 pilot plant"), and: 25 min, 178ºC, 6.2% Na_2SO_3, 2.3% Na_2CO_3 (called "point 12 pilot plant").

Pilot plant trials

The chemical composition of the raw material (Spanish hybrid poplar) can be found in reference 14. Results of physical tests on 60 g/cm^2 handsheets and board tests on 120 g/cm^2 handsheets of pilot plant pulps are presented in **Table I**

Run	4 pilot plant	12 pilot plant	Optimum	Alfa
Yield(%)	85.4	87.6	87.0	78.0
Time to 260 CSF	20	20	18	15
R30 (%)	4.74	5.38	5.87	5.72
30/50 (%)	1.8	1.83	1.7	2.32
50/100 (%)	1.33	1.03	0.8	0.73
100/150 (%)	0.32	0.22	0.12	0.12
150/270 (%)	0.22	0.29	0.22	0.22
P270 (%)	1.59	1.27	1.29	0.9
Tensile I. (N.m/g).	30.9	35.57	33.2	55.9
Burst I. (kPa.m2/g).	1.38	1.6	1.58	2.83
Tear I. (mN.m2/g).	3.73	3.87	4.08	4.16
Bulk (cm^3/g).	1.76	1.76	1.76	1.46
Sulfonic acid (mmol/kg)	47.2	58	57.05	82.7
Carboxylic acids (mmol/kg)	75.65	86.05	91.15	99.9
Total acid (mmol/kg)	122.85	144.05	148.2	182.6
RCT (kN/m)	1.31	1.24	1.19	1.36
CMT (N)	202	223	204	291
STFI (kN/m)	2.97	3.13	3.34	4.10

Table I. Pilot plant results.

Populus NSSC pulps showed in general lower physical properties than those of Eucalyptus species (2). This is obviously due to wood morphology, as poplars have weaker fibers (thinner walls) than eucalyptus.

Pilot plant yields were in general higher, and physical properties were lower than expected. Board strengths are acceptable. As Spanish poplar is not extremely dissimilar than Argentinean hybrid, these differences cannot be attributed to differences in raw materials. The sulfonic and carboxylic acid contents in pilot plant pulps were also lower than those of laboratory pulps. As predicted by the equations, the "Alfa" point conditions presented the best physical properties.

Yields determinations were made with the basket technique. Point "4 pilot plant" yield is abnormally low. This is surely due to experimental errors, as the other properties correspond better to a higher yield.

Correlations between sulfonic acids and the other responses were in general significant (yield: -0.85, tensile index: 0.94; bulk: -0.84). Concora medium tests correlations with other properties were also important (yield: -0.91; sulfonates: 0.94; carboxylates: 0.81; tensile index: 0.99). Ring crush showed similar results, and STFI compression test did not present any particular correlation.

CONCLUSIONS

- Temperature presents the most important effects on all the studied properties.
- Sodium carbonate effect on yield is important only at low sulfite charges or high temperatures.
- Sodium sulfite charge is the studied variable that has the highest influence on energy consumption, followed by temperature.
- Carbonate charge does not affect the responses in a great form as an independent variable, but it presents high interaction effects. Absence of a buffer in cooking liquors (Na_2CO_3 in this case) produces an increment of energy consumption and a reduction of pulps resistances.
- Independent variable values to obtain highest mechanical properties were located (by the equation matrix resolution), toward the maximum levels of the experimental plan (temperature of 186ºC, cooking time of 30 minutes, 11.6% of sodium sulfite and 3.05% of sodium carbonate).
- Pulp brightness decreases because of strong pulping conditions. If the pulp is going to be bleached, this means an increase in bleaching costs.
- These conditions imply an important yield reduction (80% to 72%).
- Optimum conditions minimizing the inorganic/organic solids ratio were: 26 min, 177ºC, 6.0% Na_2SO_3, 2.54% Na_2CO_3.
- Time, temperature and sulfite charge showed the greatest effects on organic materials dissolution.
- Carbonate charge acts principally through interactions.
- Pilot plant results showed higher yields and lower properties than expected. Board strengths are acceptable.

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