NSSC PROCESS OPTIMIZATION: I. PULPS QUALITY

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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. 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 minutes), 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 are: temperature of 186ºC, cooking time of 30 minutes, 11,6% of sodium sulfite and 3,05% of sodium carbonate on o.d. wood. These conditions imply important pulp brightness and yield reductions. 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/m2 handsheets and typical corrugated board tests on 120 g/m2 handsheets (CMT, RCT, STFI) were completed.

Keywords.

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

Introduction.

Kraft is unquestionably the most popular pulping process over the world. Current trends are however oriented to processes of smaller daily production capacity, that use smaller quantities of chemical products, or products that are not harmful to the environment.

Among "traditional" pulping processes, the neutral sulfite semichemical process (NSSC) survives and maintains its importance. Modern chemimechanical processes (CMP and CTMP) were developed on its chemical basis (1). Neutral sulfite semichemical 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), for example. Also bleached NSSC pulp is used to produce printing and writing papers, business forms, reply cards and tissue paper (5). It was also suggested that the NSSC is more flexible than Kraft pulping process: different chemical products can act as buffers, anthraquinone can be added, etc. It presents higher yields for similar resistance. NSSC pulps are easily bleached and they require less energy to be refined (2). Looking at F.A.O. projections (6), new installations of this pulping process will be required until the 21^{st} century

In neutral sulfite pulping a major drawback need to be resolved. In many cases, NSSC spent liquors are sent directly to water courses as 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.

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.

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 is 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. Chemical tests accomplished on wood are: extractives in hot water and in alcohol-benzene content (TAPPI standard), lignin content (Klasson), cellulose content (Seiffert), and ash content (TAPPI standard).

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). Variables studied were: cooking time; temperature; sodium sulfite, and sodium carbonate charges on wood.

Process conditions were:

- Time at maximum temperature: 10 to 30 minutes.
- Temperature: 154 to 186ºC.
- % $SO3^{=}$: 4 to 12% o.d.
- % $CO3^{=}$: 0 to 3% o.d.

Real and transformed variables according to the experimental design selected are presented in Table I.

Pulping methodology.

Pre-chemical stage: In order to reproduce the effect of the compression screw in the mill, a light mechanical stage was accomplished prior to the chemical one.

Table I: Experimental variables in real and transformed values

Run	Time	Temperature	Na_2SO_3	Na_2CO_3	Time	Temperature	Na_2SO_3	Na_2CO_3
1	15	162	6.20	0.80	-1	-1	-1	-1
2	25	162	6.20	0.80	1	-1	-1	-1
3	15	178	6.20	0.80	-1	1	-1	-1
4	25	178	6.20	0.80	1	1	-1	-1
5	15	162	9.80	0.80	-1	-1	1	-1
6	25	162	9.80	0.80	1	-1	1	-1
7	15	178	9.80	0.80	-1	1	1	-1
8	25	178	9.80	0.80	1	1	1	-1
9	15	162	6.20	2.30	-1	-1	-1	1
10	25	162	6.20	2.30	1	-1	-1	1
11	15	178	6.20	2.30	-1	1	-1	1
12	25	178	6.20	2.30	1	1	-1	1
13	15	162	9.80	2.30	-1	-1	1	1
14	25	162	9.80	2.30	1	-1	1	1
15	15	178	9.80	2.30	-1	1	1	1
16	25	178	9.80	2.30	1	1	1	1
17	10	170	8.00	1.55	-2	0	0	0
18	30	170	8.00	1.55	2	0	0	0
19	20	154	8.00	1.55	0	-2	0	0
20	20	186	8.00	1.55	0	2	0	0
21	20	170	4.40	1.55	0	0	-2	0
22	20	170	11.60	1.55	0	0	2	0
23	20	170	8.00	0.05	0	0	0	-2
24	20	170	8.00	3.05	0	0	0	2
25	20	170	8.00	1.55	0	0	0	0
26	20	170	8.00	1.55	0	0	0	0
27	20	170	8.00	1.55	0	0	0	0
28	20	170	8.00	1.55	0	0	0	0
29	20	170	8.00	1.55	0	0	0	0

Chemical stage: It was carried out in a 4-lt. stainless steel digester, having a vacuum connection and two heating systems. The primary indirect heating system consisted in saturated steam, using always a steam temperature near to the set point one. The second heating device was a liquor recirculation piping with a heat exchanger, consisting in four 600 watts heating wires controlled independently one from the other.

Temperature measurements were accomplished with a controlled PT 100 thermo-resistance. Time at maximal temperature was calculated on line with a software attachment, using the H factor at time intervals of 0.5 min (7). The activation energy used was 32 000 cal/mole. It was calculated from values obtained for NSSC cooks at long and short times (8).

Cooking liquors were prepared from concentrated solutions of sodium sulfite and sodium carbonate. 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. Intensive and standardized washings were done previous the last weighting. As duplicates of yield values showed great dispersions, organic materials balances were made in spent liquors to recalculate them (9).

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 60 ºC. 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 pulps yields corresponded to Productos Pulpa Moldeada mill conditions for thin bleached boards. The time required to reach to 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 Mac Nett fiber classification, physical tests on 60 g/m^2 handsheets (tensile, burst and tear strength, bulk, and brightness), all using TAPPI standards.

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 (Table III). 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. They were all cooked at 178 ºC. Sulfonic and carboxylic acids contents were determined on them using conductimetry (10). 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 liters digester and a 12" Sprout Waldrom 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*) was:

- Extractives in hot water: 2.3%
- Extractives in alcohol-benzene: 2.6 %
- Lignin content (Klasson): 24.2 %
- Lignin content (soluble): 3.0 %
- Cellulose content (Seiffert): 41.8 %
- Hemicelluloses (difference): 26.1 %
- Ash content: 0.84%

Table II presents the obtained results. The coefficients of the equations concerning the responses variations in function of independent variables are presented in Table III. The correlation coefficients between responses are shown in Table IV. Yield, energy, and physical properties are plotted in function of the most significant parameters for each property in Figures 1 to 6.

Table II: Cooking results and pulp properties (260 CSF)

Run	**Yield** (%)	**Energy** (MJ/kg)	**Initial CSF (ml)**	**Shives** (%)	**Time to 260 CSF**	**R30** (%)	**30/50** (%)	**50/100** (%)	**100/150** (%)	**150/270** (%)	**P270** (%)	**Brightness** (%)	**Tensile I.** (N.m/g).	**Burst I.** (kPa.m^2/g).	**Tear I.** (mN.m^2/g)	**Bulk** (cm^3/g
1	92.0	0.41	560	3.96	32.0	0.49	2.26	3.27	1.09	1.00	1.39	53.8	18.2	0.73	1.46	2.15
2	90.8	0.88	638	5.96	36.0	2.21	2.85	3.03	0.52	0.73	0.66	52.1	23.9	0.94	2.08	2.03
3	88.5	1.04	545	2.48	20.5	1.62	3.09	2.91	0.87	1.07	0.44	46.7	24.5	1.16	2.33	1.80
4	86.2	0.97	435	2.84	10.5	2.87	3.39	1.51	0.94	0.60	0.69	44.0	36.8	1.87	3.86	1.79
5	87.2	0.87	560	4.60	18.5	1.50	3.60	2.92	0.54	1.20	0.24	54.7	24.6	1.09	2.41	2.01
6	85.9	0.87	575	3.52	21.5	1.84	3.07	3.00	1.05	1.03	0.01	53.4	30.6	1.47	2.25	1.90
7	83.5	0.54	516	3.68	14.0	2.16	3.02	2.62	0.92	0.67	0.61	48.8	34.9	1.91	3.49	1.62
8	81.8	0.55	461	3.24	10.0	2.14	2.97	2.02	0.58	0.72	1.57	48.1	44.4	2.22	3.33	1.62
9	91.4	1.03	502	3.20	10.5	1.28	2.87	2.23	1.49	0.92	1.21	53.4	25.6	1.12	2.64	2.11
10	88.2	0.74	575	4.04	20.0	1.58	3.43	2.86	0.99	0.82	0.32	49.7	26.5	1.33	2.56	1.85
11	85.2	0.55	622	14.96	11.0	3.89	3.49	1.06	0.55	0.59	0.42	44.5	35.1	1.57	3.16	1.77
12	82.3	0.55	590	11.64	10.0	5.17	2.57	0.85	0.45	0.43	0.53	41.5	46.2	2.10	3.58	1.63
13	91.1	0.71	502	3.84	13.0	2.14	3.26	2.38	0.93	1.02	0.70	52.0	28.0	1.39	2.75	1.85
14	86.0	0.61	530	3.80	14.5	2.34	2.97	2.32	0.83	1.42	0.12	50.0	37.2	1.90	3.05	1.70
15	82.1	0.44	606	9.24	8.5	5.15	2.70	0.97	0.38	0.45	0.35	47.0	43.4	2.41	3.61	1.61
16	79.9	0.60	461	4.08	10.5	2.65	3.43	2.30	0.65	0.68	0.29	44.4	53.2	2.72	3.34	1.52
17	90.2	1.05	606	3.60	24.0	2.05	3.57	2.49	0.78	0.92	0.19	52.6	24.9	1.27	2.47	1.84
18	86.1	0.64	502	3.28	12.0	2.53	2.94	2.28	0.79	0.91	0.55	48.8	39.6	1.90	3.51	1.72
19	93.9	0.87	590	4.80	24.0	0.90	3.24	2.93	1.07	1.07	0.79	55.9	19.7	0.86	1.93	2.19
20	82.1	0.61	461	3.36	9.0	3.44	2.92	1.40	0.51	0.56	1.17	44.1	46.4	2.23	3.61	1.62
21	91.0	0.88	530	2.48	19.0	0.97	2.97	3.33	1.06	1.23	0.44	47.7	25.4	1.27	2.27	1.86
22	84.4	0.56	502	2.92	9.0	3.07	3.57	1.59	0.81	0.82	0.14	49.1	43.3	2.40	3.35	1.60
23	88.8	1.02	575	2.40	25.5	1.21	2.04	4.09	0.84	1.21	0.61	49.6	29.7	1.57	2.62	1.77
24	87.1	0.81	474	2.48	11.0	2.41	1.36	3.48	1.14	0.67	0.94	50.0	40.5	1.98	3.17	1.69
25	86.3	0.70	516	3.80	15.0	1.05	2.02	3.48	2.45	0.64	0.36	50.4	45.1	2.20	3.07	1.68
26	86.8	0.46	575	6.76	17.0	2.23	3.40	2.50	0.66	0.74	0.47	50.8	34.4	1.57	2.98	1.93
27	86.4	0.54	575	6.40	17.0	1.84	3.41	2.15	0.63	0.71	1.26	49.0	38.2	1.78	2.80	1.79
28	86.2	0.62	560	5.89	19.0	1.27	4.16	2.13	0.63	0.79	1.02	50.2	40.2	1.86	2.79	1.81
29	-	0.62	545	5.88	15.5	3.08	3.91	1.24	0.58	0.58	0.61	51.3	35.0	1.66	2.63	1.88

Table III: Coefficients of the regression equations between dependent and independent variables.

	Yield	**Energy**	**Initial CSF**	**Time to 260 CSF**	**Tensile I.**	**Burst I.**	**Tear I.**	**Bulk**	**Brightness**
Independent term	86.8	192.6	22.4	16.5	34.3	1.67	2.87	1.80	49.9
Time (A)	-1.2	-16.2	-	-	3.9	0.37	0.18	-	-1.0
Temperature (B)	-2.8	-24.6	1.4	-4.2	6.6	0.73	0.45	-0.14	-3.2
Sulfite (C)	-1.7	-27.2	-	-2.5	4.0	0.55	0.20	-0.08	0.6
Carbonate (D)	-0.5	-23.7	-	-3.9	3.3	0.33	0.19	-0.04	-0.8
AB	-	-	2.3	-	-	-	-	-	-
AC	-	-	-	-	-	-	-0.17	-	-
AD	-0.4	-	-	-	-	-	-	-	-
BD	-0.7	-	-2.3	2.2	-	-	-0.13	-	-
CD	0.7								
D^2	-	15.9	-	-	-	-	-	-	-
R^2	**0.94**	**0.67**	**0.58**	**0.75**	**0.88**	**0.89**	**0.87**	**0.79**	**0.93**

Table IV: Correlation coefficients between responses

	Yield	Energy	Time to 260 CSF	Tensile I.	Burst I.	Tear I.	Bulk	Brightness
Yield	1.00	0.72	0.67	-0.89	-0.89	-0.80	0.86	0.70
Energy	0.72	1.00	0.60	-0.73	-0.70	-0.67	0.62	-
Time to 260 CSF	0.67	0.60	1.00	-0.73	-0.75	-0.85	0.67	-
Tensile Index	-0.89	-0.73	-0.73	1.00	0.97	0.84	-0.86	-0.67
Burst Index	-0.89	-0.70	-0.75	0.97	1.00	0.86	-0.92	-0.65
Tear Index	-0.80	-0.67	-0.85	0.84	0.86	1.00	-0.82	-0.73
Bulk	0.86	0.62	0.67	-0.86	-0.92	-0.82	1.00	0.74
Brightness	0.70	-	-	-0.67	-0.65	-0.73	0.74	1.00

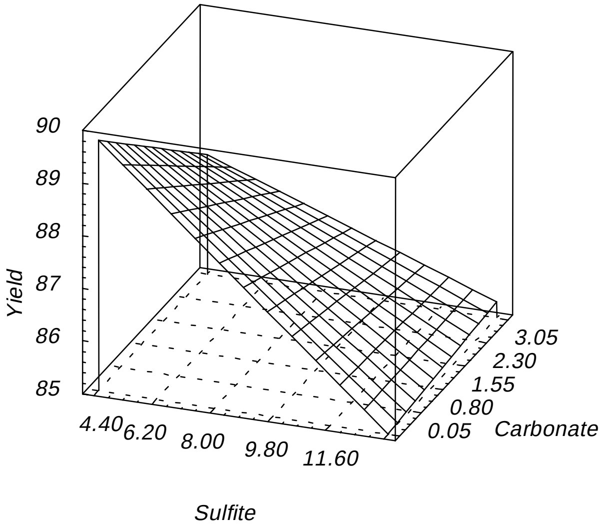

Figure 1: Variation of yield with sulfite and carbonate charges.

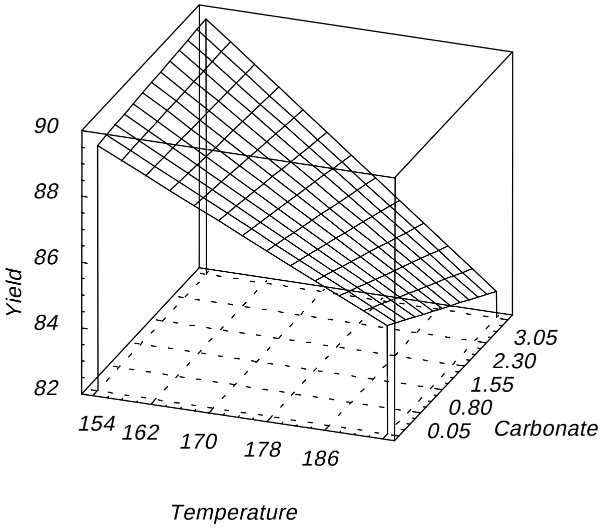

Figure 2: Variation of yield with temperature and carbonate charge.

The derived equations show a very good adjustment with experimental data (R^2 in Table III). Temperature exhibits the greater influence on yield and physical properties. Sulfite charge is the most important parameter for energy.

Bauer Mac Nett pulps fractions show a low correlation with physical properties, excluding R30 mesh fraction that possesses a correlation coefficient of R =0.7 with burst.

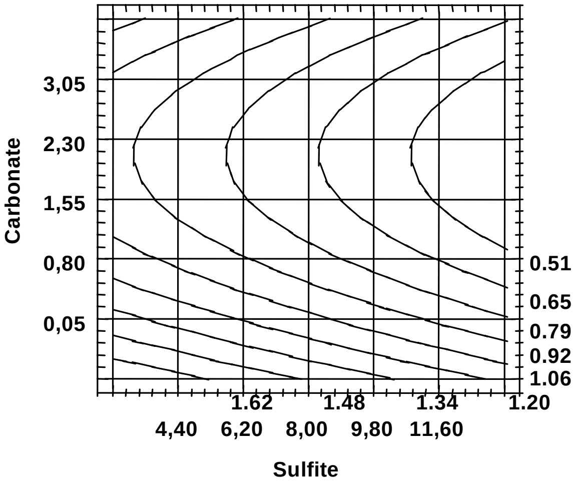

Figure 3: Energy variation with sulfite charge and carbonate charge.

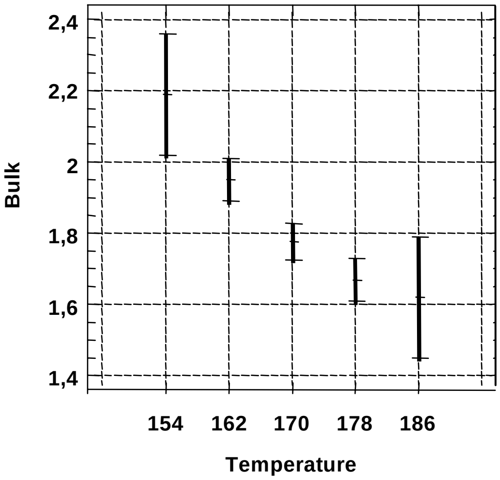

Figure 4: Bulk vs. Temperature

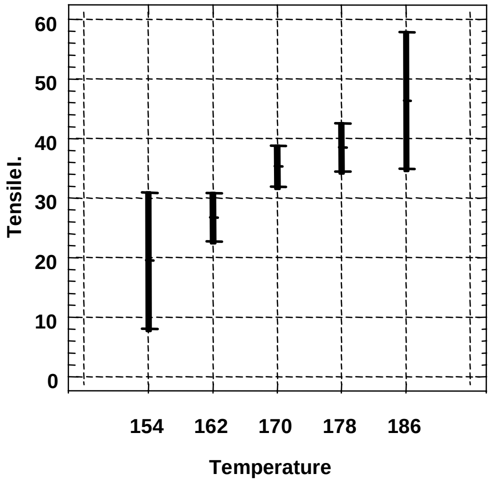

Figure 5: Tensile Index vs. Temperature.

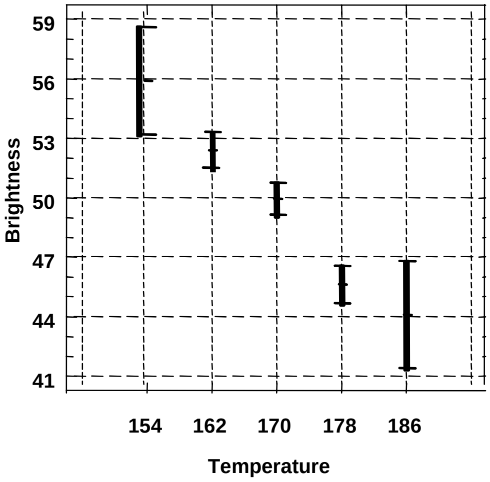

Figure 6: Brightness vs. temperature

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 (Table III). 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 (11) indicates that yields obtained at 154 and 162 ºC behave as a homogeneous group. The same reasoning was valid at 178 and 186 ºC.

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 as a consequence 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 (12). 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 (see regression coefficients of their equations in Table III).

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 seem 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 (Table III) is illustrated in Figure 6.

Sulfonic and carboxylic acids content in pulps.

Table V presents the sulfonic and carboxylic acids in pulps.

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

Table V: Sulfonation and carboxylation degree of the selected pulps.

Run	Sulfonic acids (mmol/kg)	Carboxylic acids (mmol/kg)	Total acids (mmol/kg)
3	67.5	97.6	104.1
4	64.6	88.6	153.2
7	91.2	107.4	198.6
8	93.9	103.1	197.0
11	68.5	107.2	175.7
12	65.5	95.0	160.5
15	91.0	110.6	201.6
16	127.1	106.9	234.0

Table VI: Mean effects of variables for sulfonic and carboxylic acids in pulps (factorial design, temperature: 178 ºC).

	Sulfonic acids	**Carboxylic acids**
Independent term	83.65	102.0
Time (A)	4.1	-3.7
Sulfite (B)	17.1	4.9
Carbonate (C)	4.4	2.9
AB	5.6	-
AC	4.2	-
BC	3.9	-

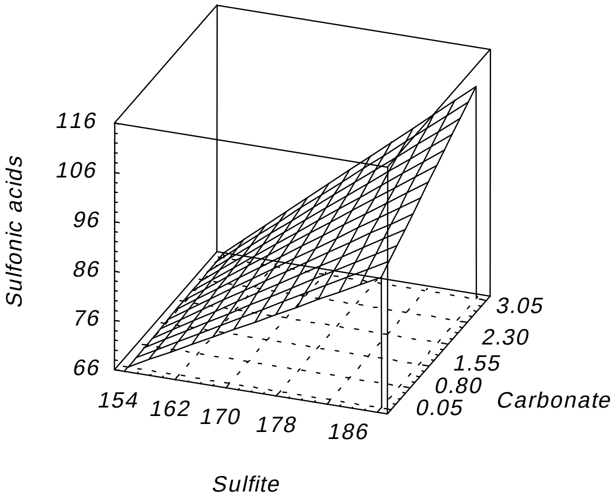

Figure 7: Sulfonic acids content in pulps
(Sulfite-Carbonate interaction)

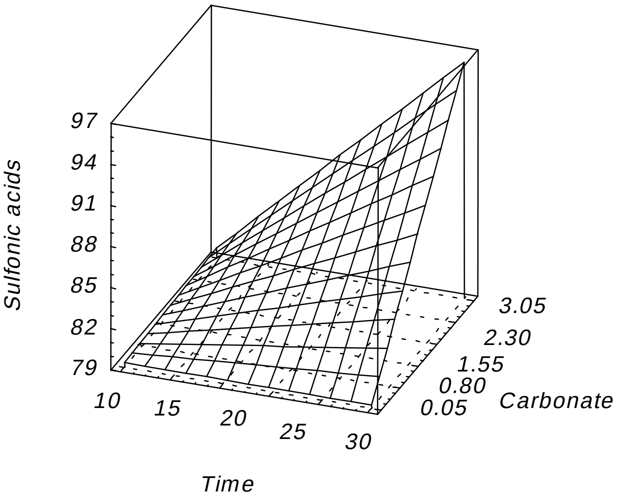

Figure 8: Sulfonic acids content in pulps
(Time-Carbonate interaction)

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.

Selection of best Runs

The resolution of the matrix of founded equations presented conditions near point 12 as the optimum, based on Productos Pulpa Moldeada reference. Optimum conditions minimizing the inorganic/organic solids ratio (13) were: 26 min, 177 ºC, 6.0 % Na_2SO_3, 2.54% Na_2CO_3 (named "optimum"). Best conditions maximizing physical properties were: 30 min, 186 ºC, 11.6 % Na_2SO_3, 3.05% Na_2CO_3 (named "Alfa"). The points selected to verify the scaling up variations were: 25 min, 178 ºC, 6.2 % Na_2SO_3, 0.8% Na_2CO_3 (called "point 4 pilot plant"), and: 25 min, 178 ºC, 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*) was:

- Extractives in hot water: 2.67%
- Extractives in alcohol-benzene content: 1.94 %
- Lignin content (Klasson): 22.9 %

- Lignin content (soluble): 1.91 %
- Cellulose content (Seiffert): 43.3 %
- Hemicelluloses (difference): 27.5 %

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 VII.

Table VII: Pilot plant results

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 acids (mmol/kg)	47.2	58	57.05	82.7
Carboxylic acids (mmol/kg)	75.65	86.05	91.15	99.9
Total acids (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

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. As Spanish poplar is not extremely dissimilar than Argentinean hybrid, these differences can not 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 corerlations 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 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).
- Pulp brightness decreases as a consequence 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%).
- Pilot plant results showed higher yields and lower properties than expected.

Literature cited.

1. von Koeppen, A., Paper Trade J. "Chemimechanical Pulps from Hardwood Using the NSSC [Neutral Sulfite Semichemical] Process", 170, no. 11: 49-51 (Nov. 1986).
2. Phillips, F. H.; Logan, A. F.; Balodis, V. and Ward, J. V., Appita J. "Studies on Pulpwood Resources in Southern Tasmania. (2). Kraft-AQ, Soda-AQ, and NSSC Pulps", 44, no. 3: 173-178 (1991).
3. Odom, J. J., TAPPI Pulping Conf. (Orlando) "Sulfite Pulping: Probable Replacement of the Kraft Process", (Book 2): 597-602 (Nov. 3-7, 1991).
4. Farrington, A. and Hickey, B. L., Appita J. "Wood Sources for the Port Huon Mill: NSSC Pulping of Some Young Eucalypt Species", 42, no. 6: 419-423 (Nov. 1989).
5. NLK Consultants, Technology Update "BSCP Process. Potential for Commercial Application", 1(2): 4-6 (Dec. 1992).
6. FAO, Enquête 1995-2000 "Capacités de la pâte et du papier", , Rome 1996.
7. Lovelady, J. S., TAPPI Pulping Conf. (Orlando) "NSSC Hardwood Yield and Yield Measurement and Forecast: Mill's Perspective", (Book 2): 603-612 (Nov. 3-7, 1991).
8. Wilder, H.D. and Han, S.T. Tappi J."A Comparison of the Kinetics of the Neutral Sulfite and Kraft Pulping Processes", 45 (1): 1-9 (1962)
9. Area, M.C., Felissia, F.E., Nuñez, C.E., Venica, A. and Valade, J.L., Symposium: « Structure and Properties of Lignocellulosic Materials », "Upgrading Spent Liquors from NSSC Process", Fifth Chemical Congress of North America, Cancun, Nov. 11-15, 1997.
10. Katz, S., Beatson, R.P., and Scallan, A.M., Sven. Papperstidn "The determination of strong weak acidic groups in sulfite pulps", 6:R48-53, 1984.
11. Walpole, R.E. and Myers, R.H., "Probability and statistics for Engineers and scientists", Mc Millan Publishing Company, forth edition, 1992.
12. Area, M.C. and Valade, J.L., ATIPCA "Aspectos químicos del pulpado CTMP de latifoliadas", 32 (1): 29-32, 1995.
13. Area, M.C., Felissia, F.E., Venica, A. and Valade, J.L., "NSSC process optimization: II. Spent liquors", unpublished, 1997.

Aknowledgments.

Reference values: Ricardo Repetti, Productos Pulpa Moldeada., Cipolleti, Río Negro, Argentina.
Board tests: Instituto de Tecnología Celulósica, UNL, Santa Fe, Argentina.
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