# Pressurized grinding of chemically treated wood

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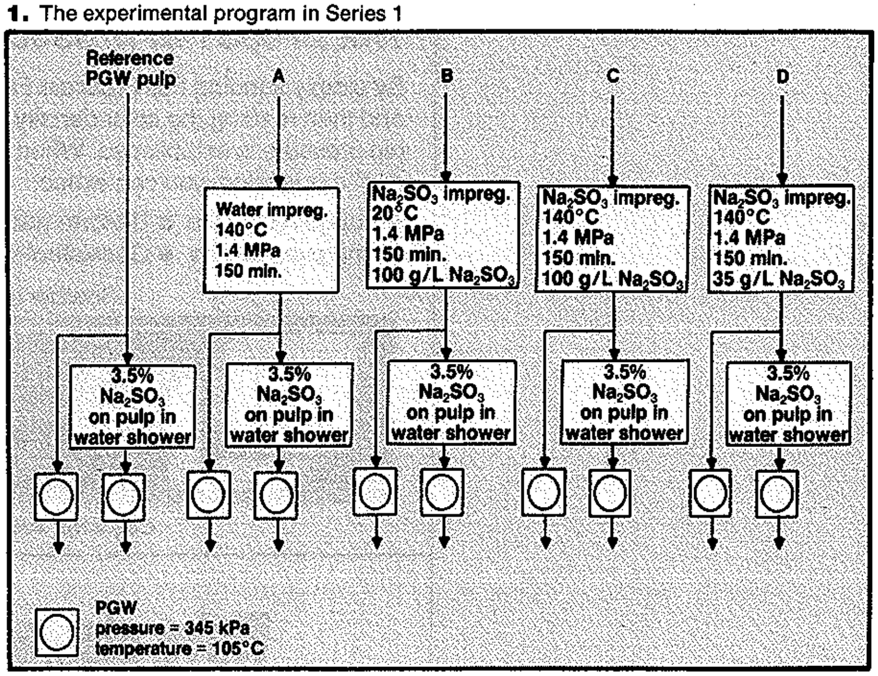
**ABSTRACT** *Mild sulfonation of softwood prior to mechanical defibration yields pulps with enhanced strength properties. Development of the chemigroundwood process has been hampered by the dimensions of the raw material to be impregnated. This study shows that impregnation with sodium sulfite solutions at elevated temperature is successful at short impregnation times. Pressure grinding of the mechanically treated wood did produce CPGW pulps that in many respects are comparable to CTMP. Compared to the reference PGW pulps, the CPGW pulps had a higher long fiber content and 50–100% higher tensile and tear strengths, but optical properties suffered, as expected.*

**KEYWORDS**
Chemical analysis
Grinding
Mechanical properties
Impregnation
*Picea abies*
Sulfonation

**1.** The experimental program in Series 1




Interest among paper and linerboard manufacturers in utilizing chemically pretreated, pressurized groundwood (CPGW) is limited. There are a few exceptions, however, such as grinding of hardwoods preimpregnated with sodium carbonate. The principal drawback of the CPGW process as compared with the CTMP process is the long impregnation time required as a result of the size and form of the raw material, which is logs.

Impregnation of wood with alkaline sodium sulfite is relatively easy compared with impregnation of wood with white liquor. Sulfonation takes place at a rather low temperature, and sodium sulfite has proven to be a suitable chemical for producing mechanical pulps, especially CTMP.

Studies on impregnation of Finnish spruce with sodium sulfite followed by pressure grinding of the impregnated wood raw material have been performed on a laboratory scale at the Finnish Pulp and Paper Research Institute. A method for determining the effectiveness of the impregnation under different conditions was developed in conjunction with the study.

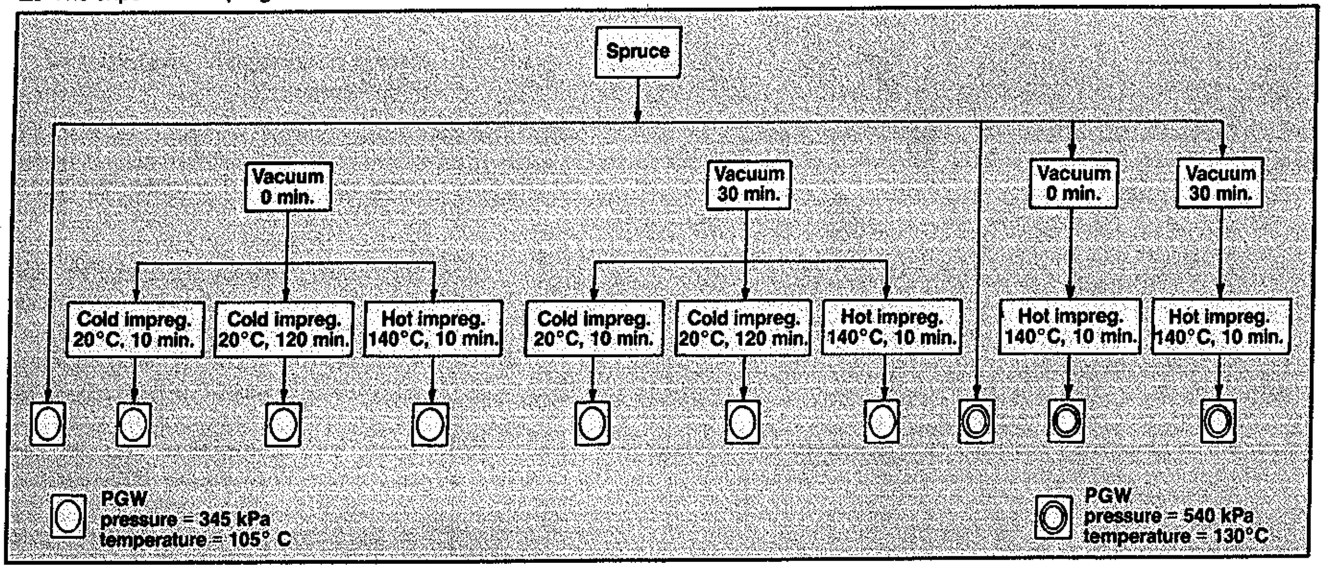
## Experimental Program

Impregnation of wood and the subsequent grinding of the sulfonated raw material were studied in two different experimental series. In the first series, comparisons were made between impregnation with hot water, cold sulfite, and hot sulfite at two different concentrations. In addition, the influence of sulfite-containing shower water on the quality of pulps produced as PGW or CPGW was studied. In this series, a long impregnation time of 150 min was used. The experimental program is given in **Fig. 1**.

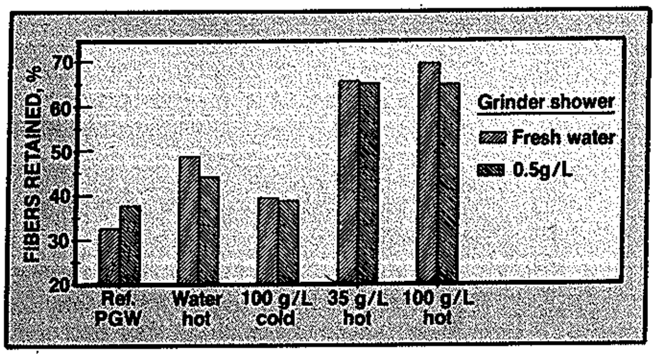
As shown in **Fig. 2**, four variables were studied in the second series:

1. Vacuum treatment of wood in the impregnation vessel

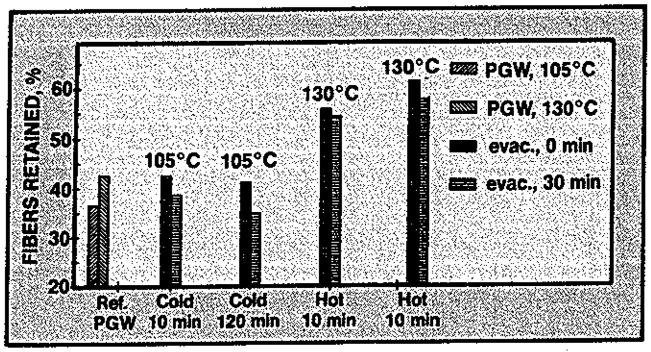
**2.** The experimental program in Series 2



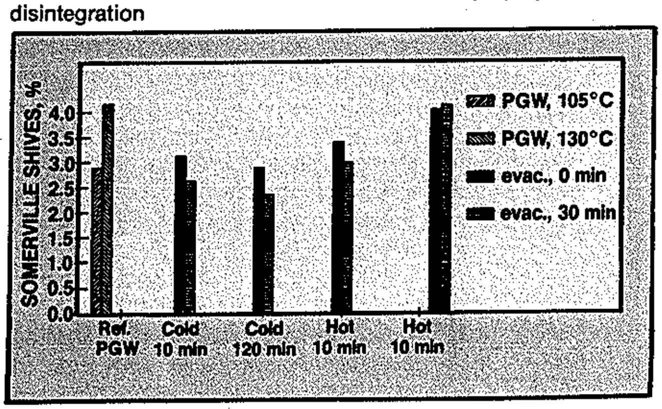

**3.** Bauer-McNett long fiber fractions of reference and CPGW pulps in Series 1



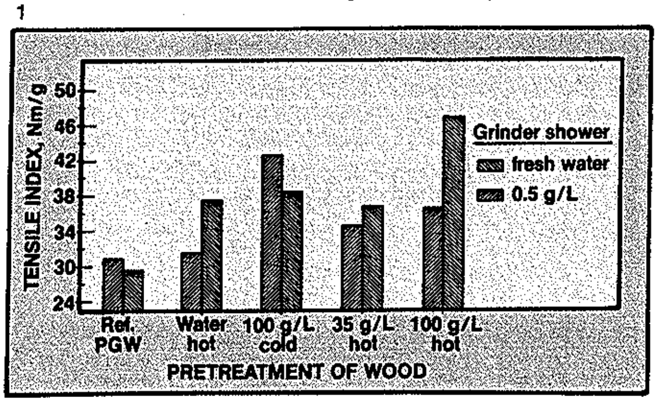

**4.** Bauer-McNett long fiber fraction of reference and CPGW pulps in Series 2




**5.** Somerville shives of the PGW and CPGW pulps prior to hot disintegration




**6.** Tensile index measured on 52-g/m² laboratory sheets in Series 1




2. Impregnation temperature
3. Impregnation time
4. Grinding temperature.

## Results

### Sulfur content of impregnated wood and CPGW pulp

Data on analyses of the sulfur content of wood after impregnation and also on the washed pulp in Series 1 are given in **Table I**. The sulfite liquor readily impregnated the wood raw material. The sodium sulfite charge at the two concentrations of 35 g/L and 100 g/L (140°C) resulted in 3% and 9% sulfite contents in the wood respectively, calculated as $Na_2SO_3$ on o.d. wood.

The sulfur contents in the washed pulps were 0.9% and 2.0%, respectively, expressed as a percentage of sodium sulfite on o.d. pulp. A high sulfite charge was achieved with cold impregnation liquor (100 g of $Na_2SO_3$ /L)—that is, 7% sodium sulfite on o.d. wood. Washed pulp, however, contained only 0.7% sodium sulfite.

Using sodium sulfite in the shower water (3.5% on wood) increases the sulfur content of the washed pulp. The amount corresponds to sodium sulfite

**I. Sodium sulfite content of impregnated wood and washed pulp, Series 1**

| | | Impregnation | | Grinding |
|---|---|---|---|---|
| Trial | Treatment | $Na_2SO_3$ dosage, g/L | Analyzed $Na_2SO_3$, % o.d. wood | Analyzed $Na_2SO_3$, % o.d. pulp |
| 1 | Ref. PGW | 0 | ... | 0.04 |
| 2 | Ref. PGW* | 0 | ... | 0.24 |
| 3 | Hot water | 0 | 0.1 | 0.04 |
| 4 | Hot water* | 0 | 0.1 | 0.24 |
| 5 | Cold sulfite | 100 | 7.4 | 0.67 |
| 6 | cold sulfite* | 100 | 7.4 | 0.79 |
| 7 | Hot sulfite | 35 | 3.3 | 0.94 |
| 8 | Hot sulfite* | 35 | 3.3 | 1.06 |
| 9 | Hot sulfite | 100 | 8.7 | 2.00 |
| 10 | Hot sulfite* | 100 | 8.7 | 2.08 |

*Sulfite at a concentration of 0.5 $Na_2SO_3$/L was added to the shower water.

**II. Sodium sulfite content of impregnated wood and washed pulp, Series 2**

| | Impregnation | | | Grinding | |
|---|---|---|---|---|---|
| Trial | Time, min | Temp., °C | Analyzed $Na_2SO_3$, % o.d. wood | Analyzed $Na_2SO_3$, % o.d. pulp | $CH_2Cl_2$ extract, % |
| | | | | Shower water 105°C | |
| 1 | Ref. PGW | ... | 0.07 | 0.04 | 0.48 |
| 2 | 10 | 20 | 3.5 | 0.56 | 0.40 |
| 3 | 120 | 20 | 4.8 | 0.67 | 0.40 |
| 4 | 10 | 140 | 4.5 | 0.91 | 0.39 |
| 5 | 10* | 20 | 4.2 | 0.51 | 0.30 |
| 6 | 120* | 20 | 5.3 | 0.59 | 0.32 |
| 7 | 10* | 140 | 6.0 | 0.99 | 0.33 |
| | | | | Shower water 130°C | |
| 8 | Ref. PGW | ... | ... | 0.04 | 0.51 |
| 9 | 10 | 140 | 4.6 | 0.99 | 0.35 |
| 10 | 10* | 140 | 5.6 | 0.95 | 0.43 |

* The wood was vacuum treated prior to impregnation.

contents of 0.2% and 0.1–0.15%, depending on whether untreated or sulfonated wood was ground.

Experiments carried out in the second series (**Table II**), show that vacuum treatment of the wood in the impregnation vessel slightly improves the cold impregnation results (Experiments 2, 3, 5, and 6).

The effect on impregnation efficiency is stronger when impregnating with hot liquor (Experiments 4, 7, 9, and 10). Increasing the impregnation time improves performance when impregnating with cold liquor. The best impregnation results in the study were achieved with hot liquor, as reflected specifically in the sodium sulfite content of the washed pulps, with the numerical results being in the range of 1% sodium sulfite on wood.

In the best cases, dichloromethane extractives were reduced from a concentration of 0.5% to 0.3%.

**Energy consumption properties of CPGW pulps**

Untreated and chemically treated wood samples had roughly the same specific energy consumption under similar process conditions—1.1±0.2 MW·h/ton (**Tables III and IV**). Because the number of experiments was restricted, no attempt was made to optimize the grinding of the chemically treated wood. The freeness of the CPGW pulps produced in the first series was thus higher than for the reference PGW pulps—300–400 mL and 100–150 mL, respectively. Also, in the second series pulp produced through hot impregnation had a higher freeness than the other pulps in the series, at 200–350 mL compared to 150–250 mL.

The results show that sulfonation of the wood raw material reduces the fiber-to-fiber bonding. The CPGW pulps produced a large amount of unbroken fiber and a small amount of fine fraction material.

**Fiber properties of PGW and CPGW pulps**

Impregnation of the wood raw material, especially hot impregnation, increased the amount of long fibers. In Bauer-McNett classification, the upper fraction, retained by a 595-μm screen (28 mesh), increased from 40% to 65%. The middle fraction, passing through a 595-μm screen but retained by a 74-μm screen (200 mesh), was reduced by about 50% from the original level of 40%. The fines fraction, passing through a 74-μm screen, was reduced from 25% to 15% on average. These percentages are valid for the first test series (**Fig. 3**).

In the second test series, the influence of hot impregnation was reflected as an increase in the long fiber fraction as compared with data for the PGW reference pulps. The long fiber fraction corresponded to 55–60% and 35–45%, respectively, for the PGW pulps (**Fig. 4**).

The hot impregnation did have a surprisingly modest influence on the amount of Somerville shives, especially in light of the corresponding Bauer-McNett analyses data (**Fig. 5**). The use of hot shower water (130°C) during the grinding operation did, however, increase the shives content by 1%. The Somerville shives contents

**III.** Experiment results of pressure grinding of sodium sulfite impregnated wood, Series 1

| | *Impregnation* | | *Grinding* | |
|---|---|---|---|---|
| *Trial* | *Treat-ment* | *$Na_2SO_3$ dosage, g/L* | *Sp. energy consump-tion, MW-h/ton* | *Freeness, mL CSF* |
| 1 | ref. | . . . | 1.09 | 110 |
| 2 | ref. | . . . | 1.02 | 163 |
| 3 | water | . . . | 1.19 | 192 |
| 4 | water | . . . | 1.30 | 142 |
| 5 | cold | 100 | 1.33 | 95 |
| 6 | cold | 100 | 1.09 | 109 |
| 7 | hot | 35 | 0.91 | 414 |
| 8 | hot | 35 | 0.96 | 293 |
| 9 | hot | 100 | 0.91 | 348 |
| 10 | hot | 100 | 0.97 | 368 |

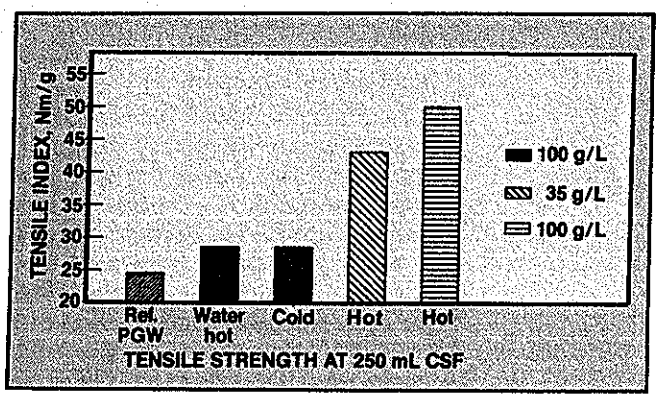
Sulfite at a concentration of 0.5 g $Na_2SO_3$/L was added to the shower water.

**IV.** Experiment results of pressure grinding of sodium sulfite impregnated wood, Series 2

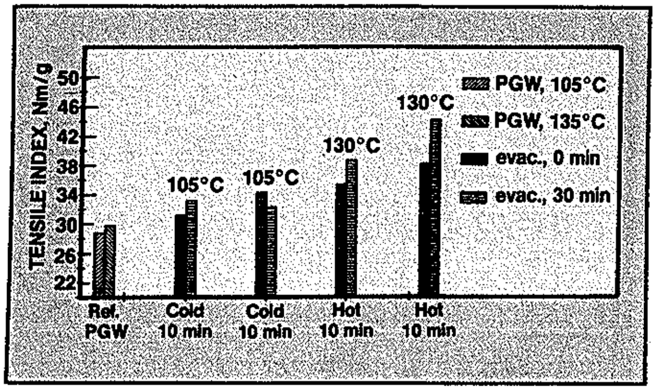
| | *Impregnation* | | *Grinding* | |
|---|---|---|---|---|
| *Trial* | *Time, min* | *Temp., °C* | *Sp. energy consump-tion, MW-h/ton* | *Freeness, mL CSF* |
| | | | Shower water 105 °C | |
| 1 | ref. | . . . | 1.08 | 203 |
| 2 | 10 | 20 | 1.02 | 238 |
| 3 | 120 | 20 | 1.03 | 185 |
| 4 | 10 | 140 | 1.05 | 344 |
| 5 | 10* | 20 | 1.10 | 165 |
| 6 | 120* | 20 | 0.94 | 203 |
| 7 | 10* | 140 | 0.97 | 341 |
| | | | Shower water 130 °C | |
| 8 | ref. | | 0.97232 | |
| 9 | 10 | 140 | 0.97 | 311 |
| 10 | 10* | 140 | 1.13 | 204 |

*The wood was vacuum treated prior to impregnation.

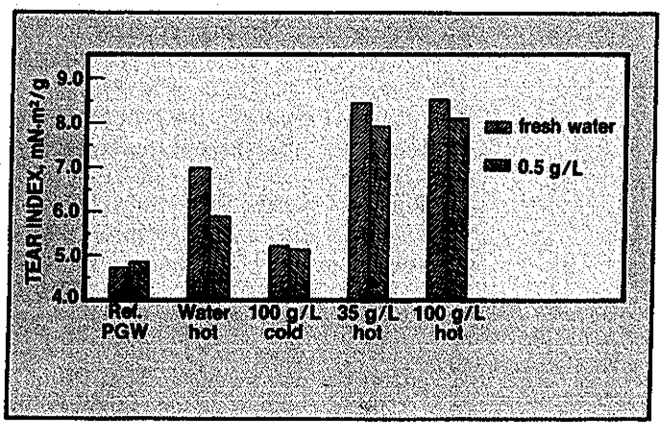
**7.** Extrapolated tensile strength values of pulps in Series 1



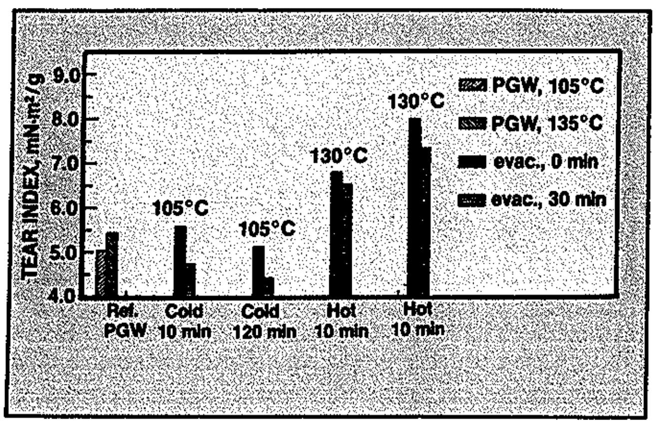

**8.** Tensile index measured on 52-g/m² laboratory sheets in Series 2



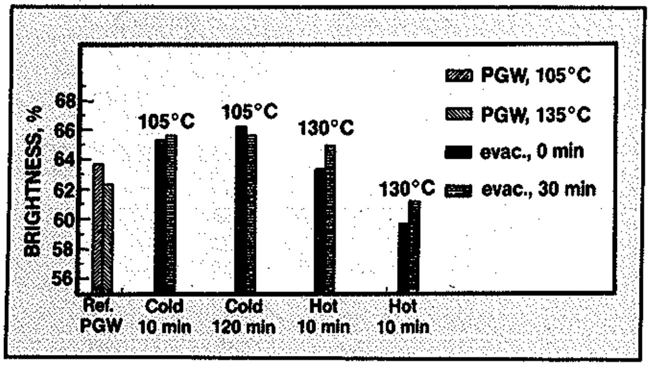

**9.** Tear index measured on 52-g/m² laboratory sheets in Series 1



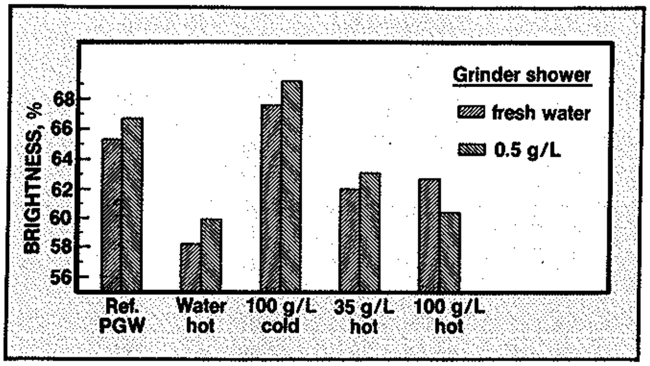

**10.** Tear index measured on 52-g/m² laboratory sheets in Series 2



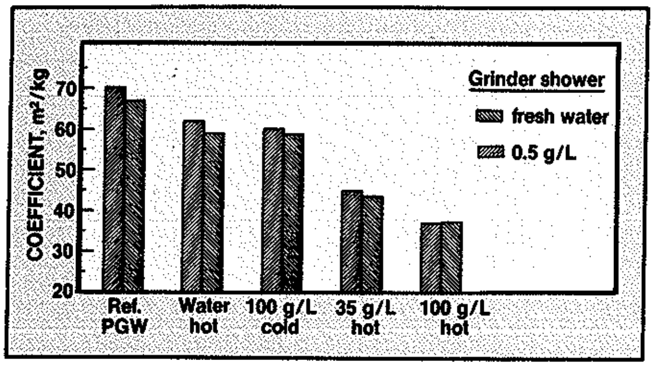
**11.** Brightness of the PGW and CPGW pulps in Series 2



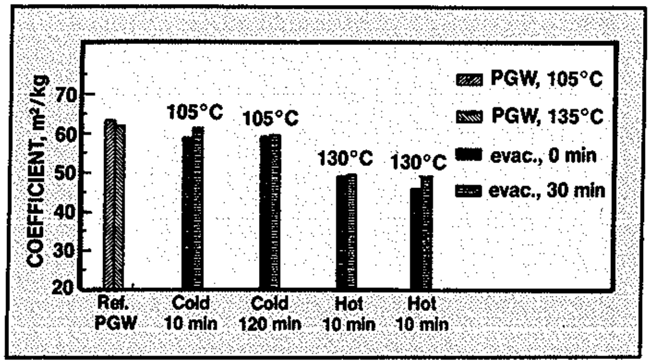

**12.** Brightness of the PGW and CPGW pulps in Series 1



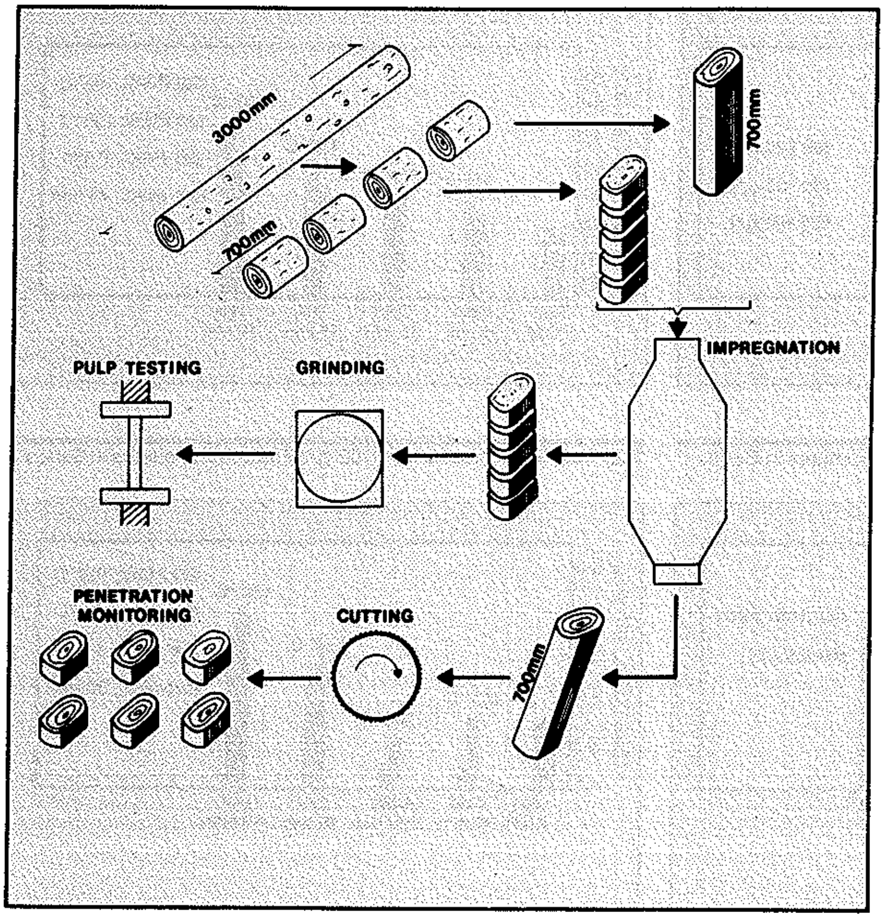

**13.** Light-scattering coefficient of the PGW and CPGW pulps in Series 1




**14.** Light-scattering coefficient of the PGW and CPGW pulps in Series 2




**15.** Wood handling of the raw material used in this study.




of PGW and CPGW pulps were both reduced to 1.5% after hot disintegration.

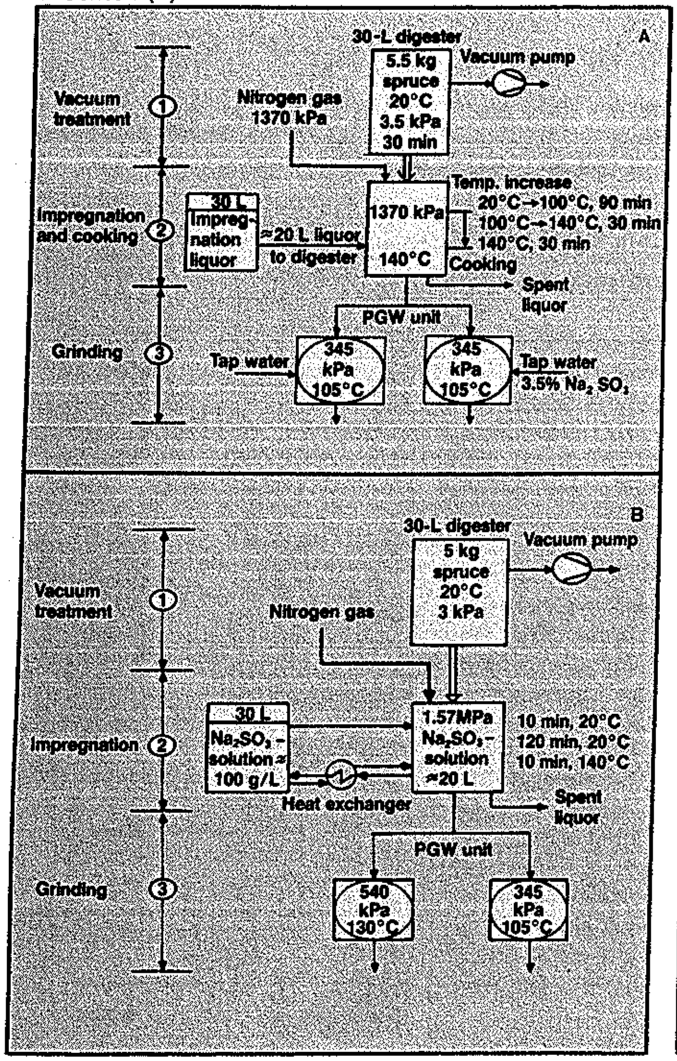
### Pulp strength properties

The effect of sulfite treatment of the wood raw material is reflected in the strength properties of the pulp. The tensile strength for the CPGW pulps in the first series is high (**Fig. 6**), especially considering the high freeness of the pulps (**Fig. 7**). Hot water impregnation and impregnation with cold sulfite liquor improved the tensile strength of the pulps equally by about 15%, whereas hot impregnation, especially with strong sodium sulfite liquor concentration, doubled the tensile strength figures for the pulp. In both series, impregnation with hot sulfite liquor enhanced the tensile strength of the pulps (**Fig. 8**).

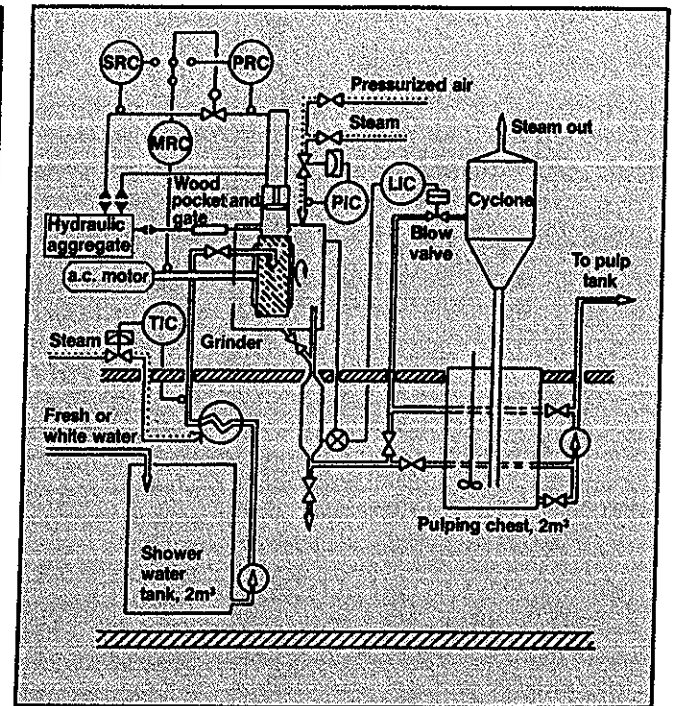
In the first series, tear strength for the CPGW pulps in some cases reached 8 mN·m²/g, compared with 5 mN·m²/g for the reference PGW pulps (**Fig. 9**). Using sulfite in the shower water seems to slightly reduce the tear strength for CPGW pulps. Results from the second series showed that cold impregnation with sulfite did not affect the tear strength (**Fig. 10**).

Hot impregnation did, however, in-

**16.** The impregnation and grinding scheme in Series 1 (A) and in Series 2 (B)




**17.** The pressurized grinder equipment used in the study




| | |
|---|---|
| Grinding stone, mm | |
| width | 120 |
| diameter | 500 |
| Grinding zone, mm | 100 x 150 |
| Peripheral speed, m/s | 5–50 |
| Power, kW | 110 |
| Grinder casing pressure, kPa | 0–500 |
| Production, kg/h | 5–50 |

Hydraulic feed of wood, alternatively at constant grinding pressure, constant grinding rate, and constant motor load.

crease tear strength from STa3-m²/g to 7 mN·m²/g. Vacuum treatment also reduced tear strength.

As expected, the use of hot shower water (130°C) in grinding increased the tear strength for the pulps produced.

### Optical properties

Data from Series 2 show that cold impregnation is favorable to pulp brightness. Brightness improved by 2 units (**Fig. 11**). The brightness of pulps produced using hot-impregnated wood was about the same as the brightness of the reference pulps. The combination of hot-impregnated wood and hot shower water yielded pulps with considerably lower brightnesses. The pulp with the highest brightness in Series 1 was produced with cold impregnation. In this series, the long impregnation time of 150 min had a negative effect on the brightness of the CPGW pulps (**Fig. 12**).

The negative effect of the chemical treatment on the light-scattering properties of the pulps was evident.

In Series 1, the light scattering coefficient declined from 70 to 45 and 37, respectively, for mild and strong impregnation liquor concentrations (**Fig. 13**). In Series 2, the light-scattering coefficient declined from 65 for the reference pulps to 50 for pulps produced with hot impregnation (**Fig. 14**).

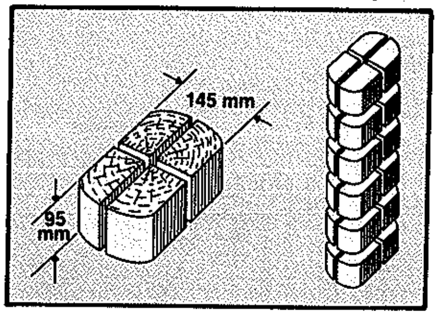
## Conclusions

Sodium sulfite impregnation of wood raw material for subsequent grinding is successful under relatively mild impregnation conditions. This is true even if the wood has rather large dimensions. Impregnation with 140°C liquor for 10 min resulted in fast penetration of the wood by the impregnation liquor. The resulting effect was considerable improvement in pulp properties.

Pressure grinding of chemically treated wood did produce CPGW pulps with properties that in many respects are comparable with CTMP pulps. The Bauer-McNett long fiber fraction and the tear strength for the CPGW pulps improved about 50%, and the tensile strength by 50–100% as compared with the reference pulps, the PGW pulps. As expected, sulfite treatment of the wood had an adverse effect on the light-scattering properties of the pulps. Scattering declined by some 25–50%.

Vacuum treatment of wood raw material in the impregnation vessel improved liquor penetration, but the actual effect on the strength properties of the pulp was negligible. Grinding with a high shower water temperature was especially effective in improving the strength properties of the CPGW pulps.

**18.** Wood blocks trimmed to fit the grinding zone and sliced for chemical analysis




## Experimental procedures

### Wood raw material

Local spruce (*Picea abies*) was used in the study. The impregnation vessel allowed experiments with log sizes up to a diameter of 180 mm and a length of 700 mm. The experiments aimed at studying the effectiveness of impregnation in logs of a rather large size. Both fresh and partly dried wood was used in the experiments.

The 3-m logs were barked and trimmed on two sides to produce 145-mm-wide beams. (The length of the grinding zone of the unit used is 150 mm.) The beams were cut either into 700-mm-long pieces for the impregnation vessel or into 95-mm-long pieces for grinding, as shown in **Fig. 15.** (The width of the grinding zone is 100 mm.)

### Impregnation and cooking

Impregnation and sulfonation of the wood was performed in a 30-L vessel equipped with forced liquor circulation. Phases involved in the chemical treatment were:

1. Filling the vessel with wood
2. Vacuum treatment (optional)
3. Addition of cold (20°C) or hot (140°C) impregnation liquor
4. Pressurizing the vessel using nitrogen gas
5. Cooking (sulfonation), starting either with a temperature raising period or directly from full temperature
6. Removal of the impregnation liquor after the completed cooking phase.

The liquor was circulated through the external heat exchanger in all experiments regardless of the temperature to be achieved. The sodium sulfite concentrations in the liquor were 35 g/L and 100 g/L. The impregnation phases are depicted in **Fig. 16.**

### Grinding

The impregnated logs were kept in sealed plastic bags until the actual grinding began. Grinding was performed with the pilot unit facilities available at the Finnish Pulp and Paper Research Institute. The grinder, which can be operated under normal pressure or in a pressurized state, was supplied by Oy Tampella Ab. **Figure 17** depicts the grinding unit. Grinding was performed under the conditions given in **Table V.**

Clean tap water was used to maintain the grinding consistency at 1.4%. A few experiments were done with a sulfite-containing shower at a sodium sulfite concentration of 0.5 g/L. All experiments were performed in the pitless run mode.

### Wood and pulp analyses

The impregnated wood species designated for sulfur analyses **(Fig. 18)** were stowed in sealed plastic bags immediately after the impregnation step and before cooling. The cooled wood samples were carefully dried for 24 h at 50°C in an oven equipped with efficient ventilation. The wood samples were cut into 3-cm-wide slices, chipped in a laboratory chipper, dried, and milled. The amount of sodium sulfite penetrating the wood during impregnation was calculated from the total sulfur amount measured in the wood meal samples.

It was possible to monitor the penetration of the wood by sulfite liquor with a relatively simple procedure. After impregnation, the wood beam was cut at different locations perpendicular to the length. The exposed surfaces were colored with suitable pH-sensitive indicator solutions, the best choice being an alizarin–methanol solution.

The pulps were screened using a vibrating laboratory screen with 4 mm holes. The properties of the screened pulps were measured. Strength and optical properties were measured using 52-g/m$^2$ sheets prepared with recirculated white water. Dichloromethane extractives was measured on 500-g/m$^2$ samples prepared in a Buchner funnel. These samples were not wet-pressed prior to drying.

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