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# MILL SCALE EXPERIENCES OF COMBINED SULPHITE PRE-TREATMENT AND HIGH INTENSITY REFINING OF SPRUCE

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## ABSTRACT

The effects of low dosage sulphite pre-treatment and high intensity refining were evaluated for production of thermomechanical pulp in two mill scale trials using Norway spruce (Picea abies) at the Braviken paper mill (Holmen Paper AB, Sweden).

Chips were mechanically pre-treated in a screw press (Impressafiner MSD 500) before impregnation with $Na_2SO_3$ solution at pH 9. Pulp samples for different dosages of $Na_2SO_3$ (0% ~1.21%) were taken from the blow line after the primary RGP68 double disc refiners. Approximately 50% of added sulphite was bound to the pulp after refining.

For pulps refined with equal specific energy consumption; density, tensile index, elongation and brightness were increased while light scattering and shive content were decreased by the sulphite pre-treatment. The effects of the sulphite pretreatment on tensile index were proportional to the dosage of sulphite. The specific energy consumption was reduced by 15% to a certain tensile index for chips pre-treated with 1.21% $Na_2SO_3$. Further analyses showed that the sulphite pre-treatment did not affect the distribution of the Bauer-McNett fractions for pulps refined with equal specific energy consumption but did increase fibre delamination/internal fibrillation as measured by Simons' staining.

The specific energy consumption for pulps produced with sulphite pre-treatment (1.24% $Na_2SO_3$) and double disc refining were 30% lower than for pulps produced in a two stage single disk refiner line (no pre-treatment), when compared to similar tensile index and light scattering coefficient.

**Keywords:** Thermomechanical pulp, Sulphite, Chip pre-treatment, High intensity refining, Energy reduction

## INTRODUCTION

In 2008 Holmen Paper AB started a new thermomechanical pulp (TMP) line in the Braviken paper mill with the ambition to increase pulp quality and decrease energy consumption. Included in this installation was the first mill scale Impressafiner operating on 100% spruce. The Impressafiner facilitates chemical pre-treatment via a high liquor uptake for chips after compression.

It is well established that sulphite pre-treatment of spruce chips prior to refining affects the fibre separation mechanisms and fibre properties of mechanical pulps. Sulphonation decreases the softening temperature of lignin which promotes fibre separation to occur closer to the middle lamella, rendering longer and more intact fibres.[1,2]

Increasing refining intensity seems to have the opposite effect to sulphonation on fibre separation. Fibres defibrated and refined at higher intensity tend to be more thin-walled and have large cracks in the fibre wall [3]. This suggests that fibre separation occurs deeper in the secondary fibre wall when the refining intensity is increased.

The combined effects of sulphite pre-treatment and high intensity refining have been shown to reduce energy consumption by 33% without any adverse effects on pulp properties [4]. Additional studies have shown that the individual gain in tensile index for both sulphonation and high intensity refining are additive (at certain specific refining energy) [5]. Both studies described were performed in pilot scale.

The aim of this study was to evaluate the combined effects of sulphite pre-treatment and high intensity refining at mill scale. Our ambition was to reduce the energy consumption during the production of mechanical pulps suitable for printing grade papers.

## EXPERIMENTAL

### Mill trials, processing and raw material

Full scale trials were conducted at the new TMP line (production rate 720 air dry ton (adt)/day) in the Braviken mill (Holmen Paper AB, Norrköping, Sweden). The raw material used was a mixture of round wood and saw mill chips from Norway spruce (Picea abies). Chips were fed from a conventional chip washer to a steaming bin (90 °C, ~15 min) and then through a rotary valve to a pressurised RT-conveyor (1.8 bar(g), 3~10 seconds). Thereafter chips were compressed in an Impressafiner MSD 500 (Andritz). Directly after compression, chips were submerged in the impregnation liquid where different dosages of $Na_2SO_3$ were added. The chips were kept in an atmospheric pre-heater (no steam added) for 10~15 minutes and thereafter fed through a plug screw to the pressurised refiner (4.5 bar(g)). Pulps were produced in three parallel double disc refiners (RGP68DD, Metso) at 1500 r/min. The refiner plates were 72″ in diameter using p-segments (DN72N816-817) and c-segments (DO52B036-037). The refiner production rate was measured by flow and consistency after the stand pipe succeeding each refiner.

Pulp samples were taken from the blow line directly after the refiner. Five pulp samples were collected during a period of 15 minutes for each chemical dose and refining energy. The samples were mixed and analysed three times with methods shown in Table 1.

**Table 1 Methods used for pulp analyses**

| Measurement | Method |
|---|---|
| Hot disintegration | SS-EN 2471 |
| Fibre length, width and shives | Eurocon PulpEye |
| Canadian standard freeness | ISO 5267-2 |
| Rapid Köthen (handsheet making method) | ISO 5269-2 |
| Sheet basis weight | SS-EN ISO 536 |
| Paper conditioning ratio | SS-EN 20187 |
| Optical properties | SS-ISO 9416 |
| Strength properties | SS-EN ISO 5270 |

### Addition of sulphite

Sulphite was added to chips by mixing concentrated solutions of $NaHSO_3$ and NaOH into the continuous flow of impregnation liquid. The solution was passed through a static mixer after which the pH was measured. The solution was then absorbed by the chips in the impregnator. The addition of $NaHSO_3$ was controlled by the production measurement in the Impressafiner. The addition of NaOH was adjusted to render an impregnation solution with pH 9. Sulphite doses are given as per cent $Na_2SO_3$ on oven dried basis.

### Study 1. Low dosage sulphite pre-treatment

Pulp samples were collected after the double disc refiner for different refining energy consumptions (1600~1900 kWh/adt) and dosage of $Na_2SO_3$ (0%, 0.12%, 0.24%, 0.61% and 1.21%).

During the trial, the raw material composition was changed from the normal 50/50 blend to 30% saw mill chips and 70% round wood chips. Chips were compressed in the Impressafiner with geometrical compression ratio 2.7∶1 and specific energy consumption 16 kWh/adt. Fresh water (1.2 $m^3$/adt) was used for impregnation. After impregnation, the temperature of chips was 62 °C. The production rate over the refiner was 9.5 adt/h and the consistency after refining was 30%~32%. The refining energy was changed by adjusting the plate gap.

Six pulps were chosen for additional analyses including sulphur content (SCAN-CM 57), Bauer-McNett fractionation (SCAN-CM 6∶05) and Simons staining according to Fernando and Daniel[6].

### Study 2. Sulphonation and refining intensity

The raw material composition was 50% saw mill chips and 50% round wood chips. The compression screw in the Impressafiner was changed prior to study 2. The new screw had a geometrical compression ratio of 3.6∶1. Specific energy consumption for compression of chips was 22 kWh/adt. Chips were pre-treated with 0% and 1.24% $Na_2SO_3$. The water used for impregnation was white water (1.2 $m^3$/adt). After impregnation chips had a temperature of 80 °C. The production rate over the refiner was 11.3 adt/h and the consistency after refining was 30%~38%. Pulp samples were collected after the double disc refiner for different refining energy consumptions (1300~1700kWh/adt). The refining energy was altered by adjusting the plate gap.

Reference pulps produced in single disc refiners were collected for different refining energy consumptions (1200~2000 kWh/adt) at the Braviken mill. The single disc line has two Twin 60 refiners (Sprout-Waldron) in series. Pulps were collected from blow lines after primary and secondary refiners following the same procedure as described above. The production rate was 13.1 adt/h. The consistency after the first stage refiner was 43% and after the second stage refiner 38%~43%. The refining energy was kept constant in the first stage refiner and was altered by adjusting the hydraulic pressure in the secondary refiner. No chip pre-treatment was used to produce single disc pulps.

## RESULTS AND DISCUSSION

### Study 1. Low dosage sulphite pre-treatment

For constant refining energy and increasing dose of sulphite, the plate gap of the refiner was decreased. The hydraulic pressure was only slightly reduced for the highest dose of sulphite (Table 2). The decrease in plate gap may influence fibre development during refining.

Freeness and shive content were decreased for increasing dose of sulphite, while tensile index, elongation and brightness were increased when refined with equal specific energy. Light scattering was slightly reduced (-1.6 $m^2/kg$) for the highest dose of sulphite. The other pulp properties presented in Table 2 were not affected by sulphite addition when the refining energy was kept constant.

The specific energy consumption needed to produce pulp with tensile index 47 Nm/g was reduced by 290 kWh/adt (15%) for chips pre-treated with 1.21% $Na_2SO_3$ compared to chips pre-treated without chemicals (Table 2). The lowest dose of $Na_2SO_3$ (0.1%) only had a small effect on pulp properties and refining conditions. These effects were regarded as scattering in measurements when comparing the chemical doses to similar tensile index. The refiner plate gap was increased and the hydraulic pressure was decreased for increasing dose of sulphite when pulps were refined to similar tensile index. Fibre length, fibre width, freeness and tear index were all increased while elongation and light scattering were reduced by increasing dose of sulphite, compared at similar tensile index. Shive levels were reduced for the two highest doses of sulphite. Brightness was slightly increased due to reduced light absorption with increasing sulphite dose.

The energy consumption needed to reach a certain tensile index for the reference (0% $Na_2SO_3$) was approximately 100 kWh/adt higher compared to data from other trails with double discs at Braviken [7]. The change in raw material composition for study 1 is probably one reason for the increased energy consumption.

The temperature of the chips after impregnation was 62 °C. Sulphonation reactions are rather slow at this temperature. Approximately 50% of the added sulphite was bound to pulps after refining.

The temperature of the chips after impregnation was 62 °C. Sulphonation reactions are rather slow at this temperature. Some of the added sulphite was removed from the chips in the plug screw prior to the refiner. Sulphonation reactions occur more rapidly in the pressurised environment (~155 °C) after the plug screw. Approximately 50% of added sulphite was bound to the pulp after refining (Table 3).

The amount of sulphur found in pulps when no chemicals were added was 210~240 mg/kg, which originates from natural sulphur content in wood and also sulphonic groups in the refiner dilution water.

Distribution of the Bauer-McNett fractions was not affected by addition of sulphite (Table 3). The fine fraction (<200) did not change in a distinct way in response to either increased refining energy or degree of sulphonation. The percentage of fibres with high degree of delamination/inernal fibrillation was significantly increased by both increased refining energy and addition of sulphite (highest dose).

**Table 2 Pulp properties and refining conditions for different dosages of $Na_2SO_3$**

| | Interpolated to SEC 1750 kWh/adt | | | | | Interpolated to tensile index 47 Nm/g | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| $Na_2SO_3$ added /% | 0.00 | 0.12 | 0.24 | 0.61 | 1.21 | 0.00 | 0.12 | 0.24 | 0.61 | 1.21 |
| Fibre length /mm | 0.94 | 0.94 | 0.92 | 0.92 | 0.92 | 0.88 | 0.87 | 0.89 | 0.92 | 0.99 |
| Fibre width /μm | 27.2 | 27.2 | 27.0 | 27.0 | 27.0 | 26.3 | 26.3 | 26.6 | 27.0 | 27.9 |
| CSF /mL | 137 | 136 | 124 | 129 | 117 | 91 | 87 | 105 | 129 | 161 |
| Shives/(number/g) | 398 | 398 | 372 | 330 | 271 | 344 | 367 | 366 | 330 | 299 |
| Density/ ($kg/m^3$) | 461 | 464 | 469 | 477 | 490 | 476 | 482 | 474 | 477 | 474 |
| Tensile index /(Nm/g) | 42.8 | 43.1 | 45.2 | 47.0 | 50.4 | 47.0 | 47.0 | 47.0 | 47.0 | 47.0 |
| Tear index/ ($Nm^2/kg$) | 7.00 | 6.99 | 7.04 | 7.05 | 7.01 | 6.78 | 6.61 | 6.94 | 7.05 | 7.39 |
| Elongation/% | 1.94 | 1.92 | 2.00 | 2.08 | 2.12 | 2.11 | 2.10 | 2.06 | 2.08 | 2.03 |
| Light scattering /($m^2/kg$) | 60.0 | 59.0 | 60.4 | 59.0 | 58.4 | 64.3 | 63.2 | 62.4 | 59.0 | 55.0 |
| Light absorption /($m^2/kg$) | 1.72 | 1.59 | 1.53 | 1.45 | 1.36 | 1.75 | 1.62 | 1.55 | 1.45 | 1.34 |
| Brightness (ISO 457) /% | 64.3 | 64.7 | 65.4 | 66.0 | 66.7 | 65.1 | 65.6 | 65.7 | 66.0 | 66.0 |
| SEC Impressafiner + refiner/(kWh/adt) | 1750 | 1750 | 1750 | 1750 | 1750 | 1897 | 1881 | 1803 | 1750 | 1608 |
| Hydraulic force /t | 58.3 | 57.1 | 58.8 | 57.2 | 56.4 | 62.6 | 63.1 | 60.2 | 57.1 | 53.7 |
| Plate gap/mm | 0.78 | 0.77 | 0.76 | 0.75 | 0.71 | 0.69 | 0.69 | 0.72 | 0.75 | 0.79 |

**Table 3 Pulp properties for different refining energies and dosage of $Na_2SO_3$**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| SEC Impressafiner + refiner /(kWh/adt) | | 1600 | 1600 | 1870 | 1870 | 1840 | 1840 |
| $Na_2SO_3$ added/% | | 0.00 | 1.21 | 0.00 | 0.24 | 0.61 | 1.21 |
| Sulphur in pulp after refining as $Na_2SO_3$ /% | | 0.08 | 0.67 | 0.09 | 0.17 | 0.33 | 0.68 |
| Density /($kg/m^3$) | | 446 | 473 | 472 | 476 | 490 | 501 |
| Tensile index /(Nm/g) | | 38.9 | 46.8 | 46.1 | 49.0 | 50.2 | 52.5 |
| Light scattering/ ($m^2/kg$) | | 56.7 | 54.8 | 64.1 | 64.6 | 59.7 | 60.5 |
| Bauer-McNett fractions/% | >16 | 23.7 | 24.6 | 18.7 | 17.7 | 19.4 | 18.1 |
| | 16~30 | 23.5 | 24.1 | 24.2 | 23.0 | 23.3 | 24.3 |
| | <200 | 24.9 | 22.7 | 25.1 | 27.4 | 26.6 | 26.3 |
| Degree of D/IF of Fibre Cell Walls (Simons Staining of Bauer-McNett 16-30 fraction)/% | Non | 57.0 | 44.5 | 48.0 | 50.5 | 43.0 | 34.8 |
| | Low | 26.3 | 26.3 | 27.5 | 26.8 | 28.0 | 26.3 |
| | High | 16.8 | 29.3 | 24.5 | 22.8 | 29.0 | 39.0 |

We suggest the following explanation for results in study 1. The addition of sulphite in the range applied does not affect the amount of fine material created during refining. However, the sulphite addition renders fibres with higher delamination/internal fibrillation which are more flexible [6]. These fibres produce a denser sheet with higher tensile index and slightly reduced light scattering. Untreated pulps demand higher refining energy consumption to reach a certain tensile index compared to sulphite pre-treated pulps. Therefore, sulphite pre-treated pulps have lower light scattering compared to untreated pulps refined to similar tensile index.

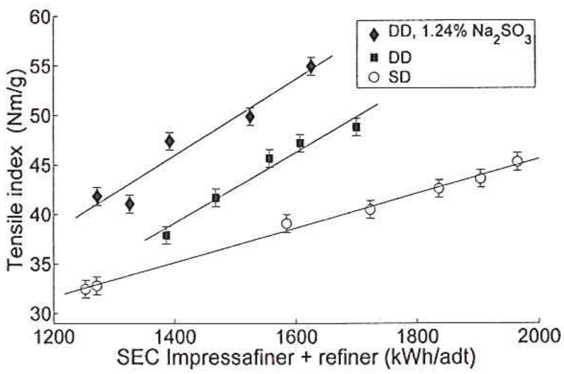


**Figure 1 Tensile index development for pulps produced by: sulphite pre-treatment and double disc refining (DD, 1.24 % $Na_2SO_3$), double disc refining (DD) and single disc refining (SD)**

**Study 2. Sulphonation and refining intensity**

Pulps produced in the double disc refiner had higher tensile index than pulps produced in single disc refiners, when compared at equal specific energy consumption (Figure 1). One reason may be a higher refining intensity in double disc refining. Also, the compressive pre-treatment of chips in the Impressafiner have been shown to reduce the energy consumption in the order 6%~8% [8]. Compared at similar tensile index, double disc pulps had shorter fibres, less shives, higher elongation and higher light scattering than single disc pulps (Table 4). Similar changes in pulp properties have been presented earlier for increased refining intensity[3].

Pulps produced with sulphite pre-treatment (1.24% $Na_2SO_3$) and double disc refining reached a tensile index of 45 Nm/g with 580 kWh/adt (30%) lower refining energy compared to single disc pulps (Figure 1). The sulphite pre-treatment reduced the light scattering for double disc pulps. However, the light scattering was slightly higher (2.3 $m^2/kg$) than for single disc pulp at similar tensile index (Table 4). Furthermore, the sulphite pre-treated double disc pulps had lower shive content and higher brightness than single disc pulps.

**Table 4 Pulp properties (tensile index 45 Nm/g)**

| Refiner type | DD | DD | SD |
|---|---|---|---|
| $Na_2SO_3$ added /% | 0.00 | 1.24 | 0.00 |
| Fibre length/mm | 1.03 | 1.13 | 1.33 |
| Fibre width/μm | 27.7 | 28.7 | 27.6 |
| CSF/mL | 147 | 221 | 164 |
| Shives/(number/g) | 330 | 196 | 696 |
| Density/($kg/m^3$) | 480 | 481 | 462 |
| Tear index/($Nm^2/kg$) | 7.61 | 7.92 | 8.57 |
| Elongation /% | 1.98 | 1.87 | 1.80 |
| Light scattering/($m^2/kg$) | 56.9 | 50.7 | 48.4 |
| Brightness (ISO 457)/% | 62.9 | 64.7 | 59.8 |
| SEC Impressafiner + refiner/ (kWh/adt) | 1563 | 1379 | 1962 |

## CONCLUSIONS

Sulphite pre-treatment increased delamination/internal fibrillation making fibres more flexible. These fibres produced denser sheets with higher tensile index and slightly reduced light scattering at a certain specific energy consumption. The specific energy consumption needed to produce pulp with tensile index

47 Nm/g was reduced by 290 kWh/adt (15%) for chips pre-treated with 1.21% $Na_2SO_3$ compared to chips pre-treated without chemicals. Fibre length, fibre width, freeness and tear index were all increased while elongation and light scattering were reduced by increasing dose of sulphite, compared at similar tensile index.

The double disc refiner produced pulp with higher tensile index compared to single disc pulp at similar specific energy consumption. Double disc pulps also had a higher light scattering compared to the single disc pulp at equal tensile index.

Pulps produced with sulphite pre-treatment (1.24% $Na_2SO_3$) and double disc refining reached a tensile index of 45 Nm/g with 580 kWh/adt (30%) lower refining energy compared to single disc pulps. Furthermore, light scattering and brightness was increased while shive content was reduced for sulphite pre-treated double disc pulp compared with single disc pulp at similar tensile index.

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