Reduction of Refining Energy during Mechanical Pulping

Using Pressurised Chip Compression and Sulphite Pre-treatment

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Reduction of Refining Energy during Mechanical Pulping - Using Pressurised Chip Compression and Sulphite Pre-treatment

Abstract

The effects of pressurised compressive chip pre-treatment and low dosage sulphite pre-treatment were evaluated for production of thermomechanical pulp in mill scale trials using Norway spruce (*Picea abies*) at the Braviken paper mill (Holmen Paper AB, Sweden). The general aim of the study was to improve the energy efficiency during the production of mechanical pulps suitable for news and improved news grade papers.

The pressurised compressive chip pre-treatment performed in an Impressafiner, resulted in a reduced acetone extractive content for first stage blow line pulp by up to 24%. Furthermore, pulp produced from mechanically pre-treated chips had higher tensile- and tear indices, elongation and light scattering and lower freeness compared to pulps from untreated chips produced with equal total specific energy consumption. The total specific energy consumption was reduced by 120 kWh/bone dry ton (6%) at equal tensile index, when pulps were produced together with the Impressafiner pre-treatment.

Sulphite pre-treatment increased tensile index, elongation, density and brightness and reduced light scattering and shive content compared to pulps produced with only mechanical pre-treatment at equal specific energy consumption. The increase in tensile index and reduction in light scattering followed linear relations to the dosage of sodium sulphite in the measured dosage range (0-1.2% Na_2SO_3). The addition of ~1.2% sodium sulphite gave a sulphur content in pulp of ~0.67% (as Na_2SO_3) and reduced the specific energy consumption by 210-320 kWh/bdt (12-15%) when compared at equal tensile index. However, light scattering was not retained for this energy reduction.

Further analyses showed that sulphite pre-treatment did not significantly affect the distribution of the Bauer-McNett fractions or the fibre length for pulps refined with equal specific energy consumption but did increase fibre delamination/internal fibrillation as measured by Fernando and Daniel's (2010) version of Simons' staining.

The specific energy consumption for pulps produced with sulphite pre-treatment (1.2% Na_2SO_3) and double disc refining were 650 kWh/bdt (30%) lower than for pulps produced in a two stage single disc refiner line (no pre-treatment), when compared at a similar tensile index and light scattering coefficient.

Keywords: Chip pre-treatment, double disc refiner, energy efficiency, Impressafiner, low dosage, Norway spruce, refining intensity, single disc refiner, sulphite pre-treatment, thermomechanical pulp.

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Contents

List of Publications

This thesis is based on the work contained in the following papers, referred to by Roman numerals in the text:

I Erik Nelsson, Christer Sandberg, Lars Hildén and Geoffrey Daniel (2011). Pressurised compressive chip pre-treatment of Norway spruce with a mill scale Impressafiner. *Accepted for publication in Nordic Pulp and Paper Research Journal*.

II Erik Nelsson, Lars Hildén, Christer Sandberg, Dinesh Fernando and Geoffrey Daniel (2011). Mill scale experiences of combined low dosage sulphite pre-treatment and high intensity refining of spruce (manuscript). A shorter version of the paper is published in Proceedings of International Mechanical Pulping Conference 2011, Xi'an, China, June 26-29, 182-186.

III Jonas Hafrén, Erik Nelsson, Hans C. Gerritsen and Arjen N. Bader (2011). Optical properties of low sulfite pretreated thermomechanical pulp. *Submitted to Holzforschung*.

The contribution of Erik Nelsson to the papers included in this thesis was as follows:

I Planning and execution of performed mill scale trials. Presentation and interpretation of obtained results. Review of the literature and writing the manuscript.

II Planning and execution of performed mill scale trials. Presentation and interpretation of obtained results. Review of the literature and writing the manuscript.

III Planning and execution of performed mill scale trials. Provided material and process information for microscopy studies. Provided optical data for lab sheets and helped with the interpretation of these results.

Abbreviations

barg	Bar guage
bdt	Bone dry ton
COD	Chemical oxygen demand
CSF	Canadian standard freeness
CTMP	Chemi-thermomechanical pulp
DD	Double disc
ML	Middle lamella
MSD	Modular screw device
Na_2SO_3	Sodium sulphite
$NaHSO_3$	Sodium bisulphite
NaOH	Sodium hydroxide
P	Primary cell wall
PM	Paper machine
RMP	Refiner mechanical pulp
rpm	Revolution per minute
RT	Retention time
S1	Outer layer of the secondary cell wall
S2	Middle layer of the secondary cell wall
S3	Inner layer of the secondary cell wall
SD	Singe disc
SEC	Specific energy consumption
$-SO_3^-$	Sulphonate
TMP	Thermomechanical pulp

1 Introduction

1.1 Background

During the production of news and improved news grade papers it is desirable to reach high opacity at sufficient strength properties with a minimal use of electrical energy. For this purpose, the thermomechanical pulp (TMP) process provides pulp with a suitable combination of light scattering and strength properties (Höglund and Wilhelmsson 1993). However, the use of electrical energy in this process is high.

One approach for reducing the use of electrical energy in the TMP process is chip pre-treatment prior to refining. Various types of pre-treatment techniques have been evaluated throughout the history of TMP, including: mechanical pre-treatment (Frazier and Williams 1982), sulphite pre-treatment (Atack *et al.* 1978), alkaline hydrogen peroxide pre-treatment (Bohn and Sferrazza 1989), oxalic acid pre-treatment (Akhtar *et al.* 2002), enzymatic pre-treatment (Peng *et al.* 2005) and others. However, so far, no chip pre-treatment technique has been widely adapted in mill scale production of TMP from spruce.

In 2008 Holmen Paper AB started a new TMP line at the Braviken paper mill with an ambition to increase pulp quality and decrease energy consumption. Included in this installation was the first mill scale Impressafiner operating on 100% spruce chips. The Impressafiner is a pre-treatment screw-press where chips are compressed at high strain in a pressurised environment. During the compression, water and extractives are pressed out of the chips. An impregnation step is located directly after the compression zone. The liquor uptake for chips in this position is high, which makes it a suitable process step for addition of chemicals. The new TMP line in Braviken is therefore a

suitable installation for studies of both mechanical and chemical chip pre-treatments in mill scale.

The mechanical pre-treatment of the Impressafiner was evaluated during the first part of this thesis work. Later, the aspect of adding a chemical treatment to the impregnation step subsequent to the Impressafiner was evaluated. Low dosage sulphite pre-treatment was chosen as a first candidate for this evaluation. It has been observed that low dosage sulphite pre-treatment gives a maximum in tensile index and light scattering at a sulphur content of ~0.2% (as Na_2SO_3) compared to other sulphite dosages at equal specific energy consumption (Axelson and Simonson 1982; Westermark *et al.* 1987; Svensson *et al.* 1994). The low sulphur content needed to reach this effect is an attractive feature of this pre-treatment concept. Low sulphur content implies low sulphite dosage and therefore less yield loss and low chemical costs.

1.2 Primary objectives of the study

- Evaluation of mechanical- and low dosage sulphite pre-treatment of spruce chips with focus on reducing the electrical energy used during refining while retaining or improving pulp properties.

- Contribute to the understanding of the mechanisms behind the reported and observed effects for further development of chip pre-treatment and refining processes.

1.3 Hypotheses

The hypotheses evaluated in this thesis work have been studied earlier. However, evaluation of these hypotheses in mill scale are scarce and have never been done in mill scale combining Impressafiner pre-treatment and Norway spruce as raw material. The hypotheses included:

- By introducing a mechanical chip pre-treatment with relatively low frequency and high amplitude prior to refining, an enhanced fibre development, e.g. increased tensile index, can be achieved at similar specific energy consumption.

- There is a maximum for tensile index and light scattering when applying low dosage sulphite pre-treatment at a sulphur content of ~0.2% (as Na_2SO_3) compared to other sulphite dosages at equal specific energy consumption.

1.4 High consistency chip refining

1.4.1 Structure and ultrastructure of Norway spruce (*Picea abies*)

Wood is a highly hierarchical composite built from a number of characteristic cell types. One of these cell types is the longitudinal tracheid (normally referred to as "fibre") which constitutes 90-95% of the wood volume in Norway spruce. The fibres in Norway spruce are long and slender, often 100 times greater in length than in width. For Norway spruce, the average fibre length and width varies between 2-4 mm and 20-40 µm respectively (Sjöström 1993).

In wood, the fibres are joined together by a matrix called the middle lamella (ML). The very thin primary cell wall (P) is the outermost cell wall layer and separates the secondary cell wall from the ML. The secondary wall is divided into three layers known as S1, S2 and S3 secondary wall layers. The S2 layer is much thicker than the S1 and S3 layers (*Figure 1a*). Each of the secondary wall layers shows a characteristic microfibril angle (*i.e.* orientation of the cellulose microfibrils) (*Figure 1b*).

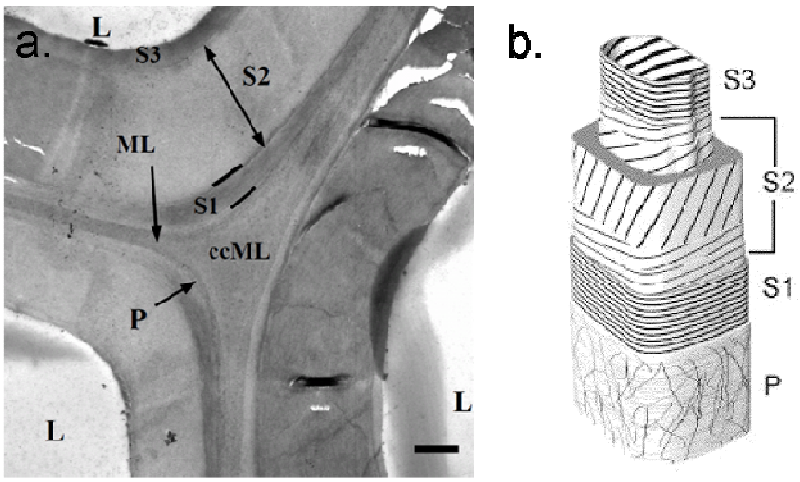

Figure 1. (a) Transmission electron microscopy image of a transverse section from Norway spruce showing the different cell wall layers (P, S1, S2, S3) and the ML. L denotes cell lumen and ccML denotes the middle lamella cell corner (Fernando 2007). Bar: 0.5 µm. (b) Proposed cell wall model of a Norway spruce fibre, showing microfibrilar orientation in different cell wall layers (Brändström 2002).

As well as the difference in ultrastructure of the cell wall layers, there is also a variation in chemical composition. *Figure 2* shows a very general overview of how the three major polymers in wood (cellulose, hemicelluloses and lignin) are distributed over a typical fibre cell wall. The difference in chemical composition and ultrastructure between the cell wall layers affects fibre

separation and development during mechanical pulping, this will be described below.

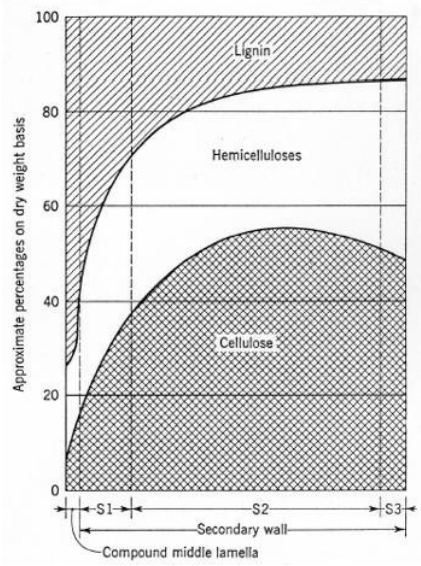

Figure 2. Distribution of cellulose, hemicelluloses and lignin across the fibre cell wall (Panshin and de Zeeuw 1980).

1.4.2 Wood softening

The softening of wood is related to the transition of lignin from a brittle glass-like form to an elastic rubber-like form. This transition occurs as the temperature is increased but is also dependent on loading frequency. The temperature interval for the glass transition has been calculated to be from 100°C to roughly 170°C at the frequency of a commercial refiner (10 kHz) (Irvine 1985).

When the temperature is increased within the glass transition range, the tensile modulus of lignin is reduced. Since the lignin concentration of the fibre cell wall is unevenly distributed with a higher concentration in the middle lamella, a change in the tensile modulus of lignin will affect the fracture mechanism during refining. Refining at temperatures below the glass transition range will lead to fractures both parallel and transverse to the fibre direction, resulting in low fibre length. As the temperature is increased, fractures parallel to the fibre direction will be favoured. A further increase in temperature towards the higher end of the temperature interval of the glass transition will promote fractures to occur predominantly in the middle lamella, resulting in lignin capsulated fibres (Irvine 1985).

Figure 3 demonstrates how the phenomena described above affects the fibre separation in different refining processes. In the refiner mechanical pulp (RMP) process, chips with a temperature below or around 100°C are fed into

the refiner and therefore, much of the fibre separations occur deep in the S2 layer, also leading to a large proportion of fractures perpendicular to the fibre axis. At the elevated temperatures in the TMP process, where chips are pre-heated to a temperature above 100°C, fibre separation occurs further out in the region of the S1 layer (Salmén *et al.* 1999) (*Figure 3*).

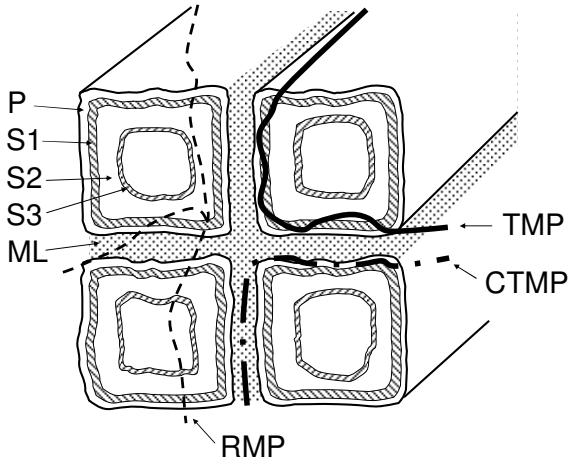

Figure 3. Schematic drawing over fibre separation in the three different processes, RMP, TMP and CTMP. The cell wall layers are denoted: Primary cell wall (P), Secondary cell wall layers (S1, S2 and S3) and middle lamella (ML). Redrawn from Salmén *et al.* (1999), who adapted the drawing after Franzén (1986) and Htun and Salmén (1996).

1.4.3 Refining intensity

Refining intensity can be defined as the specific energy per bar impact (Miles and May 1990, Miles 1991). The refining intensity is increased as the specific energy consumption increases but is also affected by the pulp residence time and segment design. Changing the rotational speed of the refiner or type of refiner (*i.e.* single disc, double disc) changes both the residence time and number of impacts and thus affects refining intensity. Chips and pulps defibrated and refined at higher intensity tend to have an increased content of thin-walled fibres and fibres with longitudinal cracks in the fibre wall (Kure and Dahlqvist 1998, Fernando *et al.* 2011). Other attributes of pulps refined at higher intensity is an increased proportion of fine material, increased light scattering and reduced fibre length when compared to pulps refined at lower intensities at equal specific energy consumption (Kure *et al.* 1999, Muhić 2010).

1.4.4 Criteria used for reduced energy consumption in this thesis

In this thesis special attention is paid to the properties tensile index and specific light scattering coefficient. The reason for this is that these two properties are

strongly correlated with the refining energy in the primary refiner of the new TMP-line in Braviken. Also, these properties are essential for low grammage printing grade papers (Höglund and Wilhelmsson 1993). A sufficiently high tensile index is needed to avoid breaks during both paper production in the paper machine and during the printing process. A sufficiently high specific light scattering is needed to produce paper with desired opacity and brightness. It is however possible to adjust both the tensile index and light scattering of the paper by adding kraft pulp or fillers at the paper machine but these possibilities are not investigated in this thesis.

The specific energy consumption needed to produce pulp is therefore defined at a certain tensile index or light scattering in this thesis. In order to reduce the refining energy, tensile index and light scattering of the pulp must be maintained or improved.

1.5 Mechanical chip pre-treatment

1.5.1 Pressurised compressive chip pre-treatment

During the production of TMP, wood chips are separated into individual fibres (*i.e.* defibration) and further treated to produce suitable fibres for paper making (*i.e.* fibrillation). In a recent review article on mechanical pre-treatment, Gorski *et al.* (2010) concludes that defibration and fibrillation are two fundamentally different mechanisms and should preferably be conducted separately. This theory was first proposed by Salmén *et al.* (1985). Traditionally, defibration of chips is achieved by cyclic compression and shear in the breaker bar zone of the primary refiner, with a frequency in the kHz range (Becker *et al.* 1977). Salmén *et al.* (1985) showed that a reduction in the frequency of the compression cycles would increase the effectiveness of the structural breakdown of wood. Salmén *et al.* (1985) also concluded that, the effectiveness of structural breakdown is greatest in the initial compression cycles and increases with amplitude irrespective of temperature. These findings suggest there could be more energy effective conditions for defibration than those in the breaker bar zone.

Frazier and Williams (1982) reported that TMP and chemi-thermomechanical pulp (CTMP) produced from axially pre-compressed wood blocks had superior bonding potential and fibre length retention compared to TMP and CTMP produced from untreated wood chips. Commercial solutions for compressive pre-treatment of chips have also been developed, including treatments in BIVIS (Clextral) or PREX (Metso) screws.

In 1997, Andritz introduced the MSD RT-Pressafiner (MSD=modular screw device, R=retention, T=temperature) with a pressurised inlet (1.5 barg) and compression ratio of 5:1 (Sabourin 2000). The compression zone of the MSD RT-Pressafiner is a screw press with an increasing shaft diameter where the chips are compressed as they are fed forward through the narrowing gap between the shaft and the outer casing. During this process, water and extractives are pressed out of the chips through holes in the outer casing. The steam pressurised inlet is necessary at high-compression condition in order to achieve a high level of fibre separation without fibre breakage (Sabourin 2000). This corresponds with results from Koran (1981), who showed that fibre separation performed at increased temperature would decrease fibre breakage and increase the creation of new fibre surfaces resulting in reduced energy consumption per separated fibre. By adding an impregnation screw directly after the compression zone in the MSD RT-Pressafiner, the equipment was named MSD RT-Impressafiner (hereafter referred to as "Impressafiner").

During the Impressafiner pre-treatment, chips are subjected to one or a few compressions cycles during a minimum time frame of one second. This gives a maximum frequency for the pre-treatment of one or a few Hz. The frequency during refining is much higher, *i.e.* in the kHz range (Becker *et al.* 1977). The specific energy consumption during refining is in the order of 100 times greater than during the Impressafiner pre-treatment. The number of compression cycles during refining can be assumed to be in the order of 1000 times greater than during the Impressafiner pre-treatment. Therefore, the amplitude of the compression cycles is most likely higher during the Impressafiner pre-treatment compared to that during refining.

Results from an industrial installation of the Impressafiner operating on southern pine, predominantly loblolly pine (*Pinus taeda*), have shown its ability to reduce DCM extractives in pulp by ~25%, reduce chemical oxygen demand (COD), stabilise the motor load in refiners and reduce the total specific energy consumption by ~115 kWh/bdt (Sabourin *et al.* 2002). A pilot plant comparison between Norway spruce and Scots pine (*Pinus sylvestris*) showed that pressurised compressive chip pre-treatment led to an increase in tensile and tear indices for pine but not for spruce (Robertsen *et al.* 2001). Another pilot scale study performed with high intensity refining of Norway spruce indicated that pressurised compressive chip pre-treatment reduced the specific energy by about 7% when compared at certain Canadian standard freeness (CSF) (Kure *et al.* 1999).

1.5.2 Extractive removal and refining

As described above, extractives are pressed out from chips during the Impressafiner pre-treatment resulting in a lower extractive content for pulps produced from pre-treated chips. The impact of extractives on the energy efficiency of thermomechanical pulping has been debated for the last decade. Reme and Helle (2001) compared the difference in response to refining between Norway spruce and Scots pine. They concluded that the initial defibration requires almost the same energy for both Norway spruce and Scots pine but when refined further, Scots pine demands much more energy to a given freeness or strength. They attributed the difference to the much higher level of extractives in Scots pine. They suggested that extractives may reduce friction in the refiner and thus increase the need for energy to reach a given freeness. A relation between extractive content and friction between wood and steel has also been suggested by Svensson *et al.* (2006). Persson *et al.* (2005) also studied the difference in response to refining between Norway spruce and Scots pine using chips that were to a large extent depleted of extractives during storage and transport. All levels of extractives were reduced, especially the large fraction of triglycerides. The difference in refining energy between Scots pine and Norway spruce was much lower than in the study performed by Reme and Helle (2001) where fresh samples were used. Persson *et al.* (2005) concluded that extractives are probably the main obstacle for using pine in mechanical pulping but that differences in response to refining due to different cell wall properties probably also play a role. This structural difference between Norway spruce and Scots pine was later confirmed by Daniel *et al.* (2009).

Heum *et al.* (2005) added different extractives to Norway spruce primary pulp in further refining. The specific energy demand to reach a given freeness level was unchanged upon the addition of extractives but the specific energy demand to reach a given strength increased. The authors indicated that further studies are needed to conclude whether this is due to reduced friction in the refiner or not. Illikainen *et al.* (2007) performed shear stress analyses on pulp and found no difference when extractives were removed. Hildén and Persson (2007) found no correlation between seasonal maxima of acetone soluble extractives and specific energy needed to reach a given pulp strength. However it is well known that extractives, especially extractives with long carbon chains have a negative impact on strength of paper from a TMP process (Sundberg *et al.* 2000, Kokkonen *et al.* 2002).

The impact of extractives on paper properties makes it difficult to isolate the effect of removing extractives prior to refining. It is not possible to rule out a negative impact of extractives on specific refining energy to a given quality.

Also, beneficial effects such as improved paper machine runnability and decreased pitch deposition may be expected through a reduction of extractives.

1.6 Sulphite pre-treatment

1.6.1 Chemi-thermomechanical pulping

During the production of CTMP from softwood, wood chips are usually impregnated with 2-4% Na_2SO_3 at pH 9-10 and pre-heated at 120-140°C with a retention time of 2-15 min. This usually yields sulphur contents in the range 0.4-1.2% (as Na_2SO_3) prior to refining (Lindholm and Kurdin 1999). The major chemical mechanism for sulphonation of wood under these conditions is the introduction of sulphonate groups into benzylic (α-) carbon of phenyl propane units in lignin (Lindholm and Kurdin 1999). The introduction of sulphonate groups in the lignin polymer affects the dynamic mechanical properties of wood. In the sulphur content range 0.3 to 2.8% (as Na_2SO_3), the softening temperature of black eastern spruce (*Picea mariana*) was decreased by about 2°C for every 0.1% increase in sulphur content (as Na_2SO_3) (Atack *et al.* 1978).

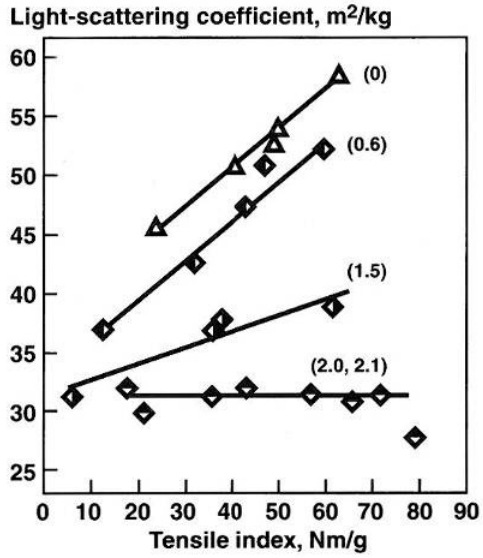

Figure 4. Light scattering vs. tensile index for different sulphonate content (% $-SO_3^-$) denoted in brackets (1% $-SO_3^-$ = 1.57% Na_2SO_3) (Atack *et al.* 1980).

Sulphonation under CTMP conditions shifts fibre separation towards the middle lamella so that separation occurs predominantly in the area of the primary cell wall and middle lamella (Lai and Iwamida 1993) (*Figure 3*). Fibre separation in the area of the middle lamella is favourable for products such as paper board and tissue where the objective is to produce pulp with high bulk

and low amount of shives at a certain Scott bond (paper board) or tensile strength (tissue) (Höglund and Wilhelmsson 1993). Such pulps are however not suitable for printing grade papers due to the inferior light scattering in relation to tensile index (Höglund and Wilhelmsson 1993). Atack *et al.* (1980) measured the relationship between light scattering and tensile index for different degrees of sulphonation (*Figure 4*) and attributed the lower light scattering for sulphonated pulps to an extensive collapse of long fibres and lower amounts of fines.

1.6.2 Acid sulphite pre-treatment

The degree and rate of sulphonation reactions are affected by the pH of the impregnation liquor. Under neutral conditions, sulphonation occurs only in the phenolic units in lignin. Under acid conditions, both phenolic and etherified sites are sulphonated (Gellerstedt 1976).

The kinetics of sulphonation reactions under both acid and neutral conditions have been investigated by Heitner *et al.* (1982) and Beatson *et al.* (1984). The rate determining step for sulphonation at pH 7 was found to be the nucleophilic addition of the sulphite anion to quinone-methide. The formation of quinone methide from a phenolic hydroxyl group in lignin is fast. The rate for this mechanism is therefore dependent on the sulphite concentration of the impregnation liquid (Heitner *et al.* 1982).

At pH 4, sulphonation proceeds partly through the same mechanism as for pH 7 and partly through an additional mechanism only possible under acid conditions. The latter proceeds though a rate determining cleavage of a benzyl-O-ether or benzyl-OH bonds which may then form a benzyl carbonium ion. Since the nucleophilic addition of the sulphite anion to the carbonium ion occur much faster than the formation of the carbonium ion, the rate of this reaction is not dependent on the sulphite concentration of the impregnation liquid (Beatson *et al.* 1984).

Sulphite pre-treatment under acid conditions has been shown to improve both strength and optical properties of CTMP from black spruce (*Picea mariana*) (Argyropoulos and Heitner 1991; Stationwala 1992). Decreasing the pH of the impregnation liquid to pH 4.2 increased both tensile index and light scattering at certain specific energy consumption compared to TMP and more alkaline CTMP (*Figure 5a, b*). The increase in tensile index was attributed to an increased interbonding of the long fibre fraction and to an increase in fines content (Argyropoulos and Heitner 1991). The latter may also explain the increase in light scattering.

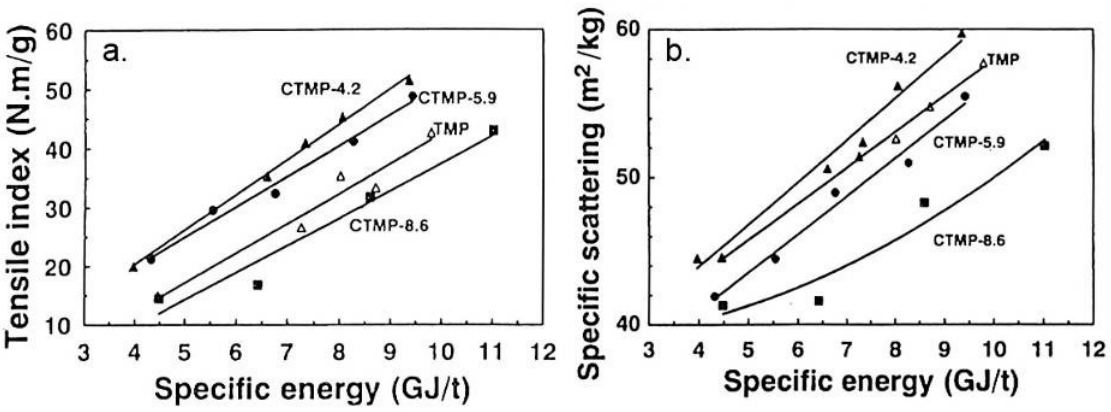

Figure 5. Tensile index (a) light scattering (b) vs. specific energy consumption for black spruce TMP and CTMP impregnated at different pH as denoted by the figures after "CTMP-". Sulphur content as Na_2SO_3 for: CTMP-4.2 = 0.76%; CTMP-5.9 = 0.79%; CTMP-8.6 = 0.63% (Argyropoulos and Heitner 1991).

1.6.3 Low dosage sulphite pre-treatment

Low dosage sulphonation (sulphur content < 0.4% as Na_2SO_3) under slightly alkaline conditions prior to refining has been shown to affect pulp properties differently compared to the effects seen in the dosage range of the CTMP process (Axelson and Simonson 1982). A maximum was observed for both tensile index and light scattering at 0.2% sulphur content (as Na_2SO_3) when compared at certain specific energy consumption (*Figure 6*). Similar results were later obtained by Westermark *et al.* (1987) and Svensson *et al.* (1994).

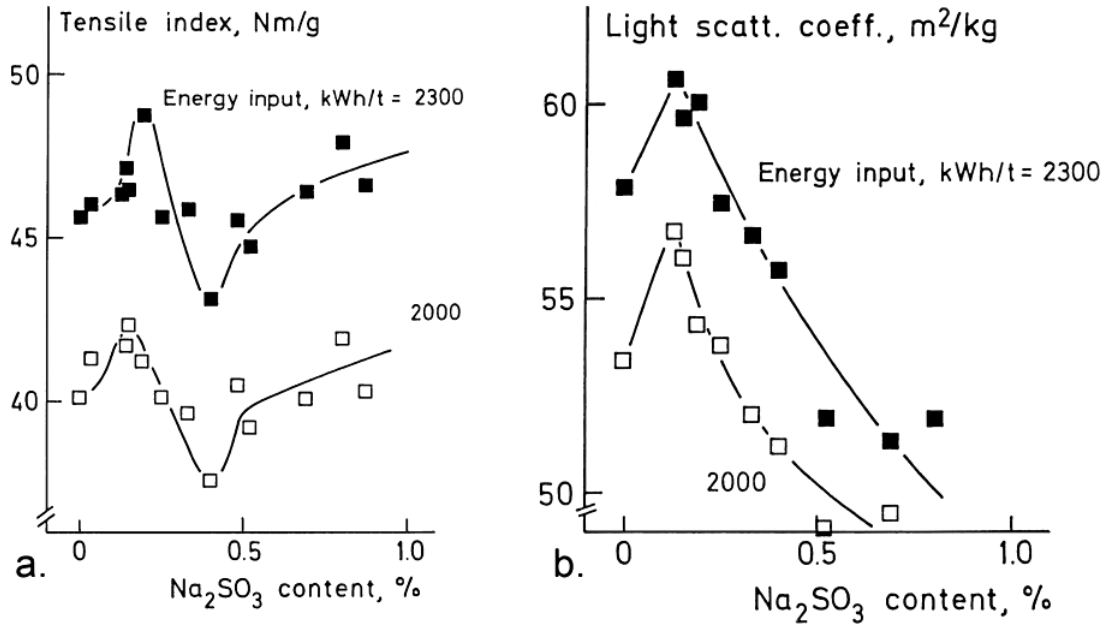

Figure 6. Tensile index (a) and light scattering (b) vs. sulphur content (as Na_2SO_3) at certain specific energy consumption. A maximum in tensile index and light scattering was observed at a sulphur content of 0.2% (as Na_2SO_3) (Axelson and Simonson 1982).

The explanation offered by Axelson and Simonson (1982) to the maximum in tensile index and light scattering was that a sulphur content of 0.2% (as Na_2SO_3) gave a suitable decrease in the softening temperature in relation to the temperature and frequency of the breaker bar section and the refining zone of the refiner used in the study.

It was later found that there is a selective sulphonation of the primary cell wall layer for low sulphite dosages (Westermark *et al.* 1987). It was also found that sulphonation affects the softening temperature of the middle lamella and the primary cell wall layer differently. As the softening temperature of middle lamella is decreased by sulphonation, the softening temperature of the primary cell wall layer is increased from a value below that of the middle lamella. The high protein content of the primary cell wall is suggested as the explanation to the differences between the primary cell wall layer and middle lamella (Östberg and Salmén 1988). However, relating this effect to the maximum in tensile index observed by Axelson and Simonson (1982) is rather speculative.

By comparing the microscopic appearance of fibre-fibre fractures for wood with different sulphur contents, it was possible to relate the tensile index maximum at a sulphur content of 0.2% (as Na_2SO_3) with fibre surfaces where the middle lamella was almost completely removed. These surfaces also had very thin, thread-like fragments that were not present at higher sulphonation levels (Westermark *et al.* 1987, Johansson *et al.* 1997).

2 Materials and methods

Full scale trials were conducted at the Braviken paper mill, Holmen Paper AB, Norrköping, Sweden. The raw material used in all trials was a mixture of round wood and sawmill chips from 100% Norway spruce (*Picea abies*). The majority of the trials presented in this thesis were conducted in the TMP-line installed in 2008, here referred to as the "double disc line". A reference study was also performed in a TMP-line installed during the mid-eighties, here referred to as the "single disc line".

All results presented in this thesis are based on measurements either before or directly after the first or second stage of refining. Therefore, detailed descriptions of the processes will only be given up until these stages.

2.1 Description of the processes

2.1.1 Raw material

The raw material was supplied to the Braviken mill both as round wood and as sawmill chips. The majority of the round wood was first thinnings and top logs collected in the region of Braviken. The round wood was debarked and chipped in the mill using standard process equipment. Sawmill chips were delivered by various sawmills located in the region of Braviken. The double disc line is normally supplied with 50% round wood, and 50% sawmill chips. The single disc line is normally supplied with 100% round wood chips. However, some variations occur in the chip ratios, depending on the supply of sawmill chips and on the operational status of the debarking and chipping process.

2.1.2 The double disc line

An outline of the double disc line is shown in *Figure 7*. The double disc line was installed to replace an older line from the seventies.

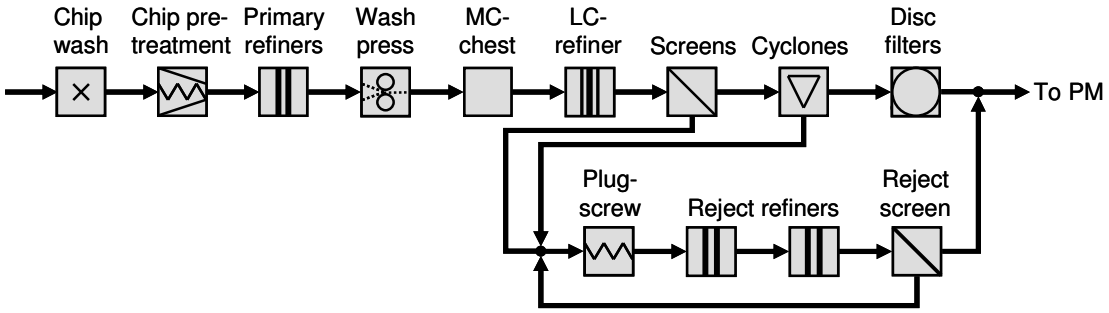

Figure 7. Overview of the double disc line.

Chip pre-treatment

Figure 8 outlines details of the chip pre-treatment equipment included in the double disc line. For production of chips and pulps without pre-treatment (later referred to as "reference chips" and "reference pulps"), chips were fed directly from the chip washer to the refiner pre-heater (*Figure 8*). For production of chips and pulps with chip pre-treatment, chips were fed from the chip washer to a steaming bin (90°C, ~15 min.) through a rotary valve to the pressurised RT-conveyor (0.3-1.8 barg, 3-10 sec). Thereafter chips were compressed in the Impressafiner with geometrical compression ratio 2.7-3.6:1 and specific energy consumption 20-28 kWh/bdt and directly submerged in the impregnator (liquor uptake: 0.9-1.8 m^3/bdt) and transferred to the refiner pre-heater (10-15 min, no steam added). Before refining, both reference and pre-treated chips were fed through a plug screw into the steam pressurised environment where chips are pre-heated prior to refining (4.6 barg, ~155°C, ~6 sec). Samples of reference chips were collected after the chip wash. Samples of pre-treated chips were taken out after the impregnator.

Double disc refining

Pulps were produced in RGP68DD (double disc) refiners (Metso) using p-(DN72N816-817) and c-segments (DO52B036-037). There are three parallel primary DD-refiners in the line. The refiner plates were 72" in diameter and rotated at 1500 rpm each in opposite directions. The refiner housing pressure was 4.6 barg and the dilution water was controlled by an automatic consistency controller based on the production rate and motor power. The production rate was calculated from pulp flow and pulp consistency in the standpipe situated directly after the blow line. The set point for the plate gap was controlled by an

automatic specific energy consumption controller based on production rate and motor power. Pulp samples were collected from the blow line directly after the refiner.

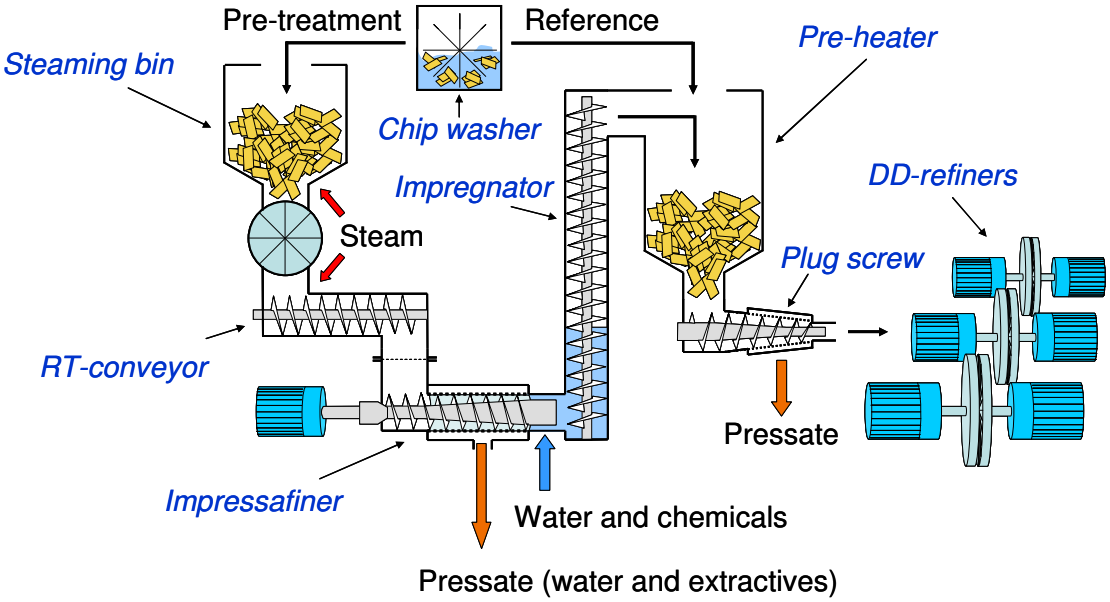

Figure 8. Detailed description of the chip pre-treatment equipment located in the double disc line. After the chip wash, it is possible to direct chips to the pre-treatment equipment or to the pre-heater.

Addition of sulphite

Sulphite was added to chips by mixing concentrated solutions of sodium bisulphite ($NaHSO_3$) and sodium hydroxide (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 Impressafiner production rate, which was determined by the rotational speed of the ingoing screw conveyor. The addition of NaOH was adjusted to render an impregnation solution with pH 9. Sulphite doses are given as percent Na_2SO_3 on oven dried basis.

2.1.3 The single disc line

The single disc line has two Twin 60 refiners (Sprout-Waldron) in series. Pulps were collected from blow lines after primary and secondary refiners.

2.2 Pressurised compressive chip pre-treatment

A number of trials were performed to evaluate different aspects of the mechanical chip pre-treatment. The Impressafiner setting for these trials are presented in Table 1.

The effects of mechanical pre-treatment on chip structure and water uptake were assessed by comparison between pre-treated and non-treated material.

The extractive removal was measured by comparing the extractive content of pulps produced with and without pre-treatment. Measurements of extractive removal were performed on four different occasions. On each occasion, the extractive removal was measured for different Impressafiner settings (Table 1) while the set points for refiner production rate, specific energy consumption and consistency were kept constant. The compression ratio was changed by changing the design of the compression screw in the Impressafiner.

The extractive content was determined by extraction with acetone using a Soxtec system followed by gravimetric measurement of the extractive amount, according to SCAN-CM 49:03.

Table 1. *Impressafiner settings for studies of mechanical pre-treatment.*

Study	Compression ratio	SEC Impressafiner (kWh/bdt)	RT-Pressure* (barg)
Microscopy and water uptake	3.6:1	25.0	1.8
Extractive removal (Jun 2009)	2.7:1	19.6 - 22.2	0.7 - 1.0
Extractive removal (Aug 2009)	2.7:1	17.2 - 18.9	1.2 - 1.8
Extractive removal (Sep 2009)	3.0:1	13.4 - 21.0	0.3 - 1.8
Extractive removal (Feb 2010)	3.6:1	19.5 - 22.9	1.8
Pulp properties and refining energy	3.6:1	24.0	1.8

*Pressure in the RT-conveyor situated at the inlet of the Impressafiner.

The effect of the Impressafiner pre-treatment on pulp properties and specific energy consumption was measured by comparing pulps produced with and without pre-treatment. The temperature in the refiner pre-heater increased from 65°C to 78°C after the start of the Impressafiner. Due to an increase in bulk density for pre-treated chips, the refining production rate increased from 9.7 bdt/h to 10.4 bdt/h (+6.6%) after the start of the Impressafiner. The specific refining energy consumption was varied from 1700 to 2150 kWh/bdt to produce refining curves for reference and pre-treated pulps. Consistency after refining was 39-41% for reference and 36-38% for pre-treated pulps.

2.3 Low dosage sulphite pre-treatment

2.3.1 Low dosage sulphite pre-treatment

Pulp samples were collected after the double disc refiner for different refining energy consumptions (1750-2100 kWh/bdt) and dosage of Na_2SO_3 (0, 0.12, 0.24, 0.61 and 1.21%). The chemical additions and Impressafiner settings are presented in Table 2. During the trial, the raw material composition was changed from the normal 50/50 blend to 30% sawmill and 70% round wood chips. After impregnation, the temperature of the chips was 62°C. The production rate over the refiner was 8.6 bdt/h and the blow line consistency after refining was 30-32%.

Samples of chips and pulp were allowed to cool down to room temperature for a few hours before they were frozen. The samples were then analysed for sulphur content (SCAN-CM 57:99).

Table 2. *Chemical additions and Impressafiner settings for studies of sulphite pre-treatment.*

Na_2SO_3 added (%)	$NaHSO_3$ (kg/bdt)	NaOH (kg/bdt)	Compression ratio	SEC Impressafiner (kWh/bdt)	Impregnation water
Low dosage sulphite pre-treatment					
0.00	0	0	2.7:1	18	Fresh water
0.12	1	0.3	2.7:1	18	Fresh water
0.24	2	0.6	2.7:1	18	Fresh water
0.61	5	1.4	2.7:1	18	Fresh water
1.21	10	3.8	2.7:1	18	Fresh water
Sulphonation and refining intensity					
0.00	0	0	3.6:1	24	PM* White water
1.24	10.2	3.6	3.6:1	24	PM* White water

*Paper Machine

2.3.2 Sulphonation and refining intensity

Pulp samples were collected after the double disc refiner for different refining energy consumptions (1450-1900 kWh/bdt). Chips were pre-treated with 0 and 1.24% Na_2SO_3. The chemical addition and Impressafiner settings are presented in Table 2. After impregnation the chips had a temperature of 80°C. The production rate over the refiner was 10.2 bdt/h and the blow line consistency after refining was 30-38%.

Pulps produced in the single disc line were collected for different refining energy consumptions (1300-2200 kWh/bdt). The production rate was 11.8 bdt/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 changed by adjusting the hydraulic pressure in the secondary refiner. No chip pre-treatment was used in the single disc line.

2.4 Chip samples and analyses

Water uptake

The water content of the chips was determined by dividing wet weight by oven dry weight after the following steps: 1) from process, frozen and thawed; 2) additional impregnation for 30 min. in the laboratory. Additional impregnation was performed at room temperature with distilled water in an air tight container connected to a vacuum pump. Before the wet weight was determined, excess water was removed from the chips by placing the chips on a mesh for 1 min. The mesh was then placed on a dry tissue for 1 min. to remove any remaining excess water from the chips.

Sulphur content

Chip samples pre-treated with only mechanical pre-treatment and with different dosage of Na_2SO_3 (*i.e.* 0.24, 0.61 and 1.21%) were collected directly after the impregnator. The samples were allowed to cool down to room temperature for a few hours before they were frozen. Chip samples were then analysed for sulphur content (SCAN-CM 57:99).

Microscopy

Chips of representative size with no pre-treatment, with only mechanical pre-treatment and with different dosage of Na_2SO_3 (*i.e.* 0.12, 0.24, 0.61 and 1.21%) were selected for microscopy. Transverse sections (~10 µm thick) were cut from the central parts of non-embedded chips using a Leitz sledge microtome and mounted on glass slides. Thereafter, the sections were stained with aq. 0.01% acridine orange for 2 min., washed briefly (1 min.) in distilled water and covered with cover slips. Sections were examined using a wide-field Leica DMRE fluorescence microscope fitted with mercury lamp and I3 513808 filter-cube (Leica, excitation 450-490 nm excitation, emission > 515 nm). Images were digitalized using a CCD camera (Leica DC 300F) and a digital imaging system (Leica Microsystems, Wetzlar, Germany).

2.5 Pulp samples and analyses

Five pulp samples were collected during a period of 12 min. for each process setting (*i.e.* for each mechanical pre-treatment setting, chemical dose or

specific energy consumption). The samples were mixed, frozen and analysed three times using the following methods: Hot disintegration (ISO 5263-3), length weighted fibre length and shives (Eurocon PulpEye), Canadian standard freeness (CSF) (ISO 5267-2), Rapid Köthen labsheets without white water circulation (ISO 5269-2, DIN 54358), density (ISO 534), tensile index and elongation (ISO 1924-2), tear index (ISO 1974), specific light scattering coefficient and light absorption coefficient (ISO 9416) and brightness (ISO 2470).

Length weighted fibre length is hereafter referred to as "fibre length", specific light scattering coefficient as "light scattering" and specific light absorption coefficient as "light absorption".

Additional analyses were also performed on chosen pulps, including: extractive (SCAN-CM 49:03) and sulphur content (SCAN-CM 57:99), fibre charge of whole pulp (SCAN-CM 65:02), Bauer McNett fractionation (SCAN-CM 6:05), Simons´ staining according to Fernando and Daniel (2010) and spectra of the diffuse reflectance factor (ISO 2469). The pH was also measured for some of the pulps by the following method. Twelve gram high consistency pulp (~30% dry content) was diluted with 60 mL deionised water. The sample was dewatered in a Büchner funnel after 15 min. and the pH measured on the eluate.

3 Results and discussion

In this chapter, results from mill scale trials with pressurised chip compression and sulphite pre-treatment are presented and discussed. First, the effects of mechanical pre-treatment on chip structure, extractive content and pulp properties are presented. Second, the effects of low dosage sulphite pre-treatment on chip structure, pulp, fibre and optical properties are given. Third, a comparison is made between pulp produced in the double disc line with sulphite pre-treatment and pulp produced in the single disc line. Thereafter, possible relations between chip pre-treatment and refining conditions are discussed. Finally some aspects of mill scale trials concerning reproducibility are presented.

3.1 Pressurised compressive chip pre-treatment

3.1.1 Microscopy and water uptake

Impressafiner pre-treatment resulted in partial disintegration of chips into material consisting of splinters and fragmented chips with cracks running along the longitudinal fibre axis (*Figures 9 and 10*).

Figure 9. Images showing the visible difference between reference chips (left) and pre-treated chips (right).

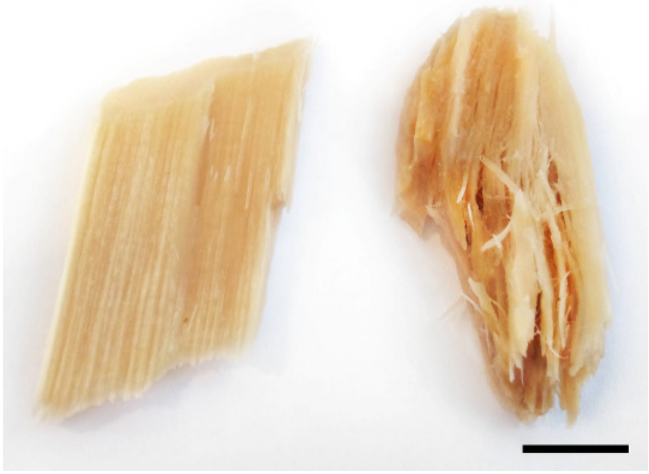

Figure 10. Image showing the visible difference between a reference chip (left) and pre-treated chip (right). Bar: 1 cm.

Observations on transverse sections from the pre-treated chips typically showed two types of cracking/splitting in the chip structure: i) splits located between both the fibres and rays running along the interphase between the middle lamella-primary wall regions and the secondary wall layer (*i.e.* S1) (*Figure 11a*, arrows); and ii) splits located in fibres at the interphase between middle lamella, primary cell wall and inner secondary wall layers (S1/S2) (*Figure 11b and c*, arrows). These cracks frequently occurred along the entire fibre axis in the longitudinal direction from the tips to central regions and were present in both the late- and earlywood although they were more prominent in the latter. With the splits located between the primary and secondary cell wall layers, it is likely that these sites of weakness were exaggerated during sectioning as shown by the orientation of the weaken S2 layer in the fibre lumen (*Figure 11b and c*). It was not possible to determine exactly where the splits occurred at the magnification used, although it is presumed to be in the region of the primary wall and the S1 layer. Kure *et al.* (1999) studied pressurised compressive chip pre-treatment of Norway spruce in pilot scale and reported similar fractures in the area between the middle lamella, the primary wall and S1 layer. The changes in chip and fibre structure noted presumably reflect the pressurised compression in the Impressafiner.

The changes in chip and fibre structure caused by pre-treatment should allow better penetration of fluids into the chips. Table 3 shows the water content of reference and pre-treated chips. The Impressafiner pre-treatment and the successive impregnation in the impregnator increased the water content of the chips with 0.27 m^3/bdt. Further impregnation in the laboratory showed that the lower water content of the reference chips did not only depend on insufficient water impregnation in the chip wash. Pre-treated chips showed highest water content after laboratory impregnation. Part of the increase

depends on the increase in surface area due to the reduced chip size. However the large pre-treated chips, handpicked to resemble the size distribution of the reference chips, also showed higher water content than reference chips after laboratory impregnation (Table 3). Therefore, the increase in water content for pre-treated chips can most likely be associated with changes in fibre structure accomplished by the Impressafiner pre-treatment.

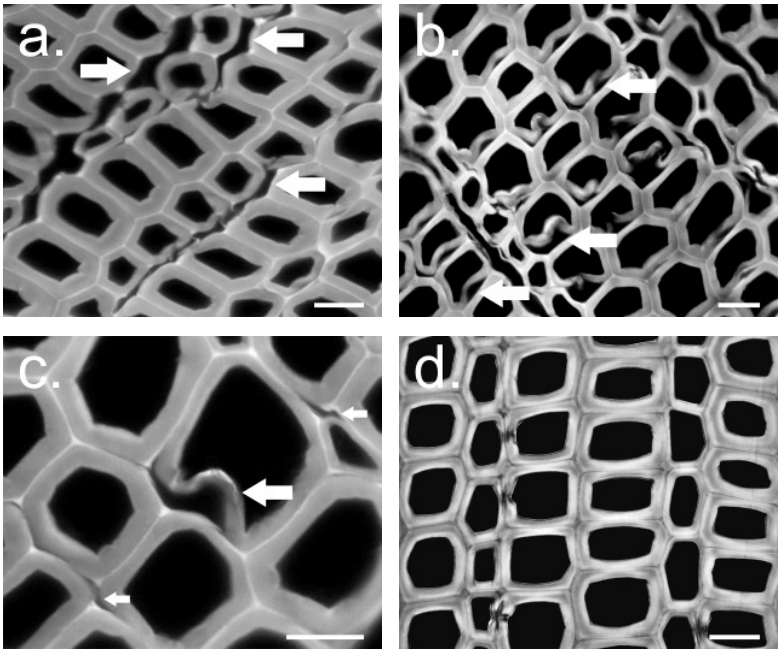

Figure 11. Transverse sections from Impressafiner pre-treated (a-c) and non-pre-treated (d) spruce chips. (a) Splits located between fibres (arrows) in middle lamella-primary cell wall layer regions; (b) Repeated orientation of the secondary wall into the fibre lumen (arrows) during sectioning due to weakness induced during the pre-treatment process; (c) Higher magnification of the splits seen in b. indicating weakness at the middle lamella/S1 interphase (arrows); (d) Section from a non-pre-treated spruce chip, no splits or orientation of the secondary wall into the fibre lumen are visible. Bars: a-d, 25 µm.

Table 3. *Water content of chips, m^3/bdt (dry content)*

Chip type	From process	Laboratory impregnation
Reference chips	1.36 (42.4%)	1.66 (37.6%)
Pre-treated chips	1.63 (38.1%)	2.09 (32.4%)
Pre-treated large chips	1.74 (36.6%)	1.92 (34.3%)

3.1.2 Extractive removal

The aim of these measurements was to measure the reduction in extractive content of pulp after primary refining with Impressafiner pre-treatment. Since some extractives are removed in both the plug screw and in the refiner, a comparison of chips before and after Impressafiner pre-treatment will not give the correct answer to this question.

The highest measured reduction of extractive content (acetone soluble) was 24% (*i.e.* from 1.40 to 1.07%) for pulps produced with pre-treated chips compared to reference pulps produced without pre-treatment (*Figure 12*). There was some scattering in the data, but a decrease of 19% could be statistically proven with 95% certainty. This reduction was achieved with the Impressafiner settings: Compression ratio 3.6:1, specific energy consumption 25 kWh/bdt and RT-preassure 1.8 barg. It should be noted that the acetone extract may also contain other groups than the lipophilic extractives, such as lignans.

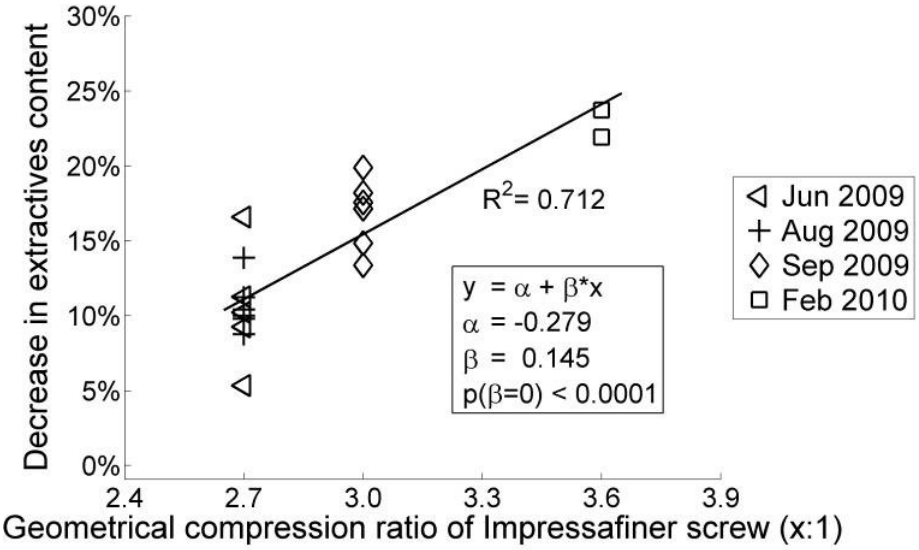

Figure 12. Decrease in extractive content (*i.e.* acetone soluble) for pulps produced from pre-treated chips compared to reference pulps. The low p-value for β=0 shows there was a significant decrease in extractive content when the geometrical compression ratio of the Impressafiner screw was increased.

The geometrical compression ratio of the Impressafiner compression screw had a significant effect on the decrease in extractive content (*Figure 12*). However, since the data was collected on different occasions, there could be some bias due to variation in raw material and process conditions. The extractive content for the reference pulps was between 1.19-1.40%. It was not possible to find any correlations between the extractive removal and the specific energy consumption of the Impressafiner or the RT-pressure of the pre-treatment in the study (data not shown).

No analyses were performed on the chemical composition of the extractives in this study. Tanase *et al.* (2010) however showed that the Impressafiner pre-treatment primarily removed resin acids from the resin canals, while the extractives found in parenchyma cells were not removed to the same extent. Tanase *et al.* (2010) evaluated the same Impressafiner installation as in this

study; operating with geometrical compression ratio 2.7:1. The authors showed an extractive removal of 15% when spruce chips were compared before and after the Impressafiner. However Tanase *et al.* (2010) measured the extractive content by extraction with a mixture of cyclohexane and acetone (9:1), followed by GC analysis and therefore their results are not directly comparable to the results presented in this study.

3.1.3 Pulp properties and refining energy

Several trials were performed to evaluate the effects of mechanical pre-treatment on pulp properties. No significant effects on pulp properties were observed when the Impressafiner was operated with compression ratio 2.7:1 and 3:1. However this may be a result of not using a proper evaluation technique during these trials.

Two trials were performed after the installation of the compression screw with compression ratio 3.6:1. These trial were performed with a more accurate evaluation technique (as described in "Materials and methods") and with better control of process parameters. The fist trial indicated that the Impressafiner pre-treatment reduced the specific energy consumption by ~100 kWh/bdt when compared at equal tensile index. However there was some process related problems during this trial and therefore these results are not presented here. The results from the second trial with compression ratio 3.6:1 are presented below.

Pulp produced from pre-treated chips had higher tensile index, light scattering and elongation than reference pulp produced with the same total specific energy consumption (*Figure 13b-d*). Approximately 120 kWh/bdt (*i.e.* 6%) less energy was needed to reach a tensile index of 47 Nm/g for pre-treated chips. There was no apparent difference in fibre length for pre-treated pulps (*Figure 13a*). The specific energy consumption in the Impressafiner was 24 kWh/btd which is included in "Total SEC" in *Figure 13*. The results obtained are consistent with earlier findings for Norway spruce in pilot scale (Kure *et al.* 1999).

Specific trials have been conducted in the double disc line to evaluate the effects of consistency, production rate and pre-heater temperature on pulp properties. No significant effects could be found for these parameters (Muhić 2010). Therefore, the changes in production rate (9.7 to 10.4 bdt/h), pre-heater temperature (65 to 78°C) and consistency (39-41% to 36-38%) after the start of the Impressafiner should not have affected the energy efficiency in the refiner.

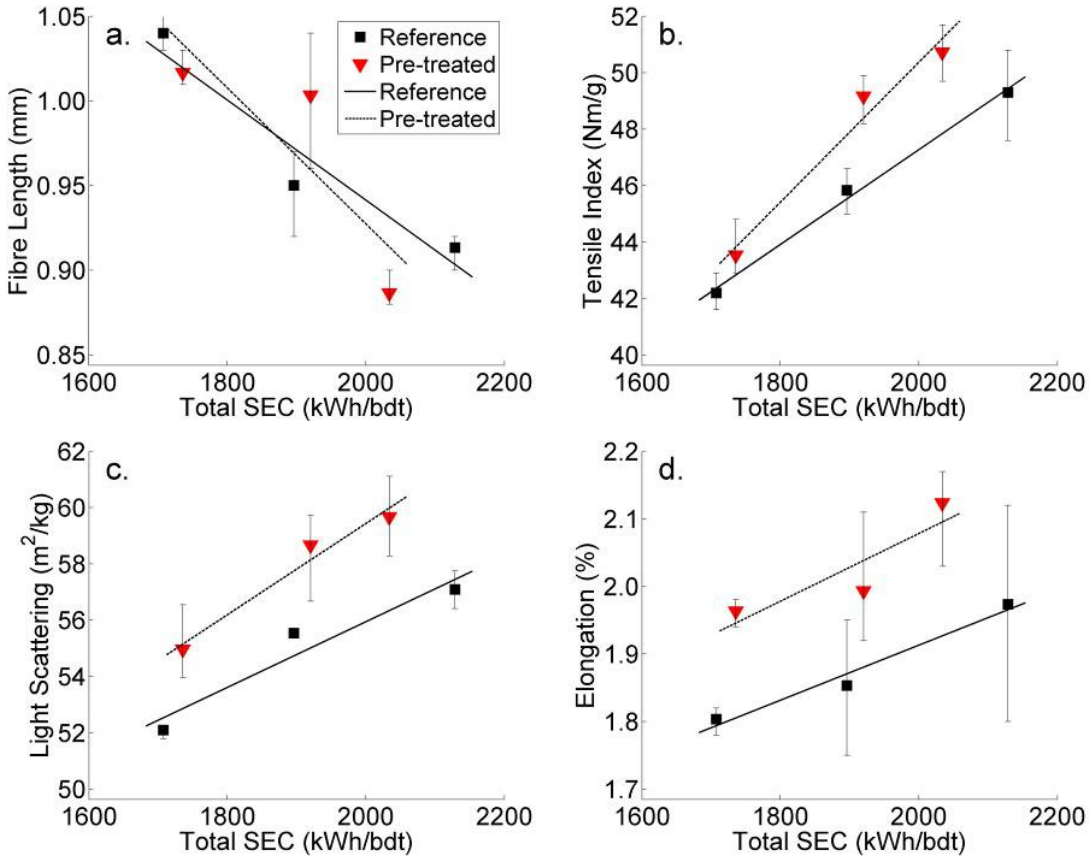

Figure 13. Fibre length (a), tensile index (b), light scattering (c) and elongation (d) vs. total specific energy consumption (SEC). Total SEC includes the electrical energy consumption in the Impressafiner and refiner. Data points are mean values of three measurements. Error bars show the minimum and maximum values of the three measurements.

3.1.4 Chip pre-treatment and energy efficiency

The Impressafiner pre-treatment reduced the extractive content and caused structural changes in chips. One or both these effects are most likely the cause for the increase in energy efficiency during refining of pre-treated chips. However, it is not possible to say with certainty if one effect was more important than the other from the results in this study.

It is well known that extractives, especially those with long carbon chains have a negative impact on the strength of paper from TMP processes (Sundberg *et al.* 2000, Kokkonen *et al.* 2002). However, a reduction in the extractive content by 24% corresponds to a rather low absolute reduction of extractives low due to the low amount of extractives in the raw material. Therefore, it is not possible to say with certainty if the reduction in the acetone extractive content from 1.40 to 1.07% was the cause for the increase in tensile index for pulps produced from pre-treated chips.

As outlined above, another plausible explanation for the improved energy efficiency during refining of pre-treated chips could be the weaknesses or splits

created between the primary wall and the secondary wall layers during the Impressafiner pre-treatment. It is hard to find literature that only describes the effects of such structural changes in chips on energy efficiency since the mechanical chip pre-treatment techniques used to change the chip structure in such a way also affect the extractive content. However Salmén *et al.* (1985) showed that, reducing frequency and increasing amplitude of the initial compression cycles would increase the effectiveness of the structural breakdown of wood. Given these results, it should be possible to achieve a more energy efficient structural breakdown of chips with the Impressafiner compared to refining due to the lower frequency and higher amplitude of the compression cycles.

It should be noted that structural breakdown of wood does not directly imply a desirable fibre development for papermaking. However, pulp produced from pre-treated chips had similar fibre length and increased tensile index compared to pulp produced from untreated chips at certain specific energy consumption. Such effect on pulp properties may imply that the structural breakdown caused by the Impressafiner was beneficial for fibre development. The increase in energy efficiency for production of pulps with mechanical chip pre-treatment may therefore be addressed to a more energy efficient mechanical treatment in the Impressafiner.

3.2 Low dosage sulphite pre-treatment

3.2.1 Sulphonation

The amount of sulphur found in chips without sulphite addition was 60 mg/kg (*i.e.* 0.02% as Na_2SO_3), which originates from the natural sulphur content in wood. Pulps without sulphite addition had a sulphur content of 210-240 mg/kg (*i.e.* 0.08-0.09% as Na_2SO_3) (*Figure 14*), which originates from the natural sulphur content in wood and also sulphur containing compounds in the refiner dilution water. The refiner dilution water is a mix of fresh water and paper machine white water which may contain sulphur containing compounds originating from the dithionite bleaching step further down the process.

The temperature of the chips after impregnation was 62°C at which sulphonation reactions are expected to be rather slow. Chip samples were collected directly after the impregnation and were then allowed to cool down to room temperature for a few hours before they were frozen. Much of the sulphonation of chip samples probably occurred after the samples were collected. Approximately 60-80% of added sulphite was chemically bound to chips, where the lowest dosage had the highest conversion ratio (*Figure 14*).

After impregnation, chips were fed to the pre-heater which may be better described as a retention bin since steam is normally not added in this position. The retention time in the pre-heater was 10-15 min. and the temperature of the chips did not change during this time. It may be assumed that some sulphonation occurred here. 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 after the plug screw (~155°C, 6 sec) and it is assumed that most of the sulphonation reactions occurred here and during refining. The pulp samples were allowed to cool down for a few hours before they were frozen and some sulphonation reactions may also have occurred during that time.

Approximately 55-70% of added sulphite was found as sulphur content in pulp after refining. However if the sulphur content of sulphite pre-treated pulps are subtracted by the amount of sulphur found in pulp produced without sulphite addition the conversion ratio is only 37-50%.

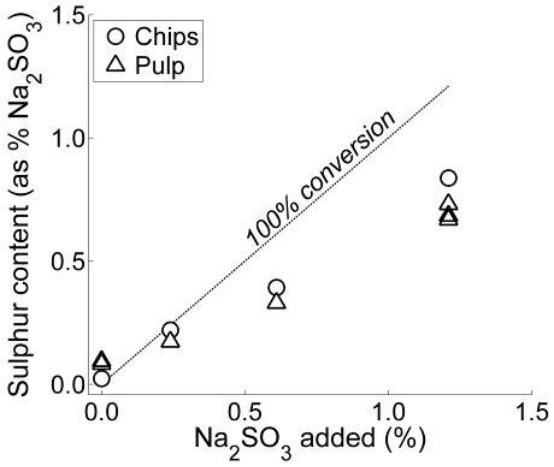

Figure 14. Sulphur content in chips and pulp (as % Na_2SO_3) vs. % Na_2SO_3 added.

3.2.2 Microscopy study of sulphonated chips

As showed earlier, chips pre-treated in the Impressafiner without sulphite addition typically showed two types of cracking/splitting in the chip structure: i) splits located between both the fibres and rays running along the interphase between the middle lamella-primary wall regions and the secondary wall layer (*i.e.* S1) (*Figure 15a*, arrows); and ii) splits located in fibres at the interphase between middle lamella, primary cell wall and inner secondary wall layers (S1/S2) (*Figure 15b*, arrows).

Introduction of sulphite at pH 9 into the impregnation liquid resulted in irregular and mild swelling of the secondary wall into the cell lumen for all dosages (*i.e.* 0.12-1.21% Na_2SO_3) (*Figure 15c-f*). Swelling of the secondary S2 wall layer into the fibre lumen was most obvious in the latewood fibres as

reflected by the inward buckling of the S2 and S3 wall layers (*Figure 15c*, arrows). Additional effects of sulphite addition noted were an overall loss of chip and fibre integrity (*Figure 15d and f*). It was not possible to determine any major differences in morphological effects between cross-sections from the different dosages of sulphite addition.

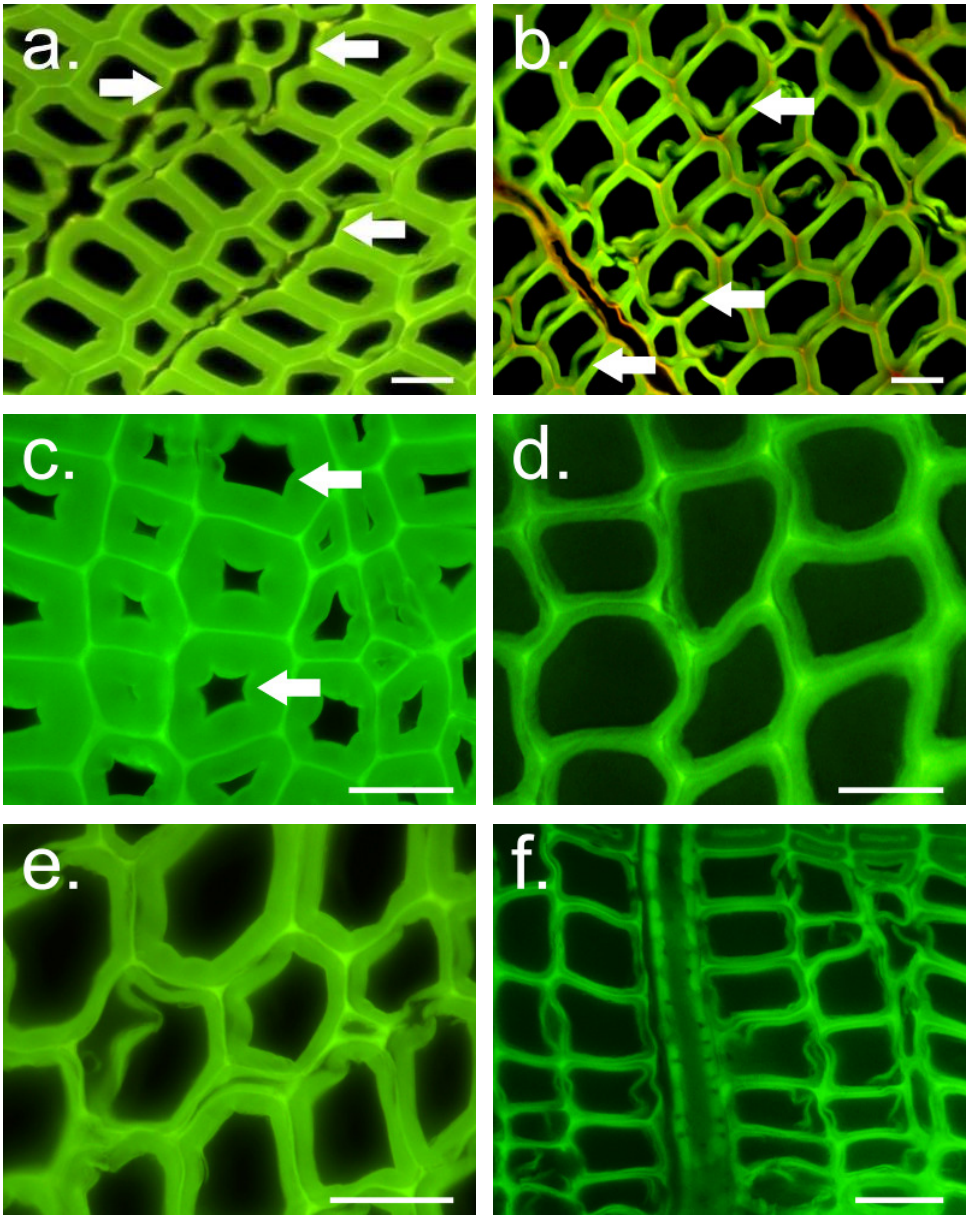

Figure 15. Transverse sections from chips pre-treated in the Impressafiner (a) and (b) and with different additions of Na_2SO_3. (c) 0.12%, (d) 0.24%, (e) 0.61%, (f) 1.21%. (a) and (b) were also presented in *Figure 11* and are presented here again to enable comparison between mechanical pre-treatment and pre-treatment with sulphite addition. It should be noted that the compression ratio in the Impressafiner was 3.6:1 for (a) and (b) and 2.7:1 for (c)-(f). Bars: a-f = 20 µm.

These observations indicate that Impressafiner pre-treatment opens up the internal structure of the chips, inducing cracks and splits between the cellular

elements thereby allowing for a more rapid influx of the impregnation fluid. It was also shown in Paper I that Impressafiner pre-treated chips were able to absorb more water compared to non-pre-treated chips. The changes in internal structure caused by the Impressafiner pre-treatment may therefore lead to more rapid and even sulphonation throughout the chips. It is probable that the impregnation fluid is redirected to additional impregnation pathways through the cracks and splits produced during mechanical pre-treatment. It may be speculated that such a change in the impregnation pathway could also affect the sites of sulphonation (*i.e.* the distribution of sulphonation reactions between the middle lamella, primary and secondary cell walls). However, further studies are needed in order to investigate if such an effect actually exists.

3.2.3 Pulp properties

The most prominent effect of sulphite pre-treatment was an increase in tensile index (*Figure 16a*). The highest dose of sulphite (*i.e.* 1.21% Na_2SO_3) increased the tensile index with 7.6 Nm/g compared to non-sulphonated pulps at specific energy consumption 1950 kWh/bdt. When compared at tensile index 47 Nm/g, the specific energy consumption was reduced with ~320 kWh/bdt (*i.e.* ~15%) for pulps pre-treated with 1.21% Na_2SO_3 compared non-sulphonated pulps.

The relationship between density and tensile index was not affected by sulphonation (*Figure 16b*). Light scattering was slightly reduced (*i.e.* 1.6 m^2/kg) by the highest addition sulphite compared to non-sulphonated pulps at equal specific energy consumption (*Figure 16*c). However, when compared at equal tensile index, the highest dosage of sulphite (1.21% Na_2SO_3) had a light scattering that was 9.1 m^2/kg lower than for non-sulphonated pulps (*Figure 16d*). Of the 9.1 m^2/kg, 7.6 m^2/kg was a result of the reduction in specific energy consumption needed to reach similar tensile index.

Fibre length was not affected by addition of sulphite when compared at similar specific energy consumption (*Figure 16e*). This is somewhat surprising since there are numerous reports from pilot scale trials showing increased fibre length for sulphite pre-treated pulps when refined at equal specific energy consumption (Argyropoulos and Heitner 1991; Atack *et al.* 1980; Axelson and Simonson 1982). However, Chagaev *et al.* (2005) showed that fibre length was not affected by sulphite pre-treatment during high intensity refining.

The two highest dosages of sulphite (*i.e.* 0.61, 1.21% Na_2SO_3) reduced the shive content at the lower energy level (*i.e.* ~1780 kWh/bdt) compared to non-sulphonated pulps (*Figure 16f*).

Axelson and Simonsson (1982) reported a maximum in tensile index and light scattering at a sulphur content of 0.2% (as Na_2SO_3), which was later confirmed by Westermark *et al.* (1987) and Svensson *et al.* (1994). In our

study, tensile index and light scattering followed linear relations with the amount of sulphite added without any maximum in the low dose range (*Figure 16a, c* and Paper II).

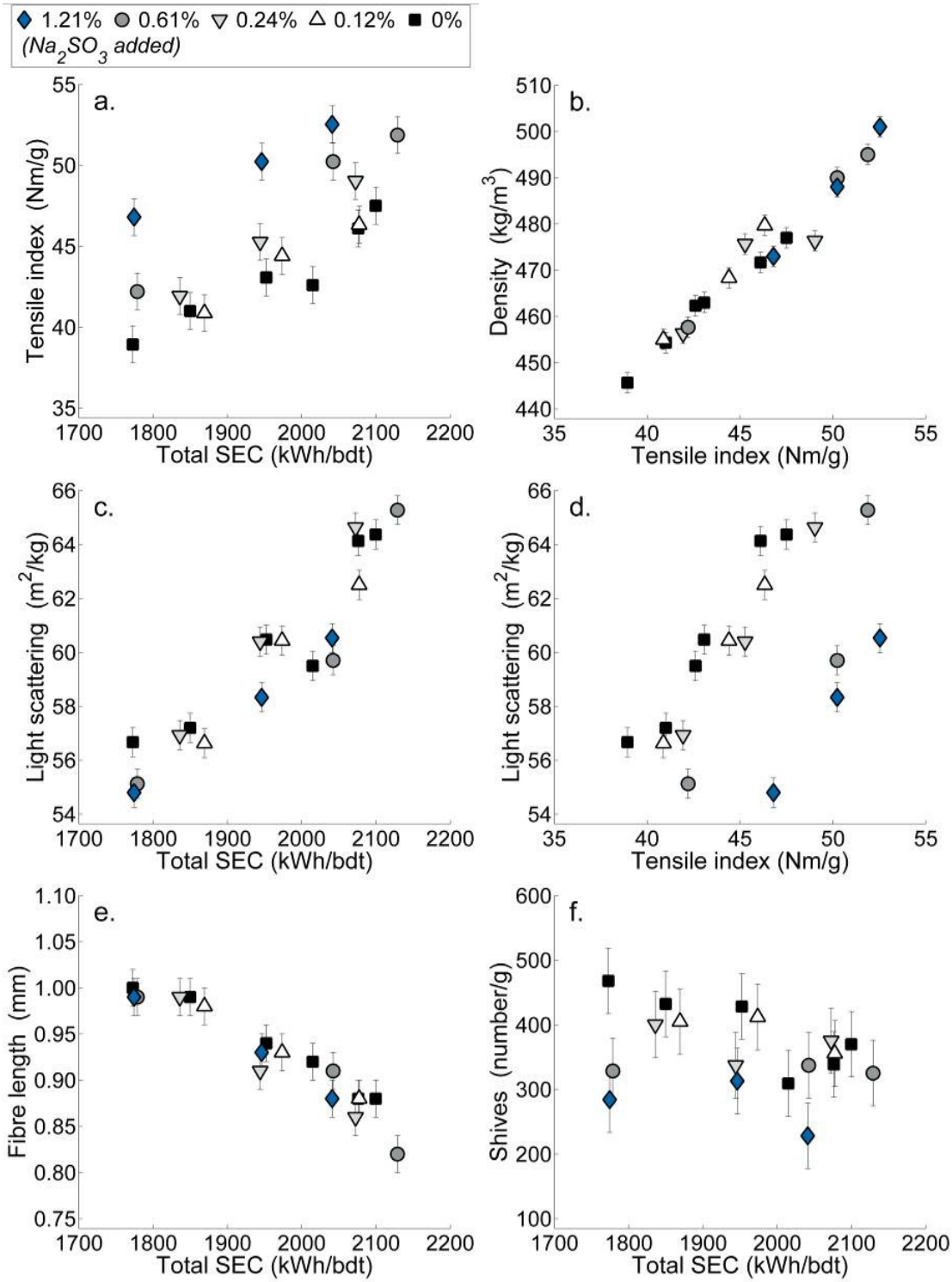

Figure 16. Tensile index (a), light scattering (c), fibre length (e) and shives (f) vs. specific energy consumption and density (b) and light scattering (d) vs. tensile index for different additions of Na_2SO_3. Error bars show 95% confidence intervals.

The difference in results between this study and previous studies are outlined in *Figure 17*. All studies were performed using Norway spruce at similar pH. However, our study was performed using a different impregnation technique (Impressafiner pre-treatment) and with a mill scale high intensity refiner. The explanation for the maximum in tensile index and light scattering offered by Axelson and Simonson (1982) was that a sulphur content of 0.2% (as Na_2SO_3) gave a suitable decrease in the softening temperature in relation to the temperature and frequency of the breaker bar section and the refining zone of the refiner used in the study. It is therefore assumed that the different results obtained in this study are caused by the different refining conditions. Nevertheless, it is not possible to rule out the effects of the Impressafiner pre-treatment as a cause to these differences through opening up the chip structure for chemical influx as described earlier.

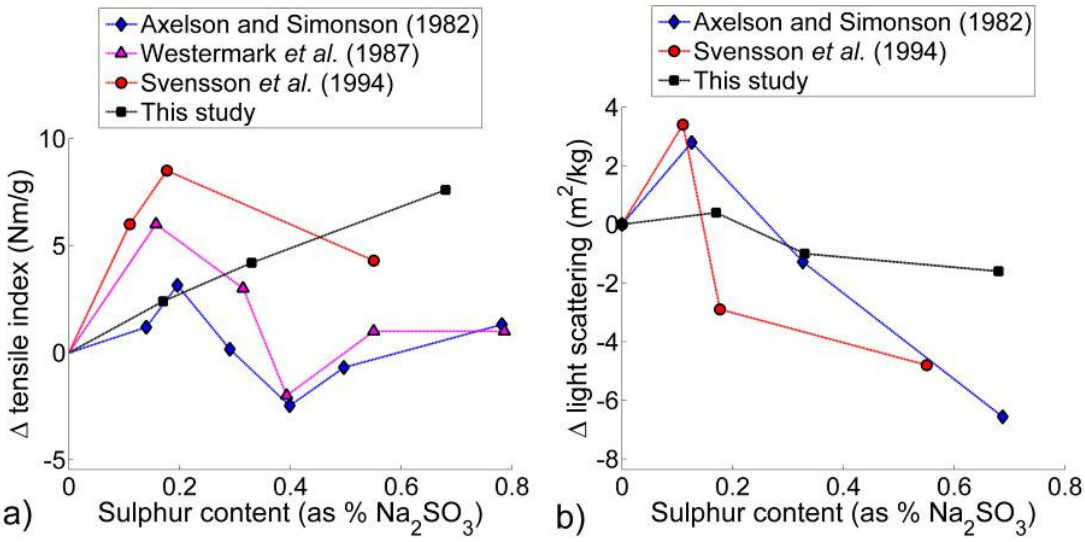

Figure 17. Comparison of changes in tensile index (a) and light scattering (b) at certain specific energy consumption vs. sulphur content (as % Na_2SO_3) for different studies. The lines between data points in (a) and (b) do not symbolise linear relations between data points.

3.2.4 Fibre properties

Table 4 shows detailed properties for six individual pulps produced with different dosage of sulphite at two energy levels. The addition of 1.21% Na_2SO_3 at pH 9 increased the pulp pH from ~5.7 to ~7.2 after refining. For the low energy pulps (1780 kWh/bdt) fibre charge was increased by 40 mmol/kg by the addition of 1.21% Na_2SO_3 which corresponds to an increase in sulphonic acid groups of 0.5% (as Na_2SO_3).

Distribution of the Bauer-McNett fractions was not significantly affected by addition of sulphite at certain specific energy consumption (Table 4). The

proportion of the fine fraction (< 200) and the 16-30 fraction did not change in any distinct way in response to either increased refining energy or degree of sulphonation. The proportion of the fine fraction (< 200) is expected to increase when the refining energy is increased, although the lack of a correlation between the two may be explained by the rather uncertain measurement of the fine fraction. By applying a linear regression model to the data in Table 4, it was possible to show that only the specific energy consumption and not the degree of sulphonation had a significant effect on the proportion of the > 16 fraction (Paper II).

Table 4. *Pulp properties for different refining energies and dosage of Na_2SO_3*

Total SEC (kWh/bdt)		1780	1780	2080	2080	2040	2040
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
Pulp pH after refining		5.72	7.01	5.75	6.00	6.31	7.23
Fibre charge in whole pulp (mmol/kg)		70	110	71	80	95	96
Bauer-McNett fractions (%)	<200	24.9	22.7	25.1	27.4	26.6	26.3
	30-200	27.9	28.6	32.0	31.9	30.7	31.3
	16-30	23.5	24.1	24.2	23.0	23.3	24.3
	>16	23.7	24.6	18.7	17.7	19.4	18.1
Degree of D/IF of Fibre Cell Walls (Simons´ staining of Bauer-McNett 16-30 fraction) (%)	High	16.8	29.3	24.5	22.8	29.0	39.0
	Low	26.3	26.3	27.5	26.8	28.0	26.3
	Non	57.0	44.5	48.0	50.5	43.0	34.8
Fibre length (mm)		1.00	0.99	0.88	0.86	0.91	0.88
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

Fernando and Daniel's (2010) version of Simons´ staining of the 16-30 fraction was used to assess the degree of delamination/internal fibrillation (D/IF) of the pulps (Table 4). Sulphite pre-treatment showed an effective increase in D/IF. Simons´ staining measures the accessibility of interior surfaces in the fibre cell wall through selective staining of fibres containing pores larger than 5 nm (Yu *et al.* 1995). This type of internal fibre development has been shown earlier to correlate positively with the whole pulp tensile index (Stone *et al.* 1968; Fernando *et al.* 2011). Stone *et al.* (1968) suggested that this correlation was a result of an increased flexibility and collapsibility for fibres with increased D/IF. Fibre flexibility and collapsibility have been described as dependant on the fibre dimensions (fibre cell wall thickness and fibre width) together with the elasticity (Young's modulus) of the fibre cell wall (Paavilainen 1993).

Increasing the amount of pores larger than 5 nm in the fibre cell wall (as measured by Fernando and Daniel's (2010) method of Simons´ staining) should correlate with a decrease in the elasticity of the fibre cell wall and thereby with increasing flexibility and collapsibility of the fibre.

By using a linear regression model it was possible to show that the percentage of fibres with high D/IF was significantly increased by both increased specific energy consumption and increased degree of sulphonation. An increase in the pulp sulphur content by 0.1% (as Na_2SO_3) gave an equivalent increase in the percentage of fibres with high D/IF as increasing the refining energy with 100 kWh/bdt (Paper II).

The effective increase in D/IF caused by sulphonation is probably the explanation to the increase in tensile index and density for sulphite pre-treated pulps. However, an increase in fibre charge is also known to increase these properties (Barzyk 1997). The increase in density could also explain the lower light scattering at certain amount of fine material for the sulphonated pulps when compared to non-sulphonated pulps. When a sheet is densified without changing the amount of fine material or the degree of external fibre fibrillation, less light reflective surfaces are available to scatter light. It should be noted that a large range of the pore sizes measured by Simons' staining are much too small to scatter light in the visual spectra.

3.2.5 Optical properties

Sulphite pre-treated pulp had lower light absorption compared to non-sulphonated pulps (*Figure 18a*). The decrease in light absorption resulted in increased brightness (*Figure 18b*) even though there was a slight decrease in light scattering for sulphite pre-treated pulps. Brightness is measured as the diffuse reflectance factor using a filter or a mathematical function having an effective wavelength of 457 nm. The light absorption is calculated from the diffuse reflectance factor and opacity measured with a filter or calculated with a mathematical function having an effective wavelength of 557 nm.

In *Figure 19a*, the diffuse reflectance factor is presented for the wavelength range 420-700 nm. Spectra for pulps from two different refining energy levels, with and without sulphite pre-treatment are shown. The diffuse reflectance factor was higher for sulphite pre-treated pulps. *Figure 19b* shows the difference in spectra (by subtraction) between sulphonated and non-sulphonated pulps for the two different refining energy levels. The difference was most pronounced between 450 and 550 nm, corresponding to a region where lignin has a high optical activity. This can be explained by degradation of chromophoric structures in lignin during sulphonation.

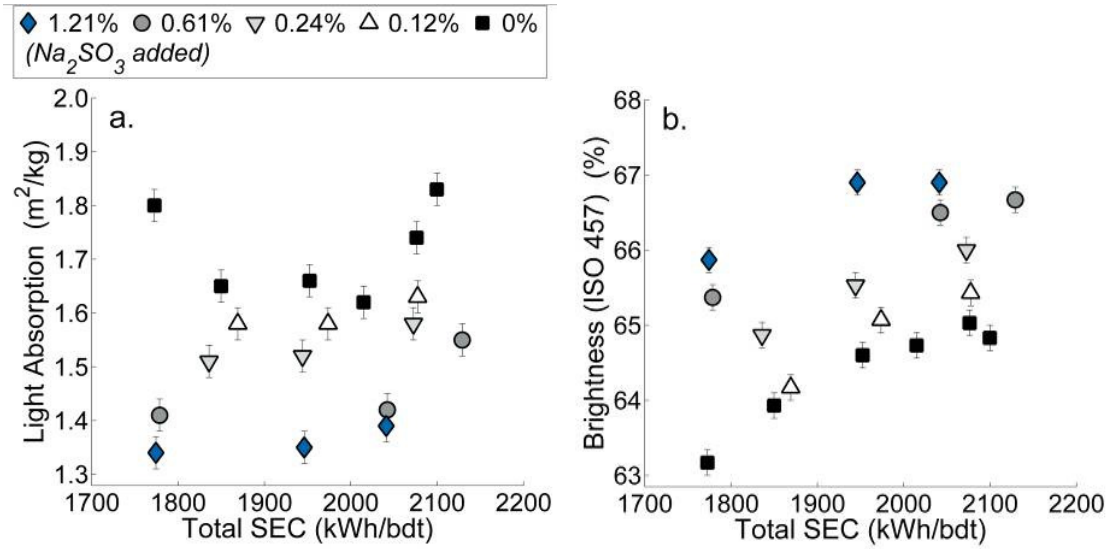

Figure 18. Light absorption (a) and brightness (b) vs. total specific energy consumption for different additions of Na_2SO_3. Error bars show 95% confidence intervals.

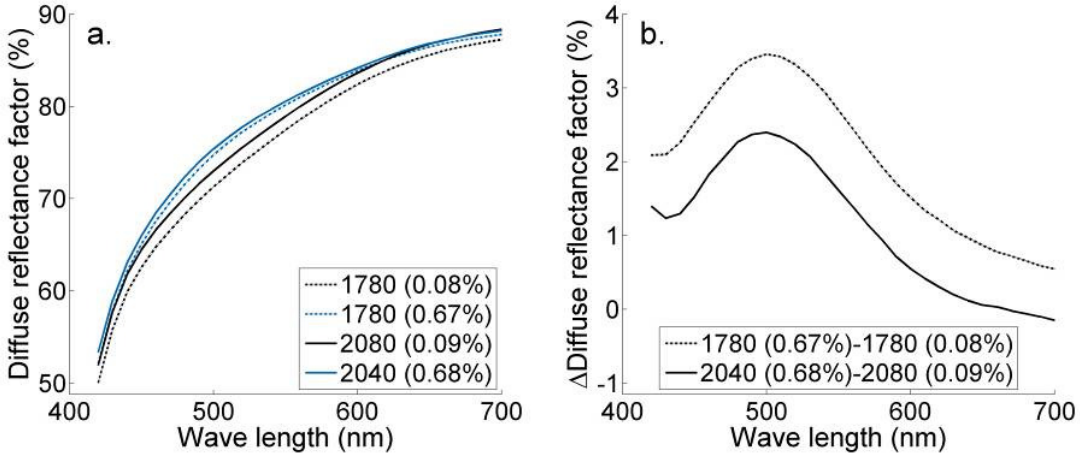

Figure 19. a) Diffuse reflection intensity measured over 420-700 nm for four of the pulps included in Table 4. The legend shows total specific energy consumption and sulphur content as % Na_2SO_3 in brackets. b) Difference in diffuse reflection intensity between sulphonated and non-sulphonated pulps measured over 420-700 nm.

3.2.6 Sulphonation and refining intensity

High opacity is an important benefit of mechanical pulps aimed for printing grade papers. It is therefore not desirable that light scattering at a certain tensile index is reduced by sulphite pre-treatment as seen in *Figure 16d*. A possible solution to this problem is to increase the amount of fine material of sulphite pre-treated pulps and thereby increase the surface area available to scatter light inside the sheet. Muhić *et al.* (2011) showed that it was possible to increase the light scattering of double disc pulps by ~15 m^2/kg at a certain tensile index and specific energy consumption by increasing refining intensity through a change

in segment design to a more "feeding" segment (Turbine^{TM}). Since the increase in light scattering was accompanied by a reduction in both fibre length and freeness it can be assumed that the increased light scattering to a large extent was a result of an increased proportion of fine material. The study by Muhić *et al.* (2011) was also performed in the double disc line at Braviken. Unfortunately the Turbine^{TM} segments have not yet been combined with sulphite pre-treatment at the mill. However, in an attempt to illustrate how sulphite pre-treatment and increased refining intensity can be combined to reduce specific energy consumption and at the same time conserve tensile index and light scattering, sulphite pre-treated pulps refined in the double disc refiner were compared to pulps from a 2-stage single disc line with lower refining intensity.

Pulps produced in the double disc refiner with only mechanical pre-treatment had higher tensile index than pulps produced in single disc refiners, when compared at equal specific energy consumption (*Figure 20a*). The specific energy consumption was 440 kWh/bdt (20%) lower for double disc refining together with mechanical pre-treatment when compared to single disc refining at tensile index 45 Nm/g (Table 5). Based on the evaluation of the mechanical pre-treatment presented above (*Figure 13b*), it may be assumed that the mechanical pre-treatment was responsible for reducing the specific energy consumption by approximately 120 kWh/bdt. This leaves a difference in specific energy consumption of 320 kWh/bdt (15 %) between double disc and single disc refining when compared at equal tensile index. One reason for this difference may be the higher refining intensity in double disc refining. However, it should be noted that the single disc line was supplied with 100% round wood chips in contrast to the 50% sawmill and 50% round wood chips used in the double disc line. Increasing the part of sawmill chips will probably increase tensile index and reduce light scattering at certain specific energy consumption (Corson 1984).

Addition of 1.24% Na_2SO_3 to the pre-treatment further increased the tensile index of the double disc pulps by 7.2 Nm/g at certain specific energy consumption (*Figure 20a*). The tensile index of sulphite pre-treated double disc pulp was 13.5 Nm/g higher compared to single disc pulp at 1700 kWh/bdt (*Figure 20a*). The light scattering of double disc pulps without sulphite addition was 10.3 m^2/kg higher than for single disc pulp when compared at 1700 kWh/bdt (*Figure 20b*). The addition of 1.24% Na_2SO_3 reduced light scattering for double disc pulps by 1.8 m^2/kg at certain specific energy consumption (*Figure 20b*).

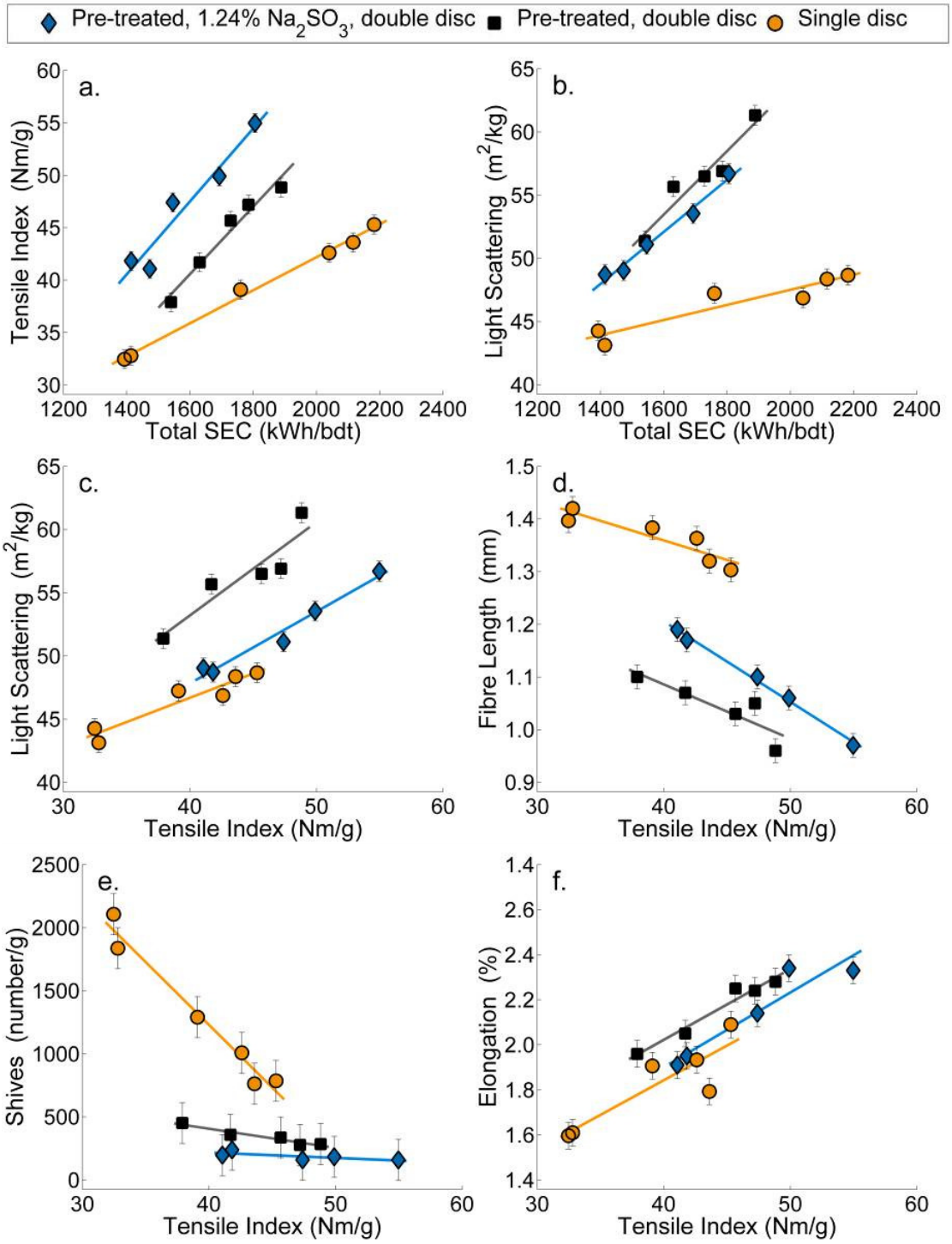

Figure 20. Tensile index (a) and light scattering (b) vs. total specific energy consumption and light scattering (c), fibre length (d), shives (e) and elongation (f) vs. tensile index. Error bars show 95% confidence intervals.

Compared at similar tensile index, double disc pulps had shorter fibres, less shives, higher elongation and higher light scattering than single disc pulps (Table 5). Similar changes in pulp properties have been presented earlier for

increased refining intensity (Kure and Dahlqvist 1998). Pulps produced with sulphite pre-treatment (1.24% Na_2SO_3) and double disc refining reached a tensile index of 45 Nm/g with ~650 kWh/bdt (30%) lower refining energy compared to single disc pulps (Table 5). The sulphite pre-treatment reduced the light scattering for double disc pulps by 6.2 m^2/kg when compared at tensile index 45 Nm/g. However, the light scattering was still higher (2.3 m^2/kg) than for single disc pulp at similar tensile index (Table 5). The relation between light scattering and tensile index is shown in *Figure 20c*. Other attributes of the sulphite pre-treated double disc pulps when compared at equal tensile index was lower fibre length and shive content and higher brightness and slightly higher elongation than single disc pulps (*Figure 20d-f*, Table 5).

Table 5. *Pulp properties interpolated to 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
Total SEC (kWh/bdt)	1737	1532	2180

3.3 Chip pre-treatment and refiner plate gap

A smaller refiner plate gap was needed to reach the same specific energy consumption for pre-treated chips compared to reference chips (*Figure 21*). The decrease in plate gap for pre-treated chips depends partly on the changes in consistency, production rate and pre-heater temperature after the start of the Impressafiner. Further trials are needed to measure the exact effect of the Impressafiner pre-treatment on plate gap. However, the decrease in plate gap for pre-treated chips was observed in every trial with mechanical pre-treatment and was larger than what could be explained by the changes in the process parameters above.

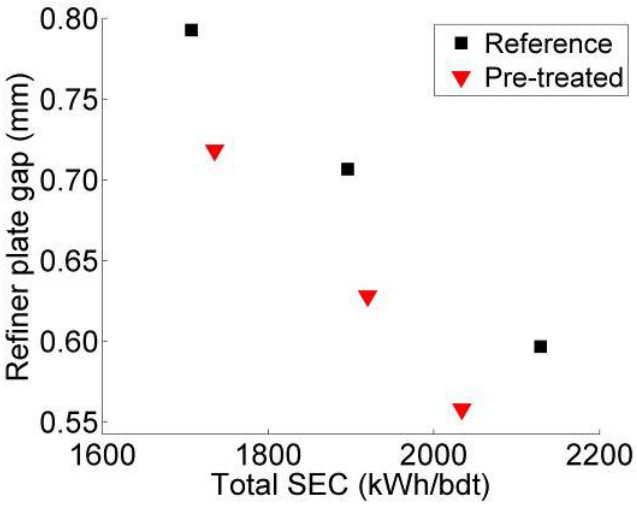

Figure 21. Refiner plate gap vs. total specific energy consumption for non-pre-treated chips and mechanically pre-treated chips.

According to Miles and Omholt (2008), the refiner plate gap for a certain grove depth, is a function of the uncompressed density and the stress-strain curve of the incoming material and also the refining intensity (*Eq 1)*. If fibres are collapsed during compression in the Impressafiner pre-treatment it seems reasonable that the unstrained density for these fibres would be higher than for fibres in the reference chips. Hence, this could provide an explanation for the decreased plate gap seen for pre-treated chips. The effect could be compared to the conditions in a secondary refiner where a smaller plate gap is needed to reach the same specific energy consumption compared to the primary refiner. An increased unstrained density for pre-treated fibres offers a basic explanation for the decrease in plate gap, however more studies are required to establish if this is the only reason.

Equation 1. Function for the plate gap at constant grove depth (Miles and Omholt 2008).

$$G = \frac{b}{\ln(b\rho_0 e_{sp}) - a} - 1$$

Where:

G = plate gap

ρ_0 = initial unstrained density of incoming material

e_{sp} = specific energy per impact (refining intensity)

a, b = intercept and slope, respectively, of the stress-strain curve for incoming material

A similar effect was also observed for the sulphite pre-treatment. The plate gap at certain energy consumption for mechanically pre-treated chips was further reduced upon the addition of sulphite (*Figure 22a*). There were no significant changes in consistency, production rate and pre-heater temperature upon the addition of sulphite. Further, the decrease was not a result of an increase in the hydraulic force acting on the refiner plates (*Figure 22b*). Assuming that the addition of sulphite did not affect the distribution of pulp in the plate gap, the decrease in plate gap should be a result of a change in the stress/strain relationship for the pulp due to the sulphonation of lignin. As mentioned above Miles and Omholt (2008) showed that the stress/strain relationship of the refined material is one of the factors affecting the plate gap. Measurements on sulphonated white spruce (*Picea glauca*) have shown that both the stress plateau and the toughness are reduced linearly to the sulphonate content (Mao *et al.* 2004). It has also been shown that sulphonation (1.6% as Na_2SO_3) of eastern black spruce (*Picea mariana*) slightly reduces the storage modulus in the glass transition phase for loadings in the transverse direction (Heitner and Salmén 1994). The storage modulus describes the elastic behaviour of viscoelastic materials. It is not trivial to relate these measurements performed on wood blocks and at low frequencies to the conditions in a mill scale refiner where the wood is divided into individual fibres and where the frequency is much higher. However, the changes in the compression behaviour of wood caused by sulphonation may be the explanation to the decrease in plate gap.

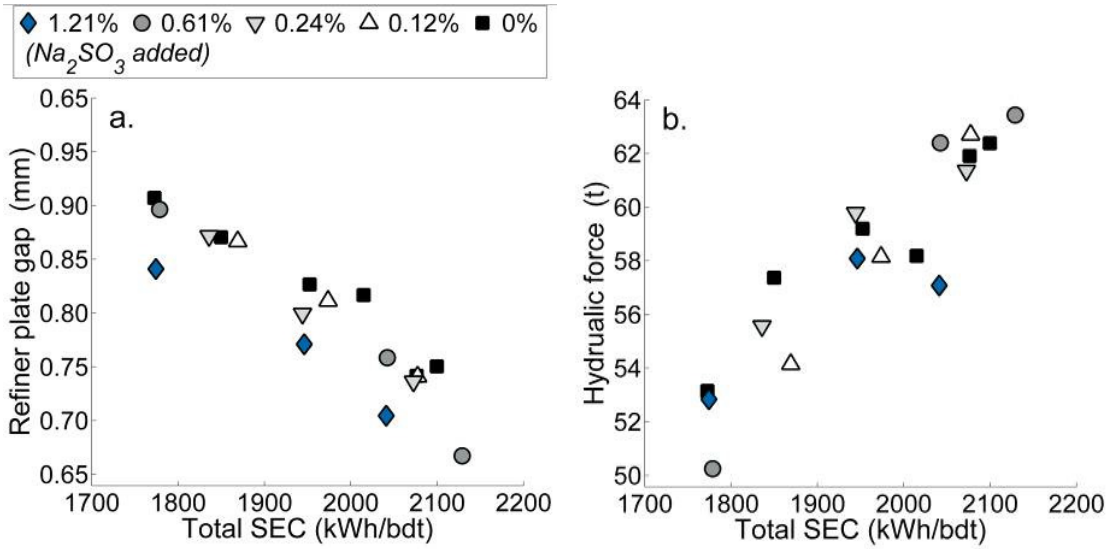

Figure 22. Refiner plate gap (a) and hydraulic force (b) vs. total specific energy consumption for mechanically pre-treated chips and for mechanically pre-treated chips with different dosages of sulphite.

It is not possible to say if the reduction in plate gap caused by mechanical and sulphite pre-treatment had a significant effect on pulp properties in the trials performed in this thesis. Reducing the plate gap has been suggested to increase the refining intensity (Tienvieri *et al.* 1999). If so, the reduction in plate gap may have affected pulp properties and thereby the energy efficiency.

Muhić *et al.* (2010) studied the effects of increasing the refiner housing pressure in the double disc line at Braviken. A similar decrease in plate gap as described above was observed for higher housing pressures. Contrary to observations by Kure *et al.* (2000), pulps refined at the higher housing pressure had reduced fibre length compared to pulps refined with lower housing pressure at certain specific energy consumption. The reduction in plate gap was given as a possible explanation for fibre length reduction (Muhić *et al.* 2010).

3.4 Reproducibility in mill scale trials

In *Figure 23*, tensile index and specific energy consumption is presented for pulps produced with only mechanical pre-treatment from the three trials presented in this thesis. There was a good agreement in tensile index development between the two trials performed with compression ratio 3.6:1 (a. and b. in *Figure 23*). The tensile index at certain specific energy consumption was lower in the trial performed with compression ratio 2.7:1 (c. in *Figure 23*) compared to the other trials. The difference in compression ratio may explain a part of the difference in tensile index development. As mentioned earlier, the trial performed with compression ratio 3.6:1 showed a reduction in specific energy consumption by ~120 kWh/bdt to a certain tensile index when compared to pulps produced without mechanical pre-treatment. Similar results were not obtained with compression ratio 2.7:1. The raw material blend was also changed from the normal 50/50 blend to 70% round wood and 30% sawmill chips in the trial denoted as c. in *Figure 23*. This together with other unknown process variations may explain the poor tensile index development in this trial.

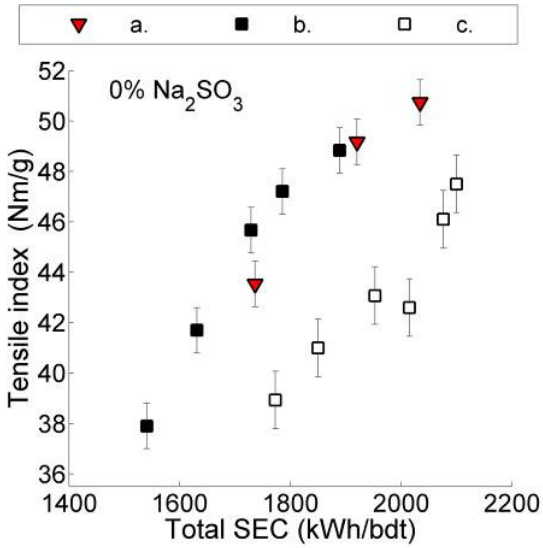

Figure 23. Tensile index vs. total specific energy consumption for the trials performed in the double disc line with only mechanical pre-treatment. (a) and (b) compression ratio 3.6:1, (c) 2.7:1. (a) is found in *Figure 13b*, (b) in *Figure 20a*, (c) in *Figure 16a*. Error bars show 95% confidence intervals.

Although, there was a difference in tensile index development between the two trials where sulphite pre-treatment was evaluated (b. and c. in *Figure 23*), the changes in pulp properties and process conditions caused by sulphite pre-treatment was almost identical (Table 6).

Table 6. *Changes in pulp properties and process conditions caused by addition of sulphite compared to pulps produced without sulphite addition at equal specific energy consumption.*

Study	Low dosage sulphite pre-treatment (c. in *Figure 23*)	Sulphonation and refining intensity (b. in *Figure 23*)
Na_2SO_3 added (%)	1.21%	1.24%
Fibre length (mm)*	*-0.02*	*-0.01*
Fibre width (µm)*	*-0.20*	*0.08*
CSF (mL)	-20.0	-23.8
Shives (number/g)	-127	-178
Density (kg/m^3)	29.0	32.0
Tensile index (Nm/g)	7.60	7.20
Tear index (Nm^2/kg)*	*0.01*	*-0.03*
Elongation (%)	0.18	0.13
Light scattering (m^2/kg)	-1.60	-1.82
Light absorption (m^2/kg)	-0.36	-0.38
Brightness (ISO 457) (%)	2.40	2.25
Hydraulic force (t)	-1.90	-0.51

*Properties not affected by the sulphite addition.

4 Conclusions

Pressurised compressive chip pre-treatment and low dosage sulphite pre-treatment was evaluated for Norway spruce (*Picea abies*) using an Impressafiner and double disc refiner in mill scale.

The mechanical pre-treatment in the Impressafiner reduced the electrical energy consumption needed to reach a tensile index of 47 Nm/g by 120 kWh/bdt (6%). The increase in energy efficiency for production of pulps with mechanical chip pre-treatment may be addressed to a more energy efficient mechanical treatment in the Impressafiner due to higher amplitude and lower frequency of the initial compression cycles. However, it is not possible to rule out effects of reduced extractive content for pulps produced with the Impressafiner pre-treatment.

Sulphite pre-treatment gave a linear dose response in tensile index and light scattering to the addition of sulphite (up to an addition of 1.2% Na_2SO_3). The maximum in tensile index and light scattering at sulphur content of 0.2% (as Na_2SO_3) reported by Axelson and Simonson (1982) was not seen in this study. Low dosage sulphite pre-treatment increased delamination/internal fibrillation of fibres indicating increased fibre flexibility. These fibres produced denser sheets with higher tensile index and slightly reduced light scattering at certain specific energy consumption. The specific energy consumption needed to produce pulp with a certain tensile index was reduced by 210-320 kWh/bdt (12-15%) for chips pre-treated with ~1.2% Na_2SO_3 compared to chips pre-treated without chemicals. However, this led to a reduction in light scattering for sulphite pre-treated pulps when compared at equal tensile index.

The combination of increased refining intensity (double disc refiner) and mechanical and sulphite pre-treatments reduced the energy consumption by 650 kWh/bdt (30%) and produced pulps with higher light scattering (*i.e.* 2.3 m^2/kg) at certain tensile index (*i.e.* 45 Nm/g) when compared with pulps produced with lower refining intensity in single disc refiners. However, it

should be noted that the single disc line was supplied with 100% round wood chips in contrast to the 50% sawmill and 50% round wood chips used in the double disc line. This may have increased the energy consumption at certain tensile index in the single disc line.

5 Future work

The results presented in this thesis show that it is possible to reduce the specific energy consumption in the TMP process with approximately 6% without affecting important pulp properties. This was accomplished by applying a pressurised compressive pre-treatment of chips prior to refining. However, the energy efficiency of the TMP process needs to be increased further to ensure a profitable production of printing grade paper in the future.

The sulphite pre-treatment applied in this thesis showed potential for an increase in energy efficiency of the TMP process to a certain tensile index but did not increase the energy efficiency to a certain light scattering. However, when comparing sulphite pre-treated double disc pulps to pulps produced in an older single disc line, a large increase in energy efficiency was seen without reducing tensile index or light scattering. Unfortunately, it is not enough to reduce the specific energy consumption by 30% based on a comparison with a TMP line built three decades ago. The results from this comparison may however show that a combination of sulphite pre-treatment and changed refining conditions could be a successful approach to increase energy efficiency of the TMP process further from its present state.

Increasing the refining intensity in the double disc refiner by changing the segment design has shown to increase light scattering at certain specific energy consumption (Muhić 2011). It would therefore be very interesting to measure the effect of sulphite pre-treatment together with an increased refining intensity in the double disc refiners.

The pH of the sulphite impregnation liquor was not varied during the trials presented in this thesis. Sulphite pre-treatment performed under acidic conditions has been shown to increase light scattering at certain specific energy consumption (Argyropoulos and Heitner 1991). It would therefore also be interesting to investigate the effects of pH during sulphite pre-treatment in the Impressafiner.

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