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Role of equipment configuration and process chemicals in peroxide-based ATMP refining of spruce

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KEYWORDS: ATMP, TMP, Energy reduction, Fibre characterisation, Fines, Hydrogen peroxide, Magnesium hydroxide, Refiner bleaching.

SUMMARY: Pilot scale refining of White spruce using ATMP (Advanced Thermomechanical Pulp) process was studied. Conventional TMP process, where first stage refiner was equipped with low-intensity segments, was the first reference used in the trial. Another reference was a TMP process with modified equipment configuration (ATMP (aq.)). Modifications consisted of mechanical pre-treatment of chips in Impressafiner and Fiberizer prior to first stage refining at elevated intensity. TMP and ATMP (aq.) references were compared to the ATMP (Mg+P) process which had the same equipment configuration as the ATMP (aq.) and where fibre development in refining was further enhanced using hydrogen peroxide and magnesium hydroxide.

The main goal of the trial was to separate the effects of equipment configuration from the effects of process chemicals in ATMP refining. Impact on the development of individual fibre properties, properties of fines fraction, whole pulp properties and laboratory sheet properties was studied and linked to the energy efficiency in refining

Electrical energy demand, needed to reach the tensile index of 30 Nm/g could be reduced by 0.42 MWh/odt (28 %) compared to conventional TMP process when mechanical pre-treatment and refining at elevated intensity (ATMP (aq.)) were utilized. Refining energy reduction was 0.49 MWh/odt (33 %) when ATMP (Mg+P) concept was used. The content of shives was considerably lower for ATMP and ATMP (aq.) pulps compared to the TMP reference. ATMP (Mg+P) also had higher brightness compared to the references, an increase by 10 ISO % for a 26 kg/odt hydrogen peroxide charge.

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The ATMP process was recently described in pilot and mill scale (Hill et al. 2009; Hill et al. 2010; Johansson et al. 2011; Gorski et al. 2011a; Gorski et al. 2011b). One of the features of ATMP is the combination of mechanical pre-treatment (using Impressafiner and Fiberizer) and high-intensity first

stage refining (achieved by feeding refiner segment pattern and/or elevated refiner speed). Another feature of the ATMP process is the introduction of chemicals after defibration. This is possible due to the mechanical RTF (Retention, Temperature and Fiberization) pre-treatment, during which the defibration of chips into fibres and fibre bundles takes place. The initially used chemical was sodium bisulphite which gave a significant reduction in electrical energy demand and some brightness improvement (Hill et al. 2009; 2010; Johansson et al. 2011). A partial ATMP process with sodium bisulphite (with pre-treatment, consisting of only the Impressafiner and not the Fiberizer) is in operation at Norske Skog Pisa mill in Brazil since 2005 and the results are confirmed in mill scale (Hill et al. 2009; 2010).

A reduction of the electrical energy demand to reach a tensile index of 25 Nm/g was reported to be in the range of 1.1 MWh/odt for Loblolly pine (Johansson et al. 2011) when an ATMP process was compared to a reference TMP process. The corresponding value for White spruce was 0.7 MWh/odt (Gorski et al. 2011a) when a tensile index of 50 Nm/g was targeted. Energy reduction by at least 0.5 MWh/odt for pine and 0.3 MWh/odt for spruce was proposed to be caused by the action of chemicals added in the first stage refiner in the ATMP process and the rest (0.6 and 0.4 MWh/odt respectively) was proposed to be attributed to the process equipment configuration (Johansson et al. 2011; Gorski et al. 2011a). However, ATMP concept was compared only to a TMP reference in these studies. Thus, it was not possible to separate the influence of the altered equipment configuration from the influence of chemicals on energy demand and pulp properties. Only a comparison to earlier studies, where the effect of the equipment configuration was discussed, could be made. In the current study, an ATMP (Mg+P) process was compared not only to a conventional low-intensity TMP but also to a reference TMP with mechanical pre-treatment and high-intensity first stage refining (ATMP (aq.), exactly the same equipment configuration as the ATMP (Mg+P) process itself). This way, a separate study of the effect that the added chemicals had on the produced pulp was possible.

TMP is known for its combination of excellent optical properties and good strength. It is desirable

to maintain this favourable combination of properties also when reducing the energy demand in refining. Addition of chemicals to the refining process was shown to alter the character of TMP making it more “CTMP-like”, i.e. the for printing papers very important light scattering coefficient was not maintained on the same level due to unfavourable defibration of chips caused by softening of the lignin in the middle lamellae (Franzén 1986; Cisneros et al. 1992; Richardson 1998; Salmén et al. 1999; Koljonen et al. 2001), see Fig 1. The character of the ATMP and TMP was studied thoroughly using statistical methods to evaluate, whether the achieved reduction of the energy demand came with a change in the character of the produced pulp (Johansson et al. 2011; Gorski et al. 2011a). No difference in the character of ATMP and TMP was found in earlier studies, i.e. light scattering ability (as well as several other important properties such as density and elongation) was found to be retained, compared at equal tensile index.

It was earlier shown that the reduction of energy demand and change in pulp properties in the ATMP concept can be explained by more energy-efficient development of individual fibre properties (Gorski et al. 2011a). External fibrillation, flexibility and cross-sectional characteristics were earlier shown to be suitable indicators for effects of refining process on fibres (Atack 1981; Koran 1981; Karnis 1994; Kure 1999; Reme 2000; Corson 2001). Significant differences between long fibre characteristics of third-stage ATMP and TMP pulps were documented

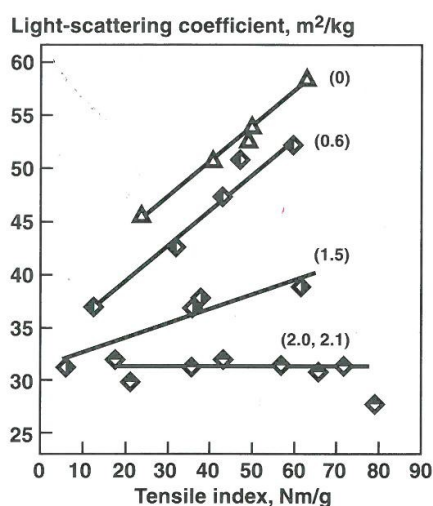


Fig 1. Impregnation of chips with sulphonating chemicals, softening the wood structure, changes the character of the produced pulp. In this figure, this is illustrated by lower light scattering ability of the CTMP pulp compared with TMP at equal tensile index. Numbers in brackets denote different charges of sulphite, used for chip impregnation (Atack et al. 1980).

in earlier studies (Hill et al. 2009; Gorski et al. 2011a). The studied characteristics included the specific surface area index, fibre bendability and axial fibre splitting. Fibre coarseness and mean fibre wall thickness differed insignificantly for the third stage pulps, but had different development throughout the compared ATMP and TMP processes (Gorski et al. 2011a).

The contribution of fines fraction quality and amount to sheet properties was thoroughly studied earlier. The fines fraction plays an important role in sheet consolidation and affects important paper properties such as tensile strength, elongation and light scattering ability of paper (Holl, Brecht 1939; Brecht, Klemm 1953, Giertz 1977; Lindholm 1980a; 1980b; Mohlin 1980; Corson 1980; Heikkurinen, Hattula 1993; Rundlöf 1996). Decrease in the long fibre fraction mass upon refining is normally approximately 30 % which correlates well with the amount of fines fraction in a typical mechanical pulp used for production of printing papers (Höglund, Wilhelmsson 1993, Rundlöf 1996). The fines fraction in mechanical pulps is not uniform; a distinction is normally made between flake-like fines and fibrillar fines also referred to as primary fines and secondary fines (Giertz 1977; Luukko et al. 1997). A considerable amount of ray cells is also usually included in the fines fraction. Primary, or flake-like, fines originate from the outermost parts of the fibre wall within the middle lamellae. These are created upon shattering of the middle lamellae when chips defibrate to fibres. Primary fines contribute to paper properties by improved light scattering, but have only a minor influence on the strength. Secondary, or fibrillar, fines originate from within the S2 fibre wall and contribute with improved strength and decreased light scattering compared to the flaky primary fines (Heikkurinen, Hattula 1993; Karnis 1994; Kure 1999). It was earlier shown that secondary fines have greater bonding ability compared to primary fines (Corson 1989).

Addition of different amounts of fines fraction to a model long fibre fraction was shown to lead to an increase in density, tensile index and light scattering as well as a decrease in air permeability of laboratory sheets (Brecht, Klemm 1953, Lindholm 1980a; Corson 1980; Retulainen et al. 1993; Moss, Retulainen 1995; Rundlöf 1996). The mechanism of densification and improved strength is thought to be formation of new bonds between fibres by fines through covering the area around fibre joints (Retulainen, Nieminen 1992). Fines also fill out the space between the fibres, thereby contributing to increased density and decreased air permeability through the fibre network

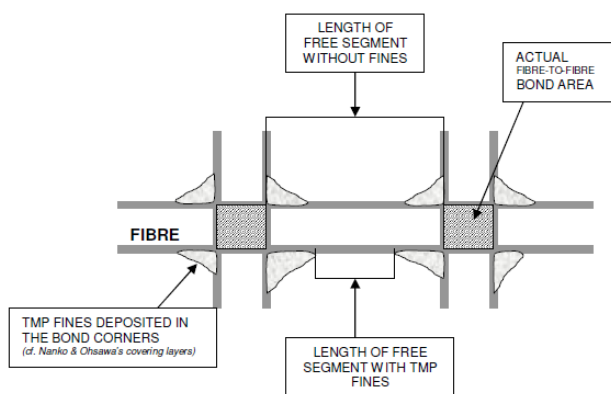


Fig 2. A schematic illustration of how TMP fines contribute to fibre bonding by settling in the corners of bonded areas; and to fibre segment activation by decreasing the effective length of free segments between interfibre bonds (Vainio 2007).

(Brecht, Klemm 1953). In addition, some fines behave as loosely bonded filler material and this, together with their large external surface area, was shown to improve the light scattering of paper (Luukko, Paulapuro 1999). A schematic illustration of how fines contribute to the properties of TMP sheets according to Vainio (2007) can be found in Fig 2. Differences were found in the influence of fines with different origin within the fibre (Heikkurinen, Hattula 1993), fines originating from different mechanical pulping processes (Lindholm 1980b) and fines, produced using different intensities in refining (Kangas et al. 2004; Vehniäinen 2008).

It was earlier shown by statistical analysis that the amount of fines did not differ significantly between different ATMP concepts and a reference TMP, when compared at equal tensile index (Johansson et al. 2010; Gorski et al. 2011a). Hence, it is not the quantity of fines that could cause difference in the properties of pulp, but rather the quality of fines, generated in different process concepts studied. The quality of ATMP fines was, however, not investigated earlier.

Objectives in this paper were:

- To determine the impact of the equipment configuration and the addition of chemicals in the ATMP process on the properties of individual fibres and long fibre fraction sheets.
- To study the character of the pulp produced using all three refining concepts (TMP, ATMP (aq.) and ATMP (Mg+P)).
- To investigate the impact of refining concepts on the quality of fines fraction of all studied processes.
- To determine if the improvement in the quality of fines and fibres translate into the improvement of sheet properties and thus indicate more energy-efficient refining.

Materials and Methods

The pilot plant

Pilot trials were conducted at Andritz pilot plant in Springfield, Ohio, United States. The trial consisted of refining using three different process concepts, see Table 1. White spruce from Wisconsin, USA, was used as raw material. Refining was conducted in two stages; second stage was run at three energy levels. The TMP refining was conducted without mechanical pre-treatment and using retaining (low-intensity) segment pattern. Preheating for all concepts was conducted at casing temperature. A rotational speed of 1800 rpm was used in the first stage TMP refining. Mechanical pre-treatment, consisting of Impressafiner and Fiberizer units, was used in ATMP (aq.) and ATMP (Mg+P) refining. The rotational speed of the first stage refiner was elevated to 2300 rpm and the segment pattern was changed to expelling (higher intensity). In ATMP (Mg+P) refining, process chemicals were also added through the inner ring dilution water into the first stage refiner. DTPA was charged at the Impressafiner outlet where the pressure was relieved for both ATMP (aq.) and ATMP (Mg+P). Chemical recipes are given in Table 1 (charges on oven dry weight of pulp). Pulp was stored in drums between the refining stages. Retention time in the drums varied between 30 and 120 minutes. The same segments, Durametal 36604, were used for all trials, but the direction was switched for ATMP (aq.) and ATMP (Mg+P). Durametal 31604 segments were used in the second stage refiner. Refiner variables for the trials are given in Table 2.

Physical testing, pulp and whole pulp sheets

Laboratory testing of whole pulp and whole pulp sheets was conducted according to TAPPI standards at the Andritz Pilot Plant laboratory, Springfield, Ohio, USA. CSF (Canadian Standard Freeness) was determined according to T227 standard. Fractionation in Bauer McNett device was performed according to T233. Handsheets (approximately 60 g/m²) were prepared according to T205 from pulp, hot disintegrated according to the same standard method. Strength properties of the handsheets were evaluated according to T220 standard. Optical properties were evaluated according to T218 (brightness) and T425 (light scattering).

Table 1. Pulp and chemicals in the pilot trial (pH is measured on first stage blowline pulp, 0.3% DTPA was added at the Impressafiner outlet in all ATMP trials).

Pulp	Chemical recipe	pH
TMP	No chemicals	5.4
ATMP (aq.)	No chemicals	5.5
ATMP (Mg+P)	Mg(OH) ₂ 1.4 % + H ₂ O ₂ 2.6%	7.4

Table 2. Refining variables during the pilot trial.

	Impressafiner*	Fiberizer SD 36-1CP	1 st stage refiner SD 36-1CP	2 nd stage refiner DD 401
TMP trials				
Preheating time (s)	Not used	Not used	120	0
Speed (rpm)	-	-	1800	1200
Casing pressure (bars)	-	-	2.8	Atm.
Casing temperature (°C)**	-	-	142	Atm.
Segment pattern	-	-	Hb***	-
ATMP trials				
Preheating time (s)	15	0	10-15	0
Speed (rpm)	38	1800	2300	1200
Casing pressure (bars)	1.5	1.7	5.2	Atm.
Casing temperature (°C)**	0	138	161	Atm.
Segment pattern	-	Ex***	Ex***	-

* Volumetric compression in the Impressafiner was 5:1 and retention time 15s

** Casing temperature calculated under the assumption of saturated steam conditions

*** Ex (expel) segment pattern gave higher intensity compared to Hb (holdback)

Shive content was determined using Pulmac Fractionator with a 0.10 mm slot screen. Charge of the pulp fractions was determined using methylene blue absorption method (Fardim et al. 2002; Fardim, Holmbom 2003).

The ATMP concept

The mechanical pulping concept called ATMP (Advanced Thermomechanical Pulp) was used in this pilot trial by Hill et al. (2009; 2010) and Johansson et al. (2011), Gorski et al. (2011a). The concept consists of several additional unit operations compared to standard TMP refining: mechanical pre-treatment in a compression screw (Impressafiner) followed by defibration in a Fiberizer unit prior to primary stage refining at higher intensity (together called RTF pre-treatment, Retention-Temperature-Fiberization). During the RTF pre-treatment, initial defibration of chips to coarse fibres and fibre bundles takes place. These are refined in a primary stage refiner, where process chemicals are added. This way, it is possible to add chemicals selectively to the fibre development phase. Unfavourable CTMP-like defibration of chips (fracture planes in the reactive outer parts of the fibre walls) due to impregnation chemicals is avoided since the defibration in ATMP process is conducted prior to the introduction of process chemicals. Primary refining is conducted at higher intensity using feeding refiner plats and/or elevated refining speed.

Fibre and fines quality characterisation

Fibre properties were characterised at the level of defined length populations and individual particles for different process stages and pulping concepts used in this study. Long fibre characteristics such as sedimentation specific surface area index (S^3A index), fibre bendability, and coarseness were

quantified for the long fibre Bauer McNett fractions (P16/R30), while cross-sectional dimensions, shape, fibre collapse index and the degree of axial splitting were quantified for fraction R50. The characterisation was performed according to the same procedures as described earlier (Gorski et al. 2011a).

Based on a series of sedimentation measurements, the sedimentation specific surface area index (S^3A index, σ) was calculated for the initial phase of settling according to *Eq. 1* (Wakelin 2004). Value of 1150 kg/m^3 was used for apparent fibre density (ρ_f) and 998 kg/m^3 for water density (ρ_a). Value of $0.0031 \text{ m}^3/\text{kg}$ was used for fibre specific volume (α), 5.55 for channel factor (k) and 0.001002 kg/ms for fluid viscosity (μ). Velocity (v) and concentration (C) were measured directly. All values are taken from the reference

$$v = \frac{(1 - \alpha \cdot C)^3 \cdot (\rho_a - \rho_f) \cdot g}{k \cdot \sigma^2 \cdot \mu} \cdot \frac{1}{C^2} \quad [1]$$

Fibre flexibility and coarseness were assessed from FiberMaster bendability measurements (Karlsson et al. 1999). Changes in the straightness of the fibres, when subjected to different hydrodynamic conditions, are the principle behind the bendability analysis. Bendability and coarseness measurements were performed for the same fibre population as subjected to the sedimentation test. At least 5 parallel measurements were carried out for each sample, and results were reported for the defined length interval 1.5 to 3.0 mm (the interval denoted "B3" by the FiberMaster).

Fibre cross-sectional characteristics were determined according to the previously described method (Reme et al. 2002). Approximately 1000 fibres per sample evaluated. Binarization, filtration and editing of SEM-BEI images of fibre cross-

sections were performed using a combination of manual and automatic image processing and analysis routines programmed in Image Java. The fibre collapse index was calculated for each individual intact fibre in R50 fraction based on fibre perimeter, total area and lumen area according to principles, described earlier (Jang et al. 1996).

Laboratory sheets were produced using a model fibre fraction, mixed with fines from all three processes. For this purpose, pulps were separated using a Britt Dynamic Drainage Jar (BDDJ) with 100-mesh screen according to T261 (Britt 1973). Second stage pulps, refined with approximately similar energy input (2.26, 2.28 and 2.22 MWh/odt), were used for the experiment. Approximately 100 O.D. g of pulp was used for each fractionation and the cut-off between separated fractions was approximately 25% fine material and 75% fibre fraction. Laboratory sheets were made using the BDDJ fibre fraction, i.e. the fraction, not passing through the 100 mesh screen. The fines fraction was mixed with model long fibres (separated in a BDDJ from the commercial disc filter pulp, CSF ~ 40 ml, from Norske Skog Saugbrugs mill which produces supercalendered paper. The mill uses TMP process to produce pulp from Norway spruce using two-stage mainline refining followed by screening and hydrocyclone fractionation. The rejects are treated in a two-stage reject refining line and mixed with accepts from the fractionation process). Due to practical reasons, it was not possible to produce sheets using only the fines fraction. Mixing was performed on weight basis, 30% of fines were used for each pulp type. Laboratory sheet properties were tested according to standard laboratory testing procedures described in the next paragraph. Even if fractions, obtained using BDDJ, were not the same, as used in the fibre characterisation study (separated in Bauer McNett device), the general behaviour of the fractions would still be similar and the results were helpful in understanding the changes in whole pulp quality and energy demand in refining.

Standard laboratory sheets were prepared according to ISO 5269:2:1998 (using recirculated white water for model sheets with fines and direct drain for long fibre sheets). The sheets were dried restrained. Optical properties of the sheets were tested according to ISO 2470:1999 and ISO 2471:1999. Strength properties were tested according to ISO 1924:2-1994. Results in the graphs are shown with 95% confidence interval, where possible.

Results and discussion

Refiner operation

The accumulated specific energy demand (SEC) in refining during the pilot trial is given in *Table 3*. In an earlier study, it was established that the error in the measurement of specific energy demand is approximately 1.5% using the same pilot equipment as in the current study (Johansson et al. 2011). Corresponding freeness values measured on pulp after each refining stage are given in brackets.

Table 3. Accumulated electrical energy demand (in MWh/odt) and CSF (in ml); corresponding CSF values in brackets

	Imp.	Fib.	1 st	2 nd stage	
TMP	-	-	1.17 (360)	2.14 (110) 2.26 (90)	2.39 (80) 2.68 (50)
ATMP (aq.)	0.04	0.24 (770)	0.83 (500)	1.73 (170) 1.92 (120)	2.09 (80) 2.28 (50)
ATMP (Mg+P)	0.04	0.24 (770)	0.89 (460)	1.86 (110) 2.03 (80)	2.22 (50) 2.39 (50)

Pulp character

The TMP process became dominant for the production of printing paper due to the fact that TMP has good strength properties combined with excellent optical properties (high light scattering ability). It is highly undesirable to change the favourable relationship between those properties – any modification of the TMP process is successful only when none of the important properties are sacrificed, i.e. the character of the TMP must be preserved when trying to reduce the energy demand in refining.

To establish the character of produced pulp ANOVA (analysis of variances) was used to determine how much of the change in each one of such critical pulp and paper properties as for example light scattering coefficient, density and elongation correlated with a change in the tensile index (the dependent variable). This method of determining the pulp character was described in more detail earlier (Johansson et al. 2011; Gorski et al. 2011a). Results show that there were no significant differences in the character of the three studied pulps. This is in agreement with previous findings (Johansson et al. 2011, Gorski et al. 2011a).

As can be seen in *Fig 3*, the content of shives is dramatically lower in ATMP (Mg+P) and ATMP (aq.) compared to the reference TMP. It was shown earlier that the content of shives was significantly lower in ATMP compared to a TMP reference (Johansson et al. 2011; Gorski et al. 2011a). Results from this study show that the decrease in the shives content was caused by compressive pre-treatment combined with high intensity refining and not the addition of process chemicals since the content of

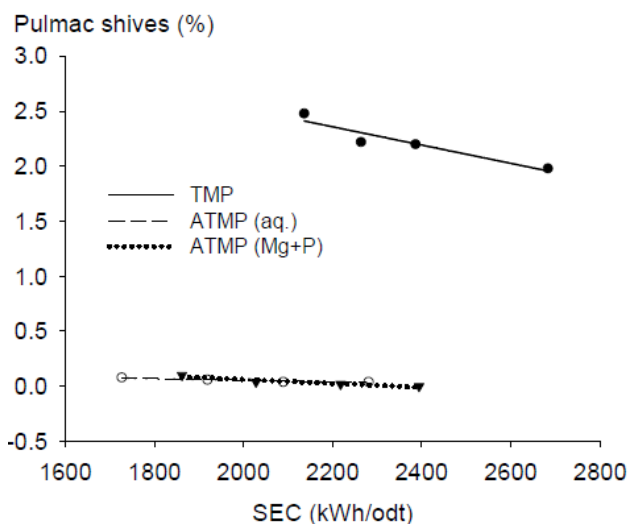


Fig 3. Shives content of the pulps (Pulmac 0.10 mm screen, weight %).

shives for ATMP (aq.) and ATMP (Mg+P) process was approximately similar and very low. High intensity refining was previously shown to decrease the content of shives. When the rotational speed of the refiner was increased from 1800 rpm to 2600 rpm, the content of shives in Norway spruce pulp decreased from 3.1 to 0.2 measured using the same Pulmac screen as in this study (Kure et al. 1999). Similar results were reported for a mix of spruce and pine raw material (Sabourin 2000). Introduction of Impressafiner pre-treatment prior to the high-intensity refining did not decrease the content of shives further (Kure et al. 1999). This is consistent with later findings where the introduction of Impressafiner pre-treatment prior to TMP refining did not contribute to decreasing the shives content significantly (Sabourin et al. 2002). The influence of a combined pre-treatment in both Impressafiner and Fiberizer on the shives content has not been reported earlier. Since very low shives contents were measured in the ATMP (aq.) and ATMP (Mg+P) pulps in this study, it is possible that Fiberizer has a positive effect on shive content reduction. This would be logical since increasing the uniformity of the size distribution of the material in the feed should increase the stability of the refiner operation and thus possibly also lead to decreased content of shives in the pulp.

The development of tear index as a function of tensile index can be seen in Fig 4. The results show that there is no significant difference between TMP, ATMP (aq.) and ATMP (Mg+P). This is consistent with previous results (Johansson et al. 2011; Gorski et al. 2011a) and confirms that all three pulp types have same basic character.

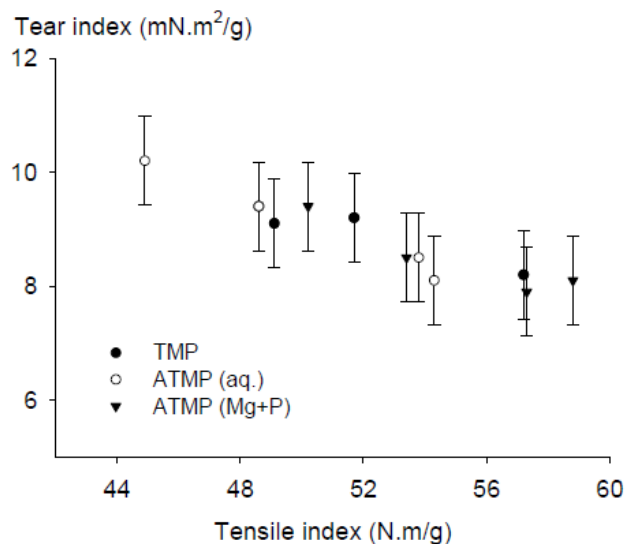


Fig 4. Tear index as a function of tensile index of handsheets.

Fibre properties

It was earlier concluded that a difference between pulps, produced according to ATMP concept (involving both mechanical pre-treatment, high-intensity refining and addition of chemicals in first stage refining) and low-intensity TMP reference with respect to several important fibre characteristics was already established after first stage refining (Gorski et al. 2011a). The results also pointed at possibly different fibre development mechanisms upon further refining.

A clarification of the mechanisms, which govern separate and combined development of fibre and fines, is of particular interest for the further development of the ATMP process. To clarify these mechanisms, it is important to separate the contributions from the mechanical pre-treatment and refining at higher intensity (ATMP (aq.)) on one hand and the addition of process chemicals (ATMP (Mg+P)) on the other hand. In this study, emphasis was again put on quantification of both first stage level and further development of fibre properties, considered important for the quality for the final product, Figs 5 to 12.

As the accumulated specific energy demand varied for the different process stages and concepts, a quantitative process comparison was based on linear inter- and extrapolations. For most of the characteristics the development throughout the refining process plotted versus the specific energy demand was well approximated by linear functions. The TMP had received 0.34 and 0.28 MWh/t more specific energy in the first refining stage compared to ATMP (aq.) and ATMP (Mg+P), respectively.

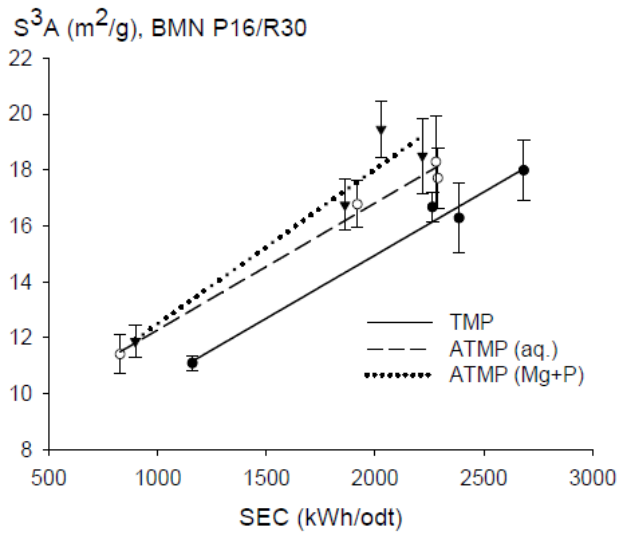


Fig 5. Development of long fibre fraction specific surface area.

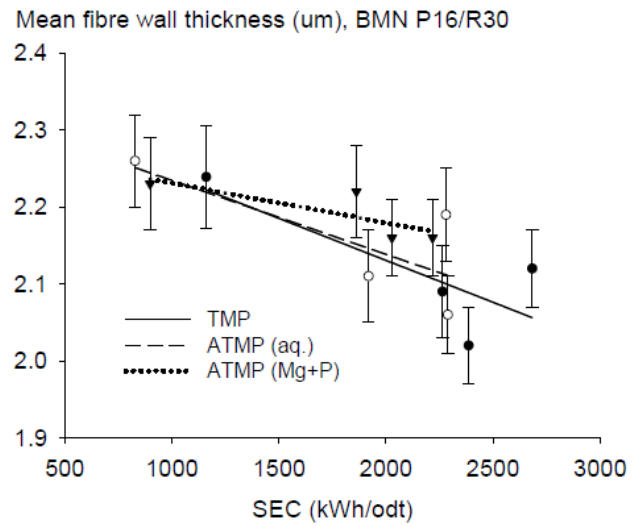


Fig 8. Development of mean fibre wall thickness.

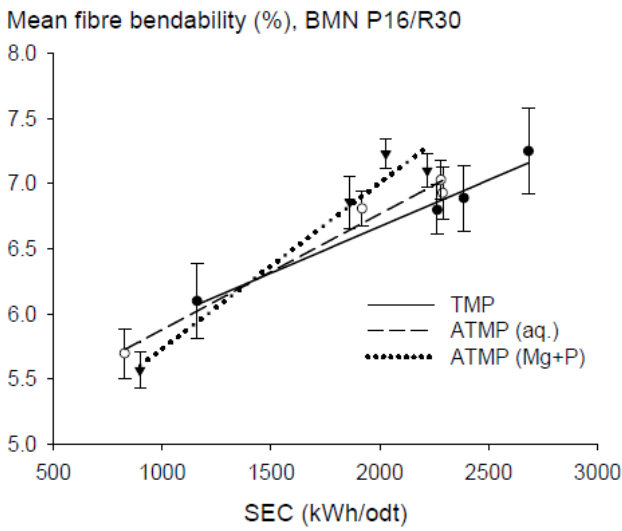


Fig 6. Development of long fibre fraction bendability.

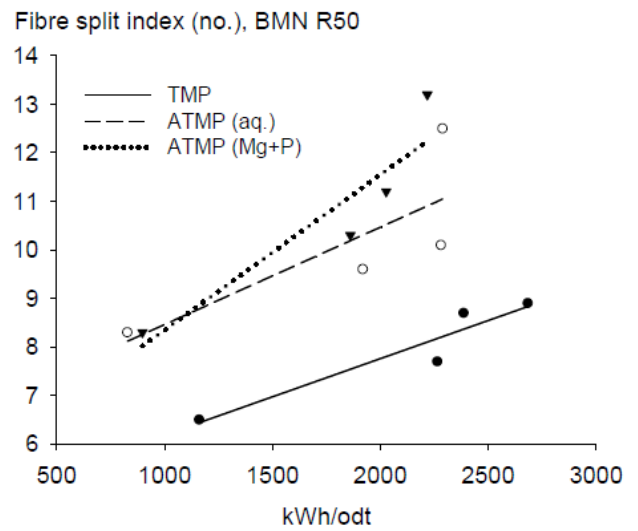


Fig 9. Development of fibre split index.

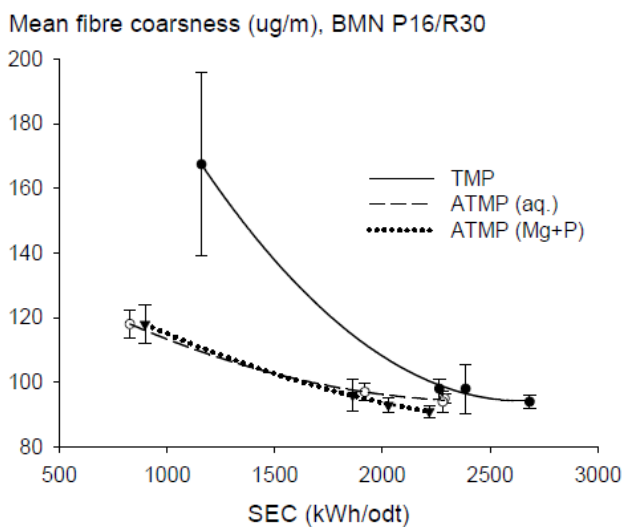


Fig 7. Development of long fibre fraction mean coarseness.

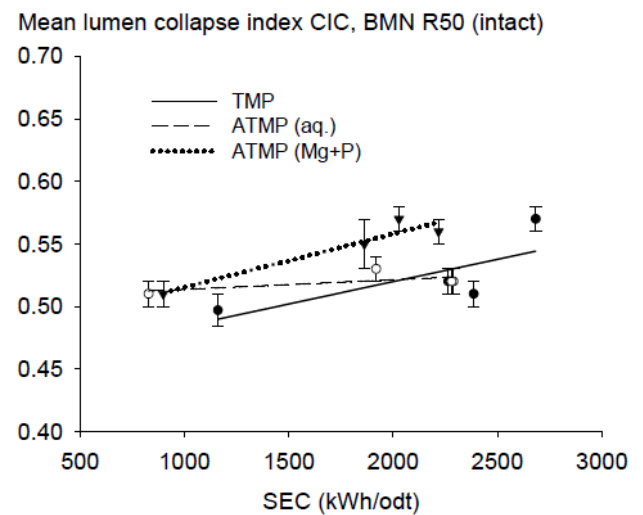


Fig 10. Development of fibre collapse index.

Table 4. Mean values for the fibre characteristics, interpolated at two energy input levels (0.85 MWh/odt and 2.25 MWh/odt).

Fibre characteristic	Unit	Value at 0.85 MWh/odt			Value at 2.25 MWh/odt			Relative change (0.85 to 2.25, %)		
		TMP	ATMP (aq.)	ATMP (Mg+P)	TMP	ATMP (aq.)	ATMP (Mg+P)	TMP	ATMP (aq.)	ATMP (Mg+P)
S ³ A index	m ² /g	9.8	11.5	11.6	16.1	18.3	19.3	+39	+37	+40
Bendability B3	%	5.8	5.7	5.6	6.8	7.0	7.4	+14	+18	+25
Coarseness	µm/m	180	118	118	108	93	89	-67	-27	-33
Fibre wall thickness	µm	2.26	2.24	2.32	2.12	2.10	2.17	-7	-7	-7
Fibre wall area	µm ²	155	155	155	143	145	147	-9	-7	-5
Fibre split index	no.	6.1	8.2	8.0	8.2	11.0	13.0	+26	+26	+38
Lumen area	µm ²	217	207	212	193	209	180	-12	+1	-18
Collapse index	%	48.2	51.6	50.3	53.8	52.5	55.9	+10	+2	+10

The total specific energy input after this stage did not differ substantially between the pulps though, with 1.17 MWh/t for TMP, 1.11 MWh/t for ATMP (aq.) and 1.17 MWh/t for the ATMP (Mg+P), *Table 3*. Mean fibre wall area and fibre bendability were similar for the TMP and the ATMP (Mg+P)/ATMP (aq.) fibres, when inter-/extrapolated to a common SEC level of 0.85 MWh/t, *Table 4*. Differences in mean fibre wall thickness were not significant. The specific surface area index was significantly higher for ATMP (Mg+P)/ATMP (aq.) compared to TMP fibres, with differences of up to 1.8 m²/g (19%). ATMP (aq.) and ATMP (Mg+P) contained about 20% more split fibres at this stage. The mean fibre coarseness was 35% higher for first stage TMP than for the respective ATMP (Mg+P) and ATMP (aq.) fibres, and the mean fibre collapse index was between 4% and 7% lower for the TMP. These results agree well with the principal findings from a previous ATMP study on White spruce, except for the fibre wall thickness (Gorski et al. 2011a).

ATMP (Mg+P) and ATMP (aq.) had similar fibre properties at 0.85 MWh/t SEC with respect to S³A index, bendability, fibre coarseness, mean fibre wall area as well as mean fibre collapse index. The ATMP (Mg+P) had somewhat higher mean fibre wall thickness than ATMP (aq.), but the difference was not statistically significant. The fibre split index and the collapse index were somewhat higher (approximately 3%) for ATMP (aq.) than ATMP (Mg+P) fibres.

Further progression of the fibre development with increasing SEC after the first stage refining was quantified as the difference in results obtained by interpolation to two levels of specific energy demand, *Table 4*.

Both specific surface index and bendability were significantly increased. No major differences were found with respect to S³A index development, even though the ATMP (Mg+P) demonstrated the greatest progression of the three concepts. Bendability was developed most effectively for

ATMP (Mg+P), followed by ATMP (aq.) and TMP, *Fig. 5* and *6*. Mean fibre wall thickness reduction and fibre spitting progressed at the same rate for all three process concepts, while the reduction of fibre coarseness and mean fibre wall area was most effective for TMP. It should be taken into consideration that the uncertainty of the first stage TMP coarseness measurement was high, *Figs 7* and *8*. The mean lumen area was decreased, and the mean fibre collapse index as calculated from cross-sectional analysis was significantly increased for TMP and ATMP (Mg+P), while no development in fibre cross-sectional shape characteristics was observed for the ATMP (aq.), *Table 4* and *Fig 10*.

It was earlier pointed out that the S³A index is influenced by a number of fibre properties, and can be interpreted as a measure of external fibrillation only when fibre wall density, fibre width, and fibre length are comparable (Gorski et al. 2011a). Wakelin (2004) stated that "...for an equal apparent density and an equal degree of fibrillation...there will be a lower sedimentation index for larger diameter fibres."

The possible dissolution of fibre wall components by process chemicals applied in the ATMP (Mg+P) concept and resulting potential changes in fibre wall density were not verified by measurements in this study. The fibre diameter was assessed from perimeter and width measurements. Fibre dimensions were obviously affected by the early process treatment, and clear differences in the fibre length and width distributions were observed, even for the defined length fraction P16/R30. ATMP (aq.) fibres were longer than ATMP (Mg+P) and TMP fibres (not shown), while the TMP had clearly narrower fibres after first stage refining, *Fig 11*. These fibre width measurements agree with SEM-based analyses of the fibre perimeter (*Fig 12*) and provide indications for structural changes within the fibre walls, likely in terms of internal fibrillation and fibre wall delamination, as introduced by the ATMP (aq.) process. The ATMP (aq.) pulp had similar fibre coarseness values as ATMP (Mg+P) at

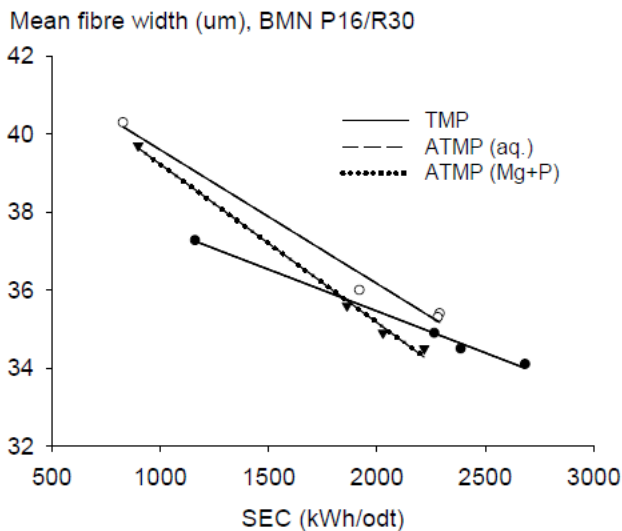


Fig 11. Development of mean fibre width based on FiberMaster measurements of BMN fraction P16/R30.

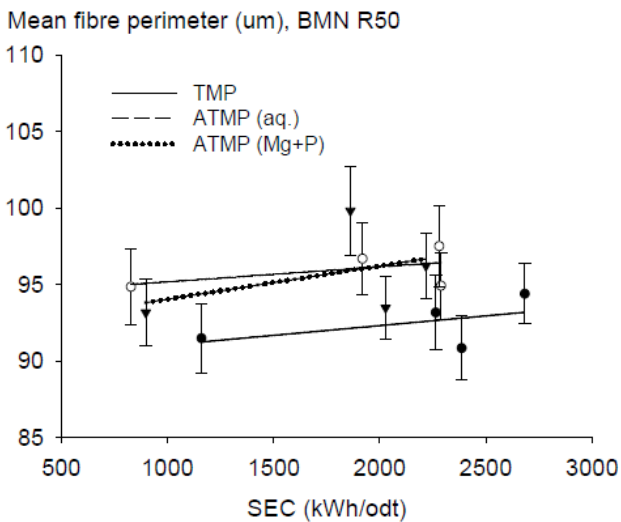


Fig 12. Development of mean fibre perimeter based on SEM and image analysis of BMN fraction R50.

all process stages, Fig 7. Differences in mean fibre wall thickness were not significant between these two fibre types, Fig 8. Bearing in mind the high fibre width values of ATMP (Mg+P) it can thus be assumed that it's favorable specific surface area index values are rather a consequence of good external fibrillation than lower fibre wall density. Based on these results it can be concluded that it is the mechanical pre-treatment and the high intensity refining (ATMP (aq.) process) that provides the foundation for the advantageous fibre quality development in the novel ATMP refining concept. The mechanical action in the Impressafiner and Fiberizer combined with further first stage refining at elevated intensity dramatically improved the disintegration of chips to individual fibres and additionally initiated structural changes to the fibre walls, which are quantifiable already after the first refining stage.

These newly created surfaces are accessible for the chemicals, introduced in the first stage refiner. The primary effect of the hydrogen peroxide and magnesium hydroxide, applied in this study, was a further enhancement of the fibre flexibility (assessed as FiberMaster bendability) when refining progressed at increasing levels of specific energy demand. In addition, a reduction of the fibre wall thickness, typical for TMP refining and commonly related to external peeling, fibrillation and fines generation, was less pronounced in the ATMP (Mg+P) process compared to TMP and ATMP (aq.) refining and statistically not significant. These findings are in agreement with previous results (Gorski et al., 2011a). In connection with the specific surface area index measurements it was suggested that external fibrillation is likely to have had developed more efficiently in ATMP (Mg+P) refining. The fibre wall thickness development of the ATMP (Mg+P) can thus be interpreted as a combination of external peeling accompanied by simultaneous swelling of the chemically modified fibres. The amount of axially split fibres was somewhat higher in ATMP (Mg+P) compared to ATMP (aq.) and TMP respectively, Fig 9. This finding correlates with previous results (Gorski et al. 2011a) and suggests that process chemicals influence fibre splitting. Exact mechanism of that is not yet known.

Quality of the BDDJ fibre fraction (R100)

Fibre length distribution of fractionated long fibre and fines measured with PQM1000 is shown in Fig 13. The length class distributions of fines and long fibre fractions were similar for all three studied processes.

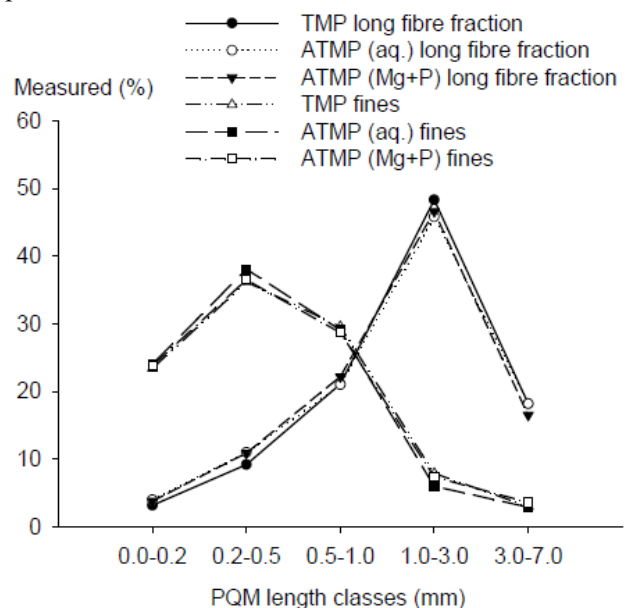


Fig 13. Distribution of long fibre and fines length classes (PQM1000) for TMP, ATMP (aq.) and ATMP (Mg+P) fractionated into two fractions using a BDDJ (100 mesh screen).

Laboratory paper sheets were made using the BDDJ long fibre fraction. Tensile index, light scattering and elongation of BDDJ long fibre fraction sheets from TMP, ATMP (aq.) and ATMP (Mg+P) pulps, manufactured at similar energy inputs, are shown as a function of apparent sheet density in *Figs 14, 15 and 16*. Long fibre fraction sheets, made of the ATMP (aq.) pulp, had 40 kg/m³ (12%) higher apparent sheet density compared to the long fibre fraction of TMP. Sheets made of ATMP (Mg+P) long fibre fraction had 15 kg/m³ (4%) higher apparent sheet density compared to ATMP (aq.).

Long fibre fraction sheets made of the ATMP (aq.) pulp had 5 Nm/g (26%) higher tensile index compared to the long fibre fraction sheets made of TMP. Sheets made of ATMP (Mg+P) long fibre fraction had 2.2 Nm/g (10%) higher tensile index compared to ATMP (aq.) sheets. These results agree with the fibre characterisation results. Specific surface area of the fibres increased by 12 % for ATMP (aq.) and 17 % for ATMP (Mg+P) compared to the TMP reference, *Fig 5*. The corresponding figure for flexibility was 3 % and 8 %, *Fig 6*. Measured differences in fibre specific surface area index and bendability between TMP and ATMP pulps were thus translated into sheet properties, and reflected the enhanced efficiency in fibre development due to the use of mechanical pre-treatment, higher intensity refining and addition of chemicals. The effect of the equipment configuration (mechanical pre-treatment and higher refining intensity) seems to be more pronounced compared to the effect of process chemicals.

The increase in light scattering was 2 m³/kg for ATMP (aq.) compared to TMP and another 1 m³/kg for ATMP (Mg+P) compared to ATMP (aq.). This was probably caused by increased external and internal fibrillation of the fibres as a result of refining (which also led to increased sheet density due to more flexible fibres). Elongation of the sheets increased with density for TMP, ATMP (aq.) and ATMP (Mg+P) as well. This suggests that, even when the sheet structure became denser, a more flexible network was created through increased flexibility of fibres and higher bond strength, which is supported by individual fibre characterisation.

These results confirm significant differences in fibre quality as developed in the investigated TMP, ATMP (aq.) and ATMP (Mg+P) processes. The mechanical pre-treatment and refining at elevated intensity in the ATMP (aq.) concept contributed to significantly improved fibre properties, which were additionally enhanced by the addition of process chemicals in the ATMP (Mg+P) concept.

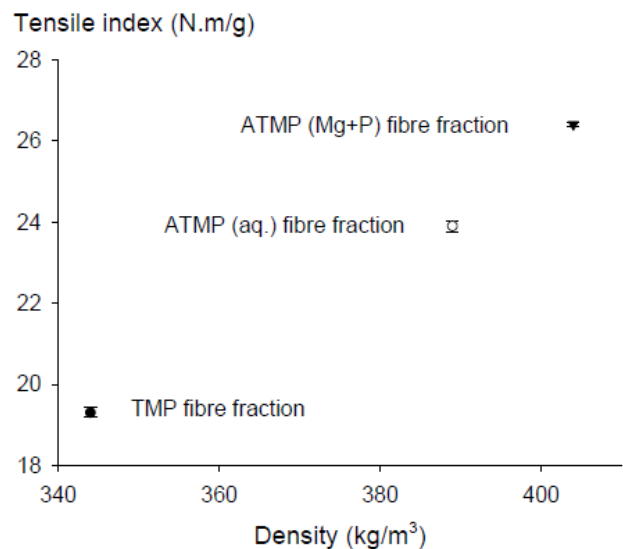


Fig 14. Development of BDDJ long fibre fraction (R100) tensile index as a function of apparent sheet density.

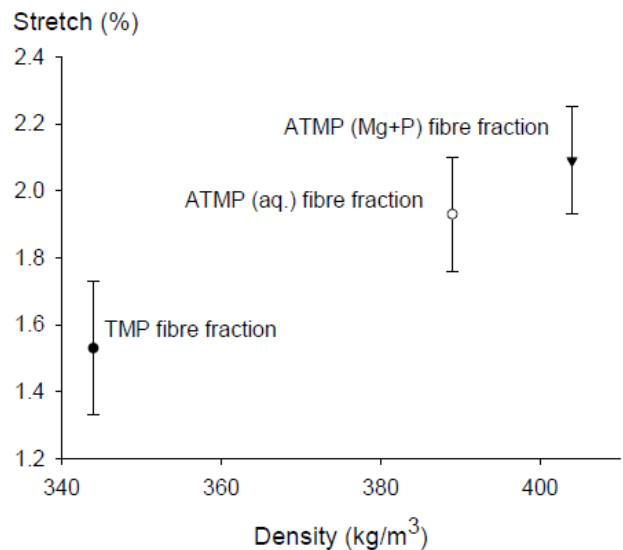


Fig 15. Development of BDDJ long fibre fraction (R100) elongation as a function of apparent sheet density.

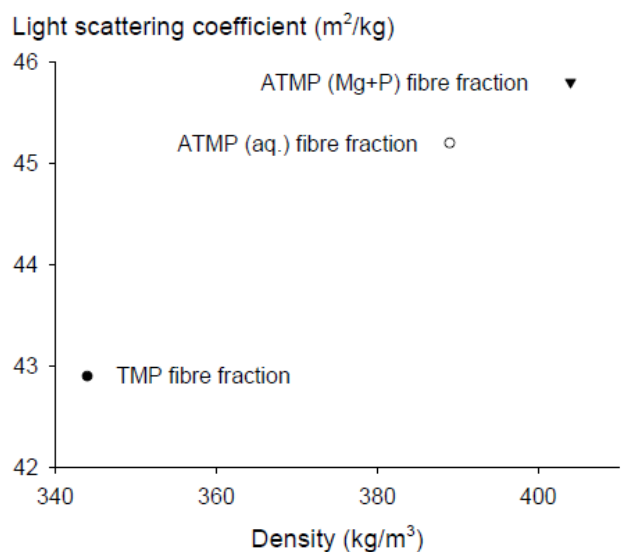


Fig 16. Development of BDDJ long fibre fraction (R100) light scattering as a function of apparent sheet density.

Quality of the BDDJ fines fraction (P100)

It is well-documented in the literature that the addition of fines to long fibre fraction leads to increased fibre bonding, light scattering and sheet strength. In this study, the effect of the amount of added fines was not studied. Instead, the relative effects of fines, originating from the three different process concepts, were investigated and compared to each other.

ATMP (aq.) and ATMP (Mg+P) fines increased the tensile index of model fibre sheets more compared to TMP fines, *Fig 17*. Tensile index increased with 2.5 Nm/g (6 %) when ATMP (aq.) fines were added instead of TMP fines and another 1.5 Nm/g (10 % in total) when ATMP (Mg+P) fines were added. At the same time, the density of sheets, made with addition of ATMP (aq.) fines, was 14 kg/m³ higher compared with sheets, made with addition of TMP fines. When ATMP (Mg+P) fines were added, no increase in density could be measured compared to sheets with ATMP (aq.) fines. Differences in elongation between sheets made with addition of the three different fines types were not significant, *Fig 19*. Light scattering increased with addition of fines from all three studied pulps. However, the increase in light scattering was larger for TMP fines compared to ATMP (aq.) fines. The addition of ATMP (Mg+P) gave the smallest increase in light scattering. This is in agreement with previously discussed literature, where flaky primary fines were found to improve light scattering, while fibrillar secondary fines have negative effect on light scattering. Since pulps, used in this study, were refined with more than 2 MWh/odt, the fines fraction can be presumed to have high fibrillar content and thus contributed less to the increase in light scattering.

It was earlier shown that an equal proportion of TMP and SGW (Stone Groundwood) fines contribute to tensile strength of laboratory sheets in slightly different ways due to the different peeling mechanisms, involved in forming of fines in TMP and SGW processes (Lindholm 1980b). In this study, all three fines types were probably of similar origin, since the starting material was the same and so were the dimensions of refined pulp fibres. Fines in this study came from pulps, refined using approximately equal energy input. It is known that the energy efficiency in refining of these three pulps differed. Compared at equal tensile index, no differences in the fines content and average fibre length were found – later in this paper, it is shown that different amounts of energy are needed to produce TMP, ATMP (aq.) and ATMP (Mg+P) with equal strength properties. Tensile index was used to compare the energy demand in refining of pulps in

this study, and it would be interesting to compare the properties of fines, originating from pulps with equal tensile index. Unfortunately, these samples were not available.

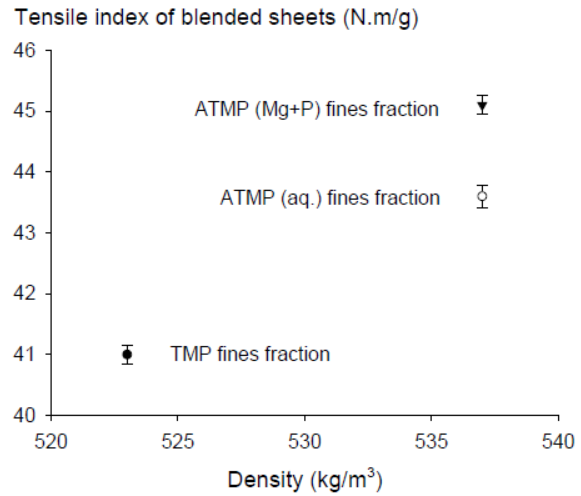


Fig 17. Tensile index development of model sheets with BDDJ fines fraction (P100) as a function of apparent sheet density.

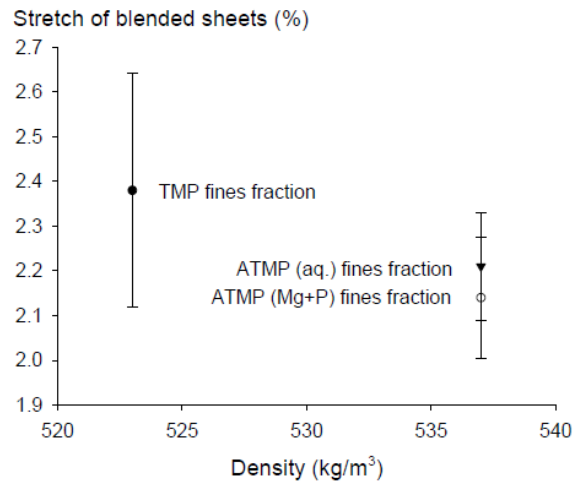


Fig 18. Elongation development of model sheets with BDDJ fines fraction (P100) as a function of apparent sheet density.

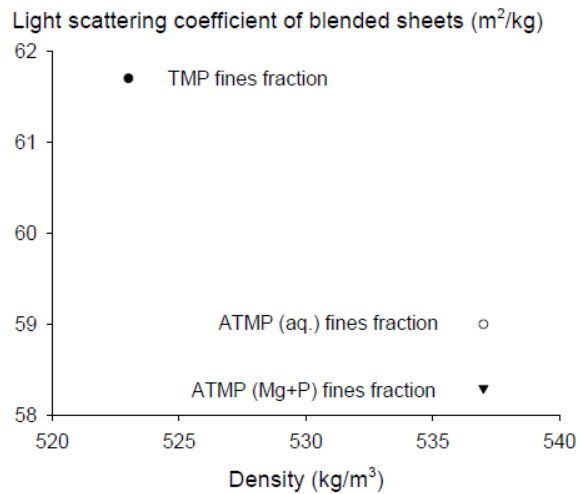


Fig 19. Light scattering index development of model sheets with BDDJ fines fraction (P100) as a function of apparent sheet density.

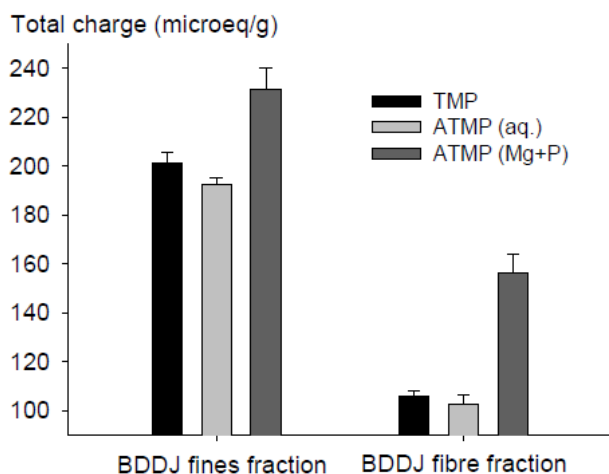


Fig 20. Charge of BDDJ fines (P100) and long fibre (R100) fractions

Carboxylation of the fines fraction, which caused higher charge (Fig 20), could have contributed to better bonding ability of the fines fraction. The charge of ATMP (Mg+P) fines was found to be 30 $\mu\text{eq/g}$ higher compared to TMP and ATMP (aq.) fines.

It was earlier found, that addition of TMP fines to a model long fibre sheets increased the tensile index and density of the sheets, while addition of polyelectrolyte (for example C-PAM) improved the tensile index, but did not affect the density of the sheets or the elastic modulus (Koljonen et al. 2003; Lindström et al. 2005; Vainio 2007). It is interesting that the effect of ATMP (Mg+P) fines on the model fibre sheets reminds somewhat of the effect polyelectrolytes were reported to have on the sheet properties. Increasing density was earlier found to increase the bond strength, improvement in bonding caused by higher bonded area and number of bonds (Retulainen et al. 1993; Retulainen et al. 1998). This seems to be true for TMP and ATMP (aq.) fines, produced in refining, conducted without addition of chemicals. Addition of the highly charged ATMP (Mg+P) fines, on the other hand, seems to lead to additional improvement of sheet properties without influencing the density of the sheet. A possible explanation could be that charged ATMP (Mg+P) fines form a layer inside the bonded area between the fibres, improving the bond strength. This would also explain the fact that density of the sheets does not seem to be affected by ATMP (Mg+P) fines; while fibres are drawn closer to each other, forming new bonds, the presence of ATMP fines in the bonded area between the fibres would counteract the increase in density. It is also possible that ATMP (Mg+P) fines form complexes with other fine fragments around fibre bonds, making them stronger. The mechanism of this is not

yet understood and more detailed studies of this phenomenon should be undertaken in the future.

Energy efficiency and handsheet strength

In Fig 21 and Table 5, the development of the tensile index of laboratory sheets with increased refining is shown for all three processes studied in this paper. The reduction of energy demand when compressive pre-treatment (Impressafiner + Fiberizer) was used together with high intensity refining (feeding segments and elevated rotational speed) was approximately 0.42 MWh/odt or 28% compared to the low-intensity TMP reference at a tensile index of 30 Nm/g.

A reduction of the energy demand by up to 30% when elevated refining intensity was used in combination with mechanical pre-treatment in Impressafiner was reported (Kure et al. 1999; Sabourin 2000; Johansson, Dahlqvist 2001; Sabourin et al. 2003). Reduction of the energy demand by another 15% was reported when Fiberizer unit was introduced in the pre-treatment in addition to the Impressafiner (Sabourin et al. 2003). Thus, compared to these previously documented results, the figure regarding the energy reduction obtained in this study is rather small. It was earlier proposed that high energy input in the first stage refining could contribute to a more energy-efficient first stage refining action (Sabourin et al. 1994). In this study, the TMP was refined with 1.17 MWh/odt while ATMP (aq.) and ATMP (Mg+P) were refined using 0.83 and 0.89 MWh/odt respectively. Energy-efficiency from high energy input in the first stage could be connected to the special conditions that exist in the first stage refining, such as the high coefficient of friction (Isaksson et al. 1997, Illikainen et al. 2007).

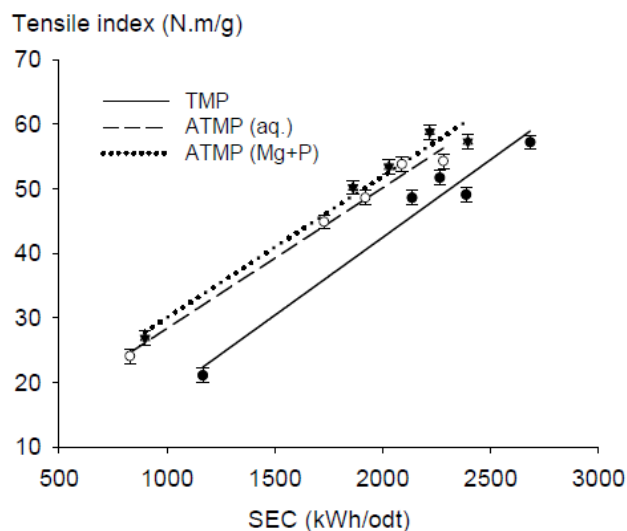


Fig 21. Tensile index as a function of specific energy demand.

Table 5. Electrical energy demand in refining at equal tensile index. Maximum error is based on previous calculations (Johansson et al. 2011).

Pulp	Energy demand	Reduction
At 30 N.m/g	MWh/odt	MWh/odt (%)
TMP	1.49±0.02	Reference
ATMP (aq.)	1.07±0.02	- 0.42 (28)
ATMP (Mg+P)	1.00±0.02	- 0.49 (33)
At 50 N.m/g	MWh/odt	MWh/odt (%)
TMP	2.32±0.03	Reference
ATMP (aq.)	2.00±0.03	-0.32 (14)
ATMP (Mg+P)	1.91±0.03	-0.31 (18)

The contribution of chemicals to the energy reduction in pine ATMP process was proposed to be in the range of 0.5 MWh/odt (at tensile index 25 Nm/g) for pine and 0.3 MWh/odt (at tensile index 50 Nm/g) for spruce (Johansson et al. 2011; Gorski et al. 2011a). In this study, the difference in energy demand between the ATMP (aq.) and ATMP (Mg+P) processes was less than 0.1 MWh/odt. However, the results of individual fibre characterisation showed that ATMP (Mg+P) fibres had higher specific surface area, bendability, split fibre index and collapse index compared to the ATMP (aq.) pulp at a given SEC level. In other words, less specific energy was needed to achieve a given quality level of these fibre properties with the ATMP (Mg+P) concept compared to ATMP (aq.) refining. These results suggest that process chemicals do have a positive effect on the development of fibre properties in ATMP (Mg+P) refining. In addition, both BDDJ fibre fraction sheets made of ATMP (Mg+P) and mixture sheets manufactured from model long fibres and ATMP (Mg+P) fines had significantly higher tensile index compared to the ATMP (aq.) and TMP sheets respectively. These findings strengthen the hypothesis that process chemicals influence the fibre development and energy demand in ATMP (Mg+P) refining.

Chemical mechanisms for the influence on the strength properties were proposed earlier (Gorski et al. 2011a). Hydrogen peroxide and magnesium hydroxide introduce new carboxylic groups into fibres. Since fibre surfaces are readily available in fiberized material with its open structure, the reactions probably happen very fast and influence the refining efficiency already in the first stage refiner. Some of the increased energy efficiency could also possibly be explained by changes in the coefficient of friction of swollen carboxylated fibres. Mechanisms of this should be investigated in more detail.

The reduction of the specific energy demand to reach a given tensile index in ATMP (Mg+P) refining, compared to the TMP reference, was

considerably higher in a previous study (0.65 MWh/odt at tensile index 30 Nm/g), conducted for the same wood species (Gorski et al. 2011a). There is a possibility, that unknown factors influenced the performance of chemicals in this study and caused a reduction of their performance. Indeed, this has been confirmed in a separate investigation of bleaching efficiency in the peroxide-based ATMP concept, conducted on pulps from the same pilot trials. A much lower content of residual hydrogen peroxide (1 % compared to 1.9 %) was found in blowline ATMP, produced in this study compared to previous trials (Gorski et al. 2011b). Based on these previous results, there is a possibility that the effect of chemicals on fibre development and energy efficiency in an ATMP process can be significantly higher than was shown in the present study (Hill et al. 2009; 2010; Johansson et al. 2011; Gorski et al. 2011a).

Optical properties of handsheets

Fig 22 shows that brightness of produced ATMP increased by approximately 10 ISO% compared to the TMP reference. This increase is lower than earlier observed with similar hydrogen peroxide charge (Gorski et al. 2011a). A separate investigation of the bleaching efficiency in the ATMP process was performed (Gorski et al. 2011b). It was discovered that pulp from the present study also had poor bleachability and low content of residual hydrogen peroxide when bleached in conventional laboratory bleaching process. The poor bleachability seemed to be related to some unknown material properties of the batch of wood used in the current study.

Light scattering was discussed earlier during the assessment of the pulp character. It was concluded from the statistical analyses that there are no significant differences in light scattering coefficient between TMP, ATMP (aq.) and ATMP (Mg+P) compared at equal tensile index, Fig 23.

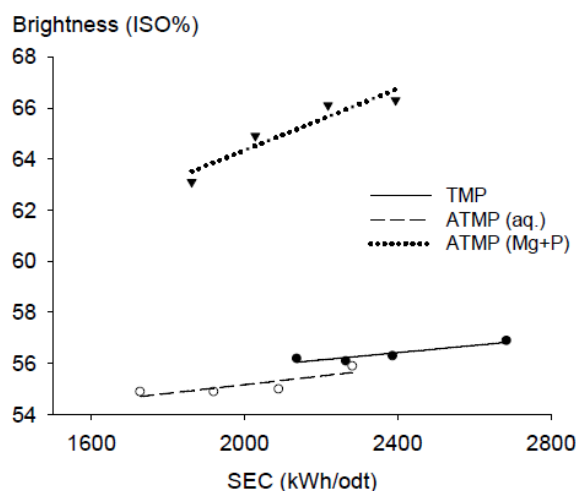


Fig 22. ISO brightness of pulps.

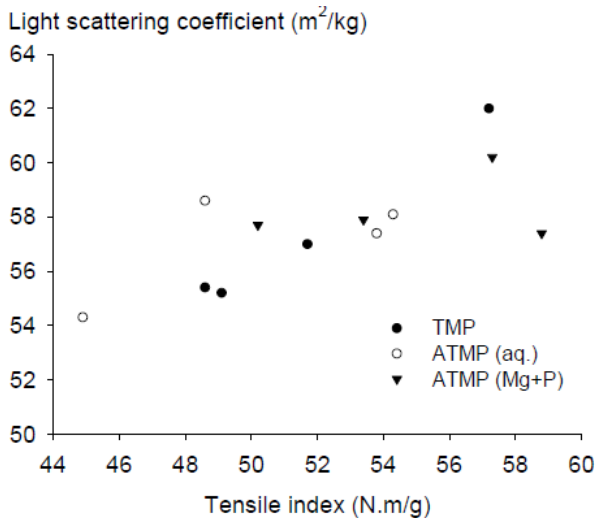


Fig 23. Light scattering coefficient of handsheets ($R^2=0.84$ and $P=8.1 \cdot 10^{-8}$).

Conclusions

- Both the process configuration (mechanical pre-treatment combined with refining at high intensity) and the addition of chemicals were shown to contribute to the spruce pulp quality and refining energy efficiency in the peroxide-based ATMP process.
- Mechanical pre-treatment and high-intensity refining had a dominant influence compared to the selected chemical combination and dosage in this study. However, based on previous publications, it is possible that the unexpectedly poor performance of the ATMP (Mg+P) process chemicals was a result of unknown disturbances.
- TMP, ATMP (aq.) and ATMP (Mg+P) had similar overall character assessed by statistical analysis of such variables as sheet light scattering, elongation and density.
- The highly effective reduction of shives in the spruce ATMP process compared to TMP was mainly due to the mechanical pre-treatment combined with high-intensity first stage refining and not a result of the addition of process chemicals.
- Both the equipment configuration and the addition of chemicals gave significant contributions to the development of individual fibre properties such as external and internal fibrillation and split fibre index. The equipment configuration seemed to have had the dominant effect. This was further verified by measurement of BDDJ fibre sheet properties such as tensile index, light scattering, density and elongation.
- Results of fibre development in the ATMP process documented in this study agree well with previously reported findings.

- ATMP (aq.) and ATMP (Mg+P) fines fractions contributed stronger to the improvement of handsheet tensile strength compared to TMP fines when added in similar proportions to model long fibres. Model fibre handsheet density increased upon addition of ATMP (aq.) fines compared to TMP fines. However, the density was the same for handsheets with ATMP (aq.) and ATMP (Mg+P) fines. Light scattering coefficient and elongation increased more upon addition of TMP fines compared to ATMP fines. ATMP (Mg+P) fines seemed to have slightly different character and contributed with stronger specific bonding at equal sheet density, possibly as a result of their increased charge due to the chemical treatment.

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