Peroxide-based ATMP refining of spruce: energy efficiency, fibre properties and pulp quality

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

SUMMARY: Pilot scale refining of White spruce using a modified TMP refining process (ATMP – Advanced Thermomechanical Pulp) was studied. ATMP combines selective wood disintegration by mechanical pre-treatment and refining at elevated intensity with chemical treatment after defibration (in this study hydrogen peroxide, alone and in combination with alkali).

The electrical energy efficiency and pulp quality using ATMP were evaluated and compared to a conventional TMP process. One goal was to retain the combination of optical properties and strength of typical TMP. Fibre properties (structural dimensions, external and internal fibre development) as well as the amount of split fibres resulting from TMP and ATMP processes were compared.

The results indicate an electrical energy efficiency improvement potential of 0.65 MWh/odt (34%) at tensile index 30 N.m/g of ATMP compared to reference TMP. All ATMP pulps retained their TMP character, i.e. the relationships between light scattering coefficient, density, elongation properties and strength that are important for printing paper. ATMP had a significantly lower content of shives and higher external and internal fibre development compared to TMP at equal energy application. The long fibre fraction of ATMP also contained significantly higher amount of split fibres. The brightness of ATMP produced with addition of hydrogen peroxide (28 kg/odt) and magnesium hydroxide (14 kg/odt) was 14 ISO % higher compared to TMP.

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Substantial reduction of electrical energy in refining is needed in order to secure the long term operation of many TMP mills. With rapidly growing energy costs the problem of high energy demand has become acute in recent years.

Advanced Thermomechanical Pulp process in pilot and mill scale was recently described (Hill et al. 2009, Hill et al. 2010, Johansson et al. 2011). The process combines mechanical pre-treatment and high-intensity first stage refining (using feeding refiner segments and/or elevated refiner speed). Another feature of the ATMP process is the introduction of chemicals after defibration. This is possible due to the RTF (Retention, Temperature, and Fiberization) mechanical pre-treatment in Impressafiner and Fiberizer units during which the defibration of chips takes place. The initially used chemical was sodium bisulphite which gave significant reduction in electrical energy demand and some brightness improvement. A partial ATMP process with sodium bisulphite is now in operation at Norske Skog Pisa mill in Brazil and the reduction in energy demand and improvement of pulp quality are confirmed in mill scale (Hill et al. 2009, 2010).

Further energy efficiency and brightness improvements were achieved when hydrogen peroxide was used in ATMP refining (Hill et al. 2010). A pilot refining trial with Pinus taeda (Loblolly pine) was conducted earlier using the ATMP process configuration with hydrogen peroxide and magnesium hydroxide (Johansson et al. 2011). Energy efficiency was improved by approximately 1.1 MWh/ODT or 42% calculated at equal tensile index (25 Nm/g) compared to TMP. The increase in pulp brightness was approximately 14 ISO % with 25 kg/odt peroxide charge and the amount of shives in ATMP was significantly lower than in TMP.

The main objectives of this study were:

- To evaluate the energy efficiency improvement potential of a peroxide-based ATMP process when used on spruce raw material
- To evaluate the role of alkali source (sodium hydroxide and magnesium hydroxide) in hydrogen peroxide ATMP refining
- To compare the development of fibre properties in ATMP refining to the TMP process
- To suggest possible mechanisms explaining the energy efficiency improvement in ATMP refining compared to conventional TMP

In this study, ATMP process with pre-treatment consisting of Impressafiner and Fiberizer units was used. A chemical system similar to the one used previously, consisting of hydrogen peroxide and

alkali (Johansson et al. 2011) was applied. Refining intensity was elevated using feeding segment pattern, but not higher rotational speed. The speed was kept at 1800 rpm both for the TMP reference and ATMP. Most of the TMP lines in the world today use low-intensity refining and thus a low-intensity TMP reference is appropriate.

Due to differences in wood morphology and chemistry (Reme 2000, Yuan et al. 2006, Fernando, Daniel 2008) different results can probably be obtained when the ATMP process is used on spruce and pine raw material. This was reported to be so in the case of mechanical pre-treatment, where a larger energy reduction could be achieved in subsequent refining of pine compared to spruce (Robertsen et al. 2001, Gorski et al. 2009). Since mechanical pre-treatment is an important part of ATMP refining, it is reasonable to believe that the reduction in energy demand could also be somewhat lower when spruce raw material is used instead of pine in the ATMP process. Reduction of energy demand by 30% or 0.6-0.8 MWh/odt was reported when mechanical pre-treatment was used together with undisclosed chemical treatment on spruce raw material (Sabourin et al. 2003). It is important to study the refining energy efficiency for spruce since most of mechanical pulp today is produced from spruce-like fibres. A prerogative for utilizing the energy reduction is that the character of produced ATMP is similar to the character of TMP, i.e. that the optical properties and strength of the pulps as well as other properties such as density and elongation are preserved on the same level using the ATMP concept. It has previously been shown to be possible for pine (Johansson et al. 2011). One goal of this study was to show that this also is the case for spruce raw material.

The chemical system chosen for this study (based on hydrogen peroxide) was the one that showed best potential for pine raw material. Advantages of this system include higher brightness, greater reduction of energy demand and better produced pulp quality (Johansson et al. 2011). Granted that the chemical system was not optimized, it is possible that optimized conditions would lead to another outcome of the trials. However, the fact that the chemical system chosen for this study does not introduce sulphur into the process and can be run at mildly alkaline conditions is also of great advantage from environmental and equipment point of view.

Tower bleaching at high consistency with alkaline hydrogen peroxide has been an established bleaching technology since the mid eighties. It is well-known that in order to optimize the stochiochemistry of peroxide bleaching, the initial pH should be in the range of 11.2-11.7 depending on temperature. Thus, without an alkali source, the pH in conventional bleaching is too low and stable perhydroxyl anions are not formed in sufficient quantities (Agnemo 1981, Moldenius 1983). Traditionally, sodium hydroxide has been used as an alkali source. Recently, however, the use of magnesium hydroxide has increased. Its advantages include lower generation of COD, higher light scattering coefficient and no need for stabilizers compared to when sodium hydroxide is used (Johnson et al. 2002, Leduc et al. 2008, Ni 2005, Ni and He 2010). One important difference is that magnesium hydroxide is not fully dissolved and thus is added as slurry. Hydroxyl ions are consumed first by the peroxide and then successively by the wood material while the slurry is gradually dissolved.

The role of the alkali source in refiner bleaching is rarely discussed in the literature. It is also not clear whether the chemical reactions involved in bleaching and reactions that lead to the reduction of electrical energy demand in ATMP are the same. Since the hydrogen peroxide ATMP concept seems to have a good potential to improve the energy efficiency in refining, the role of the alkali source is investigated in this study to further improve the process understanding. Additions of hydrogen peroxide alone as well as in combination with magnesium hydroxide and sodium hydroxide are investigated while refining variables such as intensity and pre-treatment are kept constant. Hydrogen peroxide is used without alkali in one of the trials to evaluate if energy reduction and bleaching is possible due to elevated temperature inside the refiner which may lead to formation of enough perhydroxyl anion even if no alkali is present. This has been shown to be possible earlier (Presley, Hill 1996, Rämö 2003). Addition of sodium hydroxide leads to significantly higher pH compared to addition of magnesium hydroxide and this is why a comparatively low charge (5 kg/odt) of sodium hydroxide was attempted in this study. Goal was to have approximately similar pH in both magnesium hydroxide and sodium hydroxide based ATMP processes.

In order to find explanations to the improvements in energy efficiency and pulp quality achieved by ATMP compared to TMP, properties of fibres were characterised at the level of defined length populations and individual particles for selected pulps. External fibrillation, flexibility and cross-sectional characteristics have been shown to be suitable indicators for effects of refining process on fibres (Atack 1981, Koran 1981, Karnis 1994, Kure 1999, Reme 2000, Corson 2001). It was earlier proposed that chemicals added between chip defibration and fibre development stages in ATMP

refining selectively enhance the fibre development process (Johansson et al. 2011). Undoubtedly, mechanical pre-treatment and elevated refining intensity also have an effect on fibre development in refining. However, the effects of chemicals and process conditions were not separated in this study and ATMP process was compared to a TMP reference. The development of fibres has been traditionally divided into internal (flexibility) and external (surface) development (Koran 1981, Kano et al. 1982, Marton, Eskelinen 1982, Lammi, Heikkurinen 1997, Corson 2001). Also, the content of longitudinally split fibres in a pulp has been found to be of importance for surface properties of printing paper linked to the fibre collapsibility (Kure 1999, Reme 2000, Johansson, Dahlqvist 2001). In this study, these variables are assessed in order to improve understanding of the effect that the ATMP process has on fibre properties. The effects of fibre properties on the pulp and laboratory sheet properties are also investigated using traditional pulp and paper analysis. The potential for reducing the energy demand in refining is then assessed based on the target variable (tensile index) and the improvement in other properties caused by the ATMP process.

Materials and Methods

The pilot plant

The pilot trial was conducted at the Andritz pilot plant in Springfield, Ohio, United States. The trial consisted of refining five pulps, see *Table 1*. White spruce (Picea glauca) from Wisconsin, USA, was used as raw material. Refining was conducted in three stages; the third stage was run at three energy levels. In all ATMP trials, chips were pre-compressed in an Impressafiner and defibrated further in a Fiberizer prior to refining. The principal process layout is schematically shown in *Fig 1*. All chemicals except the chelating agent (DTPA) were charged in the first stage refiner (through the dilution water added in the inner ring). DTPA (0.3%) was charged at the Impressafiner outlet,

Table 1. Pulps 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 2.8 bar	No chemicals	5.4
ATMP (Mg+P) 3.5 bar	$Mg(OH)_2$ 1.4% + H_2O_2 2.8%	7.3
ATMP (Mg+P) 5.2 bar	$Mg(OH)_2$ 1.3% + H_2O_2 2.8%	7.2
ATMP (Na+P) 5.2 bar	NaOH 0.5% + H_2O_2 3.5%	7.0
ATMP (P) 5.2 bar	H_2O_2 2.8%	5.2

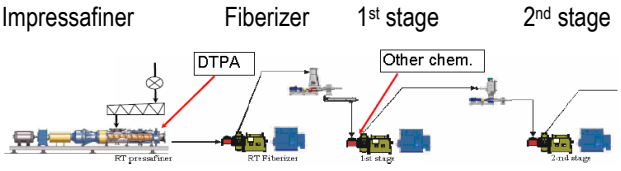

Fig 1. Pilot plant refiner configuration for ATMP production (no mechanical pre-treatment or chemicals were used for TMP).

where the pressure was relieved. Chemical recipes are given in *Table 1* (charges on oven dry weight of pulp). The pulp was stored in drums between the refining stages with retention times varying between 30 and 120 minutes Refiner variables for the trials are given in *Table 2*. All pulps (TMP and ATMP) were refined using the same rotational speed in the first stage, but the segment patterns were holdback for TMP and expel (feeding) for ATMP. The same segments, Durametal 36604, were used in both cases but the direction was switched. Temperatures were calculated from refining pressures using saturated steam tables. Unfortunately, the refining system was not equipped with accurate enough gap measurements which can also be seen in earlier studies (Senger et al. 2006). Analysis of the accuracy in the measurement of specific energy demand using the same equipment was performed earlier (Johansson et al. 2011). The same calculation can be used in this study and errors in SEC presented in *Table 5* are based on it.

The ATMP concept

The ATMP concept consists of two additional unit operations compared to the TMP: mechanical pre-treatment in a compression screw (Impressafiner) followed by defibration in a Fiberizer unit (together called RTF pre-treatment, Retention-Temperature-Fiberization) prior to primary stage refining at higher intensity. During the mechanical pre-treatment, initial defibration of chips to coarse fibres and fibre bundles takes place. These are refined in a first stage refiner where chemicals are charged. This way, an enhancement of fibre development with chemicals is possible, see *Fig 2*. At the same time, unfavourable CTMP-like defibration (fracture planes in the reactive outer parts of the fibre walls) due to impregnation of whole chips with chemicals is avoided. Primary refining is conducted with higher intensity using feeding refiner segments and/or elevated refining speed. In this study, higher intensity was achieved by switching to feeding segment pattern in the ATMP trials.

Table 2. Refining variables during the pilot trial

	Impressafiner*	Fiberizer SD 36-1CP	1st stage refiner SD 36-1CP	2nd stage refiner DD 401	3rd stage refiner DD 401
TMP trials					
Preheating time (s)	Not used	Not used	120	0	0
Speed (rpm)	-	-	1800	1200	1200
Casing pressure (bars)	-	-	2.8	Atm.	Atm.
Casing temperature (°C)**	-	-	142	Atm.	Atm.
Segment pattern	-	-	Hb***	-	-
ATMP trials					
Preheating time (s)	15	0	10-15	0	0
Speed (rpm)	38	1800	1800	1200	1200
Casing pressure (bars)	1.5	1.7	5.2 (3.5)****	Atm.	Atm.
Casing temperature (°C)**	0	138	161 (148)	Atm.	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)*

**** *Casing pressure of 3.5 bars (148 ºC) was used during one of the ATMP (Mg+P) trials. Lower casing pressure was tested to evaluate the effect on the energy demand*

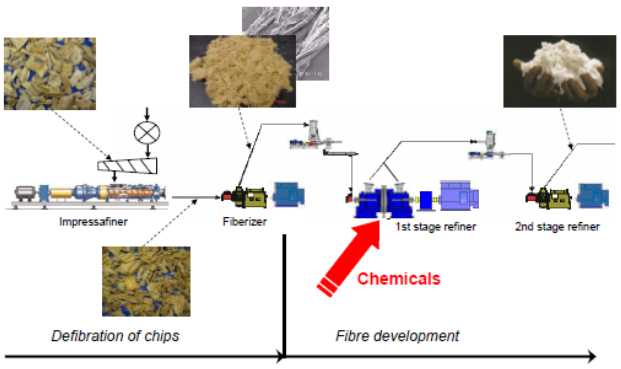

Fig 2. Combination of selective wood disintegration and targeted addition of chemicals in the ATMP process.

Physical testing

All laboratory testing was conducted according to TAPPI standards at the Andritz Pilot Plant laboratory, Springfield, Ohio, USA. CSF was determined according to the T227 standard. Pulp was fractionated in Bauer McNett according to T233. Handsheets (approximately 60 g/m^2) were prepared according to T205 from pulp disintegrated according to the same standard method. Strength properties of the handsheets were evaluated according to T220 standard (TEA was determined according to T494). Optical properties were evaluated according to T218 (brightness), T425 (light scattering) and T425/T220 (light absorption). Shive content was determined using Pulmac Fractionator with a 0.10 mm slot screen. Error is provided where possible and the bars denote 95% confidence interval.

Fibre characterisation

Four tertiary pulp samples were selected to investigate how each chemical strategy used in the ATMP concept influenced the fibre properties compared to the TMP reference. It is desirable to compare fibre properties for pulps refined to approximately equal tensile index since it is used to evaluate the refining energy demand in this study. However, not enough samples could be obtained during the pilot trials to choose pulps with equal tensile index. Thus, pulps were compared at equal energy application and tertiary ATMP (Mg+P) refined at lower primary refiner casing pressure was chosen for the fibre study. For TMP and ATMP (Mg+P) trial series, pulps from all three refining stages were characterised as well to enlighten the mechanisms of fibre properties development during refining.

Fibres from P16/R30 fraction collected after standard Bauer McNett fractionation (SCAN M6:69) corresponding to approximately 2-3 g oven dry material were diluted in ca. 1100 ml de-ionized water of 21 ºC and allowed to settle in a scaled measuring cylinder. The sedimentation rate of the suspension was recorded in the Excel worksheet template referred to by Wakelin (2004) for the initial phase of settling, where the height of the fibre-water interface decreases at constant velocity. The test was repeated for three more consistency levels, after the suspension was progressively diluted. The sedimentation specific surface area index (S^3A index) was calculated according to *Eq 11* in (Wakelin 2004) with values of 1150 kg/m^3 for fibre apparent density and 998 kg/m^3 for water density. A value of 0.0031 m^3/kg was used for

fibre specific volume, 5.55 for channel factor and 0.001002 kg/ms for fluid viscosity.

Fibre flexibility was assessed from FiberMaster bendability measurements. 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 subjected to the sedimentation test. At least 5 parallel measurements were carried out for each sample, and results are reported for the well defined length interval 1.5 to 3.0 mm ("B3").

Fibre cross-sectional characteristics were determined according to the method described by Reme et al. (2002). A combination of automatic and manual editing was applied for assessment of fibre dimensions and the degree of fibre splitting. The unambiguous classification of the fibre material into intact and split fibres is difficult. Therefore, a characteristic called "fibre split index" was used, which denotes the area proportion of split and fragmented fibres relative to all fibre material examined in a given population. Given that the fibres in the examined image are of same length and wall density, the area proportion would equal the weight proportion. Image analysis was performed using ImageJ. In case with ATMP, where chemicals may have influenced the fibre wall density, it is possible that this may have affected the fibre split index measurement somewhat.

Results and discussion

Refiner operation

Accumulated SEC (Specific Energy Consumption) in refining during the pilot trial is given in *Table 3*. Corresponding freeness values measured for pulps after each refining stage can be found in brackets.

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

	Imp.	Fib.	1st	2nd	3rd stage
TMP	-	-	0.94 (630)	1.90 (290)	2.45 (180) 2.69 (120) 2.97 (80)
ATMP (Mg+P) 3.5	0.05 (-)	0.30 (730)	0.97 (490)	1.89 (160)	2.41 (90) 2.59 (70) 2.90 (40)
ATMP (Mg+P) 5.2	0.05 (-)	0.30 (730)	0.92 (430)	1.81 (160)	2.33 (90) 2.53 (60) 2.71 (50)
ATMP (Na+P)	0.05 (-)	0.30 (730)	0.96 (590)	1.92 (240)	2.46 (140) 2.68 (110) 2.92 (70)
ATMP (P)	0.05 (-)	0.30 (730)	0.84 (460)	1.84 (160)	2.45 (180) 2.69 (120) 2.97 (80)

Energy demand to produce pulp with desired quality is further discussed in "Energy efficiency and handsheet strength".

Pulp character

The TMP process became dominant for the production of printing paper due to the fact that pulp, produced using this process, 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 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. In this study it means that the relationship between tensile index (used to evaluate energy demand) and other important pulp and sheet properties must be similar for both TMP and ATMP. Earlier it was established that ATMP pulp produced from pine has the same character as reference TMP (Johansson et al. 2011).

To establish the character of the produced ATMP, seven important properties of the pulps were chosen, see *Table 4*. P-values determined by ANOVA (analysis of variance) for all the five pulps simultaneously (5 refining stages for each pulp, 25 data points) were used as a main indicator of whether there was a significant difference in the character of properties of ATMP compared to TMP. Tensile index had been chosen as a base for comparison since it was later used to define the energy demand in refining. A low P-value indicates a small probability of significant difference in character. 95% confidence interval means that a P-value must be smaller than 0.05. Analysis of variances is a standard statistical method and is well described elsewhere (Box et al. 2005). It is also previously described in this specific application in more detail (Johansson et al. 2011) and is widely used in other disciplines for detecting statistical differences within data populations (Moen et al. 2010).

Table 4. Coefficients of determination of linear regressions for three energy levels of all five pulps, TMP and ATMP (25 data points are plotted together and compared at equal tensile index)

Property	Unit	P-value
Density	kg/m^3	$4.0*10^{-14}$
Light scattering coefficient	m^2/kg	$7.8*10^{-10}$
Canadian Standard Freeness	ml	$1.1*10^{-17}$
Fibre length (length weighted)	mm	$2.9*10^{-4}$
BMN P200 fraction (weight)	%	$1.2*10^{-4}$
Elongation	%	$1.8*10^{-8}$
Tensile energy absorption	J/m^2	$1.8*10^{-15}$

Physical properties like density, tensile energy absorption and elongation all show very similar character for TMP and ATMP processes. This means that the TMP character of the pulp was preserved using the ATMP process and thus that similar mechanisms of sheet densification and fibre network formation probably contribute to the physical properties in both cases.

The light scattering coefficient was similar for TMP and ATMP compared at similar tensile index. This is important because it means that the light scattering of ATMP is kept on the same level as in TMP reference in spite of the use of chemicals. The hypothetic explanation as to why the light scattering is retained in ATMP refining is that during conventional CTMP refining, where whole chips are impregnated with chemicals, defibration occurs closer to the lignin-rich middle lamellae which is unfavourable from the light scattering point of view (Heikkurinen et al. 1993, Höglund, Wilhelmsson 1993, Norgren 2008). In ATMP this is avoided since initial defibration occurs during the RTF pre-treatment and chemicals are charged only afterwards. Mechanical pre-treatment also makes it possible to reduce the transport problems when chemicals are applied – a large specific surface area is available for chemical reaction on fiberized material compared to charging chemicals on whole chips. All this is in agreement with the results obtained using the ATMP process on pine raw material (Johansson et al. 2011).

Earlier studies showed that refining at increased intensity leads to fibre shortening, i.e. the character of the pulp is altered. TMP and ATMP in this study were shown to have very similar character with respect to average fibre length and fines content. This could indicate that mechanical pre-treatment makes the material more suitable for refining at higher intensity. This has been proposed in earlier studies (Kure et al. 1999, Sabourin et al. 2003). Possible reason could be that fiberized material has much less variation in size distribution compared to whole wood chips. This should improve the stability of the feed to the primary refiner which is very important in higher intensity refining, known to lead to a smaller refiner gap.

Fig 3 shows that tear index decreased slightly with increasing tensile index for all studied ATMP. For TMP, however, tear index passed through a distinct maximum first and then decreased sharply with increasing tensile index. Earlier studies of accuracy in the tear index measurement show that a difference of at least 0.8-1 mNm^2/g must be obtained for the results to be significant (Strand et al. 1989). Thus, the differences in the tear index between the different ATMP pulps appear insignificant. The difference in the tear index between ATMP and TMP appear significant at lower tensile index, but the difference is not significant at higher tensile index. Tear index is known to correlate well to average fibre length and content of fines. Since the statistical analysis showed that there is no significant difference between those variables for TMP and ATMP and the accuracy of tear index measurement is relatively low, the difference in tear index development between TMP and ATMP could be considered insignificant or very small. It is well-known that development of tear index passes through a maximum when plotted against tensile index. This is clearly seen for the TMP reference, but no maximum is evident for any of the ATMP pulps. The absence of tear index maximum for ATMP could be explained by the fact that it was passed earlier in the development of tensile index, something that reflects a faster and more energy-efficient development of pulp quality. No difference in tear index between TMP and ATMP was found in an earlier study when pine was used as raw material (Johansson et al. 2011).

The shives content of ATMP pulps measured after second stage refining was lower than measurable by means of the Pulmac method see *Fig 4*. The difference between ATMP and TMP was especially pronounced at lower energy demand suggesting a much more efficient fibre separation already in the first stage during ATMP refining. ATMP produced using hydrogen peroxide alone had only slightly higher content of shives after first stage refining, but it was still much lower than the TMP reference. This feature can be used to simplify or eliminate screening of pulp. Different mechanisms can be

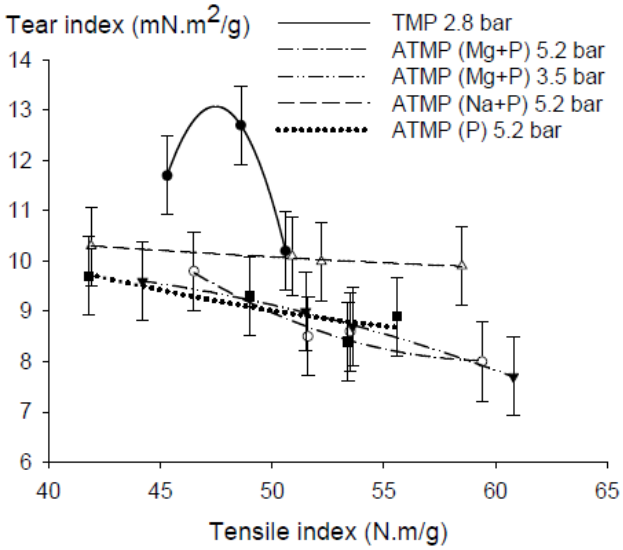

Fig 3. Relationship between the tear and tensile indexes of handsheets.

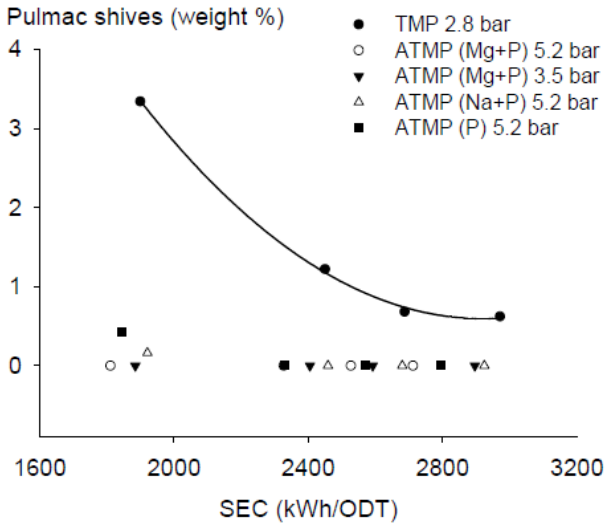

Fig 4. Shives content of the pulps (Pulmac 0.10 mm screen, weight %).

proposed to contribute to lower shives content in ATMP. Firstly, ATMP was refined using directional (expel) refiner segment pattern in the first stage refiner which could have caused more efficient shive elimination. This has been shown before for spruce raw material (Kure et al. 1999, Sabourin 2003). Secondly, first stage refining was performed with addition of chemicals known to cause swelling and dissolution of fibre components and thus weakening of the fibre structure. It is thus possible that shive elimination is a result of more efficient fibre separation assisted by chemicals. CTMP, where chemicals are used to impregnate whole chips, is known for its lower shive content compared to TMP (Åkerlund, Jackson 1984). To further study this effect, experiments that separate the contribution of higher intensity, mechanical pre-treatment and addition of chemicals in the first stage refining should be designed.

Reduction in shives content using the ATMP concept was much higher in this study (almost no shives were detected at all at higher energy levels) compared to when pine was used as raw material (0.1-0.2 % shives were detected at higher energy levels). Reference pine TMP contained much less shives at lower energy levels (around 1%) compared to around 3% which was detected in spruce TMP in this study (Johansson et al. 2011). This difference could be explained by the pine morphology. It has been shown that much more energy is needed for refining of pine compared to spruce, but the defibration of pine chips is more efficient (Fernando, Daniel 2008). Same mechanisms could also be valid for defibration of shives in refining.

Fibre properties

Direct measurements of fibrillation and fibre flexibility have proven difficult for mechanical pulps. External fibrillation can be assessed from sedimentation measurements, given that other important influencing fibre properties such as apparent density, outer diameter and fibre length are kept constant (Wakelin 2004). The possible dissolution of fibre wall components by process chemicals applied in the ATMP concept and resulting potential changes in fibre wall density has not been verified by measurements in this study. Changes in the sedimentation specific surface area index may thus not only reflect changes in external fibrillation.

Settling measurements for mechanical pulp fibres were first described by Marton and Robie (1969) who investigated the influence of temperature, consistency, coarseness, fibre length and specific surface area. Wakelin (1999, 2004) developed the method further and suggested sedimentation measurements for individual Bauer McNett fractions in order to assess fibre quality aspects affecting e.g. surface and tensile strength and linting propensity. Building on sedimentation studies by Sell et al. (1988), Wakelin related initial settling velocity to stock concentration by applying Stokes law and liquid permeability theory: `*The sedimentation velocity is a balance between the opposing forces of gravity and buoyancy; and the fluid drag acting on the external surface area. This balance can be characterised by the fibre specific surface area*´ (Wakelin 2004).

All investigated ATMP concepts (with addition of peroxide only or peroxide combined with sodium or magnesium hydroxide) had third stage pulp fibres with higher specific surface area indexes than the reference TMP. Differences were statistically significant at the 95% level of confidence between TMP and ATMP (Mg+P) fibres, *Figs 5a* and *5b*. The S^3A index values ranged between 17.0 m^2/g for the TMP and 19.7 m^2/g for the ATMP (P) fibres (an increase by 14%). Wakelin (2004) gives some reference values for different pup types and freeness levels see *Table 5*. It has to be kept in mind that the reference data is based on different refining processes and raw materials.

Table 5. Sedimentation specific surface area index values obtained for the long fibre fraction P14(16)/R30 in this study and given in the reference (Wakelin 2004).

Pup type	S^3A index (m^2/g)	CSF (ml)
Present study (White spruce)		
TMP 3rd stage	17.0	82
ATMP (P) 3rd stage	19.7	78
ATMP (Na+P) 3rd stage	19.0	73
ATMP (Mg+P) 3rd stage	19.5	42
Reference (Norway spruce)		
Mainline TMP	10.2	93
Refined reject	15.0	96
Magazine TMP	24.0	30

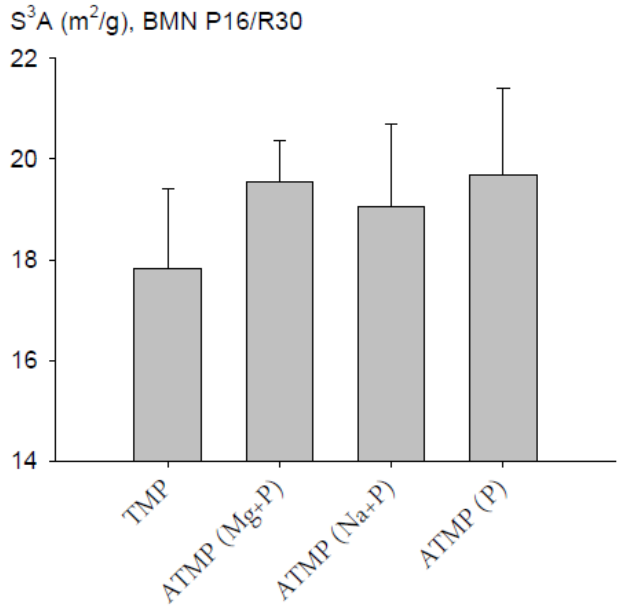

Fig 5a. Specific surface area index of long fibres from the P16/R30 BMN fraction (third stage pulps with approximately similar energy demand).

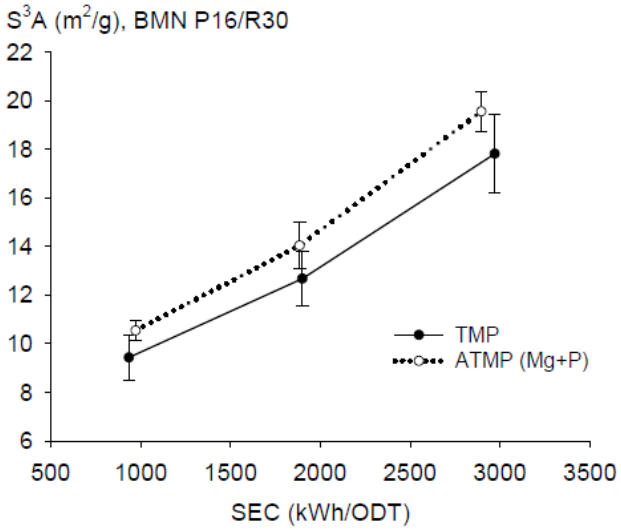

Fig 5b. Specific surface area index of long fibres from the P16/R30 BMN fraction (all three stages of refining for ATMP (Mg+P) and TMP).

As for the specific surface area index development in the ATMP (Mg+P) and TMP concepts, a difference of 1.1 m^2/g S^3A index appeared to be set already after the first refining stage and was extended to 2.5 m^2/g S^3A index after the third refining stage, *Figs 5a* and *5b*.

Swelling of fibres is known to promote external fibrillation during beating of chemical pulp (Cress, Bialkowski 1931, Giertz 1957). This is explained by the fact that swelling softens the fibres which fibrillate upon beating. Without swelling, the fibres are stiff and brittle and beating leads to cutting. For mechanical pulp though, it was proposed that external fibrillation can be achieved without swelling (Kang 2007).

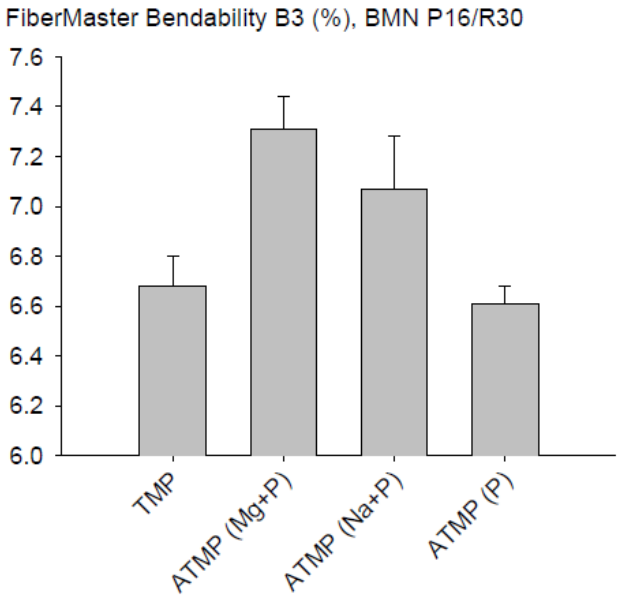

Fig 6a. FiberMaster bendability of fibres from the P16/R30 BMN fraction (third stage pulps with approximately similar energy demand).

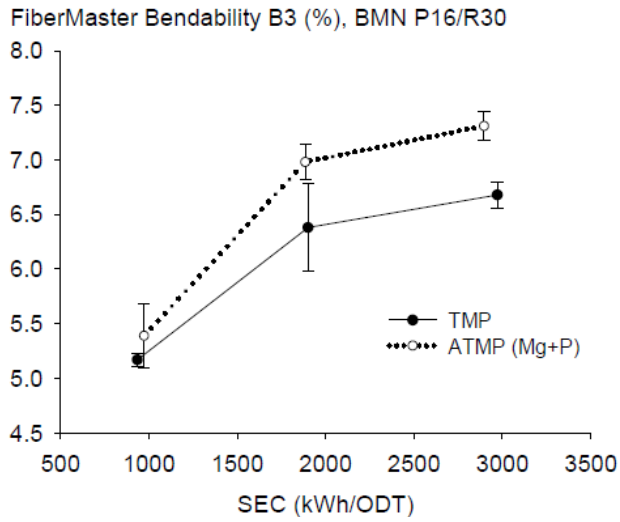

Fig 6b. FiberMaster bendability of fibres from the P16/R30 BMN fraction (all three stages of refining for ATMP (Mg+P) and TMP).

Two out of three investigated ATMP concepts had third stage pulp fibres with significantly higher bendability than the reference TMP fibres, compared to approximately the same level of specific energy application, *Fig 6a*.

The bendability of the ATMP fibres was higher already after the first refining stage, but here the flexibilization seemed to progress at a faster rate upon second and third stage refining for ATMP than TMP, resulting in 0.64 units higher bendability for the third stage (Mg+P) ATMP fibres, *Fig 6b*. It was earlier proposed that internal fibrillation in mechanical pulps develops later in the refining process because it is restricted by the lignin-rich

structure of the outer fibre walls (Stone et al. 1968, Eriksson et al. 1991).

Third stage ATMP (Mg+P) contained nearly 1.3 times as much split fibre material as the reference TMP or any other ATMP pulps in fraction R50, *Figs 7a* and *7b*. Again the level of fibre splitting was set at the first refining stage and additional splitting appeared to progress throughout subsequent stages at the same rate for ATMP and TMP. Both the TMP and ATMP series were refined at 1800 rpm in the primary refining stage. However, high intensity directional feeding segment pattern was used for the ATMP series and low intensity segment pattern for the TMP series. Increased intensity of first stage refining was earlier found to cause increased fibre splitting (Kure, Dahlqvist 1998, Kure et al. 1999, 2000, Reme 2000, Johansson, Dahlqvist 2001, Sabourin 2003). Of all characterized ATMP pulps, only ATMP (Mg+P) showed significant increase in the split fibre amount compared to TMP. It thus appears likely that the increase is a result of the addition of magnesium hydroxide and hydrogen peroxide rather than the higher intensity segments used in ATMP refining. This contradicts to some extent earlier findings where bidirectional segments with the same pattern (Durametal 36004) operated in expel mode were found to produce significantly more fibres with split circumferences compared to operation in holdback mode (Kure et al. 2000). The proportion of split fibres was shown to be more then doubled when a combination of higher intensity expel segment pattern and increased rotational speed of the refiner (from 1800 rpm to 2300 rpm) was used compared to a conventional TMP process where the refiner was operated at holdback mode with 1800 rpm rotational speed (Johansson, Dahlqvist 2001).

Since hydrogen peroxide was used for several ATMP series, it seems that it was the addition of $Mg(OH)_2$ and not peroxide that increased the amount of split fibres in the pulp. The effect is somewhat unexpected and not previously encountered in the literature. One explanation could be that $Mg(OH)_2$ which was not entirely dissolved in the pulp suspension caused some kind of abrasive treatment of fibres due to its crystalline form. Abrasive treatment of fibres during refining with the help of for example grits on the segments was previously shown to increase the degree of fibre splitting (Samboon 2009). However, the mechanical action must be very different when abrasive particles are used in a refiner compared to an abrasive segment surface and this matter should be studied more to be able to draw any conclusions.

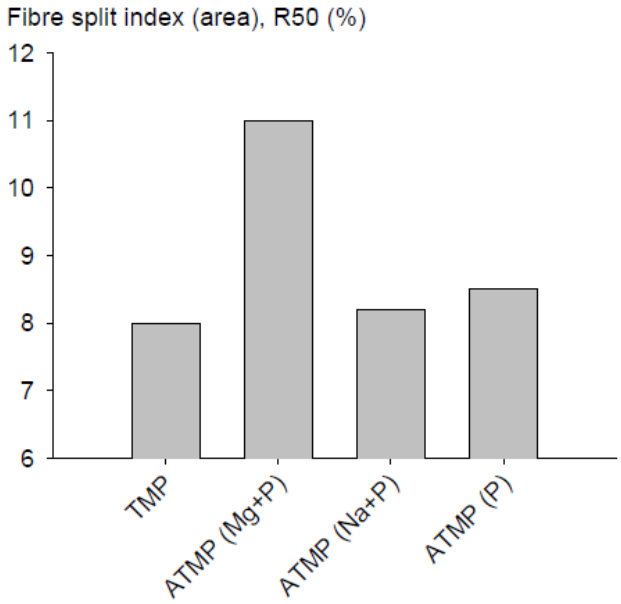

Fig 7a. Area-based degree of fibre splitting, fraction BMN R50 (third stage pulps with approximately similar energy demand).

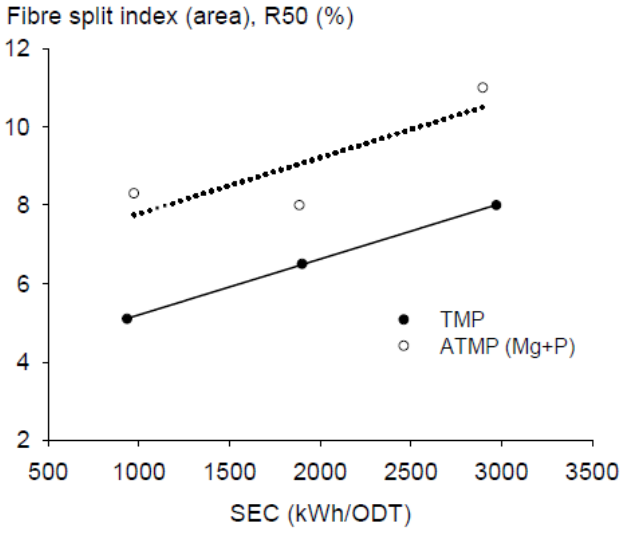

Fig 7b. Area-based degree of fibre splitting, fraction BMN R50 (all three stages of refining for ATMP (Mg+P) and TMP).

Fibre coarseness was significantly reduced upon both (Mg+P) ATMP and TMP refining, *Figs. 8a* and *8b*. Coarseness reduction upon progressing refining appeared more effective for TMP than ATMP. ATMP (Mg+P) fibres had lower coarseness after the first and second refining stage than the TMP fibres. The measurement uncertainty was large though, especially for the coarse first stage TMP. Differences in fibre coarseness for the different third stage pulps were not statistically significant. The improvements in specific surface area index and bendability as shown for several of these pulps compared to the reference TMP are thus not a result of lower coarseness, but suggest improved external fibrillation and beneficial alterations to the fibre wall structure.

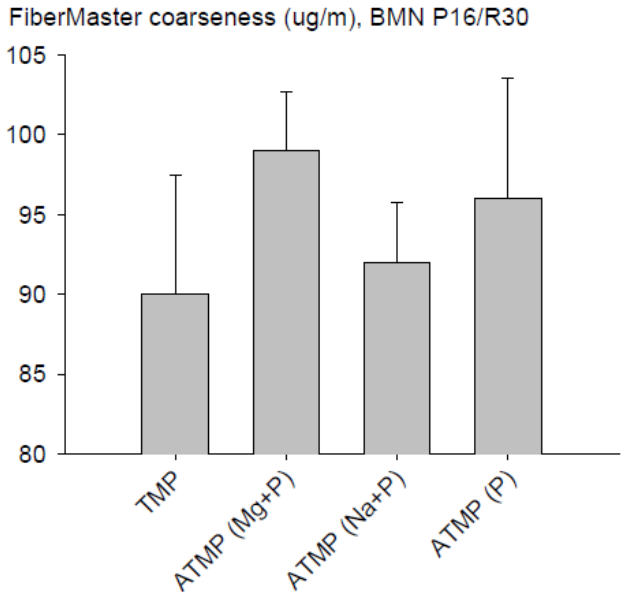

Fig 8a. Coarseness of fibres from the P14/R30 BMN fraction (third stage pulps with approximately similar energy demand).

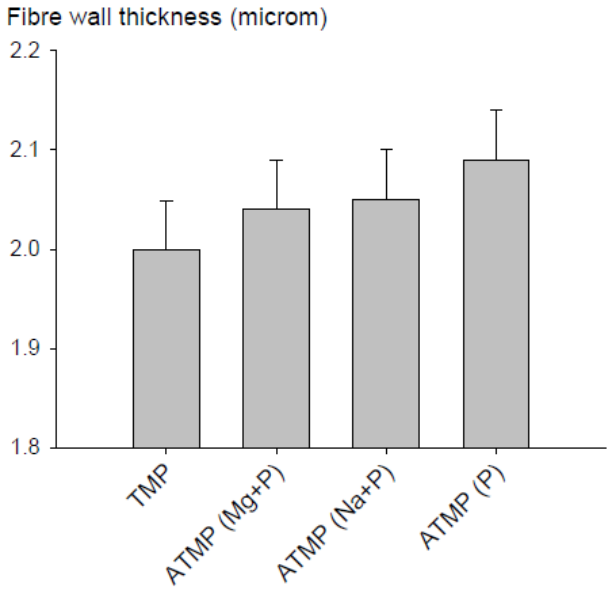

Fig 9a. Mean wall thickness development upon refining for TMP and ATMP (third stage pulps with approximately similar energy demand).

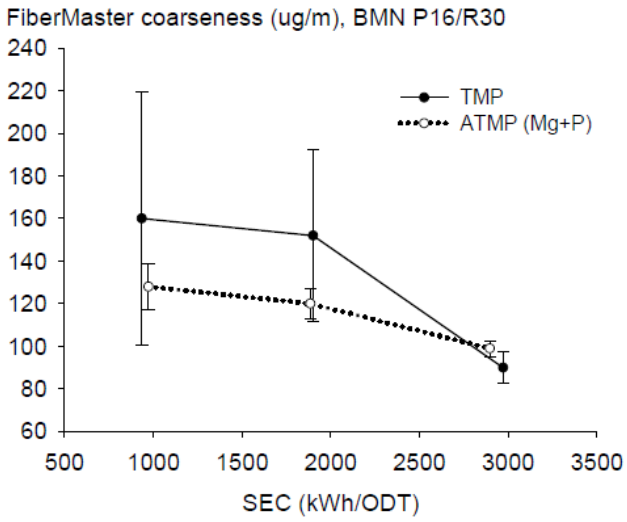

Fig 8b. Coarseness of fibres from the P14/R30 BMN fraction (all three stages of refining for ATMP (Mg+P) and TMP).

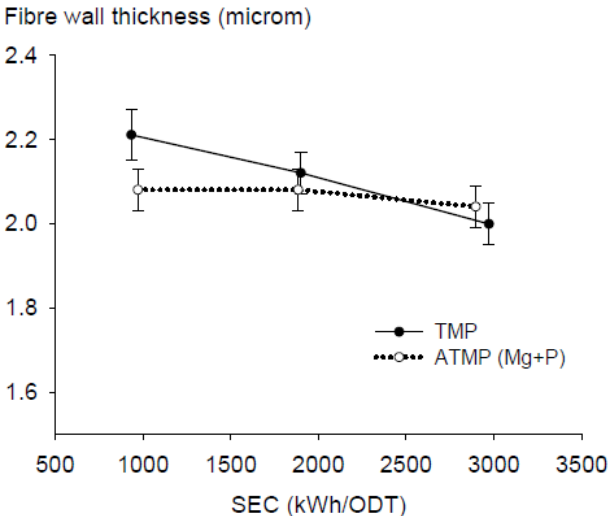

Fig 9b. Mean wall thickness development upon refining for TMP and ATMP (all three stages of refining for ATMP (Mg+P) and TMP).

In high consistency refining of mechanical pulps, a reduction of fibre coarseness with increasing SEC is a well-described phenomenon, attributed to peel-off mechanisms (Karnis 1994). An increase in fibre flexibility and collapsibility as well as a reduction in fibre wall thickness would be related trends of the progressing fibre wall peeling with increasing specific energy application.

The fibre wall thickness development for the reference TMP produced in this study confirms the well known mechanism of fibre wall thickness reduction by peeling off the fibre wall layers during refining (Kure 1999, Reme 2000), *Figs 9a* and *9b*. However, the intact ATMP (Mg+P) fibres did not follow the typical and significant fibre wall thickness reduction observed for the TMP refining process, something that eventually resulted in higher mean fibre wall thickness after third stage refining of the investigated ATMP concepts compared to TMP. The mean fibre wall thickness of the selected ATMP (Mg+P) was already significantly reduced after the first refining stage compared to TMP, which confirms the coarseness observations shown in *Figs 8a* and *8b*.

Figs 5 to *9* show that differences in important fibre properties such as splitting, external fibrillation, bendability and mean fibre wall thickness/coarseness between TMP and ATMP were obviously manifested and quantifiable already after the first refining stage. The results also indicate that the

development of these properties may have progressed in a different manner upon successive refining between ATMP and TMP pulps. However, the available data does not yet allow for distinguishing the effects of the individual process steps (mechanical pre-treatment, high intensity refining and chemical addition) of the ATMP process concept. To achieve this, complete refining curves for mechanically pre-treated pulps refined at elevated intensity with and without chemical addition should be compared to TMP. At this stage, it is clear that the ATMP process yields fibres with better external and internal fibrillation at equal energy application pointing at a potential for reducing the energy demand in refining.

Fibre wall reduction and decreasing fibre coarseness upon high consistency mechanical pulp refining is commonly attributed to external peeling. For the ATMP (Mg+P) refining process a significant coarseness reduction was observed, while the mean fibre wall thickness remained unchanged. Dissolution of fibre wall components may have taken place and decreased the fibre weight (coarseness). An unchanged mean fibre wall thickness from first to third refining stage may be the result of absent external peeling, or a combination of external peeling and simultaneous swelling of the fibre wall, due to introduced charge. It appears likely that fibre peeling took place, since the development of the sedimentation specific surface area index from first to third stage as well as the fractional composition were similar for ATMP and TMP.

More research is necessary to understand the reasons for the significant fibre wall thickness reduction already after the first ATMP (Mg+P) refining stage, and to verify the presented observations on fibre development in the novel ATMP refining process. Also, the influence of refining intensity (feeding segment pattern and elevated rotational speed of the refiner) and addition of chemicals must be studied separately.

Energy efficiency and handsheet strength

As can be seen from *Fig 10* and *Table 6*, a significant reduction in electrical energy demand to reach equal tensile index is possible using the ATMP concept compared to the TMP reference. Of all tested chemical strategies, the combination of hydrogen peroxide and magnesium hydroxide offered the largest reduction of energy demand required to reach a certain tensile index. This chemical strategy combined with refining at higher casing pressure in the first stage refiner (5.2 bars) yielded approximately 0.65 MWh/ODT or 37% reduction in electrical energy demand compared to

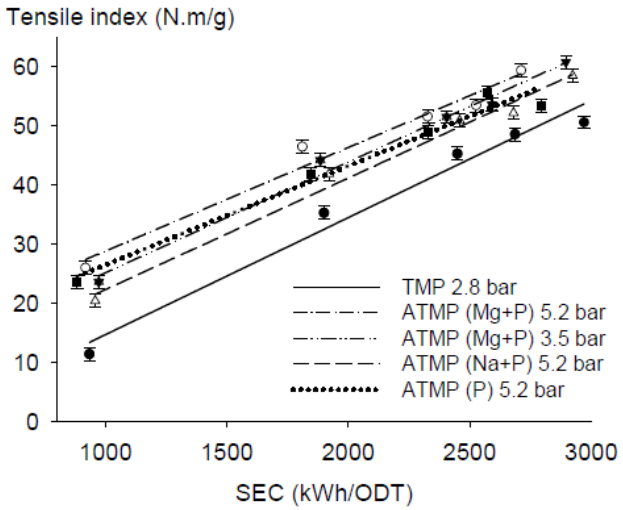

Fig 10. Tensile index as a function of specific energy demand.

Table 6. 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 2.8 bar	1.78 ± 0.03	Reference
ATMP (Mg+P) 3.5 bar	1.25 ± 0.02	- 0.53 (30)
ATMP (Mg+P) 5.2 bar	1.13 ± 0.02	- 0.65 (37)
ATMP (Na+P) 5.2 bar	1.40 ± 0.02	- 0.38 (21)
ATMP (P) 5.2 bar	1.21 ± 0.02	- 0.57 (32)
At 50 N.m/g	MWh/odt	MWh/odt (%)
TMP 2.8 bar	2.79 ± 0.04	Reference
ATMP (Mg+P) 3.5 bar	2.35 ± 0.03	- 0.44 (16)
ATMP (Mg+P) 5.2 bar	2.20 ± 0.03	- 0.59 (21)
ATMP (Na+P) 5.2 bar	2.46 ± 0.04	- 0.33 (12)
ATMP (P) 5.2 bar	2.40 ± 0.04	- 0.39 (14)

TMP at tensile index 30 Nm/g. It was earlier established that produced ATMP is of the same character as TMP and fractional composition was a part of the analysis. This indicates that the increased strength of ATMP compared to TMP at equal refining energy did not come from, for example, an increased content of fines but was rather a result of a more energy-efficient refining action. This could also be concluded from the fibre characterisation. More energy-efficient development of external and internal fibrillation in ATMP refining seems to have resulted in better strength properties of laboratory sheets and can be used to achieve a reduction in refining energy demand – target properties can be obtained at lower SEC using ATMP concept. The difference in energy efficiency between TMP and ATMP was detected already after the first stage refining and was maintained throughout the second and third stages. Thus, it seems that it was the addition of chemicals in the first stage refining together with increased refining intensity and not the chemical reactions during the storage time

between first and second stages that caused the reduction in energy demand.

When the same chemical strategy was combined with refining at lower pressure (3.5 bars), electrical energy demand in refining to reach tensile index 30 N.m/g was decreased with 0.53 MWh/odt or 30%. The energy efficiency seemed to be slightly lower when casing pressure was decreased. There can be a number of explanations to this. One is that the residence time of pulp in the refiner decreases with decreased casing pressure (Miles et al. 1991). Another is that lower temperature at lower casing pressure could decrease the efficiency of chemical reactions inside the refiner. At lower casing pressure, the steam volume inside the refiner is higher resulting in a larger gap since refiner was run under hydraulic force control during the trials. The findings are consistent with earlier published results (Muhić 2010).

Addition of peroxide alone or peroxide combined with sodium hydroxide into the first stage refiner in ATMP process resulted in energy reduction with 0.38-0.57 MWh/odt or 21-32%. Since no high-intensity TMP reference with mechanical pre-treatment was manufactured during this trial, there is a possibility that chemicals did not contribute to the energy efficiency at all in this case. Reduction of energy demand of this magnitude is comparable with what was earlier achieved using compressive pre-treatment and high-intensity refining without the addition of chemicals (Kure et al. 1999, Sabourin 2003, Sabourin et al. 2003).

There are different mechanisms that could contribute to the energy reduction in ATMP refining compared to the TMP. One of those mechanisms is associated with the separation of defibration and fibre development and the use of higher intensity segments in the first refining stage.

Mechanical pre-treatment of chips prior to refining has been earlier shown to reduce the refining energy demand. Energy reduction in the order of magnitude of 0.2 MWh/odt or 10% is normally reported and the pre-treatment is reported to give better results when applied to pine compared to spruce. Improvement of energy efficiency in refining is proposed to be caused by introduction of cracks in S1 and S2 fibre walls which make subsequent defibration of chips easier. Some of the contribution could also come from the removal of extractives from chips which has previously been proposed to reduce the energy demand in refining (Johansson 2000, Reme, Helle 2001, Heum 2005, Svensson 2007). This subject was recently covered in a review (Gorski et al. 2009).

Increased intensity in refining has previously been reported to increase the energy efficiency. This probably happens due to a more energy-efficient straining of lignin component of the fibre wall (Goring 1963, Höglund et al. 1976, Salmén et al 1985, 1997, Sabourin et al. 2003) which dissipates energy in a less efficient way if frequency of the impacts is increased (Booker, Sell 1998, Corson 2001). Elevated refining intensity by means of increasing the rotational speed has been proposed earlier (Miles et al. 1991). Energy reduction by approximately 0.3-0.4 MWh/odt or 15% has been reported for the RTS process where the intensity of refining was increased by elevating the rotational speed of the refiner from 1800 to 2300 rpm compared to conventional TMP (Muenster, Dahlqvist 1995, Sabourin et al. 1996, Martin et al. 2000, Sabourin 2001). The use of high-intensity refiner segments has been shown to decrease the energy consumption in refining by 0.1-0.4 MWh/odt or 10-20% compared to low-intensity segments (Huhtanen et al. 2004, Muhić 2010). Changing refining mode from holdback (used for refining of the TMP reference in this study) to expel (used for refining of ATMP) was reported to cause the residence time in the refiner, measured using cross correlation calculations of high frequency pressure sensors, to decrease from approximately 3500 msec to 1000 msec (Senger et al. 2006). Thus specific energy per impact increased 3-5 times. Experiments were carried out in the same refiner using same segment pattern as in this study. The higher refining casing pressure used for ATMP refining in this study would increase the residence time somewhat.

Results from using increased refining intensity by means of elevated refiner speed and feeding refiner segments have also been described together with using mechanical pre-treatment (Sabourin et al. 2001). The pre-treatment was though only carried out in an Impressafiner unit compared to the ATMP process where both an Impressafiner and a Fiberizer are used allowing for complete chip defibration prior to first stage refining. Reduction of energy demand for mixed north-eastern softwood species was 0.7 MWh/odt compared to 1.2 MWh/odt which was shown to be possible using the ATMP process (Johansson et al. 2011).

Clearly, there is a large contribution from mechanical pre-treatment and higher intensity in the first stage refining in the ATMP concept. However, this cannot explain the energy reduction between different ATMP series in this trial. The only difference between ATMP (Mg+P) and ATMP (Na+P) is that different chemicals were used; the pre-treatment and intensity were the same. The difference in the energy consumption between those two series was 0.27 MWh/odt. Even larger difference was observed between ATMP series with

different chemicals when pine was used as raw material (Johansson et al. 2011). The only plausible explanation is that there is a contribution of chemicals to the energy reduction in ATMP refining and it is in the magnitude of at least approximately 0.3 MWh/odt for spruce raw material.

When casing pressure was increased from 3.5 to 5.2 bars, a higher energy-efficiency could be achieved (energy reduction compared to TMP increased from 0.53 MWh/odt or 30 % to 0.65 MWh/odt or 37 %). The influence of refining pressure and temperature was recently investigated in mill scale (Muhić et al. 2010). Results showed energy reduction by 80-150 kWh/odt measured at equal tensile index when pressure was increased from 4.6 to 7.1 bars. Energy reduction obtained in this study (160 kWh/odt) was in the same order of magnitude. A number of mechanisms explaining the energy reduction were proposed. Changed fibre residence time inside the refiner at lower gap caused by lower steam volume could be one explanation. A possibility is also that reduced steam volume lead to less turbulence inside the gap (Huhtanen 2004).

The chemical mechanism of enhanced fibre development contributing to higher tensile index is probably the reactivity of wood towards chemicals used in ATMP refining. Wood fibres consist of a blend of ionic polymers and their stiffness and glass transition temperature govern the mechanical properties such as their swelling behaviour and, ultimately, how they respond to the refining action (Hammar et al. 1995). The softening and swelling behaviour of lignin is considered to determine the softening behaviour of wood and wood fibres (Franzén 1986, Hammar et al. 1995). This is because lignin has a glass transition temperature which is strongly influenced by the presence of carboxylic and sulphonic groups while the glass transition temperatures of hemicellulose and cellulose are far outside the temperature range used in TMP refining (Salmén, Ljunggren 1994, Hammar et al. 1995). Higher amounts of charge caused by higher amounts of carboxylic groups in lignin increase water uptake and consequently the swelling of the material (Salmén, Berthold 1997). Fibre swelling and degradation is probably what contributes to the enhanced fibre development upon refining in the ATMP process.

Increased ionic content of sulphonic and/or carboxylic acid groups leads to softening of lignin component of the fibres (Salmén 1995). It is well-known that introduction of those groups into wood chips leads to softening of the middle lamellae regions and a "CTMP-like" defibration pattern. Once the CTMP fingerprint (i.e. separation of fibres close to the middle lamellae) is set, the pulps are difficult to use in printing paper production due to decreased opacity (Franzén 1986, Fineman 1986). The ATMP process allows to perform defibration at "TMP-like" conditions, separating fibres in the S1 and S2 fibre walls with creation of enough fine and middle fraction material (Hill et al. 2010, Johansson et al. 2011) while the fibre development can be enhanced by the softening action of chemicals added after the initial defibration of chips. These fibres remain to be of TMP character when further refined.

An attempt to separate the effects of chemical treatment from the effect of increased refining intensity and mechanical pre-treatment should be made in the future. This can be done by including an ATMP reference without addition of any process chemicals in the study

Optical properties of handsheets

Fig 11 shows that ATMP had similar light scattering compared to TMP at equal tensile index. Light scattering of the sheets was discussed earlier during the assessment of character of the pulps because this property was important for evaluation of the defibration mechanisms during refining. Typical result of chemical pre-treatment of wood chips is that light scattering decreases at similar tensile index (Atack et al. 1980).

Fig 12 shows that brightness increased by approximately 14 ISO% when the ATMP concept utilizing hydrogen peroxide and alkali source was used. The increase was slightly higher when pressure in the first stage was increased. This suggests that positive effect of increased per-hydroxyl anion formation due to increased temperature was more dominant than thermal darkening reactions known to happen in pulp when the temperature is increased. There was no significant difference in brightness between ATMP where magnesium hydroxide was used in combination with lower pressure in the first stage and ATMP where sodium hydroxide was used as alkali source. Use of hydrogen peroxide alone gave about half of the brightness increase obtained using alkali sources, which is somewhat surprising since traditionnally very insignificant bleaching response is obtained if no alkali source is used. A possible reason for this is that the temperature in a refiner is much higher than is traditionally used during bleaching. It is generally accepted that hydroxide ion concentration, found under conventional bleaching conditions at pH of around 11, is needed to optimize the perhydroxyl ion equilibrium and thus the bleaching effect of hydrogen peroxide. Perhydroxyl anion is the active species in chromophores elimination (Andrews, Singh 1979, Pan et al. 1994).

However, it has also been shown that the formation of perhydroxyl anion is enhanced at elevated temperatures due to a shift in hydroxide ion equilibrium towards the hydroxide ion formation (Presley, Hill 1996, Rämö 2003). In other words, hydroxide ion formation is increased when the temperature is increased thus also increasing the formation of perhydroxyl anion, the active bleaching species. Our results suggest that enough perhydroxyl anions were formed to achieve at least a partial bleaching effect even if no additional source of hydroxide anions was present in a refining system.

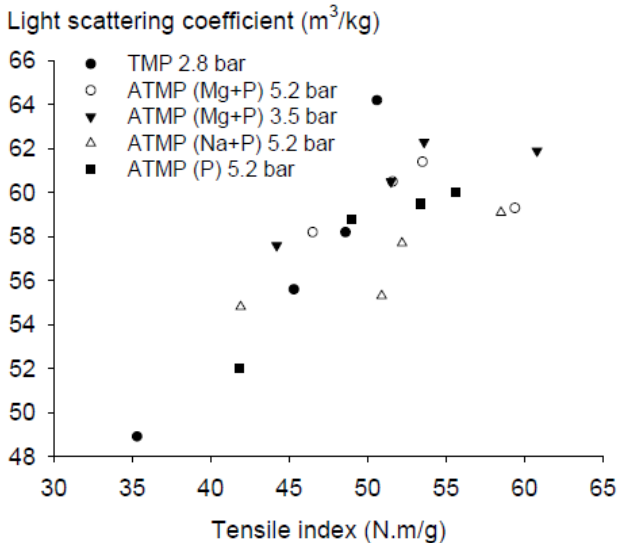

Fig 11. Light scattering coefficient of handsheets made of TMP and ATMP (R^2=0.81 and P=7.8*10^{-10}).

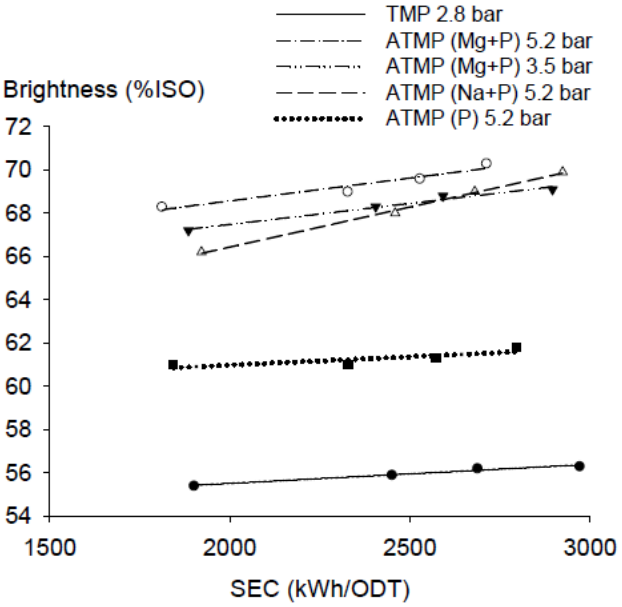

Fig 12. ISO brightness of pulps.

It is important to distinguish between the effects of alkali and hydrogen peroxide on fibres. Alkali merely ionizes the native carboxylic acid groups already present in the hemicellulose. It also deacetylates hemicellulose (galactoglucomannan) and demethylates pectins (Sjöström 1989, Pranovich et al. 2003, Konn 2006, Saarimaa 2007). Hydrogen peroxide, on the other hand, is responsible for introducing new carboxylic acid groups in lignin through oxidation (Thornton et al. 1993, Pranovich et al. 1994, Sundberg 1999) and it seems also to be able to defragment the lignin network leading to lower degree of cross-linking (Gierer et al. 1993, Gierer et al. 1994). This leads to some dissolution of low-molecular lignin compounds, but the effect of hydrogen peroxide is still considered to be of lignin-preserving nature and non-delignifying character (Chang, Allan 1971, Gierer, Imsgard 1977, Gratzl 1986, 1987).

Hydrogen peroxide bleaching is associated with yield loss and generation of COD which is important when considering an alternative process for TMP manufacturing. This matter needs to be addressed and a separate article on the bleaching performance of the ATMP process will be published by the authors. A fair comparison of ATMP process would be to a TMP process where pulp is bleached to reach equal brightness levels. This would also generate certain amount of COD and yield loss and an investigation is needed to study if that amount would be different between ATMP and bleached TMP.

Conclusions

- Electrical energy demand at equal tensile index was improved by approximately 0.65 MWh/odt compared to TMP using an ATMP concept on White spruce with unoptimized chemical system. Contribution from the added chemicals was at least 0.3 MWh/odt.
- ATMP and TMP had similar character and all pulp and paper properties important for printing paper production were on the same level or better with ATMP concept compared to TMP at similar tensile index.
- All ATMP pulps had very low content of shives already in the first stage compared to the TMP reference. Pulp brightness increased with 14 ISO % for ATMP manufactured with addition of magnesium hydroxide and hydrogen peroxide.
- ATMP process was shown to enhance the development of fibres through increased flexibility, specific surface area and proportion of split fibres at similar total energy demand.

Acknowledgements

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