

Improvement of energy efficiency in TMP refining by selective wood disintegration and targeted application of chemicals

Lars Johansson, Jan Hill, Dmitri Gorski and Patrik Axelsson

KEYWORDS: ATMP, TMP, Energy reduction, Hydrogen peroxide, Magnesium hydroxide, Sodium bisulphite, Refiner bleaching.

SUMMARY: A pilot refining trial on Loblolly pine (*Pinus taeda*) was conducted using a novel process configuration called ATMP (Advanced Thermo-mechanical Pulp). In this process, selective wood disintegration due to mechanical pre-treatment of chips and increased refining intensity is combined with targeted application of chemicals after defibration. Standard TMP was compared to ATMP where different chemical strategies were applied. These strategies employed active chemicals known to cause sulphonation, carboxylation/oxidation and degradation of fibre components in order to assist the refining process.

Main goal of the study was to evaluate the potential of decreasing the energy demand in refining evaluated at equal tensile index compared to a TMP reference. Another goal was to produce pulp where the TMP character, i.e. good optical properties combined with good bulk and strength properties, was fully preserved. The objective was also to study other quality aspects of the pulp produced using the ATMP process and compare these to properties of conventional TMP.

Energy demand in refining was decreased with at least 0.6 MWh/odt compared to TMP at tensile index 25 Nm/g when ATMP concept was used. Maximum achieved reduction in the refining energy demand was 1.1 MWh/odt (42%) compared to the TMP reference at tensile index 25 Nm/g. Statistical analysis showed that pulp, produced using the ATMP process, retained all its important properties such as light scattering, density and elongation on the same level as control TMP, i.e. it had the same character. All pulps produced using the ATMP process had very low shive content. Treatment with hydrogen peroxide and magnesium hydroxide was the most successful chemical strategy tested during the pilot trial. It was most successful both in respect to improved optical properties (14 ISO % increase in brightness could be achieved by adding 25 kg/odt hydrogen peroxide in the first stage refiner) and reduction of energy demand in refining.

ADDRESSES OF THE AUTHORS: **Lars Johansson** (lars.johansson@pfi.no): Paper and Fibre Research Institute (PFI) AS, NO-7491, Trondheim, NORWAY. **Jan Hill** (jan@qtab.se): QualTech AB (previously nsiFOCUS), SE-282 21, Tyringe, SWEDEN. **Dmitri Gorski** (dmitri.gorski@norskeskog.com): nsiFOCUS, Norske Skog ASA, NO-1756, Halden, NORWAY and Mid Sweden University, Fibre Science and Communication Network (FSCN), SE-85170, Sundsvall, SWEDEN. **Patrik Axelsson** (patrik.axelsson@norskeskog.com): Norske Skog Saugbrugs, NO-1756, Halden, NORWAY.

Corresponding author: Dmitri Gorski

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.

Refining of pine raw material is known for its high energy demand and poor resulting pulp quality compared to spruce. Pine typically requires 30% more energy in refining than spruce (Brill, Strand 1985, Sabourin et al. 1996, Reme 2000, Yuan et al. 2006). It is also more difficult to bleach (Yuan et al. 2006). These differences have been suggested to be caused by higher amount of extractives in pine (Reme 2000) and morphology of pine fibres (Yuan et al. 2006, Fernando, Daniel 2008). Reduction of energy demand in refining of pine species is a prerogative for their successful use as raw material in mechanical pulping in the future.

ATMP process offers a possibility to reduce the energy demand in refining (Hill et al. 2009). Values in excess of 0.8 MWh/odt or 25% have been reported (Hill et al. 2009, 2010). Process outline includes defibrating pre-treatment in Impressafiner and Fiberizer as well as elevated first stage refining intensity by means of using more aggressive feeding segments and higher rotational refiner speed. These process modifications, together with the addition of chemicals in the first stage refiner after pre-treatment, result in improvement of energy efficiency in refining and quality of pulp.

Mechanical pre-treatment in an Impressafiner was previously reported to reduce the energy demand in subsequent refining by approximately 0.1-0.2 MWh/odt or 5-15% (Gorski et al. 2009). Potential

for energy reduction seems to be greater for pine raw material (Robertson et al. 2001) where such values as 0.2 MWh/odt (8%) for a mix of spruce and pine (Sabourin 2000, 2003) and 0.35 MWh/odt (14%) for Loblolly pine (Sabourin 1998) were reported. The results were also confirmed in mill scale for pine (Sabourin et al. 2002). The reason for reduced energy demand was proposed to be formation of cracks in S1/S2 fibre walls which promote more energy-efficient refining (Sabourin 2000, Kure et al. 1999, Gorski et al. 2009). It is however also possible that the reduction of extractives content in chips, which is observed after Impressafiner pre-treatment, contributes to the energy efficiency in sub-sequent refining (Reme 2000, Tanase et al. 2009, Gorski et al. 2009).

Elevated refining intensity was shown to lead to decreased refining energy demand on both spruce and pine raw materials. One way of increasing refining intensity is to increase the rotational speed of the refiner (most common increase is 300-800 rpm). Typical reduction of energy demand for both pine and spruce is then approximately 0.25-0.40 MWh/odt or 10-20% (Muenster and Dahlqvist 1995, Kure et al. 1999, Sabourin 2000, Sabourin 2003). This was also shown in mill scale (Aregger 1997, Fuglem et al. 2001). Increased rotational speed causes shorter residence time inside the refiner and thus higher intensity. It was earlier reported that specific energy per impact increased from 0.2 to 0.3 kJ/kg/impact when rotational speed of the refiner was increased from 1800 to 2300 rpm. This corresponds to a decrease in residence time from 3.5 to 2.9 seconds. The study was conducted using the same segment model and pilot refiner as in the present study (Senger et al. 2006). More aggressive refiner segment pattern is another way of increasing the intensity. Here, reduction of energy demand by 0.1-0.5 MWh/odt or 10-20% was reported (Huhtanen et al. 2004, Muhić 2010). The mechanism was proposed to be smaller refining gap causing higher intensity. Specific energy per impact was reported to increase from 0.3 to 0.6-1.0 kJ/kg/impact when plate pattern was changed from holdback to expel. This corresponds to a decrease in residence time from 2.9 to 1.0 seconds (Senger et al. 2006). Improved energy efficiency due to elevated refining intensity was proposed to be the result of more energy-efficient straining of lignin component of the fibre wall with less elastic and more plastic deformation (Goring 1963, Höglund 1976, Salmén et al. 1985, 1997, Sabourin et al. 2003). This leads to less dissipation of energy as heat (Booker, Sell 1998, Corson 2001).

Elevated refining intensity combined with mechanical pre-treatment in Impressafiner was

reported to reduce refining energy demand by approximately 0.3-0.8 MWh/odt or 10-30% (Kure et al. 1999, Sabourin 2000, Johansson and Dahlqvist 2001, Sabourin et al. 2003). When Impressafiner pre-treatment was used and rotational speed of the refiner was increased from 1800 to 2600 rpm, reduction in energy demand by 0.2-0.3 MWh/odt or 16-17% was reported (Sabourin 2001b, Sabourin et al. 2003). It can be concluded that the two methods of reducing the energy demand (pre-treatment of chips in Impressafiner and elevated intensity in first stage refining) seem to be rather additive. Indeed, even some synergetic effects were reported. For example, mechanical pre-treatment was suggested to make the chips more susceptible to harsher conditions at high intensity thus leading to less fibre shortening and higher tear index (Sabourin 1998, Kure et al. 1999, Sabourin et al. 2002).

Further opportunities were reported if a Fiberizer unit was added to the mechanical pre-treatment. Fiberizer is essentially a small moderately pressurized refiner where defibration of pre-treated chips into fibre bundles takes place (Sabourin et al. 2003, Hill et al. 2009, 2010). Energy demand in subsequent refining was reported to be reduced by up to 0.8 MWh/odt or 30% when a combination of Impressafiner and Fiberizer pre-treatment was used prior to first stage refining at elevated intensity. Comparison was made to a TMP reference. Reduction in the energy demand was 0.35 MWh/odt or 15% when compared to a TMP with Impressafiner pre-treatment. It is known that high intensity refining leads to smaller refining gap. Thus, a uniform feed is crucial to achieve stable and effective refiner operation. This could be one of the reasons for increased energy efficiency when pre-treatment is expanded with a Fiberizer unit in addition to an Impressafiner. Another possibility that opens up is addition of chemicals to coarse fibre bundles (material after the Fiberizer) instead of wood chips or macerated wood chips after Impressafiner. Fiberized material has much larger surface for reaction and fracture planes between the fibres are to a large degree already formed (Sabourin et al. 2003, Hill et al. 2009, 2010).

Adding chemicals to the refining process could be another way of reducing the energy demand. Application of chemical strategies in order to achieve energy reduction has been attempted many times in the past. There are several such possibilities in a traditional refining process:

- Chips can be impregnated with chemicals (Beath and Mihelich 1977, Axelsson, Simonson 1983, Bohn, Sferrazza 1989, Argyropoulos, Heitner 1991, Stationwala 1994, Richardson, Murton 2008)

- Chemicals can be added to chip refiners (Ryberg 1978, Hartler 1980, Richardson 1996, Richardson et al. 2001, Harrison et al. 2008)
- Chemicals can be added to pulp refiners subsequent to the first stage refining (including reject refiners) (Gummerus, Bhabanisankar 1986, Gorski et al. 2010)
- Pulp can be impregnated in an inter-stage treatment (Barnet et al. 1980, Franzén 1983, Strunk et al. 1989, Nurminen and Sundholm 1995, Bian et al. 2007, Bian et al. 2008, Walter 2009)

Most of the energy demand in a paper mill today is concentrated to the main line refiners – some mills even utilize only one stage in their pulp production. Hence, it is favourable to implement chemical strategy as early in the process as possible if a reduction in overall energy demand in refining is the goal. Ideally, it should be applied before or in the first stage refining where most of the electrical energy often is consumed. The challenge, associated with the use of chemicals in refining of TMP, intended for production of printing paper, has always been the negative effect that chemical softening has on the defibration of chips. Chemical softening leads to defibration in the middle lamellae; it changes the character of the TMP pulp which becomes more “CTMP-like” and less suitable for production of printing paper where high opacity is crucial (Barnet et al. 1980, Franzén 1983, Fineman 1986, Janknecht et al. 1990, Yuan et al. 2006).

Substantial reduction of energy demand seems to be possible if chemicals are added to the process after mechanical pre-treatment stage consisting of Impressafiner and Fiberizer (Sabourin et al. 2003; Hill et al. 2009, 2010). When mechanical pre-treatment is used prior to the addition of chemicals, it seems to be possible to avoid the earlier described negative effects that chemicals have on defibration of chips. This is because chip defibration is to a large degree completed before chemicals are added and it may be possible to selectively enhance the fibre development process.

If energy reduction values reported to be possible to achieve using different combinations of mechanical pre-treatment and intensity are added up, they sometimes can exceed 50-60%. However, the above mentioned methods do not seem to be entirely additive since no trials where energy demand was reduced by more than 0.7-0.8 MWh/odt or 30% have been reported (Sabourin 1998, Kure et al. 1999, Johansson and Dahlqvist 2001, Sabourin et al. 2003). A lot of experiments were performed on spruce since spruce-like wood species are by far the most common to be used in mechanical pulp production. It seems though that mechanical pre-

treatment and refining at increased intensity yield best results when applied on pine raw material. Pine ultrastructure could be one of the causes of such difference (Fernando, Daniel 2008).

This study explores the advantages of combining the above mentioned mechanical modifications of the refining process with selective addition of chemicals in refining of Loblolly pine. A combination of full mechanical pre-treatment (Impressafiner and Fiberizer) and increased first stage refining intensity (both by means of increased rotational speed and more aggressive segment pattern) is used in the trials. Chemicals in the ATMP process are applied to defibrated but not developed fibres after mechanical pre-treatment. The objective is to establish if ATMP process, used on pine raw material, can potentially allow production of pulp with TMP character and significantly lower electrical energy demand. Some of the tested chemicals, for example hydrogen peroxide, may also offer additional quality improvements such as pulp brightness increase. CTMP, where chemicals are used for chip impregnation, is known for its low shive content (Åkerlund and Jackson 1984). If chemicals can be successfully used in ATMP process without altering the character of the pulp, it could be possible to achieve decreased shive content as well.

Materials and Methods

The pilot plant

A pilot study consisting of five refining trials was conducted at Andritz pilot plant in Springfield, Ohio, United States. Loblolly Pine (*Pinus taeda*) wood from Tennessee, USA, was used for all trials. Refining was conducted in three stages; third stage was run at three energy levels. In ATMP refining, chips were pre-compressed in an Impressafiner and defibrated further in a Fiberizer prior to the first stage refining which was run at elevated rotational speed (2300 rpm compared to 1800 rpm for TMP). Duramet 36604 segments were used in Fiberizer and first stage refiner. Optimized operation conditions in Impressafiner and Fiberizer were determined in earlier studies (Sabourin 2000, Sabourin et al. 2003). For equipment set-up in each trial see *Fig 1*. All chemicals except the chelating agent (DTPA) were charged to the primary stage refiner (through the dilution water added in the inner ring), see *Table 1*. Acetic acid was added together with acetate buffer to provide same pH as bisulphite. With bisulphite, pH of 4.4 was reached while pH of 4.8 and 4.7 was reached for acetic acid and acetic acid with hydrogen peroxide respectively. DTPA (0.3%) was charged at the Impressafiner outlet where the pressure was released. All charges are

given on oven dry weight of pulp. Pulp was stored in drums between the refining stages with retention time varying between 30 and 120 minutes. Unfortunately, the refining system was not equipped with accurate enough disk gap measurements and this information is not available. Refiner variables are given in *Table 2*. Casing temperatures were calculated from casing pressures, using saturated steam tables and do not reflect the pressure profile along the refiner segments but can rather be used for comparison. Higher local temperatures are achieved in the refiner gap.

Production was kept constant throughout the trials and refining energy demand was calculated as an average, based on motor power demand, bulk density and moisture content of the material. Measurement of the production rate is relatively inaccurate in mill scale; errors of up to 10% are not uncommon. However, production rate could be measured with much higher accuracy during the pilot trials. Error analysis in the measurement of production rate, and thus also specific energy demand, was performed (*Appendix 1*) and the error was established to be approximately 1.5%.

The ATMP concept

A mechanical pulping concept called ATMP (Advanced Thermomechanical Pulp) was used in four out of the five trials described in this paper. Configuration of the ATMP process consists of two extra units, i.e. Impressafiner and Fiberizer. Mechanical pre-treatment of chips in a high-

Table 1. Pulps and chemicals in the pilot trial (pH was 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	5.8
ATMP (B)	NaHSO ₃ 1.7%	4.4
ATMP (AA)	AcOOH 4.0%	4.8
ATMP (Mg+P)	Mg(OH) ₂ 1.5% + H ₂ O ₂ 2.5%	7.0
ATMP (AA+P)	AcOOH 4.2% + H ₂ O ₂ 2.2%	4.7

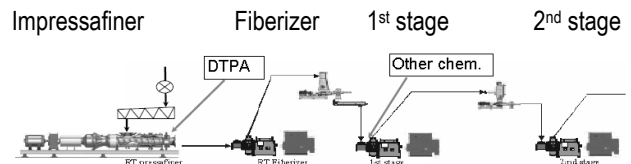


Fig 1. Pilot plant refiner configuration for ATMP production (no mechanical pre-treatment or chemicals were used for TMP).

compression screw (Impressafiner) followed by defibration of chips to fibres and fibre bundles in a Fiberizer unit is used prior to primary stage refining of the fiberized material. Chemicals are charged into the primary refiner with dilution water after defibration, *Fig 2*. Approximately 150-250 kWh/odt were applied to achieve chip defibration during the mechanical pre-treatment. The reactions with chemicals, added in the primary refiner, were directed to fibre surfaces, exposed during the pre-treatment.

Table 2. Refining variables during the pilot trial

	Impressafiner*	Fiberizer SD 36-1CP	1st stage refiner SD 36-1CP	2nd stage refiner SD 36-1CP	3rd stage refiner DD 401
TMP trials					
Preheating time (s)	Not used	Not used	150	0	0
Speed (rpm)	-	-	1800	1800	1200
Casing pressure (bars)	-	-	2.8	2.8	Atm.
Casing temperature (°C)**	-	-	142	142	Atm.
Segment pattern	-	-	Hb***	Hb***	-
ATMP trials					
Preheating time (s)	15	10	10-15	0	0
Speed (rpm)	38	1800	2300	1800	1200
Casing pressure (bars)	1.5	1.7	5.8	2.8	Atm.
Casing temperature (°C)**	-	130	164	142	Atm.
Segment pattern	-	Ex***	Ex***	Hb***	-

* 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)

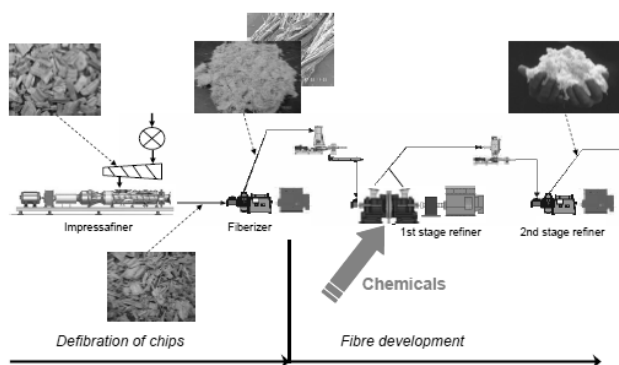


Fig 2. Combination of selective wood disintegration and targeted addition of chemicals in the ATMP process.

Physical testing

Pulp from Springfield was transported to Halden, Norway, and put in cold storage until testing. CSF (Canadian Standard Freeness) was measured according to ISO 5267:2:2001 method. Fibre length was determined optically, using PQM1000 (Sunds Defibrator). Amount of shives was measured using a 0.10 mm slotted Pulmac screen.

Standard laboratory sheets with recirculated white water were manufactured according to ISO 5269:2:1998. 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 are presented, where possible, with error bars, denoting 95% confidence interval.

Results and discussion

Refiner operation

Specific energy demand in the pilot trial refining is given in Table 3. The corresponding Canadian Standard Freeness (CSF) values, measured on pulp after each refining stage, can be found in brackets. Loblolly pine is extremely difficult to refine (Leask 1987, Burkett, Tapio1990, Harris 1993, Sabourin et al. 2001, Yuan et al. 2006, Yuan et al. 2009) and attempts to produce standard TMP with the same low freeness as ATMP at maintained fibre length and strength properties failed. Energy demand to produce pulp with required quality is discussed further under “energy efficiency and handsheet strength”.

Pulp character

TMP process became dominant for production of wood containing printing paper due to the fact that TMP pulp has good strength properties combined with high light scattering ability which provides good opacity even at low basis weight (Höglund, Wilhelmsson1993, Heikkurinen et al. 2009). It is thus essential to preserve this character of TMP

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

	Imp.	Fib.	1 st	2 nd	3 rd stage
TMP	-	-	0.72 (740)	1.35 (640)	2.09 (310) 2.37 (170) 2.61 (165)
ATMP (B)	0.04	0.22	0.69 (750)	1.55 (400)	2.38 (140) 2.62 (100) 2.81 (80)
ATMP (AA)	0.04	0.24	0.55 (750)	1.11 (510)	1.89 (200) 2.07 (150) 2.18 (110)
ATMP (Mg+P)	0.04	0.25	0.60 (740)	1.35 (210)	2.19 (90) 2.44 (70) 2.67 (50)
ATMP (AA+P)	0.02	0.22	0.55 (750)	1.12 (470)	1.73 (155) 1.90 (120) 2.13 (70)

when trying to reduce the energy demand in refining. In this work, energy demand was evaluated at equal tensile index (the dependent variable) since the tensile index is normally the target variable for newsprint paper machine operation. Pulps, produced in this study, are considered to be of similar character if other important properties change in similar way with increasing tensile index (i.e. increasing energy input in refining). If, for example, the statistical analysis shows that there is no significant difference in light scattering coefficient compared at equal tensile index for ATMP and TMP, the authors conclude that ATMP and TMP have similar character of the light scattering index development. In practice it means that light scattering is preserved in ATMP compared to TMP while a reduction of electrical energy demand is achieved. Statistical methods used (analysis of variations and principal component analysis) are described in more detail in Appendix 2.

To establish the character of produced ATMP, eight important properties of the pulps were chosen, see Table 4. Their development was compared at equal tensile index for ATMP together with the TMP reference. The properties were not independent – a change in the size of fines fraction would for example lead to a change in average fibre length. Each of the properties was assessed using analysis of variations (ANOVA). P-values were calculated using ANOVA analysis to determine the significance of the results. A P-value of 0.01 means that there is a 1% of variation in the independent variable that did not correlate with the variation in the dependent variable (tensile index) for all pulps. Since a confidence interval of 95% was used to determine the significance, P-values had to be lower than 0.05 for the difference in character to be considered insignificant.

Table 4. Coefficients of determination of linear regressions for three energy levels of all five pulps, TMP and ATMP (15 data points are plotted together and compared at equal tensile index)

Property	Unit	P-value
Density	kg/m ³	3.4*10 ⁻⁸
Light scattering coefficient	m ² /kg	2.6*10 ⁻⁴
Z-strength	kPa	7.7*10 ⁻⁸
Canadian Standard Freeness	ml	7.7*10 ⁻⁸
Fibre length (length weighted)	mm	1.2*10 ⁻²
BMN P200 fraction (weight)	%	2.6*10 ⁻²
Elongation	%	8.8*10 ⁻⁵
Tensile energy absorption	J/m ²	5.4*10 ⁻⁹

There was no significant difference in any of the pulp properties shown in Table 4. Low P-values show that over 97-99% of variation in the studied properties correlate with the changes in the tensile index. The fact that ATMP and TMP have similar fractional compositions is probably what causes the preserved light scattering ability, illuminating the importance of conducting the defibration of chips prior to the addition of chemicals if the TMP character is to be preserved. Reduction of the amount of fines and middle fraction has almost always been reported for pulps made from impregnated chips (Argyropoulos, Heitner 1991, Stationwala 1994) witnessing about CTMP-like defibration which is unfavourable for printing papers.

A multivariate analysis (PCA, Principal Component Analysis) was also performed and confirmed the results of the ANOVA. Using the same pulp properties, a score plot and a loading plot were created, see Figs 3 and 4. Score plot in Fig 3 shows that there is some clustering between the different pulp batches, but the overall picture is that all pulps have approximately the same character while most of the variations come from increasing the specific energy demand. The level of variation can be described as low also due to the high R² and Q² values of the plot. In the loading plot, Fig 4, it can be seen that all studied pulps (both TMP and ATMP) behave very similarly and show negative correlation of approximately similar magnitude between strength properties, middle fraction and fines content on one hand and fibre length, long fibre content, tear index and CSF on the other hand. This is logical, since an increase in strength properties (caused by refining) leads to decrease in CSF, tear index, long fibre content and fibre length. High R² of the model serves as another indication of the unchanged character of ATMP compared to TMP, which are both included in the same multivariate analysis. Small variation in the data, indicated by high R², implies that values of properties of all pulps in this analysis (both TMP and ATMP) undergo similar change in relation to each other.

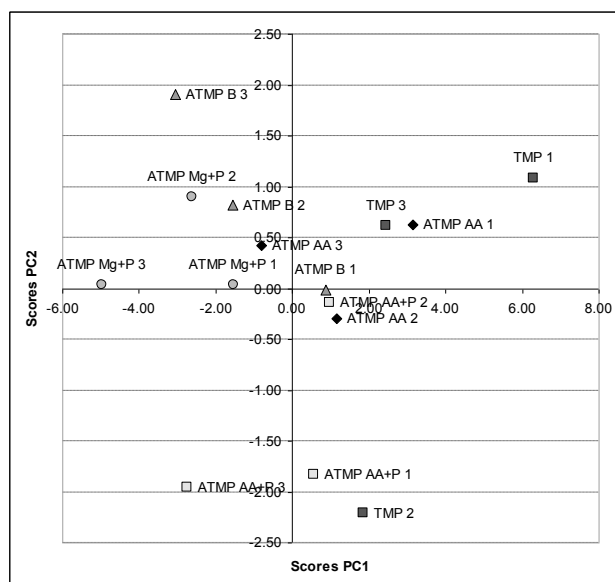


Fig 3. Score plot of the pulp properties, R²(cum) = 0.79, Q²(cum) = 0.51

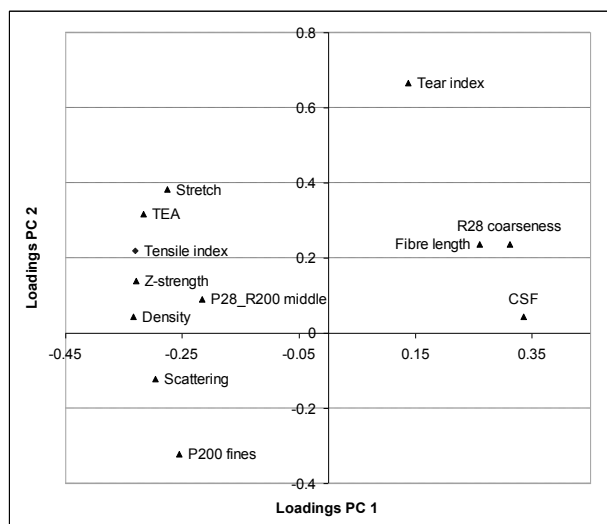


Fig 4. Loading plot of the pulp properties. R²(cum) = 0.79, Q²(cum) = 0.51

It is proposed that the character of ATMP pulps was preserved due to the choice of the addition point for chemicals to the refining process (after the defibration of chips already was completed). It seems to be an advantage to use as little energy as possible for chip defibration, i.e. in the Impressafiner and Fiberizer. This is because addition of chemicals to already refined low-freeness pulp is conflicting with the objective of reducing overall energy demand, since most of the energy is consumed in the main line refining. It has been early concluded that this approach would not give a significant improvement in energy-efficiency of the TMP process since the advantages are limited when starting the chemical treatment on already refined pulp (Chang et al. 1979). It is also known

that chip defibration requires very little energy while most of it is converted in fibre development.

Impregnation of whole wood chips poses a severe transport problem – ideally, we want only to impregnate the region of fibre walls where fracture will lead to favourable defibration and development of desired fibre surface and fibre fractions. This favourable surface for TMP fibres is considered to be S1/S2 where it is easiest to promote external fibrillation and fines can be generated efficiently (Heikkurinen 1993). However, the largest concentration of reactive groups in fibres is mostly in hemicelluloses in the outer fibre walls (Fardim et al. 2002, Ek et al. 2009 and Konn 2006), lignin (Zhang et al. 1994, Ek et al. 2009) and pectin and protein (Ek et al. 2009, Konn 2006, Stevanic and Salmén 2007). Most of these compounds are located in the middle lamellae and primary cell wall of the fibres, the concentration of lignin for example decreases drastically in S1 compared to S2 fibre wall (Panshin and DeZeeuw 1970). Carboxylation, sulphonation and other reactions which induce softening, swelling and breaking of bonds in fibres happen mostly in the outer parts resulting in weakening of the middle lamellae and primary wall structure (Hammar et al. 1995, Stevanic and Salmén 2007). Hence, attempted whole chip impregnation usually results in unfavourable defibration in or closer to the highly reactive middle lamellae or primary wall. This impacts the quality of the pulp negatively from the point of view of printing paper production; more Asplund-like fibres with a “CTMP fingerprint” are produced (Franzén 1986, Cisneros et al. 1992, Richardson 1998, Salmén et al. 1999, Koljonen et al. 2001). Pulp, produced using the ATMP process, has TMP character and retains it also when further refined. The reason for this is probably that the unfavourable chip defibration is avoided even when chemicals are used to soften the fibre structure in the ATMP process, see Fig 5.

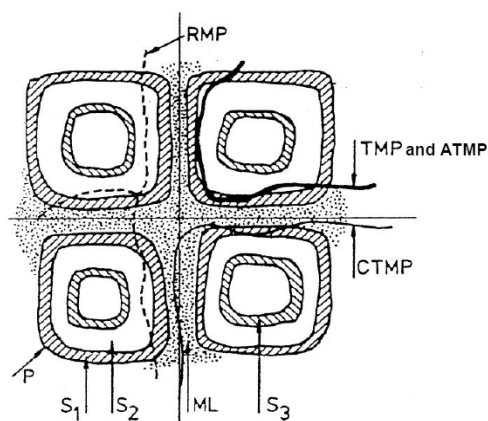


Fig 5. Fracture zones in wood chips during different chemical and mechanical treatments (modified from Htun, Salmén 1996, originally from Franzén 1986)

Fig 6 shows that all ATMP pulps contain less shives than the TMP reference, which suggests a more efficient fibre separation. This could be due to the elevated intensity and pressure or the chemical treatment. Reduction of shives using compressive pre-treatment and elevated intensity in refining has been reported before (Stationwala et al. 1994, Kure et al. 1999, Sabourin et al. 2003). Elevated pressure and temperature was previously reported to reduce shives content in refining due to easier chip defibration when the temperature is elevated above glass transition point of lignin (Nurminen 1999, Muhic et al. 2010). Shives have been named a common cause for, among other, web breaks (Höglund et al. 1976, Gregersen 1998). Keeping in mind that all ATMP series were refined using the same process layout and intensity, it seems that magnesium hydroxide in combination with hydrogen peroxide was the most effective in decreasing the shives content. Acetic acid seems to be less effective than the combination of acetic acid and peroxide. In CTMP, where chemicals are used to impregnate chips, the amount of shives is normally much lower compared to TMP (Åkerlund and Jackson 1984). This is probably a result of a more efficient fibre separation when the middle lamellae lignin is softened by chemicals and it could also be the mechanism of shive elimination during the production of ATMP. Effects of elevated intensity and addition of chemicals on shive reduction could be separated in the future if ATMP trials without the addition of chemicals are performed.

In Fig 7 it can be seen that there is no significant difference in tear index of TMP and ATMP compared at similar tensile index. Shapes of the curves are typical for tear-tensile plots and pass through a maximum. Long fibre content is consi-

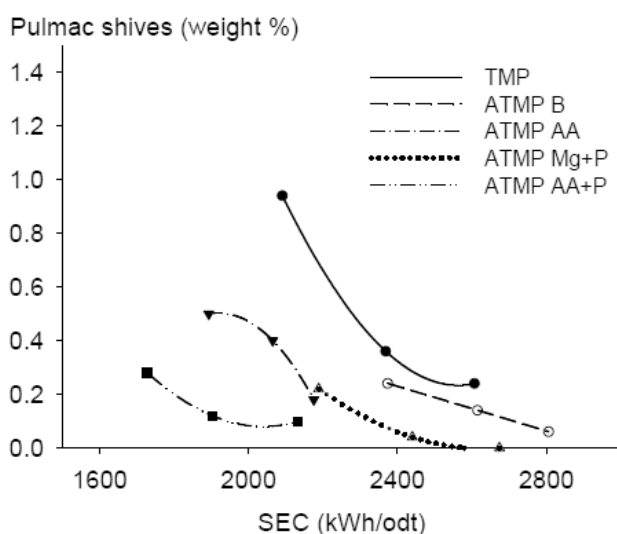


Fig 6. Shives content of the pulps (Pulmac 0.10 mm screen, weight %).

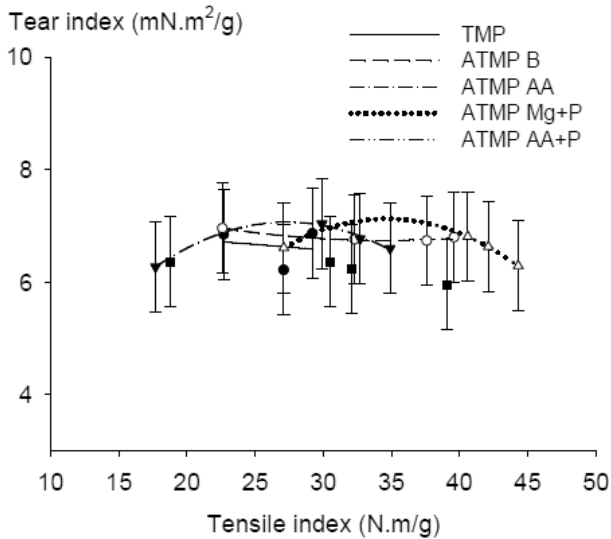


Fig 7. Relationship between the tear and tensile indexes of handsheets.

dered to be important for tear index and since statistical analysis earlier showed that there is no difference in long fibre content between TMP and ATMP, the results can be considered consistent.

Energy efficiency and handsheet strength

Tensile index of handsheets is plotted against electrical energy demand in refining in Fig 8. All ATMP pulps have lower energy demand when compared at equal interpolated tensile index, see Table 5. ATMP with addition of hydrogen peroxide and magnesium hydroxide shows highest energy reduction of 1.1 MWh/odt or 42%.

Reduction of energy demand by 0.4-0.5 MWh/odt using the same process configuration as in the current study, but without the addition of chemicals, was reported earlier (Sabourin et al. 2003). All ATMP trials in this study were performed using the same equipment configuration and refining conditions. Addition of bisulphite gave lowest reduction in the energy demand of 0.6 MWh/odt, which is consistent with earlier findings (Hill et al. 2009). Hence, it can be suggested that the additional reduction of energy demand by 0.5 MWh/odt probably comes from the chemicals, added to the first stage refiner. Two high intensity stages (high-intensity single disk primary refiner and double disk secondary refiner) were used in ATMP refining compared to the TMP reference, where only on high intensity refining stage was used (double disk secondary refiner). However, it was earlier shown, that only the use of high intensity in first stage refining has considerable impact on the energy efficiency and development of pulp properties in refining (Falk et al. 1987, Johansson, Dahlvist 2001). Second stage refining at high and low intensity were shown to have approximately similar effect.

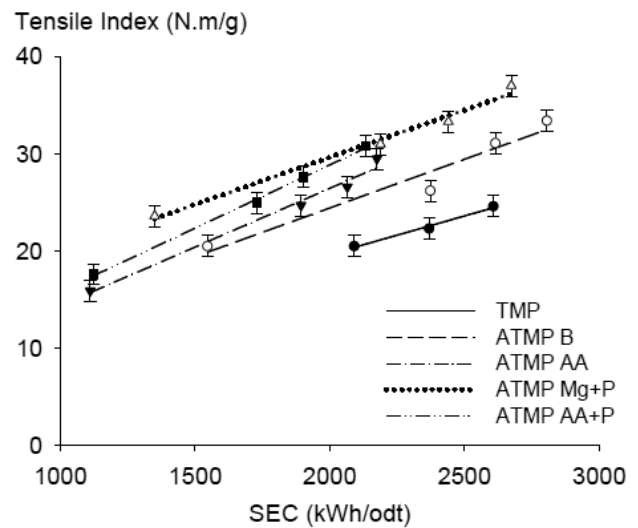


Fig 8. Tensile index as a function of specific energy demand.

Table 5. Electrical energy demand in refining at equal tensile index (25 N.m/g).

Pulp	Energy demand	Reduction
At 30 N.m/g	MWh/odt	MWh/odt (%)
TMP	2.65±0.04	Reference
ATMP (B)	2.05±0.03	- 0.6 (23)
ATMP (AA)	1.85±0.03	- 0.8 (30)
ATMP (Mg+P)	1.55±0.02	- 1.1 (42)
ATMP (AA+P)	1.75±0.03	- 0.9 (34)

Unexpectedly, addition of acetic acid gave a significant reduction in the energy demand by 0.8 MWh/odt (0.9 MWh/odt in combination with hydrogen peroxide). This is difficult to explain since no charge-inducing and swelling reactions are known to happen in fibres due to the presence of acetic acid. Results also suggest that the tensile index develops faster in second and third refining stages for ATMP, produced with addition of acetic acid – slopes of both curves (with and without peroxide) are steeper. At higher specific energy demand, addition of acetic acid and hydrogen peroxide seems to be almost as effective as addition of magnesium hydroxide and hydrogen peroxide. This is not easily explained. Pulp where acetic acid was added had rather low pH (4.7-4.8) and it is reasonable to expect that fibres were in their proton form. This alone should, however, not have led to improved energy efficiency since it was shown earlier that fibres are more swollen in their sodium form (Engstrand et al. 1991). Another explanation could be the ability of acetic acid with acetate buffer to influence the content of calcium ions in fibres. Oxalic acid pre-treatment was earlier shown to reduce the energy demand in refining somewhat (Swaney et al. 2003). This was explained by the removal of calcium ions from the fibres by oxalic acid, which makes the fibres more suitable for

refining. Acetic acid was shown to be able to dissolve calcium carbonate (Ciccone 2003) and thus a similar effect as after treatment with oxalic acid could be present.

It can also be argued that acetic acid did not have any effect on the energy reduction at all, but is was rather the RTF pre-treatment and increased refining intensity that that contributed to the energy efficiency in refining of ATMP (AA) and ATMP (AA+P). However, it has never been shown that this combination of mechanical pre-treatment and high intensity refining can reduce the energy demand by as much as 0.8-0.9 MWh/odt – values of 0.4-0.5 MWh/odt were reported (Sabourin et al. 2003, Sabourin 2003). In addition, ATMP with bisulphite addition was earlier shown to be more energy-efficient than reference TMP, also when exactly similar equipment configuration was used. Thus, it is not very plausible that all of the reduction in energy demand in the trials where acetic acid was used was attributed to the refining conditions. More trials on how ATMP refining influences fibre development will be made in the near future and an explanation to the action of acetic acid may come up later. Unfortunately, no good explanation can be offered for the time being.

The slopes of the other three curves (TMP, ATMP B and ATMP Mg+P) in *Fig 8* did not change much in relation to each other throughout the refining process. It could mean that most of the effect that the chemicals have on pulp is realized already in the first stage refiner and the reduction in energy demand originates there. This could be explained in several ways.

- It is in the first stage refiner that the outer, lignin-rich and reactive, regions of the fibre walls are stripped off the fibres. Chemicals probably react mostly with the outer parts of the fibres and hence it is the removal and development of those which should be affected most
- Efficiency of the refining action depends on the condition of the material inside the refiner, but also on the refiner itself. Coefficient of friction is by far highest in the first stage refiner (Isaksson et al. 1997, Illikainen et al. 2007) and it can be suggested that maximum effect can be gained from the addition of chemicals in the first stage

It could however also be that the chemicals, applied to the fibres in the first stage refiner, reacted during the storage between refining stages and this led to reduction of energy demand in the second stage. This was supported by freeness measurements, *Table 3*, where it can be seen that

pulps after the second refining stage seem to have had larger difference in CSF values compared to the first stage pulps. Pulps from the first refining stage were too coarse for handsheet manufacture. This needs to be investigated in future studies.

Statistical analysis revealed linear relationships between the tensile index and the density of the sheets for both TMP and ATMP. Densification of a paper sheet can occur through a number of mechanisms:

- Change in fractional composition (for example a larger fines fraction and shorter average fibre length) can lead to densification of the structure due to smaller particle size. Increased amount of fines in sheets is known to lead to increased strength properties (Moss and Retulainen 1995)
- More conformable and collapsed fibres can be drawn closer to each other in the process of sheet formation and drying with a larger bonded area as a consequence. This causes a denser structure but also lower light scattering (since fewer surfaces are available in the sheet) and elongation

As shown earlier, the fractional composition of ATMP develops similar to the TMP compared at equal tensile index. The higher degree of densification of ATMP is thus probably a result of pulp properties, developed during refining. Relationship between the light scattering and tensile index is fairly linear for TMP and ATMP sheets, which also suggests that the sheet densification mechanisms are similar in both TMP and ATMP refining. More conformable fibres, softened by induced charge and drawn closer to each other, would increase the density of the sheet. However, it would also lead to decreased light scattering coefficient since there would be more bonded surface. Higher degree of fibrillation was earlier found to affect the light scattering abilities of paper sheets (Reme et al. 1998) and it is thus possible that loss of light scattering due to higher bonded area is compensated for by increased fibrillation (larger surface of the unbonded parts of the fibres compensate for loss of light scattering surface due to larger bonded area).

The three major objectives of mechanical pulping are (Atack 1981):

- to reduce wood to its constituent fibres
- to retain the integrity of a considerable fraction of these fibres
- to induce maximum amount of flexibility and fibrillation into the separated fibres and fine fibre fragments

These objectives have been later confirmed by a number of authors (Koran 1981, Kano et al. 1982, Marton, Eskelinen 1982, Lammi, Heikkurinen 1997, Corson 2001). Since the fractional composition of TMP and ATMP is similar compared at similar tensile index (as shown by the statistical analysis) but the specific energy required to reach a certain tensile index is much lower in the ATMP process (Fig 7), it must mean that fibre fractions develop in a more energy-efficient manner. Apart from the particle size distribution, both the flexibility and the external fibrillation of fibres influence densification behaviour of paper sheets – more flexible fibres can easier form bonds with each other and increased external fibrillation leads to increased bond strength. In the future, fibre properties of pulps, produced using the ATMP process, should be studied in more detail. Mechanisms of tensile index development seem to be similar in sheets made of ATMP and TMP.

It was earlier proposed that only two independent pulp variables are needed for characterization of mechanical pulps, the long fibre content and the specific surface of the fibres (Forgacs 1963). During refining, fibre walls are consequently stripped off and fibres are made more flexible with larger and more fibrillated surface (Kure 1997). Higher intensity in refining leads to more efficient peeling of the fibre walls (Kure et al. 1999). It is thus reasonable to propose that some of the mechanisms, that govern the development of fibre properties described above, are enhanced by the presence of chemicals in ATMP refining. Chemical reactions in wood fibre structure such as carboxylation, sulphonation and oxidative degradation influence the softening behaviour of the fibres (Iwamida et al. 1980, Hartler 1980, Östberg, Salmen 1988, Olander et al. 1990, Heitner, Salmén 1994, Salmén 1995, Hammar et al. 1995, Konn 2006) and could lead to a more efficient development of fibre properties in ATMP compared to TMP refining. Addition of chemicals in the refining process was also shown to influence such important property as the coefficient of friction inside the refiner (Engstrand et al. 1995, Hill et al. 2010) which in its turn was proposed to have a strong influence on the energy efficiency (Remé 2000, Svensson 2007, Gorski et al. 2010). How the increased softening of the material or decreased friction contributes to a more energy-efficient development of pulp properties is not entirely understood. One possibility is that the amount of elastic deformation in the softer material is decreased and instead, more plastic deformations are achieved. This would increase the energy efficiency in refining according to previous results (Koran 1981). However, the coefficient of friction is

challenging to measure. Its calculation can be attempted in the future, when better measurement of refiner variables such as axial thrust, gap and so on is possible. Another possibility is that removal of outer parts of the softened fibres and their external and internal fibrillation can proceed in a more-energy efficient way due to weakening of the material as a result of swelling. Individual fibre properties should be studied in the future to better understand the mechanisms behind the energy efficiency in ATMP refining.

To investigate and isolate the roles of chemical addition and refining conditions (higher intensity and mechanical pre-treatment), a thorough characterisation of all contributing mechanisms should be carried out in the future. In this article, the overall effects of the chemical strategies on pulp and paper properties are described and the most promising strategy is chosen for further investigation. It is also important to point out that the trials in this study are performed in pilot scale in a batch process – no optimization of the chemical recipes is performed since this can only be done online in a process, where step changes in chemical dosage are possible.

It has been shown in many cases that the amount of energy converted in fibre separation and development constitutes only a small fraction of the total energy demand in refining. Recent advances in the modelling of the refining process have showed that entropy-related conversion of the refining energy (i.e. refining energy, used to induce changes in the material) is less than 5% of the total energy applied, while the enthalpy-related part (i.e. energy converted to heat) is over 95% (Karlström et al. 2008, Eriksson, Karlström 2009). This suggests, that even if entropy-related energy demand would be eliminated (i.e. fibre development would be achieved without using any energy at all), a reduction of total energy demand with 42% would not be possible without a substantial reduction in the enthalpy-related energy conversion. In fact, most of the energy reduction, achieved in the ATMP process, is probably due to minimized conversion of energy to heat. This could have been achieved due to several different mechanisms. Minimizing elastic deformation of the material (which leads to heat generation not inducing any change in the material) and maximizing the plastic deformation (which induce a change in the material, i.e. the development of fibre properties) could be one of the mechanisms. The above mentioned effect on the coefficient of friction due to the addition of process chemicals could be another one. These effects are challenging to study, but it can be done if enough information about the variables in the refining system is recorded accurately in the future.

Optical properties of handsheets

Brightness of handsheets can be seen in *Fig 9*. Addition of bisulphite increased the brightness of the pulp by approximately 4 ISO%. Addition of acetic acid decreased the brightness by approximately 10 ISO%. Small part of the decrease could be counteracted by charging hydrogen peroxide together with the acetic acid. Addition of hydrogen peroxide and magnesium hydroxide increased brightness by approximately 14 ISO%. The brightness increase, achieved by adding magnesium hydroxide and hydrogen peroxide, is consistent with mill scale results of refiner bleaching with same chemical system reported earlier (Gorski et al. 2010).

Light scattering index of ATMP was earlier in this study shown to be similar to that of TMP using multivariate analysis and other statistical tools (see section where the character of the pulps is discussed). *Fig 10* shows light scattering of the pulps produced in this trial as a function of tensile index of the sheets. No loss of light scattering, typical for CTMP, is evident for sheets produced from ATMP.

Since magnesium hydroxide was used in ATMP (Mg+P), there is a possibility that it was present in the pulp and later also paper sheets in form of crystals, which could potentially have led to an increase in light scattering. However, the statistical analysis and results in *Fig 10* clearly show that there was no difference in light scattering measured at equal tensile index for ATMP produced using bisulphite or acetic acid either. The fact that light scattering is preserved at the same level as in TMP for all ATMP pulps suggests that it is rather the defibration mechanism and not a higher content of

magnesium hydroxide in some of the sheets that preserved light scattering coefficient. This should, however, be investigated further. Another possibility is that the refining at higher intensity, used in the ATMP process, led to higher light scattering but the addition of chemicals lowered it back to the reference TMP level. An ATMP reference run without addition of chemicals would help to answer this question.

Chemical system that reduced energy demand in refining of the ATMP (Mg+P) was also an efficient bleaching agent. This offers new possibilities for a more efficient process layout since the need to bleach refined pulp can be reduced or even eliminated altogether. With an optimized chemical system it may be possible that even better bleaching results may be achieved. 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. Naturally, a fair comparison of ATMP process would be to a TMP process where pulp has been bleached to reach equal brightness levels as described in this study. 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.

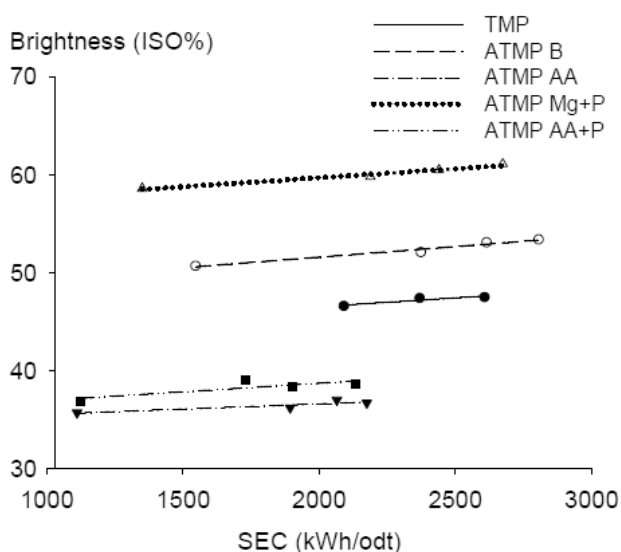


Fig 9. ISO brightness of pulps

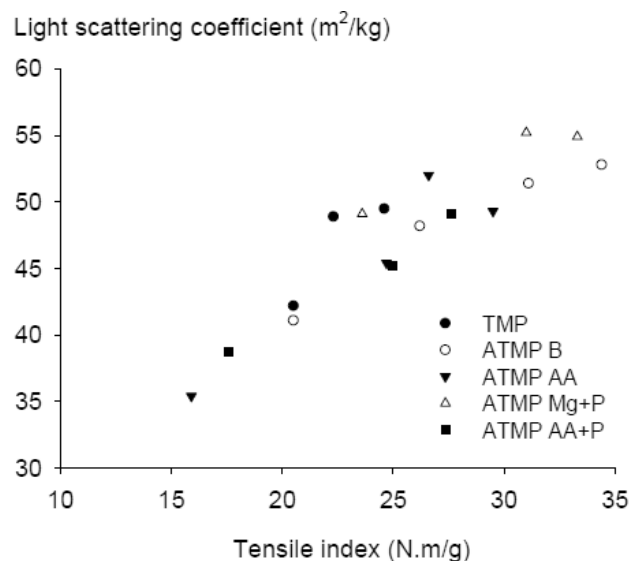


Fig 10. Light scattering coefficient of handsheets made of TMP and ATMP ($R^2=0.81$ and $P=7.8 \cdot 10^{-10}$).

Conclusions

- Using the ATMP concept, it was possible to produce pulp which required a significantly lower specific energy application than TMP to reach equal tensile index. The reduction of the energy demand ranged from 0.6 to 1.1 MWh/odt and was caused by a combination of altered refining conditions and addition of chemicals.
- All pulps produced using the ATMP concept had very low shive content.
- Results show that pulps, produced using ATMP process, have the same basic character as TMP in all aspects. Preserved light scattering at lower electrical energy demand makes ATMP process applicable in production of paper, where optical properties play major role and where CTMP traditionally can not be used (e.g. magazine paper and newsprint)
- ATMP, produced using hydrogen peroxide and magnesium hydroxide, had approximately 14 ISO% higher brightness compared to the TMP reference

Appendix 1

Experimental error in the calculation of the specific energy demand

Specific energy demand in the pilot trials was calculated according to *Equation 1*.

$$SEC = \frac{\text{power} \times \text{time}}{\text{production}} \quad [1]$$

The numerator consists of the logged motor power multiplied by the time it took to produce a batch of pulp during the trial (typically 15-20 minutes). Gauge error in the measurement of power is approximately 1% (sampling frequency is 1 Hz throughout the trial and the signal is completely digital) and it is an independent variable. Time can be assumed to be measured accurately. Production calculation is based on the dry weight of a raw material sample. Prior to sampling, a careful homogenization of the raw material is carried out to minimize the sampling error. Chips, incoming to the pilot plant, are mixed, soaked in water and drained before the measurement is performed. This procedure is repeated after Impressafiner and Fiberizer runs. Thus, the sampling error can be approximated with the error in the determination of dry contents (independent variable), typically around 0.5%. Determination of the production error consists of two terms. The other one is the volumetric error in the amount of chips, delivered by the transport screw into the refiner. This error can also be considered to be an independent

variable. The screw is powered by an Allen-Bradley Bulletin 1557 variable frequency AC drive which operates with an accuracy of approximately 1% (according to specification). Thus, the error in calculated energy demand depends on the error propagation which can be described by *Eq 2* where x , y and z are the error terms.

Since the *Eq 1* consists only of multiplication and division, the *Eq 2* can be rewritten as *Eq 3*. Using the *Eq 3*, it is possible to calculate errors in the determination of specific energy demand used in *Table 5*, see *Table 6*.

$$\Delta SEC = \sqrt{\left(\frac{\partial SEC}{\partial x} \times \Delta x\right)^2 + \left(\frac{\partial SEC}{\partial y} \times \Delta y\right)^2 + \left(\frac{\partial SEC}{\partial z} \times \Delta z\right)^2} \quad [2]$$

$$\frac{\Delta SEC}{SEC} = \sqrt{\left(\frac{\Delta x}{x}\right)^2 + \left(\frac{\Delta y}{y}\right)^2 + \left(\frac{\Delta z}{z}\right)^2} \quad [3]$$

Table 6. Experimental errors in the determination of energy demand calculated at equal tensile index (interpolated)

Pulp	Energy demand	Error
At 25 N.m/g	MWh/odt	MWh/odt
TMP	2.65	0.04
ATMP (B)	2.05	0.03
ATMP (AA)	1.85	0.03
ATMP (Mg+P)	1.55	0.02
ATMP (AA+P)	1.75	0.03

The calculated error in the energy demand for the equipment, used in this study, was 1.5%.

Appendix 2

Two statistical methods were used in this study to evaluate the character of produced pulp. Character here was defined as the ability to preserve all important pulp qualities at similar level for ATMP compared to control TMP, evaluated at equal target property (i.e. tensile index). Statistical methods, used here, are fairly standard and are well described elsewhere (Box et al. 2005).

ANOVA

ANOVA is an abbreviation for "Analysis of Variations" and is an algorithm which can be used to determine if there are any statistically significant differences between development values in a given data set. In this study, it is used as a basis for statistical analysis in order to show if the development of produced ATMP has different character (i.e. differs significantly) compared to a control TMP. Character of the pulp was in this study defined by 8 important properties listed in *Table 4*. Those properties were evaluated using tensile index as evaluation criteria (i.e. if ATMP and TMP had significant difference in one of the properties when

compared at equal tensile index, it would be shown in ANOVA). Tensile index was used as evaluation criteria since it was used to evaluate the energy demand in refining of the pulp. Two-way ANOVA analysis built into the Excel software was used in this study.

Data sets obtained in refining trials were relatively small and comparison could normally be made by just looking at the graphic plots. Nevertheless, a proper statistical analysis with ANOVA helped to obtain more conclusive results. Since only one treatment was evaluated in this study (application of ATMP process instead of conventional TMP process), one-way ANOVA analysis could be considered. However, several energy levels were used for refining and thus a two-way analysis was necessary since the pulps were not refined applying exactly equal amounts of energy. P-value, the outcome of ANOVA, indicates how certain it is that all values belong to the same dataset (cluster). P-value of 0.03 indicates that there is only a 3% chance that there is a statistical difference between the development of values in a data set. Since 95% confidence interval is considered to be enough, P-value must be smaller than 0.05 for the differences to be considered insignificant. ANOVA analysis is often used in other disciplines to prove significant difference of data within a data set (Moen et al. 2010).

PCA

PCA, or Principal Component Analysis, is a way to reduce the dimensions of the data. Pre-processed data is projected onto a line forming the first principal component. The second component is derived from the data left after extraction of the first component. The components are orthogonal to each other and are, thus, independent. Score plot represents the samples and load plot, the derived decomposition. Ideally, the score and load matrixes contain information on all systematic variations in the data and all the noise is contained in the residual matrix. In this study, PCA was used to confirm if the data used for ANOVA form any distinct clusters which would suggest that there was a significant difference between pulps contained in those clusters. Thus, if ATMP and TMP samples would form distinct clusters in score and loading plots, this could be interpreted as statistical difference between them. Instead, they form random pattern in both cases. PCA is described in more detail elsewhere (Wold 1987, Martens and Naes 1993, Jackson 1991).

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