

## Review

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# Aspects on bond strength in sheet structures from TMP and CTMP – a review

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**Abstract:** High yield pulps (HYP), manufactured in mechanical and chemimechanical pulping processes, are mainly used in graphic papers and paper grades where a high bulk is preferable, like in paperboards. Moreover, packaging papers with very high demands on both dry and wet strength could be manufactured from HYP in a near future. Preferred bonds between fibre components (long fibres, shortened fibres and fines) in the various paper grades are quite different. In the review, plausible effects of mechanical interlocking, intermolecular interactions (“physical bonding”), hydrogen bonds, intermixing of polymers, additives and possible specific interactions in the formation of strong bonds in sheet structures from HYP are discussed. A required condition for high bond strength in sheets from HYP furnishes is that fibre components are forced into sufficiently close contact. This is to a great extent impeded if the fibre walls are too stiff. Consequently, the current review focuses on both how fibre fractions should preferably be developed for different end uses and how suitable bonds might be achieved in different paper grades. The ideal type of bonds is certainly different depending on the demands on the final paper quality.

**Keywords:** bond strength; graphic papers; mechanical pulps; packaging paper; paper strength.

## Introduction

High yield pulps (HYP), which are manufactured in stone groundwood (SGW), pressure groundwood (PGW), thermomechanical (TMP) and chemithermomechanical (CTMP) processes or variants of these, contain a mixture of wood fibres with their natural length, shortened fibres, fibre fragments and fines from outer parts of the fibre walls. Mechanical pulps (SGW, PGW and TMP) are primarily used in graphic papers (News, SC and LWC) and chemimechanical pulps (CTMP) in paper grades where a high bulk is preferable, like in paperboards. Moreover, recent research has shown that also packaging papers with very high demands on both dry and wet strength could be manufactured from HYP in a near future. The optimal mix of HYP fibre components in these three quite different grades varies a lot, as well as the most suitable type of bonds in their sheet structures. Based on studies of hand-made sheets, the nature of the various bonding mechanisms in these grades is discussed in the present review. The focus is on sheets based on pulps from TMP and CTMP processes, but the mechanisms are usually relevant also for sheets from SGW and PGW furnishes. We use the term “bond” in the sense of the contact zone that holds fibres (or particles) together on a micrometre scale, where at least a fraction of the surfaces are close enough for attractive interactions to give adhesion. Another term is fibre-fibre joint. When we mention different types of physical “bonds”, other interactions or even chemical bonds, we will specify which type is referred to (see Appendix A).

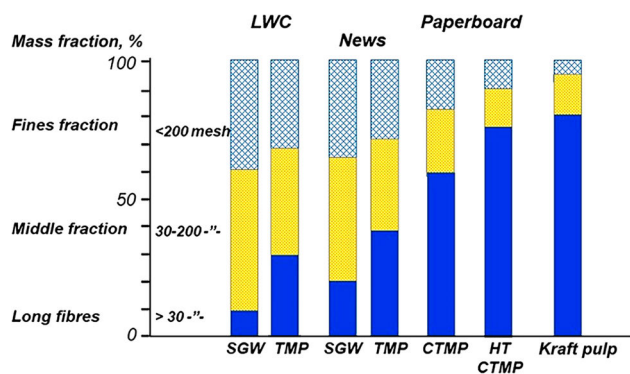
The in-plane strength of sheets from chemical pulp fibres are generally described as depending on fibre strength and length in combination with bond strength and bonded area between fibres (Kallmes and Perez 1966, Page 1969, Giertz 1973). Moreover, sheet formation, stress distribution and residual stresses in the sheets are considered to have a significant influence. An extensive review on the nature of joint (i. e. bond) strength in sheets from chemical pulp fibres was published by Lindström et al. (2005). In this review, references to previous reviews and literature dealing with the subject can be found. The nature of bonds in sheets from HYP is generally not the

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**Figure 1:** Typical composition as size fractions (% weight) from the Bauer-McNett fractionation of HYP from spruce wood for different end uses.

same as in sheets from chemical pulps due to the impact of the remaining lignin and the very heterogeneous nature of HYP fibre materials, Figure 1. The heterogeneity is especially evident in furnishes for manufacturing of graphic papers (Wood and Karnis 1991, Sundholm 1999, Heikkuri and Leskelä 1999, Ferritsius et al. 2009). The effect of fibre characteristics has been reported in many studies over the years (Atack 1972, Karnis 1994, Mohlin 1997, Kure 1999, Reme 2000, Vehniäinen 2008). How the heterogeneity affects strength properties has been dealt with in several theoretical models based on empirical observations with special focus on the strength properties of graphic papers (Forgacs 1963, Mannström 1968, Giertz 1973, Shallhorn and Karnis 1979, Strand et al. 1989, Andersson 1981a, 1981b). Extensive summaries of the literature regarding the different aspects of joints in mechanical pulp sheets can be found, for example, in the doctoral theses from Luukko (1999), Rundlöf et al. (2002), Koljonen (2004), Vainio (2007), Lehtonen (2014) and Reyier Österling (2015).

A necessary requirement for high bond strength in sheets from HYP furnishes is that fibres in the long fibre and middle fractions are forced into sufficiently close contact. This is to a great extent prevented if their fibre walls are too stiff. Consequently, the current review focuses on both how these fractions should preferably be developed for different end uses and how suitable fibre-fibre bonds might be achieved in different paper grades. The ideal type of bonds is certainly different depending on the demands on the final paper quality.

Definitions of technical terms that are used in this review can be found in the enclosed Appendix A. In Appendix B, a short description is given of the alteration of stiffness of wood and wood components (i. e. cellulose, hemicellulose and lignin) in the fibre walls of HYP under different conditions in papermaking.

## Materials and methods

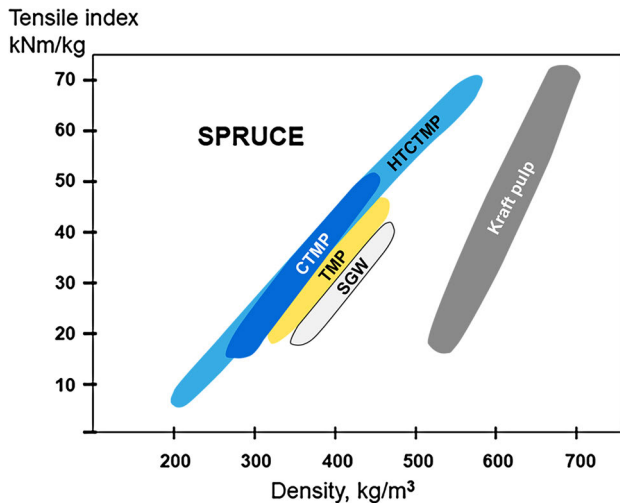
Key information is included in the text, see each reference for details.

## General aspects

### The structure of HYP sheets

Sheets formed from only the stiff long and shortened fibres in a HYP network are weak and bulky (Jackson and Williams 1979, Corson 1979, Mohlin 1979b, Lindholm 1980a, 1981). The density of such sheet structures depends on the shape of the fibres as well as their flexibility, i. e. their conformability. These fibre properties are significantly affected by mechanical treatments in pulp manufacturing processes (Karnis 1994, Mohlin 1997, Corson 1996, Kure 1999, Reme 2000). The strength of a sheet formed of the long and shortened fibres can be improved by wet-pressing in different ways, by adding fines, by fibre modifications or by enhanced press temperatures. The latter is preferably made at temperatures close to or above the lignin softening temperature. All three techniques are utilized, but to different extent to create more and stronger bonds in the three types of papers from HYP furnishes that are in focus in the current review, i. e. graphic paper, paperboard and packaging paper. Pros and cons of these densification techniques in relation to desired sheet characteristics in the different grades will be discussed in separate sections in the following.

Generally, the strength properties of sheets from HYP is related to the density of their structure (Fredriksson and Höglund 1978, Andersson and Mohlin 1980, Andersson 1981a, 1981b, Höglund and Wilhelmsson 1993). Mechanical treatment at refining or grinding is a way to make the fibre material more flexible and to generate fines, which, in combination with densification in papermaking, results in more and stronger fibre-fibre joins in the paper structure. The relation between in-plane tensile strength and density varies between HYP from different processes, Figure 2. However, all types of HYP have a higher sheet bulk (lower sheet density) than sheets from chemical softwood pulps at equal tensile strength. Whether the rupture upon loading of sheets from HYP is just linked to breaks of fibre-fibre related bonds or if it also includes breakage of single fibres is a matter of discussion in the literature (Kallmes and Perez 1966, Shallhorn and Karnis 1979). However, it is generally believed that the strength of fibre-fibre joints is the limiting factor in low bonded sheets, i. e. in newsprint papers and in paperboard, whereas fibres may start to break

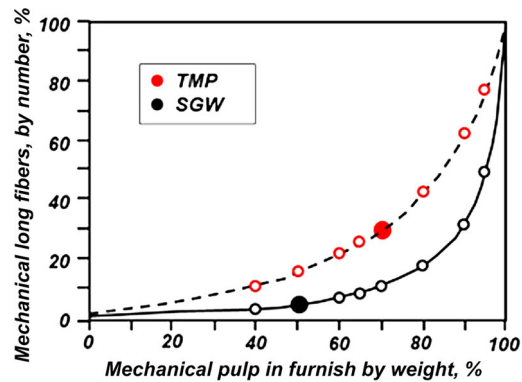


**Figure 2:** Typical differences between HYP and kraft pulp from spruce (ISO sheets).

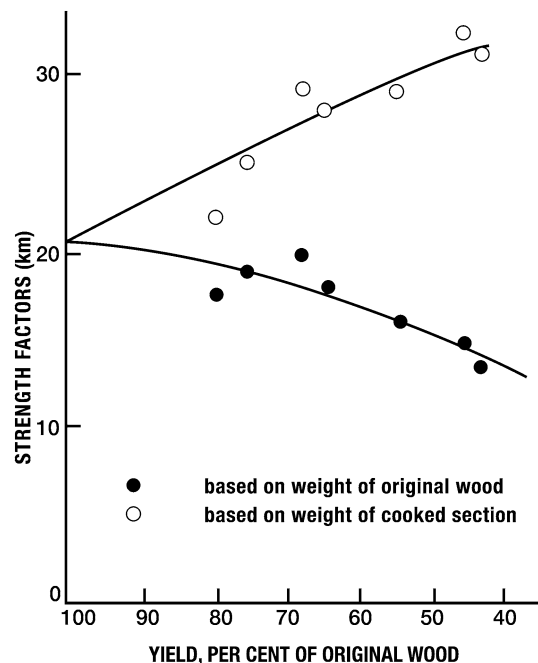
at some loading level in highly densified sheets with strong inter-fibre bonds, i. e. SC/LWC papers and packaging papers. This issue will be discussed in the following sections.

### Influence of single HYP-fibre strength on sheet strength

Fibre strength is usually estimated using two principally different methods. Single fibre measurements by loading a fibre in strain is in theory a simple method, but in practice complicated (McDonough et al. 1987, Tchepel et al. 2001). Therefore, this method is not particularly suitable for such a heterogeneous material as HYP fibres. A more suitable measurement of fibre wall strength on sheets is the zero-span technique. In zero-span tensile testing, a strip of paper is clamped between two jaws separated by a near-zero distance. Problems reported with this technique are that fibres clamped in the jaws can break and that both fibre-fibre bonds and fibre orientation are affected (Clark 1944). Nevertheless, the zero-span method has been used also for the estimation of fibre wall strength of mechanical pulp fibres (van den Akker et al. 1958, El-Hosseiny and Bennett 1985). In general, the literature reports slightly lower zero-span values for TMP sheets than for sheets from chemical pulps (Levlin 1999). This difference may in part be related to the difference in yield, i. e. fewer fibres per weight and the lower numbers of long fibres in TMP compared to that in chemical pulps. The difference in numbers of long fibres in furnishes containing combinations of typical TMP/SGW and a softwood kraft pulp is illustrated in Figure 3 for furnishes for manufacturing of LWC/SC grades.



**Figure 3:** Typical numbers of long fibres, measured using Bauer-McNett fractionation, in furnishes from spruce SGW/TMP and chemical pulps for LWC/SC grades (Wedin et al. 1992).



**Figure 4:** Strength of single fibres from conventional kraft cooked pulp of different yields. Redrawn from Stone and Clayton (1960).

Stone and Clayton (1960) developed an alternative method to estimate fibre wall strength by testing thin microtome sections of earlywood from spruce after kraft cooking to different yields. In the microtome sections, the fibres were naturally orientated in parallel and tested according to the zero-span testing technique. In the evaluation of the data, the zero-span strength was reported both based on the weight of original wood and the cooked sections, Figure 4. Based on the original weight, the strength decreased with decreased yield as expected, whereas it increased based on actual weight after cooking. In refining

in HYP processes, the thickness of fibre walls is reduced when fines from the outer layers are peeled off, which naturally also lowers the strength of a single fibre. This strength reduction could be comparable to the loss of yield in kraft cooking, as the reduction in fibre strength was shown to only be related to the total loss of lignin and hemicellulose in the fibre wall. Even if the strength of single fibres is reduced after peeling off of fine material and changes due to cracks and deformations, the tensile strength of a single HYP fibre would be higher than a single chemical pulp fibre. However, at a given grammage, the number of long fibres in sheets from chemical pulps are always more than double due to the difference in yield. This means that the total strength of all fibres in a sheet from chemical pulp, such as in a zero-span test, will usually be higher than in sheets from HYP.

In summary, it is possible that the lower tensile strength in highly bonded sheets from HYP, compared to sheets from chemical pulps, can be related to the lower percentage of long fibres. Nevertheless, it is even more important that the fibre-fibre bond strength and bonded areas in sheet structures from HYP are sufficiently high. In this respect, it seems to be important to improve fibre conformability and flexibility to achieve a high percentage of bonded area. This will be discussed in the next section.

### Influence of fibre flexibility on sheet density and strength

The flexibility of the long-fibre and middle fraction material is, as already indicated, essential for the final density of a sheet from HYP and the possibility for the fibres to come in sufficiently close contact at sheet consolidation to achieve high fibre-fibre bond strength. The native cross-sectional shape of chemical pulp fibres can easily be transformed to a flatter form in several stages in pulping and papermaking processes, e. g. refining, pressing or drying (Page 1967). The lumen volume is usually considerably reduced, and often the lumen is eliminated. This tendency of chemical pulp fibre to collapse strongly affects the sheet structure and its physical properties. The degree of collapse is dependent on pulp yield – the highest yield giving the lowest collapse. Fibre collapse in HYP fibres occurs preferentially in fibres with thin walls (Kure 1997).

The collapse of the cross-sectional shape is more difficult to achieve on HYP fibres, as most of the original stiff lignin is retained in the fibre walls. This means that these fibres preserve much of the natural stiffness of the wood. Permanent softening of lignin in fibre walls by chemical treatments, such as sulfonation, decreases fibre

stiffness (Heitner and Hattula 1988). Also other chemical treatments, like peroxide bleaching, might make it easier to partially collapse HYP fibres. Thermal softening has a more reversible effect on wall stiffness as long as the temperature is restricted to temperatures below the softening temperature of lignin (Irvine 1985).

In addition to reduction of wall stiffness, bending stiffness can be reduced by making the fibres thinner in accordance with the well-known equation

$$\text{Bending Stiffness} = E \times I \quad (1)$$

Where  $E$  = Elastic Modulus and  $I$  is the Moment of Inertia. Assuming that the material properties ( $E$ ) are approximately constant through the wall thickness of a fibre, the cross-section property that governs the bending stiffness is the area moment of inertia  $I$ . For a schematic circular fibre

$$I = \pi(r^4 - (r - t)^4)/4 \quad (2)$$

where  $r$  is the radius and  $t$  the wall thickness (Vesterlind and Höglund 2005a, 2005b). For a fully collapsed cross section (where it has been assumed that the wall thickness is small compared to  $r$ ),  $I$  is given by;

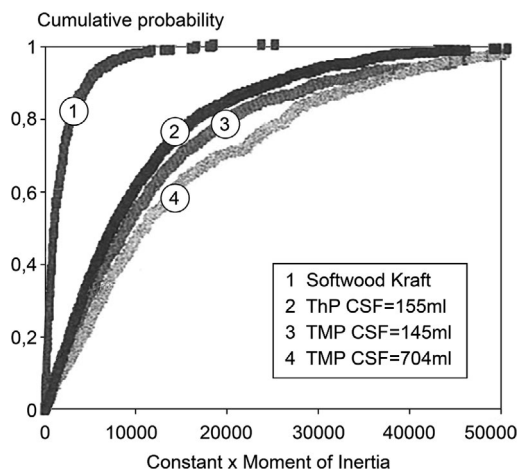
$$I = \pi r t^3 / 6 \quad (3)$$

The cross-section collapse load of a fibre ( $C_r$ ) can be estimated to be proportional to

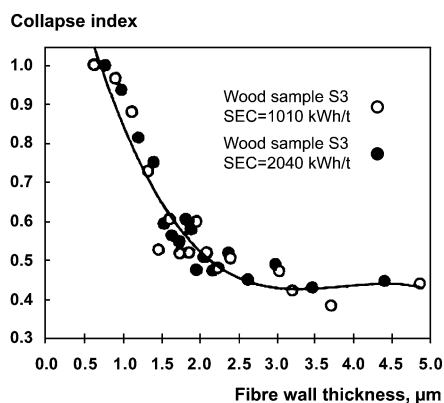
$$C_r = t^2 / (2r - t) \quad (4)$$

It should be emphasized that this estimation is based on a simplified model and the underlying assumptions might be discussed, but the model is easy to use for approximations. It shows that reduction of wall thickness in refining and grinding has a great impact on fibre flexibility and collapsibility.

Cumulative distribution curves of moment of inertia for selected pulps are shown in Figure 5, (Höglund et al. 1997). The figure is based on data from measurements in an image analysis system at SCA Research on microscopy of specimens from cross-sections of dried spruce fibres contained in the Bauer-McNett R30-mesh long-fibre fractions. The figure illustrates the significant difference in bending stiffness between fibres from softwood kraft and spruce TMP processes. TMP-fibres that have been refined to a lower freeness (higher energy input), have somewhat lower stiffness than long-fibre that are manufactured at higher freeness at much lower input of energy in refining, as more fine materials are peeled off from the outer fibre layers at higher energy input. The difference might be related both to the difference in fibre-wall thickness and in-



**Figure 5:** Bending stiffness of TMP and kraft pulp fibres (Höglund et al. 1997).



**Figure 6:** Collapse Index of spruce TMP long fibres refined at two different energy inputs (Kure 1997).

ternal cracks in the wall structure. The ThP pulp has been manufactured at higher temperature levels at preheating and defibration in the chip refining process, which might explain the reduction in the modulus. Similar differences between chemical pulp and TMP long fibre properties, as is shown in Figure 5, has also been reported in more detailed studies by Jang and Seth (1998) and Jang et al. (2001).

The degree of fibre collapse after certain mechanical treatment is of course quite different for fibres from various parts in the wood stem. Effects of wood properties on mechanical pulp properties have been studied by Tyrväinen (1995) and Corson (1997) among others. According to Kure (1997), the fibre collapse is strongly related to fibre wall thickness regardless of energy input in refining, Figure 6. This means that thick-walled latewood fibres preserve most of their shape and stiffness from the wood even after refining, whereas the shape of earlywood fibres is more easily changed. The tendency to collapse might also

be related to cracks in the fibre walls. However, cracks and fibre splitting in fibre walls are primarily frequent in earlywoods with low wall thickness (Reme et al. 1999a). Moreover, the amount of split fibres is higher in pulps manufactured under harsh conditions in refining than under more gentle conditions (Pöhler and Heikkurinen 2003). The fibril angle of the cellulose fibrils in the fibre wall might also influence the tendency for high fibre collapse. However, fibril angles are also strongly related to fibre wall thickness, which means that this effect co-correlates well with the effect of wall thickness (Jang et al. 2001).

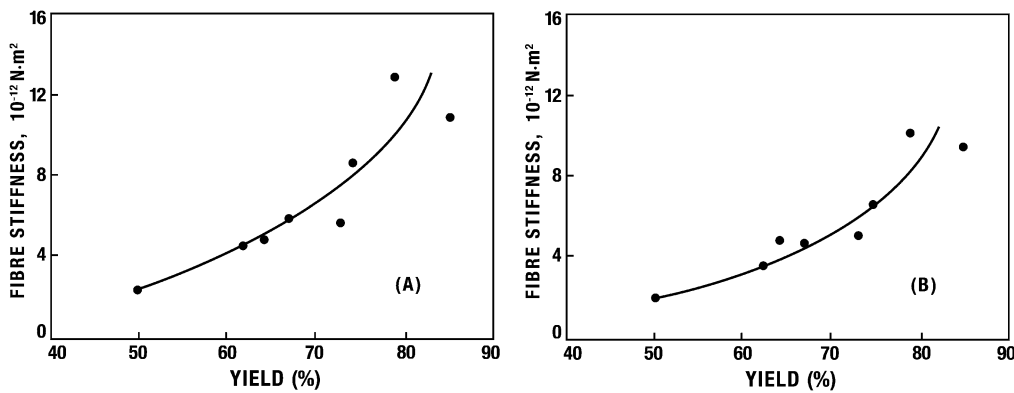
The flexibility of wet pulp fibres has been measured by Tam Doo and Kerekes (1982). They argue that wet chemical pulp fibres are 20 to 30 times more flexible than wet mechanical pulp fibres from the same wood, Figure 7. In their testing equipment, a single fibre is placed across a notched tip of a submerged capillary tube, and water is drawn into the tube to deflect the fibre. The fibre stiffness was calculated from data at its maximum deflection.

The effect of refining of spruce wood from earlywood and latewood respectively on sheet tensile index was tested by Hedberg Hu et al. (1999). The refining was made in a laboratory size refiner. The separation of earlywood and latewood was made by hand from spruce veneer. The study clearly demonstrates that thin-walled earlywood and thick-walled latewood fibres form networks with different tensile strength, Figure 8. The thin-walled earlywood fibres from the long fibre fraction (BMcN 16–30 mesh) form a network with much higher strength properties, than the thick-walled latewood fibres.

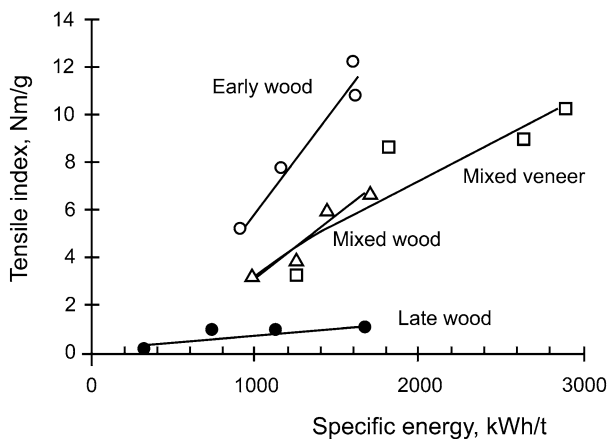
The latewood fibres were obviously too stiff even after a reduction of the wall thickness (compare results in Figure 6), which means that the sheet density of paper sheets from such very stiff fibres most probably are much lower than from earlywood fibres (Reme et al. 1999a).

The density and strength of sheets from HYP is also affected by the curl of the fibres. This was clearly visualized by Beath et al. (1966) in their studies of the so-called latency effect. In high consistency refining, fibres are twisted and curled at sufficiently high temperature under treatment in a narrow refiner gap. Cooling the fibres after refining will stiffen the lignin and hemicellulose and preserve fibres in their distorted form. Distorted materials can be found in all different fibre size fractions (Mohlin 1979a). However, if the pulp is disintegrated in a suspension at a temperature close to or above the softening temperature of lignin the fibre can be straightened. This treatment significantly improves the sheet strength (Htun et al. 1987).

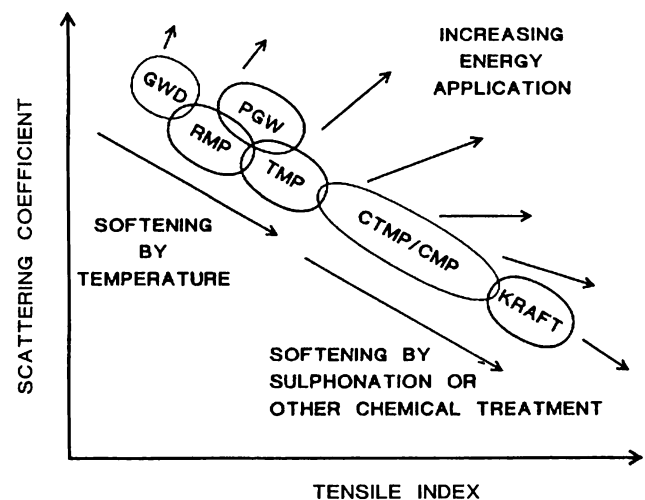
Shives is naturally the stiffest type of particles in HYP. It is of outmost importance to reduce the shive fraction to a minimum in the manufacturing of all types of HYP-



**Figure 7:** A: The effect of yield on fiber stiffness of laboratory cooked acid sulphite eastern Canadian spruce/balsam (3:3) mixture. It is interesting to note that the stiffness of mechanical pulps (estimated yield 95 %) for this species was found to be about  $30 \times 10^{-12} \text{ N}\cdot\text{m}^2$ . A: fibres from BMcN + R14 fractions. B: Fiber from BMcN 14/28 fractions. Redrawn from Tam Doo and Kerekes (1982).



**Figure 8:** Tensile Index of TMP from spruce earlywood and latewood at refining with different specific energy consumption. Redrawn from Hedberg Hu et al. (1999).



**Figure 9:** Typical relations between light scattering coefficient and tensile index in sheets from different softwood pulps (Wood and Karnis 1991).

based products. Especially in thin papers with low grammage, shives are considered to initiate breaks due to too few strong joints in the zones around a shive (Reme and Helle 2000).

In summary, to obtain sheets of high tensile strength from HYP, a requirement is that the wood fibres in their long and middle length fibre fractions are made flexible. The interaction between long fibre and fines to create sheet structures of high strength in graphic papers will be discussed next.

## Bonds in graphic paper structures

### Interaction between long fibre and fines

High quality graphic papers (News, LWC and SC papers) are characterized by a high opacity, high surface smooth-

ness and good sheet formation at a given strength level (Kartovaara 1989, Wood and Karnis 1991, Heikkurinen and Leskelä 1999, Ionides 2003, McDonald et al. 2003). To achieve these characteristics, the sheet structure must contain a mix of bonded and unbonded surfaces of the fibre components (Wood and Karnis 1991, Karnis 1994, Mohlin 1997). The unbonded areas in the sheet structure scatter light if the distance between the surfaces is not too short, i.e. less than approximately 200 nm (Alince 1986, Lindblad et al. 1989), while bonded areas add to the strength. Strong interaction between fibres, fibre fragments and fine materials is essential. The relation between light scattering and tensile index for different types of HYP is visualized in Figure 9. In sheets from mechanical pulps (SGW, PGW, TMP), both the unbonded and bonded areas increase with increasing energy input in grinding/refining

processes, i. e. both light scattering and tensile strength are improved, while bonded areas are increased much more in sheets from CTMP. At improved softening of fibres and decreased yield in pulping processes, bonded areas increase at the expense of unbonded areas. Improved bonding may above all be an effect of increased fibre flexibility, changed fibre surface structure and changed quality of fines.

The effect of fines on graphic papers strength has been described in many publications over the years – extensive papers and reviews is found in the literature (Honkasalo et al. 1983, Rundlöf 1996, Luukko 1998, 1999, Lehtonen 2014). In the literature, it has been pointed out that the quality of fines is most important during the initial phase of consolidation of the paper structure and to achieve strong final fibre-fibre bonds in wet-pressing. Giertz 1977 mentioned that fines increased the surface tension forces (Campbell's forces effect) in consolidation of a paper web. Brecht and Klemm introduced the idea that the final sheet strength after wet-pressing and drying of mechanical pulp webs to a large extent can be explained as the sum of the properties of its fractions. Many investigations have followed and given valuable contributions to our understanding of these interactions (Marton and Alexander 1963, Jackson and Williams 1979, Lindholm 1980a, 1980b, 1981, Rundlöf et al. 1995, de Silveira et al. 1995, Karnis 1996, Moss and Retulainen 1997, Luukko and Paulapuro 1999, Ferritsius et al. 2009, Reyier Österling 2015). Typical values of tensile strength on sheets from different fresh fibre material fractions, which have been separated from quite different HYP in a standard Bauer McNett fractionator, are shown in a study of by Jackson and Williams (1979), Figure 10. Properties of fractions from three TMPs, two CTMPs and one unbeaten slush SBK (semibleached kraft pulp) were studied. CTMP was manufactured both under standard mild conditions and more severe. Of the long fibre fractions from mechanical pulps, the SGW has the highest tensile strength, while the TMP with the highest CSF has the lowest. In contrast, the fine fraction from SGW has the lowest tensile strength and TMP the highest. Similar ranking between SGW and TMP fractions have been published by Lindholm (1980a). The difference in bending stiffness is probably the reason for the different properties of fibres in the long fibre fractions. The tensile strength of sheets from the different fractions of SBK and the low yield CTMP are small, whereas the differences are great in sheets from SGW and TMP.

In refining of TMP and CTMP, the qualities of the fibre fractions and the fines are developed simultaneously as most fines are peeled off from the outer surface of free fibres during treatment in the refiner gap (Heikkurinen

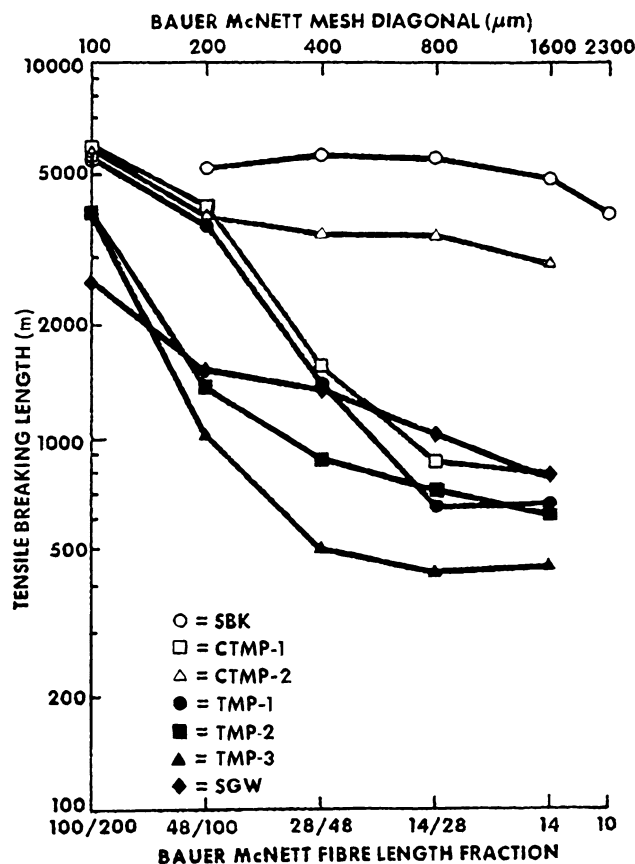
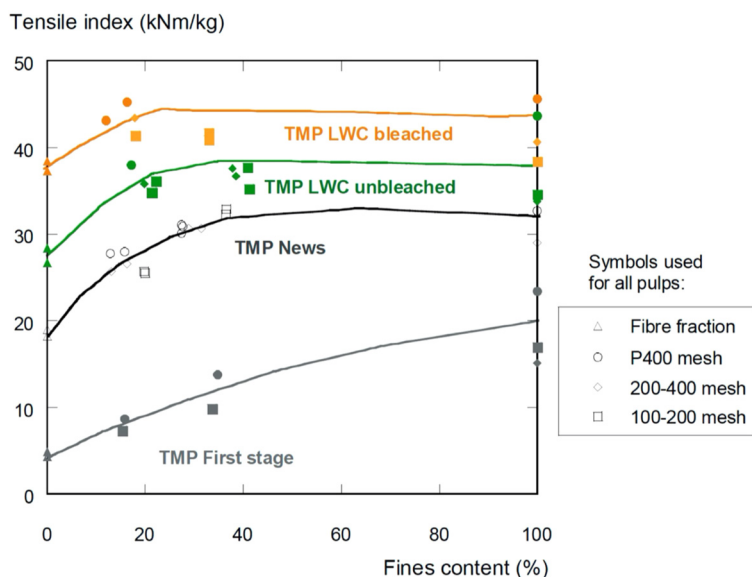


Figure 10: Tensile breaking length of Bauer-McNett fractions from SGW, TMP, CTMP and SBK pulps (Jackson and Williams 1979).

and Hattula 1993, Karnis 1994, Reme et al. 1999b). At a low energy input mostly flake-like fines particles with a high content of lignin are produced (Luukko 1999, Kangas et al. 2004, Vehniäinen 2008). In more intense treatments, fines with an increasing amount of thread-like shape will appear, presumably originating from the inner parts of the fibre wall. These types of fines are considered to give a higher contribution to the strength properties than the flake-like fines (Brecht and Klemm 1953, Giertz 1973, Luukko 1998). At the same time, the fibre walls become thinner and more flexible. Somewhat surprisingly, the correlation between tensile index and density will be developed linearly within a broad field (Höglund and Wilhelmsson 1993, Heikkurinen and Leskelä 1999, Rundlöf 2002) in spite of the difference in both long fibre and fines properties at different densities, Figure 2. As an example, sheets from HTCTMP, i. e. CTMP manufactured after pre-heating to a temperature well above the softening temperature of lignin, can be manufactured within a very broad range of densities, according to Figure 2. In the low-density domain, the mix of stiff fibres that to a high extent is covered with lignin and flake-like fines gives sheets with low



**Figure 11:** Tensile Index as a function of fines content for different spruce TMP. The unbleached first stage pulp had a freeness value of CSF 700 ml, TMP News CSF = 175 ml, TMP LWC CSF = 91 ml and bleach TMP LWC CSF = 52 ml (Rundlöf 2002). Modified ISO sheets.

density at a given tensile strength. In comparison to a kraft pulp, the density is extremely low at a given tensile strength. At high densities, a mixture of more slender and flexible fibres combined with thread-like fines results in sheets with high tensile strength. At the highest density, i. e. at the highest input of energy in the refining process, the strength approaches that of chemical pulps.

Very high sheet strength of pulp from TMP processes can be achieved at reject refining of separated long fibres in screen rooms, when the fines are peeled off and the refining proceeds further on the same fibres (Wedin et al. 1992, Höglund and Wilhelmsson 1993, Corson et al. 1997). The fines will then consist almost exclusively of thread-like particles giving a high contribution to tensile strength (Heikkurinen and Hattula 1993). Selected chemical treatment of the long fibre fraction can further improve bond strength in TMP sheet structures (Heitner et al. 1983, Gummerus 1983, Lindholm 1984, Shaw 1983, Franzen and Li 1985).

Effects of adding different amounts of fines to fibres from long fibre fractions have been frequently studied (Jackson and Williams 1979, Corson 1979, Mohlin 1979b, Lindholm 1980b, Retulainen et al. 1993a, 1993b, Rundlöf, et al. 1995, Luukko and Paulapuro 1999). In a figure from Rundlöf (2002) it is shown that the strength of sheets of long fibre fractions from TMP, which were refined to low freeness levels (CSF = 52–175 ml), is improved in a similar way, when fines from the same pulp is added, Figure 11. However, the strength of sheets from a high freeness pulp behaved somewhat differently at addition of its own fines.

The lower improvement in strength is expected as the fines fraction from the pulp in question consisted mostly of large flake-like particles with a high content of lignin (Heikkurinen and Hattula 1993). However, further refining actions of these fines would probably have resulted in much improved strength.

Results similar to Figure 11 are shown in investigations by Lindholm, Figure 12. He showed that the strength of sheets from TMP long-fibres fraction is somewhat lower than that of sheets from SGW and PGW. However, the more fibril-like fines from TMP add more to the final strength. Tensile index (and sheet density) typically increases steeply at lower additions of fines and then level out at about the same level as the original pulp. This seems to support the conclusion that the presence of fines assists the consolidation of the sheet.

Figure 11 indicates that fines and fibres develop simultaneously with increasing energy input in refining. Clearly, the amounts of thread-like particles in the fines increase, but the flake-like fraction seem to develop too. Westermark and Capretti (1988) found that a fines fraction enriched in flake-like particles from a TMP of newsprint quality gave about the same tensile index as the original pulp, much higher than the flake like fines shown for the high freeness pulp in Figure 11. Their experiment was repeated with the same result for another sample of the unbleached TMP for LWC in Figure 11 (unpublished data).

Luukko and Paulapuro (1999) have shown how fibrillar fines from TMP strongly improve density and strength of a TMP sheet network, Figure 13A, while flake-like fines

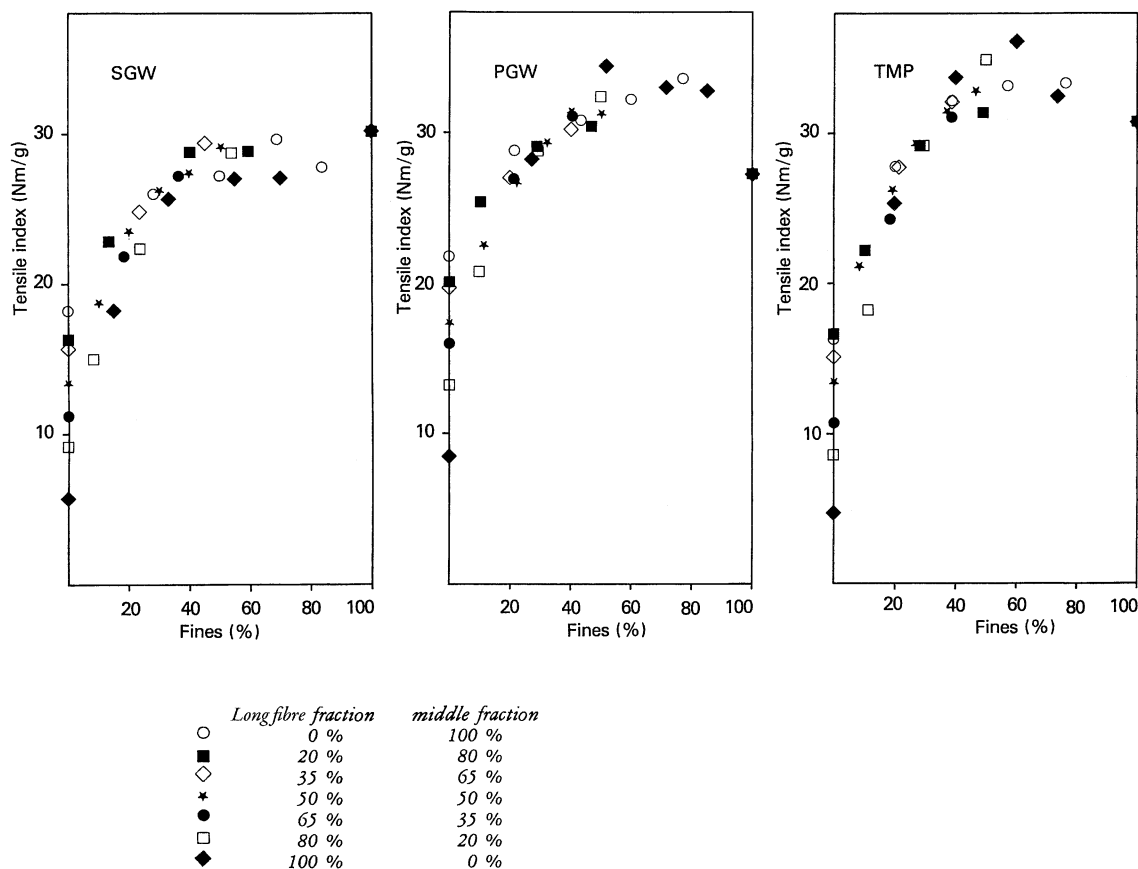


Figure 12: Improvements of tensile index when adding fines to fibre fractions (Lindholm 1980a). ISO sheets.

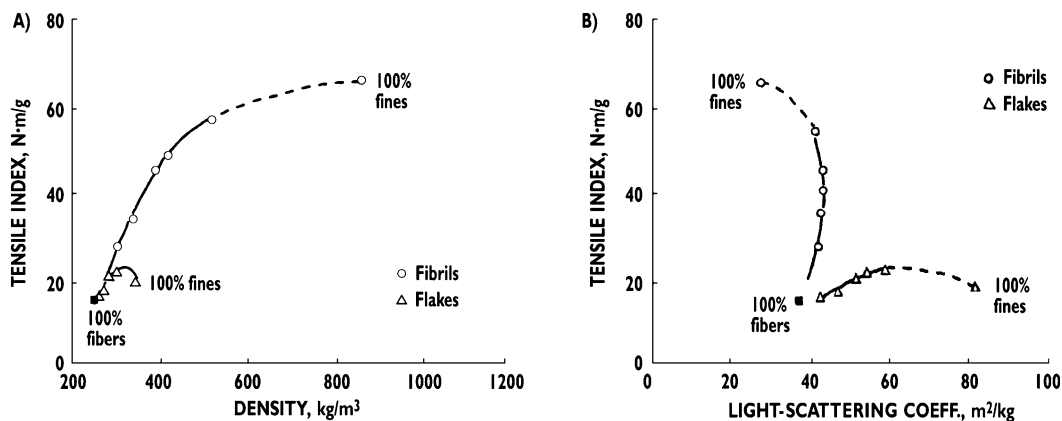
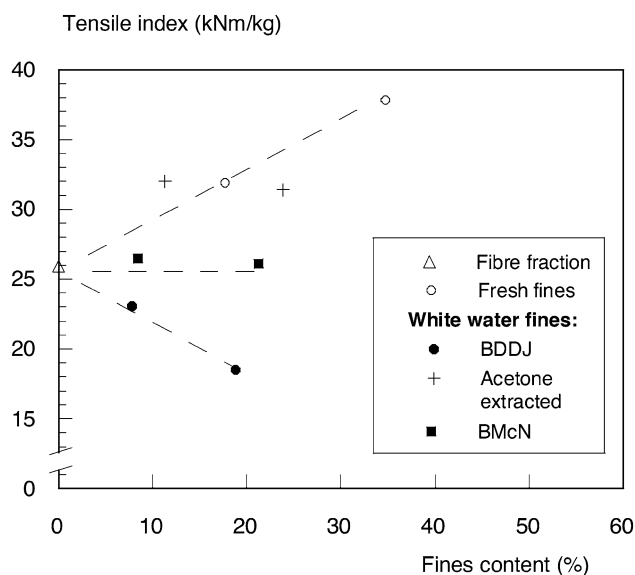


Figure 13: A: Effect of adding fibrillar fines and flake-like fines (R300/R400) on the tensile index and density of the fines/fibre mixture sheet. B: Effect of particle type (fibre and flake (R300/R400 fractions) on the tensile and light-scattering coefficient of the fibre/fibre mixture sheet (Luukko and Paulapuro 1999).

that are manufactured at exceptionally low energy input in refining (i. e. at very high CSF) primarily just increase light scattering ability, i. e. add mostly unbonded surfaces to the structure, Figure 13B. They also showed that the separated fibrillar TMP fines could improve density and strength almost as efficiently as fines from bleached chemical pulp.

The different effects of fines from bleached chemical pulps and TMP on tensile strength in relation to light scattering has also been studied by Retulainen et al. 1993a, 1993b, among others.

In contrast to the effects of the fresh fines showed in Figures 11 and 12, added fines that have been contaminated



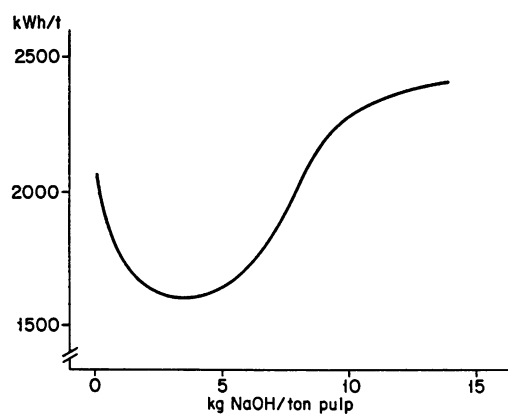
**Figure 14:** Tensile index of modified ISO-handsheets of fresh fines and differently treated fines from white water added at the same fresh fibre fraction. The procedure used to fractionate the fines from the white water sample is shown as “BDDJ” for Drainage-Jar and “BMcN” for Bauer-McNett fractionators (Rundlöf et al. 1995 and 2000a).

by extractives in mill white water can reduce the strength of the fibre fraction. This is shown in Figure 14, which also shows how the strengthening effects of fines are recovered after extraction. The weakening of paper strength by wood resin has been studied in more detail by Rundlöf et al. (1995, 2000a, 2000b, 2000c) and Sundberg et al. (2000), among others.

There are at least two types of particles in the fines fraction of fresh pulps that do not contribute properly to sheet strength: ray cells (Westermarck and Capretti 1988, Wood and Karnis 1992) and fines from knots (Sahlberg 1995). These particles may even decrease the strength, when added in low amounts.

### Influence of charges, counter ions and swelling on strength properties

Already during the eighties, it was shown possible to improve the TMP chip-refining process by adding alkali to neutralize the wood fibres during defibration and fibrillation occurring in the pressurized refiner. Even a small amount of sodium hydroxide (3–4 kg/t) is enough to give a strength increase at the same specific energy consumption, or a reduction in energy demand by about 20 % to the same strength, Figure 15 (Engstrand et al. 1991a). This NaOH addition corresponds to the amount of carboxyl acid



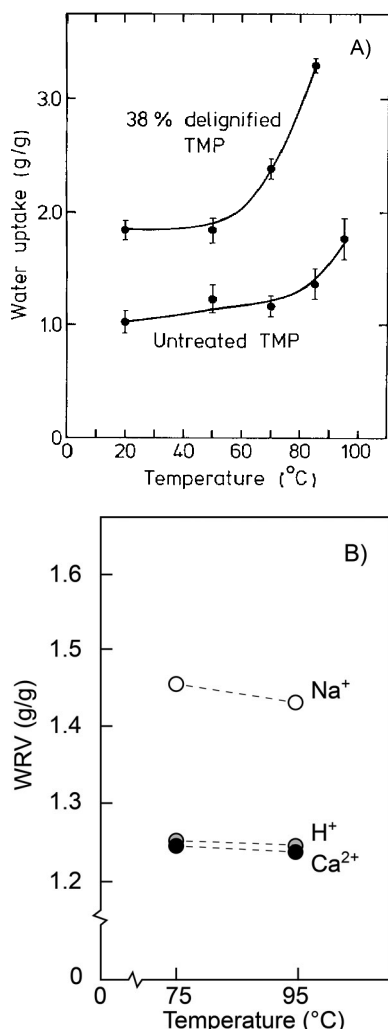
**Figure 15:** Specific energy consumption measured at a tensile index of 32 kNm/kg as function of sodium hydroxide charge to the plug-wiper water. This TMP was manufactured in a pilot mill as follows: accept fraction from spruce wood chips was vapor steamed at 100 °C, 15 minutes, whereafter it was water-impregnated to a dry content of 39 %, preheated at 127 °C. The chips were thereafter refined in one refiner step to pulp of different freeness degrees.

groups in spruce wood (in the range 75–100 μmol/g) and would therefore dissociate the major part of the carboxylic acid groups. However, these effects may be difficult to implement in mill systems (see below).

Figure 15 shows one strong indication of the ionic polymer character of wood and mechanical pulp, further supported in later publications, see e.g. Hammar et al. (1995). One hypothesis is that the effect of neutralization in the refiner is related to swelling, water uptake, of the fibrous material. It should be pointed out that alkaline conditions also lead to chemical modifications which may contribute to sheets strength in other ways than by increasing fibre charge. It has later been shown that the chemical environment in the pulp passing through the gap of high consistency refiners can influence gap distance as well as the gap temperature profile (Engstrand et al. 1995).

An interesting sulfonation related effect on the strength/energy balance in refining was found by Axelsson and Simonsson (1982 and 1983) when investigating the influence of low additions of sulfite, where about 2 kg sodium sulfite addition per ton of pulp resulted in a local minimum in energy demand to a certain tensile index. When the sulfite charge is only about one tenth of the amount used in conventional CTMP (20–25 kg/t) nearly all sulfite is quickly converted to sulfonate groups bound to the lignin and the softening should be very localized. This was also what was found, as the lignin in the primary wall gets a far higher degree of sulfonation than the bulk part of the lignin (Westermarck et al. 1987).

For TMP, swelling has been studied by means of the solute exclusion technique and by means of the WRV tech-



**Figure 16:** A: water uptake as function of temperature for TMP and partly delignified TMP, both in sodium form, measured by means of solute exclusion technique (Eriksson et al. 1991). B: Water retention value at 3000 G at elevated temperatures for peroxide treated TMP fibres, total charge level of 269  $\mu\text{mol/g}$ , in proton form as well as in sodium and calcium form (replotted from Fjellström et al. 2013).

nique (water retention value at 3000 G, see Eriksson et al. 1991 and Fjellström et al. 2013). Figure 16A shows that the water uptake of the fibrous material increases with temperature which is interpreted as the softening of the fibre wall increases the possibility to swell. Figure 16B shows a clear effect of ionic form at elevated temperatures, Na<sup>+</sup> as counterion gives a higher WRV than Ca<sup>2+</sup> and H<sup>+</sup>. This difference is negligible at room temperature.

For chemical pulps, increasing swelling of fibres has been related to increasing sheet strength (see e. g. Scallan and Grignon 1979, Lindström 1992 and references therein). Not only dissociation of the carboxyl groups but also the valence of the counter ions is of importance. Normally

both swelling potential and tensile strength decrease in the order:

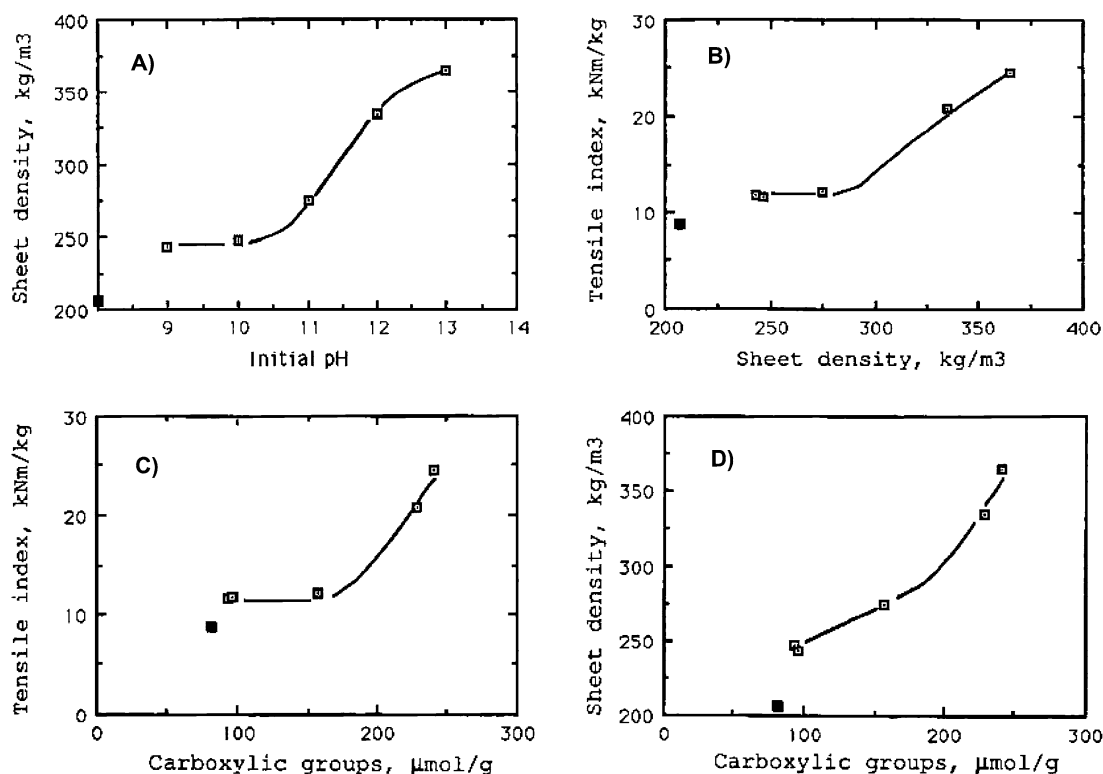
$$\text{Na}^+ > \text{Ca}^{2+} \approx \text{Mg}^{2+} > \text{H}^+ \geq \text{Al}^{3+}.$$

This is shown valid also for mechanical and chemimechanical pulp fibres treated with alkali and therefore containing increased amounts of charged groups. Sheet strength properties can then be increased by a favorable counter ion form, corresponding to the same order as shown above for chemical pulps (Katz et al. 1981). In this case, the effect of counter ions is studied separately using one and the same fibrous material. Therefore, it is safe to assume that the increase in strength is caused by the ion exchange which give an increase in the overall bonding in the sheet. But it remains to be clarified to what extent this is due to an increased contact area and/or stronger bonding in the contact points.

As shown above, another important aspect related to bonding in mechanical pulp sheets is the amount of charge itself, for example when the number of carboxylic groups in lignin are increased by means of modifications to the hydrogen peroxide bleaching. This is hard to study separately, without also having other contributions to sheets strength from the chemical modifications needed to increase the amount of charged groups. In the case shown in Figure 17, the pH in peroxide bleaching is increased resulting in increased charge (mainly in lignin) and this leads to an increased densification of the sheets which begins at a pH above 10. This study was performed on TMP without its fines fraction just to be able to analyse the influence of carboxylic groups on sheet properties and it should be emphasized that the amount of carboxyl groups increases with increasing initial pH of the peroxide bleaching. In this case the peroxide charge was kept at 4 % and sodium hydroxide charge varied to control the pH (Engstrand et al. 1991b and 1991c). An increased sheet density naturally increases the fibre-fibre contact area, which increase sheet strength. To what extent the increased charge contribute to stronger fibre-fibre joints was not studied.

The first study showing how mechanical pulp strength properties could be enhanced by means of increased alkalinity in peroxide bleaching was made by Moldenius (1983). It should be pointed out that peroxide bleaching studies in laboratory scale are in general performed in deionized water while the laboratory sheets in most cases are manufactured in tap water. Due to this, the acidic groups in the fibres are mainly in calcium form.

Unfortunately, the quite strong potential to improve strength properties is in many (most) cases counteracted by the fact that the full-scale systems are recirculating process waters. This effect is prevalent in hydrogen peroxide



**Figure 17:** Norway Spruce based TMP fibre fraction (fines removed with a 300 μm Celleco filter) before peroxide bleaching. Bauer McNett fractions; > 16 mesh 51.6 %, 16–30 mesh 23.0 %, 30–50 mesh 15.5 %, 50–200 mesh 4.1 % and < 200 mesh 5.9 %. A: Sheet density as a function of initial pH. B: Tensile index as a function of sheet density. C: Tensile index as a function of carboxyl group content. D: Sheet density as a function of carboxyl group content.

bleaching as shown below, but mill systems may also lead to difficulties in utilising the local minima in energy consumption shown above.

Circulating process waters may contain large amounts of wood extractives. The higher pH in bleaching results in destabilization of colloidal droplets containing lipophilic extractives (Sundberg et al. 1996) which are then deposited onto the surfaces of fibres and fines (Rundlöf et al. 2000b and 2002), where extractives most probably decrease the strength of bonding and cause lower sheet strength when the surface coverage exceeds some critical level (see Figure 14, Rundlöf et al. 1995, Sundberg et al. 2000 and Rundlöf et al. 2000c).

The net result of the alkaline addition in hydrogen peroxide bleaching can be that bonding is reduced instead of improved, Figure 18 (Engstrand et al. 2019). The laboratory sheet tensile index is reduced already after dilution in the first standpipe and the largest reduction is normally over the peroxide bleach tower and the second standpipe after which the strength is not changed much but for the increase over the post refiner. As the alkali charge increases with increasing peroxide charge the decrease in tensile index tend to become larger. Most of this decrease is an effect

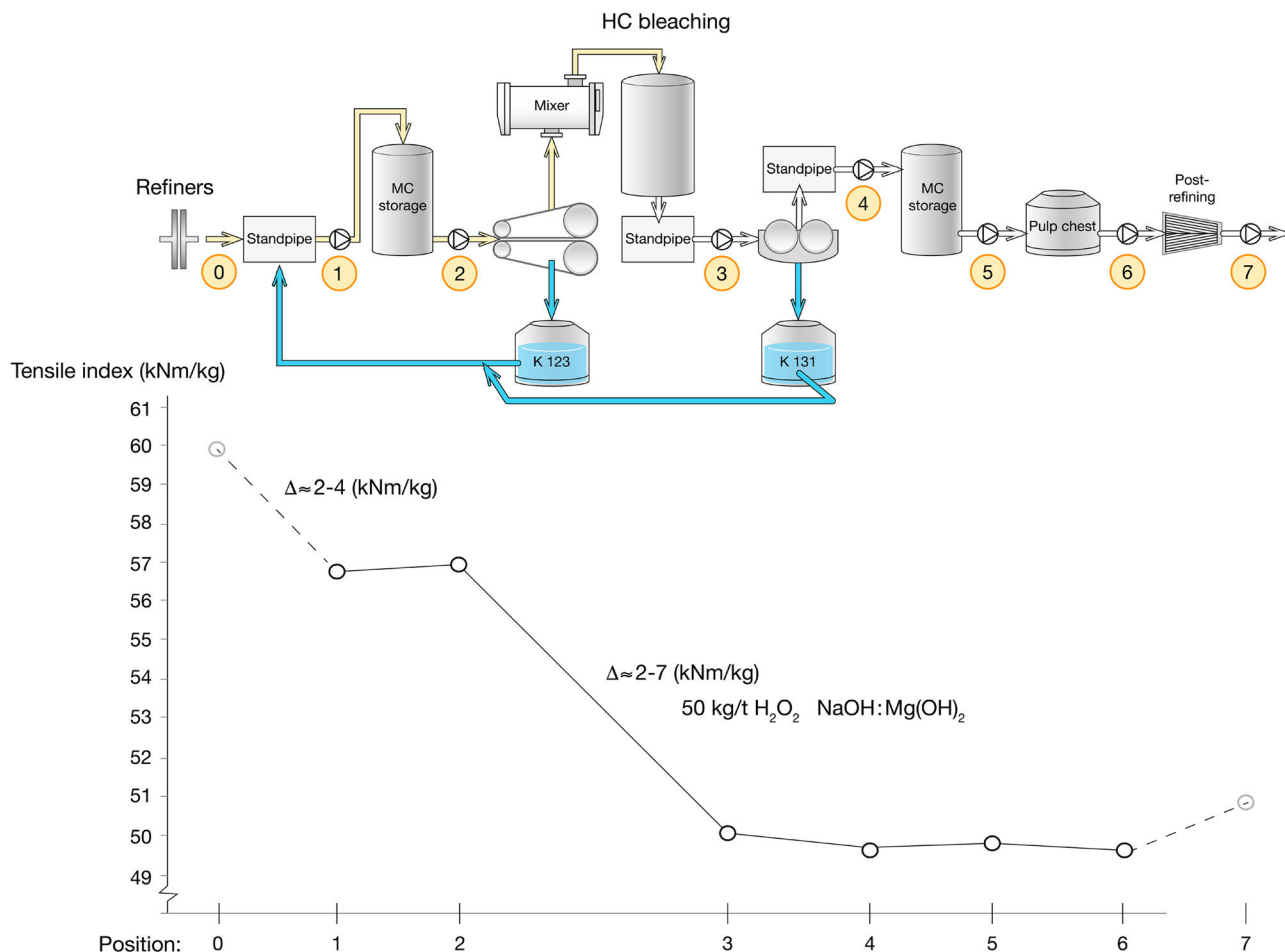
of decreased bonding between the fibrous material and the major part of the tensile index can be restored by washing the fibres in acetone.

This means that there is a great opportunity for paper and paperboard producers to improve strength properties or to reduce refining energy demands provided that we find process solutions making it possible to get full control over resins and counter ions.

## Bonding mechanism

During pressing and drying under conventional conditions in papermaking, bonds are formed in graphic papers based on mechanical pulps when moist fibres of natural length, shortened fibres, fibre fragments and fines are brought into sufficiently close contact.

In the initial phase of dewatering of wet HYP webs in papermaking, surface tension pulls the fibre particles together under the influence of surface tension as water is removed (Lyne and Gallay 1954), which is known as the Campbell effect (Campbell 1959). Fines are considered to



**Figure 18:** The figure shows tensile index as a function of unit operation starting from secondary stage high consistency refining (30–40 %) point 0, via standpipe point 1, where pulp is diluted to 8–10 % over MC-storage tower point 2, to band-press 40 % consistency/mixer/HC-bleaching to standpipe point 4 where pulp is diluted to 8–10 % and sent to MC-storage point 5, stored in pulp chest point 6 and post-refined at low consistency 4 % point 7.

increase the surface tension force during the consolidation phase (Giertz 1977). When pressed to a dry content of about 50 % the strength of the network starts to increase and bonds are established (Lyne and Gallay 1954), see also below in section “Effects of moisture content and temperature at consolidation of sheet structures from spruce CTMP”. The bonds can be of different kinds.

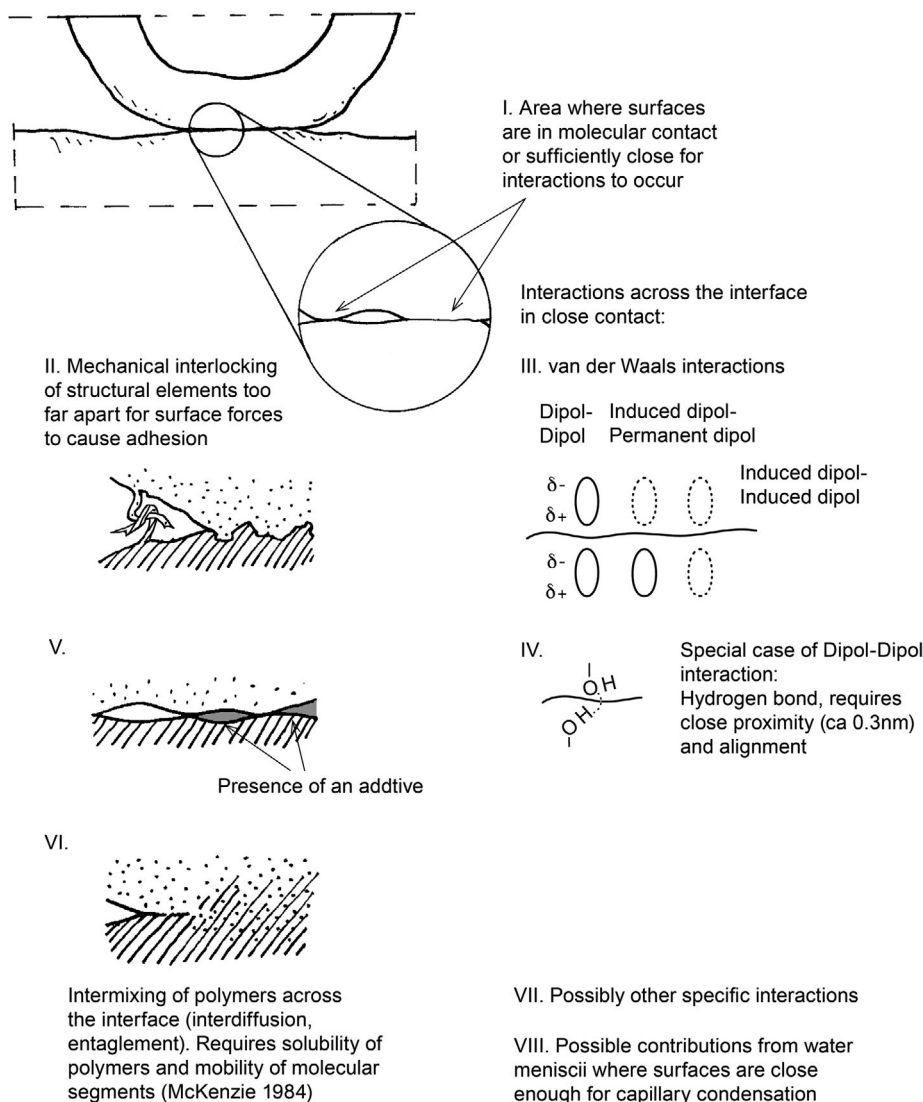
As stated above, we use “bond” in the sense “adhesive bond” – the area where fibres and particles are joined on a micrometre scale (also called “fibre-fibre joint”). Figure 19 gives an overview of a hypothetical cross-section of a dry fibre-fibre bond (based on Figures 5–7 in Lindström et al. 2005) showing possible interactions (I–VIII) which are likely candidates to hold the dry fibrous HYP-material together. This figure is intended as an overview for the sake of discussion, a more thorough survey is beyond the scope of this review. For cellulose fibres this can be found elsewhere (see e. g. Lindström et al. 2005). To our knowl-

edge, the literature is still lacking a detailed analysis of the molecular interactions between surfaces of mechanical pulp fibres/particles. We also note that different terms are used in the literature. To avoid confusion, we use the terminology given by Israelachvili (1997), see also Appendix A. Possible interactions shown in Figure 19:

**I General aspects.** An important factor is the area inside the fibre-fibre bond where surfaces are close enough for attractive interactions to be relevant. For molecular contact, this means distances of molecular dimensions (in the range of 1 nm or closer). In air, van der Waals interactions may also be significant over longer distances, in the order of magnitude of 10 nm. About 30 nm has been indicated for pure cellulose surfaces (Hirn and Schennach 2015). In the following, we use “close contact”, for surfaces close enough for attractive interactions to be relevant.

The area in close contact inside the bond may be smaller or larger than the projected area of the fibre-

## Adhesion between fibres in a dry fibre-fibre bond



**Figure 19:** Overview of cross-sections of dry fibre-fibre bonds (based on Figures 5–7 in Lindström et al. 2005).

fibre bond. Surface roughness decreases the area in close contact inside the joint (as indicated by Torgnysdotter 2006). On the other hand, fibrillated fibre surfaces may increase this contact area, since fibrils attached to the surfaces provide much additional surface available for interaction.

The light scattering coefficient is often used as an indication of “bonded” area in a sheet. This may be useful as a relative measure and for engineering purposes, but is not a direct measure of the area in close contact. As a rule of thumb, light scattering becomes less efficient at dimensions smaller than half the wavelength of light, about 200–400 nm (see e. g. Alince 1986), which is much longer than the distances for intermolecular interactions.

Bodies in contact may conform to each other to create additional contact provided that they are flexible enough (Johnson et al. 1971). Viscoelastic or plastic deformations may therefore increase adhesion. As stated above, all elements in HYP, from fibres to fines, contribute to the bonds. This includes fibrils attached to fibres/particles and surface layers of polymers.

**II Mechanical interlocking.** On a macroscopic scale, this physical entanglement is akin to Velcro™ or a jigsaw puzzle. It may contribute to adhesion, but without intermolecular interactions, and do not require as close contact between surfaces. Mechanical interlocking could contribute to the energy needed to separate fibres, possibly by entanglement of fibrils, which dry in a locked position or

suchlike. If mechanical interlocking occurs, it is likely in combination with areas where molecular interactions are relevant.

III Intermolecular interactions (“physical bonding”) take place between molecules that affect each other but remain discrete entities. This is in contrast to chemical bonding where electron charge distributions of uniting atoms change completely and merge to become something else, i. e. reactants form a new molecule. Three types of interactions contribute to van der Waals forces: between permanent dipoles (dipole-dipole or Keesom interaction) or permanent dipoles and induced dipoles (Debye interaction) and between induced dipoles (London or dispersion forces). For two bodies or surfaces interacting, the pair potentials between the molecules in each body are summarised, which means that much of the interactions are non-specific. The term physical bonding does not necessarily refer to specific bonds formed between two molecules but rather interactions between surfaces of bodies, which is dependent on their size and geometry.

IV Hydrogen bonds. A particularly strong and directional dipole-dipole interaction (III) that may form between O–H groups (or N or F) if sufficiently close and aligned. According to Israelachvili (1997), it used to be believed that hydrogen bonding was a form of quasi-chemical bond, but nowadays dipole-dipole interaction is a widely accepted view.

It is often claimed that hydrogen bonds are holding paper together, but the contribution of hydrogen bonds to fibre-fibre adhesion is not firmly established and still a matter of discussion (see Lindström et al. 2005 and references therein). Hydrogen bonds require close proximity and alignment of the interacting groups – McKenzie (1984) mentions a distance of ca 0.3 nm. Hydrogen bonding could take place between cellulose surfaces if sufficient mobility of cellulose chains, softness of surface layers and contact time allow O–H-groups to get in position. For HYP fibres and particles, the situation is different, since available surfaces contain all wood components to varying extents. Hydrogen bonding seems less probable than for pure cellulose surfaces and would hardly be possible between lignin-covered surfaces. Furthermore, van der Waals interactions present between all surfaces in close contact in air seems to be sufficient to hold fibres together, also for cellulose fibres as estimated by Hirn and Schennach (2015).

V Intermixing of polymers from both sides across the interface require mobility and mutual solubility of polymer segments (McKenzie 1984). Intermixing increases the possibility of contact between polymers and therefore molecular interactions. If complete intermixing occurs, the formation of the fibre-fibre bond would be similar to

welding, where opposing surfaces melt and blend together to form a common interfacial zone.

VI Additive(s) present between fibre surfaces may provide additional contact and/or stronger interactions. In addition, the presence of what is normally seen as a contaminant (like wood extractives, see Figure 14) could enhance adhesion by filling cavities where surfaces would otherwise be too far apart and provide additional contact.

VII Possibly other specific interactions, such as ionic bonds where charged groups on opposing surfaces share a divalent ion or even chemical bonds, if conditions make chemical reactions possible.

VIII Water menisci may form where surfaces are close enough for capillary condensation, and contribute in a way similar to the effect of water during drying.

Finally, it should be pointed out that Figure 19 shows possible mechanisms holding a dry fibre-fibre bond together. The formation of the fibre-fibre bonds begins in the wet state. Surface softness and flexibility of HYP material will affect how surface-layers adjust to increase close contact (I), as well as possibilities for polymers to intermix (VI). The character of the wet surfaces, conditions when the bonds are formed (pressing, drying) and also the conditions when the bonds are broken, are all needed for a fuller picture of adhesion in fibre-fibre bonds (a general recommendation by Kendall 2001).

These bonds between mechanical pulp fibres that finally hold the fibre together in graphic papers are frequently referred to as hydrogen bonds in the literature (see e. g. Nissan and Sternstein 1964). In light of the above discussion, other mechanisms such as van der Waals interactions (III) seem probable, with a possible contribution from hydrogen bonding (IV). The bond strength and bonded area in the network increases if the drying phase is formed during a concurrently applied pressure. Otherwise, HYP webs will be consolidated in a more bulky form under the influence of the least flexible fibres in the fractions with long and shortened fibres. This mechanism is discussed in more detail below in section “Effects of moisture content and temperature at consolidation of sheet structures from spruce CTMP”. At an enhanced temperature during pressing and drying, i. e. at a temperature close to or above the softening temperature of lignin, more and stronger bonds can be established (see also below in section “Bonds between HYP fibres due to lignin and hemicellulose flow”).

In a TMP used in manufacturing of graphic papers, typically 30–40 % of the pulp is classified as fines. The average length of fines increases and the lignin content decreases continuously at increased energy consumption in refining (Heikkurinen and Hattula 1993). The fines pro-

duced at low energy input in the refiner have a much higher lignin content than the mean lignin content in the pulp. At high energy input the proportions are the opposite. As both the character of long fibre (mainly flexibility and fibre surface structure) and fines (mainly slenderness and lignin content) are changed simultaneously in refining it is difficult to separate the effect of each of them in consolidation and bonding of the sheet structure. As pointed out above, densification and tensile strength increase in parallel. However, under conventional conditions in papermaking, higher bond strength is reached between fibrillated fibre surfaces and fibrils from inner parts of the fibre walls, rather than between surfaces that are partly covered with lignin. This belief is also prevalent in the literature and finds support in observations by Gupta et al. (1962), showing that the adhesion between surfaces of wet lignin is low or non-existing at temperatures below the softening or tack temperature of lignin. No doubt, fibre surfaces and fines that are covered with lignin add more to the percentage of unbonded surfaces in the sheet structure, which is also necessary to achieve high light scattering in the graphic paper structures. However, there is still reason to note that almost all unbonded lignin surfaces in HYP are linked to fibre materials with low flexibility. At consolidation, pressing and drying of paper webs under conditions where the lignin is softened, preferably at a temperature close to or above the softening or tack temperature region, bonds between lignin surfaces can be very strong (VI), see also below in section “Improvement of dry and wet tensile index of sheets from spruce CTMP”. It is likely that some type of entanglements of polymer chains is involved under such conditions, which will be discussed in more detail below in section “Bonds between HYP fibres due to lignin and hemicellulose flow”. To some extent this type of bond may also exist in graphic papers (McKenzie 1984, Pelton et al. 2000).

## Bonds in bulky paperboard plies

### Introduction

The market for graphic papers is declining in many regions in the world because of the increased competition from electronic media. However, the trend is the opposite for paperboards. A smart use of HYP in paperboard products gives the opportunity to improve both the function and quality (e. g. bending stiffness) of light-weight paperboard products at reduced costs (Fineman 1985, Bengtsson 2005). Most research and development of HYP tech-

nologies at institutes and universities have historically been focused on improving properties and processes in manufacturing of graphic paper grades, which have been the main product from mechanical pulps. However, lots of research and development in the past years have also focused on questions and issues of importance in development of CTMP for paperboard grades. Compared to pulps for graphic papers, these pulps can be manufactured at a much lower energy input in the refining stage. CTMP research in this field at Mid Sweden University has been reported in theses (Vesterlind 2006, Pettersson 2007, Klinga 2007), presented at international conferences (Nygren et al. 2003, Pettersson et al. 2005, Klinga et al. 2005, Vesterlind and Höglund 2005a, 2005b, Norgren and Höglund 2011, Norgren et al. 2014a, 2014b) and in scientific journals (Pettersson et al. 2005, 2006, Vesterlind and Höglund 2006, Klinga et al. 2007, 2008, Norgren and Höglund 2014, Norgren et al. 2014a, 2014b, Pettersson et al. 2015). Based on the most interesting results from these publications, the bonding mechanisms in sheets made of CTMP to be used in paperboard plies are discussed in this review together with fundamental aspects reported in the literature.

The importance of reducing the stiffness of fibres in the long-fibre and shortened fibre fractions in mechanical pulps at manufacturing of thin graphic papers was discussed in previous sections. This seems to be a prerequisite requirement for high bond strength in these paper grades. By contrast, the high stiffness of HYP long and shortened fibres are utilized as an advantage in products, where a high sheet bulk at a certain strength level is demanded. As an example, the bending stiffness is improved significantly when paperboards are designed to have outer plies with a high in-plane strength and tensile stiffness, such as those from chemical pulp fibres, and a bulky middle ply with stiff HYP fibres as a main component (Fellers et al. 1983, Fineman 1985, Bengtsson 2005), Figure 20. In this respect, the best HYP for the middle ply is the one that gives the best combination of high sheet bulk and z-directional fibre-fibre bonds (i. e. high out-of-plane strength). A certain level of fibre-fibre out-of-plane bond strength is always needed to prevent delamination in a bulky ply structure.

### Stiffness of CTMP fibres from spruce wood and some hardwoods

There is a huge variation in terms of fibre morphology and wood polymer composition between softwoods (conifers) and hardwoods (deciduous), but also between different

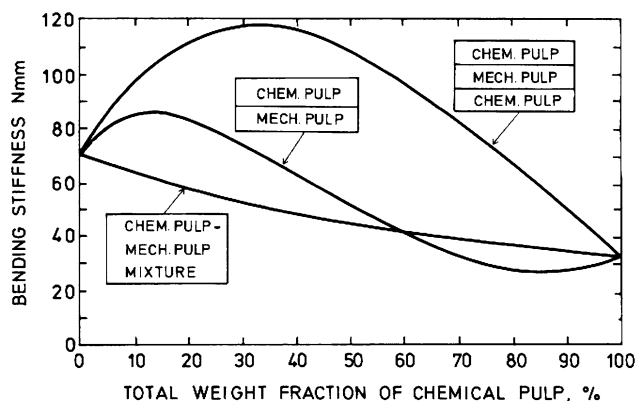


Figure 20: Multilayer sheet structure design for high bending stiffness (Fellers et al. 1983).

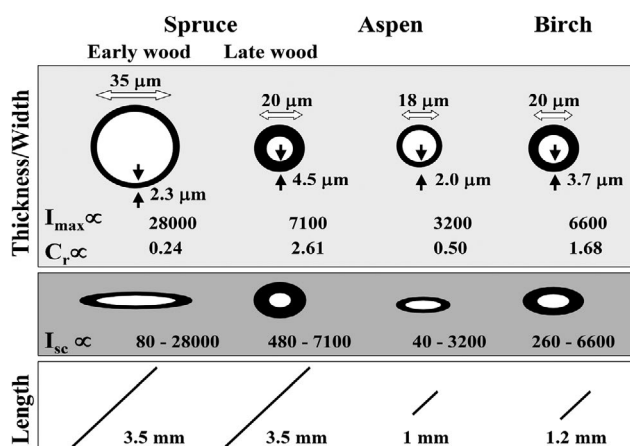


Figure 21: Schematic illustration of typical fibre dimensions and degree of possible fibre shape collapse at refining of various types of wood species (Vesterlind and Höglund 2005b).

families of trees. Figure 21 shows how fibre dimensions usually vary. Softwood earlywood has long, slender fibres with a large fibre width and a large lumen, while softwood latewood has a smaller fibre width and a significantly thicker fibre wall. Hardwoods generally have shorter fibres with a small fibre width and lumen area. The different fibre shapes will of course have a great impact on their stiffness.

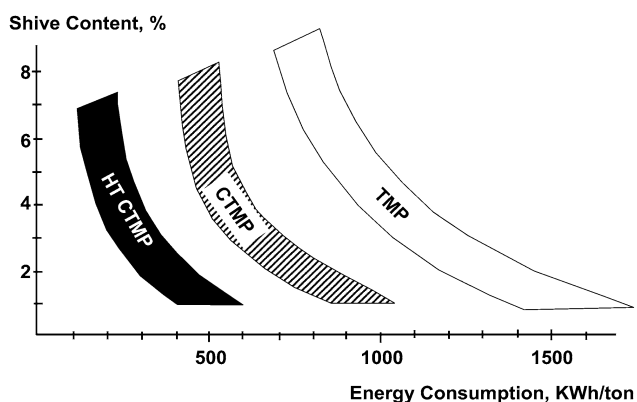
Figure 21 shows estimated bending stiffnesses in terms of area moment of inertia,  $I$ , calculated from a simplified model, where the cross-sectional fibre-wall area is assumed to be in the shape of a circle. In the model the material properties such as the E-modulus is assumed to be the same regardless of type of wood, and to be constant throughout the thickness of the fibre-wall. The bending stiffness has its maximum when the fibre shape is intact,  $I_{max}$ . A large fibre cross section area like on spruce earlywood and a thick fibre-wall, like in spruce latewood or in

birch wood, is highly beneficial for a high bending stiffness. However, at refining in TMP and CTMP processes, the fibres are made more and more flexible and their shapes are gradually changed, which is reflected in reduced bending stiffness,  $I_{sc}$ . This reduction is most pronounced on earlywood spruce fibres, whereas fibres with small fibre widths but thick fibre walls are less affected.

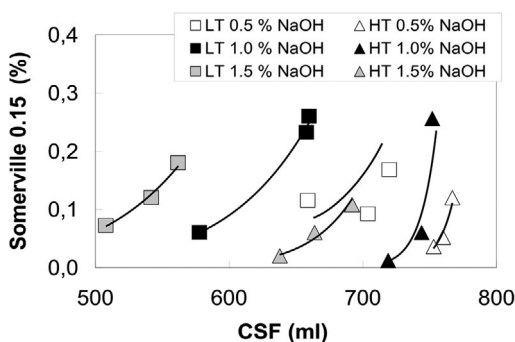
Figure 21 also includes a measure of the collapse resistance,  $C_r$ , which is proportional to the compression load needed to collapse the fibre structure. It increases with increased fibre-wall thickness and decreased fibre-width. This means that fibres, such as birch and latewood spruce fibres are most resistant to collapse, whereas fibres from aspen and earlywood spruce are more easily collapsed in compression. The impact on stiffness of a partly distorted structure is greater for fibres with a thin fibre-wall. This is illustrated by the term  $I_{sc}$  (semi-collapsed or partly collapsed fibres) and shows the range of bending stiffness for un-collapsed and fully collapsed fibres. With this information at hand, the production of paper grades requiring high bulk should preferably use softwoods with a high content of latewood or hardwoods with thick fibre-walls (such as birch wood), as the wood raw material. The energy input in refining should be low to avoid severe fibre collapse, that is, a reduction of moment of inertia,  $I$ , and collapse resistance,  $C_r$ . In refining, a thick-walled hardwood fibre with a small width, like that from birch, can obviously preserve its high bending stiffness better than a medium-sized spruce fibre. Not only are there morphological differences, but there are also major differences in wood polymers between softwoods and hardwoods. For instance, the amount of cellulose is roughly the same in spruce (*Picea Abies*) and birch (*Betula Pendula*), but the amount of lignin is higher in spruce (about 27 % lignin in spruce compared to about 21 % in birch) and the hemi-celluloses are correspondingly lower. Due to the smaller amount of lignin, birch cannot be sulfonated to the same extent as spruce (Lindholm and Kurdin 1999). This obviously has implications for the processing. Adapting the chemical charge according to the lignin structure and type of hemi-celluloses is therefore of great importance. It is common knowledge that sulfonation reduces the shive content. Alkali impregnation, however, increases pulp strength at reduced bulk of hardwood CTMP in a significant way, which is not the case for softwood CTMP, and have a negative effect on yield and brightness (Koran et al. 1984, Katz et al. 1981, Jackson 1988, Jackson et al. 1988, Granfeldt et al. 2001).

At a given tensile strength, CTMP seems to give the highest bulk of all pulps in the HYP family (Höglund and Wilhelmsson 1993). The bulk in a ply from HYP can be improved by increasing the amount of fibres with high bend-

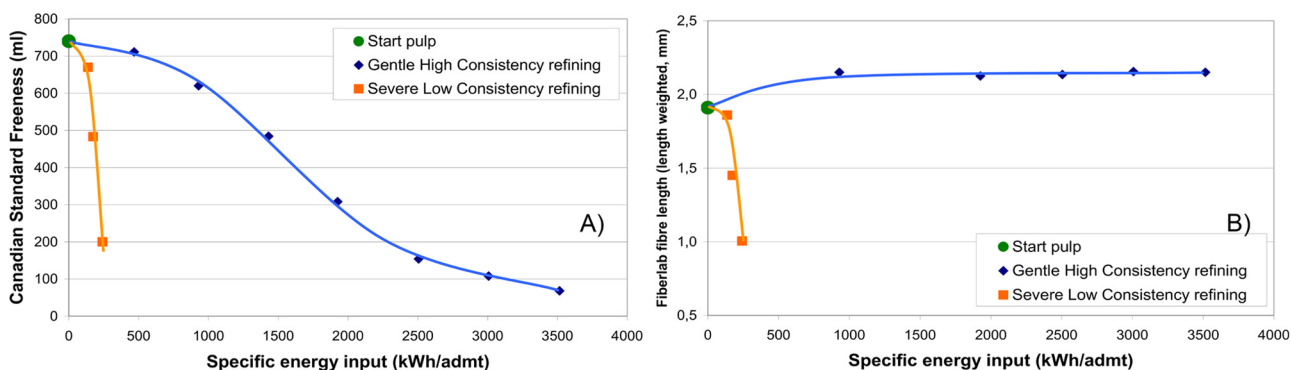
ing stiffness. The high bulk is reduced when the stiffness of the fibre walls is reduced, either by more intensive refining (i. e. refining to a lower freeness at high energy input), or by chemical means in chip softening pre-treatments or pulp bleaching. The energy input in refining during man-



**Figure 22:** Fibres are separated very efficiently at a low level of energy input in the HT CTMP process (Höglund 2002).



**Figure 23:** Somerville shive content vs. CSF in manufacturing of CTMP from birch in a mill trial. CTMP was produced both at a low pre-heating temperature denoted LT (130 °C) and at a high temperature HT (160 °C) (Vesterlind and Höglund 2006).



**Figure 24:** CSF, A and fibre length, B (optically measured at Metso Fiberlab) development during post-refining of a HTCTMP (CSF = 740 ml) from spruce. Pilot plant trials (Klinga et al. 2008).

ufacture of CTMP can be reduced to a limit, where too high shive content will cause problems. This borderline can be adjusted towards lower energy inputs by softening of wood lignin in chip pre-treatment before refining, as in the HTCTMP process, where the lignin is sulfonated at temperatures above the softening temperature range of lignin, Figure 22. To reduce the negative effect of fibre wall softening at high temperature on fibre stiffness, the chip pre-treatment temperature and time must be carefully optimized.

Vesterlind 2006 studied the combined effects of chemical impregnation ( $\text{Na}_2\text{SO}_3$  and  $\text{NaOH}$ ) and pre-heating to a high temperature prior to refining of birch chips in the manufacturing of CTMP of paperboard quality. It was shown that it is possible to manufacture also birch CTMP, like spruce CTMP, with low shive content at low energy input at preheating and refining at a temperature above the softening temperature of lignin, Figure 23. The temperature needed to achieve this effect is somewhat lower than in refining of softwood, as the lignin softening temperature generally is somewhat lower for hardwood (Becker et al. 1977, Heitner and Attack 1983, Salmén et al. 1999).

### Correlation between out-of-plane strength and bulk in CTMP based sheets

In pilot plant trials, Klinga et al. (2008) studied the effect of post-refining of spruce HTCTMP (CSF 740 ml) at both very gentle refining conditions at high consistency (HC) and under severe conditions at low consistency (LC). The HC refining was performed to gently knead the fibres to increase fibre flexibility, whereas the LC refining was made at high intensity with the intention to reduce fibre length while preserving fibre wall stiffness. Figure 24 shows the development of freeness and fibre length as a function of specific

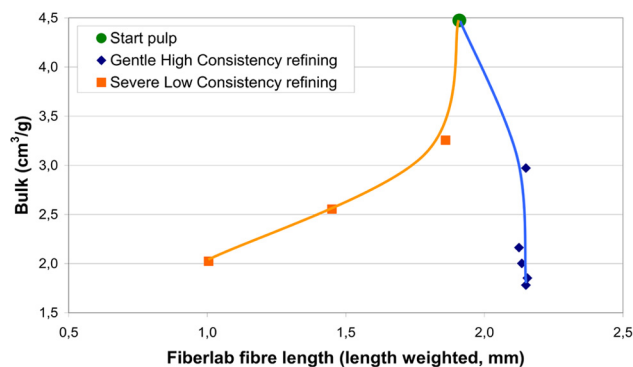
energy. Fibre length was well preserved throughout the HC refining, whereas fibre length was radically reduced in the LC refining. The small increase in fibre length between the start pulp and the first point on the HC curve is believed to be an effect of fibre straightening.

Both the HC and LC refining result in a significant reduction of bulk. The reduction was achieved by two different mechanisms, either making the fibres more flexible (HC refining) or reducing the fibre length (LC refining), see Figure 25.

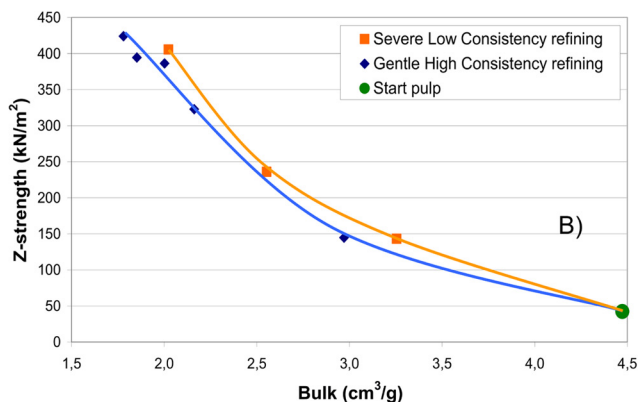
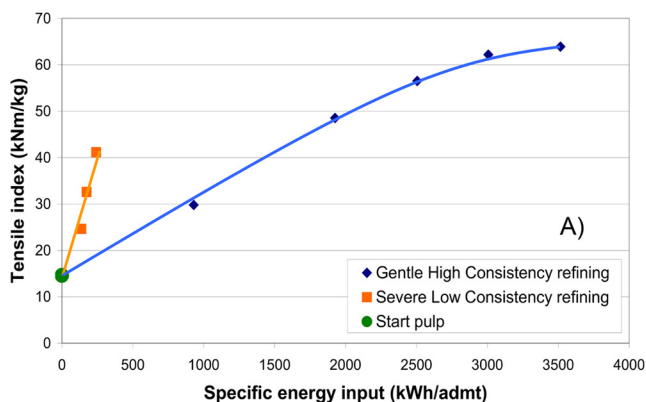
HC refining did, at high energy input, yield pulps with fibres characterized by a high flexibility, giving sheets with both a high in-plane tensile index and a high Z-strength (Figure 26). The high strength values were achieved despite the low content of fines (< 20 %) and the high content of sulfonated lignin on the fibre surfaces, which is a characteristic of fibres from HTCTMP processes. Strong bonds were formed when the sulfonated lignin surfaces were brought into contact. The in-plane tensile index did

not reach such high values in the LC refined pulps, which can be expected due to the reduction in fibre length, compare Figure 25. The Z-strength, however, reached remarkably high values despite the extremely low energy input. Furthermore, the bulk of the LC refined pulps was as good as, or even slightly better, than the HC refined pulps, when compared at a given Z-strength. The difference in fibre length obviously has no impact on the relationship.

In summary, the study by Klinga et al. shows that it is possible to achieve high out-of-plane strength in sheets from spruce HTCTMP in two different ways. Either fibre surfaces, containing a high share of sulfonated lignin, are brought into close contact by making the fibre more flexible, like in HC refining, or by reducing the fibre length, as in LC refining. However, the in-plane strength (tensile index) is much better in sheets from HC-refined pulps, where the fibre length is not reduced on purpose. As expected, the in-plane sheet structure becomes stronger when fibres in its network have joints in more places along its length.



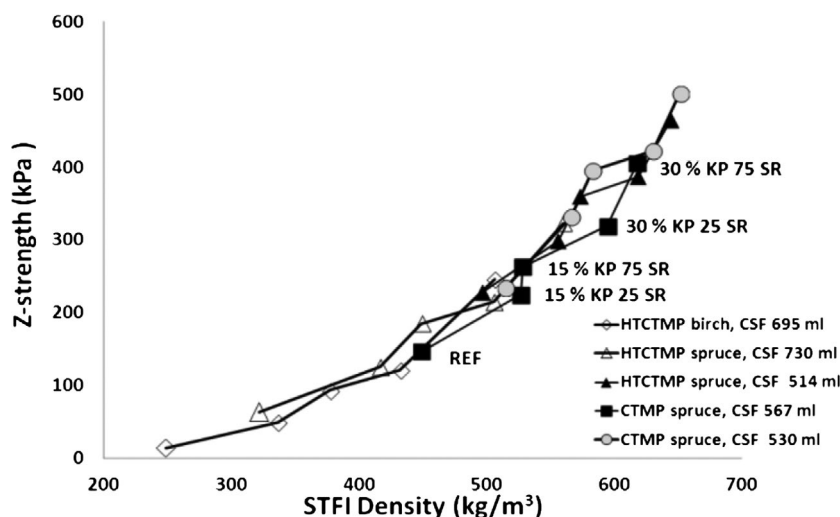
**Figure 25:** Sheet bulk (Tappi sheets) as a function of fibre length at post refining of HTCTMP (CSF = 740 ml) from spruce (Klinga et al. 2008).



**Figure 26:** A: Tensile Index vs. Energy Input in refining; B: Z-strength vs. Bulk in sheets from HC and LC refined spruce HTCTMP (Tappi sheets) (Klinga et al. 2008).

### Out-of-plane strength in CTMP based sheets – effect of energy input in refining and addition of flexible kraft pulp fibres

In the previous section it was shown how the out-of-plane strength of sheets made from CTMP can be improved either by a more intense refining in HC refining or by fibre length reduction, as in LC refining. These measures result in a simultaneous densification of the sheet structure. Admixture of flexible reinforcement chemical fibres is another way to improve out-of-plane strength. The effects of adding softwood kraft pulp fibres in CTMP furnishes are, for instance, shown in a paper by Pettersson et al. 2017,



**Figure 27:** Z-strength vs. density of different CTMP and HTCTMP combined with kraft pulp in 15 % and 30 % ratios. The different additions of kraft pulps are marked for only one pulp. Sheets ( $150 \text{ g/m}^2$ ) made on a Formette Dynamic Sheet Former from Fibertech AB (Pettersson et al. 2017).

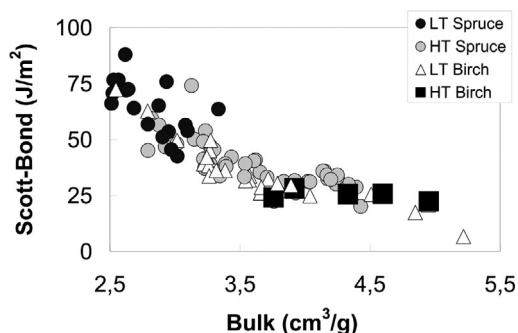
see Figure 27. Five CTMP or HTCTMP, which were chosen to cover a broad range of properties (CSF 530–730 ml), were blended with 15 % or 30 % kraft pulp respectively. Two different degrees of beating of the kraft pulp were tested, 25 SR and 75 SR. The former represents a conventionally used degree of beating for reinforcement pulps, whereas 75 SR represents an extremely tough treatment.

As expected, the best reinforcement effect, in terms of out-of-plane strength, measured as Z-strength, was achieved with the most intensely beaten chemical pulp fibres. These fibres are certainly also those that proved to be the most flexible. The reinforcement effect is marked in Figure 27 for only one pulp, but the effects followed a similar pattern for all the tested CTMP. The effects of making the CTMP pulp fibres more flexible or adding flexible chemical fibres are similar in terms of the effect on the relationship between Z-strength and density. The Scott-Bond, not shown here, develops in a similar manner.

In plies in paperboard composites, it is desirable to increase the Z-strength (i. e. internal bond strength) without simultaneous increase in sheet density. This can obviously not be done either by a more intense refining, fibre length reduction in LC refining, or by adding flexible kraft pulp fibres.

### Out-of-plane strength in CTMP based sheets – comparison of spruce and birch CTMP

Despite the quite different characteristics of spruce and birch fibres and different refining conditions in manufac-



**Figure 28:** Scott-Bond vs. Bulk in a mill trial. ISO sheets (Vesterlind and Höglund 2005a, 2005b).

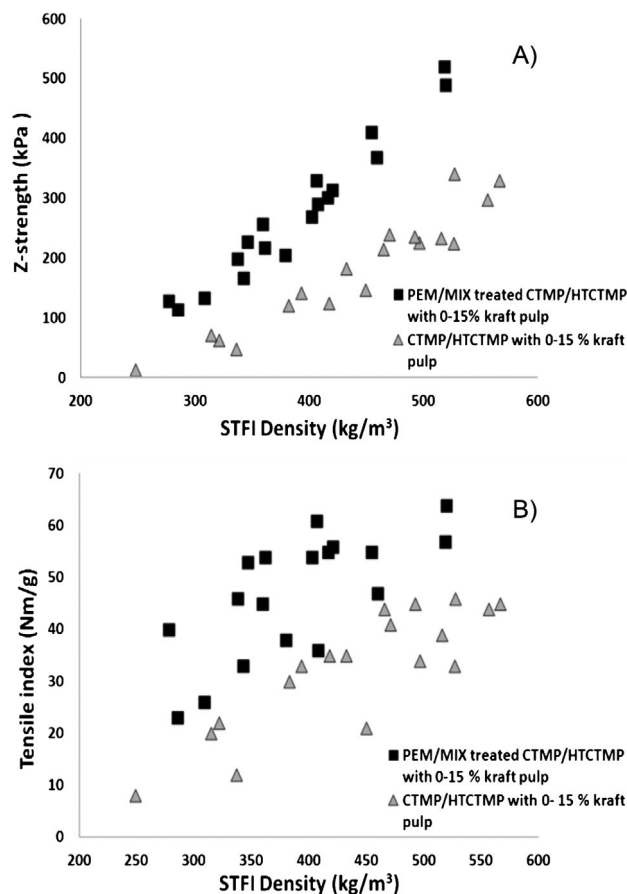
turing, CTMP from spruce and birch are found along almost the same curve in a Scott-Bond vs. bulk plot, Figure 28. Pulps with the label LT are refined after preheating to a temperature close to or below the softening temperature of lignin, whereas pulps labelled HT are preheated at a temperature well above the softening temperature of lignin. The out-of-plane bond strength at a given density is evidently remarkably similar even though fibre surface structures should be quite different considering the different type of wood and chemical and thermal treatments. The most important factor for a certain out-of-plane bond strength is obviously to bring the fibres into close contact at consolidation and pressing of the sheet structure.

Methods that improve out-of-plane bond strength in sheet structures based on HYP without reduction in bulk will be discussed in the following sections.

## Effects of fibre surface modification using mixtures of cationic starch and CMC

It is obviously difficult to change the strong relationship between out-of-plane strength and density in sheets from HYP by changes in pulping processes. However, treatments of fibre surfaces have shown to be very efficient. It is common in papermaking from chemical pulp furnishes to add strength additives to improve the molecular adhesion in fibre/fibre contact zones (Lindström et al. 2005). In numerous studies, the paper strength-enhancing effect of cationic starch has been demonstrated when adsorbed to the negatively charged pulp fibres (Moeller 1966, Howard and Jowsay 1989, Wågberg and Björklund 1993, Wågberg and Kolar 1996). It has been suggested that starch increases both the specific joint strength (Howard and Jowsay 1989), the molecular contact area, and the number of efficient fibre-fibre joints (Moeller 1966). Both in-plane and out-of-plane properties can be considerably improved this way (Eriksson et al. 2005). Furthermore, it has been shown to be possible to improve sheet strength without or with only a slight reduction of bulk at addition of the strength improving agents onto the fibre surfaces, while protecting the inner fibre wall layers from being affected (Wågberg et al. 2002). Especially the polyelectrolyte multi-layering technique (PEM), that is, consecutive treatment with cationic and anionic polyelectrolyte, will provide a significant improvement in both the tensile and Z-strength (Decher 1997). This technique has been shown to be very efficient also to achieve high fibre-fibre bond strength between CTMP fibres at only a minor densification of the sheet (Pettersson et al. 2006, Pettersson 2007). Papers made from high freeness CTMP, with a low amount of bonding fines have a relatively low in-plane and out-of-plane sheet strength. PEM technology has been shown to be very advantageous in creating ductile fibre joints in bulky sheet structures containing a high share of stiff fibres. As an alternative to the PEM technique, a mixture of the same polymers can be applied in a single stage according to a pre-MIX concept (Pettersson et al. 2015). This method also makes it easy to apply the technology in mill scale. This type of treatment is less efficient on HYP aiming for manufacturing of graphic papers, as these pulps contain a high percentage of fines that adsorb too much of applied polymers to be of economic interest (Lundström 2009).

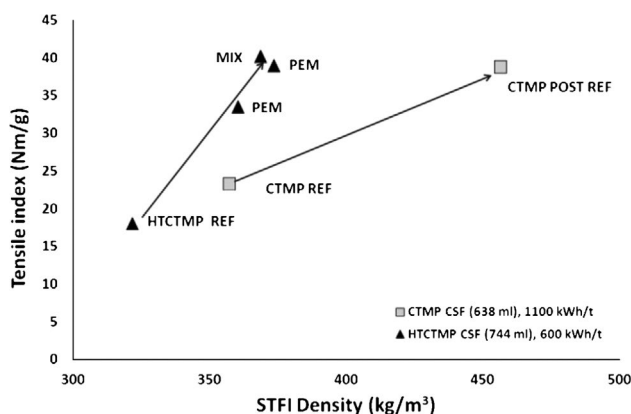
In a study by Pettersson et al. (2017), sheets were made from CTMP and HTCTMP fibres that were surface treated according to the PEM technique, where a multilayer structure consisting of cationic starch/CMC/cationic starch was formed. The CTMP and HTCTMP used are the same as



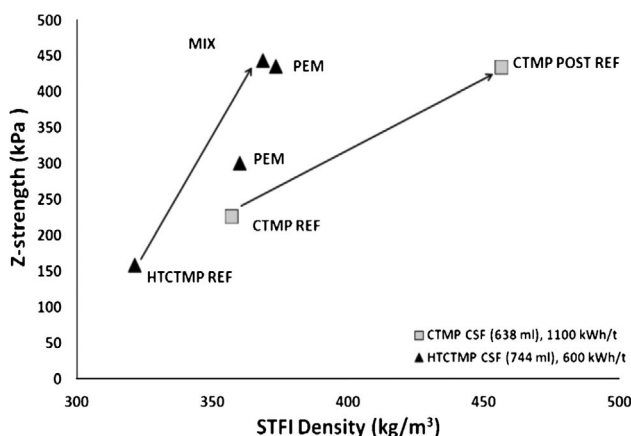
**Figure 29:** A: Z-strength and B: Tensile Index vs. density of different CTMP and HTCTMP with and without PEM/MIX treatments, reinforced with kraft pulp (25 SR) in 0–15 % ratios. Sheets (150 g/m<sup>2</sup>) made on a Formette Dynamic Sheet Former from Fibertech AB (Pettersson et al. 2017).

those in Figure 27. Alternatively, fibre surfaces were treated according to the pre-MIX concept, using the same amount of chemicals but in a single stage of treatment. Strength data for the sheets made from these pulps, with pulps furnished reinforced with 15 % kraft pulps beaten to 25 SR, are presented in Figure 29. It shows that it is possible to double the Z-strength at a certain sheet density after PEM or pre-MIX treatment of CTMP/HTCTMP using 40 kg/ton cationic starch and 2 kg/ton CMC. The effect on Scott-Bond strength, not shown here, is similar to that on Z-strength.

The results, shown in Figure 29, from evaluations on hand-made sheets have been confirmed in a pilot plant trial at Innventia EuroFEX (Pettersson et al. 2015). The significant increase in tensile index with just a minor increase in sheet density, both at additions of starch-CMC-starch as multilayers (PEM) or after a pre-mixing of the polymers (MIX), is shown in Figure 30. The effect of three layers,

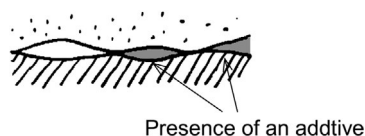


**Figure 30:** Tensile index vs. density of HTCTMP from spruce, with and without PEM/MIX treatments, reinforced with 20 % kraft pulp compared with post-refined CTMP (Pettersson et al. 2015).

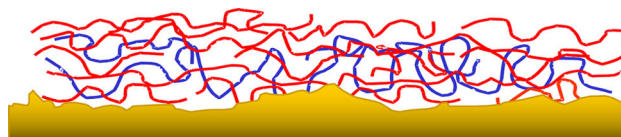


**Figure 31:** Z-strength vs. density of HTCTMP from spruce, with and without PEM/MIX treatments, reinforced with 20 % kraft pulp compared with post-refined CTMP (Pettersson et al. 2015).

containing starch-CMC-starch, is almost exactly the same as at addition of a mixture of the polymers. Obviously, both techniques can be used and will provide the same result if the amount of chemicals is the same. The low consistency (LC) post-refined CTMP at additional energy consumption of about 200 kWh/ton increased the tensile index to almost the same value as for the PEM/MIX treatment but gave an undesirable increase in sheet density. The LC refining appears to affect the tensile index in a manner similar to that of addition of kraft pulp (compare results in Figure 27). The density of sheets from the surface treated HTCTMP-based furnish is about 20 % lower at the same tensile index. Z-strength is improved in a similar way as the tensile index, Figure 31. The sheets made from the HTCTMP treated with 3 layers of starch-CMC-starch or according to the MIX concept had the same Z-strength as the



**Figure 32:** Type V bond in Figure 19.



**Figure 33:** Illustration of three applied layers of polymers on a fibre surface (e. g. starch-CMC-starch).

post-refined reference CTMP, but at a much lower density (approximately 20 %). The refining energy required for the manufacturing of the HTCTMP was only about 600 kW/h as compared to about 1100 kW/h at the high consistency refining of the reference CTMP (standard board quality). The reference pulp was also post refining in a low consistency stage with an energy input of 200 kWh/ton to achieve the same Z-strength as that for the treated HTCTMP. The energy reduction was thus about 50 % in this example to achieve the same Z-strength value.

The results clearly show that it is possible to produce bulky paper structures on a completely new level of in-plane and out-of-plane properties from high freeness CTMP and HTCTMP, which have been surface treated with cationic starch/CMC according to the PEM or pre-MIX technique. Evidently, the fibre-fibre bond strength is considerably improved in such sheet structures.

## Bonding mechanisms between surface modified fibres

The presence of polymer layers on the surfaces of the relatively stiff fibers, illustrated in Figure 32, may contribute to adhesion in at least two ways: additives may give additional contact areas (Type I bond in Figure 19) by filling gaps between fibre surfaces (Type V bond in Figure 19); and/or the additive may in itself form a stronger bond than the pure fibre-fibre adhesion. Obviously, these mechanisms make the fiber-fiber bonds stronger with just a minor effect on sheet density.

To be able to build thick layers of polymers on fibre surfaces, two polyelectrolytes with different charge may be added. They can be formed with the PEM technique for consecutive adsorption as illustrated in Figure 33. The

figure illustrates a simplified view of two polyelectrolytes of opposite charge that have been added one after the other in sequence, e.g. cationic starch – anionic CMC – cationic starch. In this way, it is possible to recharge the fibre several times and get an increased amount of polymer adsorbed onto the fibre surface (Pettersson et al., IMPC 2005). This PEM technique, adding the polyelectrolytes in sequence, gives a mixed bulk containing both components with a surface-layer enriched in the polyelectrolyte added the last time (Decher 1997). Alternatively, differently charged polyelectrolytes can be applied as a mixture. As has been shown in previous sections, both ways give similar strength improvements of the sheet structure.

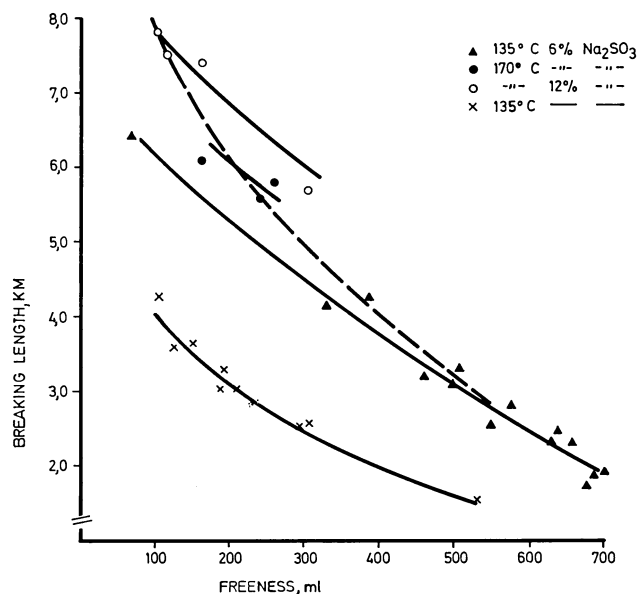
## Strong bonds in packaging papers

### Background and general aspects

In products with very high demands in terms of in-plane strength (i. e. linerboard and fluting), HYP is rarely used, except for in small quantities as in added recycled papers. However, results from recent research, which will be discussed in this section, indicate that it could be feasible to manufacture even extraordinarily strong paper grades from HYP in the future.

HYP sheets with quite high in-plane strength can already be manufactured from softwood as well as hardwoods. High strength sheets from softwood CTMP can be achieved under conventional papermaking conditions, if its fibres are refined to sufficiently high flexibility (Höglund and Bodin 1976, Fredriksson and Höglund 1978, Klinga et al. 2008), Figure 34. However, the refining needed to reach the high fibre flexibility is unfortunately very energy demanding. Therefore, the use of softwood CTMP in paper products with remarkably high requirements on strength has so far not been of interest for economic reasons. To achieve high in-plane strength from hardwoods CTMP, the yield must be reduced to quite low levels (Heitner and Attack 1983, Koran et al. 1984, Granfeldt et al. 2001), which is not attractive from the perspective of environmental concerns. Therefore, to get very high strength sheets from HYP, i. e. close to that from softwood kraft pulps, it is desirable to find new technologies to densify the sheet and achieve high bond strength in a more efficient way than under conventional papermaking conditions.

It is known that the strength of HYP containing papers can be improved significantly by changing the con-



**Figure 34:** Tensile strength (breaking length) of spruce CTMP, manufactured from  $\text{Na}_2\text{SO}_3$ -impregnated chips after preheating to 135 °C or 170 °C. The broken line shows double TMP tensile strength. ISO sheets (Höglund and Bodin 1976).

ventional pressing and drying conditions of papermaking. At press temperatures below or close to the softening temperature of lignin, an enhanced pressure seems to have a somewhat greater impact on sheet density and strength than press temperature (Chagaev et al. 1999, Nygren et al. 2003). However, it has been shown that at densification of CTMP-based moist sheets in a press nip at high pressure, the tensile strength can be improved by at least 10–15 % by increasing the press temperature from 25 to 100 °C (Pettersson et al. 2017). Additionally, the study showed that tensile strength of sheets close to that from kraft pulps can be achieved from a furnish of spruce CTMP fibres that have been surface modified with a mixture of cationic starch/CMC and reinforced with 20 % kraft pulp. Furthermore, it was predicted that a nip press temperature well above the softening temperatures of wet lignin would enhance densification of lignin-rich webs even more.

The positive effect on strength at compression of a wet web of lignin-rich pulps at a temperature well above the softening temperature of lignin in manufacturing of hardboards has been known and applied since the beginning of 1920s (Mason 1927, Asplund 1956). The strengthening effects of press-drying at enhanced temperatures of lignin-containing paper, mostly high-yield kraft pulps, has also been well known for a long time (Setterholm 1979, Setterholm and Koning 1984, Back 1984, Karlsson and Paltakari 2008). In press-drying, papers are dried under restraint due

to an applied pressure in the Z-direction, while the paper is in contact with hot surfaces preferably with temperatures over the softening temperature of wet lignin. At and above this temperature, moist fibre walls of high yield pulps are efficiently softened (see Appendix B). Moist lignin might be tacky and bond strength between fibres with lignin-containing surfaces in close contact is improved (Gupta et al. 1962, Horn 1979, Michell et al. 1983, Pynnönen et al. 2002). Lehtinen developed the press-drying concept into a continuously working process, the so-called Condebelt drying process (Lehtinen 1984, Lehtinen 1995, Schlegel et al. 1999). In this process, the paper web is fed between two steel belts. The belt on top is heated and water cools the belt under the web, where the evaporated steam from the paper web condenses and is transported out of the press-drying unit. For good strength properties, a paper sheet temperature of about 140 °C is reported to be needed on the top belt, i. e. a temperature within the softening temperature range of wet lignin. Typically, sheets are press-dried in the sheet dryness interval 55–80 %. In Impulse Drying (Wahren 1982), another high temperature drying concept that has been in focus at pilot plant trials on and off for a long period, water is removed from the paper web in a hot nip press on the paper machine by the combined action of mechanical pressure and intense heat ( $T > 200$  °C), (Arenander and Wahren 1983, Luciano and Martinez 2001). Solid content in pilot plant studies after an impulse drying unit stage has been reported to be about 65 % (Rigdahl et al. 1999). Sheet strength improvements have been reported from such Impulse Drying experiments as well (Orloff and Sobczynski 1993, Krook et al. 1996).

A common experience from full scale or pilot plant papermaking trials on lignin-containing webs, where a high z-directional pressure is applied in combination with a temperature close to or above the softening temperature of wet lignin, is that paper strength and paper smoothness are significantly improved. Quality improvements are more obvious with furnishes containing pulps with a high content of lignin, like CTMP.

### Effects of moisture content and temperature at consolidation of sheet structures from spruce CTMP

Despite the known effects of temperature and moisture on the viscoelastic properties of hemicellulose and lignin (Goring 1963, Becker and Noack 1968, Höglund et al. 1976, Irvine 1984, Irvine 1985, Salmén 1984), most sheet forming, pressing and drying are made at room temperature in

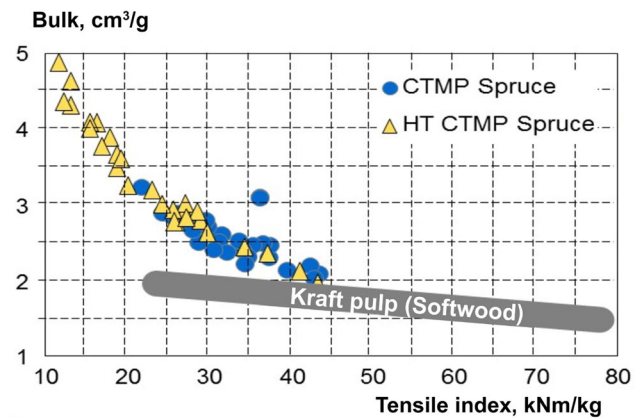
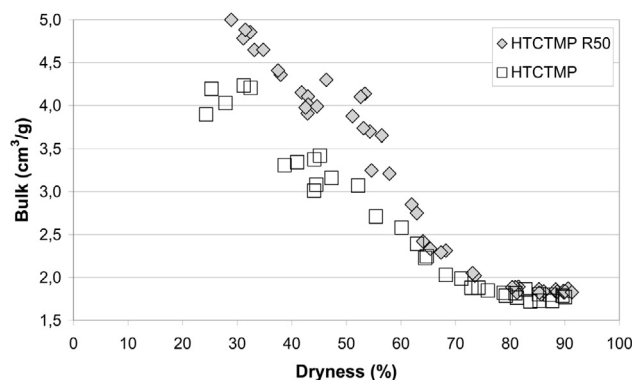


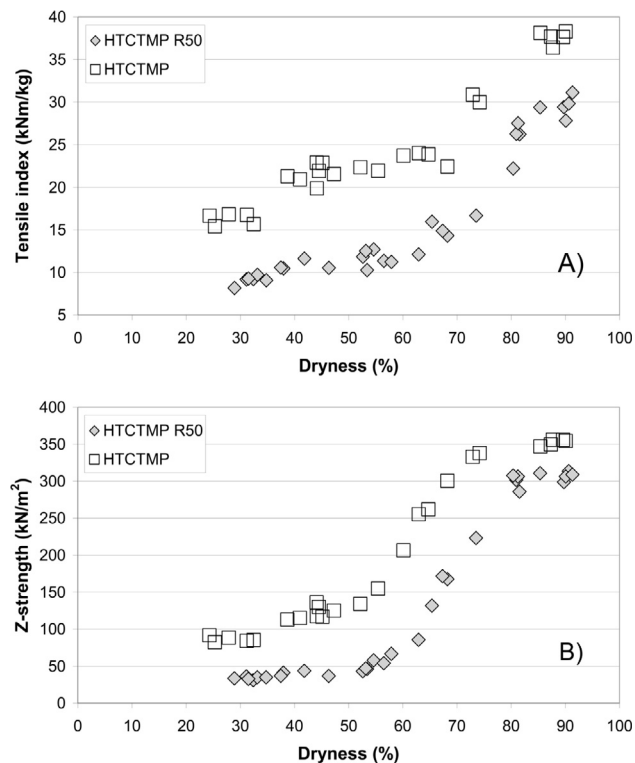
Figure 35: Bulk vs. tensile index for CTMP and HTCTMP from spruce. ISO sheets (Höglund 2002).

laboratory evaluations of sheet properties from HYP. It is obvious that temperature, pressure and moisture content during pressing and drying operations affect the properties of sheets with a high lignin content very differently from sheets that are made from pulps that are free from or have a low content of lignin (Nygren et al. 2003). These effects have been analysed in detail in several studies (Klinga et al. 2005 and 2007, Norgren and Höglund 2011, Norgren et al. 2014a, 2014b). Klinga et al. (2007) studied the effect of moisture content during press-drying at a temperature close to the softening temperature of lignin on sheets from TMP and a HTCTMP that had been manufactured at a very high CSF (699 ml). HTCTMP is characterized by a somewhat higher content of long fibres and a lower content of fines than standard CTMP, Figure 1. Energy consumption to separate fibres and achieve low shives content is very low (about 500–600 kWh/ton), Figure 22. In the manufacturing process chips are preheated to about 175 °C before refining, which means that a major part of the long fibre surfaces is covered with sulfonated lignin. Evaluation according to the ISO standard method ( $T = 25$  °C) of this type of HYP shows that the sheet strength can be exceptionally low and the bulk extremely high, Figure 35.

The press-drying of HTCTMP in the trials by Klinga et al. (2007) was done in a modified Rapid-Köthen dryer at an applied pressure of 400 kPa and a press temperature of 93 °C, i. e. at higher pressure than the standard test method. The dryness values reported in Figures 36–37 were obtained immediately after an initial press-drying stage. After the press-drying stage all sheets were dried in a conditioning room under standard conditions to final dryness before the physical properties were tested. The figures show sheet properties, after conditioning, as a function



**Figure 36:** Bulk (measured on conditioned sheets) at press-drying (pressure 400 kPa and temperature 93 °C) to different dryness of HTCTMP (CSF = 699) and its R50 Bauer-McNett fraction (Klinga et al. 2007).

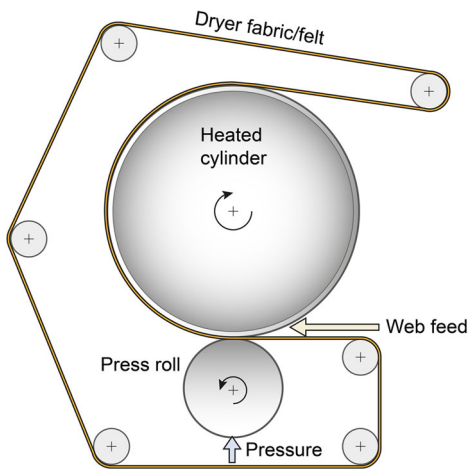


**Figure 37:** Tensile index, A, and Z-strength, B (measured on conditioned sheets) at press-drying (pressure 400 kPa and temperature 93 °C) to different dryness of HTCTMP (CSF = 699) and its R50 Bauer-McNett fraction (Klinga et al. 2007).

of dryness after the initial press-drying stage for both the whole pulp and its long fibre fraction (R50). Even though these fibres are very stiff, the sheets undergo a remarkable densification compared to the densities that were obtained on ISO-sheets (Figure 35) when the sheets were under pressure up to high dryness levels, Figure 36. When

the press-drying phase was terminated at low dryness levels, the fibres seem to spring back to the original shape, whereas the fibres are locked in their positions when the sheets were press-dried to higher dryness levels. When the fibres are forced to be in contact during pressing at the enhanced temperature to a sufficiently high dryness levels, it is possible to achieve significantly improved sheet strength both at in-plane and out-of-plane strength direction in spite of the high content of lignin on the fibre surfaces, Figure 37. Under these press-drying conditions, the development of sheet strength is similar to the development of tensile strength under conventional drying conditions of sheets from sulphite pulps, as has previously been reported by Lyne and Gallay (1954). As chemical pulp fibres are more flexible than fibres from a HYP, chemical fibres are more easily brought into close contact.

Both the in-plane strength, measured as tensile strength, and out-of-plane strength, measured as Z-strength for the HTCTMP sheets are shown in Figure 37. The press-drying stage gave a significant increase in both tensile index and Z-strength for both the whole pulp and its long fibre fraction. A most interesting finding was that strength was not continuously improved with increased dryness, especially at development of Z-strength. There was a distinct inflection of the curve, when dryness reached a dryness level of ~50 %. The most significant dryness interval for internal strength development was found to be between 50 % and 75 % d. c. Similar improvements of sheet strength were achieved also for two tested TMP (CSF = 44 ml and 250 ml), not shown here, even if the change is most pronounced for the HTCTMP. Similar results as reported here were also achieved in studies by Norgren and Höglund (2014). It seems obvious that quite different bulk, in-plane and out-of-plane sheet strengths can be achieved depending on the conditions during drying and pressing of furnishes based on HYP. This is especially significant for HTCTMP, which has been manufactured at a low energy input in refining (i. e. paperboard grade). According to Nygren et al. (2003), the difference in sheet properties for HTCTMP is higher than in sheets from a TMP, which has been manufactured at a high energy input in refining (i. e. of graphic paper grade). The effect of fines is visible in Figures 36–37. As the content of fine material in a paperboard ply from CTMP is low, strong bonds between surfaces from fibres in the long-fibre fraction must be quite common. In a graphic paper made of mechanical pulps, the fine materials are more involved in formation of strong bonds in the paper structure as is described in previous sections, Figures 11 and 12. This means that strong bonds are generated in a completely different way in an original bulky paperboard ply from CTMP with a low content of

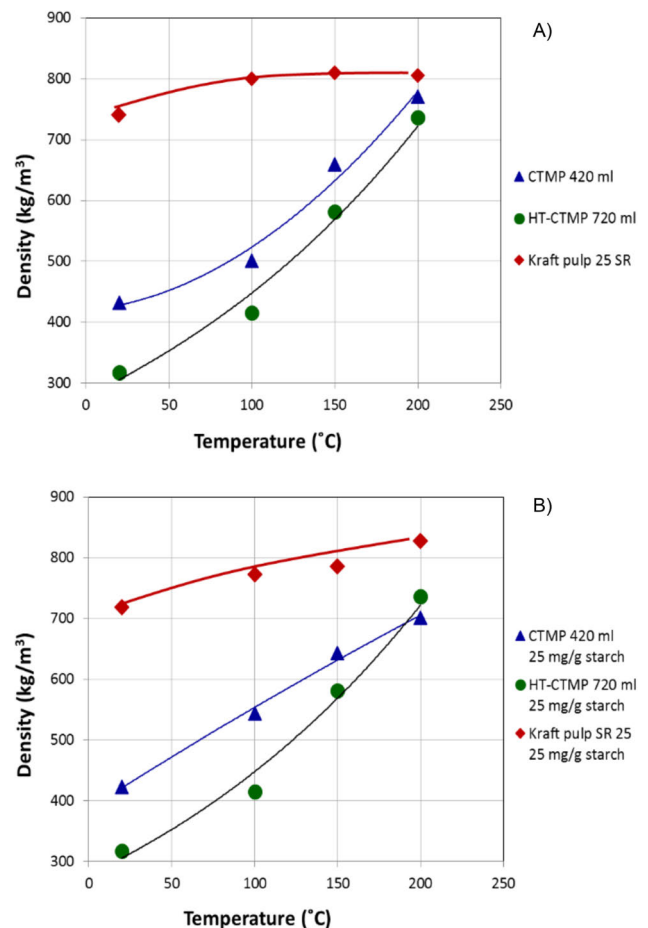


**Figure 38:** The pilot press drying machine. Moist sheets were inserted on a fabric between the press roll and the heated cylinder. The diameter of the cylinder and the press roll was 0.8 and 0.2 metre respectively. The feeding rate was 1 m/min (Norgren et al. 2018).

finer, than in a graphic paper based on mechanical pulp with a high content of fines. In the study by Klinga et al., the effect of pressing and drying at a temperature close to the softening temperature of lignin is visible. Techniques to achieve extremely strong bonds in sheets that are press-dried at temperatures well above the softening temperature range of lignin will be discussed in the following sections.

### High bond strength in sheets from spruce HYP after press-drying at temperatures well above the softening temperature of wet lignin

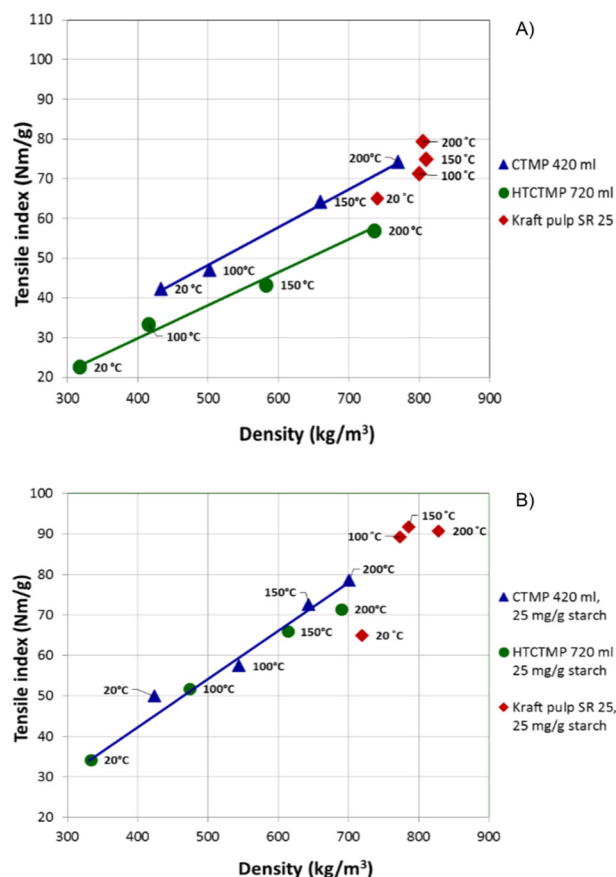
In studies by Norgren et al. (2014a, 2014b) and Joelsson (2020), sheets from HYP were press-dried in a cylinder press, Figure 38, at cylinder temperatures well above the softening temperature of wet lignin. The effects on sheet properties were compared with those from a laboratory beaten bleached kraft pulp. Furthermore, in the study by Norgren et al. (2014a, 2014b), the effect of surface modification of HTCTMP (CSF 729 ml) and CTMP (CSF 420 ml) fibres with just a low dosage of cationic starch was evaluated. The dry content of sheets fed into the press nip was approximately 40 % throughout the whole study. This dry content was selected to be able to demonstrate the full potential of press-drying in the selected temperature range. As shown in Figure 37, sheets are most efficiently consolidated to a fixed structure during press-drying in the dry content interval 50–75 %.



**Figure 39:** Density vs. press nip temperature of sheets made from two different spruce HT-CTMP and CTMP (CSF 720 ml and 420 ml respectively) and a kraft pulp (25 SR). In B) the fibres were treated with 25 mg/g starch (Norgren et al. 2018).

### Densification of sheets from spruce CTMP

The densification effects on sheet structures as a result of increased press nip temperature well above the softening temperature of wet lignin is shown in Figure 39. The effect is very pronounced both for sheets containing HTCTMP and CTMP fibres, i. e. the sheets containing lignin rich fibres, whereas sheets from the kraft pulp are almost unaffected by the press temperature. The relative increase in density is the greatest on sheets from the high freeness HTCTMP, where the density is more than doubled when the press nip temperature is increased from 20 to 200 °C. A sheet density close to that of the kraft pulp sheets is obtained at a press nip temperature of 200 °C. Obviously, the softening of the HYP fibres makes it possible to bring the moist fibre material in close contact, and strong fibre-fibre bonds are formed under the present conditions, i. e. pressure at press nip temperatures well above the softening



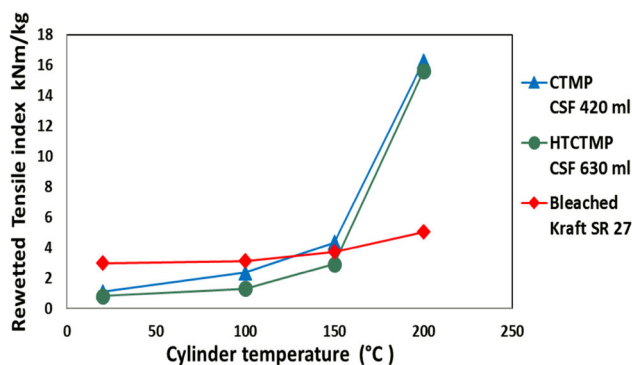
**Figure 40:** Tensile Index versus sheet density of sheets made from two different spruce HT-CTMP and CTMP pulps (CSF 720 ml and 420 ml respectively) and a kraft pulp (25 SR) (Norgren et al. 2018).

ing temperature of wet lignin at suitable moisture content.

After fibre surface modification with cationic starch, the densification effect is very similar to that without surface treatments. The densification is obviously primarily linked to the softening of fibre walls.

### Improvement of dry and wet tensile index of sheets from spruce CTMP

With increased density, due to higher temperature in pressing and drying, the tensile index of HYP sheets are substantially improved, whereas the tensile index of sheets from the kraft pulp is much less affected, Figure 40. Sheets from surface treated CTMP have a tensile strength at the same level as the untreated reference kraft pulp at the highest press temperature. Even sheets from the surface treated HTCTMP reached almost the same level. Sheet strength from the surface treated CTMP and HTCTMP fibres



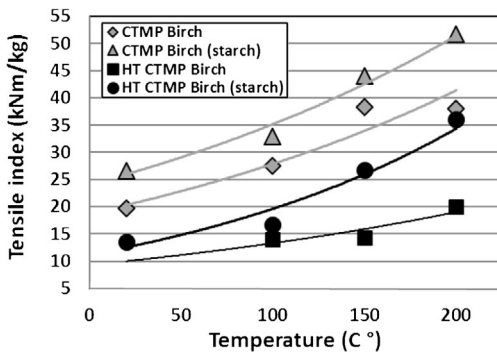
**Figure 41:** Rewetted tensile index for CTMP, HTCTMP from spruce and a softwood bleached kraft pulp. Sheets were soaked 1 minute in water before testing (Höglund et al. 2018).

is on similar levels at equal densities. The bond strength between fibres in the lignin rich sheet structures is obviously very high after the press stage at the highest press temperatures. This might be related to the changed character of the moist lignin, which becomes very tacky at temperatures above the softening temperature of lignin (Gupta et al. 1962). As the number of fibres in the HTCTMP or CTMP webs is just about half of that in the kraft pulp sheets, due to the difference in pulp yields, the strength of fibre-fibre bonds between the lignin rich fibres might be even higher than fibre-fibre bonds between bleached kraft pulp fibres.

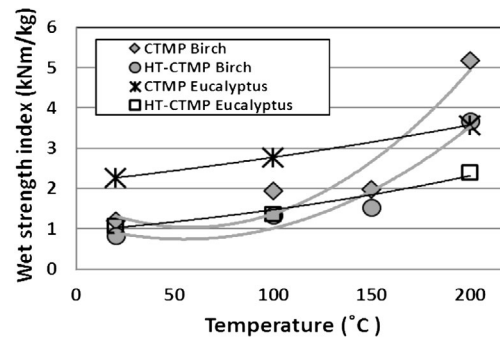
The bond formed between fibres, where the lignin has become very tacky, is also very resistant to moisture, Figure 41, contrary to the fibre-fibre bond in the sheets from the bleached chemical pulp, which was also found by others (Norberg and Back 1968, Swenson et al. 1986, Seth et al. 1985). Obviously, the bonds between the lignin-covered fibres are quite different from the usual bonds in paper materials that are manufactured under conventional paper-making conditions. This will be discussed in the following.

### Strength improvement of sheets from hardwood CTMP

In a 2018 conference paper to Appita, Pettersson et al. aimed to demonstrate the effect on sheet strength of improved fibre-fibre bond strength between HYP fibres from hardwoods. Fibre-fibre bond strength was enhanced both by surface treatments with cationic starch and by pressing in a hot press nip at temperatures well above the softening temperature of wet lignin. Special attention was given to the possibilities of upgrading the strength properties of



**Figure 42:** Tensile index versus cylinder temperature of press-dried sheets made of HT-CTMP and CTMP (CSF 660 ml and 570 ml). In some of the trial points pulps are treated with 25 mg/g starch (Pettersson et al. 2018).



**Figure 43:** Re-wet strength index versus cylinder temperature of press-dried birch and eucalyptus CTMP and HT-CTMP (Pettersson et al. 2018).

very bulky sheet structures containing stiff HYP fibres from hardwoods, which have been manufactured with low energy consumption in refining.

To improve the tensile strength of hardwood pulps, which have been manufactured at a low energy input in refining, consolidation of the sheet structure has to be enhanced. This was done in three ways. Either by fibre surface treatment with cationic starch, by pressing of the sheets in a hot press nip or by a combination of the two measures. Figure 42 illustrate that these treatments can considerably improve the tensile strength. A combination of the two measures is especially efficient. The original tensile index of about 10 kNm/kg was enhanced to 35 kNm/kg for the HTCTMP and from 20 kNm/kg to 50 kNm/kg for the CTMP. The improvements in tensile index can be related to a higher densification of the sheet structure. However, the effects of fibre surface modification were clear even at low contemporaneous densification. CTMP and HTCTMP from eucalyptus urophylla were also studied in this paper, and the results from these pulps followed the same pattern as for birch pulps. The tensile strength after rewetting can also be improved by pressing the hardwood HYP sheets in a hot press nip, Figure 43. However, the improvements are not as impressive as for HYPs from softwoods (Samuelsson et al. 2018). The reason might be the lower content of lignin in combination with a shorter fibre length.

## Bonds between HYP fibres due to lignin and hemicellulose flow

The nature of fibre-fibre bond at press-drying of high yield kraft pulps in the yield range of up to about 70 % has been studied by several authors (Horn 1979, 1988, van Byrd

1979, Michell et al. 1983, Michell 1984, Back 1984, Seth et al. 1985). In these publications the flow of hemicellulose and lignin are discussed as well as feasible interactions between these components. Horn (1979) suggests that hemicellulose, which flows at a lower temperature than lignin, might be primarily responsible for the strength improvement whereas lignin seals the bonds formed by the hemicellulose. This conclusion is also supported in a study of fibre surfaces using ESCA on sheets from press-drying at temperatures below and above the softening temperature of lignin by Koubaa et al. (1996). Seth et al. (1985) investigated properties of press-dried sheets made from pulps that covered a wide range of fibre conformabilities and yields. At the same density or light scattering coefficient, the press-dried sheet had almost the same dry properties as the wet-pressed sheet, which was pressed at room temperature, although there was a trend towards slightly higher tensile strength at press-drying. This indicates that the effect from press-drying is just an extension of the effects of wet pressing. However, sheet densities were obtained more easily in press-drying than by beating or refining. The main effect of press-drying was the significant improvement in strength properties of the wetted sheets. The improvements in wet-strength properties at pressing at high temperatures was the greatest for pulp with the highest content of lignin. They claimed that the increase in wet-strength properties upon press-drying seems to be caused by the flow of the hemicellulose-lignin matrix at the elevated temperature. In a publication from the same authors, they also show that the press-dried sheets had a higher amount of collapse fibres, which give rise to a higher sheet density and higher fibre-fibre contact areas (Michell et al. 1983). A somewhat different result was achieved in an investigation by Lehtonen (2014), showing that light scattering at the same tensile index was significantly higher at wet pressing than at press-drying

at lower pressing pressure than in the investigations by Seth et al. (1985). The lower light scattering at press-drying may be related to the different bonding mechanisms. Similar results as those of Seth et al. (1985) regarding the effect of lignin content on wet strength has been showed by Höglund et al. (2018) and in the thesis by Joelsson (2020).

In press drying at temperatures above the softening temperatures of lignin, factors other than fibre flexibility may control inter-fibre bond formation (Horn 1979). Fibre-fibre bond strength at press-drying of sheets from HYP fibres is certainly closely linked to the softening or flow of lignin. According to Gupta et al. (1962), strong adhesion between lignin surfaces is only possible at temperatures above the softening (tacky) temperature of lignin. The softening of native wet lignin happens within a relative broad span in temperature (Irvine 1985). The sudden improvement in wet strength correlates quite well with the temperature where wood lignin is fully softened, i. e. the transition into the rubbery region (see Appendix B). Some sort of entanglement between softened wood polymers might be part of the mechanism as a result of hemicellulose and lignin flow during pressing at these temperatures. Hydrogen bonds, intermolecular van der Waals bonds or covalent cross-linkage may then be established at high dry content during press-drying (Back 1967, McKenzie 1984, Seth et al. 1985).

McKenzie (1984) has suggested that the diffusion theory proposed by Voyutskii (1963) could be applied to describe the mechanism when strong bonds are formed in press-drying under conditions where hemicellulose and lignin flow. According to this theory, the essential steps would be:

- areas of inter-fibre contacts are enhanced by surface tension forces
- the sheet structure is further compacted in wet pressing to a high density
- microfibrils are drawn in towards the fibre crossings by the retreating water menisci, when remaining water evaporates, and mobile molecular segments from opposing surfaces intermingle
- intermolecular bonds (eventually interchain hydrogen bonds) are finally formed, when moisture content is below a critical level.

The theory may be correct, as it is well in line with experimental findings. However, more studies of the fundamental mechanisms in press-drying of HYP webs is necessary for clearer evidence of the bonding mechanism at press-drying at temperatures well above the softening temperature of lignin.

In summary, the results from the studies of pressing at temperatures well above the softening temperatures of lignin show that there is an as of yet unexploited potential in using spruce HTCTMP and CTMP in manufacturing of products in addition to the traditional ones, i. e. graphic papers and paperboard products, where the requirements on dry and wet strength are very high. Sheet characteristics from HYP webs can be changed within a broad range, as the physical and chemical properties of lignin are marked different at varying temperatures. To be able to form sheets at high densities from HYP webs in a cost-efficient way, it is evident that lignin preferably has to be softened to temperatures well above the tack and softening temperatures of lignin.

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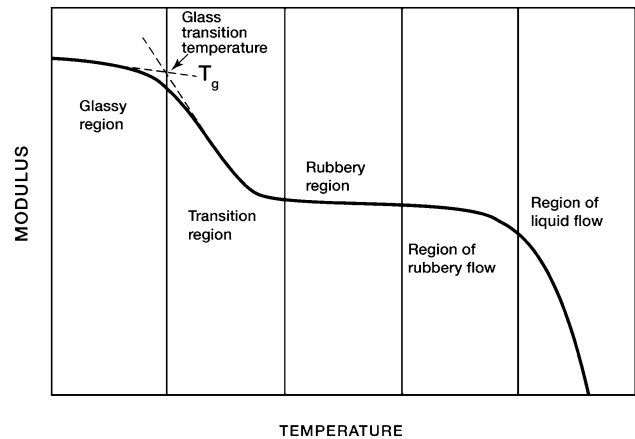
**Funding:** The Kamprad Family Foundation.

**Conflict of interest:** The authors declare that there is no conflict of interest.

## Appendix A. Definitions of terms used in the review

- Adhesion – The state of sticking together, being attached.
- Area in close contact – Area in sufficiently close proximity for intermolecular forces to give adhesion (see Figure 19).
- Bond – Used here in a general sense, the contact zone which holds fibres (or particles) together on a micrometer scale, where at least a fraction of the surface is close enough for attractive interactions to give adhesion. See fibre-fibre bond/joint.
- Bonded area – See area in close contact. Area in sufficiently close proximity for molecular interactions to cause adhesion (McKenzie 1984, Kendall 2001).
- Campbells force – Surface tension force that draw moist solid surfaces, that are free to move, together (Campbell 1959).
- Chemical bond – covalent bond formed in a chemical reaction where molecular groups react. Electron charge distributions of uniting atoms change completely and merge to become something else.

- Entanglement – Intermixing of polymers across the interface between fibre material in close contact.
- Fibre-fibre bond (joint) – Similar to bond, i. e. area where two fibres are joined/bonded together. At least part of this area in sufficiently close proximity to adhere (McKenzie 1984).
- Glass transition phenomena – Rotational or translation motions of polymer segments, which are either directly thermally activated or made possible by the removal of existing restraining bonds (Irvine 1984).
- Hemicellulose flow – Condition where the entanglements in the polymer structure begin to slip (Irvine 1985). The hemicellulose flows at a lower temperature than lignin (Horn 1979).
- Hydrogen bonds – Unusually strong dipole-dipole interaction between hydrogen –H and strongly electronegative atoms such as –O, oxygen (or –N or –F). Requires close proximity and alignment of O–H groups (interacting groups).
- Interlocking – Mechanical interlocking of structural elements too far apart for surface forces to cause molecular adhesion.
- Interdiffusion – See entanglement.
- Joint – Similar to bond.
- Lignin flow – Condition where the entanglements in the polymer structure begin to slip (Irvine 1985).
- Softening temperature – The middle temperature in the transition region between the glassy and rubbery region (see Figure 49). Above the softening temperature the polymer become soft, deformable and sometimes tacky (Gupta et al. 1962).
- Softening temperature of wet lignin – A temperature of about 90–100 °C in softwoods at a loading rate about 1 Hz (Goring 1963, Höglund et al. 1976, Back and Salmén 1982, Irvine 1985). A somewhat lower temperature for hardwoods (Becker et al. 1977, Olsson and Salmén 1997). Softening temperature increases to about 135 °C under conditions like those in a fast press pulses in a paper machine (Williams et al. 1955, Irvine 1985).
- Tack temperature – May, in wood, be analogous with softening temperature (Gupta et al. 1962, Goring 1963).
- Transition region – Region where the polymer stiffness decreases from a glassy to a rubber-like elasticity (Irvine 1985).
- van der Waals bonds – Interactions between permanent and/or induced dipoles. The electron charge distributions are perturbed, but the interacting groups remain discrete entities.



**Figure 44:** Transformation of elasticity for an amorphous, uncross-linked polymer at increased temperature (Irvine 1984).

## Appendix B. Viscoelastic properties of wood and wood components

Wood is a complex composite material of amorphous polymers, lignin and hemicellulose with partly crystalline cellulose as a reinforcement. Wood strength properties are dependent on, among other things, the glass transition of the amorphous components, to an extent determined by their concentrations and structural arrangements in various local entities (Back and Salmén 1982).

The modulus of an amorphous, uncross-linked polymer (like lignin, hemicellulose and the amorphous components in wood cellulose) is strongly affected by temperature. Figure 44 describes the different phases in its transformation from a stiff to a fluent material (Irvine 1984). The definition of glass transition temperature ( $T_g$ ) is visualized in the figure. In technical literature the term “softening temperature” is commonly used. In our paper we refer softening temperature to the inflection point in the transition region between the glassy and rubbery region, i. e. in the middle of the transition region (see Figure 49), where the internal friction reaches its maximum according to tests in a torsional pendulum test equipment (Atack 1972, Höglund et al. 1976), Figure 45.

Water is an efficient softener of lignin and hemicellulose, while cellulose is unaffected by moisture content as well as temperature in the region of interest in papermaking, as illustrated in Figure 46. As more moisture can be absorbed in the hemicellulose structure than in lignin, the softening temperature of wet hemicellulose is much lower than in wet lignin (illustrated in Figure 47). This means

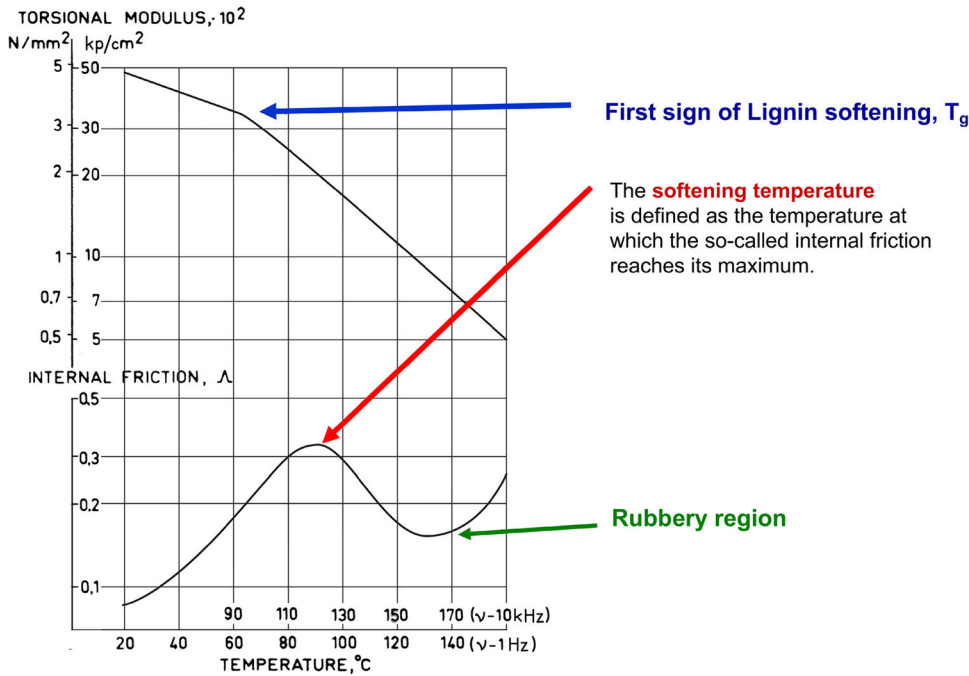


Figure 45: Softening of spruce wood in a saturated steam environment. Adapted from Höglund et al. 1976.

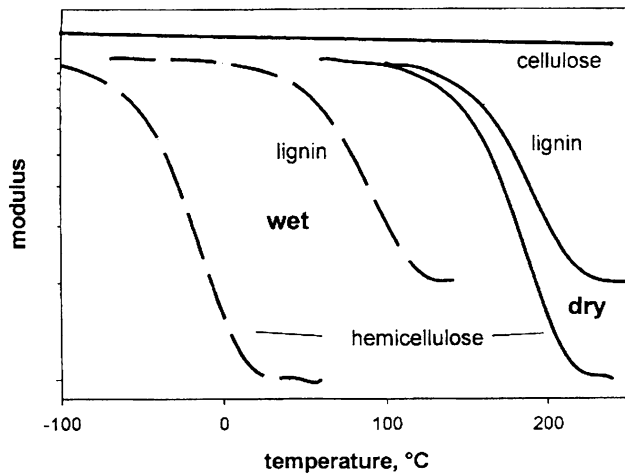


Figure 46: Softening of dry and wet lignin and hemicellulose at a low loading rate. Based on (Salmén 1982).

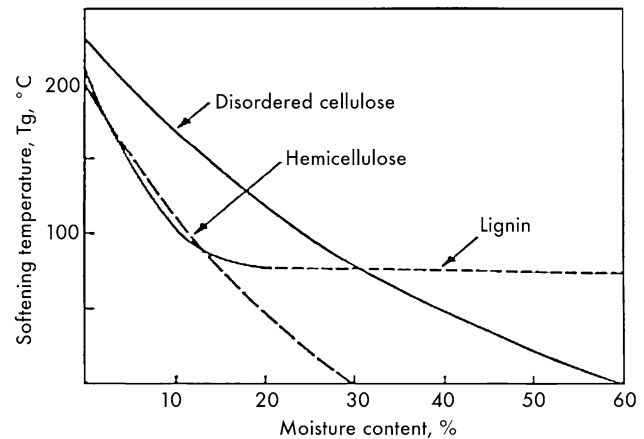


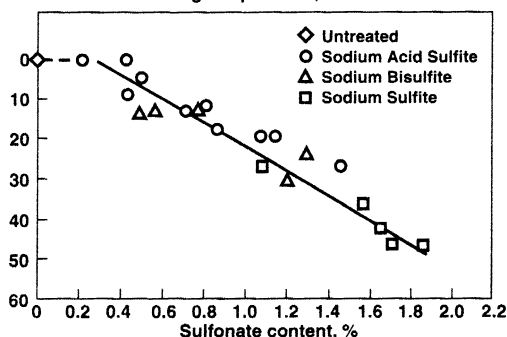
Figure 47: Softening temperature of lignin, hemicellulose and disordered cellulose at a low loading rate (Salmén 1990).

that the stiffness of wood and pulp fibres with a high content of lignin (HYP) to a high extent is controlled by the properties of lignin.

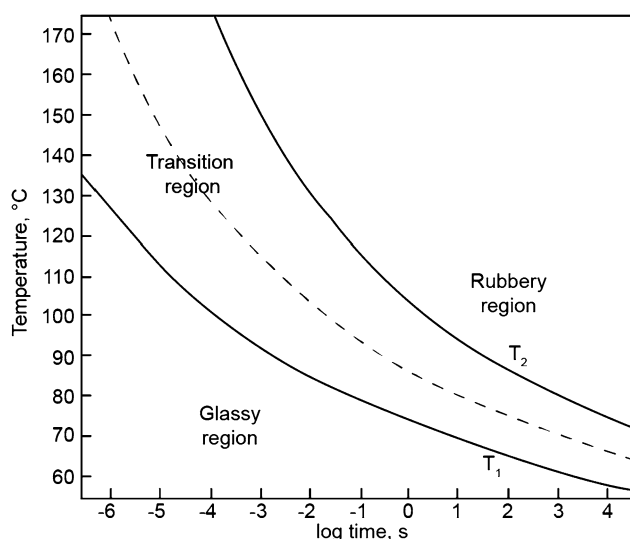
The softening of lignin can be changed by impregnation with chemicals. In the manufacturing of different kinds of CTMPs this is preferably made through sulfonation of the lignin at pre-treatment before the refining stage, Figure 48.

In addition to the influence of temperature, moisture and additions of chemicals, the viscoelastic properties of HYP fibres are also a function of time, i. e. loading rate or loading frequency (Bueche 1962, Irvine 1985). The dependence on wood/lignin modulus, i. e. the stiffness status, of temperature and loading frequency is summarized in Figure 49.

Decrease of softening temperature, °C



**Figure 48:** Decrease of softening temperature with sulphonation of wood lignin at a low loading rate (Heitner and Attack 1983).



**Figure 49:** The effect of loading frequency on the elasticity of softwood.  $T_1$  shows the change of glass transition temperature and  $T_2$  the change of the temperature, where wood/lignin is change-over into the rubbery form. Rubbery flow is assumed to occur at a temperature above the  $T_2$  border line. The broken line marks the development of softening temperature. Redrawn from Irvine 1985.

## References

- Alinec, B. (1986) Light-scattering of pigments in papermaking. *Pap. Puu* 68(8):545–547.
- Andersson, M. (1981a) Z-strength in pulp characterization – Chemical pulps. *Sven. Papp.tidn.* 84(3):R6–R14.
- Andersson, M. (1981b) Aspects of z-strength in pulp characterization. *Sven. Papp.tidn.* 84(6):R34–R42.
- Andersson, M., Mohlin, U.-B. (1980) Z-strength of mechanical pulps. *Pap. Puu* 62(10):583–605.
- Arenander, S., Wahren, D. (1983) Impulse drying adds new dimension to water removal. *Tappi J.* 66(9):123–126.
- Asplund, A.J.A. (1956) The beginning and development of the fibre board industry. *Sven. Papp.tidn.* 59(12):441–448.
- Attack, D. (1972) On the characterization of pressurized refiner mechanical pulps. *Sven. Papp.tidn.* 75(3):89–94.
- Axelsson, P., Simonsson, R. (1982) Thermomechanical pulping with low addition of sulfite. Part 1. Effects of mild sulfite treatment of spruce chips prior to defibration. *Sven. Papp.tidn.* 85(15):132–139.
- Axelsson, P., Simonsson, R. (1983) Thermomechanical pulping with low addition of sulfite. Part 4. A mill scale trial. *Sven. Papp.tidn.* 86(86):149–151.
- Back, E.L. (1967) Thermal Auto-Crosslinking in Cellulose Material. *Pulp Pap. Can.* 10:T165–T171.
- Back, E.L. (1984) A review of press drying. In: OFE meeting Berlin, sept 1984 and in STFI-Tech. Report D:24.
- Back, E.L., Salmén, L. (1982) Glass transitions of wood components hold implications for molding and pulping processes. *Tappi J.* 65(7):107–110.
- Beath, L.R., Neill, M.T., Masse, F.A. (1966) Latency in Mechanical Wood Pulps. *Pulp Pap. Can.* 67(10):T423–T430.
- Becker, H., Höglund, H., Tistad, G. (1977) Frequency and temperature in chip refining. *Pap. Puu* 59(3):123–130.
- Becker, H., Noack, D. (1968) Studies on Dynamic Torsional Viscoelasticity of Wood. *Wood Sci. Technol.* 2(3):213–230.
- Bengtsson, G. (2005) CTMP in production of high-quality packaging board. In: *Int. Mech. Pulp. Conf.*, Oslo, Norway. pp. 7–13.
- Brecht, W., Klemm, K. (1953) The Mixture of Structures in a Mechanical Pulp as a Key to the knowledge of its Technological Properties. *Pulp Pap. Can.* 1(52):72–80.
- Bueche, F. *Physical Properties of Polymers*. Interscience, New York, 1962.
- Campbell, W.B. (1959) The mechanism of Bonding. *Tappi J.* 42(12):999–1001.
- Chagaev, O., Stationwala, M.I., Allen, R. (1999) The role of fiber collapse in Mechanical Pulping. In: *Int. Mech. Pulp. Conf.*, Houston, USA. pp. 155–169.
- Clark, J.d.A. (1944) The ultimate strength of pulp fibers and the zero-span tensile test. *Paper Trade J.* 118(1):29–34.
- Corson, S. (1979) Thermomechanical Pulp Properties at a Function of the Fibre and Fines Fractions. In: *Int. Mech. Pulp. Conf.*, Toronto. pp. 49–56.
- Corson, S.R. (1996) Development of TMP fibre and pulp properties. *Appita J.* pp. 525–531.
- Corson, S.R. (1997) Tree and fibre selection for optimal TMP quality. In: *Int. Mech. Pulp. Conf.*, Stockholm, Sweden. pp. 231–240.
- Corson, S.R., Wakelin, R.F., Lloyd, M.D. (1997) The furnish development strategies. Part II: Sheet properties. *Pulp Pap. Can.* 98(1):41–44.
- de Silveira, G., Zhang, X., Berry, R., Wood, J.R. (1995) Location of fines in mechanical pulp handsheets using scanning electron microscopi. In: *Int. Mech. Pulp. Conf.*, Ottawa, Canada. pp. 85–92.
- Decher, G. (1997) Fuzzy nano-assemblies: Toward layered polymeric multi-composites. *Science* 277(5330):1232.
- El-Hosseiny, F., Bennett, K. (1985) Analysis of the zero-Span Tensile Strength of Paper. *J. Pulp Pap. Sci.* 11(4):J121–J127.
- Engstrand, P., Ferritsius, R., Rundlöf, M., Paulsson, M. (2019) Decrease in strength along a process line for SC paper. In: *Fundamental Mechanical Pulp Research Seminar, FMPS2019*, Norrköping, Sweden.
- Engstrand, P., Ottestam, C., Salmén, L., Pettersson, R., Sjogren, B., Htun, M. (1991b) In: *Properties of ionic polymers, natural and*

- synthetic. Eds. Salmén, L., Htun, M. STFI Stockholm. pp. 157.
- Engstrand, P., Sjogren, B., Olander, K., Htun, M. (1991c) In: Proc. 6th Int. Symp. on Wood and Pulping Chem., Melbourne. Vol. I. p. 75.
- Engstrand, P.O., Karlström, A., Nilsson, L. (1995) The impact of chemical addition on refining parameters. In: Int. Pulp. Conf., Ottawa/St Paul, Canada. pp. 281–286.
- Engstrand, P.O., Pettersson, R., Htun, M., Hammar, L.Å. (1991a) Method of reducing the energy consumption at the refining of cellulose containing material. U.S. Patent No. 5,007,985. 16 Apr. 1991.
- Eriksson, I., Haglund, I., Lidbrandt, O., Salmén, L. (1991) Fiber swelling favoured by lignin softening. *Wood Sci. Technol.* 25(2):135–144.
- Eriksson, M., Pettersson, G., Wågberg, L. (2005) Application of polymeric multilayers of starch onto wood fibres to enhance strength properties of paper. *Nord. Pulp Pap. Res. J.* 15(4):256–265.
- Fellers, C., de Ruvo, A., Htun, M., Carlsson, L., Engman, C., Lundberg, R. Profitable use of pulps and processes, Carton Board. STFI, Stockholm, 1983.
- Ferritsius, O., Ferritsius, R., Reyier, S. (2009) The influence of process design on the distribution of fundamental fibre parameters. In: Int. Mech. Pulp. Conf., Sundsvall, Sweden. pp. 160–168.
- Fineman, I. (1985) Let the paper product guide the choice of mechanical pulp. In: Int. Mech. Pulp. Conf., Stockholm. pp. 203–214.
- Fjellström, H., Engstrand, P., Htun, M. (2013) On the relationship between wood fibre wall swelling, charged groups, and delamination during refining. *J-FOR* 3(4):30–34.
- Forgacs, O. (1963) The Characterization of Mechanical Pulps. *Pulp Pap. Can.* 64(C):T89–T118.
- Franzen, R., Li, K. (1985) Upgrading thermomechanical pulp by selective long fibre treatment. *Appita J.* 38(3):195–204.
- Fredriksson, B., Höglund, H. (1978) Chemimechanical pulp in different paper grades. *Appita J.* 31(5):365–370.
- Giertz, H.C. (1973) Basic difference between mechanical and Chemical pulps. In: Int. Mech. Pulp. Conf., Session II, Stockholm, Sweden. pp. 6:1.
- Giertz, H.C. (1977) Basic raw material properties and their significance in mechanical pulping. In: Int. Mech. Pulp. Conference, Session I, Helsinki, Finland. pp. 37–46.
- Goring, D. (1963) Thermal Softening of Lignin, Hemicellulose and Cellulose. *Pulp Pap. Can.* 64(12):T517–T527.
- Granfeldt, T., Falk, B., Denton, B., Åkerlund, G. (2001) Aspen CTMP-where are we now and where is it heading? In: Int. Mech. Pulp. Conf., Helsinki, Finland. pp. 315–327.
- Gummerus, M. (1983) Possibilities of upgrading TMP by sulphite treatment of the screen rejects. *Pap. Puu* 65(4):216–223.
- Gupta, P.R., Pezanowich, A., Goring, D. (1962) The Adhesive Properties of Lignin. *Pulp Pap. Can.* 63(1):T21–T31.
- Hammar, L.-Å., Htun, M., Ottestam, C., Salmén, L., Sjögren, B. (1995) Utilising the ionic properties of wood polymers in mechanical pulping. In: Int. Mech. Pulp. Conf., Ottawa, Canada. pp. 183–187.
- Hedberg Hu, S., Otterstam, C., Salmén, L., Svensson, B. (1999) Refining of early- and latewood. STFI report TF 141, STFI Stockholm, Sweden.
- Heikkurinen, A., Hattula, T. (1993) Mechanical pulp fines – characterization and implications for defibration mechanisms. In: Int. Mech. Pulp. Conf., Oslo. pp. 294–308.
- Heikkurinen, A., Leskelä, L. (1999) The character and properties of mechanical pulps. In: Papermaking Science and Technology, Book 5. Eds. Gullichsen, J., Paulalolo, H. Fapet Oy, Helsinki, Finland. pp. 395–413.
- Heitner, C., Atack, D. (1983) Ultra-high-yield pulping of aspen, effects of ion content. *Pulp Pap. Can.* 84(11):365–370.
- Heitner, C., Hattula, T. (1988) Ultra-High Yield Pulping. Part VI; The effect of Sulphonation on the Development of Fiber Properties. *J. Pulp Pap. Sci.* 14(1):J16.
- Heitner, C., Karnis, A., Atack, D. (1983) Ultrahigh yield pulping – Part IV. High strength pulp from mechanical pulp rejects. In: Int. Mech. Pulp. Conf., Washington D.C., USA. pp. 27–34.
- Hirn, U., Schennach, R. (2015) Comprehensive analysis of individual pulp fiber bonds quantifies the mechanisms of fiber bonding in paper. *Sci. Rep.* 5:10503. 10.1038/srep10503.
- Honkasalo, J., Knuts, K., Laine, J., Nygren-Konttinen, A. (1983) The fines fraction of mechanical pulp – A literature review. *Pap. Puu* 64(11):693–699.
- Horn, R.A. (1979) Bonding in press-dried sheets from high-yield pulps, The role of lignin and hemicellulose. *Tappi J.* 69(7):77–80.
- Horn, R.A. (1988) Wet tensile strength of press-dried paperboard. In: Tappi Engineering Conference. pp. 435–439; Int. Mech. Pulp. Conf., Helsinki, Finland. pp. 362–375.
- Howard, R.C., Jowsay, C. (1989) The effect of cationic starch on the tensile strength of paper. *J. Pulp Pap. Sci.* 15(6):225–229.
- Htun, M., Engstrand, P., Salmén, L. (1987) The implication of lignin softening on the latency removal of mechanical and chemimechanical pulps. In: Int. Mech. Pulp. Conf., Helsinki, Finland. pp. 61–66.
- Höglund, H. (2002) Mechanical Pulp Fibers for New and Improved Paper Grades. In: 7<sup>th</sup> International Conference on new available technologies, Stockholm, Sweden. pp. 158–163.
- Höglund, H., Bodin, O. (1976) Modified thermo-mechanical pulp. *Sven. Papp.tidn.* 79(11):343–347.
- Höglund, H., Bäck, R., Falk, B., Jackson, M. (1997) Thermopulp – a new energy-efficient mechanical pulping process. *Pulp Pap. Can.* 98(6):T215–TT222.
- Höglund, H., Pettersson, G., Norgren, S., Engstrand, P. (2018) A paper or paperboard product comprising at least one ply containing high yield pulp and its production method. Swedish Patent: SE 540115 C2.
- Höglund, H., Sohlén, U., Tistad, G. (1976) Physical properties of wood in relation to chip refining. *Tappi J.* 59(6):144–147.
- Höglund, H., Wilhelmsson, K. (1993) The product must determine the choice of wood in mechanical pulping. In: Int. Mech. Pulp. Conf., Oslo. pp. 1–22.
- Ionides, G. (2003) Adding value for improved market competitiveness of mechanical printing papers. In: Int. Mech. Pulp. Conf., Quebec City, Canada. pp. 9–13.
- Irvine, G.M. (1984) The glass transition of lignin and hemicellulose and their measurement by differential thermal analysis. *Tappi J.* 67(5):118–121.
- Irvine, G.M. (1985) The Significance of the Glass Transition of Lignin in Thermomechanical Pulping. *Wood Sci. Technol.* 19:139–149.
- Israelachvili, J.N. Intermolecular and surface forces. 2nd ed. Academic Press, San Diego, CA, USA, 1997, ISBN 0-12-375181-0.
- Jackson, M. (1988) High yield pulps from Canadian hardwoods.

- Nord. Pulp Pap. Res. J., Special Issue.
- Jackson, M., Falk, B., Moldenius, S., Edström, A. (1988) The manufacture and end-use potential of high-yield pulp from eucalyptus. *Pulp Pap. Can.* 89(10):79–86.
- Jackson, M., Williams, G. (1979) Factors Limiting the Strength Characteristics of Thermomechanical Pulp. In: *Int. Mech. Pulp. Conf.*, Toronto, Canada. pp. 37–48.
- Jang, H.F., Seth, R.S. (1998) Using confocal microscopy to characterize the collapse behaviour of fibres. *Tappi J.* 81(5):167–174.
- Jang, H.F., Weigel, G., Seth, R.S., Wu, C.B. (2001) The effect of fibril angle on the transverse collapse of papermaking fibres. In: *Int. Mech. Pulp. Conf.*, Part 1, Helsinki. pp. 27–33; *Pap. Puu* 84(2):112–115.
- Joelsson, T. High strength paper from high yield pulps by means of hot-pressing. Licentiate thesis 167. Mid Sweden University, 2020, ISBN 978-91-88947-39-0.
- Johnson, K.L., Kendall, K., Roberts, A.D. (1971) Surface Energy and the Contact of Elastic Solids. *Proc. R. Soc. Lond. Ser. A* 324(1558):301–313.
- Kallmes, O., Perez, M. (1966) New theory for the load-elongation properties of paper. In: *Consol. Pap. Web, Trans. Symp.* pp. 779–800.
- Kangas, H., Pöhler, T., Heikkurinen, A., Kleen, M. (2004) Development of mechanical pulp fibre surfaces as a function of refining energy. *J. Pulp Pap. Sci.* 11(30):294–308.
- Karlsson, M., Paltakari, J. (2008) Introduction to paper drying and its principles. In: *Papermaking Science and Technology, Book 9, (Papermaking Part 2, Drying)*, Fapet Oy, Helsinki, Finland. Chapter 1. pp. 27–31.
- Karnis, A. (1994) The Mechanism of Fiber Development in Mechanical Pulping. *J. Pulp Pap. Sci.* 10(20):J280–J288.
- Karnis, A. (1996) The role of latent and delatent mechanical pulp fines in sheet structure and pulp properties. *Pap. Puu* 77(8):491–497.
- Kartovaara, I. (1989) Mechanical pulp as coating base paper raw material. Strength, weaknesses and development needs. In: *Int. Mech. Pulp. Conf.*, Part 1, Helsinki, Finland. pp. 25–35.
- Katz, S., Liebergott, N., Scallan, A.M. (1981) A mechanism for alkali strengthening of mechanical pulps. *Tappi J.* 64(7):97–100.
- Kendall, K. Molecular addition and its application. Kluwer Academic/Plenum publishers, New York, USA, 2001, ISBN 0-306-46520-5.
- Klinga, N. The Influence of Fibre Characteristics on Bulk and Strength Properties of TMP and CTMP from Spruce. Licentiate thesis 23. Mid Sweden University, 2007, ISBN 978-91-85317-52-3.
- Klinga, N., Höglund, H., Sandberg, C. (2007) Paper properties of TMP and CTMP handsheets from spruce, as affected by solid content from pressing. *Nord. Pulp Pap. Res. J.* 22(4):475–480.
- Klinga, N., Höglund, H., Sandberg, C. (2008) Energy efficient high quality CTMP for paperboard. *J. Pulp Pap. Sci.* 34(2):98–106.
- Klinga, N., Sandberg, C., Höglund, H. (2005) Sheet properties of high yield pulp related to different pressing and drying conditions. In: *Int. Mech. Pulp. Conf.*, Oslo, Norway. pp. 344–348.
- Koljonen, K. Effect of surface properties of fibres on some paper properties of mechanical and chemical pulp. Doctoral thesis. Helsinki University of Technology, Helsinki, Finland, 2004, ISSN 1795-2409.
- Koran, Z., Lo, S.N., Valade, J.L. (1984) Strength properties of birch and aspen sulphite pulps in the yield range of 77–94 %. *Pulp Pap. Can.* 85(2):39–41.
- Koubaa, A., Riedl, B., Koran, Z. (1996) Surface Analysis of Press Dried CTMP Paper Samples by Electron Spectroscopy for Chemical Analysis. *J. Appl. Polym. Sci.* 61(3):545–552.
- Krook, R., Stenström, S., Hollmark, H. (1996) Some effect of press drying on paper properties. *Nord. Pulp Pap. Res. J.* 11(3):141–151.
- Kure, K.-A. (1997) The alteration of wood fibres in refining. In: *Int. Mech. Pulp. Conf.*, Stockholm, Sweden. pp. 137–150.
- Kure, K.-A. On the relationship between process input variables and fibre characteristics in thermomechanical pulping. Doktor ingenioravhandling 1999:45. NTNU, Trondheim, Norway, 1999.
- Lehtinen, J.A. (1984) Method of continuous drying of a paper or other porous web and a drying device for applying this method, USA Patent No. 4,461,095.
- Lehtinen, J.A. (1995) Condebelt drying: Quality results and process development. *Pap. Technol.* 36(10):67–71.
- Lehtonen, L.K. Elucidating the nature of bonding in mechanical pulps. Doctoral thesis. Institute of Paper Science and Technology, Atlanta, Georgia, USA, 2014.
- Levlin, J.-E. (1999) Aim of pulp and paper testing. *Pap. Sci. Technol.* 17:11–17.
- Lindblad, G., Iversen, T., Bergenblad, H., Rigdahl, M. (1989) Light scattering ability of coatings as a tool to protect paper and board against photo-oxidation. *Nord. Pulp Pap. Res. J.* 4(4):253–257.
- Lindholm, C.-A. (1980a) Comparison of some papermaking properties of groundwood, pressure groundwood and thermomechanical pulp by means of artificial blends of pulp fractions. Part 1. Primary results. *Pap. Puu* 62(10):593–600.
- Lindholm, C.-A. (1980b) Comparison of some papermaking properties of groundwood, pressure groundwood and thermomechanical pulp by means of artificial blends of pulp fractions. Part 2 The fines fractions. *Pap. Puu* 62(12):803–808.
- Lindholm, C.-A. (1981) Comparison of some papermaking properties of groundwood, pressure groundwood and thermomechanical pulp by means of artificial blends of pulp fractions. Part 3. The fiber fractions. *Pap. Puu* 63(8):487–492, 495–497.
- Lindholm, C.-A. (1984) Determining optimum combinations of mechanical pulp fractions – Part 3. Effect of chemical modification of the long fibre fraction. *Pap. Puu* 66(11):657–665.
- Lindholm, C.-A., Kurdin, J.A. In: *Mechanical Pulping, Book 5*. Ed. Sundholm, J. 1999, ISBN 952-5216-05-5. pp. 76–77 and 227.
- Lindström, T. (1992) Chemical factors affecting the behaviour of fibres during papermaking. *Nord. Pulp Pap. Res. J.* 7(4):181–192.
- Lindström, T., Wågberg, L., Larsson, T. (2005) On the nature of joint strength in paper – A review of dry and wet strength resins used in paper manufacturing. In: *13<sup>th</sup> Fundamental Research Symposium*, Cambridge, UK. pp. 457–562.
- Luciano, M.F.C., Martinez, D.M. (2001) On the characterization of the delamination process during impulse pressing. *Nord. Pulp Pap. Res. J.* 16(4):362–368.
- Lundström, L. Polyelectrolyte multilayers of cationic and anionic starch and their use for improving the strength of papers made from mechanical pulps. Licentiate thesis. KTH, Department of Fibre and Polymer Technology, 2009, ISBN 978-91-7415-294-4.

- Luukko, K. (1998) On the characterization of mechanical pulp fines. A review. *Pap. Puu* 80(6):441–448.
- Luukko, K. Characterization and Properties of Mechanical Pulp Fines. Chemical Technology Series, No. 267. Acta Polytechnica Scandinavica, 1999, ISSN 1239-0518.
- Luukko, K., Paulapuro, H. (1999) Mechanical pulp fines: Effect of particle size and shape. *Tappi J.* 82(2):95–101.
- Lyne, L.M., Gallay, W. (1954) Studies in the Fundamentals of Wet Web Strength. *Tappi J.* 12(37):698–704.
- Mannström, B. (1968) On the characterization of mechanical pulps. In: *Int. Mech. Pulp. Conf., Atlanta, USA.* p. 35.
- Marton, R., Alexander, S.D. (1963) Properties of Fiber Fractions from Chemical and Mechanical Pulps. *Tappi J.* 46(2):65–70.
- Mason, W.H. (1927) Pulp and paper from steam exploded wood. *Paper Trade J.* 84(8):131–136.
- McDonald, D., Miles, K., Amiri, R. (2003) The nature of the mechanical pulping process. In: *Int. Mech. Pulp. Conf., Quebec City, Canada.* pp. 1–8.
- McDonough, T.S., Aziz, S., Rankin, K.L. (1987) The Strength of Mechanical Pulp Fibers. In: *Inst. Paper Chem., Wisconsin, USA. IPC Technical Paper Series.* p. 7.
- McKenzie, A.W. (1984) The structure and properties of paper. Part XXI: The diffusion theory of adhesion applied to interfiber bonding. *Appita J.* 37(7):580–583.
- Michell, A.J. (1984) Press drying of paper – An overview. *Appita J.* 37(4):325–329.
- Michell, A.J., Seth, R.S., Page, D.H. (1983) The effect of press-drying on paper structure. *Pap. Puu* 65(9):536–541.
- Moeller, H. (1966) Cationic starch as wet end strength additive. *Tappi J.* 49(5):211–214.
- Mohlin, U.-B. (1979a) The contribution of different pulp fractions to the latency effect in thermomechanical pulps. In: *Int. Mech. Pulp. Conf., Toronto, Canada.* pp. 13–23.
- Mohlin, U.-B. (1979b) Properties of TMP Fractions and the Importance for the quality of Printing Papers. In: *Int. Mech. Pulping Conf., Toronto, Canada.* pp. 57–84.
- Mohlin, U.-B. (1997) Fibre Development During Mechanical Pulp Refining. *J. Pulp Pap. Sci.* 1(23):J28–J33.
- Moldenius, S. Some aspects of hydrogen peroxide bleaching of mechanical pulps. Doctoral thesis. Royal Institute of Technology, Stockholm, Sweden, 1983, ISBN 99-0363040-9.
- Moss, P.A., Retulainen, E. (1997) The effect of fines on fibre bonding: Cross-sectional dimensions of TMP fibres at potential bonding sites. *J. Pulp Pap. Sci.* 23(8):J382–J388.
- Nissan, A.H., Sternstein, S.S. (1964) Cellulose-fiber bonding. *Tappi J.* 47(1):1–6.
- Norberg, K.G., Back, E.L. (1968) Effect of Hot Pressing Temperature on the Properties of Hard and Semi-Hard Fibre Building Boards. *Sven. Papp.tidn.* 71(15):774–787.
- Norgren, S., Höglund, H. (2011) Consolidation of fibre-fibre bonds in TMP and CTMP based papers. In: *Int. Mech. Pulp. Conf., Xi'an, China.* pp. 448–453.
- Norgren, S., Höglund, H. (2014) Consolidation of fibre-fibre bonds in TMP and CTMP based papers. *Pap. Technol.* 55(3):6–10.
- Norgren, S., Pettersson, G., Höglund, H. (2014a) High strength papers from high yield pulps. *Pap. Technol.* 56(5):10–14. *Int. Mech. Pulp. Conf., Helsinki, Finland.*
- Norgren, S., Pettersson, G., Höglund, H. (2014b) High strength papers from high yield pulps. *Pap. Technol.* 56(5):10–14.
- Norgren, S., Pettersson, G., Höglund, H. (2018) Strong paper from spruce CTMP – Part II: Effect of pressing at nip press temperatures above the lignin softening temperature. *Nord. Pulp Pap. Res. J.* 33(1):142–149.
- Nygren, O., Bäck, R., Höglund, H. (2003) On characterization of Mechanical and Chemi-mechanical Pulps. In: *Proc. International Mechanical Pulping Conference, Quebec City, Canada.* pp. 97–104.
- Olsson, A.-M., Salmén, L. (1997) The effect of lignin composition on the viscoelastic properties of wood. *Nord. Pulp Pap. Res. J.* 12(3):140–144.
- Orloff, D.I., Sobczynski, S.F. (1993) Impulse drying pilot press demonstration: Ceramic surfaces inhibit delamination. *Pap. Technol.* 34(10):24–32.
- Page, D.H. (1967) The collapse behavior of Pulp Fibers. *Tappi J.* 50(9):449–455.
- Page, D.H. (1969) A theory for the Tensile Strength of Paper. *Tappi J.* 52(4):674–681.
- Pelton, R.H., Zhang, J., Rundlöf, M., Wågberg, L. (2000) The Effect of Charge Density and Hydrophobic Modification on Dextran-based Paper Strength Enhancing Polymers. *Nord. Pulp Pap. Res. J.* 15(5):440–445.
- Pettersson, G. Influence of polymeric multilayer treatment on out-of-plane properties of CTMP-based paperboard. Doctoral thesis 40. Mid Sweden University, Sundsvall, Sweden, 2007, ISBN 978-91-85317-78-3.
- Pettersson, G., Höglund, H., Norgren, S., Engstrand, P., Peng, F., Granfeldt, T., Bergström, J., Joelsson, T. (2018) Upgrading the strength properties of sheets from hardwood high yield pulps. In: *Appita – Pan Pacific Fibre Value Chain Conference.* pp. 142–147.
- Pettersson, G., Höglund, H., Sjöberg, J., Peng, F., Bergström, J., Solberg, D., Norgren, S., Hallgren, H., Moberg, A., Ljungqvist, C.-H. (2015) Strong and bulky paperboard sheets from surface modified CTMP manufactured at low energy. *Nord. Pulp Pap. Res. J.* 30(2):318–324.
- Pettersson, G., Höglund, H., Wågberg, L. (2005) Bulky papers with high internal bond strength. In: *Int. Mech. Pulp. Conf., Oslo, Norway.* pp. 7–13.
- Pettersson, G., Höglund, H., Wågberg, L. (2006) The use of polyelectrolyte multilayers of cationic starch and CMC to enhance strength properties of papers formed from mixtures of unbleached chemical pulp and CTMP. Part I and II. *Nord. Pulp Pap. Res. J.* 21(1):115–128.
- Pettersson, G., Norgren, S., Höglund, H. (2017) Strong paper from spruce CTMP – Part I. *Nord. Pulp Pap. Res. J.* 32(1):54–58.
- Pynnönen, T., Hiltunen, E., Paltakari, J., Laine, J.E., Paulapuro, H. (2002) Good bonding for low-energy HT-CTMP by press drying. *Pulp Pap. Can.* 105(3):T57–T61.
- Pöhler, T., Heikkurinen, A. (2003) Amount and character of splits in fibers cell wall caused by disc refining. In: *Int. Mech. Pulp. Conf., Quebec, Canada.* pp. 417–423.
- Reme, P.A. Some effects of wood characteristics and the pulping process on mechanical pulp fibres. Doktor ingenioravhandling 2000:21. NTNU, Trondheim, Norway, 2000.
- Reme, P.A., Helle, T. (2000) The fibre characteristics of shives initiating web rupture. *Nord. Pulp Pap. Res. J.* 15(4):287–291.
- Reme, P.A., Johnsen, P.O., Helle, T. (1999a) Changes induced in early- and latewood fibres by mechanical pulp refining. *Nord. Pulp Pap. Res. J.* 14(3):256–262.
- Reme, P.A., Kure, K.-A., Gregersen, Ö.W., Helle, T. (1999b) Optimal

- Mechanical Fibres for Improved Publication Papers: Targets and Treatments. In: Int. Mech. Pulp. Conf., Houston, Texas, USA. pp. 171–182.
- Retulainen, E., Moss, P., Nieminen, K. (1993b) In: Transactions of the 10th Fundamental Research Seminar, Vol. 2. Pira International, Surrey, U-K. p. 727.
- Retulainen, E., Nieminen, K., Nurminen, I. (1993a) Enhancing strength properties of kraft and CTMP fibre network. *Appita J.* 46(1):33–38.
- Reyier Österling, S. Distributions of Fibre Characteristics as a Tool to Evaluate Mechanical Pulps. Doctoral Thesis 221. Mid Sweden University, Sundsvall, Sweden, 2015, ISBN 978-91-86694-66-1.
- Rig Dahl, M., Bäckström, M., Hedström, C.-G., Kilian, M., Talja, R. (1999) Impulse technology – A new way to manufacture paper. In: 6<sup>th</sup> International Conference on New Available Technology. SPCI, Stockholm. pp. 442–449.
- Rundlöf, M. Quality of fines of mechanical pulp. Licentiate thesis, TRITA-PMT report 16996:3. 1996, ISSN 1104-7003.
- Rundlöf, M. Interaction of dissolved and colloidal substances with fines of mechanical pulp – Influence of sheet properties and basic aspects of adhesion. Doctoral thesis. Royal Institute of Technology, Stockholm, Sweden, 2002, ISSN 1104-7003.
- Rundlöf, M., Eriksson, M., Ström, H. (2002) Effect of Mannanase and Lipase on the properties of colloidal wood extractives and their interaction with mechanical pulp fines. *Cellulose* 9:127–137.
- Rundlöf, M., Htun, M., Höglund, H., Wågberg, L. (2000a) The Importance of the Experimental Method when Evaluating the Quality of Fines of Mechanical Pulps. *J. Pulp Pap. Sci.* 26(9):301–307.
- Rundlöf, M., Htun, M., Höglund, H., Wågberg, L. (2000b) Mechanical Pulp Fines of Poor Quality – Characteristics and Influence of White Water. *J. Pulp Pap. Sci.* 26(9):308–316.
- Rundlöf, M., Höglund, M., Htun, M., Wågberg, L. (1995) Effect of Fines Quality on Paper Properties – New Aspects. In: Int. Mech. Pulp. Conf., Ottawa, Canada. pp. 109–118.
- Rundlöf, M., Sjölund, A.-K., Ström, H., Åsell, I., Wågberg, L. (2000c) The effect of dissolved and colloidal substances released from TMP on the properties of TMP fines. *Nord. Pulp Pap. Res. J.* 15(4):256–265.
- Sahlberg, U. (1995) Influence of knot fibers on TMP properties. *Tappi J.* 78(5):162–168.
- Salmén, L. Temperature and water induced softening behaviour of wood fiber based materials. Doctoral thesis. KTH, Stockholm, 1982, A22810463B.
- Salmén, L. (1984) Viscoelastic Properties of in Situ Lignin under Water Saturated Conditions. *J. Mater. Sci.* 19:3090–3096.
- Salmén, L. (1990) On the interaction between moisture and wood fibre materials. In: Mat. Res. Soc. Symp. Proc., Pittsburg, Vol. 197. Eds. Cauldfield, D.F., Passaretti, J.D., Sobczynski, S.F. pp. 193–201.
- Salmén, L., Lucander, M., Härkönen, E., Sundholm, J. (1999) Fundamentals of mechanical pulping. The character and properties of mechanical pulps. In: Papermaking Science and Technology, Book 5, Eds. Gullichsen, J., Paulalolo, H. Fapet Oy, Helsinki, Finland.
- Samuelsson, T., Pettersson, G., Norgren, S., Svedberg, A., Höglund, H., Engstrand, P. (2018) Development of strong and water-resistant packaging materials from high yield pulps – fundamental aspects. In: Int. Mech. Pulp. Conf., Trondheim, Norway.
- Scallan, A.M., Grignon, J. (1979) The effect of cations on pulp and paper properties. *Sven. Papp.tidn.* 82(2):40–47.
- Schlegel, J., Rökman, B., Saari, J. (1999) Condebelt drying – board properties BM clothing and runnability. *Pap. Technol.* 40(10):29–41.
- Seth, R.S., Michell, A.J., Page, D.H. (1985) The effect of press-drying on paper strength. *Tappi J.* 68(10):102–107.
- Setterholm, V.C. (1979) An overview of press drying. *Tappi J.* 62(3):45–46.
- Setterholm, V.C., Koning, J.W. (1984) Press-dried paper. *Appita J.* 37(5):361–365.
- Shallhorn, P.M., Karnis, A. (1979) Tear and Tensile Strength of Mechanical Pulps. In: Int. Mech. Pulp. Conf., Toronto, Canada. pp. 25–36.
- Shaw, A.C. (1983) The Opco process applied to TMP screen rejects. *Pulp Pap. Can.* 84(5):32–35.
- Stone, J.E., Clayton, D.W. (1960) The use of Microtome Sections for Measuring the change in Strength of Spruce fibers due to Pulping. *Pulp Pap. Can. Oct. 1960:T475–T484.*
- Strand, B.C., Jackson, M., Mokvist, A. (1989) Improving the Reliability of Pulp Quality Data Through Factor Analysis and Data Reconciliation. In: Int. Mech. Pulp. Conf., Helsinki, Finland. pp. 362–375.
- Sundberg, A., Holmbom, B., Willför, S., Pranovich, A. (2000) Weakening of paper strength by wood resin. *Nord. Pulp Pap. Res. J.* 15(1):46–53.
- Sundberg, K., Thornton, J., Holmbom, B., Ekman, R. (1996) Effect of wood polysaccharides on the stability of colloidal wood resin. *J. Pulp Pap. Sci.* 22(72):26.
- Sundholm, J. (1999) What is mechanical pulping. In: Papermaking Science and Technology, Book 5. Eds. Gullichsen, J., Paulalolo, H. Fapet Oy, Helsinki, Finland. pp. 17–21.
- Swenson, R.S., Macdonald, D.M., Ring, M., Field, J.H., Hall, F.K. (1989) Kraft liner board and method of producing Kraft liner board from bleached or unbleached Kraft pulp, TMP pulp, SCMP or sulfite pulp. European Patent Application: 0219643 A1.
- Tam Doo, P.A., Kerekes, R.J. (1982) The flexibility of wet pulp fibers. *Pulp Pap. Can.* 83(2):T37–T41.
- Tchepel, M., Provan, J.W., Nishida, A., Briggs, C. (2001) A procedure for measuring the flexibility of single wood-pulp fibres. In: Int. Mech. Pulp. Conf., Part 2, Helsinki. pp. 295–304.
- Torgnysdotter, A. Fibre/fibre joints: their characterization and influence on different paper strength properties. Doctoral thesis. KTH, Stockholm, Sweden, 2006, ISSN 1652-2443.
- Tyrväinen, J. (1995) The influence of wood properties on the quality of TMP made from Norway spruce (*Picea Abies*) – Wood from old-growth forest, first-thinnings and sawmill chips. In: Int. Mech. Pulp. Conf., Ottawa, Canada. pp. 23–33.
- Vainio, A. Interfibre bonding and fibre segment activation in paper – Observations on the phenomena and their influence on paper strength properties. Doctoral thesis, Laboratory of Paper and Printing Technology Report, Series A29. Helsinki University of Technology, 2007, ISBN 978-951-22-9057-4, ISBN 978-951-22-9058- (electr.).
- van Byrd, L. (1979) Press drying. Flow and adhesion of hemicellulose and lignin. *Tappi J.* 62(7):81–84.
- van den Akker, J.A., Lathrop, A.L., Voelker, M.H., Dearth, L.R. (1958) Importance of fiber strength to sheet strength. *Tappi J.* 41(8):416–425.

- Vehniäinen, A. Single fiber properties – a key to the characteristic defibration pattern from wood to paper fibers. Doctoral thesis, Helsinki, Finland. 2008, ISSN 1457-6252.
- Vesterlind, E.-L. High Temperature CTMP from birch. Licentiate thesis 11. Mid Sweden University, 2006, ISBN 978-91-85317-20-9.
- Vesterlind, E.-L., Höglund, H. (2005a) Chemimechanical pulp made from birch at high temperature. In: SPCI International Conference, Stockholm, Sweden, 14-16 June 2005.
- Vesterlind, E.-L., Höglund, H. (2005b) Low energy birch CTMP in bulky paper products. In: Int. Mech. Pulp. Conf., Oslo, Norway. pp. 119–125.
- Vesterlind, E.-L., Höglund, H. (2006) Chemithermomechanical pulp made from birch at high temperature. Nord. Pulp Pap. Res. J. 21(2):216–221.
- Voyutskii, S.S. Autohesion and Adhesion of High Polymers. Interscience Publishers, J. Wiley and Sons Inc., New York, USA, 1963.
- Wahren, D. (1982) Method and apparatus for the rapid consolidation of moist porous web. USA Patent no. 4,324,613.
- Wedin, P.-O., Falk, B., Fredriksson, B., Bäck, R., Höglund, H. (1992) Double disc refined TMP for LWC Paper. In: Proc. 4<sup>th</sup> International Conference, New Available Techniques and Current Trend, Part 1, Bologna, Italy. pp. 281–296.
- Westermarck, U., Capretti, G. (1988) Influence of ray cells on the bleachability and properties of CTMP and kraft pulps. Nord. Pulp Pap. Res. J. 3(2):95–99.
- Westermarck, U., Samuelsson, B., Simonson, R., Pihl, R. (1987) Investigation of a selective sulfonation of wood chips. Nord. Pulp Pap. Res. J. 2(4):146–151.
- Williams, M.L., Lander, R.F., Ferry, J.D. (1955) The temperature dependence of relaxation mechanisms in amorphous polymers and other glass-forming liquids. J. Am. Chem. Soc. 77(14):3701–3707.
- Wood, J.R., Karnis, A. (1991) Future furnish requirements for newsprint and mechanical printing papers. Pulp Pap. Can. 92(1):T21–T25.
- Wood, J.R., Karnis, A. (1992) Linting propensity of mechanical pulp. Pulp Pap. Can. 93(7):T191–T198.
- Wågberg, L., Björklund, M. (1993) Adsorption of cationic potato starch on cellulosic fibres. Nord. Pulp Pap. Res. J. 8(4):399.
- Wågberg, L., Forsberg, S., Johansson, A., Juuntti, P. (2002) Engineering of fibre surface properties by application of the polyelectrolyte multilayer concept, Part I. Modification of paper strength. J. Pulp Pap. Sci. 28(7):222.
- Wågberg, L., Kolar, K. (1996) Adsorption of cationic starch on cellulosic fibres. Tappi J. 59(12):121.