Miscellaneous

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Dewatering properties of pulps made from different parts of a Norway spruce (*Picea abies*)

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Abstract: A single Norway spruce tree (*Picea abies*) was manually fractionated into heartwood, sapwood, juvenile wood and branches. These fractions were chemically pulped, individually, in laboratory scale. The pulps were characterized and investigated in relation to dewatering behavior and sheet strength properties. An unbleached and unbeaten commercial kraft pulp from softwood fibers was used as a reference, and the fractionated pulps were within the same range in all tested properties. The fractionated pulps were then compared with each other, and fiber characteristics were used to explain differences in dewatering and strength. Heartwood pulp results in stronger and stiffer papers that are harder to dewater. Sapwood pulp gives more open network structures resulting in easy dewatering and high air permeance, although with lower strength properties compared to heartwood. Pulp from Juvenile wood gives s quite strong but brittle sheets, with efficient dewatering. Pulp from branches gives paper sheets with efficient dewatering, air permeance and relatively high elongation of break but lower strength. The results show that there is definitely potential for utilizing more parts of the trees for pulp and paper making, especially when tailoring the raw material origins after preferred paper properties.

Keywords: compression wood; dewatering; heartwood; Norway spruce; sapwood.

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Introduction

Modern forestry aims for efficient utilization of the raw material harvested from the forest. That means maximizing the use of most different parts of the tree for generating as much profit as possible. In the way softwood forestry is carried out today in Scandinavia, there are mainly four fractions produced:

- Thick logs of good quality are obtained on the final harvesting; this fraction goes to sawmills for producing planks etc., and this process generate...
- Sawmill chips. Formed residues when the round log is converted to planks etc. This is used for pulping
- Pulpwood, commonly consists of logs of young trees harvested during thinning, logs of the upper part (“tips”) of trees in final harvesting, but also thick logs, that are unsuitable for saw mill uses for different reasons, such as bended stems, partly rotten wood and logs with spiral grown wood.
- Branches and ultimate tips are mostly used as fuel.

Thus, the pulp and paper industry use mainly two fractions, the sawmill chips and the pulp wood, and not surprisingly, they have different properties. Therefore, the pulp mill can balance the use of the different raw materials for obtaining suitable properties.

However, this technical division of trees does not correspond to a biological-based division of different wood qualities. Biologically, several types of wood exist in a softwood tree:

- Juvenile wood exists in young trees, in the upper part of a tree, and in the central part of mature logs. It has shorter fibers and thinner cell walls.
- Mature wood exists on the outer part of the thick stem, and it has thicker cell walls and longer fibers than juvenile wood.
- Heartwood exists in the inner part of the thicker stem. It is a completely dead tissue, and is dryer and has a partly different chemical composition than
- Sapwood that exists in outer part of thick stems and all the way through a younger stem. Although most cells are dead, with only parenchyma cells are living, it is a biologically active tissue involved in water and

mineral transport, and storage of nutrition. This is reflected in a higher moisture content and other types of extractives than in the heartwood
- Compression wood is formed when the tree is subjected to mechanical stress, and branches consist largely of compression wood. It differs chemically from normal wood in the aspect of lignin structure and hemicellulose composition, and the fibers are also smaller.

It shall be noted that these aspects are overlapping, and wood can be both juvenile wood and heartwood for instance. In this experiment the tip fraction will consist of wood that is sapwood and juvenile wood, the "sapwood fraction" will consist of wood that is sapwood and mature wood, and the "heartwood fraction" will consist of wood that is heartwood and a mixture of juvenile and mature wood. All of them can be contaminated by overgrown branches, with compression wood. The branches consist of wood that is juvenile wood and partly compression wood.

The sawmill chips consist thus mainly of mature sapwood, but can also contain reaction wood in the form of overgrown branches. The pulp wood has a more complex composition, and even if juvenile wood dominates, the fraction can also contain mature wood – both sapwood and heartwood – as well as reaction wood.

Most wood fibers are very disposed to cling to each other and form flocks; this is called flocculation in paper manufacturing. Wood fibers, especially virgin fibers, are also extremely efficient in retaining water. Flocculation is mostly not preferable when producing homogenous paper sheets, with a few exceptions when air permeance is of importance in the paper product. During paper production, the fibers must therefore be separated by dilution and shear forces. To avoid flocculation, solid contents as low as 0.2 % dryness are commonly used. Aspects of fiber flocculation depend on the fiber crowding factor (Kerekes and Schell 1992), and different fiber types give varying formation and, consequently, other drainage and dewatering properties. Due to the heavy dilution in the early stages of paper production, vast amounts of water are removed by dewatering in several steps in a paper machine. Dewatering is a well-known bottleneck that affects both production- and energy efficiency. Numerous strategies to increase dewatering rates are needed for paper production: optimal machine design, forming fabric design, reduction of rewetting, application of wet-end chemicals, et cetera (Kuhasalo et al. 2000, Norman 2000).

The water removal in the paper machine is defined by diminishing returns, where each unit of removed water makes it harder to remove another, which implies that energy savings early in the process can be very beneficial later in the process (Attwood 1962, Baldwin 1997, Ramaswamy 2003). The last dewatering step in any paper machine is drying, and the drying sections are the main consumers of energy in the papermaking process. Higher dryness early in the process means less water to evaporate and thus reduced energy demand in the drying section (Kuhasalo et al. 2000, Norman 2000, Wahlström 2001, Ramaswamy 2003, Sjöstrand 2020). The reason for the diminishing returns of dewatering is the location of the water in the sheet structure. Water outside of the fibers in the sheet is most easily removed, followed by water in the fiber lumen, and the most difficult water to remove is located within the fiber walls (Stenström and Nilsson 2015).

To capture the entire dewatering process before drying and to investigate the fibers' influence three dewatering tests will be made: (i) drainage according to Schopper-Riegler which mimic the early gravitational dewatering in the forming section, (ii) dryness and air penetration during vacuum dewatering in laboratory scale to simulate the dry vacuum suction boxes on the paper machine, and (iii) water retention value to get knowledge about water content in fiber lumen and fiber walls (Stenström and Nilsson 2015, Sjöstrand 2020).

The last years progress in artificial intelligence and different forms of spectroscopic characterization might lead to possibilities for better defined and more homogeneous raw material fractions from the forestry to the industry. In order to investigate this potential, a single tree of Norway spruce (*Picea abies*) was harvested and fractionated for heartwood, mature sapwood, juvenile wood, and branches/reaction wood. The different fractions were characterized and pulped using kraft cooking. In this work, we investigate the dewatering and tensile strength properties of these pulps.

Materials and methods

Wood and chip preparation

A single Norway spruce (*Picea abies*) tree from Umeå forest area in Sweden was collected (63° 48′ 43.2102″ N, 20° 14′ 9.564″ E) and was used in our research studies. The tree's age was determined by counting the year rings, and it was found that the spruce tree's age was 58 years. The diameter of the tree was approximately 28 cm. The spruce tree was debarked and separated into four sections manually using an electrically operated sawmill machine (F2 Logosol, Härnösand, Sweden). The four sections of the spruce tree were sapwood, heartwood, juvenile wood (top portion of the tree) and the branches. All the tree parts except the branches were chipped in the MoRe Research chipper (Bruks Mekaniska AB, Sweden). All the chips obtained were classified in a chip classifier which had a series of trays (Ø 45 mm, //8 mm, Ø 13 mm, Ø 7 mm, Ø 3 mm and <Ø 3 mm) and chips retained on the 13 mm tray was used for the experimental trials. The branches were debarked first, and chips were prepared manually with a chip size of approximately 2.4 × 2.4 cm (length x breadth) and 3–4 mm thickness.

Pulping experiments

Lab-prepared kraft cooking liquor was used in all experimental trials. The cooking experiments were carried out in the electrical heated autoclave digester using glycol as the heating media. The start-up temperature was 100 °C, and the L/W-ratio was always 4.0 throughout the cook. All kraft cooking was carried out at 20 % Effective alkali and 35 % sulfidity. The ramping time to reach the kraft cooking temperature was 1 °C/min, and the final cooking temperature used in our experiments was 165 °C. The kraft cooking time used in our experiment ranged from 1.5 h to 4 h cooking time at 165 °C and the same cooking conditions were carried out for each spruce tree parts. The aim here was to understand the delignification rate and the carbohydrate degradation at different cooking time.

At the end of each cook, the autoclaves were cooled rapidly by submerging them into cold water of about 10 °C to stop further reactions. Pulp and liquor samples were collected after each cook. The pulp samples obtained after cooking were washed overnight with distilled water and then disintegrated in NAF (Nordiska Armaturfabriken) water jet defibrator with 2 mm holes at a water pressure of 3 bar. The pulp obtained after defibration was dried at 45 °C in an oven to air dry equilibrium conditions. The reject yields were calculated by gravimetric measurements.

Carbohydrate analysis

Carbohydrate analysis was carried out on acetone extracted 40 mesh pulp to get extractive free pulp samples. These extractive free pulp samples were then subjected to acid hydrolysis for lignin and sugar content determination. 3 ml of 72 % H 2SO4 were added to each pulp/wood sample, then placed in a vacuum desiccator for 1 hour and 20 minutes and stirred occasionally. Thereafter, the mixtures were diluted with 84 ml of MilliQ water and then digested in an autoclave at 120 °C for 1 hour. The digested samples were filtered through a glass fiber filter using a 3-piece filter setup. The filtrates were then diluted 1:10 for sugar analysis and acid soluble lignin. The insoluble (Klason lignin) part was dried in an oven at 105 °C and weighed. Carbohydrate content was determined using a (HPAEC-PAD) Dionex ICS3000 with a pulsed amperometric detector, using a CarboPac PA1 column (Thermo scientific, USA) with an injection volume of 25 µl and a flow rate of 1 ml/min. External sugar standards were used for calibration. The results were determined as anhydrous sugars and the average results obtained after triplicate analysis were reported. The pulp samples after cooking experiments were analysed for Kappa Number (ISO 302:2015 E) and viscosity (ISO 5351:2010).

Pulp Fiber analysis

Pulp fiber analysis was carried out in PULP EYE analyzer which is a fiber classification module which works in accordance with TAPPI/ISO standards. The PulpEye fiber classification methods analysis 100,000 objects in 30 seconds and gives accurate measurement information about the pulp fiber properties like length, width, fines, coarseness, macro fibrillation, curl and kinks. The different pulps where qualitatively inspected in an optical microscope to provide a visual basis as support for the discussion, although the conclusions in this article are based on the PULP EYE analyzer results.

Dewatering measurements

Dewatering of the different pulps was observed by measurements of dewatering resistance (SR) according to ISO 23714 (2014), water retention value (g water/g fiber) ISO 5267-1 (1999) and vacuum dewatering in a custom-built laboratory vacuum suction box described by Granevald et al. (2004), Figure 1. The vacuum dewatering was performed with a commercial forming fabric with a triple layered structure (SSB) and an air permeability of 325 cubic feet per minute (cfm) delivered by Albany International. Prior to the vacuum dewatering, isotropic sheets were formed in a Finnish laboratory sheet former with thorough agitation of the stock to ensure as consistent formation as possible. The sheet former has no built-in fabric so the commercial forming fabric, described above, is used when forming the sheets. The sheets were only for vacuum dewatering and were later discarded after the dryness measurements. The vacuum level was set at –40 kPa,

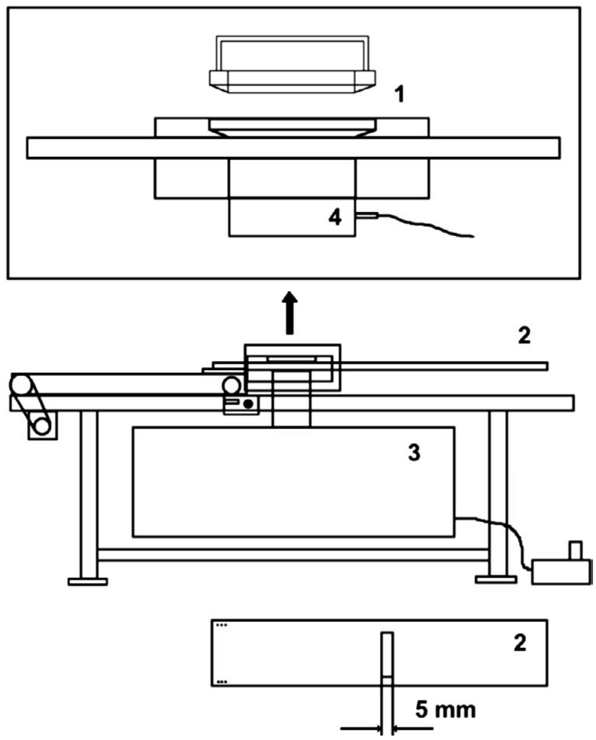

Figure 1: Schematic picture of the laboratory suction box. The machine includes: (1) sample frame, (2) moveable plate with 5 mm rectangular opening, (3) 300 L vacuum tank, and (4) a transducer logging the pressure directly underneath the sample. Redrawn from Granevald et al. (2004).

the basis weights of the sheets were 60 g/m^2, and the dwell time in vacuum was 20 ms. After each test, the dryness was measured according to ISO 638:2008 and recalculated to a Moisture ratio. The pressure difference during testing was logged with a transducer (Amtele AB, Stockholm, Sweden) located directly underneath the sample. The air volume that passed through the sample was calculated using Equation 1,

$$V_{air} = \frac{V_{tank}}{P_{atm}} * \Delta P, \tag{1}$$

where V_{air} is the volumetric flow of air through the sample (L), V_{tank} is the vacuum tank volume (L), P_{atm} is the pressure outside the tank (kPa), and ΔP is the measured pressure difference directly below the sample (kPa). All dewatering experiments were performed with four replicates for statistical significance.

Sheet formation and strength measurements

Paper sheets of 60 g/m^2 were also formed in another laboratory sheet former, plane pressed and restrained dried. The sheet former includes agitation of the stock to achieve consistent formation. The sheets were dried in a standardized climate according to ISO 187:1990 for 24 hours, tensile testing was also performed in the controlled climate. The sheets were subjected to tensile testing according to ISO 1924-3:2008. The tensile testing was performed with ten replicates according to standard.

Results and discussion

A single tree of Norwegian spruce (*Picea abies*) was fractionated into heartwood, mature sapwood, juvenile wood, and branches/reaction wood, chipped and pulped with the kraft pulping technique. The pulp produced from 4 h kraft cooking time for different spruce tree parts are shown in Table 1. It can be seen that the branches had the high residual lignin content after 4 h kraft cooking, as compared to the other spruce tree parts indicating that the pulping time need to be extended for branches to get lower lignin content. The glucose content for sapwood, heartwood and juvenile were similar; though the glucose content for the branches was low since the original branches had lower glucose content. Branches had higher xylan content in the native wood and hence the residual xylan content in the unbleached pulp was higher for the branches. Rhamnose was totally removed from the wood chips at the end of 4 h cooking time and hence it is not mentioned in Table 1. Mannose content was similar in all the pulp at the end of the cook. Negligible amount of galactose was found in the pulp indicating its severe degradation during the kraft pulping. The results from the PULP EYE analyzer are shown in Table 2. Figure 2 show selected images of the four pulps from the optical microscopy observations.

The pulps were characterized for dewatering properties, with values for all analyses made according to description in the method. The drainage (°SR), water retention values (WRV), moisture ratio after vacuum dewatering and penetrated air volume during vacuum dewatering for the pulps are shown in Figure 3. Figure 4 show tensile strength index, elongation at break, tensile energy absorption and elastic modulus for the sheets made from the pulps. The stress-strain curves for the different pulps are shown in Figure 5. Comparing the pulp fractions with the reference pulp, all measured properties are of the same magnitude; a closer interpretation of this is not possible since the fiber properties, pulp method, and wood origin are not the same. Comparison with reference pulp mainly shows that the pulp fractions could readily be used in commercial paper processes.

Table 1: Carbohydrate analysis and kappa number of 4h kraft cooking results.

	Glucose, %	Xylose, %	Arabinose, %	Mannose, %	Galactose, %	Kappa number
Sapwood	35.51	2.68	0.16	2.63	0.1	17
Heartwood	35.2	2.72	0.14	2.97	0.14	18
Juvenile	36.1	2.9	0.17	2.96	0.11	18.24
Branches	28.21	3.5	0.33	2.0	0.2	36.3

Table 2: Values for all pulp characteristics.

Analysis	Unit	Heartwood	Sapwood	Juvenile wood	Branches	Variance (%)
Fibre length	mm	2.02	1.83	2.06	1.52	2
Fibre width	μm	28.0	28.5	27.0	22.2	1
Fibre curl	%	9.5	10.3	9.6	11.0	2
Fines amount	%	3.9	8.0	3.9	3.6	2.5
Fibre weight	mg/m	0.120	0.128	0.110	0.074	3
Fibre wall thickness	μm	1.72	2.28	1.80	2.13	2
Shive content	#/g	184	124	244	192	6
Kinks	Kinks/mm	0.15	0.18	0.17	0.24	3

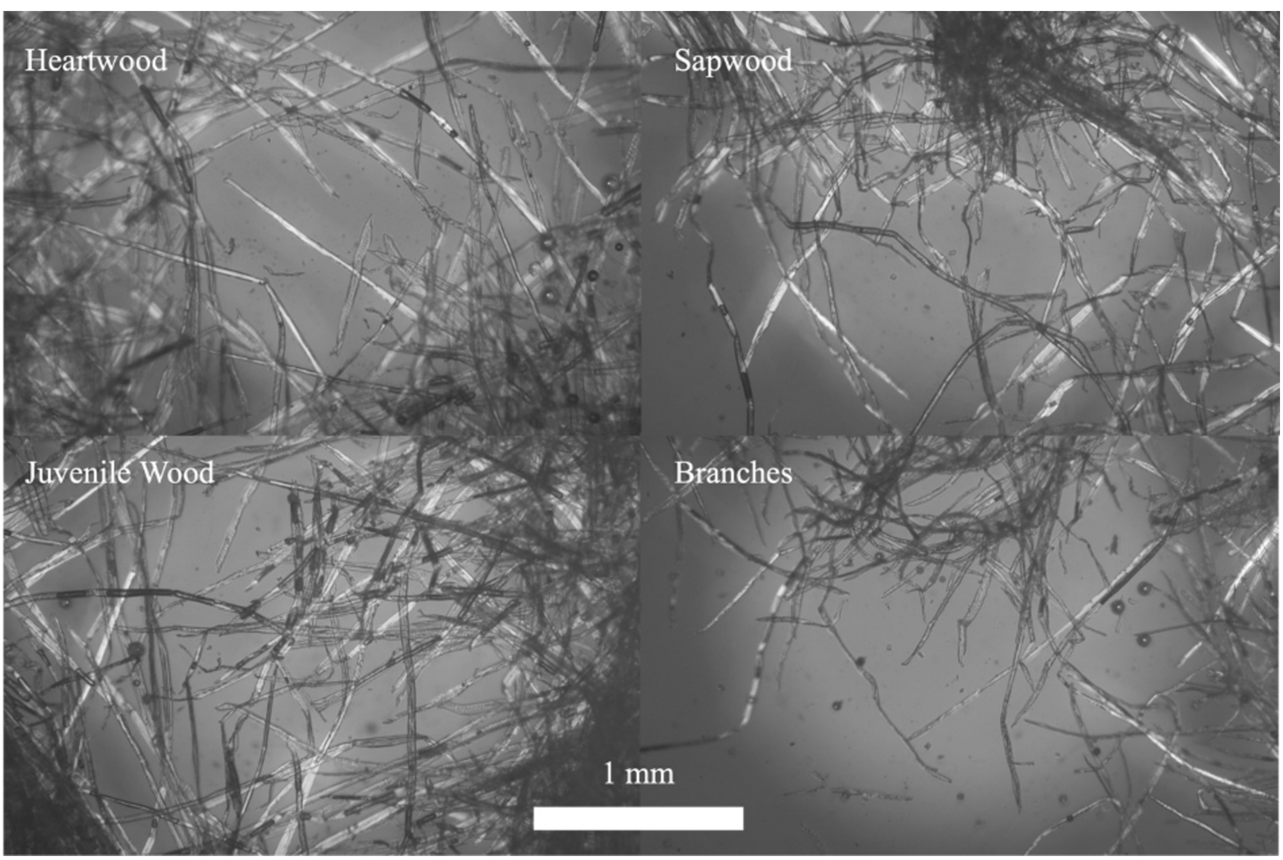

Figure 2: Optical microscopy images of the four pulp types. All four images have the same magnification and the scale is indicated by the white bar of 1 mm.

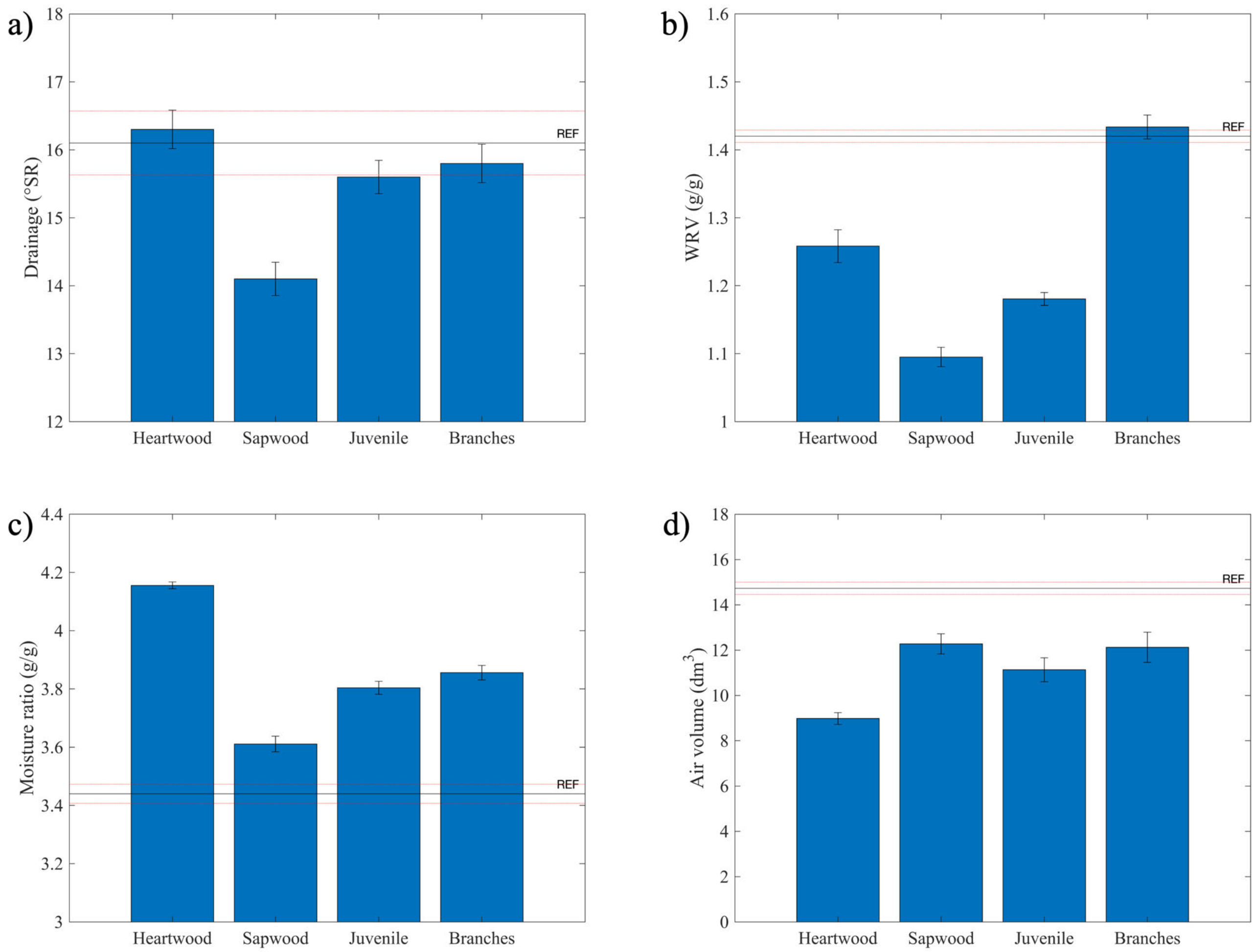

Figure 3: (a) Drainage (°SR), (b) Water retention values (g/g), (c) Moisture ratio (g/g) after 20 ms vacuum dewatering at −40 kPa, and (d) Penetrated air volume (dm3) during 20 ms vacuum dewatering at −40 kPa, for all pulps, the error bars indicate a 95 % confidence interval based on four measurements. Reference pulp mean value is shown with a solid black line with dashed red lines representing 95 % confidence interval.

The values in Table 2 are interpreted and connected to fiber flexibility by assuming that high values of fibre wall thickness mean inflexible fibers and low fibre wall thickness means highly flexible fibers. This will be relevant in the coming discussion where fibre characteristics are connected to dewatering behavior and strength properties.

Drainage (°SR) is a measurement of how easily water flows past fibers in a suspension and are highly relevant to compare with early gravitational dewatering directly after the headbox. Heartwood has a high value (Figure 3a) which means slow dewatering, which can be connected to and explained by the heartwood pulp's relatively large and flexible fibers, values seen in Table 2, impeding the flow of water (Figure 6). Sapwood has a low drainage value which means fast early dewatering, this is connected to the shorter fibers of higher fiber wall thickness that will not impede water flow to a large extent during drainage and early gravitational dewatering.

The water retention values of the pulps are shown in Figure 3b, where it is shown that branches have the highest water content after centrifugation, followed by heartwood, Juvenile and Sapwood, in that order. Water retention values are mostly connected to dewatering at the press section and the fibers' ability to hold water in the fiber lumen and fiber wall (Stenström and Nilsson 2015). There are some chemical aspects of pulp from branches that might affect fiber swelling but not the more mechanically centered dewatering of drainage and vacuum dewatering. Higher surface charges and higher xylan content in the fiber wall might contribute to the ability to absorb water and hold it during WRV centrifuging (and pressing). Branch pulp also have a thick fiber wall with low weight

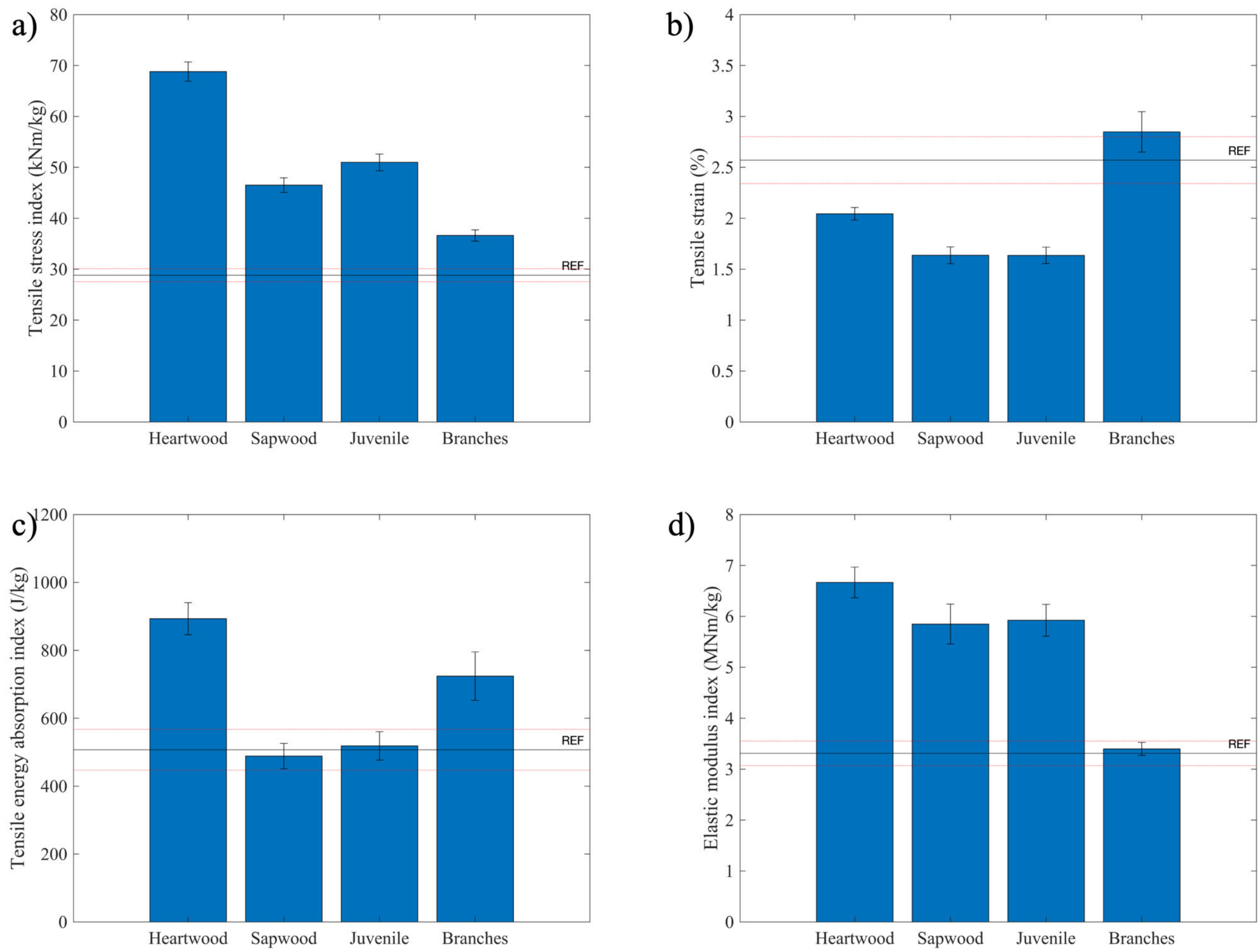

Figure 4: (a) Tensile strength index (kNm/kg), (b) Elongation at break (%), (c) Tensile energy absorption (J/kg), and (d) Elastic modulus index (MNm/kg) for sheets made of the pulps, the error bars indicate a 95 % confidence interval based on ten measurements. Reference pulp mean value is shown with a solid black line with dashed red lines representing 95 % confidence interval.

per meter possibly indicating a relatively large lumen volume and, therefore, the high WRV number (Figure 3b). As opposed to sapwood, the high fiber wall thickness and high fiber weight indicate low lumen volume and correspondingly low WRV number. Sapwood with lower flexibility in the fibers might also be more resistant to fiber wall swelling. Heartwood fibers in a tree have almost dry fibers for large portions of the year; one might argue that heartwood fibers, to some extent, have more hornification which is also bad for fiber swelling and would decrease WRV even though the other dewatering measurements are also relatively high due to the flexibility of the fibers' impact on the fiber network.

High values of moisture ratio after vacuum dewatering means higher water content; even small differences can be huge in relation to actual tons of water that need to be removed according to high production volumes (Sjöstrand et al. 2019). One extra gram of water per gram dry paper produced might seem like a small difference in dewatering in Figure 3c but will result in several tons of excess water that must be removed each hour of production in a paper machine. Heartwood pulp have long fibers with high flexibility (Table 2) and has the highest moisture ratio after vacuum dewatering, this is strongly connected to the compactness of the fiber network, much alike how beating of fibers affects dewatering and also strength. Sapwood with short, coarse fibers gives a more open fiber network, and the moisture ratio is considerably lower after vacuum dewatering (Figure 3c).

In Figure 3d, there are no significant differences in penetrated air volume during vacuum dewatering between sapwood, juvenile wood and branches but the heartwood has lower air penetration. This again, is connected to the excellent flexibility of the heartwood fibers and the closed

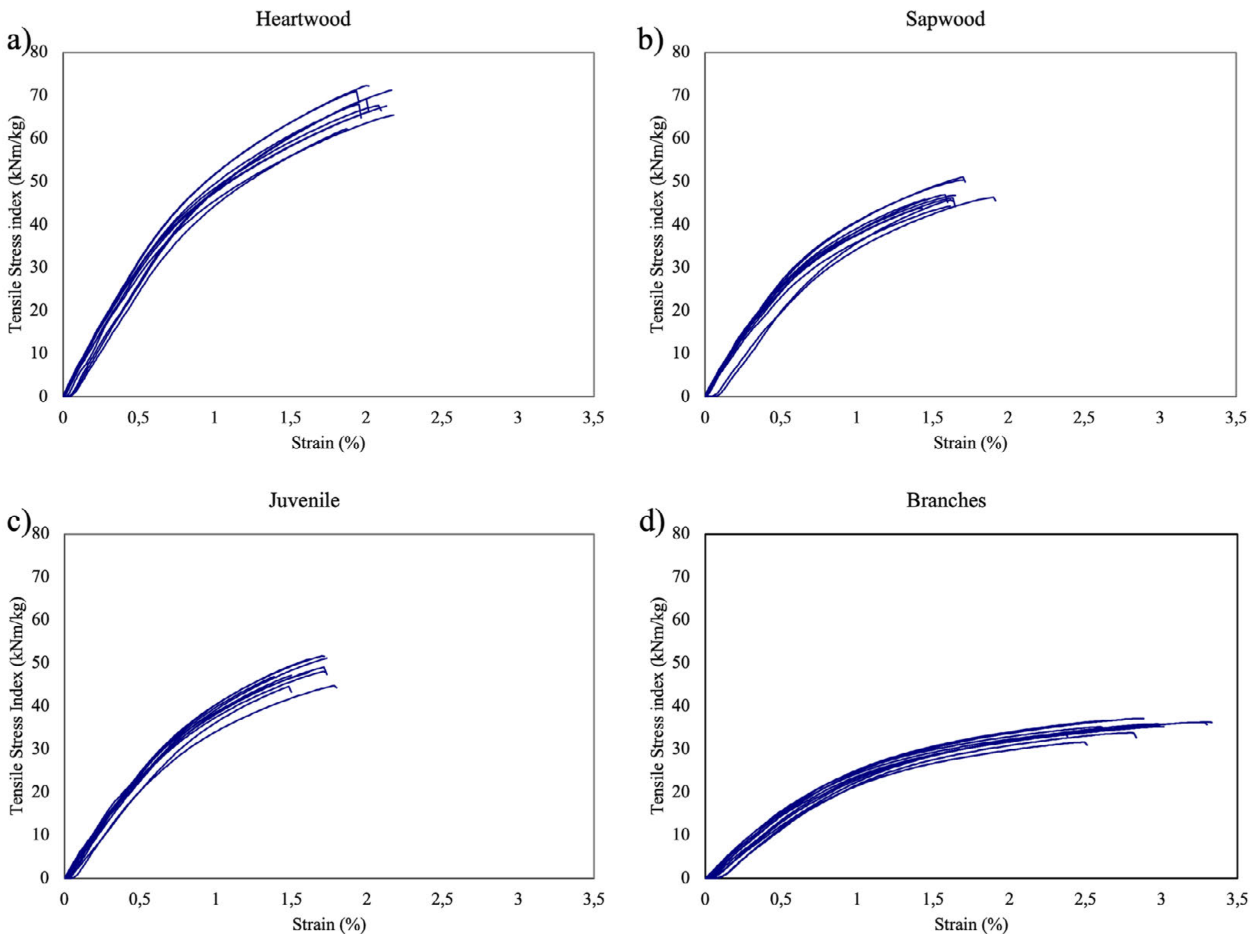

Figure 5: Stress-strain curves for Heartwood (a), Sapwood (b), Juvenile wood (c) and Branches (d).

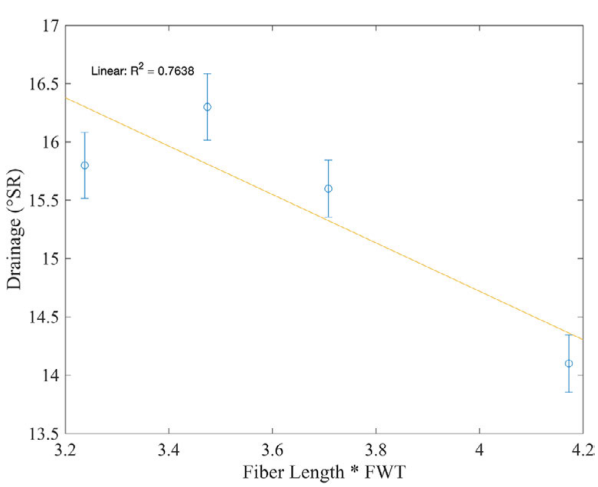

Figure 6: The product of fiber length and Fiber wall thickness plotted against drainage with a linear regression to indicate the trend.

network they provide. To further investigate the air permeance behavior, more appropriate testing can be performed, such as air permeance by Bendtsen (ISO 5636-3:2013) et cetera. The measured penetrated air volume in this study is more related to energy consumption on the paper machine than actual product properties. By this argument, it is more energy efficient to run easily dewatered sheets with shorter dwell times in vacuum, which is a well-known fact in literature (Attwood 1962, Baldwin 1997, Ramaswamy 2003).

Strength measurements from Figure 4 and 5 continue to have logical explanations and are consistent with the dewatering results. In Figure 4a, the heartwood pulp's long, wide and flexible fibers give the highest tensile strength index, while the smaller fibers of branches give the lowest strength (Figure 7). Sapwood has shorter fibers, and juvenile wood has long but less flexible ones.

In Figure 4b the flexible fibers of heartwood are also shown to provide high elongation at break; branches with lower network strength also give high elongation at break. This might be explained by the high curl and number of kinks (Table 2) that needs to be stretched before the break.

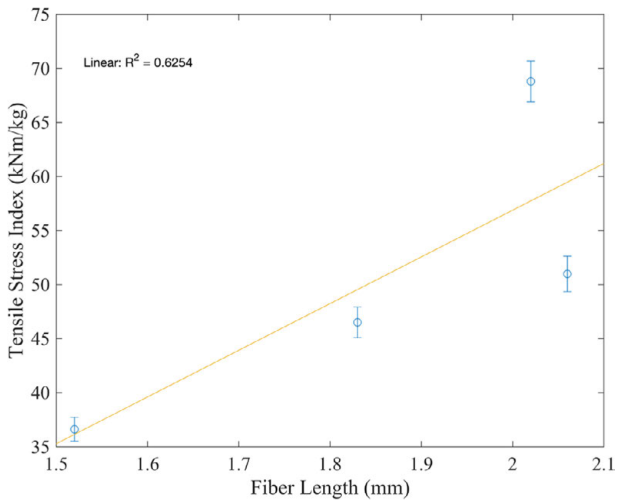

Figure 7: Tensile stress index plotted against fiber length with a linear regression to indicate the trend.

The Tensile Energy Absorption (TEA) in Figure 4c is a measurement that indicates the total energy that a sheet can absorb before failure and high strength, stiffness and elongation give high values of TEA. Heartwood is good in all categories, but it is interesting to note that also branches can take a high energy, due to their high elongation at break values.

The elastic modulus is connected to the number of fiber-fiber bonds and the strength of the network (Niskanen and Kärenlampi 1998), and heartwood has high values due to its long, wide, thick and flexible fibers. The high number of kinks and low fiber weight together with weak network of branches gives the lowest elastic modulus (Figure 4d).

Conclusions

There is definitely potential for utilizing more parts of the trees for pulp and paper making, especially when tailoring the raw material origins after preferred paper properties. The following conclusions can be made from each of the four investigated pulp types:

– Heartwood pulp consists of fibers that result in stronger and stiffer papers that are harder to dewater.
– Sapwood pulp gives more open network structures, resulting in easy dewatering and sheets of higher air permeance, although with inferior strength properties compared to heartwood.
– Pulp from juvenile wood gives quite strong but brittle sheets with quite good dewatering properties.
– Pulp from spruce branches gives paper sheets which are hard to dewater and have air permeance and at the same time also with a relatively high elongation of break, which provides high Tensile Energy Absorption values also compared with sapwood and juvenile wood.

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