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Pressurized hot water flow-through extraction system scale up from laboratory to pilot scale

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The scaling up of the pressurized hot water flow-through extraction system for biomass from laboratory scale to pilot scale by a factor of 6000 was studied. The extraction efficiency and system performance were evaluated by comparing the hemicellulose extraction kinetics, yield, and mass balances. The extractions were performed at 160 °C and 170 °C temperature for birch and spruce sawdust, respectively. Extraction time was 60 minutes. Three identical extractions were conducted with both sawdust types in laboratory and pilot scale. The hemicellulose yields were 50 wt% for birch and 80 wt% for spruce in both laboratory and pilot scale. Relative standard deviations for hemicellulose yields were < 10% between extractions, so the extraction parameters from the laboratory scale setup can be used in the pilot scale extractions.

Introduction

Growing demands for an increased use of renewable resources also brings along the needs of effective biomass fractionation methods. Currently woody biomass is mainly used for pulping, in construction, or as energy. The demand for paper has decreased in developed countries and there is space for new products from wood. Different biorefinery process concepts are proposed to produce chemicals and fuels from biomasses. These processes utilize batch, flow-through, or counter- and co-current systems[1] with different chemicals and pH ranges.[2] Biorefineries should preferably utilize non-food biomass sources and they need effective pretreatment and fractionation techniques, which are sustainable and do not strain the environment. Before moving to build an industrial plant, pilot scale systems has to be used to test pretreatment and fractionation concepts.

Water and biomass can be found almost all around the globe. Pressurized hot water extraction utilises plain water as a solvent, hence no other chemicals are needed. Additionally, woody biomass for the extractions can be found in local communities. Water is an environmentally friendly extraction media[3] and it is extensively used in the industry. Water could be recycled after extractions by purification and filtration steps[4] and reused in further extractions. Large amounts of sawdust are produced annually in sawmills. This residue can be used as a raw material source for industrial scale extractions.

Pressurized hot water extraction (PHWE) at subcritical temperatures can be used to modify the physical properties of water.[5] The dielectric constant of water decreases at subcritical temperatures and therefore water can dissolve more semi-polar compounds. The temperature also enhances the penetration capability of water into the sample matrix by lowering the viscosity of water. The increased diffusivity improves the mass transfer of dissolved compounds, thus improving extraction efficiency.[6]

Hemicelluloses are polysaccharides, which are found in cell-walls of plants. Unlike cellulose, polymeric hemicelluloses from wood are not used in large quantities in the industry. Hemicelluloses are mainly converted into sugars, chemicals, fuels, or sources of heat and energy. The major hemicellulose in birch is xylan, which consists of xylose and 4-*O*-methylglucuronic acid.[7] Spruce hemicelluloses are mostly consisting of galactoglucomannans.[8] Extracted xylans could be used to produce xylo-oligosaccharides for food additives and nutraceuticals,[9] as moisture barriers with composites,[10] foams, and gels.[11] Other proposed usages for xylans and galactoglucomannans (GGM) could be in food packaging as barriers or aerogels.[12,13] Acetylated galactoglucomannan can be used to produce oxygen barriers[14] and birch extracts could also be used to produce oxygen barriers.[15]

PHWE flow-through extractions have been used to extract many different biomasses, such as flax shives,[16] corn stover,[17,18] birch,[19] and different plant species[20] from the agricultural and forestry sectors.

The aim of these experiments was to scale up pressurized hot water (PHW) flow-through extractions from laboratory scale to pilot scale. The PHW flow-through extraction system has earlier been used in laboratory scale to extract acetylated xylan from birch[21,22] and galactoglucomannan from spruce sawdust.[23] These lab scale systems gave fundamental information about PHWE flow-through systems. Scale-up to larger scale is an important step to evaluate processes before industrial scale. The main objective of these experiments was to evaluate PHWE flow-through systems operation in pilot scale for recovery of hemicelluloses from birch and spruce sawdust.

Experimental

Sawdust

Birch and spruce sawdust were collected from the Finnish sawmills at Koskisen Oy (Hirvensalmi) and Metsä Wood (Renko), respectively. Spruce sawdust was taken from the process line in October and birch sawdust in September. Both sawdust batches were stored in the dark in closed plastic bags at -20 °C. Before extraction, sawdust was warmed up to room temperature. The same unscreened sawdust was used in both laboratory and pilot scale extractions.

To determine particle size distribution of raw sawdust from birch and spruce wood, the portion of sawdust was air-dried and sieved with 8 different sieves (Retch, Germany) (Table 1). The distribution percentage shows how much sawdust each sieve retained. Fractions obtained after sieving were then oven-dried (105 °C). Most of the birch sawdust particles were larger than 0.5 mm, with more than 50% in the size range of 1–2 mm. The size distribution of the spruce sawdust was broader, with major part of sample distributing between 0.2–4 mm.

Table 1. Particle size distribution of the birch and spruce wood sawdust (wt%).

Sieve	Birch	Spruce
Pan	<0.1	<0.1
0.05 mm	1.0	0.9
0.1 mm	1.7	2.0
0.2 mm	9.2	11.7
0.5 mm	22.6	23.3
1 mm	51.0	29.9
2 mm	8.2	23.1
4 mm	4.3	8.7
8 mm	2.0	0.4

Laboratory scale PHW flow-through extraction system

The extraction set-up for the laboratory scale experiments is shown in Figure 1. The volume of the extraction vessel (Alimetrics, Finland) was 50 mL and the height and diameter were 60 and 34 mm, respectively. The length to diameter–ratio of 1.8:1 was the same for both the laboratory and pilot scale extraction vessels. Operating parameter limits for the laboratory PHW extraction system are for pressure 0–400 bar, for temperature 10–250 °C, and for flow-rate 0.3–9 mL/min.

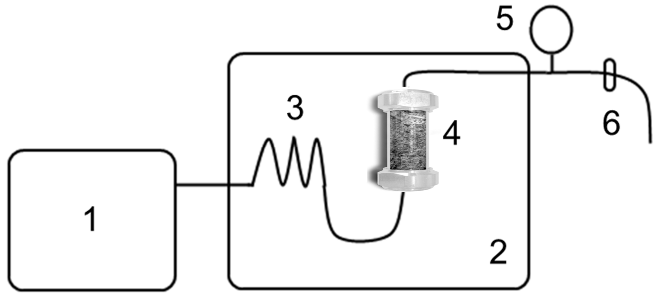

Figure 1. Scheme of the laboratory scale extraction system, 1) HPLC pump 2) GC-oven 3) pre-heating capillary 4) extraction vessel 5) manometer 6) valve.

The extraction vessel was placed inside an oven (employed from gas chromatography HP 5890 series 2) to control the temperature of the extraction vessel. A pump (employed high performance liquid chromatographic pump, Merck Hitachi, L-6200) was used to pump the De-ionized water (Maxima) through the pre-heating capillary and extraction vessel. The pressure of the extraction system was controlled with a valve and monitored with a manometer. Extracts were collected into plastic bottles in ten minute fractions (40 mL) for a total of 60 minutes. At the beginning of the PHW flow-through extractions with birch sawdust, water was pumped into the extraction vessel with a flow rate of 6.7 mL/min, until the extraction vessel was filled with water.

During pilot scale extractions of spruce sawdust it was noticed that the pre-steaming decreased temperature differences in the sawdust bed. With pre-steaming, the sawdust bed and the extraction vessel was heated more evenly. After the effect of steaming was noticed, all spruce extractions were done with pre-steaming. Water was allowed to evaporate to steam during the filling stage of the laboratory scale extraction vessel by lowering the pressure inside the extraction vessel. Pre-steaming is commonly used in the pulp industry to remove the air from chips and to heat them more uniformly.[24] Pre-steaming can also arrange the chips in the chip bed more evenly. With birch extractions, the evaporation of water was prevented by keeping the pressure in the laboratory scale system over 20 bars. During the extraction phase, a constant flow rate of pre-heated water at 3.3 mL/min was used for both birch and spruce extractions.

Pilot scale PHW flow-through extraction system

The pilot scale system consisted of a 300 L extraction vessel, a pump, heaters, heat exchangers, and a hot water reservoir (Figure 2).

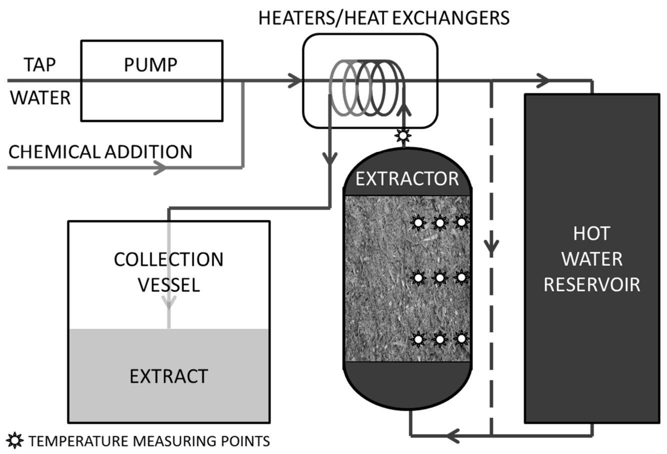

Figure 2. A scheme of the pilot scale extraction system. Stars in the scheme indicate points where the temperature inside extraction vessel is measured.

The pilot scale flow-through extraction system was built by Viitos-Metalli Ltd. at Heinola, Finland. The system operates at pressures of 0–25 bar, temperatures of 10–225 °C, and flow-rates of 6–100 L/min. The power of the pump (KSB) and heater elements (Loval Oy) are 18.5 kW and 150 kW, respectively. The pipe heat exchanger system was built at Viitos-Metalli Ltd. The plate heat exchangers were obtained from Alfa-Laval. The flow rates of water and extract inside the extraction system were controlled by 5 valves and the pressure was monitored at 3 points (not shown). Extraction temperature was monitored with thermocouples at nine different points at three different heights inside the extraction vessel. The thermocouples were placed inside hollow metal rods, which were placed into the extraction vessel. The thermocouples were within 10 cm from each another. There was also one thermometer in the outgoing pipe to measure the temperature of the extract.

Thermocouples were connected to the computer with the Labview (National instruments, USA) data collection hardware. Collection software was self-programmed with Labview programming language to monitor and collect the temperature data during the extraction.

The extract was collected in a container with a volume of one cubic meter. The container was placed on a scale to monitor the weight of the extract during and after the extraction. All pilot scale extractions were done using tap water. Spruce sawdust was pre-steamed similarly as in the laboratory scale extractor by letting the hot water evaporate in atmospheric pressure. The hot steam (100 °C) heated the sawdust inside the extraction vessel and in addition decreased the channeling of water flowing through the large sawdust bed. The vents were closed and the extraction vessel was pressurized in the end of heating phase.

Extractions

PHW flow-through extractions were conducted using similar relative vessel dimensions, sample amounts proportional to vessel volumes, and similar extraction conditions both in the laboratory and pilot scale systems as shown in Table 2. The same batch of sawdust was used both in the laboratory and in the pilot scale extractions. Extraction parameters for spruce and birch were chosen by combining information from the test extractions and from the previous work done with laboratory scale system.[21–23] Three identical extractions were conducted for birch and spruce sawdust both in the laboratory and the pilot scale.

Table 2. The extraction and extraction vessel parameters for laboratory and pilot scale systems.

	Laboratory scale *(0.05 L)*		**Pilot scale** *(300 L)*	
	Spruce	**Birch**	**Spruce**	**Birch**
Temperature	170 °C	160 °C	170 °C	160 °C
Pre-steaming	Yes	No	Yes	No
Loading (dry sawdust)	8.4 g	11.9 g	55 kg	71 kg
Wood to water -ratio	1:22	1:16	1:22	1:16
Flow rate	3.3 mL/min		20 L/min	
Length/diameter	60/34 mm		1040/590 mm	
Length to diameter -ratio	1.8:1			
Residence time	12 min			
Extraction time	60 min			

Extractions were performed at 160 °C and 170 °C for birch and spruce, respectively. Birch sawdust was extracted at lower temperature to extract mainly xylans from sawdust, while keeping the amount of extracted lignin as low as possible. Spruce sawdust was extracted at higher temperature since less softwood lignin is released during the PHW extraction.[21,23] In the pilot scale the extraction flow rate was set at 20 L/min, preserving the scale up ratio from the laboratory scale. The extraction time was 60 minutes in both cases.

Residence time of water inside the extraction vessel was calculated (Eq. 1) for the laboratory and the pilot scale extraction systems to get equal contact time between water and the sawdust at both scales. Residence time was set to be 12 minutes in all extractions.

Equation 1. Residence time.

$$\tau = \frac{V_{vessel} - V_{dry\,sawdust}}{Q_{water}}$$

τ is residence time of water in the extraction vessel (s), V_{vessel} is the volume of the extraction vessel (m^3), $V_{dry\ sawdust}$ is the volume of the dry sawdust solids (m^3), and Q_{water} is the flow rate of water (m^3/s).

The density of solids,[24] which was used for birch and spruce sawdust, was 1.530 kg/L. The residence time could be controlled by certain water flow rate and the amount of the sawdust in the vessel. First the pilot scale extraction vessel was packed full with spruce or birch sawdust. Then the amount of the sawdust used in the laboratory scale extraction vessel was calculated by keeping the residence time constant. The differences between the loadings are due to the different densities of the sawdust from different kind of wood species. This affected the wood to water ratios, which were 1:16 (w/w) for birch and 1:22 (w/w) for spruce.

Analysis of sawdust and extraction residues

All calculations of wood were done on dry basis. Sawdust samples were taken before and after extraction. Sawdust samples were freeze-dried to a constant weight before sample analysis. To analyse the moisture contents the aliquots from pilot scale sawdust samples were oven-dried at 105 °C. The laboratory scale sawdust samples were freeze-dried due to small sample amount. Weight loss was calculated by measuring the weight and moisture content of the sawdust before and after the extraction. If the samples were not analysed immediately, they were stored in a dark freezer at -20 °C in closed polyethylene bags.

The amount and composition of the hemicelluloses in the sawdust and extraction residue were determined by acid methanolysis.[25] Acid methanolysis at 100 °C for 5 hours depolymerizes the hemicelluloses and pectins of the sample into monomeric sugars and form methyl glycosides. An internal standard resorcinol was added into the samples and the calibration samples. Samples were silylated and analysed by Shimadzu GC-2010 (Shimadzu, Kyoto, Japan) gas chromatograph (GC). The results are reported as amounts of anhydrosugars.

The amount of cellulose in wood was determined by using acid hydrolysis.[26] Samples and cotton linters (Sigma Aldrich) as calibration standard were hydrolysed and samples were left overnight. Next day samples were autoclaved at 121 °C for 1 hour. After neutralization with $BaCO_3$ the internal standard sorbitol was added into the samples. Samples were silylated overnight and analysed with the same GC-FID used in the hemicellulose analysis. The results are reported as amounts of anhydrosugars.

Lignin content was determined as Klason lignin.[27] Wood extractives were determined gravimetrically from sawdust. Sawdust was extracted using an Accelerated Solvent Extractor (Dionex, USA) with acetone/water (95/5, v/v). Extracts were dried first under nitrogen flow and then in vacuum oven at 40 °C to dryness.

Analysis of extracts

To monitor the repeatability of the extractions, samples were collected in 10 minutes fractions during both the laboratory scale and the pilot scale extractions. Total extraction time was 60 minutes and it started when the first drop of the extract reached the collection container. The amount of extracted hemicelluloses in extracts was monitored and estimated using a hand-held refractometer (VWR). If the samples were not analysed right after the extraction, they were placed in a freezer at -20 °C. Samples were mixed thoroughly before analyses. Total dissolved solids (TDS) were determined in aliquots of extracts after oven-drying at 105 °C.

The extracted hemicelluloses were analysed as solid samples. Extracts were frozen (-20 °C) and then freeze-dried to dryness. Hemicelluloses in the dried samples were depolymerized with acid methanolysis at 100 °C for 3 hours.

The average molar mass (M_w) of the extracted hemicelluloses in the extracts was determined using a HPSEC with a refractive index detector (RID) (Shimadzu Corp., Tokyo, Japan) and a multiangle laser scattering (MALLS) detector (miniDAWN, Wyatt Technology, Santa Barbara, CA, USA). A HP-SEC system had a guard column (Ultrahydrogel 6 mm x 40 mm; Waters, Milford, MA, USA) and two main columns (UltrahydrogelTM linear 8.9 mm x 300 mm; Water, Milford, MA, USA) connected in series. The eluent was 100 mM $NaNO_3$ with flow rate 0.5 mL/min. A dn/dc value of 0.15 mL/g for xylan and galactoglucomannan was used for molar mass calculations.

Lignin determination was done by adding the aliquot of sample to 0.1 M NaOH solution and measuring UV absorbance at 280 nm with a Shimadzu UV-2401PC (Shimadzu, Kyoto, Japan). Extinction coefficient 18.5 L/g·cm was used for birch and 20.3 L/g·cm for spruce. Extinction coefficients were calculated from values obtained from the previously isolated and purified PHWE lignin. The pH of the extracts after extraction was measured at room temperature with a pH-meter (Five easy, Mettler-Toledo).

The amount of the extracted hemicelluloses was estimated with a refractometer (VWR, Finland) using °Brix scale. Few droplets of extract were placed on the refractometer and °Brix value was read from the scale. The amount of the extracted hemicelluloses was calculated from the °Brix results with a correction factor that was calculated for birch xylan and spruce galactoglucomannan. The correction factor was calculated by comparing results from acid methanolysis and °Brix values (Figure 3). With hemicelluloses concentrations over 3 °Brix, the accuracy of the °Brix measurement suffered slightly because of the increased lignin content.

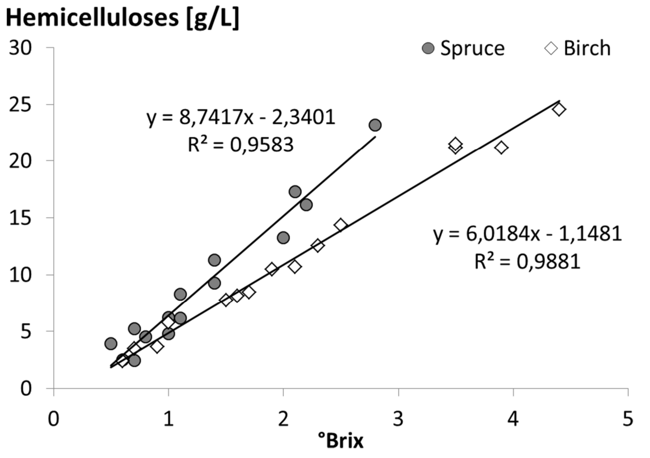

Figure 3. Linear regression of °brix and extract's hemicelluloses concentration for birch and spruce obtained by acid methanolysis.

Results and discussion

Chemical composition of the raw material

Cellulose, hemicelluloses, lignin, and extractives content were analysed from the birch and spruce sawdust. The results are shown in Table 3.

Table 3. The chemical composition of the birch and spruce sawdust.

[wt%] of wood	Birch	Spruce
Cellulose	39	42
Hemicelluloses	30	23
Lignin	21	26
Extractives	3	3
Other compounds	7	6
Sum	**100**	**100**

Spruce sawdust contained more cellulose and lignin and less hemicelluloses compared to birch sawdust. The other compounds include the acetyl groups of the hemicelluloses and minor amounts of proteins and ash. They were calculated by subtracting the sum of known compounds from the total.

Temperature and pH profile of the PHW extraction

Temperature is the most important single factor which affects the hemicelluloses yield with plain water.[21,23] The average temperature profiles for birch and spruce extractions are presented in Figures 4a and 5a, respectively.

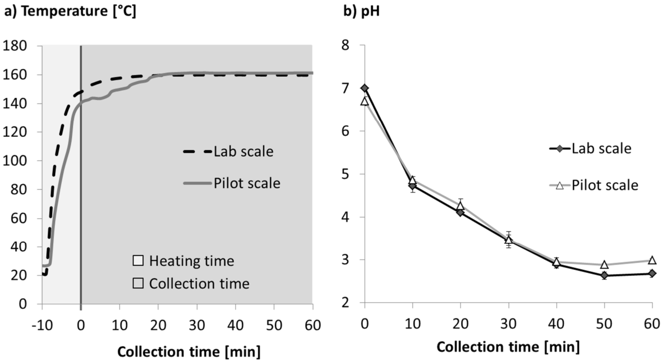

Figure 4. Birch extraction a) temperature and b) pH profiles in both laboratory and pilot scale extractions. Pilot scale temperature is an average of nine measuring points.

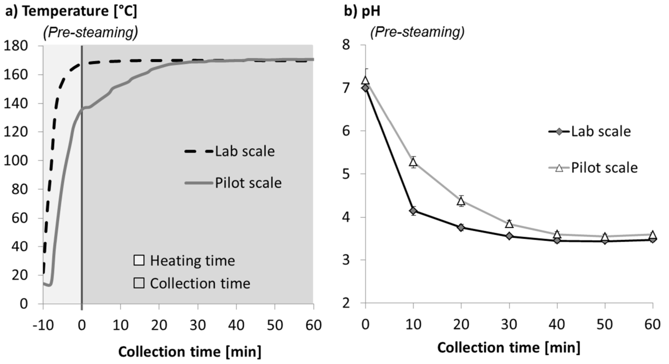

Figure 5. Spruce extraction a) temperature and b) pH profiles of laboratory and pilot scale extractions. Pilot scale temperature is an average of nine measuring points.

The extraction vessel heats up faster in the laboratory scale than in the pilot scale. In the laboratory scale the extraction vessel is placed inside the oven, which heats both the extraction vessel and the water flow. Pre-steaming further enhances the heating. The pilot scale does not have any similar external heating system as the laboratory scale system. The pilot scale system warms only by the in-flow of the preheated water. Figures 4b and 5b show the effects of the temperature on the pH of the extract. The faster the temperature rose, the more the pH decreased. The decrease in the pH results from the acetic acid[28] and uronic acids, which are liberated from the hemicelluloses and pectin chains during the autohydrolysis.[21,23]

Hemicelluloses

The main product obtained from PHW extraction of sawdust is a hemicellulose-rich fraction. The amount of the extracted hemicelluloses in the laboratory and the pilot scale are shown in Figure 6a for birch and in Figure 7a for spruce wood. The amounts and extraction rates of the hemicelluloses extracted from the birch sawdust were similar for both laboratory and pilot scale. The total yield of birch hemicelluloses obtained during the extractions was 50% with a relative standard deviation (RSD) of 3.2% for laboratory and 4.2% for pilot scale extractions.

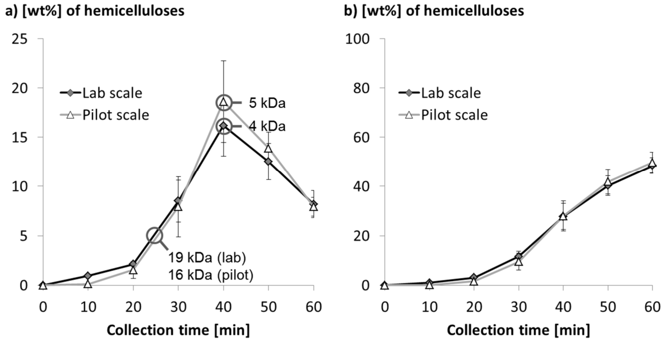

Figure 6. a) Birch hemicelluloses yield, and molar masses b) cumulative yield from the laboratory and pilot scale PHW extractions. Error bars are relative standard deviations of three parallel extractions. The amounts of hemicelluloses are measured with °Brix scale.

The yield of extracted hemicelluloses increased after 20 min and the highest concentration was obtained at 40 min of extraction time. The average molar mass (Mw) of birch hemicelluloses decreased during the extraction due to the hydrolysis.

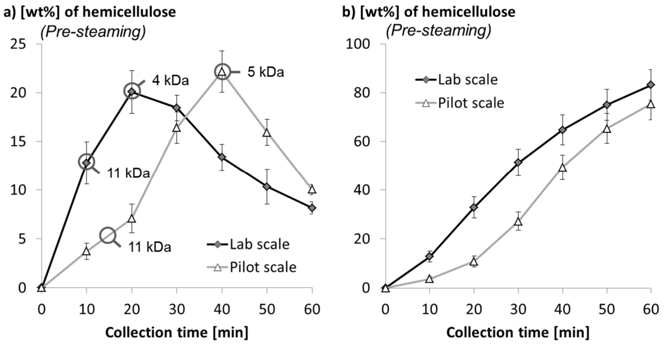

Figure 7. a) Spruce hemicelluloses yield and molar masses b) cumulative yield from the laboratory and pilot scale PHW extractions. Error bars are relative standard deviations of three parallel extractions. The amounts of hemicelluloses are measured with °brix scale.

Laboratory and pilot scale extractions gave varied results for spruce (Figure 7a). The maximum of the hemicellulose yield was reached at 20 minutes in laboratory and at 40 minutes in pilot scale. This can be due to faster warming of the laboratory scale extraction vessel as seen in Figure 5a which was followed by a lower pH of the extract (Figure 5b). It is essential to have similar temperature profiles to successfully scale up the extraction process. Nevertheless, the total hemicellulose yield settled near 80% being exactly 83% ± 6% in laboratory and 77% ± 7% in pilot scale. Probably due to better heat transfer, the extraction rate of hemicelluloses was higher in the laboratory scale experiments (Figure 7b).

The decrease of the hemicellulose molar mass (Mw) depends on the extraction temperature, pH, and the residence time inside extraction vessel[22]. With these extraction parameters, the molar mass of the hemicelluloses obtained from birch was 16–19 kDa in the beginning of the extraction and 4–5 kDa for the peak of the hemicellulose yield. For the spruce, the molar masses were 11 kDa and 4–5 kDa, respectively. The decrease in the molar mass is due to the hydrolysis of hemicelluloses.[23,29] The longer time the hemicelluloses stay at high temperature or lower pH, the more hemicelluloses are cleaved into smaller fragments.

Hemicelluloses were analysed from the original sawdust, extracted sawdust, and extract. Figure 8a and 8b show the sugar monomer ratios of the hemicelluloses in the birch and spruce samples, respectively.

The sugar ratios of extracted sawdust and extract were similar when the pilot and laboratory scales were compared (Figure 8). However, the birch sawdust extracted with the pilot scale system contained relatively more glucose and less xylose compared to the laboratory scale, which could be a result from the cooling down phase of the pilot vessel. The pilot scale extraction vessel was cooled down by running cold water through the extracted sawdust. In this phase some of the xyloses could still be extracted from sawdust. The extracted sawdust had also longer treatment time taken due to the cooling of larger 300 L extraction vessel. Acid methanolysis can cleave amorphous cellulose parts, which can be seen as excess glucose (not part of the hemicelluloses). Therefore the relative amounts of xyloses were smaller and glucose amount larger in the extracted sawdust in pilot scale. Similar effects can also be seen with spruce (Figure 8). The largest amount of glucose would be from galactoglucomannan, but small amounts of glucose could also be cleaved during the extraction from cellulose in the fibres.

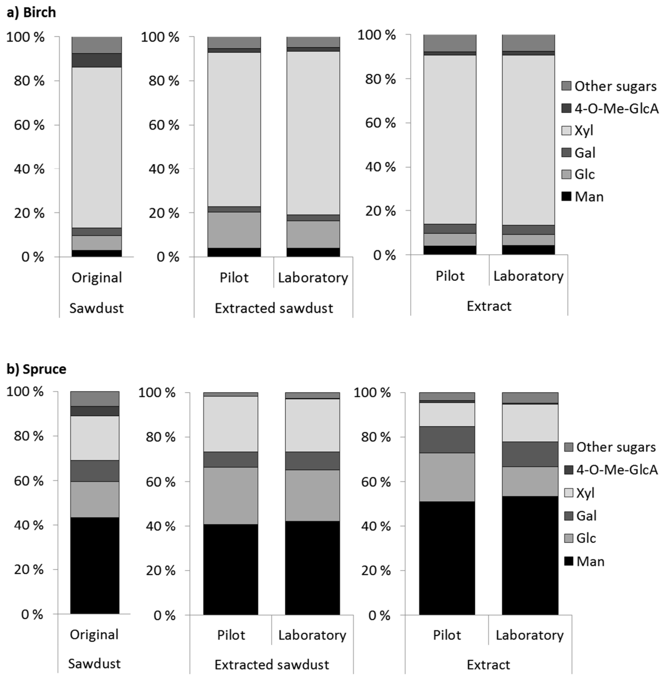

Figure 8. The hemicelluloses analysed with acid methanolysis in the a) birch and b) spruce sawdust, extracted sawdust, and extract. Other sugars include arabinose, rhamnose, glucuronic acid, and galacturonic acid.

Mass balances

During the extractions more than 20% of the birch and spruce sawdust was dissolved into the extract. During the cooling down phase in pilot scale some of the hemicelluloses and lignins were still extracted out from the sawdust. The extract collected during the cooling down phase was taken into account in mass balances (Figure 9a and 9b).

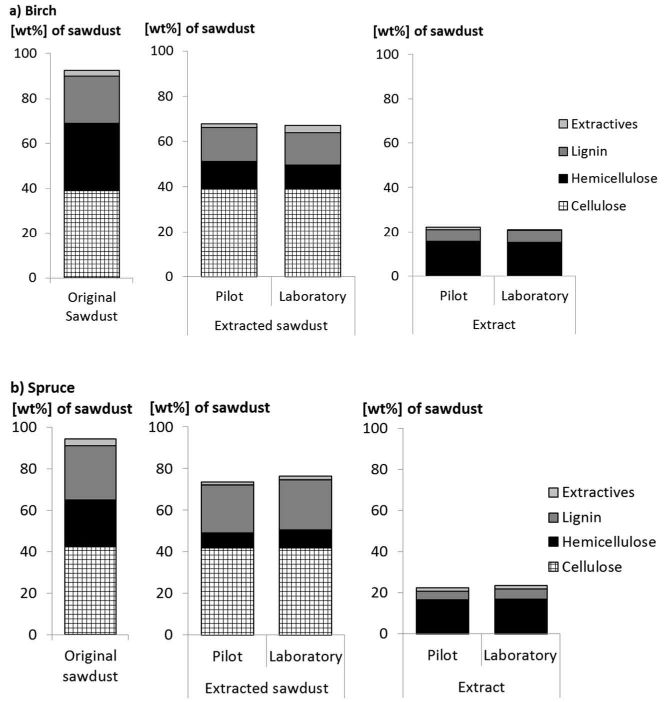

Figure 9. The mass balances of a) birch and b) spruce extractions.

There were no significant differences in mass balances between the pilot and laboratory scale extractions. Hardly any of the cellulose was dissolved in laboratory and pilot scale PHW extractions. The temperatures used in the experiments were not high enough for cellulose degradation to occur.[21,28]

The extraction yield of the hemicelluloses can be controlled by varying the extraction temperature, time and flow rate. In these experiments about 50% of birch and 80% of spruce hemicelluloses were dissolved.

In case of wood extractives, about half of the extractives were found in the extract and half was retained in the sawdust for both birch and spruce. Some part of the lignin was also extracted, but mainly the lignin remained in the sawdust. Somewhat more lignin was extracted from the birch sawdust (24%) than spruce sawdust (15%). This could be explained by the differences of the hardwood and softwood lignin structure and their cell wall topochemistry.[30]

Figure 10 shows the distribution of the wood compounds in the case when 1000 kg of sawdust would be extracted. During the extraction, acetic, uronic, and other organic acids were released and some furfurals and other compounds were formed. In the previous experiments,[21] the amount of furfurals in extracts was very small. Acetic acid and furfural can be evaporated when samples are dried and are not included in the calculations. With birch, this kind of weight loss can be larger than with spruce due to the larger amount of acetic acid released from xylans. In addition to extractives, lignin, hemicelluloses and cellulose, sawdust, and extracts contained some ash and proteins, which were not analysed. These missing compounds a.k.a. acetic acid, ash, and proteins are included as others in the Figure 10.

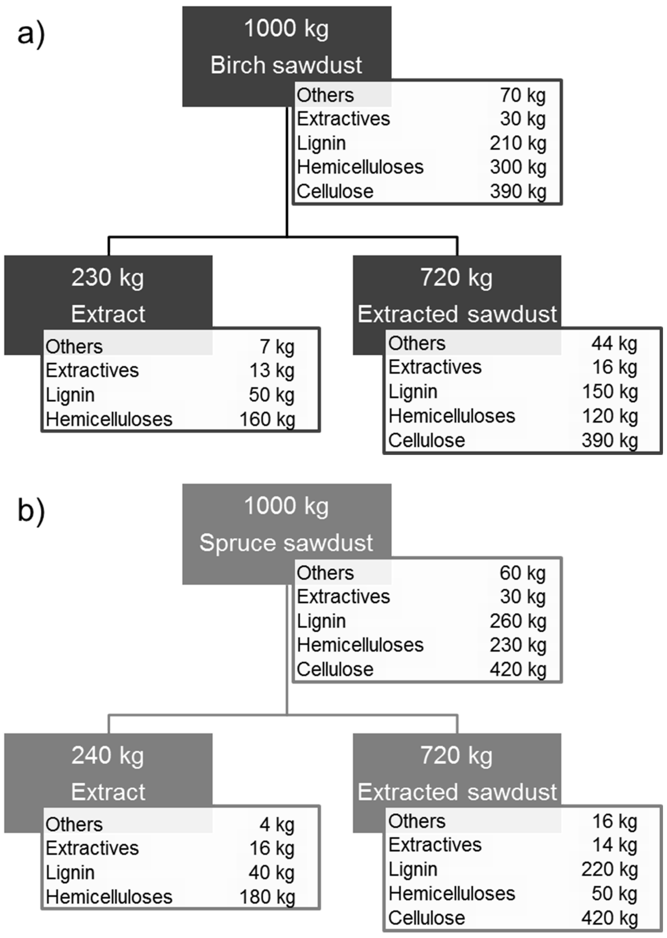

Figure 10. The distribution of the wood compounds between pilot scale extract and extracted sawdust for a) birch and b) spruce.

Performance of the pilot scale system

The information of the performance of the pilot scale system is important in further upscaling.[31] Warming and cooling down the laboratory and pilot extraction vessels were not similar, since the pilot scale flow-through extraction system had larger heat capacity, mass and the pilot scale system was heated only with hot water. The channeling of water through the sawdust bed can mostly be avoided in a smaller laboratory scale system, but in the pilot scale the channeling effect was still present. Channeling can occur if sawdust packing and movement create voids in the sawdust bed. Water moves more rapidly in these voids than in other parts of sawdust bed.

Channeling was monitored with nine thermocouples inside the extraction vessel, which were in three different heights. When the extraction vessel was first filled with sawdust and hot water was flown into the vessel, the temperature of the thermocouples near the side of the vessel heated up first (Figure 11). Also heating was not similar in every parts of the sawdust bed, indicating channeling of water. When the experiment series with the spruce sawdust was first done, the similar channeling was observed. To reduce channeling, spruce sawdust was heated with 100 °C steam before extractions. Pre-steaming reduced channeling significantly (Figure 11) and more even heating of the sawdust bed was possible. Pre-steaming could also re-arrange sawdust bed more evenly by removing voids and loosening the more dense parts of sawdust.

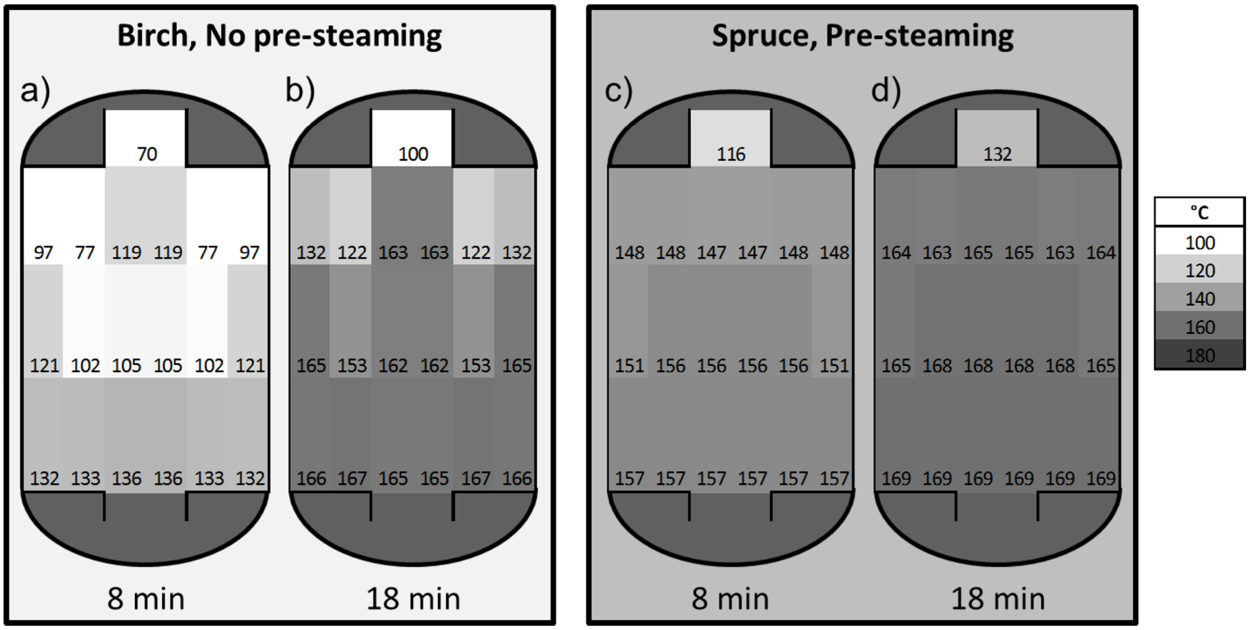

Figure 11. Temperatures from the nine thermocouples at the right side of extraction vessel are mirrored to the left side. a) 8 min and b) 18 min and spruce with pre-steaming c) 8 min and d) 18 min from the beginning of the heating phase.

Black precipitate has been reported to be formed in batch extractions at temperatures near 200 °C.[28,31] In the pilot scale experiments, lignin-derived compounds and other extracted compounds started to precipitate when the temperature of the extract decreased. Some of the formed precipitate stuck on the surfaces of the heat exchangers and pipelines. Some part of these precipitates could have been so-called pseudo-lignin from carbohydrates.[33] There was also some precipitation in the containers where the extracts were stored, especially when the birch sawdust was used.

The flow rate of the system was monitored during and after the extraction in the cooling down phase. The maximum flow rate of the system decreased after 50 runs and therefore the pilot scale extraction system was inspected thoroughly. Brown or black lignin-kind of sticky precipitate was found inside the heat exchangers. The system had to be cleaned regularly in order to keep it operating day-to-day. The cleaning was performed with AlfaCaus cleaning agent by Alfa-Laval, which contained sodium hydroxide and detergents. If the system was used tens of times continuously without cleaning the heat exchangers, it resulted into a total clogging of the system. Totally clogged heat exchangers were successfully cleared with a hot steam treatment (300 °C) followed by AlfaCaus back flush cleaning.

The typical extraction consumes the average of 230 kWh of electricity depending on the extraction temperature. Some heat can be recovered with heat exchangers. The average water consumption was 6 m^3, which includes heating and cooling down water. The excess low TDS water could be e.g. ultrafiltrated and water could be used again in the extractions. Hemicellulose extracts could also be concentrated with ultrafiltration.[4] The costs of the extraction, concentration, and purification could be decreased if the PHW extraction system could be integrated into larger industry for example pulp or sawmills.

Conclusions

The pilot scale extractions were repeatable and scalable. The PHWE system was scaled by factor of 6000, from 50 mL to 300 L. Laboratory scale extractions gave valuable information about different extraction parameters, such as extraction temperature and time. Temperature and pH profiles were different when pre-steaming was used with spruce sawdust in the pilot scale. Still overall extraction yield was similar in laboratory and pilot scale and sawdust was heated more evenly with less channelling effects. In the PHWE flow-through extraction system 50% of birch and 80% of spruce hemicelluloses were extracted. There were no significant differences in hemicellulose composition and mass balances between laboratory and pilot scale. Pilot scale extractions gave important information about overall performance and channelling, which could not be noticed in laboratory scale extractions.

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