



## Extraction of hemicelluloses from fiberized spruce wood



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### ABSTRACT

A novel mechanical pre-treatment method was used to separate the wood chips into fiber bundles in order to extract high molecular weight wood polymers. The mechanical pre-treatment involved chip compression in a conical plug-screw followed by defibration in a fiberizer. The fiberized wood was treated with hot water at various combinations of time and temperature in order to analyze the extraction yield of hemicelluloses at different conditions. Nearly 6 mg/g wood of galactoglucomannan was obtained at 90 °C/120 min which was about three times more than what could be extracted from wood chips. The extracted carbohydrates had molecular weight ranging up to 60 kDa. About 10% of each of the extracted material had a molecular weight above 30 kDa. The extraction liquor could also be reused for consecutive extractions with successive increase in the extraction yield of hemicelluloses.

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### 1. Introduction

Norway Spruce (*Picea abies*) contains 25–30% hemicelluloses, of which about 18% is present in the compound middle lamella, 19% in the S1 layer and 63% in the S2 and S3 layers of the secondary cell wall (Fengel & Wegener, 1983; Sjöström, 1993). The extractability of hemicelluloses from wood is limited, probably due to that lignin crosslink the different polysaccharides which decreases their solubility (Lawoko, Henriksson, & Gellerstedt, 2006), but a portion of these hemicelluloses is soluble in hot water or even water at room temperature (Casebier, Hamilton, & Hergert, 1969; Örså, Holmbom, & Thornton, 1997; Willför & Holmbom, 2004). The amount of water soluble hemicelluloses is large at high temperatures and long treatment times, however, hydrolytic cleavage degrades the polysaccharides at elevated conditions (Lundqvist et al., 2002; Song, Pranovich, Sumerskiy, & Holmbom, 2008). A considerably large amount of hemicelluloses can be extracted from ground wood compared to wood chips (Song et al., 2008) on the cost of low molecular weight hemicelluloses and a wet solid residue having a value corresponding to the fuel value. The dissolution is even more extensive from thermomechanical pulp (Thornton, Ekman, Holmbom, & Örså, 1994; Willför, Sjöholm, et al.,

2003). The refining process in thermomechanical pulping (TMP) vigorously cracks S1/S2 fiber walls, which prompts the dissolution of hemicelluloses from cell walls into process water. Hemicellulose from TMP process water can be fractionated with high purity by combining different techniques; for example, ultrafiltration and chromatography (Westerberg et al., 2012; Willför, Rehn, et al., 2003). Water soluble polysaccharides mainly consists of O-acetyl-galactoglucomannans (AcGGM) (Lundqvist et al., 2003; Thornton et al., 1994) with molecular weight ranging up to as high as 60 kDa (Lundqvist et al., 2002; Willför, Sjöholm, et al., 2003). Other water soluble substances include mainly acidic arabinogalactans, some xylans and lignin (Lundqvist et al., 2002; Willför & Holmbom, 2004).

Potential applications of hemicelluloses are still exploring. To date hemicelluloses have been tested for making films to be used as gas barriers with promising mechanical properties (Edlund, Ryberg, & Albertsson, 2010; Höije, Gröndahl, Tømmeraa, & Gatenholm, 2005; Mikkonen, Heikkilä, Willför, & Tenkanen, 2012), as an additive in paper making to improve paper strength properties (Hannuksela, Fardim, & Holmbom, 2003; Hartmans et al., 2009) and as hydrogels (Gabrieli, Gatenholm, Glasser, Jain, & Kenne, 2000; Lindblad, Ranucci, & Albertsson, 2001).

TMP is an energy intensive process and most of the energy is converted to heat while refining. This is a large economical and environmental problem both when TMP is used for classical pulping for paper properties and also for the method as a pre-treatment stage in biorefinery concepts. However, in recent years much work has been done to understand the mechanism of mechanical pulping on the wood structure in order to reduce the energy demand

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during refining. Mechanical pre-treatment of chips combined with increased refining intensity has been demonstrated to considerably reduce the energy demand in a novel process called advanced thermomechanical pulping (ATMP) (Johansson, Hill, Gorski, & Axelsson, 2011). The pre-treatment stage in ATMP consists of an impressafiner and a fiberizer. The impressafiner is a screw-press where chips are compressed at a high strain in a pressurized environment. A fiberizer is essentially a small moderately pressurized refiner which defibrates the wood chips into fiber bundles called as fiberized wood (Hill, Johansson, Sabourin, & Aichinger, 2009; Hill et al., 2010). The compressive pre-treatment separates the wood fibers and creates cracks in S1 and S2 fiber walls which increases the surface area and decrease the mass transport length (Kure, Dahlqvist, Sabourin, & Helle, 1999). Compressive pre-treatment prior to main-line refining saves up to 20% energy compared to conventionally produced TMP pulps (Sabourin, Aichinger, & Wiseman, 2003). Integrating a hot water extraction system together with mechanical pre-treatment in the primary stages of either mechanical or chemical pulping processes will, in addition to saving energy, produce valuable products from isolated hemicelluloses which otherwise are degraded and/or dissolved in the processing liquors (Heiningen, 2006; Lisboa, Evtuguin, Neto, & Goodfellow, 2005).

In this study, fiberized spruce wood has been subjected to hot water extractions with the goal to investigate its possible role in a biorefinery context by extracting high molecular weight galactoglucomannan (GGM) and that the solid residue could be used as a raw material in the pulping processes. Different extraction conditions of time and temperature were used in order to investigate the effect on the amount and molar mass of isolated polysaccharides. Since the aim was to isolate relatively native hemicelluloses therefore elevated treatment conditions were not studied due to the risk of polymer degradation. The extracted material was investigated for lignocellulosic composition, molar mass distribution and acetyl content. An attempt to reuse the extraction liquor containing dissolved solids from the wood has also been made in order to foresee the potential in a mill application to increase the concentration of hemicelluloses in the extraction liquor.

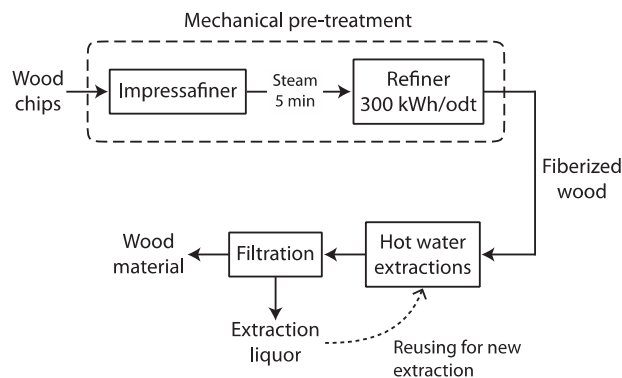
## 2. Experimental

### 2.1. Material and mechanical pre-treatment

Chips of Norway spruce (*P. abies*) were obtained directly after the impressafiner from Braviken paper mill, Holmen Paper AB in Norrköping, Sweden. Impressafined chips were steamed for 5 min with atmospheric steam and fiberized using a pilot scale 12" disc refiner (Sprout-Waldron) at Chalmers University of Technology, Sweden. The net energy input of the refiner was adjusted to 300 kWh/odt wood by adjusting the speed of the conveyer belt delivering wood to the refiner in a single pass mode. Fiberized wood was stored in the dark at  $-24^{\circ}\text{C}$  and thawed overnight at room temperature prior to further use.

### 2.2. Extractions

Distinct batches of fiberized wood were extracted with water at 30, 60 and  $90^{\circ}\text{C}$  for different times ranging from 5 to 120 min. For comparison, frozen spruce wood chips were thawed overnight and subjected to hot water extraction at  $90^{\circ}\text{C}$  for different times. For each batch of extraction, 500 ml of deionized water was first heated in a beaker to the target temperature followed by the addition of 10 g (dry weight) of fiberized wood or wood chips. The temperature drop due to the addition of wood, was recovered in 2–3 min, however a variation of  $\pm 2^{\circ}\text{C}$  was noted throughout the treatment. The water–wood mixture was stirred with a mechanical stirrer



**Fig. 1.** Schematic diagram depicting the mechanical pre-treatment of spruce wood chips followed by hot water extraction. The extraction liquor was reused multiple times with new batch of fiberized wood.

using an axial flow impeller rotating at  $\approx 400$  rpm. Subsequently, the liquid was separated from the wood using a fiber cloth sieve (Monodur PA-71, Mesh opening  $71\ \mu\text{m}$ ) under vacuum, followed by washing the wood with 100 ml deionized water. The liquid was again vacuum-filtered through a glass fiber filter (Whatman GF/A,  $1.6\ \mu\text{m}$ ) to remove any particles or colloids. The filtrate was allowed to cool and the pH was recorded at room temperature. Filtrate containing dissolved solids from wood was concentrated by vacuum evaporation using a water bath at  $50^{\circ}\text{C}$  and lyophilized.

For reusing the extraction liquor, six consecutive extractions were performed at  $90^{\circ}\text{C}$  for 120 min. The liquid containing dissolved solids from the first extraction was reused for extraction with a new batch of fiberized wood and was further used to carry four more batch-wise extractions. A 10 g (dry weight) of fiberized wood was used in each batch. Extraction liquid in each stage was filtered the above-mentioned way before subjecting to the following extraction (Fig. 1).

### 2.3. Analysis

#### 2.3.1. Gravimetric analysis

The total amount of material dissolved from the fiberized wood was calculated gravimetrically by lyophilizing the extracted aliquots.

Lignin content in the extracts was determined by subtracting the amount of Klason lignin in the remaining wood after extraction, from the lignin present in the starting fiberized wood. For lignin analysis, wood samples were dried at  $105^{\circ}\text{C}$  and ground to pass a 40-mesh screen. 200 mg of ground wood was hydrolysed employing conditions described in a standard method (SCAN, 2009). The hydrolysates were filtered and the insoluble part on the filter paper was dried and weighed giving the amount of Klason lignin.

#### 2.3.2. Carbohydrate composition

Carbohydrate composition of the lyophilized extracts was determined after acid hydrolysis (SCAN, 2009). Monosaccharide content of the hydrolysate was analyzed with high-performance anion-exchange chromatography (Dionex, Sunnyvale, CA, USA) equipped with pulsed amperometric detector (HPAEC-PAD) using a CarboPac PA-1 column ( $4 \times 250\ \text{mm}$ ). The system was equilibrated for 7 min with 260 mM sodium hydroxide and 170 mM sodium acetate followed by Milli-Q water for 6 min. Only Milli-Q water was used as eluent at a flow rate of 1 ml/min. A solution of 300 mM NaOH was added to the column effluent before the PAD cell at a flow rate of 0.5 ml/min. Data was processed with Chromeleon 7.1 software.

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