Recovery of water-soluble acetylgalactoglucomannans from mechanical pulp of spruce

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ABSTRACT: This paper presents a method for recovering acetylgalactoglucomannans from mechanical pulp at a yield of 5 kg/ton pulp, at 95% purity. The acetylgalactoglucomannans can be obtained in dry state for later use or as a concentrate for targeted use in the wet-end of the paper machine. Possible benefits of using mannans in the wet-end include improved paper machine runnability and better paper strength. This method also provides a convenient means of interstage washing in a mechanical pulp mill, with the additional benefit of possible recovery of valuable substances such as wood resin and aromatic substances, including lignans. In the method, TMP water, containing dissolved and colloidal substances, is prepared from mechanical pulp. A cationic organic coagulant, followed by filtration, serves to remove dispersed colloidal wood resin. Dissolved aromatic substances, mainly lignans and lignin, are then removed by sorption to polyacrylate resin. Further purification is accomplished by membrane filtration using a 20 kDa filter, giving a concentrate enriched in acetylgalactoglucomannans that can be used as such in the wet-end of the paper machine. Pure acetylgalactoglucomannans are recovered for later use by precipitating the acetylgalactoglucomannans in ethanol. We also discuss some alternative purification and drying steps.

Application: The method described in this paper enables mills producing mechanical pulp and wood-containing paper to improve runnability and paper strength and produce valuable by-products for commercialization.

Galactoglucomannans are the main hemicellulose type in softwoods and are normally present in amounts of 15%-20% of the wood material [1]. Of these galactoglucomannans, about 5%-10% are dissolved during mechanical pulping of spruce wood [2]. Spruce galactoglucomannans sterically stabilize pitch emulsions by forming a protective hydrogel layer around the pitch droplets [3, 4]. The galactoglucomannans also decrease the deposition tendency of pitch [5, 6]. The galactoglucomannans also sorb to cellulose-rich fiber surfaces [7] and positively affect fiber-fiber bonding, and thus paper strength [8, 9].

A major portion of the water-soluble carbohydrates in spruce wood dissolve in hot water—and even water at room temperature—after even a short treatment time at neutral or slightly acidic pH [10, 11]. The dissolution is even more extensive from spruce thermomechanical pulp (TMP), partly due to the extensive fibrillation caused by the TMP process [11]. Mainly neutral galactoglucomannans and some acidic arabinogalactans dissolve under these conditions [10, 11]. The dissolved galactoglucomannans consist of a backbone of mannose and glucose units with galactose side groups linked at C-6 to mainly mannose [11-13]. The monosaccharide unit ratio for the water-soluble spruce TMP galactoglucomannans is about 4:1:0.5-1 (mannose:glucose:galactose) [9, 10] and the degree of polymerization is about 370 [11]. The spruce galactoglucomannans are naturally acetylated, with a degree of acetyl group substitution of about 0.26, at C-2 and C-3 in the mannose units in the backbone. The solubility of the acetylgalactoglucomannans relates to the content of galactose groups and acetyl substituents [14]. This is also verified by the fact that deacetylation at high pH [15] leads to lower solubility and partial deposition onto fibers [7, 16].

Galactomannans—mostly chemically modified guar gums—have had long use in papermaking [17], but the use of wood-derived galactoglucomannans is negligible. Our group has recently developed an improved method for recovering acetylgalactoglucomannans, as well as other substances generally considered as contaminants, from wood material during mechanical pulp processing [18]. This method also covers the possibility of interstage washing, for which there is a growing interest because of the trend toward more tightly closed water systems [19] and the potential recovery of valuable by-products such as wood resin, lignans, lignin and other aromatic substances. In this paper, we present results from an application for recovering acetylgalactoglucomannans at a large laboratory scale.

EXPERIMENTAL

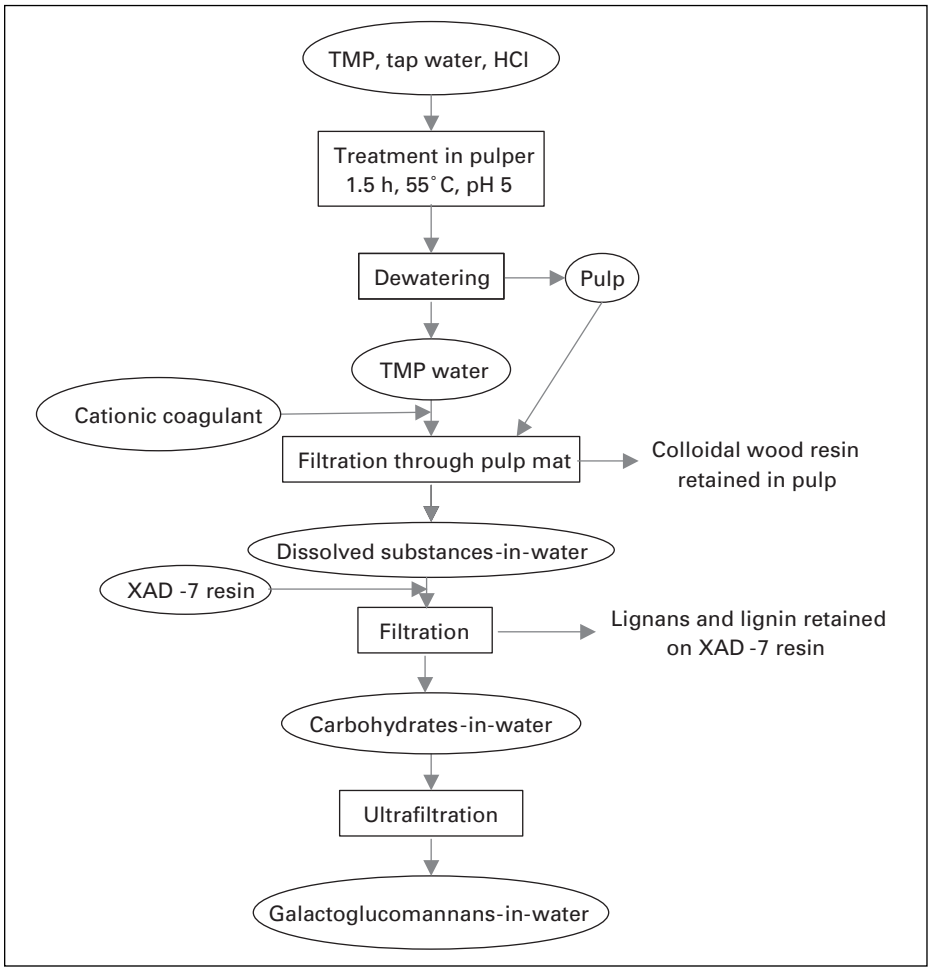
Materials

For this study, we used thermomechanical pulp produced from Norway spruce (*Picea abies*) taken after the first refiner in a Finnish mill using two-stage refining. The pulp consistency was about 40% and the pulp was stored at -24°C. The pulp freeness was 720 CSF.

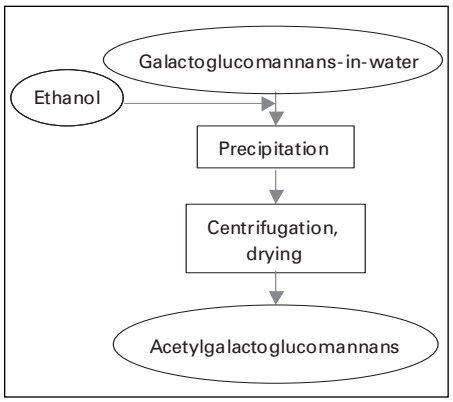
We also used technical grade Amberlite XAD-7 resin, a polyacrylate produced by Rohm and Haas UK. Other researchers recommend purifying the resin before use by sequential Soxhlet extraction with methanol, acetonitrile, and diethyl ether [20]. Because of the large amount of resin used in our experiment, we only washed it with ethanol while stirring. The washed resin was stored in a cold room in technical grade ethanol. Most of the ethanol was removed from the XAD-7 resin by filtration (suction-dry resin) before use.

Basic procedures

TMP-water, containing dissolved and colloidal substances from the pulp, was prepared by suspending 5 kg o.d. TMP in 125 L tap water (4% suspension) (**Fig. 1**). The pH was adjusted to about 5 by adding 60 mL 1 M HCl. The suspension was treated in a pulper (5.5 kW, propeller 1020 rpm) for 1.5 h at 55±5°C, and then pumped to a vat equipped with a 200-mesh metal



1. Method scheme for recovery of an acetylgalactoglucomannan concentrate from TMP.



2. Method scheme for recovery of pure acetylgalactoglucomannans in dry state for later use.

wire and a tap outlet in the bottom. We dewatered the pulp by filtration and collected the TMP water in another vat.

A starch-based cationic polymer (11 g o.d., Raifix 25035, Raisio Chemicals Oy) removed the colloidal wood resin from the water by flocculation. We added an excess of the polymer to the TMP water, which we then allowed to stand for 15 min. The TMP water was then filtered through the pulp mat, formed in the dewatering stage, until the water was clear and thus the colloidal wood resin retained in the pulp mat. Finally, we used 80 L of tap water for displacement washing of the pulp.

We added 5 kg suction-dry XAD-7 resin to the obtained water (140 L), containing only dissolved matter, and stirred the suspension for 15 min. The XAD-7 resin, now containing sorbed lignans and lignin, was removed by filtration through a vat. We further purified the obtained water (135 L), which contained mainly dissolved neutral carbohydrates, by ultrafiltration using an Ultrafilter CR 200/1 apparatus (Valmet Flootek) equipped with a membrane with a cut-off of 20 kDa. The rate of flow-through was about 4 L/h. The filter residue (1.7 L) was recovered.

We further concentrated the water (to 0.5 L) by vacuum evaporation in a water bath set at 50°C. We added 4 L of technical-grade ethanol to the concentrate and allowed the acetylgalactoglucomannans to precipitate overnight in a cold room (**Fig. 2**). The precipitated acetylgalactoglucomannans were concentrated by centrifugation at 500 g for 15 min., after which they were washed with ethanol p.a., methanol, and methyl *tert*-butyl ether (MTBE). The acetylgalactoglucomannans were dried in a vacuum drier, weighed, and analyzed.

Analytical methods

We used acid methanolysis followed by silylation and gas chromatography (GC) to analyze the carbohydrate sugar composition and amount [21].

The wood resin and the lignans were determined as groups, i.e., free fatty acids, resin acids, lignans, sterols, steryl esters, and triglycerides, after extraction of the aqueous samples with MTBE, by silylation and GC analysis [22].

We measured the UV absorbance at 280 nm on the aqueous samples, after extraction with MTBE, to estimate the concentration of lignin [22].

The average molar mass was determined on a multi-angle laser light scattering apparatus (DAWN-DSP-F, WYATT Technologies) combined with a Waters GPCV 2000, having two Shodex columns (OH-PAC) in series [7].

The degree of acetylation of the final product was determined by dissolving an exact amount of acetylgalactoglucomannans (70-80 mg) in distilled water (40 mL). We adjusted the pH of the aqueous sample to 11 with 0.5 M sodium hydroxide and then mixed the sample for 2 h at 60°C. This ensured complete deacetylation of the acetylgalactoglucomannans. The sample was allowed to cool down before titration to starting-pH with 0.05 M sulfuric acid. We calculated the degree of acetylation from the amounts of added base and acid, taking into account the amount of galacturonic acid in the final product.

RESULTS AND DISCUSSION

TMP water

In the present study, we chose to use TMP sampled after the first refiner. This has a high freeness number, which facili-

	MANNOSE	GLUCOSE	GALACTOSE	XYLOSE	ARABINOSE	GALACTURONIC ACID	OTHER*	SUM
TMP fibers, kg/ton TMP	108	44	26	55	14	16	15	278
Dissolved carbohydrates, kg/ton TMP								
TMP water	5.1	2.4	1.7	0.2	0.4	0.5	0.3	10.6
Carbohydrates-in-water	4.4	2.0	0.8	0.1	0.2	0.3	0.1	7.9
Galactoglucomannans-in-water	3.6	0.9	0.5	<0.1	<0.1	0.1	<0.1	5.1
Dissolved carbohydrates, mole-%								
TMP water	47	23	15	2	5	5	3	
Carbohydrates-in-water	56	26	10	1	2	4	<1	
Galactoglucomannans-in-water	70	18	9	1	<1	2	<1	

*Glucuronic acid, 4-O-methyglucuronic acid, rhamnose

I. Amount of monosaccharide units in TMP fibers and amounts and mole-% of these units in different waters obtained by the study method.

tated the subsequent filtrations. Efficient pulp washing (deresination), at a lower cost for the washing equipment, can also be achieved by washing the pulp after the first refiner [23-25]. TMP water was prepared by extraction of TMP in a large pulper. In a pre-study, we found that more carbohydrates were dissolved from TMP sampled after the second refiner than after the first refiner [26]. We observed no significant difference in the amount of other dissolved and colloidal substances.

The pH was adjusted to about 5 to ensure a high yield of dissolved acetylgalactoglucomannans and a low yield of other substances. The yield of dissolved carbohydrates from TMP is lower at pH 8 than at pH 5, mainly due to deacetylation and partial resorption of dissolved galactoglucomannans to the fibers [18, 26]. The yield of dissolved wood resin, lignans, and lignin is higher at pH 8 than at pH 5.

The treatment time was 1.5 h at 55±5°C. At this temperature and treatment time, the dissolution of carbohydrates, and especially acetylgalactoglucomannans, is sufficient and the dissolution of other substances minimized [10-11, 18, 26].

The total yield of dissolved carbohydrates in the TMP water was about 10 kg/ton o.d. pulp (**Table I**). This amount was about 4% of the total amount of non-cellulosic sugar units in the TMP. The dissolved carbohydrates were present as poly- and oligosaccharides and the amount of dissolved monosaccharides—mainly fructose, glucose, and arabinose (altogether <1.5 kg/ton TMP)—was negligible. The total yield of dissolved acetylgalactoglucomannans in the TMP-water was about 7 kg/ton TMP. This is assuming a ratio of mannose:glucose:galactose of 4:1:0.5 and that all mannose is part of the acetylgalactoglucomannans. Other dissolved polysaccharides were mainly acidic arabinogalactans, xylans, and pectic acids, which agrees well with earlier reports [10, 11].

Some wood resin, lignin material, and lignans were also dissolved or dispersed into the TMP-water (**Table II**). We considered these substances as contaminants to be removed in the subsequent treatments.

Removal of colloidal material

The colloidal material, consisting mainly of wood resin (pitch) and some microfines, was removed by adding a starch-based cationic coagulant to the water, followed by filtration through the TMP mat formed on the filter in the dewatering stage. Other coagulants, such as polyaluminum chlorides, may also be used [27]. This treatment was successful in removing almost all of the wood resin (Table II), leaving only trace amounts in the water. These trace amounts were removed in the subsequent ultrafiltration step. The treatment also removed about 20% and 45% of the amount of dissolved lignin and lignans, respectively.

A combination of filtration through a glass fiber filter and microfiltration through a 0.1 mm filter is efficient in removing colloidal substances [11, 26, 28]. Membrane filtration is used for removing colloidal material in inter-stage washing and could be applied in this process [18]. These two filtration techniques were not applicable in the now-tested large laboratory scale, but they are worth considering when up-scaling the process further. However, clogging of the filters, followed by a lower final yield of acetylgalactoglucomannans, may decrease the usability of these methods. Microflotation has also been tested for removal of colloidal material. However, the efficiency of removing pitch from unbleached TMP-water has been low, probably due to the strong steric stabilization of the colloidal pitch droplets [26, 29]. Microflotation also seems to remove a substantial amount of the now desired acetylgalactoglucomannans.

	WOOD RESIN kg/ton TMP FREE FATTY ACIDS	RESIN ACIDS	SITOSTEROL	STERYL ESTERS	TRIGLYCERIDES	LIGNIN kg/ton TMP	LIGNANS kg/ton TMP
TMP water	0.1	0.3	0.09	0.5	1.2	3.2	1.3
Dissolved substances-in-water	<0.01	0.03	0.03	0.02	<0.01	2.5	0.7
Carbohydrates-in-water	<0.01	0.03	0.01	0.01	<0.01	0.7	0.2
Galactoglucomannans-in-water	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

II. Amounts of wood resin component groups, lignin, and lignans in different waters obtained by the study method.

Removal of dissolved lignin and lignans

Dissolved lignin and lignans were removed by adsorption to XAD-7 resin. The XAD-7 resin treatment removed about 70% of both the remaining dissolved lignin and lignans from the water (Table II). The remaining lignin and lignans were removed from the water in the subsequent ultrafiltration. Our pre-study showed that the sorption efficiency of XAD-7 (polyacrylate) resin was superior to that of XAD-4 (polystyrene-divinyl benzene) resin for lignans [30]. Both XAD-7 and XAD-4 were now tested and the selectivity for lignin and lignans was about equal for both types of resin, when using the present experimental set-up. However, the sorption efficiency for lignans was slightly higher than for lignin when the XAD-7 resin was packed in a column and the TMP-water was allowed to flow through the column [26]. Adsorption to resin should also be efficient for other aromatics, such as stilbenes and flavonoids.

Membrane filtration could also be applied to remove lignin and lignans, but high-molar-mass polysaccharides could cause an undesired clogging of the membranes.

Further purification by ultrafiltration

We further purified the water, containing mainly dissolved neutral carbohydrates (Tables I and II), using a membrane filtration apparatus equipped with a membrane with a cut-off value of 20 kDa. The filter residue (1.7 L), containing mainly acetylgalactoglucomannans with a yield of about 5 kg/ton TMP, was recovered. A filter with a cut-off value of 30 kDa was tested in a pre-study, but with an unreasonably large decrease in the final yield. We also noticed that the temperature in the filtration apparatus increased during the ultrafiltration. This may affect the filter and the cut-off value can be slightly higher in the end than in the beginning of the filtration.

The relatively low rate of flow-through (4 L/h, about 34 h total for 135 L) in the experiment can be up-scaled by using larger filtration apparatus and several in series with sequentially smaller cut-offs. Such equipment is already commercially available (Valmet Flootek).

Mills could use the ultrafiltered water (containing mainly dissolved acetylgalactoglucomannans) in the wet end to stabilize pitch or to enhance the paper strength. It is not recommendable to use the water obtained before the ultrafiltration step, because it then contains salts and low-molar-mass oligosaccharides and monosaccharides. Paper mill bacteria thrive on low-molar-mass carbohydrates and may adversely affect paper machine operation and paper quality.

The molar galactoglucomannan purity of the dissolved polysaccharides was >95% (Table I). The main impurity was polysaccharides containing mainly galacturonic acid. An ion exchange resin might work to remove the impurity. However, it is possible that small amounts of impurities, i.e., sugars other than mannose, glucose, and galactose, are incorporated in the structure of the acetylgalactoglucomannans and therefore would be unnecessary and even impossible to remove.

Recovery of dried acetylgalactoglucomannans for later use

We recovered the dissolved acetylgalactoglucomannans in dry form for later use by precipitation in ethanol, centrifugation, and drying. The final yield of acetylgalactoglucomannans was 5 kg/ton TMP (**Table III**), which was about 70% of the total dissolved amount and 3% of the theoretical amount in the pulp, assuming that all mannose was present in acetylgalactoglucomannans with a ratio of mannose:glucose:galactose of 4:1:0.5. We found that the precipitation in ethanol did not improve the purity of the product or change the molar mass. The molar mass (M_w) of the final product was 29 kDa, which corresponds to an average degree of polymerization (DP) of about 180. The degree of acetylation of the final product was 0.29 and the purity was 95 mole-%, with some galacturonic acid as the main impurity. The degree of acetylation is consistent with other reports for isolated spruce acetylgalactoglucomannans [7,11,12].

Other drying techniques, such as spray drying, might also be considered. However, the applied drying technique should not alter the pH or raise the temperature too much, because the acetylgalactoglucomannans are susceptible to deacetylation and degradation under such conditions [15]. Significant deacetylation of acetylgalactoglucomannans occurs only at pH levels higher than 9. These same phenomena could also occur upon long-time wet storage.

Feasibility of recovery in an industrial scale

Integrated mechanical pulp and paper mills would definitely benefit from applying the presented method for recovery of acetylgalactoglucomannans. This is especially true if the process is built as an interstage-washing step after the first refiner and the dewatered pulp is directed back to the process. Colloidal material should be removed by flocculation and flotation or microfiltration. The wood resin is then recovered and can be used as a supplement to the present tall oil production, e.g. via addition to kraft cooking. Additionally, dissolved aromatic substances can be recovered, separated, and purified. Lignans, stilbenes, and flavonoids are potentially valuable bioactive components for pharmaceutical or functional food applications since such compounds can have antioxidative and anticarcinogenic properties. The lignin

would then be a pure, sulfur-free, and reactive product and a valuable complement to presently produced lignin products. Pilot-scale experiments for the presented method are already under way.

Mills could direct the ultrafiltered galactoglucomannan-concentrate to the wet end of the paper machine to improve the runnability and to increase the final paper strength. They could also recover the acetylgalactoglucomannans in a pure, dry state for later use, possibly even in future food and pharmaceutical applications.

CONCLUSIONS

This paper presents a method for recovering acetylgalactoglucomannans from mechanical pulp with a yield of 5 kg/ton pulp and with 95 mole-% purity. We obtained the acetylgalactoglucomannans in a dry state for later use or as a concentrate for targeted use in the wet end of the paper machine. Possible benefits of using mannans in the wet end are better paper strength and improved runnability on the paper machine. This method also provides a convenient means of interstage washing in a mechanical pulp mill, with the additional benefit of possible recovery of valuable substances such as wood resin and aromatic substances.

TMP water, containing dissolved and colloidal substances, was prepared from mechanical pulp. Dispersed colloidal wood resin was aggregated by a cationic polymer and removed by filtration. Dissolved lignans and lignin were removed by sorption to XAD-7 resin. Salts and low-molar-mass substances were removed by membrane filtration on a 20 kDa filter. The obtained concentrate was enriched in acetylgalactoglucomannans and can be used as such in the wet end of the paper machine. Precipitation of the acetylgalactoglucomannans in ethanol allowed recovery of pure acetylgalactoglucomannans for later use. We also considered alternative purification and drying steps. **TJ**

ACKNOWLEDGEMENTS

The authors wish to thank Andrey Pranovich for advice and valuable help in the method development and Tea Hannuksela for doing the molar mass determinations. We also thank Christer Eckerman and Maria Adams for their help with the work and for some of the pre-studies. Additionally, we acknowledge Raisio Chemicals Oy for access to their ultrafiltration equipment.

This work is part of the activities at the Åbo Akademi Process Chemistry Group within the Finnish Centre of Excellence Programme (2000-2005) by the Academy of Finland.

Total in pulp (theroretical*), kg/ton TMP	149
Dissolved, kg/ton TMP	7
Final yield, kg/ton TMP	5
% dissolved of total in pulp	4.7
% recovered of deissolved	71
% recovered of totoal in pulp	3.4
$\overline{M}_w$, kDa	29
Mannose: Glucose:Galactose ratio	4:1:0.5
Degree of acetylation	0.29
Carbohydrate mole-%	
Mannose	69
Glucose	17
Galactose	9
Galacturonic acid	3
Other**	<2

*Assuming that all mannose was present in acetylgalactoglucomannans with a molar ratio of Man:Glc:Gal of 4:1:0.5; **Xylose, arabinose, glucuronic acid, 4-O-methylglucuronic acid, and rhamnose.

III. Final yield and characteristics of the recovered acetylgalactoglucomannans.

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Received: December 19, 2002
Accepted: March 11, 2003

This paper is also published on TAPPI's web site <www.tappi.org> and summarized in the November *Solutions! for People, Processes and Paper* magazine (Vol. 86 No. 11)

INSIGHTS FROM THE AUTHORS

We chose to research this topic because our laboratory has had a long history of research on water-soluble hemicelluloses and pectins, so this was a natural extension of our know-how and expertise. We felt that we needed to develop and test a method with industrial importance, not only for analytical means.

Practical problems presented the biggest challenge, mostly due to the lack of suitable equipment during the up-scaling. We solved these problems by using equipment available at our facility or through our industrial partners in other projects.

One of the most interesting revelations of this study is the possibility to easily recover almost all interesting extractable substances from pulp or wood.

As the next step, we need to convince pulp and paper mills that they can benefit from applying the presented method. We will also use the now isolated acetylgalactoglucomannans for further scientific and biological experiments, especially in the areas of pitch problems and food supplements.

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