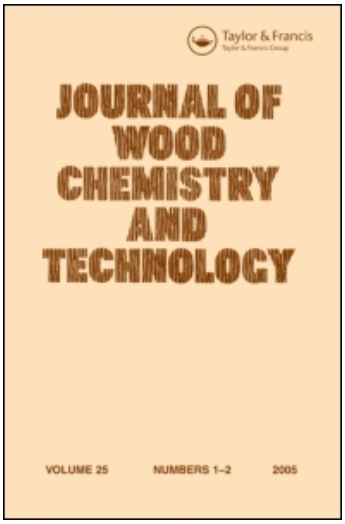

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Wood Resin in Bigtooth and Quaking Aspen Wood and Knots

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Abstract: The lipophilic extractives, that is, wood resin, in *Populus grandidentata* (bigtooth aspen) and *Populus tremuloides* (quaking aspen) sapwood, heartwood, and knots were analyzed. The amount and composition of the extractives in sapwood were similar in both species. Triglycerides, short-chain fatty acids, and steryl esters were predominant in sapwood samples. The knots of both species contained similar amounts of extractives as the stemwood, but the composition was quite different. Monoglycerides; long-chain fatty acids and alcohols, with 22 to 28 carbon atoms; hydroxy fatty acids; and ferulic acid esters of fatty alcohols were the major components in heartwood and knots, but were minor components in the sapwood.

Keywords: Aspen, *Populus grandidentata*, *Populus tremuloides*, sapwood, heartwood, knots, lipophilic extractives

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INTRODUCTION

Populus grandidentata Michx., that is, bigtooth aspen, is native to northeastern and north-central United States and southeastern Canada.[1] *Populus tremuloides* Michx., that is, trembling aspen, is the most widely distributed tree in Canada and the Northern United States. The soft, light-colored aspen wood is mostly used for kraft and chemimechanical pulp.

Rowe and Conner reviewed the extractives of several aspen species, including *P. tremuloides* and *P. grandidentata*.[2] *P. grandidentata* contained smaller amounts of ether-soluble extractives than *P. tremuloides*.[3] A homologous series of fatty acids from C12 to C28, including odd-numbered acids except C27, have been identified in *P. tremuloides*.[2] The major alcohols were C24, C26, and C27 fatty alcohols, and the sterols sitosterol and citrostadienol. The extractives included 3,5-stigmastadien-7-one (tremulone) and α- and β-amyrin, butyrospermol, 24-methylene-cyclortenol, lupeol, and α-amyrenonol. However, the lipophilic extractives in *P. grandidentata* have not been studied in detail.

The problems of pitch deposits in aspen wood pulping and bleaching caused by lipophilic extractives have been of great concern to the pulping industry for a long time.[4] The total amount of extractives in kraft pulps is not as important as is the composition of the extractives because the extractives are known to reduce pulp quality by causing odor and taste problems in paper and pulp products.

Fatty acids, resin acids, steryl esters, and glycerides form water-soluble soaps under alkaline conditions.[5] Neutral components, such as hydrocarbons, diterpenols and diterpene aldehydes, sterols, triterpenols, and certain alkali-stable steryl esters, do not form soluble soaps and have a tendency to deposit and cause pitch problems.[5] It has been estimated that the ratio of acids to unsaponifiables should be about 3:1 to achieve good deresination in kraft pulping.[6] The ratio in fresh *P. tremuloides* sapwood is about 2:1,[7] and can therefore explain the pitch problems occurring during kraft pulping of aspen.[4] The ratio is even lower in the heartwood. Seasoning increases the relative amount of unsaponifiables, but at the same time the pitch becomes more hydrophilic and the insoluble pitch particles become smaller, allowing more efficient deresination.[8]

In pulping of aspen chips, good washing of the unbleached pulp and good white water clarification are necessary to minimize the effect of the high resin levels.[5] Due to the larger amount of acidic components, the steryl ester content in pulps from fresh aspen is lower than in pulps from seasoned aspen chips.

Peng et al.[9] reported that *P. tremuloides* sterols and triterpenols were not affected by kraft pulping, although bleaching modified the structures of most resin components. Even though a significant amount of sterols, triterpenols, and their corresponding esters was removed during pulping and bleaching, a large proportion of sterols and triterpenols were left in the bleached pulp.

A knot, that is, branch base, is the part of a branch that is embedded in the tree stem. An open wound is formed when a branch is broken close to

the stem, making the knot susceptible to attack by fungi and other microorganisms. The knots of hardwoods need extra protection, because the tension wood fibers contain a gelatinous layer that is easily degraded by brown-rot fungi.[10] In recent studies, knots of softwoods (*Picea abies, Pinus sylvestris*, and several *Abies* species) have been found to contain exceptionally large amounts of phenolic substances or lipophilic extractives.[11–14] The large amount of extractives may provide protection against invading microorganisms. The same trend has been shown in smaller scale in a willow species, *Salix caprea*.[15] In a recent study, the amount of lipophilic extractives in knots of two *Acacia* species were found to be similar to (*A. crassicarpa*) or lower than (*A. mangium*) the level in the stemwood.[16]

In this work we analyzed the lipophilic extractives in the *P. tremuloides* and *P. grandidentata* heartwood, sapwood, and knots in detail. The extractives in the knots of these species have not been previously studied and detailed information of lipophilic extractives in *P. grandidentata* stemwood has been missing. To be able to prevent pitch problems in pulping of *P. grandidentata* detailed information of the lipophilic compounds at the component level is needed.

MATERIAL AND METHODS

Material

Two mature *P. tremuloides* and two *P. grandidentata* trees were felled and samples of stemwood and knots were sawn out, frozen, and shipped frozen to our laboratory. The trees were felled in August 2002 in Cape Breton, Nova Scotia, Canada. From each tree one living and one dead knot were sampled (Table 1), as well as the heartwood and sapwood. The living knots had a living branch attached, whereas the dead knots had either a dead

Table 1. Samples of *P. grandidentata* and *P. tremuloides*

	P. grandidentata		*P. tremuloides*	
	Tree 1	Tree 2	Tree 1	Tree 2
Age (years)	32	37	71	58
Number of heartwood annual rings	22	26	33	24
Length of the tree (m)	9.4	14	21	20
Stemwood disc height (m)	1.5	2.1	1.5	2.3
Diameter of the stemwood disc (cm)	18	29	29	43
Living knot disc height (m)	6.4	6.1	11	11
Dead knot disc height (m)	2.6	3.0	4.6	4.8

branch attached or the branch had fallen off. Heartwood samples were distinguished visually by light microscopy. The heartwood samples of both *P. tremuloides* trees contained infected parts, here called blackwood, that were analyzed separately. This defect was most likely due to microbial infection that is a common defect in *P. tremuloides*. The infection has been reported to occur in about 5% of the trees in this species.[17] One sapwood sample of *P. grandidentata* contained red-stained wood that was also analyzed separately.

Methods

The wood samples were splintered, freeze-dried, and ground in an analytical mill (Pulverisette 19, Fritsch). A second freeze-drying step after the grinding ensured practically complete removal of volatile compounds.

Extraction was carried out in an ASE apparatus (Accelerated Solvent Extractor, Dionex Corp.).[11] The lipophilic extractives were extracted with hexane (solvent temperature, 90°C; pressure, 13.8 MPa; two 5 min static cycles).

Fatty acids, fatty alcohols, sterols, triterpenols, and fatty acid monoglycerides were, after evaporation of the extract solutions and silylation of the extractives, analyzed by gas chromatography (GC) on a 25 m by 0.20 mm i.d. column coated with crosslinked methyl polysiloxane (HP-1) with a film thickness of 0.11 μm.[18] Heneicosanoic acid and betulinol were used as internal standards. No FID correction factors were used. The limit of quantitation was about 0.01 mg/g, but compounds at smaller amounts could be detected. These are reported as trace amounts.

Di- and triglycerides, ferulic acid esters, and steryl esters were determined by GC on a 6 m by 0.53 mm i.d. column coated with crosslinked methyl polysiloxane (HP-1) with a film thickness of 0.15 μm.[19] Cholesteryl heptadecanoate (for ferulic acid and steryl esters and diglycerides) and 1,3-dipalmitoyl-2-oleyl glycerol (for triglycerides) were used as internal standards. No FID correction factors were used. All results were calculated on a freeze-dried wood basis.

Total fatty acids, fatty alcohols, and sterols, including esterified ones, were analyzed by GC on a 25 m column as already discussed, after alkaline hydrolysis, that is, saponification, using 0.5 N KOH solution in 90% aqueous EtOH for 5 h at 70°C. After this, distilled water and one drop of bromocresol green were added. The solutions were acidified to about pH 3 with 0.5 M HCl. The acidic and neutral components were extracted three times with methyl *tert*-butyl ether. The ether extracts were combined, evaporated, and silylated. Heneicosanoic acid was used as internal standard for all compounds after alkaline hydrolysis.

Identification of individual components was made by GC-MS analysis of silylated components with an HP 6890-5973 GC-MSD instrument, using the 25 m GC column described earlier. The individual steryl esters were identified

using a 15 m by 0.25 mm i.d. column coated with crosslinked dimethyl diphenyl polysiloxane (MXT-65TG) with a film thickness of 0.10 μm.

The molar-mass distribution of the extractives was determined by high-performance size exclusion chromatography (HPSEC). The system was as follows: Tosoh Biosep TSK G3000, TSK G2500, and TSK G1500 HXL columns with a guard column and a Pharmacia LKB 2142 differential refractometric detector. The solvent (tetrahydrofuran, THF) flow was 1 mL/min and the concentration of each sample was adjusted to a concentration of 1 mg/mL of steryl esters based on GC data. The injection volume was 100 μL.

RESULTS

Extractives in *P. grandidentata*

The total amounts of lipophilic extractives were larger in the sapwood samples than in the other studied tissues (Table 2). The triglycerides comprised the largest component group of lipophilic extractives in sapwood, with concentrations of 4.1 to 10 mg/g (The average, 7.3, is reported in Table 3). Diglycerides, mainly of linoleic acid, were found only in sapwood samples. However, there were only trace amounts of monoglycerides in the sapwood. The amounts of free fatty acids were largest in the sapwood and the short-chain fatty acids (C14—C20) dominated over the long-chain fatty acids (C22—C28). Linoleic acid was the dominating fatty acid, although only trace amounts of long-chain fatty acids occurred. The structures of some of the lipophilic extractives are presented in Figure 1.

After alkaline hydrolysis, the short-chain fatty acids dominated as would be expected from the large amounts of tri- and diglycerides. Hydroxy-fatty acids that were released in hydrolysis were now observed in amounts of 0.12 mg/g. These compounds had probably been esterified with glycerol or ferulic acids. The results for alkaline hydrolysis that refer to the situation after kraft pulping are presented in Figure 2.

Table 2. Gravimetric amounts of lipophilic extractives in *P. grandidentata* and *P. tremuloides*, in mg/g

	Average of two trees					
	Sapwood	Heartwood	Black wood	Living knot	Dead knot	Red part
Populus grandidentata	16	3.7	—	8.9	5.7	100
Populus tremuloides	17	6.4	6.6	20	12	—

Table 3. Lipophilic extractives in *Populus grandidentata* determined by GC

	Sapwood	Heartwood	Living knot	Dead knot	Red
C24 monoglyceride	0.01	0.06	0.06	0.07	0.01
C26 monoglyceride	+[a]	0.08	0.11	0.14	+
Others[b]	0.01	0.12	0.11	0.10	0.03
Sum monoglycerides	0.03	0.27	0.28	0.31	0.05
Sum diglycerides	1.2				
Sum triglycerides	7.3	0.22	1.4	0.20	11
C16 acid	0.16	0.08	0.08	0.04	0.16
C18:3 acid	+	+	0.01	+	+
C18:2 acid	1.9	0.90	0.51	0.14	1.9
C18:1 acid	0.24	0.18	0.11	0.02	0.33
C18 acid	0.03	0.01	0.01	0.01	0.08
C20 acid	0.01	0.01	0.01		
Others[c]	0.16	0.02	0.04	0.02	0.35
Sum short-chain FAs	2.5	1.2	0.77	0.24	2.9
C22 acid	0.02	0.02	0.02	0.02	0.02
C24 acid	0.03	0.08	0.06	0.07	0.05
C26 acid	+	0.04	0.04	0.05	0.01
Others[d]	0.04	0.06	0.04	0.04	0.13
Sum long-chain FAs	0.09	0.20	0.16	0.18	0.21
Sum ferulic acid esters[e]		0.18	0.89	0.52	0.71
C26 alcohol	+	0.05	0.14	0.12	0.08
C28 alcohol	+	0.04	0.17	0.11	0.01
Others[f]	+	0.02	0.03	0.03	0.03
Sum fatty alcohols	0.01	0.11	0.35	0.26	0.13
24-OH-24:0 acid	+	0.01			0.01
Others[g]	+	+	+		0.03
Sum hydroxy-fatty acids	+	0.01	+		0.03
Sum steryl esters[h]	2.0	1.4	3.7	2.0	2.1
Sitosterol	0.21	0.20	0.23	0.22	0.28
Butyrospermol	0.01	0.04	0.03	0.04	0.01
Methylene cycloartanol	0.01	0.02	0.01	0.01	0.09
Citrostadienol	0.01	0.03	0.05	0.03	0.01
Sum sterols	0.24	0.28	0.33	0.29	0.39
α-amyrin	0.08	0.08	0.06	0.05	0.04
Sum triterpenols	0.08	0.08	0.06	0.05	0.04
Total extractives by GC	11	2.3	3.9	1.8	15

All results are expressed as mg/g dry wood. Averages of two trees.

[a]Trace amounts; [b]Including monoglycerides C28, 26-OH-26:0, and 28-OH-28:0; [c]Including fatty acids C12, C14:2, C14, C15, C16:2, C16:1, C17:1, C17, C19:1, C19, C20:3, C20:2, and C20:1; [d]Including fatty acids C23, C25, and C27, C28; [e]Including ferulic acid esters of fatty alcohols C24, C26, and C28; [f]Including fatty alcohols C16 and C24; [g]Including C9 dioic acid, C18-OH-18:1, C22-OH-22:0, and C26-OH-26:0 acids; [h]Including triterpenyl esters.

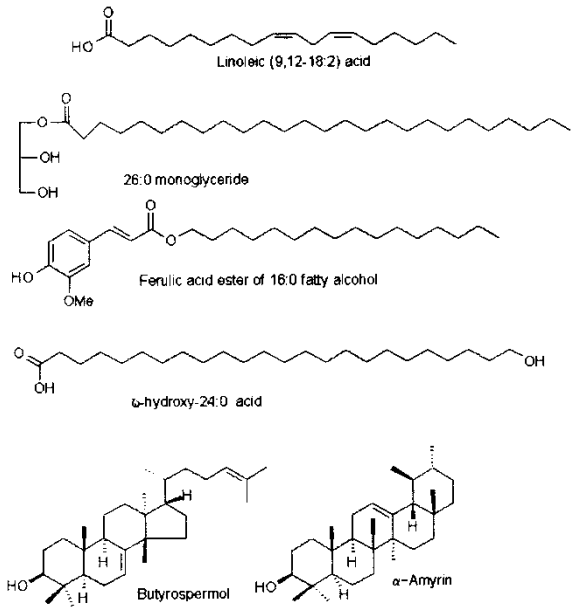

Figure 1. Structures of some lipophilic compounds identified in *P. grandidentata* and *P. tremuloides*.

The red-stained sapwood contained exceptionally large amounts of extractives, 100 mg/g, that is, 10% by weight. The stain had a similar composition of the extractives as in normal sapwood, but it also contained large amounts of branched alkanes with 28–32 C-atoms. The reddish extract remained oily after vacuum drying.

Heartwood samples contained larger amounts of monoglycerides than triglycerides, the predominant one being monoglyceride of fatty acid C26. Compared to the sapwood samples, the amounts of long-chain fatty acids were larger in heartwood, although the short-chain fatty acids dominated over the long-chained fatty acids.

During hydrolysis of the heartwood extracts, the fatty acid concentration increased markedly (Table 4). Linoleic acid (C18:2) dominated in the extracts also after hydrolysis and its amount increased two-fold. The heartwood samples contained ferulic acid esters of fatty alcohols C24, C26, and C28. Such compounds have previously been identified in *Eucalyptus globulus* stemwood,[20] and *Acacia mangium* and *A. crassicarpa* stemwood and knots.[16] The amounts of fatty alcohols increased two-fold and the fatty alcohol C26 dominated in this group after hydrolysis.

The knots contained more extractives than the heartwood (Table 2). The monoglyceride amounts were larger than the amounts of triglycerides in the knots, except for the living knot of tree 1. The fatty acid C26 was

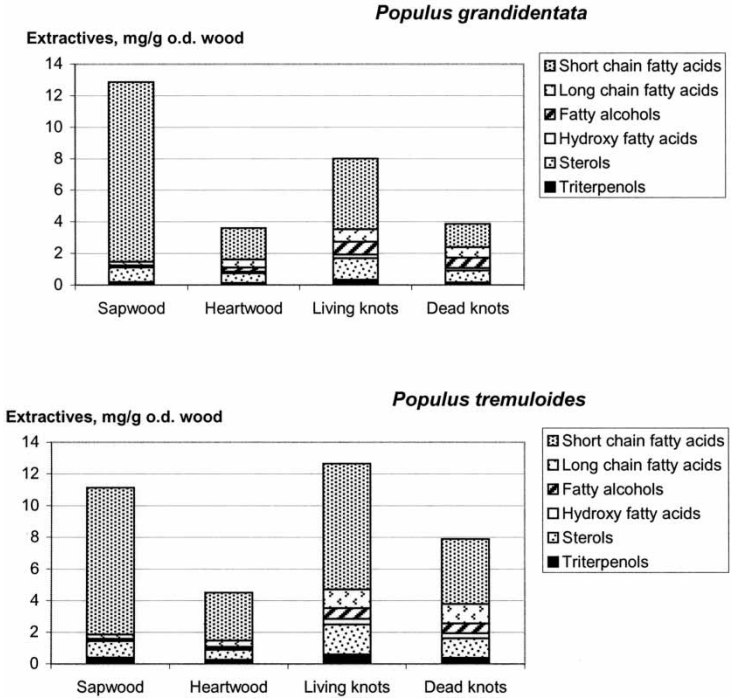

Figure 2. Lipophilic extractives in *P. grandidentata* and *P. tremuloides* determined by GC after alkaline hydrolysis. Mean values of two trees. The complete tables can be obtained from the corresponding author.

the dominating acid in the monoglycerides. The amounts of the long-chain fatty acids were similar in the knots as in the heartwood, but the knots contained more ferulic acid esters of fatty alcohols than the heartwood samples. The knots also contained larger amounts of free fatty alcohols than the stemwood samples, the alcohols C28 and C26 being most dominant.

Additional amounts of the long-chain fatty acids in the knots were released during hydrolysis and acid C26 was the main long-chain fatty acid in the hydrolyzed extracts. The knot extracts contained larger total amounts of long-chain fatty alcohols than the stemwood extracts.

The amounts of steryl and triterpenyl esters were on the same level in all samples. Fatty acid esters of butyrospermol, α- and β-amyrin, sitosterol, and methylene cycloartanol were identified in the extracts. These were quantified as a group. The amounts of free sterols were also similar in all samples. Sitosterol was the dominating compound, whereas the amounts of butyrospermol were much smaller. There were minor amounts of free triterpenols in all samples. Sterols were liberated in hydrolysis; sitosterol was predominant, followed by butyrospermol and methylene cycloartanol. Furthermore, the

Table 4. Lipophilic extractives in *Populus tremuloides* determined by GC

	Sapwood	Heartwood	Living knots	Dead knots
C18:2 monoglyceride	0.01	0.01	0.05	0.12
C24 monoglyceride	0.01	0.05	0.12	0.16
C26 monoglyceride	0.01	0.06	0.22	0.19
Others[a]	0.04	0.05	0.11	0.10
Sum monoglycerides	0.07	0.18	0.50	0.57
Sum diglycerides	0.69			
Sum triglycerides	3.1	0.17	0.96	0.48
C16 acid	0.13	0.12	0.13	0.13
C18:3 acid	+[b]	0.03	+	0.01
C18:2 acid	1.6	1.4	2.8	1.5
C18:1 acid	0.01	0.01	0.01	0.01
C18 acid	0.02	0.01	0.02	0.03
C20 acid	0.03	0.01	0.02	0.02
Others[c]	0.11	0.05	0.08	0.10
Sum short-chain FAs	1.9	1.6	3.0	1.8
C22 acid	0.03	0.04	0.04	0.06
C24 acid	0.03	0.06	0.06	0.15
C26 acid	0.01	0.03	0.07	0.13
C28 acid	+	+	0.02	0.04
Others[d]	0.04	0.04	0.03	0.03
Sum long-chain FAs	0.11	0.17	0.22	0.42
Sum ferulic acid esters[e]		0.07	0.38	0.31
C26 alcohol	+	0.03	0.14	0.15
C28 alcohol	+	0.03	0.17	0.18
Others[f]	0.03	0.01	0.02	0.03
Sum fatty alcohols	0.03	0.07	0.34	0.36
24-OH-24:0 acid	+	+	+	0.01
Others[g]	0.02	0.01	0.01	+
Sum hydroxy-fatty acids	0.02	0.01	0.01	0.01
Sum steryl esters[h]	2.5	1.7	3.4	3.3
Sitosterol	0.12	0.12	0.13	0.16
Butyrospermol	0.01	0.03	0.05	0.08
Methylene cycloartanol	0.05	0.03	0.02	0.02
Citrostadienol	+	0.01	0.02	0.02
Sum sterols	0.18	0.19	0.23	0.28
α-amyrin	0.06	0.06	0.05	0.06
Sum triterpenols	0.06	0.06	0.05	0.06
Total extractives by GC	9.4	4.2	9.2	7.5

All results are expressed as mg/g dry wood. Averages of two trees.

[a]Including monoglycerides C28, C26-OH-26, and C28-OH-28; [b]Trace amounts; [c]Including fatty acids C12, C14:2 C14, C15, C16:2, C16:1, C17:1, C17, C19:1, C19, C20:3, C20:2, and C20:1; [d]Including fatty acids C23, C25, C27, and C28; [e]Including ferulic acid esters of fatty alcohols C24, C26, and C28; [f]Including fatty alcohols C16 and C24; [g]Including C9 dioic acid, C18-OH-18:1, C22-OH-22, and C26-OH-26 acids; [h]Including triterpenyl esters.

amounts of triterpenols increased slightly during hydrolysis. No oligomeric or polymeric material of higher molar mass than triglycerides was observed upon HPSEC analysis of the extracts.

Extractives in *P. tremuloides*

The sapwood samples of *P. tremuloides* contained much more extractives than the heartwood samples (Table 2). Triglycerides and steryl esters were the predominant component groups in the sapwood samples (Table 4). Diglycerides, which were two linoleic acid (C18:2) esters, were found only in the sapwood samples. The short-chain fatty acids dominated over the long-chain fatty acids, and linoleic acid constituted the major part of the total free fatty acid fraction. There were only trace amounts of fatty alcohols in the sapwood samples.

Alkaline hydrolysis increased the amounts of all fatty acids, as was to be expected because of the large amounts of glycerides. Linoleic acid dominated in the hydrolyzed extracts. The amounts of fatty alcohols remained constant in the hydrolyzed sapwood extracts.

The heartwood of *P. tremuloides* contained much less lipophilic extractives than sapwood. This difference was due to lower amounts of triglycerides and diglycerides in heartwood. However, the amounts of monoglycerides were larger in the heartwood than in the sapwood, and monoglycerides of long-chain fatty acids (22–28 C-atoms) dominated over the short-chain fatty acids (12–20 C-atoms). Small amounts of the ferulic acid esters of fatty alcohols C24, C26, and C28 were detected in the heartwood extracts. The amounts and composition of the free fatty acids, fatty alcohols, sterol, triterpenols, and esters of sterols and triterpenols in the heartwood samples were similar to the amounts and composition in the sapwood samples.

Long-chain fatty acids were released in alkaline hydrolysis of the heartwood extracts; however, the short-chain fatty acids remained predominant. Hydrolysis released fatty alcohols and hydroxy-fatty acids in heartwood.

The total amounts of extractives in the blackwood were in range with the amounts in the heartwood.

The total amounts of lipophilic extractives were largest in the living knots of *P. tremuloides* (Table 2). The amounts of triglycerides were clearly smaller in the knots than in the sapwood, but the knots contained more monoglycerides than the stemwood samples; these monoglycerides consisted merely of long-chain fatty acids. The knots also contained larger amounts of long-chain fatty acids than the stemwood samples; this fraction consisted mostly of fatty acids C24 and C26. Small amounts of the ferulic acid esters of fatty alcohols C24, C26, and C28 were found in the knot extracts. Compared to stemwood extracts, the knots contained larger amounts of fatty alcohols, with C26 and C28 being the dominating compounds.

The knot extracts contained larger amounts of long-chain fatty acids, fatty alcohols, sterols, and triterpenols than the stemwood samples after hydrolysis. Especially large amounts of hydroxy-fatty acids were found in the knot extracts after hydrolysis.

Steryl and triterpenyl esters were found in similar amounts in all extracts (Table 5). Fatty acid esters of butyrospermol, α- and β-amyrin, sitosterol, and methylene cycloartenol were found in the extracts, although these were quantified as a group. Sitosterol dominated among the free sterols of the extracts. After hydrolysis, the amounts of butyrospermol and sitosterol were clearly larger than prior to the hydrolysis and butyrospermol became the predominant sterol.

The triterpenol α-amyrin dominated both before and after hydrolysis, whereas lupeol was found only after hydrolysis. β-Amyrin was not observed in the extracts probably because it overlaps with sitosterol in the gas chromatogram. In a recent study of *P. tremula* it was observed that the amount of β-amyrin is about 80% of the amount of α-amyrin. α-Tocopherol (vitamin E) that can prevent autoxidation of unsaturated lipids in plants was also identified in trace amounts in the extracts.[20] No oligomeric or polymeric material with higher molar mass than the triglycerides was observed upon HPSEC analysis of the extracts.

CONCLUSIONS

The comprehensive and detailed study of the lipophilic extractives, that is, resin components, in *P. tremuloides* and *P. grandidentata* has shown that:

- The lipophilic extractives in sapwood of *P. tremuloides* and *P. grandidentata* are quite similar and the pulping properties regarding pitch problems are alike.
- The heartwood of both species contains much less resin than the sapwood, mainly due to smaller amounts of short-chain fatty acids, but instead the heartwood contains more long-chain fatty acids than sapwood.
- Knots in *P. tremuloides* contain more extractives than knots of *P. grandidentata*. The resin composition in knots of both species is detrimental regarding pulping. Therefore, effective removal of knots is needed in order to facilitate pulping. This is the first study of resin in the knots of any *Populus* species.
- The lipophilic extractives in infected heartwood of *P. tremuloides* are similar to those in the sound heartwood.
- Monoglycerides of long-chain fatty acids were identified in both trees and ferulic acid esters of fatty alcohols were identified in heartwood and knot samples. Hydroxy fatty acids were found in all sample types to some extent in free form but mostly as in esterified form. These components have not previously been identified in any *Populus* species.

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