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ORIGINAL ARTICLE

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Effects of log storage and drying on birch (*Betula pendula*) wood proanthocyanidin concentration and discoloration

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Abstract In conventional drying, sawn birch (*Betula* sp.) timber darkens and reddens from the inside while the layer a few millimetres under the yellowish surface remains light in color. Lack of information concerning the chemical basis of the discoloration hinders the development of a reliable solution for this problem. In this study, the role of soluble proanthocyanidins in discoloration of birch wood was investigated because the polymerization and oxidation of these compounds are known to yield insoluble reddish compounds. Different periods of log storage affected the synthesis of soluble proanthocyanidins during conventional drying. Concentration of proanthocyanidins also correlated with changes in the color of birch wood. Discoloration appeared differently in conventionally dried and vacuum-dried wood, which indicates that the discoloration mechanism in these drying methods may differ chemically, and/or the compounds that take part in discoloration may be different at different drying temperatures.

Key words *Betula pendula* · Condensed tannins · Discoloration · Drying methods · Proanthocyanidins

Introduction

Depending on species, drying method and schedule, and heterogenity of wood material, wood discoloration that appears during drying varies from a dark layer just under the surface of a piece of sawn timber[1,2] to comphrehensive darkening throughout the wood.[3–6] The chemistry of this discoloration is, however, poorly documented. The most comphrehensive studies on this topic have been carried out for oak (*Quercus robur*, *Q. petraea*),[7–9] western hemlock (*Tsuga heterophylla*),[10–13] and radiata pine (*Pinus radiata*).[1,2]

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Although the chemical basis of wood discoloration that appears during drying is poorly known, attempts have been made to prevent discoloration by chemically treating undried wood to restrict enzymatic action in parenchymatic tissue.[14–18] To prevent discoloration, drying methods (e.g., vacuum, high frequency, and microwave drying) and drying schedules have also been developed, and pressure cycles have been applied during wood drying.[19]

The discoloring reactions that occur during drying may involve the woody tissue itself, i.e., the walls of lignified cells, or compounds located in cell vacuoles.[20,21] In cell walls, lignin may make complexes with phenolic catechin[22] and cellulose.[23] The reactions of compounds in cell vacuoles are believed to be mainly oxidation and polymerization reactions of phenolic compounds.[7,8,21,24–26] Maillard type discoloring reactions may also occur on the surface of sawn timber as the result of migration of extractives, mainly amino acids and reducing sugars.[2]

The most difficult problem in conventional drying of birch timber is discoloration. It appears in the inner part of sawn birch timber, while the outer layer, to a depth of 1–5 mm under the yellowed surface, usually remains light in color.[6,27] In this study the role of proanthocyanidins (PAs or condensed tannins) and their relation to color change were analyzed in connection with both conventional and vacuum drying.

Materials and methods

The *Betula pendula* Roth timber used in this experiment was sawn into 30 × 70 × 1200 mm boards. Boards with dimensions of 30 × 70 mm are used in the parquet industry as raw material for parquet surface lamellae. Birches with decayed pith wood were excluded, but trees with darkened wood around the pith could not be avoided. Darkened wood was not, however, used for the experiments. Because the reasons for discoloration were investigated here, the drying schedules for both conventional and vacuum processes in small laboratory kilns were used so that discolora-

Table 1. Conventional drying and vacuum-drying schedules used in the experiments

Stage of process	Conventional drying			Vacuum-drying		
	T (°C)	DF	EMC (%)	T (°C)	P	EMC (%)
Pressure lowering	–[a]	–	–	50	100	9
Heating	37	–	15	64	210	8
Drying[b]						
>70%	37	2.0	–	65	210	12
60%–70%	38	2.4	–	68	210	10.5
50%–60%	39	2.4	–	70	220	10
40%–50%	40	2.7	–	72	260	9.5
30%–40%	41	3.2	–	73	270	9.5
25%–30%	42	3.2	–	74	280	9
20%–25%	42	3.2	–	75	260	7
15%–20%	60	3.5	–	76	220	6
10%–15%	65	3.5	–	77	240	6
<10%	65	3.5	–	82	160	5.5
Conditioning	70	–	3	73	160	5
Cooling	–	–	5	40	1000	7

T, Temperature; DF, drying force; EMC, equilibrium moisture content; P, air pressure
[a] Hyphens indicate not included in schedule
[b] Based on wood moisture content

tion would occur (Table 1). In addition to kiln drying processes, low-temperature drying was performed for birch boards at normal room temperature (~20°C) and air humidity (40%–50%).

Every drying lot included two logs of 10 birches. Five lots of unstored wood, four lots of wood stored for 5 weeks, and three lots of wood stored for 10 weeks were used in conventional and vacuum drying. One drying lot each of unstored wood and wood stored for 5 weeks and two lots of wood stored for 10 weeks were used in room drying. Unbarked logs were stored in pile at the timber yard of a saw mill, to where they were transported not later than 2 days after felling. The storage period is the period between felling and sawing. One of the vacuum drying processes of unstored wood failed: the temperature remained under 40°C with several peaks of 50°C, and the pressure changed from 150 to 350 mbar to about 17% moisture content (MC) at intervals of approximately 4 h. The results of this lot are excluded from the general results of vacuum drying. The chemistry of birch wood discoloration was studied by determining the concentrations of soluble proanthocyanidins (sPAs) from: (1) fresh wood (n = 89), (2) the wood of logs stored for 5 (n = 81) and 10 weeks (n = 62), (3) wood samples taken from boards during conventional drying at 4 wood MC (35%, 30%, 25%, 20% according to the wood moisture sensors of the kiln) (n = 118), (4) conventionally dried boards (n = 228), (5) vacuum-dried boards (n = 199), and (6) boards dried in normal room conditions (n = 75) at the final MC (5%–7% for conventionally and vacuum-dried wood, 7%–9% for room-dried wood). The wood samples taken during drying were grouped into 4 classes: 40%–60% (n = 11), 30%–40% (n = 41), 20%–30% (n = 40), and 10%–20% (n = 26) according to the inner MC of the board (measured gravimetrically).

The undried wood samples and those taken during conventional drying were stored frozen until the sPAs were analyzed. According to preliminary tests, freezing did not affect the concentration of sPAs. To analyze the sPAs, the wood samples were ground into powder and extracted with acetone (95%) for 20 h at room temperature in a shaker. The extracts were then filtered, and the same samples were extracted again in the same way. According to test extractions, a third extraction of the same wood powder yielded only traces of sPAs and thus was not carried out in subsequent tests. Acetone was used for extraction because it solubilizes a large amount of the sPAs that are insoluble in methanol.[28] The concentration of sPAs was determined by converting the sPAs to colored anthocyanidins in acid solution.[29,30] A standard curve was made using commercial cyanidin chloride. The concentration (μg/g dry weight basis) of the combined extracts of the two extractions was the total concentration of sPAs of a wood sample as cyanidin equivalents. Two replicates of each board were analyzed, and their average was the final total concentration of sPAs of the sample. The concentrations of sPAs were compared by the SPSS general linear model procedure.

The color results of these boards are given by Luostarinen et al.[27] in which the same material was used. Here the color coordinates of the inner wood of the boards were used to calculate the correlation between them and the concentration of sPAs (SPSS correlation procedure).

Results

Storage and drying

The duration of the log storage period affected the trend of concentration of sPAs during conventional drying (Fig. 1). When wood was not stored or was stored for 5 weeks, the concentration of sPAs was highest when the MC was 30%–40%, after which it decreased significantly by the MC class of 20%–30%, and after that less to the final MC. During

drying, the concentration of sPAs in wood stored for 10 weeks remained significantly lower, increasing to only 63.0μg/g by the MC class of 10%–20%.

Significant differences were observed in the average concentrations of sPAs between storages at the final MC only for vacuum drying. The concentration of sPAs was highest in wood stored for 10 weeks (Fig. 1). The concentrations of sPAs between drying methods, however, differed. The concentration of sPAs was significantly higher in room-dried samples than in conventionally dried samples, and the concentration for vacuum-dried wood was significantly lower than that of conventionally dried wood. The concentration of sPAs in the unstored vacuum-dried wood of the failed lot was significantly higher, on average 67.8μg/g, with the standard error of the mean (SE) $\pm$4.60μg/g, than that of the other lots after vacuum drying. The concentration also differed significantly from that of both unstored conventionally dried wood and room-dried wood. The color of the wood from failed vacuum drying was significantly lighter (L^*), less red (a^*) and less yellow (b^*) than the color of the corresponding conventionally dried lot (vacuum: $L^* = 81.99 \pm 0.38$(SE), $a^* = 4.30 \pm 0.17$, $b^* = 18.17 \pm 0.23$; conventional: $L^* = 79.15 \pm 0.22$, $a^* = 4.87 \pm 0.11$, $b^* = 18.62 \pm 0.11$).

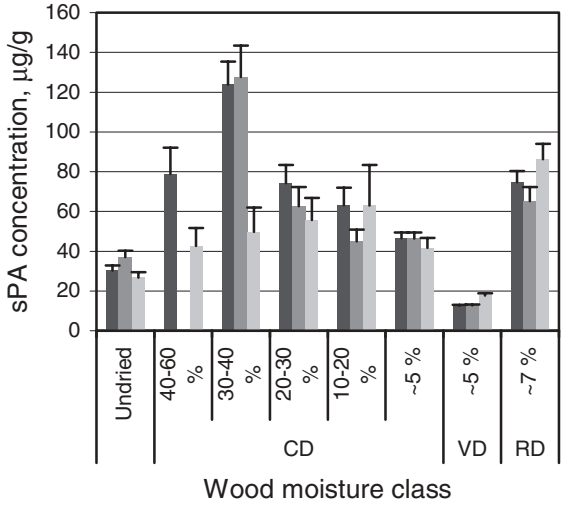

Fig. 1. Concentration of soluble proanthocyanidins (sPAs) by moisture content (MC) class and the duration of the log storage. Columns: *dark grey*, not stored; *medium grey*, stored for 5 weeks; *light grey*, stored for 10 weeks. *CD*, conventional drying; *VD*, vacuum drying; *RD*, room drying. No observations for wood stored for 5 weeks at the MC class 40%–60% were available because of the sampling method

Correlations between color and concentration of sPAs

The dynamics of sPAs could be separated into two periods in conventional drying so that the turning point was at 30% MC, which is about the saturation point for wood fiber (FSP). Thus, the correlation between $L^*a^*b^*$ color coordinates and concentration of sPAs was calculated separately for the two periods. The $L^*a^*b^*$ color coordinates correlated with the total concentration of sPAs of wood in the MC interval of "30% – final MC" so that the higher the concentration of sPAs, the lighter (L^*) was the color of the wood; and the redder (a^*) and yellower (b^*) the color, the smaller was the concentration of sPAs. Correlations for L^* and a^* coordinates were significant for unstored wood and wood stored for 5 weeks, and for b^* for wood stored for 5 weeks. However, for wood stored for 10 weeks only the L^* coordinate showed significant correlation ($P < 0.05$) with concentration of sPAs (Table 2). The relation between concentration of sPAs and colors in both vacuum-dried wood and room-dried wood were in accordance with this correlation so that when compared with conventionally dried wood, vacuum-dried wood was darker and contained less sPAs, and room-dried wood was lighter and contained more sPAs (Table 3).

Table 2. Correlations between $L^*a^*b^*$ color coordinates of inner wood of boards and concentration of soluble proanthocyanidins (sPAs)

	L^*		a^*		b^*	
Moisture class	*r*	*n*	*r*	*n*	*r*	*n*
Logs not stored						
Fresh – 30%	−0.070	28	0.282	28	−0.165	28
30% – final MC	0.441***	139	−0.286***	139	−0.145	139
Fresh – final MC	0.307***	167	−0.178**	167	−0.068	167
Logs stored for 5 weeks						
Undried – 30%	0.170	43	0.203	43	−0.149	43
30% – final MC	0.646***	105	−0.513***	105	−0.374***	105
Undried – final MC	0.244***	148	−0.106	148	−0.138	148
Logs stored for 10 weeks						
Undried – 30%	0.200	33	−0.029	33	−0.310	33
30% – final MC	0.240**	84	−0.110	84	−0.177	84
Undried – final MC	0.010	117	0.079	117	−0.029	117

Only undried and conventionally dried logs are included
r, Correlation coefficient; *n*, number of samples; MC, moisture content
** $P < 0.05$
*** $P < 0.01$

Table 3. Concentration of sPAs and $L^*a^*b^*$ color coordinates of conventionally dried, vacuum-dried, and room-dried birch wood

	sPA concentration (μg/g)	L^*	a^*	b^*
Conventionally dried	45.2 (2.02)	79.4 (0.07)	4.7 (0.03)	19.2 (0.04)
Vacuum-dried	14.1 (0.48)	78.9 (0.10)	5.6 (0.05)	19.5 (0.08)
Room-dried	77.6 (4.37)	81.7 (0.26)	4.1 (0.11)	20.5 (0.19)

Standard error of the mean (SE) shown in parentheses

Discussion

The dynamics of concentration of sPAs of birch wood during drying can be separated into two different periods. The first period started when drying commenced. The sPAs were then synthesized, a process which commonly occurs in plants during different kinds of stress.[31–33] Water loss from wood and living parenchyma of the xylem (rays, axial parenchyma) certainly causes severe stress and increases the concentration of enzymes and their substrates. Stress is also accentuated by raised temperature. For unstored wood and wood stored for 5 weeks in these conventional drying processes, the synthesis of sPAs probably went on as an enzymatic reaction before the temperature rose above 40°C,[34,35] which was reached at a MC of about 40%. The second period started at a MC of about 30% (~FSP), when the concentration of sPAs started to decrease. Thus, the periods for concentration of sPAs corresponded to color data,[27] which showed that wood first even slightly lightened to the FSP, and then it started to darken clearly.[6,36]

The dynamics of sPAs in birch wood stored for 10 weeks was different from that of the wood which was stored for 5 weeks or which was not stored at all. Only some sPAs were synthesized during drying in wood stored for 10 weeks, and the concentration was highest in the MC class of 10%–20%, after which it decreased slightly. This is due to the effect of storage on the activity and death of parenchymatic tissue. For example, the activity of peroxidase has been observed to decrease during storage to near its minimum in 1 month.[35] For these same wood samples, it was observed that the longer the storage continued, the wood color remained less red during conventional drying.[27] This means that fewer sPAs were condensed or oxidized to colored tannins. Thus, sufficiently high water content and temperature, rather than living tissues, are necessary for the reactions that cause discoloration to occur.[37] However, these reactions occur to a lesser degree in dead tissues than in living tissues.

The sPAs can be synthesized both biochemically (enzymatically) and chemically if the temperature is not too high (over 40°C) and if living parenchyma exists. The dynamics of sPAs in wood stored for 10 weeks suggests that sPAs may be synthesized at least chemically at an MC lower than the FSP, just as the transport of soluble substances occurred at an MC lower than the FSP.[38] If the difference in the synthesis of sPAs between different storage treatments is due to the presence of living parenchyma, then the biochemical synthesis of sPAs is clearly stronger than the chemical route, and any chemical synthesis in unstored wood and wood stored for 5 weeks under the FSP was hidden by condensation or oxidation of sPAs produced earlier. The prevailing concentration of sPAs may affect the magnitude of the condensation and oxidation reactions.

The fact that the concentration of sPAs in wood stored for 10 weeks increased slightly to the MC class of 10%–20% suggests that one limit for color-changing reactions in wood is a MC between 10% and 20%. The limiting MC value for wood color changes has been measured to be between 15% and 20%, under which birch wood again becomes slightly lighter,[6,27,36] and no obvious color darkening occurs if birch wood is pre-dried in a timber yard to about 15%–20% MC before kiln drying.[39] The color changes of birch wood during conventional drying correspond to those of robinetin (a flavone), which first clearly darkens when it is oxidized and coupled to form dimers, and then becomes lighter again when irradiated[40] or heated.[41] Photooxidation is known to destroy the flavonoid structure which causes lightening of the color.[41]

The concentration of sPAs in birch timber dried under normal room conditions was higher than the concentration of sPAs in undried wood at the final MC, but was lower than the concentration at the MC level of 30%–40%. This suggests that synthesis and condensation and/or oxidation of sPAs occurred, but one or all of them occurred to a lesser degree at room temperature than at elevated temperature. PAs are known to be synthesized under ambient conditions,[42] but they are condensed mainly in hot acidic conditions.[43,44] The slight color darkening of room-dried wood[27] indicates condensation and/or oxidation of sPAs in low quantities.

It has been determined previously[6,45] for conventional drying of birch wood that temperature is an important factor both in discoloration and in reactions of sPAs. This was also observed in vacuum drying after failure of a drying lot. The total concentration of sPAs was higher and the wood color was lighter in the lot which dried at lower temperature than in the other vacuum-dried lots. The vacuum drying used here yielded timber that was clearly darker than usually by this method dried birch wood. Because the drying schedule of the failed vacuum-dried lot was almost the same in terms of temperature as the conventional drying schedule to a MC of about 20%, there seems to be a difference between drying methods in condensation/oxidation of sPAs. The MC limit for the reactions appears to be between 15% and 20%, and this could also be seen in color. In the boards of the failed lot, the color of the surface layer was

darker than the inner part. This was also the case for the other vacuum-dried boards but not for the conventionally dried or room-dried boards. For the same birch boards used in the present experiment, it was observed[46,47] that in vacuum-dried boards the concentration of monosaccharides increased, which is probably partly caused by hydrolysis of hemicellulose. It was also observed[46] that particularly in the vacuum-dried birch boards the sugars migrated toward the surface of the boards so that the gradient was obvious. This kind of migration of sugars has also been observed in some softwoods.[48] Thus, in addition to sPAs, carbohydrates, both soluble and originally cell-wall bound, may also have taken part in color changes in these vacuum-dried boards. It is also possible that discoloration of the surface layer of the vacuum-dried boards is chemically different from that of the inner parts of the boards or that of boards dried conventionally, and sugars may play a different role in these cases.

The correlation between changes in birch wood color, especially reddening, and in concentration of sPAs during drying suggests that sPAs play a role in darkening of birch wood during drying. This finding is supported by the corresponding correlation in room-dried wood, and the lighter color with higher concentration of sPAs in the failed vacuum-dried lot than in other vacuum-dried lots. As the redness was significantly higher in vacuum-dried wood than in conventionally dried wood, and the color of the surface layer of the vacuum-dried boards was darker than their inner wood,[27] it is possible that both the discoloration mechanism and the compounds that take part, may differ in different drying methods, but they may also differ at different temperatures.

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