Discolouration of Sawn Birch (*Betula pendula*) Timber from Plantation Forests during Drying: The Role of Proanthocyanidins (condensed tannins) in Discolouration of Birch Wood

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Abstract

In the previous paper on discolouration of sawn birch timber from plantation forests that had been published in *Baltic Forestry*, 2004 10 (2), the effect of external factors (growing site, felling season, log storage) was discussed. In this paper, the role of proanthocyanidins in discolouration during drying is investigated. Conventional warm-air drying gave the highest concentration of proanthocyanidin in the wood. Storage of wood as logs increased the proanthocyanidin concentration in both fresh and dried wood. Proanthocyanidins obviously polymerised and oxidized to coloured compounds during drying, as the proanthocyanidin concentration was found to be lowest in the darkest reddish wood. The combination of drying temperature and prevailing moisture content of wood during the drying process seemed to be very important for formation of coloured phenolic compounds.

Key words: Proanthocyanidins, silver birch, wood discolouration, wood drying

Introduction

For many light-coloured wood species, discolouration of wood during artificial drying is a considerable problem, because it restricts the usability of sawn timber and reduces the value of the wood products. The appearance of discolouration, as well as the compounds involved and synthetized in discolouration, may vary depending on the tree species (e.g. McMillen 1975, McGinnes and Rosen 1984, Wegener and Fengel 1988, Koch and Bauch 2000). In general, chemical discolouration is thought to be a result of the oxidative and polymerising reactions of wood extractives and cell-wall compounds. In sawn birch timber, discolouration typically appears in the interior of the boards by darkening or reddish colouring of the wood, while the surface layer to a depth of 1 – 5 mm remains light (Paukkonen *et al.* 1999, Luostarinen and Luostarinen 2001). Because little information on the chemical basis of discolouration with birch is available, the means of avoiding chemical discolouration in sawn birch timber are restricted mainly to slow drying in the open air before kiln drying (Kataikko 1996) or to the use of slow and mild drying schedules, which in turn, lowers the profitability of the timber production.

In woody plants, after lignin the most common polyphenols are the proanthocyanidins (condensed tannins); and they are present in all parts of trees, also in wood (Haslam 1975). Unlike lignin, the proanthocyanidins are not structural, being located mainly in cell vacuoles. In hot and acid conditions, soluble proanthocyanidins are known to polymerise to insoluble red substances; and they may also form new oxidised and coloured complexes, sometimes called “phlobaphenes” (Hillis 1985), the structure of which may be altered by treatment of the sample, for example, by drying or storage (Stafford 1988). The amount of proanthocyanidins in wood is probably very different in different tree species; e.g. in oak wood they are known to accumulate in large quantities (Scalbert *et al*. 1989, Lavisci *et al.* 1991). Julkunen-Tiitto *et al*. (1996) found that more

than 5 % of the stems (including bark and cambium) of young silver birch saplings were composed of condensed tannins, catechin-derived condensed tannins being typical for all birch species. Catechin also has an ability to make complexes with cell-wall lignins (Kodera *et al*. 1979), which may cause discolouration of wood as well.

In both softwoods and hardwoods, seasonal changes in extractives of wood are thought to affect formation of discolouration during drying (Kreber and Byrne 1994). The intensity of discolouration of birch wood has been found to differ, in particular, between different felling seasons and storage periods of logs; the mechanism of discolouration is thought to differ depending on drying method (Luostarinen *et al.* 2002, Möttönen and Luostarinen 2002). According to our preliminary unpublished results, discolouration of birch wood during drying also differs between natural and plantation forests. However, lack of information about the formation of chemical discolouration during drying restricts the possibilities to reduce discolouration by adjusting drying schedules based on little-studied factors (e.g. origin of the raw material, felling season, storage of logs) and by developing other means of inhibiting discolouration.

The objective of this study was to investigate the role of proanthocyanidins in discolouration of sawn birch (*Betula pendula* Roth) timber from plantation forests during conventional kiln drying and vacuum drying. For the conventional warm-air drying method, changes in proanthocyanidin concentration during the drying process were investigated in order to determine the role of drying conditions in changing concentrations of soluble proanthocyanidin in birch wood.

Materials and methods

During 1999, silver birch (*Betula pendula* Roth) trees from two 33-year-old planted stands were felled in summer, autumn and winter, 20 trees each as described earlier by Möttönen and Luostarinen (2002). The drying schedules used for both drying methods were presented in an earlier publication (Luostarinen *et al*. 2002).

For the proanthocyanidin analysis, sample blocks of undried wood were taken from the upper end of ten butt logs immediately after felling and, for the stored logs, after the eight-week storage period. After the conventional and vacuum drying processes, samples of dried wood were taken from the sawn boards when the target moisture content of 5 % had been reached. In addition, samples were taken during conventional drying at moisture contents of 35, 30 and 25 % based on the readings of the moisture sensors in the kiln; and at each moisture content, one board from each planted stand was chosen. The change in concentration of soluble proanthocyanidin in the wood was also studied more closely in one conventional drying process (drying of boards sawn from logs stored for eight weeks in winter) by taking the samples from the same two individual boards at moisture contents of 55, 35, 30, 25, 20, 15 and 10 %. A gravimetric method was used to determine the moisture content of the samples accurately. In addition, to determine the initial moisture content of the boards, forty sample boards were weighted before and after each drying period.

From the undried wood blocks, wood samples from near the pith and from near the trunk surface were taken separately for analysis of proanthocyanidins. Among the dried boards were also boards sawn from near the pith and boards sawn from near the trunk surface; those two types of boards were analysed separately. If the samples were taken before or during the drying process, they were stored at –18 ºC until the proanthocyanidin was analyzed. The samples taken from dried boards were stored in plastic bags at room temperature.

The colourless, soluble proanthocyanidins were analysed by acid butanol assay, in which the proanthocyanidins are hydrolysed to anthocyanidins in hot mineral acid (HCl/BuOH) solution (Porter *et al.* 1986, Hagerman 1995). The wood samples were ground, and 0.5 g of wood powder from undried samples, or from samples taken during drying and 1.0 g of wood powder from dried samples were extracted by shaking each sample overnight with 50 ml acetone (95 %). The extract was filtered and preserved, and the wood powder was collected from the filter paper and re-extracted as before. The extracts from the two extractions were concentrated to a volume of less than 10 ml each by vacuum evaporator at 40 °C. The volumes were adjusted to 10 ml with acetone (95 %). Then 1.0 ml of the extract and 6.0 ml of the acid butanol reagent were mixed, the solution was shaken and 0.2 ml of a reagent containing iron (2 % ferric ammonium sulphate in 2 N HCl) was added. The solution was shaken vigourously, heated in closed tubes in a boiling water bath for 50 min and cooled; the absorbance was then read with a spectrophotometer (Hewlett-Packard 8453) at 550 nm. The concentration of proanthocyanidin was quantified using cyanidinchloride as standard. In the results the yield of proanthocyanidins is expressed as anthocyanidin equivalents, µg/g (dry weight).

In addition to chemical sampling, the spectral reflectance of wood was measured with a spectrophotometer (Minolta CM-2002). The results of the spectral measurements are reported in detail by Möttönen and

Luostarinen (2002). Reflectance spectra were measured from the thinly planed surface layer of conventionally and vacuum-dried boards. In addition, the dried boards were split-sawn, the inner side of the split board was planed and the reflectance spectra of the inner wood of boards were measured. L*a*b* colour co-ordinates (CIELAB) were calculated from the spectra.

The data were analysed using the analysis of variance and correlation procedures of SPSS-statistics (SPSS Inc.). Tukey's test was used to examine differences in proanthocyanidin concentration between felling seasons, storage periods of logs and locations of wood in the trunk. Pearson's product moment correlation coefficient was used to examine relationships between proanthocyanidin concentration and L*a*b* colour co-ordinates in wood.

Results

Moisture content of undried wood

Boards sawn from near the pith always had higher moisture content than those sawn near the trunk surface (Table 1). The difference between the locations was greatest in winter, when the overall moisture content of wood was lowest. During the eight-week storage period, the logs dried considerably only in summer, but the difference in the moisture content between radial locations did not change markedly.

Table 1. Moisture content (%) of undried boards sawn from different radial locations in the trunk during different felling seasons and after different storage period of logs (unstored, 0; eight weeks stored, 8). Number of boards in locations varied between 8 and 18; in each drying lot the total number of boards was 40. Standard deviation in parentheses

Drying lot	Location of board		
	Near the pith	In the middle	Near the surface
Summer 0	110.70 (9.23)	95.55 (10.43)	100.10 (13.69)
Summer 8	70.78 (14.47)	70.06 (6.82)	62.24 (11.13)
Autumn 0	87.70 (10.44)	83.28 (13.30)	79.77 (8.76)
Autumn 8	89.52 (7.05)	86.49 (10.75)	76.36 (9.66)
Winter 0	88.57 (5.97)	79.30 (6.73)	77.88 (4.87)
Winter 8	89.78 (12.78)	83.21 (7.20)	76.29 (4.32)

Proanthocyanidin concentration of wood

The proanthocyanidin concentration was markedly higher in conventionally dried wood than in undried or vacuum-dried wood (Table 2). The seasonal variation in concentration was greatest in conventionally dried wood, in which the highest concentrations were found in winter and in autumn when the wood was stored for eight weeks before drying (Figure 1). The concentration of proanthocyanidin in fresh wood was also higher in winter than in summer or autumn. Storage of logs increased the proanthocyanidin concentration of both undried and dried wood in summer and, especially in conventionally dried wood, also in autumn.

Table 2. Proanthocyanidin concentration as anthocyanidin equivalents in undried, conventionally dried and vacuum-dried wood. All felling seasons and storage periods of logs are included

	Undried wood	Conventionally dried wood	Vacuum dried wood
PA concentration, µg/g (dry wt)	13.3	49.2	11.9
Standard deviation	6.8	29.2	9.2
Min	4.8	9.2	4.2
Max	31.6	106.8	19.7

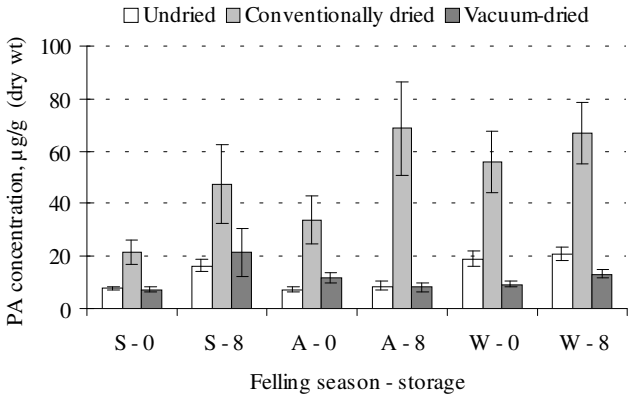

Figure 1. Proanthocyanidin (PA) concentration as anthocyanidin equivalents (±SD) in undried, conventionally dried and vacuum-dried wood in different felling seasons (summer, S; autumn, A; winter, W) after different storage periods of logs (unstored, 0; stored eight weeks, 8)

With regard to the radial location of wood, in both fresh and dried wood the proanthocyanidin concentration was highest in boards sawn near the pith of the trunk (Figure 2). The difference in proanthocyanidin concentration between radial locations was greater in conventionally dried wood than in fresh or vacuum-dried wood.

During conventional drying, the concentration of proanthocyanidin was always highest when the moisture content of wood was 20 – 35 %, i.e. near the wood-fiber saturation point (Figure 3). After that, it decreased relatively steeply until the target moisture content of wood (5 %) was reached. Although the proanthocyanidin concentration at the target moisture content was always highest in stored wood, no notable differences between unstored and stored wood were observed during drying.

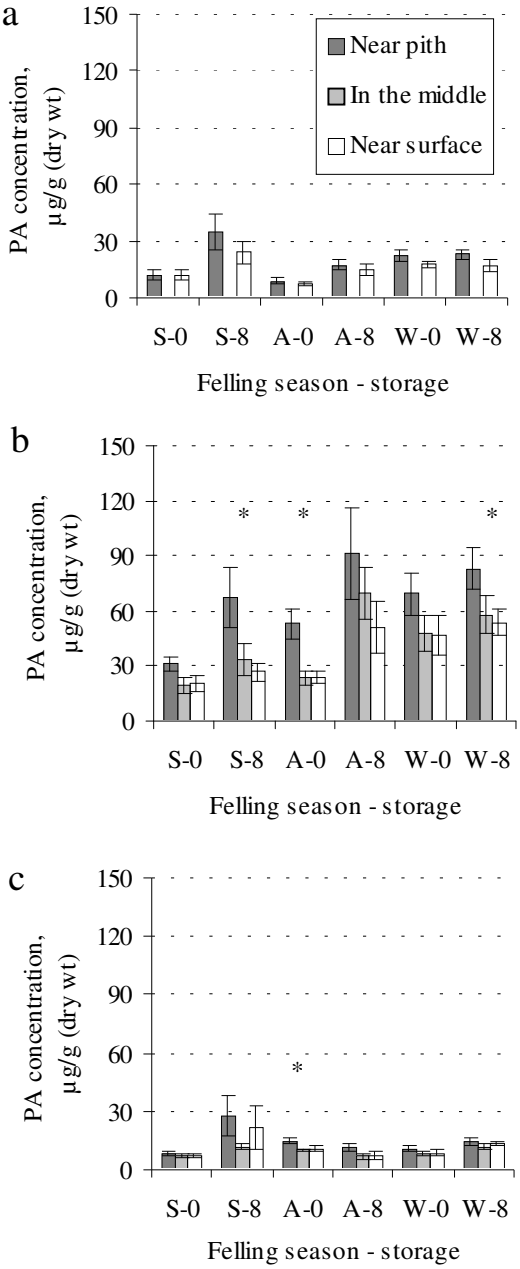

Figure 2. Variation in proanthocyanidin (PA) concentration as anthocyanidin equivalents (±SD) in undried (a), conventionally dried (b) and vacuum-dried (c) wood at different radial locations in the trunk. Felling season and storage of wood as in Fig. 1. An asterisk (*) denotes significant difference in proanthocyanidin concentration between wood near the pith and wood near the trunk surface

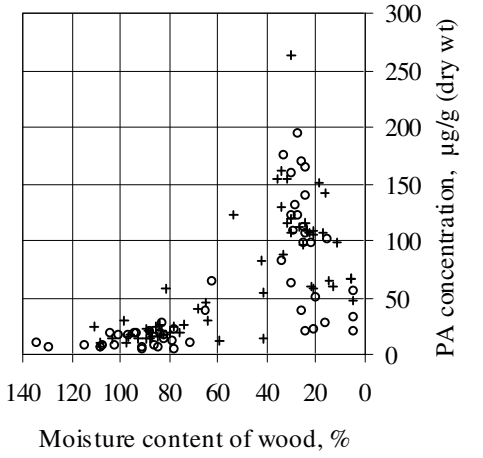

Figure 3. Proanthocyanidin (PA) concentration as anthocyanidin equivalents (±SD) in fresh wood (MC above 60 %), during drying of wood (MC between 10 % and 60 %) and in dried wood (MC below 10 %). Storage of wood as logs before drying: unstored (o), stored eight weeks (+). All felling seasons are included. The value points for dried wood are the mean values of 20 separate samples of each drying lot; otherwise they are single observations

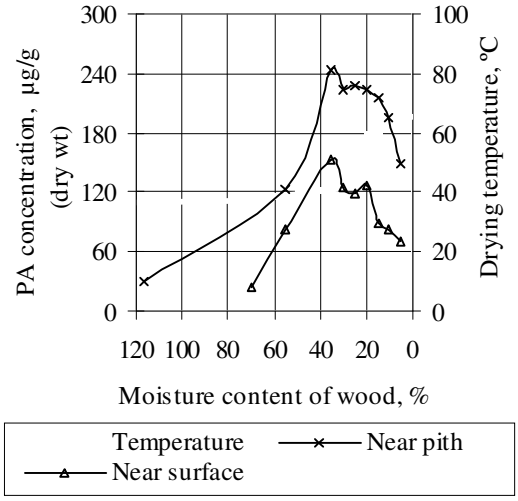

Figure 4. Proanthocyanidin (PA) concentration as anthocyanidin equivalents (±SD) in two individual sample boards during the conventional drying process

In the test made with the two individual boards, the proanthocyanidin concentration of wood increased steadily during drying until the moisture content was ca. 35 %, at which the maximum concentration was reached in both sample boards (Figure 4). At moisture content of 20 - 30 %, the proanthocyanidin concentration was constant; but when the drying temperature was raised at moisture content of 20 %, the concentration of proanthocyanidin started to decrease very steeply.

Correlation between proanthocyanidin concentration and colour of dried wood

During vacuum drying, the surface layer of the boards discoloured more than the inner wood; whereas during conventional drying, the inner wood discol-

oured more (see Möttönen and Luostarinen 2002). With regard to dried wood sawn from unstored logs, soluble proanthocyanidins correlated significantly with colour co-ordinates measured from the surface layer of both conventionally dried and vacuum-dried boards (Table 3). With different drying methods, however, the sign of the correlation coefficient differed (Table 3, Figure 5). From the colour co-ordinates measured from the inner wood of boards, after conventional drying the proanthocyanidin concentration correlated most strongly with redness and after vacuum drying with yellowness. Storage of logs affected the relationship between proanthocyanidin concentration and colour co-ordinates of dried wood, as no significant correlation was found.

Table 3. Correlation coefficients between the proanthocyanidin concentration (PA) and the colour coordinates measured from the inner wood (i) and surface layer (s) of boards after conventional drying (c) and vacuum drying (v). Storage periods of logs: unstored (0); stored eight weeks (8). Number of samples in each case was 60

	L^*_i	L^*_s	a^*_i	a^*_s	b^*_i	b^*_s
PA_{c0}	-0.03	0.58**	0.41**	-0.47**	-0.13	-0.59**
PA_{c8}	-0.11	0.16	0.33*	0.04	0.00	-0.20
PA_{v0}	-0.33*	-0.48**	0.33*	0.42**	0.40**	0.51**
PA_{v8}	-0.07	0.25	-0.08	-0.18	-0.06	-0.20

* correlation is significant at the 0.05 level
** correlation is significant at the 0.01 level

In conventionally dried wood, the radial location in the trunk affected the correlation between proanthocyanidin concentration and colour co-ordinates (Table 4). In particular, the correlation between proanthocyanidin concentration and the colour co-ordinates of the surface layer of boards increased from wood near the pith towards the trunk surface.

Discussion and conclusions

In this study the soluble proanthocyanidins in the wood of young birches clearly changed with season. Fresh wood contained significantly more proanthocyanidins during winter dormancy than during the growing season in summer or in early autumn; and after conventional drying, the differences between seasons were even emphasized. According to Mononen *et al.* (2001), in both fresh and conventionally dried wood of mature birch trees, the concentration of (+)-catechin-7-O-β-D-xylopyranoside, a phenolic compound, which is an apparent precursor of proanthocyanidins, is also highest in winter. In addition, concentrations of ether extracts (Perilä and Toivonen 1958), as well

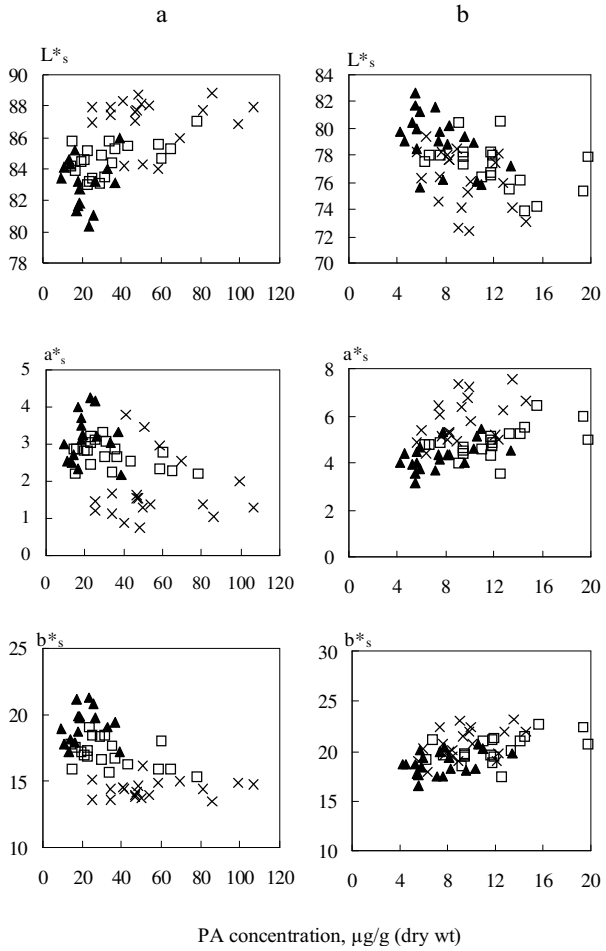

Figure 5. Relationship between proanthocyanidin concentration (PA) of unstored conventionally dried (a) and vacuum-dried (b) wood and colour coordinates (L*, a*, b*) of surface layers. Felling seasons are denoted by the following symbols: summer (▲); autumn (□); winter (×)

Table 4. Correlation coefficients between the proanthocyanidin concentration (PA) and the L*a*b* colour coordinates in boards from different radial locations in the trunk after drying. Colour coordinates: inner wood of boards (i), surface layer of boards (s)

Near the pith

		L^*_i	a^*_i	b^*_i	L^*_s	a^*_s	b^*_s
PA	conventional	-0.27	0.44**	0.10	0.02	0.08	-0.16
	vacuum	0.10	-0.20	-0.27	0.36*	-0.26	-0.24

In the middle between the pith and the trunk surface

		L^*_i	a^*_i	b^*_i	L^*_s	a^*_s	b^*_s
PA	conventional	-0.06	0.24	0.05	0.34*	-0.17	-0.41*
	vacuum	-0.41*	0.32*	0.44**	-0.54**	0.41*	0.40*

Near the trunk surface

		L^*_i	a^*_i	b^*_i	L^*_s	a^*_s	b^*_s
PA	conventional	0.02	0.24	-0.17	0.49**	-0.38*	-0.56**
	vacuum	-0.23	0.10	0.08	-0.15	0.07	0.08

* correlation is significant at the 0.05 level
** correlation is significant at the 0.01 level

as soluble sugars (Piispanen and Saranpää 2001a) of birch wood are known to differ between seasons.

During storage, the proanthocyanidin concentration of undried wood increased, especially in summer, when the conditions undoubtedly were best for enzyme-catalysed synthesis of proanthocyanidins. According to Laver and Musbah (1997), enzymatic activity related to colour formation in wood is dependent on pH and temperature. In summer, the increased proanthocyanidin concentration of undried wood was maintained in dried wood regardless of drying method. After storage, however, the dried wood was darker and more reddish (see Möttönen and Luostarinen 2002), whereas the usual trend was for the colour to become lighter and less reddish as the concentration of proanthocyanidin increased. In fact, many other compounds, for example, storage components (Piispanen and Saranpää 2001a), may be involved in colour; and the chemistry of discolouration may be more complicated in wood stored during warm weather before drying.

The fluctuation of proanthocyanidin concentration observed in wood during conventional drying in relation to temperature and moisture content of wood obviously plays a decisive role in discolouration. During the early phase of the conventional drying process the increase in soluble proanthocyanidins indicates that these compounds were formed from their precursors as a result of the mild temperature and the adequate moisture content of the wood (e.g. Botha *et al*. 1981). In addition, the lack of oxygen in moist wood during the early phase of the drying process may have inhibited oxidative transformation of phenolic compounds. Koch and Bauch (2000) also assumed for European beech wood (*Fagus sylvatica* L.) that the discolouration-forming reactions could be initiated by the oxygen potential in the wood cells during drying. The oxidised and polymerised reaction products of proanthocyanidins, which are mainly insoluble, were formed at the elevated temperature after the moisture content of wood decreased below the saturation point of the wood fiber. This was seen as a decrease in soluble proanthocyanidins at the end of the drying process. Darkening of birch wood has been observed to start at the same level, at a moisture content of 30 – 35 % (Luostarinen *et al.* 2002). However, the precursors of coloured compounds were probably formed in the inner wood of the boards above the saturation point of the wood fiber, because for discolouration of birch wood the dominating factor has been found to be the timing of heat treatment (Sundqvist 2002).

The reason for strong discolouration of wood during vacuum drying, especially in the surface layer of boards, was the exaggeratedly high temperature (65 – 82 °C). In addition, the proanthocyanidin concentration of vacuum-dried wood was low. It is probable that the enzymatic synthesis of proanthocyanidins was hindered due to the high temperature used already from the beginning of drying. The effect of temperature on proanthocyanidin concentration has been found previously during conventional kiln drying of sawn birch timber at different temperatures (Paukkonen *et al.* 1999) and during oven and vacuum drying of purple willow leaves (Julkunen-Tiitto and Sorsa 2001). The discolouration of surface layer of vacuum-dried boards at high temperature, however, is more probably caused by the enrichment of low-molecular-weight sugars and nitrogen at the surface of sawn timber during drying (Terziev and Boutelje 1998, Kreber *et al*. 1998, Piispanen and Saranpää 2001b). In addition, these soluble substances can be transported and enriched on the surface of sawn timber far below the saturation point of the fibers (Terziev 1995).

The proanthocyanidin concentration of wood was most strongly correlated with colour co-ordinates of wood measured from the surface layer (1 – 2 mm below the yellowish surface) of the boards. This indicates that the increased proanthocyanidin concentration observed in conventionally dried wood, especially in winter, was located in the surface layer of boards that remained the lightest in colour. We have previously assumed that the reason for the light surface layer in winter is premature drying of the surface layer of boards at the beginning of conventional drying at low temperature (Möttönen and Luostarinen 2002). In this phase, water that has been frozen before the beginning of drying is more pronounced in the cell cavities than in cell-wall tissue, owing to the way water freezes in wood (see e.g. Kübler 1962, Skaar 1988) and it is capable of drying out of wood easily. Mononen *et al.* (2001) found, with DSC-analyses, that the proportion of pore water (in very small pores in the cell-wall) in relation to free water (mainly in cell cavities) decreased in fresh birch wood during winter storage of logs. Thawed water in the cell cavities near the surface of boards may not have had time to be absorbed by the cell wall at the beginning of the drying process. In this study, because the surface layer of boards was able to dry considerably at low temperature in winter, the proanthocyanidins in the surface layer did not polymerise/oxidise to coloured compounds to the same extent as during other seasons.

In an earlier study with this material, the radial location of wood in the trunk was observed to affect the colour of both the surface layer and the inner wood of conventionally dried boards (Möttönen and Luostarinen 2002). According to the present study, there are also differences in the transformations of proan-

thocyanidins in boards from different locations, as both the proanthocyanidin concentration and its correlation with colour co-ordinates differs between radial locations in conventionally dried wood. The difference in the initial moisture content of undried wood, which also was observed in this study, may have caused the boards from different locations not to dry simultaneously, *i.e.* the moisture content of boards sawn from near the pith may have been higher than that of the surface boards also during the drying process when the drying temperature was raised.

The results obtained here support the conclusion that polymerisation and oxidation of proanthocyanidins plays an important role in discolouration of birch wood during drying. The drying temperature and its level during different phases of the process are decisive factors for formation of polymers or their precursors. However, in addition to proanthocyanidins, other compounds also take part in discolouration.

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ЦВЕТОИЗМЕНЕНИЕ ДРЕВЕСИНЫ БЕРЕЗЫ (*BETULA PENDULA*) ЗАГОТОВЛЕННОЙ В ИСКУССТВЕННЫХ ЛЕСАХ, В ХОДЕ СУШКИ: РОЛЬ ПРОАНТОЦИАНИДИНА (КОНЦЕНТРИРОВАННОГО ТАНИНА) В ЦВЕТОИЗМЕНЕНИИ БЕРЕЗОВОЙ ДРЕВЕСИНЫ.

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Резюме

Исследуется роль проантоцианидов в цветоизменении при сушке древесины. При традиционной сушке горячим воздухом в древесине проявляется самая высокая концентрация проантоцианидов. Хранение древесины бревнами увеличило концентрацию проантоцианидов как в свежей, так и в высушенной древесине. Проантоцианиды явно полимеризировались и окислялись до цветовых компонентов в ходе сушки, так как концентрация проантоцианидов была самой низкой в древесине, имеющей наиболее темный красноватый оттенок. Комбинация температуры сушки и преобладающее содержание влаги в древесине в ходе процесса сушки также имели большое значение для формирования цветовых фенольных компонентов.

Ключевые слова: проантоцианиды, береза повислая, цветоизменение древесины, сушка древесины