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ORIGINAL ARTICLE

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Colour of larch heartwood and relationships to extractives and brown-rot decay resistance

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Abstract Larch heartwood is appreciated for its good mechanical properties, its colour and its texture, and it is often used outdoors because of its natural durability (decay resistance). In this study the colour of larch heartwood was studied in relation to extractives and decay resistance, with the aim to estimate durability of larch heartwood from its colour. On a total of 293 trees colour in the CIE L*a*b* space (L* lightness, a* red/green axis, b* yellow/blue axis), extractives content (acetone and hot-water extractives, amount of phenolics) and the brown-rot decay resistance were determined. For calculating the relative decay resistance (x), mass loss after inoculation for 16 weeks with two fungi [*Coniophora puteana* (Schum.ex.Fr.) Karst., *Poria placenta* (Fr.) Cke, European standard EN 113] of larch heartwood samples was compared to Scots pine (*Pinus sylvestris* L) sapwood reference samples (EN 350-1). Different species [Japanese larch (*Larix kaempferi* Lamb.), Hybrid larch (*Larix decidua*x *L. kaempferi)* and European larch (*L. decidua* Mill.)], provenances and age classes (38-year, >150-year) were included. Japanese larch heartwood turned out to be significantly more reddish (higher a*-values) compared to the European larch provenances. Reddishness of the hybrids was intermediate. The red hue (+a*) was strongly correlated with the amount of phenols (r=0.84) and decay resistance (r=0.63) and therefore suitable for prediction of both parameters. The results suggest that colour measurements of larch heartwood could be of benefit in tree breeding programs and for an optimised utilization of larch timber.

Keywords *Larix* sp. · Phenols · Natural durability · Relative decay resistance · CIE L*a*b*

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Introduction

The genus *Larix* encompasses ten species that are widely distributed across the cooler regions of the northern hemisphere. In Europe the natural habitat of *Larix decidua* Mill. (European larch) is scattered and there are various geographic races (subspecies) and ecotypes, which exhibit marked differences in growth rates and other tree characteristics (Schober 1985). Larch (*Larix* sp.) is much appreciated for its mechanical properties, its colour and texture, and its natural durability (Knuchel 1954). Its strength and resistance to decay make it well suited to use as floor planking, building skids, pilings, posts and poles. Natural durability, or alternatively decay resistance, is defined as the ability of wood to resist biological degradation. It is generally low in the sapwood and high in the heartwood of some species (Eaton and Hale 1993). Heartwood formation is associated with cell death, the disappearance of storage material and an increase in extractive content (Bamber 1976). Heartwood extractives comprise terpenoids, tropolones, flavonoids, stilbenes, and other aromatic compounds (Scheffer and Cowling 1966). The significance of heartwood extractives for natural durability was demonstrated by Hawley et al. (1924), and has been repeatedly discussed in the literature

Table 1 Description of the samples and sites: name (corresponding to origin or provenance and the country of the site; *F* France, *GB* Great Britain, *D* Germany, *B* Belgium, *A_nat* natural stand in Austria, *F_nat* natural stand in France), average tree age (age), number of trees (N), and species. The old slow grown trees from natural stands are shaded in grey

Name (origin-site)			Age	*N*	Species
Ina-F			38	20	Japanese
Hybrid-F			38	29	Hybrid
Hybrid-GB			39	36	
Ruda-D			38	29	European
Ruda-F			38	20	
Ruda-B			38	20	
Zabreh-F			38	19	
Zabreh-B			38	20	
Blizyn-D			38	20	
Langau-B			38	20	
Montgenevre-F			38	18	
Langau-A_nat			160	26	European
Country	site	Latitude and longitude			a.s.l (m)
GB	Clanna	51°43′N, 2°35′W			90
F	Coat an Noz	48°31′N, 3°25′W			200
B	Na-ssogne	50°05′N, 5°20′E			320
D	Elm	52°00′N, 10°03′E			205
A_nat	Langau	47°49′N, 15°10′E			1050
F_nat	Mont-genevre	44°56′N, 6°44′E			1800

(e.g. Rudman 1963; Celimene et al. 1999; DeBell et al. 1997; Schultz et al. 1990, 1995). Toxic extractive compounds are recognized to be the most important factor (Hillis 1987) and in some durable species even low-toxic extractives may contribute synergistically to high durability (Schultz and Nicholas 2000). Besides the spatial distribution of extractives at the cellular level, impregnated in the cell walls or just in the cell lumina, wood structure itself may be of importance (Hillis 1971; Kleist and Schmitt 1999).

The colour of wood depends on chemical components interacting with light, i.e. the presence or absence of extractives (Hon and Minemura 2001). Consequently, wood colour has been related to the amount of extractives, to wood decay resistance and to wood density (Hiller et al. 1972; Nelson and Heather 1972; Wilcox and Piirto 1976).

Brown-rot decay resistance of larch (*Larix* sp.) heartwood is described as highly variable, ranging from non durable to moderately durable (class 5 to 3, EN 350-2) (Viitanen et al. 1997; Morrell and Freitag 1995; Srinivasan et al. 1999; Nilsson 1997; Jacques et al. 2002). Furthermore, for the amount of heartwood extractives, a huge variability within and between trees, across sites, species, provenances and tree age has been observed (Gierlinger et al. 2002b; Dix and Roffael 1994; Coté et al. 1966). A strong relationship between extractives content and brown-rot decay resistance has been shown (Gierlinger et al. 2002a; Windeisen et al. 2002).

Information on variation of wood colour of larch is very scarce, Simak (1957) giving some data for Slovak larches. The relationship between colour and heartwood extractives and natural durability is not known for larch wood. We hypothesize that heartwood colour in larch may be related to both and therefore could become an easy to measure indicator for extractives content and decay resistance.

Materials and methods

Material

A total of 293 larch trees grown in four plantation sites and in two natural stands were included in this investigation (Table 1). Some of the trees were from an IUFRO provenance trial in Belgium, Germany and France, with selected European larch (*Larix decidua* Mill.) provenances from the Alps (Montgenèvre, Langau), from the Sudetan mountains (Ruda, Zabreh), and from Poland (Blizyn) (Table 1). In addition, one Japanese larch (*Larix kaempferi*) origin (Ina) and two hybrids (*L.×eurolepis*) were included as well. In summary, 13 different groups of larch wood were analyzed (Table 1).

Sample preparation

From each tree, a 2-m long log was cut at breast height upwards, which was further sawn to disks and boards. For colour measurements and chemical analysis clear samples were prepared from the entire heartwood. Samples were dried at 50°C and ground with a cutting mill (Retsch, SM1) to pass a 200 μm screen (wood meal) and the fraction below 100 μm (wood powder) was separated with an analytical sieving apparatus (Retsch, AS 200 basic). For wood decay tests a set of 24 samples was prepared from each tree, half taken from the inner and half from the outer part of the heartwood. Samples (50×25×15 mm) were planed and placed in a standard climate chamber to equalise at 12% wood moisture content prior to fungal inoculation. Sixteen samples were submitted to wood decay tests and 8 were used as standards to calculate reference dry matter.

Colour measurements

Colour measurements were performed on dried wood powder (<100 μm) to minimise wood structural effects. The reflection spectrum was taken from a 12 mm measuring spot in the 400–700 nm region with a Phyma Codec 400 Vis spectrometer. The recommended CIE (Commission Internationale de lÈclarirage) color parameters L* (lightness), a* [along the *X* axis red (+) to green (−)] and b* [along the *Y* axis yellow (+) to blue (−)] were calculated (Hunt 1995) and an average value of three measurements per sample was used for further analysis.

Chemical analysis

About 3 g air-dried wood meal (100–200 μm particle size) was extracted using the *fex* IKA 200 solid extractor. Extractions were carried out with acetone (Carl Roth, 5025.2) for 6 h followed by another 6-h hot-water extraction. Extractive contents were determined gravimetrically according to TAPPI T204 om-88 (% based on dry wood) and water contents according to TAPPI T 264 om-88/8.2. From acetone extractives (ACE) and hot-water extractives (HWE) content the total amount of extractives (TOT) was calculated. The phenol content was determined in the acetone and the hot-water extracts by means of a modified Folin-Denis assay (Swain and Hillis 1959). The flavonoid taxifolin (3, 3′, 4′, 5,7-pentahydroxyflavanone) was used as a standard (Sigma Aldrich, T4512). Analyses of 100 trees were carried out with four replications on a LKB Biochrom 4060 UV-VIS spectrophotometer and the mean calculated. The amount of phenols in the ACE and HWE were summarized as the total amount of phenolics (PHE). PHE of the remaining samples was estimated by means of FT-NIR models, as shown in Gierlinger et al. (2002c).

Wood decay tests

Wood decay test were performed according to European Standard EN113 and EN350. A total of 4,688 larch samples and reference samples of *Pinus sylvestris* sapwood were tested. For each tree, 8 samples were inoculated with *Poria* (= *Postia*) *placenta* (Fr.) Cooke (strain FPRL280) and another 8 samples with *Coniophora puteana* (Schum.: Fr.) Karst. (strain FPRL11E). After 16 weeks the mycelium was removed from the samples prior to drying at 103°C to weight constancy. The mass loss was calculated and divided by the mass loss of the pine reference, resulting in a ratio called "*x*-value" as suggested in EN 350-1. Data were analysed using average values of the 8 *Poria* and 8 *Coniophora* samples.

Data analysis

The SPSS 10.0.5 software package was used for statistical analysis. Pearson correlations were employed and due to correlations between the extractive parameters themselves and between extractive contents and wood decay, a partial correlation analysis was also performed. The Partial Correlations procedure computes partial correlation coefficients that describe the linear relationship between two variables while controlling for the effects of one or more additional variables. The one-way ANOVA (analysis of variance) procedure was used to test the hypothesis that the means of the investigated groups (age groups, provenances) are equal. Additionally the Scheffé-test (at α=0.05) was applied to show which group means differ. UNSCRAMBLER software package (Version 7.6, CAMO ASA) was used to calculate partial least squares (PLS) regression models to predict the variation in wood decay resistance and phenolics by the colour parameters.

Results

A one-way ANOVA revealed significant differences in the L*a*b* values between species, provenances and sites as well as between the young trees from plantations and the old trees from natural stands (Scheffé-test in Table 2). Colour values of the trees from the same provenances grown on different sites (Ruda from D, F, B and Zabreh from F, B) were close together, whereas between trees of the same site but of different origin or species significant differences were observed (Table 2). The a*-value (describing the reddishness of the samples) showed that Japanese larch (Ina-F) and also the hybrids had a significant redder hue than the young European larch trees (Table 2, Fig. 1). Moreover, the old trees from

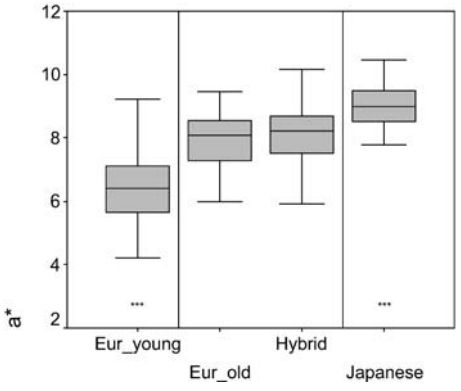

Fig. 1 Differences in the colour values a* (red hue) between European (*Eur_young*), Japanese and Hybrid larch trees from plantations and old European larch trees from natural stands (*Eur_old*) (*** differences are significant in the Scheffé-test at α=0.05)

Table 2 Homogenous subsets of the colour-values L*a*b* applying the Scheffé-test (subsets for α=0.05) (*L**luminosity, *a** red/green axis, *b** yellow/blue axis). A description of the samples and sites is given in Table 1. (*Hyb* Hybrid, *Mont* Montgenevre, *Bliz* Blizyn, *Zabr* Zabreh, *Lang* Langau)

L*						a*						b*				
Hyb-F	73.41					Lang-B	5.58					Lang-B	23.35			
Mont-nat	73.67					Ruda-D	5.60					Ruda-D	24.07	24.07		
Mont-F	75.11	75.11				Ruda-B	6.23	6.23				Ruda-F	24.14	24.14		
Bliz-D		75.83	75.83			Ruda-F	6.70	6.70				Ruda-B	24.39	24.39	24.39	
Ina-F		75.88	75.88			Zabr-B	6.72	6.72				Mont-F		24.68	24.68	
Hyb-GB		75.95	75.95			Bliz-D	6.94	6.94	6.94			Zabr-B		24.89	24.89	
Zabr-F		76.03	76.03			Zabr-F		7.14	7.14			Lang-nat		24.93	24.93	
Lang-nat		76.78	76.78	76.78		Mont-F		7.25	7.25	7.25		Ina-F		25.24	25.24	25.24
Zabr-B		77.20	77.20	77.20	77.20	Lang-nat		7.60	7.60	7.60	7.60	Bliz-D			25.49	25.49
Ruda-D			77.47	77.47	77.47	Hyb-GB			8.13	8.13	8.13	Zabr-F			25.61	25.61
Ruda-B				78.38	78.38	Hyb-F			8.25	8.25	8.25	Mont-nat				26.17
Ruda-F				78.45	78.45	Mont-nat				8.59	8.59	Hyb-GB				26.23
Lang-B					79.29	Ina-F					8.99	Hyb-F				26.27
Signifi-cance	0.347	0.075	0.429	0.380	0.069	Signifi-cance	0.064	0.060	0.098	0.075	0.053	Signifi-cance	0.230	0.086	0.055	0.239

Table 3 Pearson and partial correlation coefficients for the color values *L** (luminosity), *a** (red/green axis), *b** (yellow/blue axis), extractive contents and the average *x* -value from wood decay tests. Correlations were performed using tree values and averages of the 13 investigated groups (origin-site, Table 1) (coefficients are significant at *P* >5%, 2-tailed)

	Pearson (trees, *n* =293)			Pearson (origin, *n* =13)			Partial (trees, *n* =293)		
	L*	a*	b*	L*	a*	b*	L*	a*	b*
ACE	–0.30	0.66	0.30	n.s	0.76	n.s	n.s	n.s	n.s
HWE	–0.34	0.44	0.21	–0.63	n.s	n.s	n.s	n.s	n.s
TOT	–0.34	0.50	0.24	–0.64	0.60	n.s	n.s	n.s	n.s
PHE	–0.50	0.84	0.45	–0.66	0.93	0.63	–0.33	0.64	0.29
X	0.44	–0.63	–0.37	0.76	–0.82	–0.70	n.s	n.s	n.s

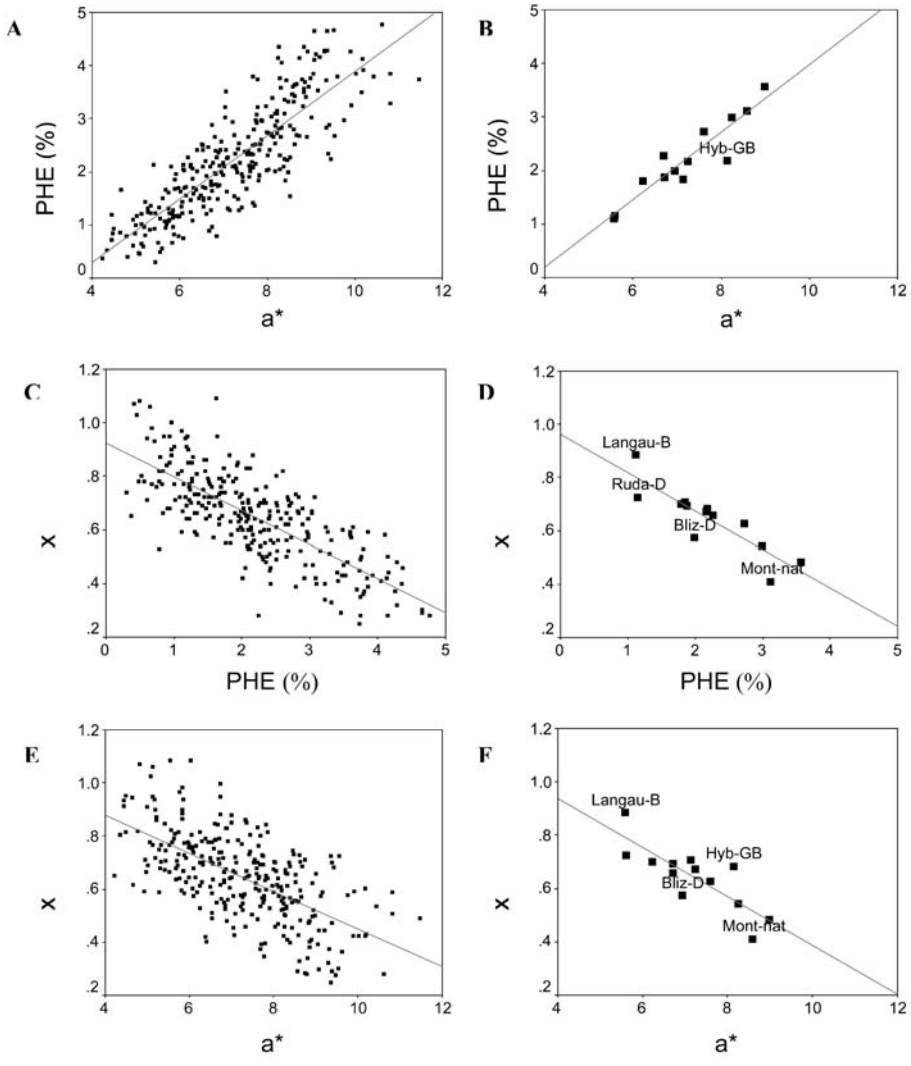

Fig. 2A–F Relationships between red hue (*a**) and amount of phenolics (*PHE*) (**A, B**), between PHE and x (relative brown-rot decay resistance) (**C, D**) and between a* and x (**E, F**). On the left side 293 individual tree values are plotted and on the right side samples were averaged according origin and site (13 groups, see Table 1)

natural European larch stands (Mont-nat, Lang-nat) had higher a*-values than the young trees of the same provenances grown on plantations (Lang-B, Mont-F) (Table 2, Fig. 1). The parameter b* corresponds to the blue to yellow axis and was found to be highest in the heartwood of the hybrids (Table 2). The wood from Ruda and Langau-B had the greatest lightness L* and the lowest a* and b* values (Table 2).

Correlation coefficients between the colour coordinates, extractive contents and the relative decay resistance (*x*-values) are listed in Table 3. Significant Pearson correlations were observed throughout using 293 individual tree values (Table 3A). From the three colour variables a* showed the highest correlations with all parameters, ranging from 0.44 with HWE to 0.84 with PHE (Table 3A). Correlations based on average values of the 13 investigated groups, showed higher coefficients, up to 0.93 between phenols and a* (Table 3B). The strong relationship, the redder (a*) the heartwood the more phenolics, is illustrated with all individual samples as well

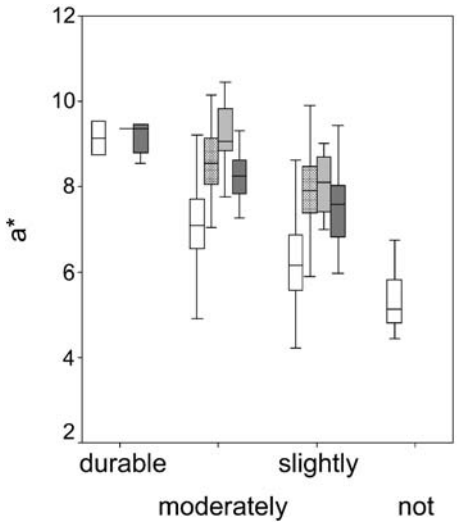

Fig. 3 The observed a*-values (red hue) of the samples grouped according to the estimated *x*-values in natural durability classes (EN350-1) (*durable* class2: *n*=8, *moderately durable* class3: *n*=102, *slightly durable* class4: *n*=164, *not durable* class5: *n*=17). (*Clear boxes* European larch young, *Cross hatched* European larch old, *light grey* Hybrid larch, *dark grey* Japanese larch)

as with group averages in Fig. 2A, B. Colour differences between origins and sites are associated with differences in the amount of phenolics. In Fig. 2C, D the strong negative correlation between relative wood decay resistance (*x*) and the amount of phenolics is shown. Finally in Fig. 2E, F it is shown that the redder the heartwood (a*), the smaller is the relative decay resistance (*x*), which means longer durability in outdoor exposure.

It was interesting to observe that in the partial correlations most significant coefficients vanished with exception of the strong correlation between a* and the phenolics (PHE, Table 3C). The strong relationship between wood decay (*x*) and a* (r =–0.63) disappeared since both parameters are linked with the phenols in heartwood of larch (Fig. 2A–D). Summing up, the amount of phenols turned out to be strongly correlated with the reddishness (a*) of larch heartwood.

The sample set was classified into durability groups based on the calculated *x*-values according to EN350-1 and significant differences between the a*-values of the four groups were found (Scheffé-test at α=0.05). The mean value of a* increased from 5.4 in the trees classified as not durable, to 6.8 in the slightly durable class, to 8.1 in the moderately durable class and up to 9.3 in the trees classified as durable. Figure 3 demonstrates the abundance of young European larch plantation trees in all 4 classes and the increase in colour within this species with higher durability. Within the classes moderately and slightly durable all four investigated groups were found with considerable differences in reddishness (a*) (Fig. 3).

Finally partial least squares (PLS) regression models were calculated to predict *x* -values (relative decay resistance) and the amount of phenolic substances (as an indicator of decay resistance) with the three colour coordinates L*, a* and b*. The amount of phenolics was predicted with a root mean square error of cross validation (RMSECV) of 0.54% (range 0.3–4.7%, the relative decay resistance (*x*) with a RMSECV of 0.126 (range 0.18–1.09).

Discussion

As expected colour of larch heartwood was very variable, significant differences were found between young and old trees and the three investigated species. We have a poor understanding of the influence of genetic and environmental factors in controlling wood colour and associated properties of extractives content and decay resistance. Wood colour may vary greatly within a given species due to different environmental conditions and silvicultural treatment (Nelson et al. 1969; Sullivan 1967; Wilkins and Stamp 1990). Mosedale et al. (1996) found that the greatest differences in most colour parameters of European oak occurred between sites rather than between species, although they reported indicators of genetic control for oak wood colour. For black walnut heartwood colour, a genetic control was found to be weak or non-existent (Rink 1987). For wild cherry, a large family variation (nearly twice that of individuals within family variation) was observed especially for the a* parameter together with high levels of heritabilities at the family level (Janin 1996). Nelson et al. (1969) suggested that soil properties are associated with wood colour of walnut, independent from effects with diameter-growth rate and tree age. In our study, greater differences were observed between provenances on the same site rather than for given provenances on different plantation sites (Table 2). This suggests that origins (genetics) had a stronger effect on the colour of larch heartwood than the growth site. Significant differences were also observed among species of larch: Japanese larch and hybrids were found to have a significantly redder hue than European larch, and hybrids had high b*-values (yellow). Additionally, an effect of age and growth rate on the heartwood colour was observed, as the slowly grown mature trees from native sites showed higher a* and b*-values, compared to the young rapidly grown plantation trees of the same provenance.

We expected that wood colour would be related with extractives content, and our data support this hypothesis. The a*-coordinate turned out to be strongly correlated with phenolics. Such relationships were found for various other species, e.g. walnut, Douglas fir, oak (Klumpers et al. 1994; Burtin et al. 1998; Dellus et al. 1997a, 1997b). The major component (about 80%) of extractable larch phenolics comprises the colourless dihydroquercetin (taxifolin) (Sasaya 1987; Babkin et al. 2001), which may act as the precursor of chromophores (Laver and Arvey 1996; Dellus et al. 1997a). Dellus et al. (1997a) found in experiments on colour of Douglas fir, which belongs together with larch as the plant material having the highest dihydroquercetin content (1–4% w/w) (Gard-

ner and Barton 1960), that the increase of the red hue with the age of the heartwood is linked with a decrease of dihydroquercetin. A model experiment suggests that the poorly extractible pigments of red-orange heartwood are formed by the enzymic reduction of dihydroquercetin, resulting in unstable leucocyanidin, which auto-oxidizes spontaneously to polymeric pigments (Dellus et al. 1997a).

A correlation was anticipated between a* and decay resistance, because of the high correlation between phenols and a* and phenols and decay resistance. Results supported this hypothesis and partial correlations confirmed that the relationship between colour and decay resistance is indirect, based on the influence of extractives on both. The influence of phenolics on wood decay resistance was also demonstrated quite recently for pine heartwood (Harju et al. 2003). Heritability of extractive contents has been reported as high, suggesting that chemical composition could be altered through tree breeding (Fries et al. 2000; Ericsson et al. 2001). We found that colour measurements on wood powder were a good indicator of phenolics and may therefore be useful in breeding for higher phenolic contents and higher decay resistance.

Classification in durability classes (Fig. 3) confirmed the results of correlation analysis, that more reddish larch heartwood (high a*-value) is more durable. But the differences in reddishness (a*) within class 3 (moderately durable) and class 2 (slightly durable) between the young and old trees and different larch species, point to additional effects on decay resistance. The Hybrid larches were found within the moderately and slightly durable classes with relatively high a*-values, compared to European and Japanese larches. Perhaps wider growth rings due to rapid growth affect decay resistance as well as phenolics. In future studies the effect of wood structure on decay resistance and on colour measurements taken from wood surfaces should be explored.

Compared to the prediction of phenolics and natural durability by Near Infrared spectroscopy (NIR) (Gierlinger et al. 2002a, 2002c), the accuracy of the models based on the colour variables was poor. The root mean square error of cross validation was doubled to 0.54% (range 0.3–4.7%) for the models predicting the phenolic content with the colour coordinates (a*, b* and L*), compared to 0.21% in the NIR-model (Gierlinger et al. 2002c). Nevertheless, these first results for the prediction of phenolic content and wood decay resistance based on the colour parameters are encouraging. Prediction might be improved through multivariate calibration models based on the whole visible spectra. A further improvement should be achieved for prediction of decay resistance by using VIS-spectra of wood surfaces, as natural durability is also influenced by wood structure.

Conclusions

The amount of phenolics has a great influence on decay resistance of larch heartwood. The red hue (a*-value) allows the rapid prediction of the amount of phenolic substances, for example from ground increment cores. Therefore, in breeding programmes tree selection for reddishness (a*) could result in more durable wood due to higher phenolics content. To classify wood for exterior timber utilization further research on colour measurements of larch wood surfaces is needed, leading to selection of potentially durable trees during wood processing.

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