Colour change of Nordic Scots pine wood under UV radiation – A laboratory approach

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ABSTRACT

Wood is affected by direct or indirect solar radiation in several outdoor and indoor end-uses. The chemical transformation of a compound into smaller compounds caused by the absorption of ultraviolet, visible, or infrared radiation is called photodegradation. In case of wooden surfaces, the amount of photodegradation can be directly expressed as, for example, changes in extractive composition, or indirectly by measuring the change in the colour of wood during exposure. When studying the ultraviolet–visible light–infrared part of the spectrum, it is the UV part that mainly affects the colour of wood. The shortest wavelengths of the UV part of spectrum are being absorbed in the atmosphere, causing that only the UV-A part of the entire UV radiation can reach the ground surface. This is the part of UV radiation that affects materials in outdoor uses. In indoor uses the effective spectrum of radiation is narrower, thus the radiation is again absorbed, now by the window glass.
The aim of this study was to determine the colour change of Nordic Scots pine (*Pinus sylvestris* L.) wood caused by UVA-351 type fluorescent lamps in rapid exposure test simulating indoor end-uses. Cyclical exposure routines at a constant temperature (+20°C) and relative humidity (65%) were used. The specimens' colour was measured three times – before exposure, approx. in the middle of the total exposure period, and after the entire exposure period. The colour was measured by spectral reflectance method by Minolta CM-2002 spectrophotometer with D_{65} standard illuminant, and the CIE L*a*b* colour coordinates were calculated.
Test material consisted of approx. 500 wood specimens from five sub-regions in Finland and Sweden. Samples were collected from stands of different forest sites and age classes to represent the geographical and site factor variation of Scots pine stands. In this paper, the results for the change in the colour difference between heartwood and sapwood during UV exposure will be presented, based on the Linear Mixed Model (LMM) analyses.

BACKGROUND

Scots pine (*Pinus sylvestris* L.) wood tends to have large variations in material properties related to silviculture and growth region. Additionally, the level and variation in those properties are most probably different compared to other competing species and substituting non-wood materials in the main market segments. Aesthetic properties and visual impression affect people's choices, when they are buying wooden products, such as interior materials or furniture. It is not so straightforward to measure people's preferences towards different looks of wood and to determine the properties that actually affect in decision-making processes. Two qualitative gross features are said to have importance in people's impression and valuation of wood: the overall blend of wood features and the presence or absence of divergent features that mismatch in the surface (Broman, 2000). In other words, questions about harmony, easiness to look at, and balance are of importance. One of the major factors causing imbalance and disturbance in wooden surfaces made of pine is the simultaneous presence of sapwood and heartwood, due to their quite large natural colour difference. In addition, the difference tends to increase with time.

Wood is affected by direct or indirect solar radiation in several outdoor and indoor end-uses. The chemical transformation of a compound into smaller compounds caused by the absorption of ultraviolet, visible, or infrared radiation is called photodegradation (IUPAC, 1996). In case of wooden surfaces, the amount of photodegradation can be directly expressed as, for example, changes in extractive composition, or indirectly by measuring, for example, the change in the colour of wood during exposure. When studying the ultraviolet-visible light-infrared part of the spectrum (wavelength approx. 10^{-9}m–10^{-3}m), it is the UV part (wavelength approx. 1–400nm) that mainly affects the colour of wood (Tolvaj and Faix, 1995 ; Müller et *al.*, 2003). The colour change caused by irradiation is correlated with the used wavelengths (Mitsui, 2004). Hon and Minemura (2001) reported that exposing wood only to UV part of irradiation intensifies the colour changes, since the colour of specimens covered with a filter that transmitted only ultraviolet light changed more than the colour of specimens without filter.

The question about the reason of colour changes of wood during UV irradiation cannot be answered in a definite way. It is said that the complex process of weathering, i.e. the change in the properties of wood especially in outdoor applications, is mostly paraphrased as photochemically initiated oxidation triggered by UV light (Tolvaj and Faix, 1995). In woods which display distinct colour change on irradiation there has been reported a link to specific extractives (Yoshimoto, 1989). The red hue of wood is commonly associated with extractive content, and correlations between redness values (a*) and the extractive contents of wood has been reported for blackbutt (*Eucalyptus pilularis*) and larch (*Larix* sp.) (Yazaki et *al.*, 1994 ; Gierlinger et *al.*, 2004). Instead, the yellowing of wood is primarily governed by the photochemistry of the essential wood components, and particularly of lignin (Yazaki et *al.*, 1994 ; Nimz, 1973). According to Hon and Glasser (1979) the substances causing the yellowing were generated by lignin and lignin derivatives, such as quinones, quinone methides, and stilbenes. Furthermore, it has been reported that the irradiation turns also cellulose yellow, which is attributed to the production of oxygen-containing groups, such as carbonyl, carboxyl, and hydroperoxide groups (Kleinert and Marraccini, 1966a, 1966b ; Hon, 1979).

The shortest wavelengths of the UV part of spectrum are being absorbed in the atmosphere, causing that only the UV-A part (approx. 320–400nm) of the entire UV radiation can reach the earth's surface. This is the part of UV radiation that affects materials in outdoor uses. In indoor conditions the effective spectrum of radiation is narrower, thus the radiation is again absorbed, now by the window glass. That part of UV radiation can be simulated, for instance, by using UVA-351 type (peak wavelength 350nm) fluorescent lamp apparatus (ASTM G154, 2000).

MATERIALS AND METHODS

A sample of 60 mature Scots pine dominated stands from mineral soils was collected in three regions in Finland (northern, south-eastern, and central inland Finland) and two regions in Sweden (south-central and southern Sweden), 12 stands from each (Fig. 1). In this paper, the regions are numbered ascending starting from the northernmost region (number 1) and ending to the southernmost region (number 5). The stands were selected randomly, and they were to represent different forest sites and age classes, as well as to cover the geographical variation of pine stands. In Finland, the sampling was based on sample plot network of the latest National Forest Inventory (NFI), in Sweden on the records of the Sveaskog Ltd. In each stand, three randomly selected Scots pine trees covering the diameter range of conventional saw log and small-diameter log trees (DBH≥14cm) were felled for sampling. 70-cm bolts were cut from the sections of butt log, middle log, and top log (at 2, 6, and 10m heights, respectively) from each sample tree. The bolts were sawn through-and-through into approx. 30-mm thick boards, and the boards were slowly dried at room temperature. The boards were numbered ascending from the pith outwards, so that the 0-board was the middle, pith enclosed board, the 1-boards were the first boards outwards from both sides of the pith, etc. The inner face of the boards was planed and a total of 486 specimens with approximate dimensions of 50x100x20mm (longitudinal, tangential, radial) were produced from the 1-boards for the colour measurements. Specimens were stored in dark storage at constant conditions (+20°C, RH 65%) before test procedure and between test phases.

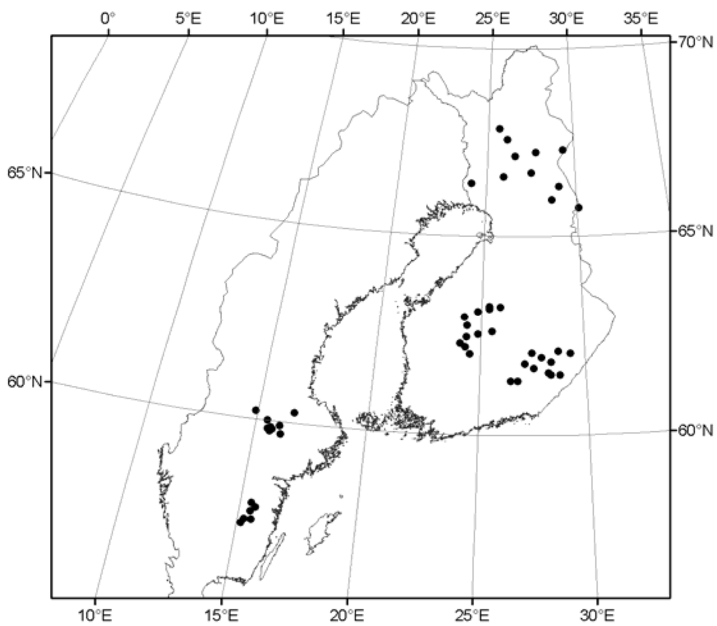

Figure 1 : Location of the sample of 60 stands in Finland and Sweden. Map: Nivala V. and Lukkarinen A., Metla.

Specimens were irradiated with UVA-351 type fluorescent lamp apparatus simulating the indoor end-use conditions. The peak wavelength of the tube was 351nm (Fig. 2), and these lamps are recommended for simulations of sunlight through window glass, since the low end cut-on of the lamps is similar to that of direct sunlight which has been filtered through window glass (ASTM G154, 2000). The tests were executed in a chamber under constant conditions with a temperature of +20°C and relative humidity of 65%. Six fluorescent tubes were positioned evenly at 1.2m height from the specimen plane. The total area of the specimen plane was approx. 6m², but the used area was outlined so that the irradiation at the position of minimum irradiation was at least 70% of the maximum irradiation on the specimen plane (EN ISO 4892-1, 1999). The measured intensities of irradiation at a wavelength of 360nm ranged between 0.366 and 0.524mW/cm² at the delimited surface plane. The specimens were randomly placed on the plane both in the beginning and in the middle of the test procedure.

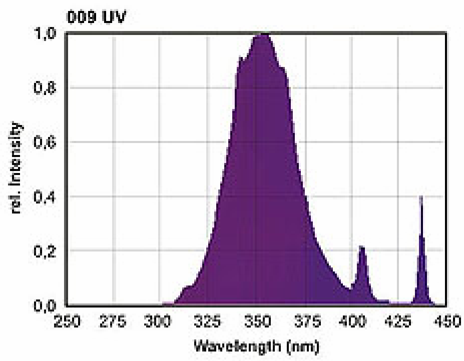

Figure 2 : Relative spectrum of the UVA-351 type fluorescent lamp (Narva LT 36W/009 UV).

For irradiation of the specimens two cyclical test procedures were used. A repeated cycle of 4h ON – 2h OFF was used for the first phase and a more intensive cycle of 5h ON – 1h OFF for the second phase of the irradiation, from t_0 to $t_{0.5}$ and from $t_{0.5}$ to t_1, respectively. Here, 0 refers to the beginning, 0.5 to the middle, and 1 to the end of the total irradiation period. Concluded from the results of the preliminary study with the above mentioned set up, a total irradiation time of 2000h was used.

In each specimen the colour of the surface was measured from four points, two points separately from sapwood and heartwood at the points of time t_0, $t_{0.5}$, and t_1. The colour was measured by spectral reflectance method with a spectrophotometer (Minolta CM-2002), using a D_{65} standard illuminant and a 2° standard observer. The specular component was included in the measurement. Each measurement of the specimens was executed exactly in the same direction. Of each measurement, a reflection spectrum in the 400–700nm region was obtained from the spectrophotometer, and the CIELAB (L^*, a^*, and b^*) colour parameters (Hunt, 1998) were computed based on the measurements. Here, L^* refers to lightness (from 0 to 100), a^* to redness (from -60 to 60), and b^* to yellowness (from -60 to 60) of the specimen. The colour coordinates of entire sapwood and heartwood area of the specimen were calculated by averaging the two measurements from each. The difference in the lightness (ΔL^*) and chroma coordinates (Δa^* and Δb^*) between sapwood and heartwood were calculated using the following formulae:

$$\Delta L^*_t = L_{st}^* - L_{ht}^*$$
$$\Delta a^*_t = a_{st}^* - a_{ht}^*$$
$$\Delta b^*_t = b_{st}^* - b_{ht}^*$$

where *s* refers to sapwood and *h* to heartwood, and *t* refers to the point of time (0, 0.5, or 1) as described earlier.

The total colour difference between sapwood and heartwood was calculated for each specimen and point of time as

$$\Delta E^*_t = \sqrt{\Delta L^{*2}_t + \Delta a^{*2}_t + \Delta b^{*2}_t}\ .$$

Variation in the colour difference between sapwood and heartwood was studied by means of linear mixed models with SPSS 13.0 for Windows software. Region, height, and the point of time were treated as fixed variables in the model, whereas tree and plot were random factors. The analysis was carried out with repeated measurements procedure. In order to model the variance-covariance structure of repeated measurements correctly, an additional variable, i.e. the combination of height and the point of time, was used as repeated factor in the model.

RESULTS AND DISCUSSION

Colour coordinates of sapwood and heartwood

The reflectance spectra of both sapwood and heartwood changed considerably during the UV irradiation (Fig. 3). In the range of visible light the reflectance decreased most between wavelengths of approx. 450 and 550nm, whereas towards the both ends of the spectrum the changes became less dramatic. Before irradiation the difference in the reflectance of sapwood and heartwood first increased till 450nm and after that decreased linearly, whereas after exposure the difference increased from 400 to 600nm and decreased with longer wavelengths. By wavelength, the difference ranged from 1.4 to 5.7%-points before irradiation and from 1.3 to 9.3%-points after irradiation, respectively. Regardless of wavelength the relative reflectance of sapwood was higher compared to one of heartwood, which indicates that the sapwood was unambiguously lighter in colour than heartwood. The variation in the relative reflection values was bigger in heartwood than in sapwood, and the variation decreased towards the both ends of the spectra of both sapwood and heartwood.

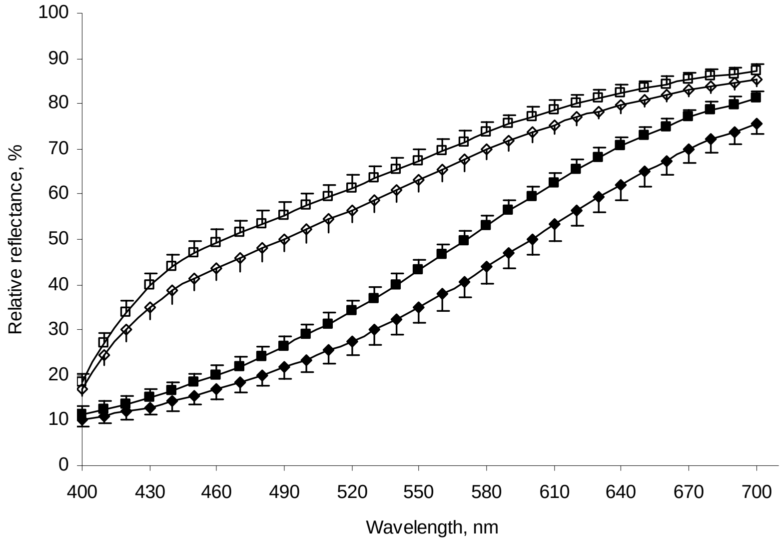

Figure 3 : Relative reflectance of sapwood (■) and heartwood (◆) in the range of visible light before (t_0, open symbols) and after (t_1, filled symbols) irradiation (average±standard deviation). For clarity the standard deviations are drawn only in one direction.

The average values of calculated colour coordinates separately for sapwood and heartwood and for each point of time are shown in Table 1. Before irradiation, the L^* of sapwood was higher and the a^* and b^* were lower compared to heartwood. Lightness of both sapwood and heartwood decreased and redness simultaneously increased during the irradiation. Instead, yellowness increased clearly in sapwood and heartwood during the first 1000h of irradiation, but decreased slightly during the latter phase of procedure. The maximum decrease in L^* was approx. 12.9 in sapwood and 16.3 in heartwood, respectively. The corresponding maximum differences in a^* and b^* were 8.4 and 18.1 in sapwood, and 9.6 and 13.4 in heartwood. As a conclusion, sapwood was lighter and less red compared to heartwood, and the differences became clearer during the exposure. Instead, the yellowness increased more in sapwood than in heartwood during the irradiation, although before UV irradiation the yellowness of sapwood was lower compared to heartwood (Table 2).

Table 1 : The colour coordinates of specimens at t_0, $t_{0.5}$, and t_1 (average±standard deviation).

	Sapwood			Heartwood		
Time	L^*	a^*	b^*	L^*	a^*	b^*
t_0	86.23±1.23	2.70±0.61	20.69±1.09	84.12±1.27	3.47±0.76	23.14±1.25
$t_{0.5}$	75.56±1.21	9.63±0.79	38.81±1.33	69.75±2.44	12.53±1.31	36.57±1.48
t_1	73.35±1.45	11.10±0.86	37.97±2.02	67.87±2.42	13.05±1.29	35.08±2.08

Despite the decrease in lightness and increase in redness of sapwood and heartwood during the test phases 1 and 2, the ΔL^* and Δa^* were highest at the time $t_{0.5}$, instead of t_1. Same phenomenon was typical also for the total colour difference ΔE^*. Only the Δb^* increased also during the test phase 2. The maximum difference between points of times was approx. 3.8 in ΔL^*, 2.2 in Δa^*, 5.4 in Δb^*, and 3.7 in ΔE^*, respectively (Table 2).

Table 2 : The differences in colour coordinates between sapwood and heartwood of the specimens at t_0, $t_{0.5}$, and t_1 (average±standard deviation).

Time	ΔL^*	Δa^*	Δb^*	ΔE^*
t_0	2.14±1.21	-0.76±0.83	-2.51±0.89	3.55±1.33
$t_{0.5}$	5.97±2.08	-2.99±1.06	2.28±1.38	7.20±2.31
t_1	5.49±2.04	-1.95±0.87	2.91±1.54	6.70±2.21

Mixed model analysis

In order to homogenize the variance of dependent variable, i.e. the total colour difference ΔE^*, a square root conversion was calculated. *Region*, *height* in the tree, and the point of *time* were set as fixed factors in the model. Also the interactions between region and point of time (*region*time*), as well as height and point of time (*height*time*) were included. *Stand* and *tree* were treated as random factors. With a given point of time, there might be some correlations in the residuals within individual trees between different heights, but no correlations between trees. In addition, the correlations of residuals might be different depending on the point of time. To take these correlations into account, the measurements were treated as repeated. To be able to model the variance-covariance structure of repeated measurements correctly the combination of *time* and *height* (*time_height*) was set up as repeated factor with a subject of *tree*stand*region* (Table 3). Both variables, *time* and *height,* had three levels, which caused that the combination variable had a total of nine levels. To ensure the fit of the model, the residuals were examined as a function of predicted values, and also the normal distribution of residuals was checked.

Table 3 : Structure of the built mixed model. Dependent variable is *sqrt(ΔE*)*.

		Number of levels	Covariance structure	Number of parameters	Subject variables	Number of subjects
Fixed effects	Intercept	1		1		
	Region	5		4		
	Height	3		2		
	Time	3		2		
	*Region*Time*	15		8		
	*Height*Time*	9		4		
Random effects	*Stand(Region)*	60	Variance components	1		
	Tree(Stand(Region))	175	Variance components	1		
Repeated effects	*Time_Height*	9	Unstructured	45	*Tree*Stand* Region*	175
Total		280		68		

With the dependent variable *sqrt(ΔE*)*, the effect of *height* was statistically insignificant. Other fixed factors, i.e. *region* and *time*, as well as the both interactions were statistically significant. Despite the insignificance of *height* the parameter was kept in the model because of the significant interaction term with *height* included (Table 4).

Table 4 : Tests of fixed effects. Df=degrees of freedom. Dependent variable is *sqrt(ΔE*)*.

	Numerator df	Denominator df	F	Sig.
Intercept	1	78.161	7999.657	0.000
Region	4	74.614	4.291	0.004
Height	2	144.853	1.789	0.171
Time	2	167.456	995.035	0.000
*Region*Time*	8	165.604	7.935	0.000
*Height*Time*	4	149.232	19.676	0.000

The square root of colour difference was highest in the region 5 and lowest in the region 2. By height, the colour difference was lowest in the section of middle logs (height 6m), and increased both in butt logs (height 2m) and top logs (height 10m). The biggest colour differences were measured at the middle of the total irradiation period ($t_{0.5}$), where the average difference was approx. 0.87 units bigger compared to the t_0. The difference decreased approx. 0.1 units between $t_{0.5}$ and t_1 (Table 5). The variance component of random effect *stand* was statistically significant (p=0.039), whereas the *tree*-level random effect was insignificant (p=1.000). The individual variance and covariance parameters of repeated effect *time_height* were not significant at 0.05 level.

Table 5 : Estimates of fixed effects. S.E.=standard error, df=degrees of freedom. *Reg*=Region (1=northern Finland, 2=inland Finland, 3=south-eastern Finland, 4=south-central Sweden, 5=southern Sweden), *Hgt*=Height (2, 6, 10m), *Time*=point of time (1=t_0, 2=$t_{0.5}$, 3=t_1). Dependent variable is *sqrt(ΔE*)*.

Parameter	Estimate	S.E.	df	Parameter	Estimate	S.E.	df
Intercept	2.707***	0.067	115.082	[*Reg*=1]*[*Time*=1]	0.427***	0.066	178.569
[*Reg*=1]	-0.192*	0.093	107.267	[*Reg*=1]*[*Time*=2]	0.039**	0.012	184.563
[*Reg*=2]	-0.368***	0.091	100.612	[*Reg*=2]*[*Time*=1]	0.289***	0.063	164.404
[*Reg*=3]	-0.289**	0.090	96.512	[*Reg*=2]*[*Time*=2]	-0.009	0.012	169.096
[*Reg*=4]	-0.139	0.089	94.223	[*Reg*=3]*[*Time*=1]	0.205***	0.062	151.898
[*Hgt*=2]	0.110**	0.036	145.531	[*Reg*=3]*[*Time*=2]	-0.015	0.011	150.714
[*Hgt*=6]	-0.017	0.034	141.458	[*Reg*=4]*[*Time*=1]	0.147*	0.061	153.275
[*Time*=1]	-0.777***	0.046	180.331	[*Reg*=4]*[*Time*=2]	0.007	0.011	149.826
[*Time*=2]	0.096***	0.010	207.703	[*Hgt*=2]*[*Time*=1]	-0.285***	0.033	149.794
				[*Hgt*=2]*[*Time*=2]	-0.014	0.009	157.144
				[*Hgt*=6]*[*Time*=1]	-0.055*	0.026	139.049
				[*Hgt*=6]*[*Time*=2]	0.001	0.009	146.635
[*Reg*=5], [*Hgt*=10], and [*Time*=3] are set to zero.				* p≤0.05, ** p≤0.01, *** p≤0.001			

Based on the pair wise comparisons, only region 2 (inland Finland) and region 5 (southern Sweden) differed from each other significantly, when the effect of *time* was ignored. In region 2, the average colour difference was 1.284 units smaller compared to region 5. At t_0 the northern Finland (region 1) differed significantly from all other regions, whereas at $t_{0.5}$ and t_1 the only significant differences were found between regions 5 (southern Sweden) and 2 (inland Finland), and regions 5 and 3 (south-eastern Finland). At t_0 the average colour differences varied from 3.290 to 4.437, at $t_{0.5}$ from 6.438 to 8.153, and at t_1 from 6.027 to 7.651, respectively (Fig. 4).

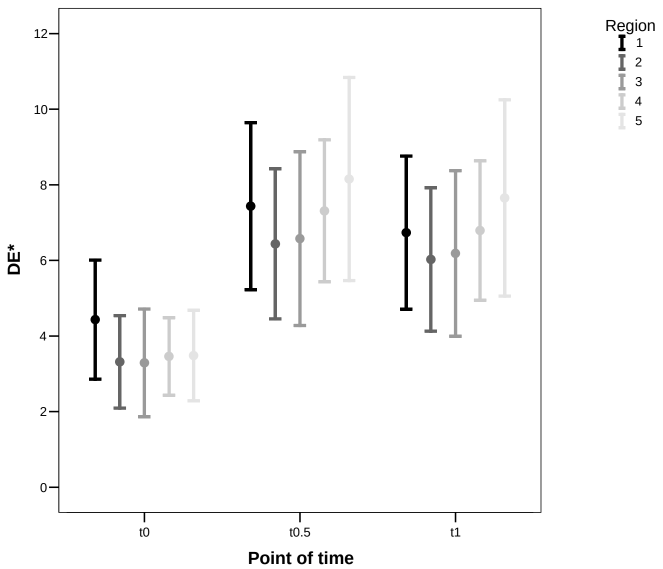

Figure 4 : The colour difference between sapwood and heartwood (*ΔE**) in different regions at t_0, $t_{0.5}$, and t_1 (average±standard deviation).

At t_0 the pair wise differences between heights were all significant, but at $t_{0.5}$ and t_1 only the 2m height differed significantly from other heights, i.e. the difference between 6m and 10m was not significant at 0.05 level. The differences between points of time were significant regardless of height and region. In each region and height the greatest colour differences between sapwood and heartwood were found at time $t_{0.5}$. In region 2 (inland Finland) the differences in ΔE^* between heights 2m and 6m, and 2m and 10m at $t_{0.5}$ and t_1 were considerably bigger compared to the other regions, even though the differences were not so clear at t_0. Before irradiation ΔE^* increased towards the top of the tree. After irradiation (at $t_{0.5}$ and t_1) the ΔE^* values decreased from 2m to 6m and 10m in all regions except region 1 (northern Finland), where the colour differences between sapwood and heartwood were almost constant between different heights (Fig. 5).

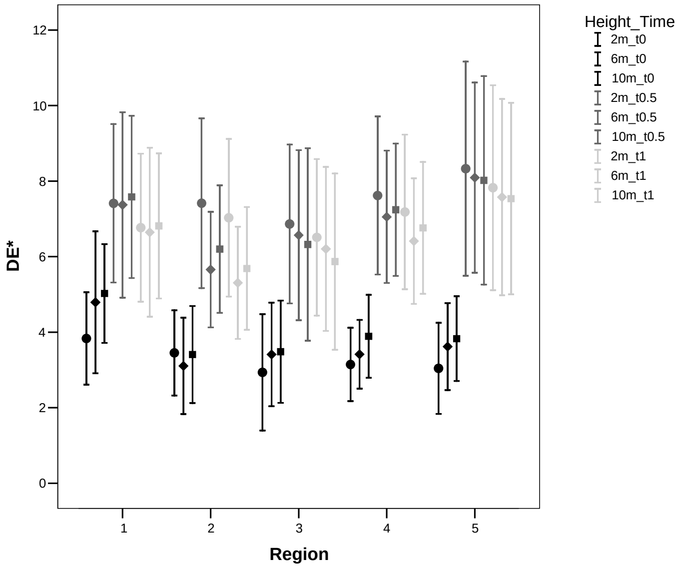

Figure 5 : The colour difference between sapwood and heartwood *(ΔE^*)* in different regions by height and time (average±standard deviation). Black=time t_0, dark grey=time $t_{0.5}$, light grey=time t_1; circles=height 2m, diamonds=height 6m, squares=height 10m.

CONCLUSIONS

UV-A irradiation caused remarkable colour changes both in sapwood and heartwood of Scots pine during 2000h exposure period. The relative reflectance of wood decreased in the visible light range of spectrum, and the decrease was dependent on the wavelength. A remarkable increase in redness and yellowness was found both in sapwood and heartwood as a consequence of UV radiation. Before and after test period the redness of wood was higher in heartwood than in sapwood, whereas the yellowness was first higher in heartwood, but during the irradiation the sapwood turned into more yellow compared to heartwood.

The total colour difference between sapwood and heartwood increased in the first test phase (from t_0 to $t_{0.5}$), but decreased during the second phase (from $t_{0.5}$ to t_1). Between phases the specimens were stored in a dark storage under constant temperature and relative humidity. The storage time was approx. 2–3 months. The colour was measured only in the end of the first phase, and next measurement was after the second phase. The colour may have changed during the storage period, which may explain at least a part of the exceptional behaviour of colour coordinates between the first and the second phase.

At t_0 the northern Finland (region 1) differed clearly from other regions. In fact, the colour differences were almost constant between regions 2 to 5 before irradiation. During and after the irradiation ΔE^* values were biggest in the southernmost region and lowest in inland Finland and south-eastern Finland, i.e. regions 2 and 3. In other words, during the service time the heterogeneity of wood material especially from southern regions tends to increase more compared to material from more northern regions. This may affect the appearance of floorings, panelling, and pieces of furniture made of Scots pine wood.

In the future, it will be attempted to improve the built mixed model by finding more background variables, such as forest site type and geographical coordinates, which affected the observed colour differences. Also the individual colour coordinates and their changes will be modelled as a function of selected explanatory variables.

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