DISCOLOURATION OF THERMALLY MODIFIED WOOD DURING SIMULATED INDOOR SUNLIGHT EXPOSURE

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This paper deals with the effect of UV light through window glass on the changes in colour of surfaces of uncoated and clear-coated thermally modified wood, and on chemical changes of surfaces of uncoated thermally modified and unmodified wood. Discoloration of the uncoated wood samples and those treated with three commercial transparent coatings (two-component polyurethane varnish, water-borne varnish, and nano-impregnation) was measured spectrophotometrically using CIELAB parameters (L^*, a^*, b^* and ΔE^*). FTIR spectroscopy was used to study chemical changes caused by UV irradiation. Colour change (ΔE^*) was recorded in all tested wood samples after exposure to UV light, and the smallest discoloration was recorded in wood samples coated with two-component polyurethane varnish. FTIR spectroscopy results show that thermal treatment and exposure to UV light modified the chemical structure of wood surface and that the thermally modified samples exposed to UV light showed similar changes as unmodified samples exposed to UV light, but less pronounced.

Keywords: Photodegradation; QUV test; FTIR; Transparent coatings; Thermally modified wood

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INTRODUCTION

Colour is a very important property in the use of wood for decorative applications, because of its aesthetic value. Wood colour is a composite property, the value of which is related to its basic chemical composition (Hon et al. 1985). The colour of wood is sensitive to light radiation and thermal treatment (Mitsui et al. 1995; Mitsui and Tsuchikawa 2005; Tolvaj and Mitsui 2005).

Thermal treatment of wood has been known for long as an effective method to modify the properties of wood (Kollman and Fengel 1965). Sometimes it is used to change the aesthetic properties of wood (Johansson and Morén 2006). A thermal treatment always results in darkening of wood, which is often attributed to the formation of coloured degradation products from hemicelluloses and extractive compounds (Sundqvist and Morén 2002). The formation of oxidation products such as quinones is also cited as a reason for darker wood colour (Tjeerdsma et al. 1998; Bekhta and Niemz 2003).

Thermally treated wood is often appreciated for its light brown to dark brown colour and is used in Croatia for parquet floorings as a substitute for some tropical hardwoods. Better dimensional stability in variable room conditions is an additional reason for the use of this new material for parquet production (Živković et al. 2008).

Unfortunately, it has been established that the brownish colour of thermally treated wood is not stable against light exposure (Jämsa et al. 2000; Ayadi et al. 2003; Deka et al. 2008).

Wood floorings are usually protected by clear coatings, which ideally would help to retain the natural appearance of wood. These coatings are sensitive to UV radiation and in addition ultraviolet (UV) light can penetrate through them to initiate photochemical reactions in the underlying wood substrate, resulting in discoloration. The discoloration of wood during light exposure is not only an aesthetical drawback, but also a good indicator of early degradation in most applications (Schaller and Rogez 2008). The extent of wood discolouration depends on incident light energy, which is more intensive in the ultraviolet spectrum (UV) than at longer wavelengths. Furthermore, it depends on photochemical stability of lignin and extractives, which are different for different wood species, on the content of oxygen or ozone in the air, and also on wood temperature and humidity. The spectral characteristics of light to which wood is exposed are particularly important. Wood discolours under outdoor conditions much faster and more intensively than indoors, because part of the UV spectrum responsible for primary photooxidation processes is absorbed by glass and therefore does not affect the wood indoors.

Unfortunately, the problem of discolouration of clear coated wood indoors is given consideration only upon complaints. Most commercial products used indoors do not contain UV absorbers or if they do their quantity or absorption range is inadequate. Recently, new products have come to the market and they are advertised to protect the natural colour of wood in interior use and to prevent greying, yellowing, and darkening of wood surfaces. There is no scientific information on the efficacy of these products.

The objective of this study was to establish the discolouration of uncoated and clear coated thermally modified wood (predominantly used for parquets) during accelerated lightfastness testing. We also studied the chemical changes in uncoated thermally modified wood and unmodified wood after UV irradiation.

EXPERIMENTAL

Materials

This study examined three wood species: beech (*Fagus silvatica* L.), ash (*Fraxinus excelsior* L.), and hornbeam (*Carpinus betulus* L.). Wood samples were thermally modified with water, steam, and high temperature using the Thermo Wood process (www.thermowood.fi). Thermally modified wood samples were obtained from a local parquet manufacturer. The dimensions of the samples were 150mm x 70mm x 18 mm (axial x radial x tangential). Beech and ash wood were represented by three types of samples: thermally modified samples at 190 °C (marks: B190, A190)) and 212 °C (marks: B212, A212) and unmodified samples (marks: BU, AU). Likewise, hornbeam wood was represented by two types of samples: thermally modified samples at 212 °C (mark H212) and unmodified samples (mark HU). The unmodified samples were exposed uncoated, and thermally modified samples were exposed coated and uncoated.

The samples were coated with commercial wood finishes: water-borne varnish with a solid content of 32%, 2k solvent-borne polyurethane varnish (PU) with a solid content of 38%, and water-borne nano-impregnation for UV light protection. Four coating systems were used: PU varnish (three layers, mark PU), PU varnish with impregnation (one layer of impregnation and two layers of varnish, mark PUI), water-borne varnish (three layers, mark AQ), and water-born varnish with impregnation (one layer of impregnation and two layers of varnish, mark AQI). All finishes were applied on wood manually by brush with a 24 hour drying time between layers. The amount of applied layer was 120 g/m^2 for varnishes, and 100 g/m^2 for impregnation.

Accelerated Lightfastness Test

The accelerated indoor sunlight exposure test of specimens was conducted in a QUV lightfastness tester (Q-Panel Company) equipped with UVA-351 fluorescent lamps. The UVA-351 fluorescent lamp is recommended for simulating sunlight filtered through window glass (Fig. 1). Three panels of each type of samples were exposed to UV light directly at the distance of 5 cm for 32 days (768 hours), and the fourth was the unexposed reference sample. The temperature of the black panel was 60 ±2 °C, and irradiation was 0.77 Wm^{-2}nm^{-1}.

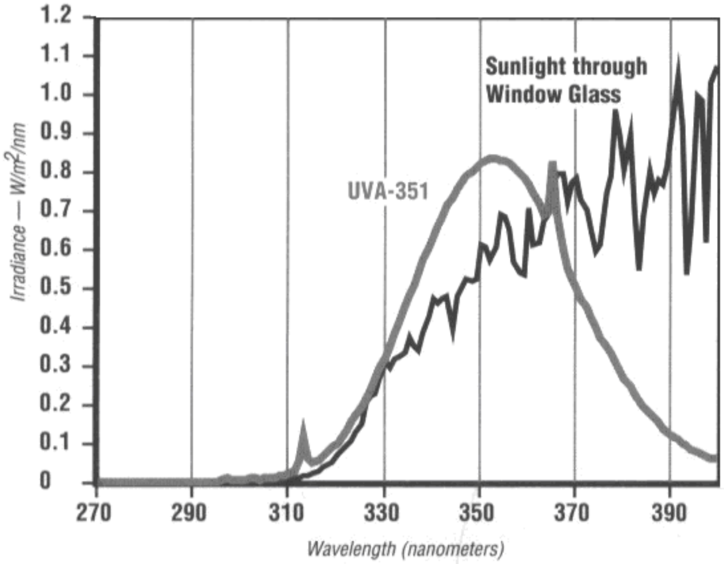

Fig. 1. UVA-351 fluorescent lamp compared to sunlight through ordinary window glass (Q-Lab)

Colour Measurements

Initial colour measurements were performed prior to any exposure to UV light irradiation. Intermediary colour measurements were performed after 1, 2, 4, 8, 16, and 32 days of light irradiation. The measurements were made with a portable spectrophotometer Microflash 100d produced by Datacolor (d/8° measuring geometry, 10° standard observer, D65 standard illuminate, xenon flash lamp source) always on the same six marked locations, and the arithmetic mean of 18 measurements was calculated for each group of samples. The overall colour change ΔE* was measured using the CIE L*a*b* colour measuring system, where L* describes the lightness, a* and b* describe the chromatic coordinates on the green-red and blue-yellow axis. ΔE* is the colour difference between the initial colour of the sample and the colour of the sample after exposure, and it is calculated as follows,

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \qquad (1)$$

where ΔL*, Δa*, and Δb* are the differences between coordinate values of exposed and unexposed area of L*, a* and b*, respectively. A low ΔE* corresponds to a low colour difference. Note that ΔE* shows the extent of colour difference but not the direction (Sehistedt-Persson 2003).

FTIR Spectroscopic Analysis

Chemical changes on unmodified and modified wood surfaces were analysed by FTIR spectroscopy. The sample for analysis was obtained by removing wood surfaces using a razor blade and screening of specific dimensions of 0.125 mm. The FTIR spectra of samples were obtained by direct transmission (KBr pellet technique) using a Shimadzu 8400S spectrometer at a wavenumber range of 4000 to 450 cm^{-1}. Each spectrum was taken as a mean value of 32 scans at a resolution of 4 cm^{-1}. KBr pellets for FTIR spectroscopy were prepared using a macro technique. Dry (120 °C in the oven for at least 1 h, followed by cooling in a desiccator over P_2O_5) and spectroscopy-grade potassium bromide (KBr) was used as a suitable alkali halide for the preparation of samples, as well as for recording standard curves. A standard Ø13 mm diameter pellet was prepared by pressing a 10 mg sample in 300 mg of KBr for 5 min under pressure of 200 bar. The obtained FTIR spectra were further processed using computer software Spectrum One (ver. 5.0.1).

RESULTS AND DISCUSSION

Colour Changes

Colour changes (ΔE*) of irradiated unmodified and thermally modified, coated and uncoated wood samples are presented in Figs. 2 and 4. The colour data of wood samples before UV light irradiation are presented in Table 1. In Fig. 2 can be seen that at the beginning of the exposure the absolute colour change increased in unmodified samples, whereas with increasing time of exposure to UV light, the absolute colour change (ΔE*) decreased. The change of parameters L*, a* and b* during the UV exposure contribute to the change of ΔE*. As can be seen in Fig. 3, the lightness (L*) decreased in the first four days of exposure and then slightly increased for beech wood samples or reached a near constant value for ash and hornbeam wood. The coordinate a* increased in the first days of exposure for ash and hornbeam wood and then maintained a nearly constant value. The coordinate a* increased in beech wood in the first four days of UV exposure and then started to decrease slightly. The coordinate b* increased in the first four days of exposure much more rapidly than a* and then started to decrease slightly with irradiation time. The increased colour change (ΔE*) during the initial period of UV exposure is a result of chromophores formation involving breakdown products from the photo-oxidation of lignin (Sharratt et al. 2009). Pandey (2005) reported that the colour changes of wood during light irradiation were correlated with lignin decay and formation of carbonyl groups generated during photodegradation of wood. The role of extractives in photodegradation reactions is not well defined. They could act as antioxidants and provide some level of protection to wood against photodegradation (Nzokou and Kamdem 2006).

Table 1. Colour Data (L*, a* b*) Before UV Light Irradiation

Type of sample	Coating system	Colour data*		
		L*	a*	b*
Ash				
unmodified	--	81.7 (1.4)	5.3 (0.1)	20.3 (1.9)
thermally modified at 190 °C	--	54.7 (1.2)	11.5 (0.5)	26,3 (1.0)
	PU	48.6 (1.7)	15.8 (1.1)	28.4 (1.4)
	PU + impregnation	49.6 (1.4)	17.0 (0.1)	28.0 (1.0)
	AQ	55.2 (1.1)	12.8 (0.2)	28,9 (0,3)
	AQ + impregnation	56.8 (1.4)	13,0 (0.3)	29,5 (1,2)
thermally modified at 212 °C	--	38,9 (1.2)	10.2 (0.5)	17.8 (0.8)
	PU	28.2 (1.2)	5.0 (0.7)	2.4 (0.4)
	PU + impregnation	29.8 (1.5)	7.9 (0.7)	5.4 (0.6)
	AQ	35.4 (1,7)	10,3 (0,7)	13.5 (1.1)
	AQ + impregnation	32.7 (1.9)	8.6 (0.4)	9.8 (0.9)
Beech				
unmodified	-	73.9 (1.0)	8.7 (0.5)	18.4 (0.4)
thermally modified at 190 °C	-	55.9 (1.5)	10.2 (0.4)	23.2 (1.1)
	PU	44.7 (0.7)	14.4 0.4)	22.7 (0.6)
	PU + impregnation	41.6 (1.3)	14.8 (0.4)	18.9 (1.0)
	AQ	46.3 (1.0)	11.9 (0.3)	22.8 (1.1)
	AQ + impregnation	45.9 (1.4)	11.2 (0.4)	21.2 (0.9)
thermally modified at 212 °C	-	43.4 (1.8)	10.6 (0.9)	19.2 (1.1)
	PU	31.0 (1.7)	8.7 (0.7)	6.6 (1.3)
	PU + impregnation	29.8 (1.8)	7.2 (1.0)	5.0 (0.6)
	AQ	33.0 (1.4)	8.3 (0.4)	10.0 (1.6)
	AQ + impregnation	32.2 (0.9)	8.5 (0.7)	9.1 (0.8)
Hornbeam				
unmodified	-	80.2 (0.8)	3.3 (0.2)	18.9 (1.7)
thermally modified at 212 °C	-	37.5 (1.2)	9.2 (0.6)	14.8 (0.4)
	PU	27.5 (1.0)	3.3 (0.5)	1.1 (0.6)
	PU + impregnation	27.9 (0.6)	4.9 (0.4)	2.5 (0.7)
	AQ	32.7 (0.3)	8.2 (0.2)	10.0 (0.6)
	AQ + impregnation	33.0 (0.3)	8.6 (0.2)	10.4 (0.2)

* Values in parenthesis are standard deviations

Beech wood samples showed the best lightfastness of unmodified samples during exposure to UV light, while colour changes in ash and hornbeam unmodified wood samples were similar, which can be related to type and amount of extractives (Fig. 2). Higher colour changes could also be noted in unmodified wood samples during the first eight days of exposure to UV light, compared to thermally modified samples, which is in agreement with the research of Ayadi et al. (2003).

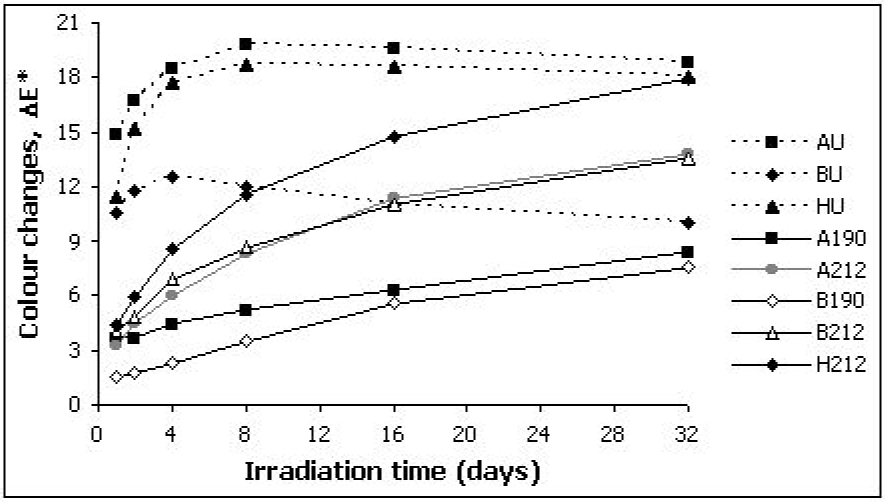

Fig. 2. Colour changes ΔE* of uncoated thermally modified and unmodified wood samples

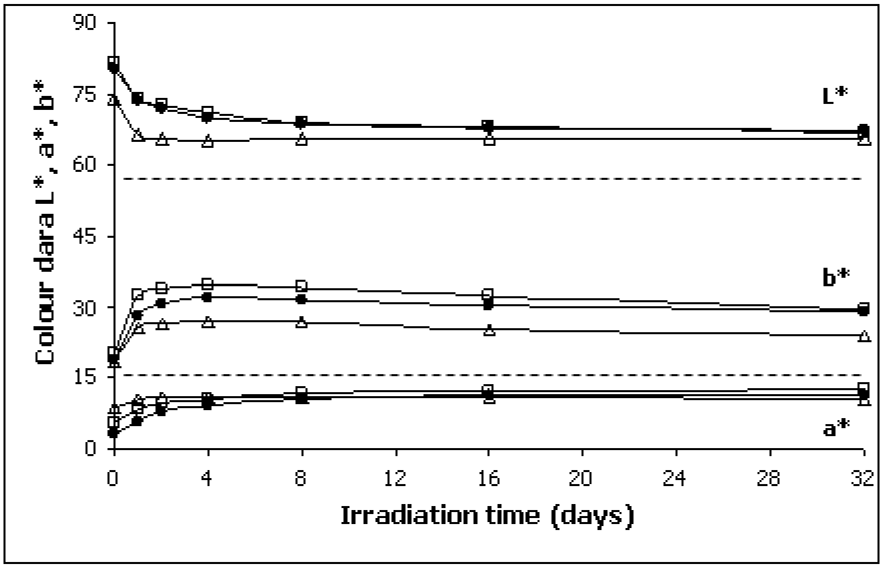

Fig. 3. Change of colour data L*, a*, and b* in unmodified ash (□), beech (Δ), and hornbeam (●) wood samples during UV exposure

The absolute colour change (ΔE*) of thermally modified samples (190 °C) coated with 2k PU varnish decreased in the first 16 days of exposure for ash wood samples and in only 8 days for beach wood samples and than began to grow. The absolute colour change of other modified samples increased during exposure to UV light. It can also be seen that the measured values of colour change (ΔE*) during the exposure to UV light were less in thermally modified samples at 190 °C compared to samples modified at 212 °C, with the exception of the samples modified at 212 °C coated with 2k PU varnish with and without impregnation (Fig. 4).

Among all the coating systems considered in this paper, the smallest colour changes during exposure to UV light were measured in samples coated only with polyurethane, and the largest in samples coated with water-borne varnish. Impregnation combined with 2kPU varnish showed decreased colour changes compared to uncoated thermally modified samples, but the colour change was lower in samples coated only with 2k PU varnish. Samples coated with a combination of impregnation and water-borne varnish showed better lightfastness than the uncoated samples and samples coated only with water-borne varnish (Fig. 4).

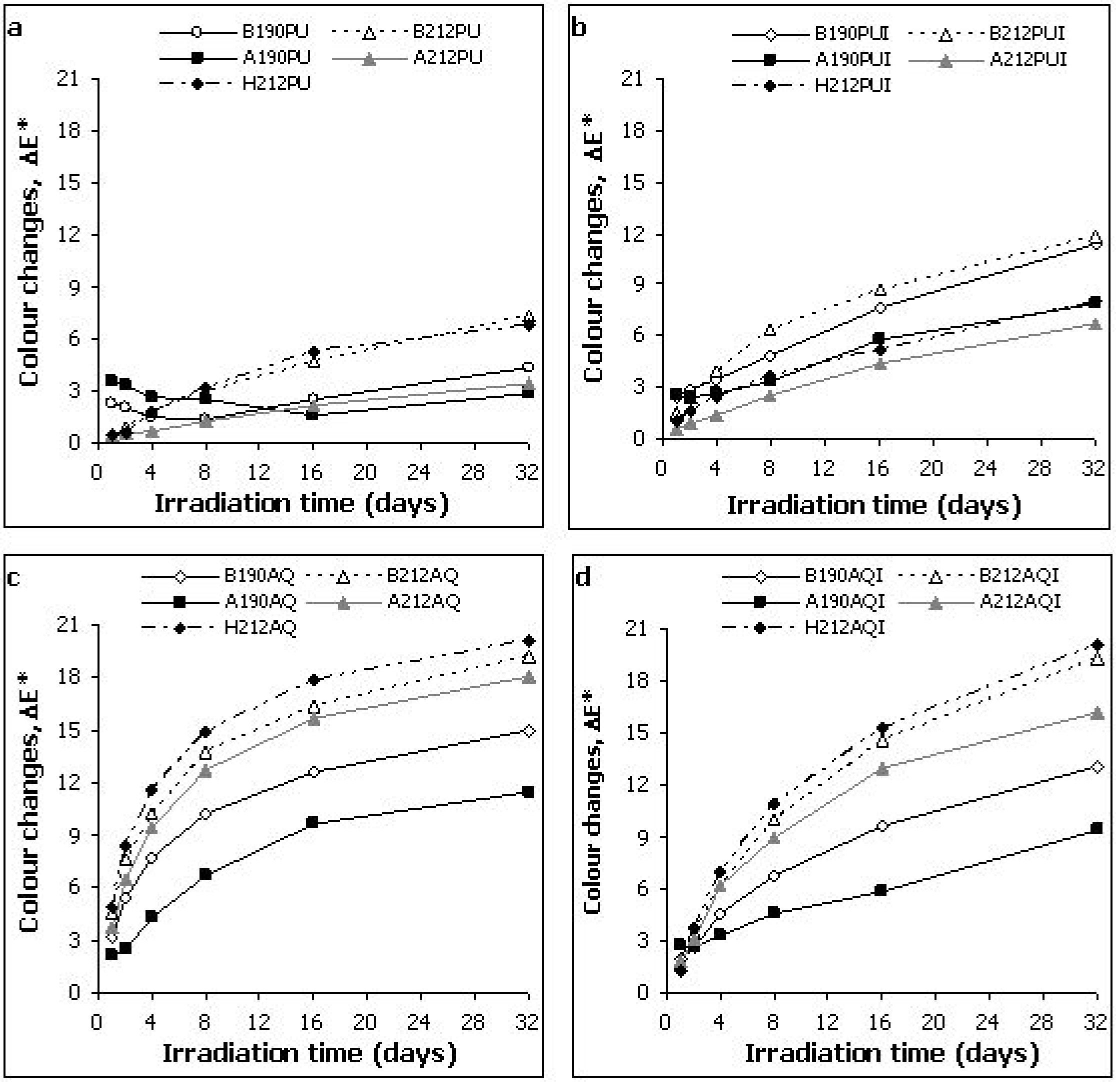

Fig. 4. Colour changes ΔE* of coated thermally modified wood samples with 2k PU varnish (a), impregnation + 2k PU varnish (b), water-borne varnish (c), and impregnation + water-borne varnish (d)

The values ΔL*, Δa*, and Δb* at the end of exposure to UV light can be seen in Fig. 5. According to ΔL* values, darkening behaviour in unmodified samples and brightening behaviour in thermally modified, coated, and uncoated samples could be observed. Ash samples, thermally modified at 190 °C and coated only with polyurethane varnish, where slightly darkening (ΔL* = -0.2) was observed, were the only exception. With respect to Δa*, the greatest changes could be seen in uncoated samples. Unmodified uncoated samples showed an increase of red hue, and thermally modified uncoated samples showed a decrease of red hue. According to Δb* values, most of the tested samples showed an increase of yellow hue. Only three uncoated thermally modified tested samples showed a decrease of yellow hue, namely: A190, B190 and B212. The impregnation in combination with polyurethane varnish increased lightening and yellow hue in the surface of wood samples after 32 days of light exposure compared to polyurethane varnish. However, the impregnation in combination with water-borne varnish increased lightening but decreased yellow hue of the sample surface compared to water-borne varnish (Fig. 5).

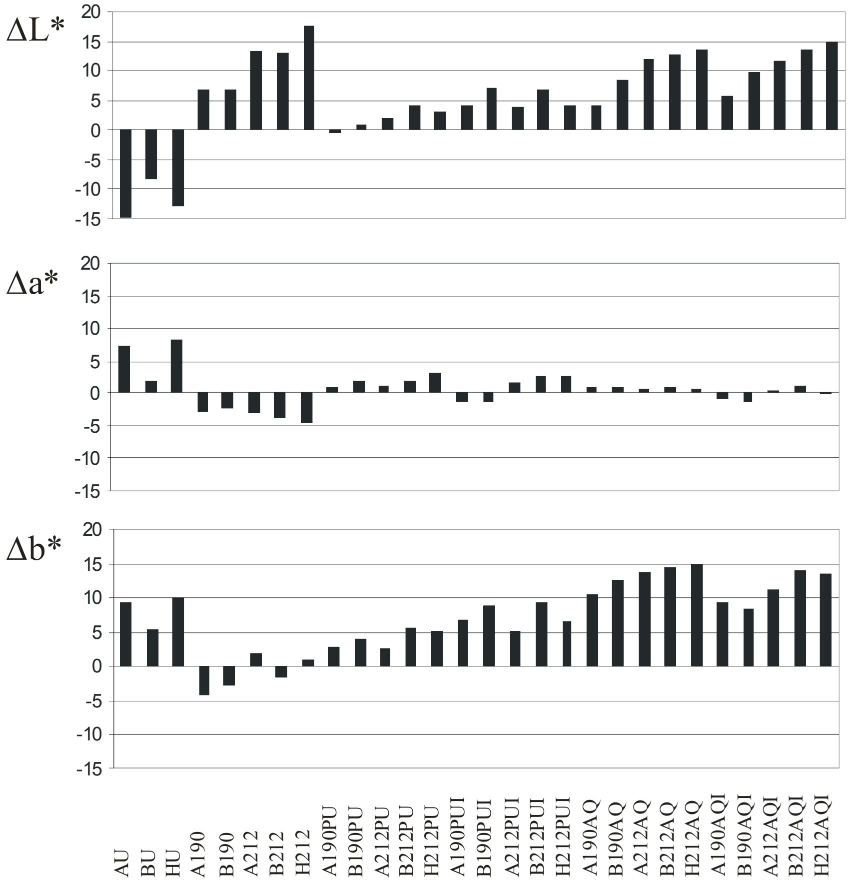

Fig. 5. ΔL*, Δa* and Δb* values after 32 days of accelerated indoor sunlight exposure

FTIR Analysis

In this research, the FTIR spectra were recorded in modified samples of different hardwood species (beech wood, hornbeam wood and ash wood). The samples of the first set were thermally modified at 190 °C and 212 °C, the samples of the second set were thermally modified and exposed to UV light, and samples of the third set were unmodified and exposed to UV light. Furthermore, FTIR spectra were recorded in unmodified samples of the same hardwoods as control (reference) samples.

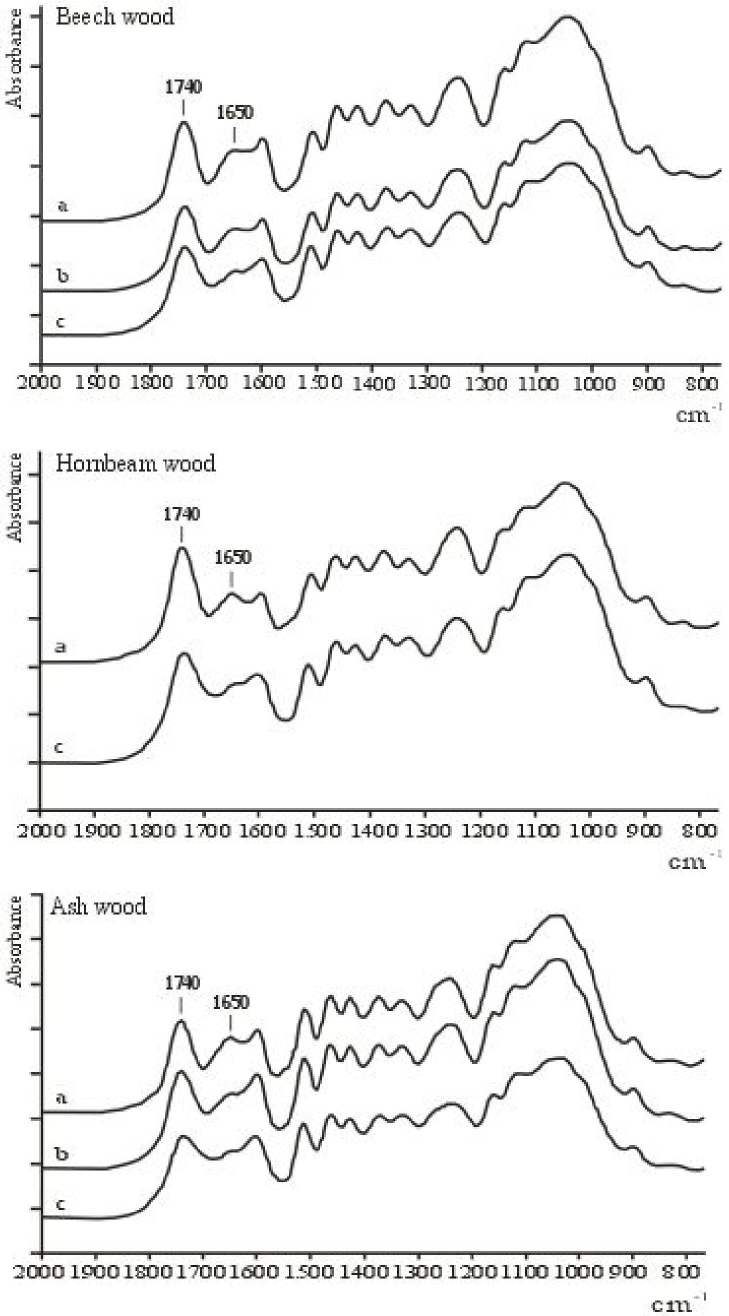

Fig. 6. FTIR spectra of unmodified (a) and thermally modified samples of different hardwood species at 190 °C (b) and 212 °C (c)

Characterization and assignment of different IR peaks of wood have been extensively studied and well documented (Pandey 1999; Faix and Bottcher 1992; Antonović *et al.* 2008). A comparison of IR spectra analyses of unmodified and modified samples of wood surfaces showed significant chemical changes between them.

FTIR spectra of unmodified and modified samples showed the same basic structure: a strong hydrogen bonded O-H stretching absorption at 3300-4000 cm^{-1} and a prominent C-H stretching absorption at 2800-3000 cm^{-1}. In the fingerprint region (750-2000 cm^{-1}) many sharp and distinctive peaks were observed, due to various functional groups contributed from wood polysaccharides (cellulose and polyoses) and lignin.

Figure 6 shows FTIR spectra of unmodified samples of beech wood, hornbeam wood and ash wood, and the same species thermally modified at 190 and 212 °C. The most significant absorption peak of the ester non-conjugated carbonyl group appears in the FTIR spectrum at 1740 cm^{-1}. The carbonyl peak was less intense in all hardwood samples thermally modified at 190 and 212 °C than in unmodified samples.

A temperature increase during thermal treatment results in deacetylation, caused by the cleavage of acetyl groups linked as an ester group to the polyoses (Carrasco and Roy 1992; Tjeerdsma and Militz 2005). However, the influence of temperature increase was even more clear in a rapidly decreasing 1650 cm^{-1} water peak (corresponding to H-O-H swinging in absorbed water) of wood treated at 190 °C, and almost complete disappearance of this peak in the spectrum of wood treated at 212 °C in all wood species. This is directly related to the decrease of the carbonyl peak mentioned above, which leads to acid catalytic hydrolysis of wood polyoses esters in the presence of water and elevated temperatures (Hakkou *et al.* 2005).

FTIR spectra of unmodified and thermally modified samples (190/212 °C) of different hardwoods exposed to UV light, as well as spectra of unmodified and unexposed samples are shown in Fig. 7. Based on the comparison with unmodified and unexposed samples, it can be clearly seen in all hardwoods that thermally modified samples exposed to UV light showed similar changes as unmodified samples exposed to UV light, but less pronounced, indicating that there was still same degradation of wood surface, which was also reported by Deka and Petrič (2008).

FTIR spectra of wood samples exposed to UV light are characterised by an increase in intensity of the carbonyl peak at 1740 cm^{-1}, a reduction in the aromatic skeletal vibration peak at 1595 cm^{-1}, and by a decrease in intensity of lignin-related peaks at 1506 cm^{-1}, 1462 cm^{-1} (CH_3 degenerate vibrations), 1425 cm^{-1} (C=C skeletal vibrations of aromatic ring), and 1245 cm^{-1} (lignin phenolic hydroxyl groups). These changes indicate delignification of the samples caused by exposure to UV light.

It could be noted that the relative decrease in lignin related peaks of thermally modified wood exposed to UV light was less in comparison to unmodified wood similarly exposed to UV light. It can also be seen in Fig. 7 that the carbonyl peak at 1740 cm^{-1} changed not only the height but the width as well during photodegradation. This means that more than one band changed, which is in agreement with the research of Tolvaj and Mitsui (2005).

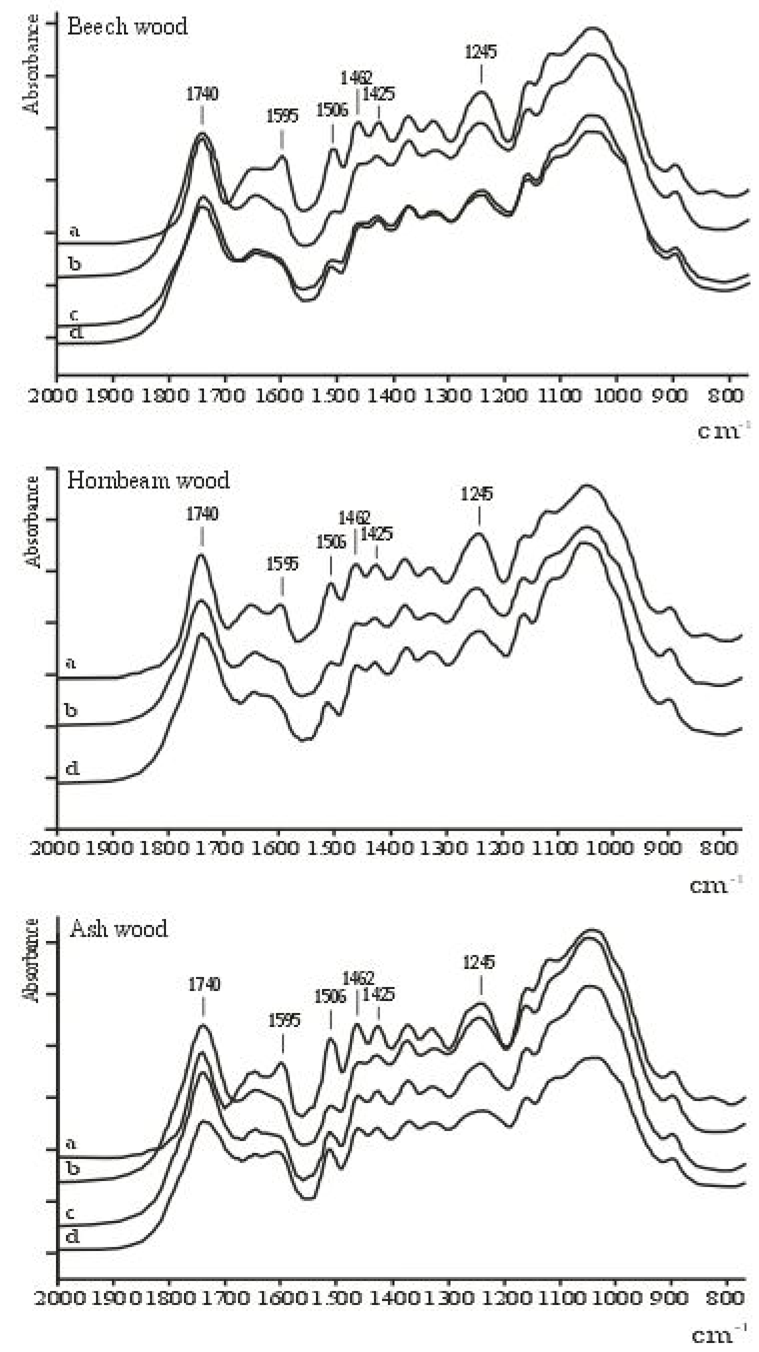

Fig. 7. FTIR spectra of different hardwoods samples: unmodified (a), unmodified and exposed to UV light (b), thermally modified at 190 °C (c) / 212 °C (d) and exposed to UV light

CONCLUSIONS

1. The measured values of colour change (ΔE*) during the exposure to UV light were less in uncoated ash, beech, and hornbeam wood samples thermally modified at 190 °C than in uncoated samples thermally modified at 212 °C.

2. In the first half of exposure to UV light, the surface of uncoated thermally modified ash, beech, and hornbeam wood samples discoloured slowly compared to uncoated unmodified wood samples.
3. Among the tested coatings, the highest lightfastness during simulated indoor exposure was recorded for 2k PU varnish. Commercial nano-impregnation in combination with 2k PU varnish did not improve lightfastness of tested wood samples, while the nano-impregnation in combination with water-borne varnish slightly decreased the colour change (ΔE*) of tested wood samples compared to wood samples coated only with water-borne varnish.
4. FTIR spectra of thermally modified ash, beech, and hornbeam wood samples exposed to UV light showed similar chemical changes as unmodified wood samples exposed to UV light, but less pronounced.

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