INFRARED SPECTRUM CHANGE OF UV IRRADIATED AND NATURAL WOOD SAMPLES DURING 12 YEARS OF STORAGE IN TOTAL DARKNESS

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ABSTRACT

The stability of the surface of UV-irradiated wood samples was investigated after 12 years of storage in total darkness at room temperature. The investigated specimens were earlywood and latewood of Scots pine sapwood, earlywood and latewood of spruce, earlywood of ash, beech and hybrid poplar. The thin (1 mm tick) samples contained only earlywood or latewood, and the tangential surfaces were used for infrared spectrum measurement. For comparison, the non-irradiated natural surface (back side) of the specimens was used for infrared spectrum measurement. Natural wood surfaces were stable during the storage. Only ether linkages in hemicelluloses showed minor degradation at 1175 cm^{-1} wavenumber. Lignin molecules remained stable during the 12-year storage period on both UV irradiated and non-irradiated side of the specimens. In contrast, UV irradiated samples suffered alterations during the 12 years of thermal treatment at low temperature (20-25°C). Hemicelluloses in photodegraded surface layers underwent thermal degradation and oxidation processes, generating new carbonyl groups. Extractives also presented absorption increase in the conjugated carbonyl region.

KEYWORDS: Hemicelluloses, extractives, photodegradation, thermal degradation.

INTRODUCTION

Photodegradation of wood species is a widely investigated phenomenon. The main factor that causes the photodegradation of wood is sunlight during outdoor exposure. The ultraviolet (UV) part causes the dominant chemical degradations (Tolvaj and Mitsui 2005, Yu et al. 2021, Cirule et al. 2021). Chemical analyses reveal that the deterioration is primarily related to the decomposition of lignin (George et el. 2005, Pandey and Vuorinen 2008, Popescu et al. 2011, Liu et al. 2017, 2019). The generated free radicals react with oxygen to produce carbonyl chromophoric groups (Tolvaj and Faix 1995, Pandey 2005, Xie et al. 2005, Timar et al. 2016). Most of the extractives located in wood material are also susceptible to photodegradation.

Colour change is the most sensitive indicator revealing the chemical changes in wood. The intensity of the colour change is high from the beginning of the light irradiation, and decreases over time (Tolvaj and Faix 1995, Pastore et al. 2004, Pandey 2005, Wang and Ren 2008, Sharratt et al. 2009, Cogulet et al. 2016). Intensive and continuous yellowing can be observed during photodegradation, along with moderate shift towards red (Oltean et al. 2008, 2010, Sharratt et al. 2009, Miklecic et al. 2011, Srinivas and Pandey 2012). Arpaci et al. (2021) found that high density species are more stable against colour changes during natural weathering than low density species.

Thermal degradation properties of wood material are also intensively studied, especially above 160°C. Steaming as hydrothermal treatment between 80-120°C has mainly industrial interest. This method modifies the unattractive and highly inhomogeneous colour of some wood species. Thermal degradation of wood below 70°C is an unstudied phenomenon.

Temperature dependence of chemical changes during thermal treatment is exponential following the Arrhenius law (Preklet et al. 2023). It means that the change is rapid if the temperature is high (above 150°C). One hour of treatment is enough to generate visible changes at these temperatures. In contrast, a one-year thermal treatment causes hardly any visible colour change in the wood if the temperature is below 40°C. Matsuo et al. (2010, 2014) studied the time-temperature superposition during thermal degradation of wood in the temperature range 90-180°C. These results and the Arrhenius equation indicates that there will always be some reaction at any temperatures, even if the temperature is low. Slow changes become observable if the experimental period is long enough. Matsuo et al. (2011) found that the colour alteration of a 921 years old historical hinoki sample stored at ambient temperature is almost equivalent to that of a sample heat treated at 180°C for approximately 6.7 h. Discolouration due to this kind of ageing is supposed to be caused mainly by mild thermal oxidation reactions.

Both extractives and hemicelluloses play an important role in colour alteration during thermal treatment. Thermal degradation of extractives and hemicelluloses are usually followed by oxidation processes. Heat treatments create free radicals which can then react with oxygen forming oxidation products such as quinines (Bekhta and Niemz 2003). Chemical reactions are quite different if oxygen is present or excluded during thermal treatment.

The long-term thermal degradation properties of wood below 40°C have not yet been investigated. The main objective of this study was to discover the chemical changes of photodegraded and untreated wood species during long-term (12 years) storage in total darkness under laboratory conditions. Infrared (IR) spectroscopy is an excellent analytical tool to monitor the chemical changes during photodegradation and thermal degradation of wood. As wood is not transparent material, the diffuse reflectance technique can be used to calculate the IR absorbance spectrum. Reflectance data can be converted to absorbance data using the Kubelka-Munk (KM) equation. It is important, however, to keep in mind that most data conversions, including the KM transformation, can generate anomalies and have validity limits (Tolvaj et al. 2011).

MATERIALS AND METHODS

The studied wood samples were prepared 12 years ago to investigate the photodegradation properties of different wood species (Tolvaj et al. 2013). The investigated conifers were Scots pine (*Pinus sylvestris* L.) sapwood and spruce (*Picea abies* Mill.). Both earlywood and latewood samples were prepared. The chosen deciduous species were ash (*Fraxinus excelsior* L.), beech (*Fagus sylvatica* L.) and poplar (*Populus x euramericana cv. Pannonia*). The size of samples was 30 x 10 x 1 mm. The samples contained only earlywood or latewood in case of conifers and earlywood for ash. The 30 x 10 mm tangential surfaces were used for IR spectrum measurement. All species were represented by 2 samples and two fixed locations were used for measurements on each sample before and after treatments.

The irradiation chamber was equipped with a doubled mercury vapour lamp. The total electric power of the applied mercury lamps was 800 W and the samples were located 64 cm from the lamps. The UV radiation was 80% of the total (UV and visible) light emission (31% UVA, 24% UVB and 25% UVC). Constant 80°C was kept in the chamber with 32-38% relative air humidity. After 24 h of UV irradiation, the samples were kept in total darkness in laboratory condition (20-25°C air temperature, 30-45% air humidity). Duration of the storage was 12 years. IR spectrum measurement was made 3 times: before UV irradiation in 2011 (natural), after 24 h UV irradiation (UV24) in 2011, after 12 years of storage in 2023 (UV24+12 years and Nat+12 years). The (Nat+12 years) results were measured on the back side of the samples. This side of the samples was not exposed to UV radiation in 2011.

Diffuse reflectance infrared Fourier transform (DRIFT) spectrum of the samples was measured before and after treatments. Measurements were carried out with an IR spectrophotometer (JASCO FT/IR 6300). The resolution was 4 cm^{-1} and 64 scans were measured and averaged. The background spectrum was obtained against an aluminium plate. Two-point baseline correction was carried out at 3800 cm^{-1} and at 1900 cm^{-1}. The spectral intensities were calculated in Kubelka-Munk (K-M) units. The spectra were normalised to the band maximum around 1375 cm^{-1}. The intensity of spectra was adjusted to 1.0 by this normalisation at maximum around 1375 cm^{-1}. This C-H band of cellulose is often used as internal standard because of its high intensity, central position and strong stability. The difference spectrum was calculated by subtracting the initial IR data from the data of the treated sample. In this case, absorption increase is represented by positive band while negative band represents absorption decrease. Details of IR data manipulations and the band assignment are described in a previous work (Csanady et al. 2015).

RESULTS AND DISCUSSIONS

The exponential temperature dependence means that high temperatures generate rapid changes and low temperatures cause slow alteration. The effect of low temperatures can be visible if the investigation period is long enough. Pretreatment can also influence the intensity of changes. In this study, chemical changes on natural and UV-treated surfaces of different wood species stored for a long time (12 years) at low temperatures (room temperature) are compared. 12-year storage of natural (untreated) samples did not generate notable absorption changes (Figs. 1-7, dotted lines). Only the low-density spruce (Figs. 2 and 4) (both earlywood and latewood) and poplar (Fig. 7) presented remarkable absorption decrease at 1175 cm^{-1},

which is associated with the C-O-C asymmetric stretching in cellulose and hemicelluloses. In our case, this alteration was caused by the splitting of ether bridges of hemicelluloses. Cellulose is known to be highly stable at ambient temperatures. The main absorption bands of lignin around 1506 and 1596 cm^{-1} showed no degradation during this treatment, indicating that lignin remained intact during the treatment.

Diffuse reflectance IR spectra of wood often present anomaly in the 1000-1200 cm^{-1} wavenumber interval (Tolvaj 2023). This anomaly is visible as a broad absorption increase on the difference spectrum in this wavenumber interval. This can be observed in Figs. 1, 3, 4 and 7 in case of "Nat+12 years" spectra. The reason of this anomaly is usually that the roughness of the sample surface is different before and after treatment. The explanation of the observed anomaly is that the difference spectrum was calculated using the absorbance spectra measured on the front surface (at the beginning of the investigation) and that measured on the back surface (after 12-year storage). Consequently, the anomaly was generated by the roughness deviation of front and back surfaces. There was no anomaly observed in case of the other spectra (UV24 and UV24+12 years).

UV irradiation generated the well-known absorption changes. These results were partly published in an earlier paper of Tolvaj et al. (2013). The band intensity of aromatic ring vibrations arising from guaiacyl lignin around 1506 cm^{-1} decreased while the intensity of unconjugated carbonyl band between 1680 and 1820 cm^{-1} increased during the irradiation (Tolvaj and Faix 1995, Colom et al 2003, Pandey 2005, Pandey and Vourinen 2008, Huang et al. 2012, Cogulet et al. 2016, Bonifazi et al. 2016). The negative absorption peak at 1506 cm^{-1} is detectable together with the absorption decrease of the guaiacyl ring breathing at 1268 cm^{-1}. Absorption intensity of syringyl lignin also decreased for deciduous species around 1596 cm^{-1}. Conifers and deciduous species also showed differences in the absorption increase of unconjugated carbonyls. Deciduous species (Figs. 5-7) presented two vell separated bands, while conifers created a broad integrated absorption intensity increase. This complex band has shoulders confirming that it is an integrated band of two or three sub-bands. The only exception was the latewood of spruce generating two well separated bands (Fig. 4). This part of the spectra for poplar (Fig. 7) shows partly separated bands. This appearance of carbonyl bands of poplar is between the appearance of carbonyl bands for coniferous and deciduous species. All investigated species presented a shoulder around 1690 cm^{-1}. The left-side main sub-band represents the absorption of CO stretching for unconjugated ketones and γ lactones generated by the oxidation after the splitting of the aromatic ring. The right-side band represents the absorption of aliphatic carboxyl groups. The intensity change of the left side band correlates with the intensity of lignin degradation (Tolvaj 2023). The new results show that intensity changes of the right integrated band (around 1705 cm^{-1}) do not correlate with intensity changes of lignin bands. This band is attributed to the photodegradation of hemicelluloses (Preklet et al. 2021, Hofmann et al. 2022).

The greatest absorption decrease generated by UV irradiation was visible at 1170, around 1138 and at 1100 cm^{-1} for all investigated species. The first decrease belongs to the asymmetric stretching of ether bond in cellulose and hemicelluloses. The second decrease belongs to the symmetric stretching of ether bond, the aromatic C-H deformation, and to the glucose ring vibration. The third band belongs also to the C-O-C ether linkage. These absorption decreases indicate the ether bond splitting and the depolymerisation of cellulose and hemicelluloses.
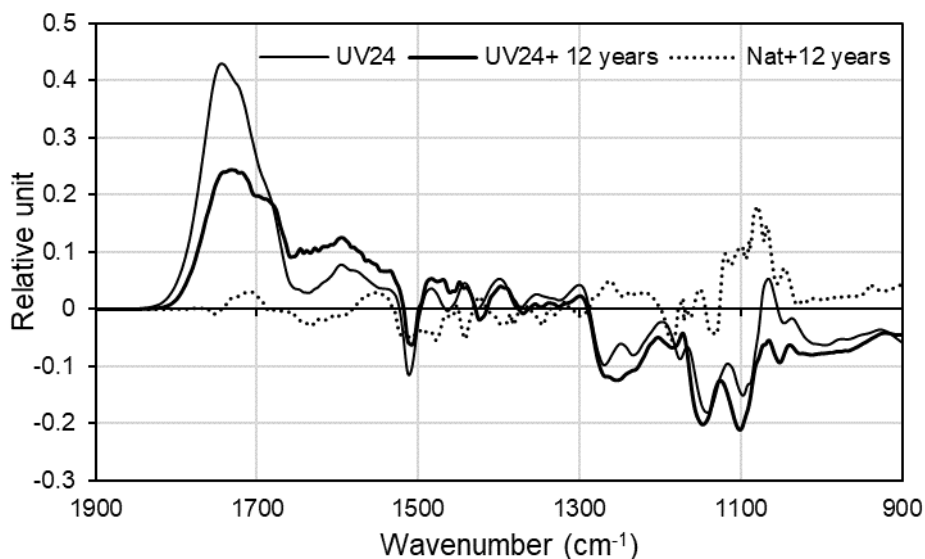
Fig. 1: Difference IR spectra of Scots pine earlywood of sapwood generated by 12-year storage in laboratory conditions in darkness (Nat+12 years), by 24 h UV irradiation (UV24) and by UV irradiation followed by 12-year storage in darkness (UV24+12 years).
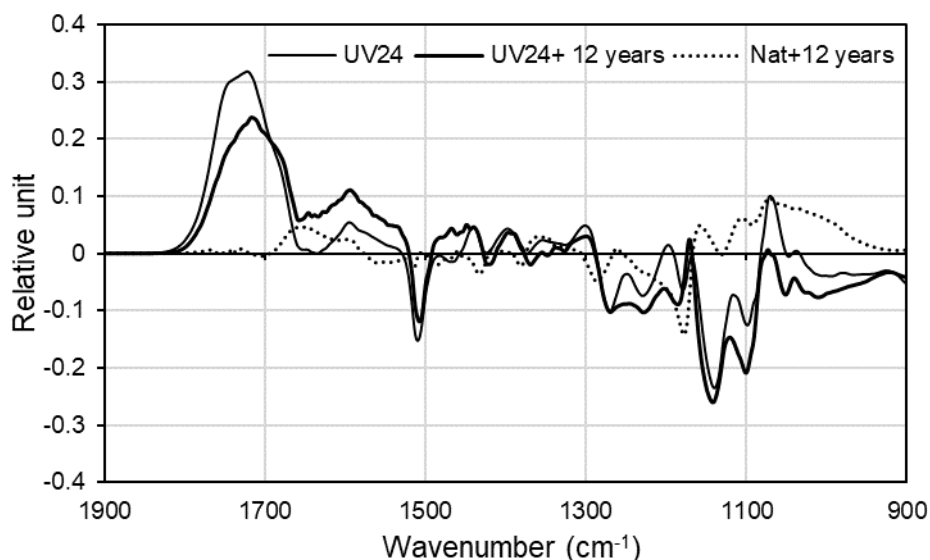

Fig. 2: Difference IR spectra of spruce earlywood generated by different treatments.

There are differences between conifers and deciduous species regarding the absorption change intensity of ether bonds. Absorption decrease at 1170 cm[-1] is hardly visible in conifers, except for spruce latewood (Fig. 4). In contrast, here is the greatest absorption decrease for ash earlywood. Absorption decrease at 1100 cm[-1] is small for deciduous species and intensive for conifers, except spruce latewood.

The 12-year storage of UV irradiated samples generated considerable and important changes. Intensity of lignin bands (around 1506 and 1596 cm[-1]) were not affected by 12-year storage confirming that there was no lignin degradation during this period. Small differences can be observed, but these were generated by baseline shift.
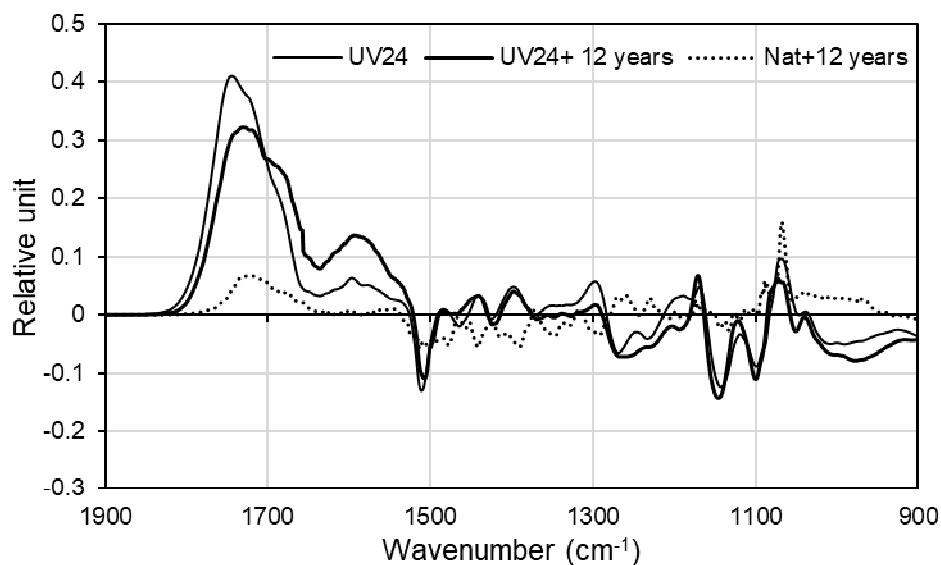
Fig. 3: Difference IR spectra of Scots pine latewood of sapwood generated by different treatments.
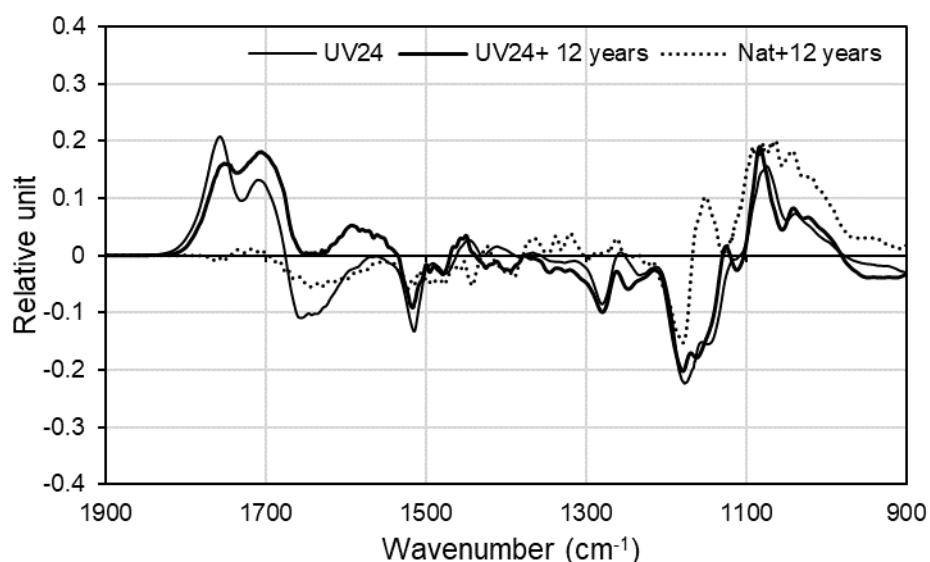

Fig. 4: Difference IR spectra of spruce latewood generated by different treatments.

The 12-year storage, however, resulted in great alterations of the broad absorption band of unconjugated carbonyls. The intensity of the left sub-band decreased for all investigated species. This decrease indicates that the carbonyl compounds formed during photodegradation were not stable enough during the long-term thermal degradation. This finding confirms previous research results demonstrating the instability of these carbonyl compounds. Rain and air humidity leached them out faster than other carbonyl compounds (Kannar et al. 2018, Bejo et al. 2019, Preklet at al. 2021, Varga et al. 2020).

Evaluation of the changes around 1706 cm[-1] is more complicated. Unconjugated carbonyl absorption band of conifers contain highly overlapped sub-bands, except spruce latewood (Figs. 1-4). As the left band showed considerable absorption decrease reducing the intensity of the whole integrated band, it is impossible to estimate the alteration of the right band.

Fortunately, spruce latewood and the investigated deciduous species present two well separated bands in the unconjugated carbonyl absorption region (Figs. 4-7).
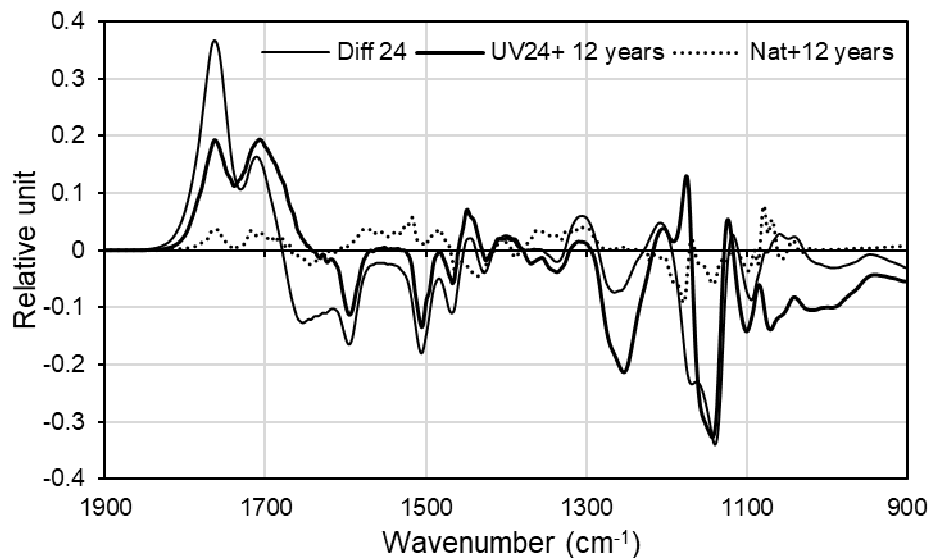

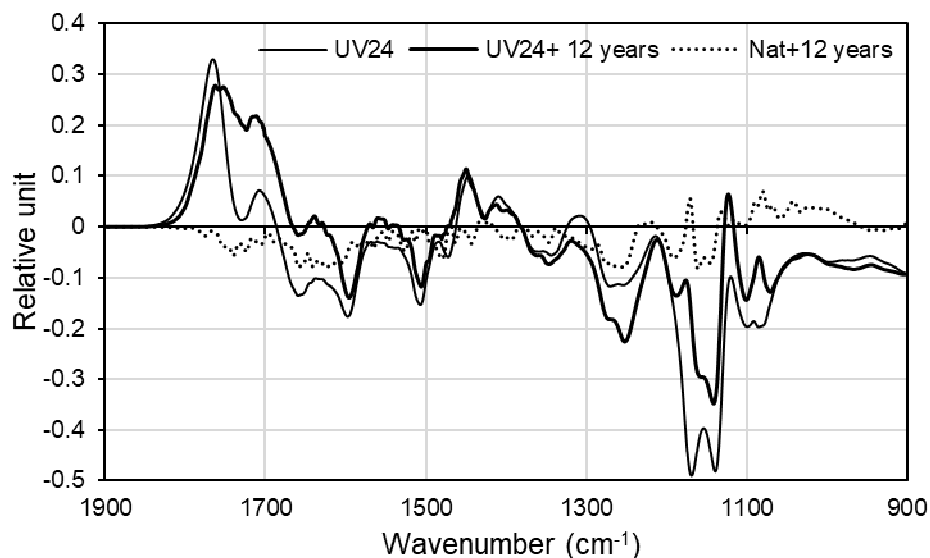*Fig. 5: Difference IR spectra of beech generated by different treatments.*

Fig. 6: Difference IR spectra of ash generated by different treatments.

These samples clearly show that the 12-year storage caused an increase in absorption in the right band. This absorption increase demonstrates that hemicelluloses underwent slow thermal degradation and the oxidation produced quinoid structures during the long storage period (Bekhta and Niemz 2003). The back side of the samples (which were not exposed to UV radiation) did not present a similar absorption increase around the wavenumber of 1706 cm^{-1} (Figs. 4-7 dotted line). Consequently, only those hemicellulose molecules generated the absorption increase around 1706 cm^{-1} which were affected and modified by the UV irradiation before the 12-year storage. All investigated conifer samples showed absorption increase in the 1540-1690 cm^{-1} wavenumber interval generated by the 12-year storage. This absorption interval belongs to the conjugated carbonyl groups, these systems are responsible for the colour of wood.
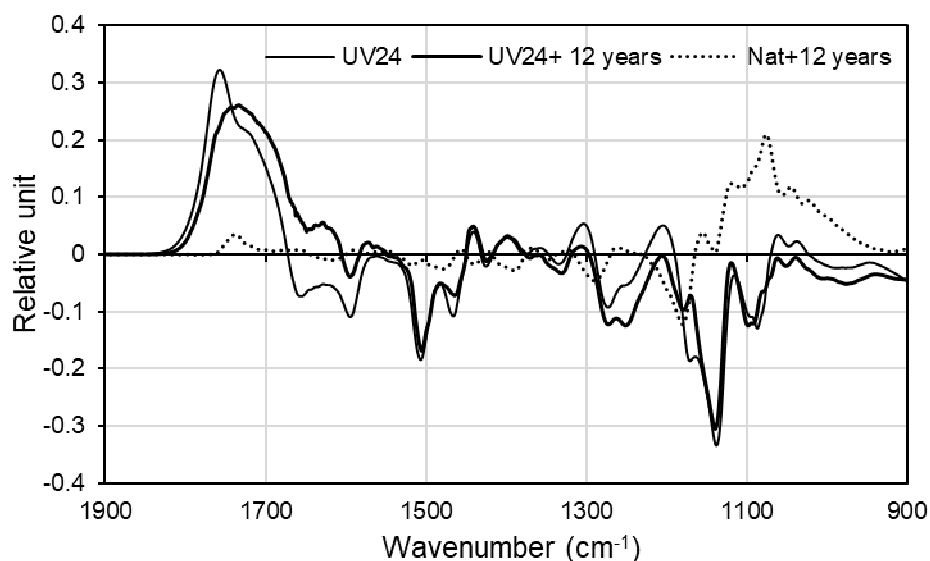
Fig. 7: Difference IR spectra of poplar generated by different treatments.

This absorption increase interval was visible between 1620 and 1690 cm[-1] for deciduous species (Figs. 5-7). Probably the same interval between 1540 and 1690 cm[-1] is the real one for deciduous species as well, but the absorption decreases of syringyl lignin around 1596 cm[-1] overlapped the neighbouring absorption increase and eliminated it. This superimposition lifted slightly the negative peak of syringyl lignin too.

Visual observation showed an increase in redness, which was highly species dependent. The reddest sample was Scots pine latewood, and the less red sample was poplar with good agreement with the absorption increase in the conjugated carbonyl region after 12-year storage. This finding is confirmed by earlier paper (Matsuo et al. 2011).

There is a small but well visible negative absorption peak around 1230 cm[-1] wavenumber generated by 12-year storage on UV irradiated samples (Figs. 3-7). This negative peak represents the reduction of acetyl groups in xyloglucan confirming the degradation of hemicelluloses (Tolvaj 2023). The xyloglucan band at 1230 cm[-1] highly overlaps the absorption band of guaiacyl lignin at 1268 cm[-1], which in the case of beech samples creates an integrated band with a maximum at 1250 cm[-1].

Coniferous and deciduous species presented different behaviour in the absorption interval of ether linkages between 1080-1170 cm[-1]. Conifers showed two main absorption decreases at 1100 and around 1140 cm[-1]. Earlywood samples of conifers showed additional absorption decrease in this wavenumber region because of the 12-year storage compared to the absorption decrease generated by UV irradiation. This decrease represents that ether linkages in cellulose and hemicelluloses were split during the 12-year storage (Figs. 1 and 2). In terms of this alteration during the 12-year storage, the band at 1100 cm[-1] was more affected than the neighbouring band around 1140 cm[-1]. The participation of cellulose in this degradation requires further chemical investigations, because cellulose is known to be stable at room temperature. Latewood of spruce did not show alteration in the absorption interval of ether linkages during the 12-year storage.

In the investigated deciduous species, UV irradiation followed by 12-year storage generated two main absorption decreases in the absorption interval of ether linkages around 1138 and 1170 cm[-1]. The UV irradiation produced negative peak at 1170 cm[-1] considerably

decreased for ash and mainly disappeared for beech and poplar during the 12-year storage. The other peak hardly changed, only ash showed little decrease. These results assume that ether linkages were created, or the UV degraded linkages were restored, during the 12-year storage. Since the samples were stored at room temperature, the thermally most sensitive hemicelluloses may be responsible for the change. The exact chemical background of this alteration needs further chemical investigations.

CONCLUSIONS

UV irradiated and natural surfaces of Scots pine, spruce, beech, ash and poplar samples were stored in room conditions for 12 years to study the long-term durability of photodegraded wooden surfaces. Natural wood surfaces proved to be fairly stable during the storage. Only ether linkages in hemicelluloses showed minor degradation. In contrast, UV irradiated samples underwent alterations during the 12-year storage period. Extractives and hemicelluloses showed chemical changes. Lignin molecules remained stable during 12 years of storage. Hemicelluloses in the photodegraded surface layers underwent thermal degradation and oxidation processes, forming new carbonyl groups. The number of ether linkages absorbing at 1170 cm^{-1} also increased in deciduous species. Extractives presented an increase in absorption in the conjugated carbonyl region.

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