



Chemical changes of steamed timber during short-term photodegradation monitored by FTIR spectroscopy

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Abstract

Black locust (*Robinia pseudoacacia* L.), beech (*Fagus sylvatica* L.), poplar (*Populus x euramericana* cv. *pannonia*) and spruce (*Picea abies* Karst.) wood samples were treated in saturated steam at 100, 110 and 120 °C for 2 days. Steamed and unsteamed (control) specimens were irradiated using a UV emitter mercury lamp in order to determine the chemical changes generated by UV irradiation. The main goal of the research was to find out if steaming can improve the UV resistance of wood. Chemical changes were determined by diffuse reflectance FTIR spectroscopy. Steaming deacetylated the hemicellulose molecules of hardwood species. Steamed spruce specimens showed the same photodegradation properties as the unsteamed ones. Steaming reduced the photodegradation sensitivity of lignin considerably for black locust only. Steamed hardwood specimens presented greater absorption increase at 1705 cm⁻¹ compared to unsteamed samples, which corresponds to the photodegradation of hemicelluloses.

1 Introduction

Steaming is an environmentally friendly colour modification method to create a warm brown wood colour. There are some wood species having unattractive or highly inhomogeneous natural colour. This appearance reduces the usability of these species for visible constructions. Customers appreciate aesthetic wooden surfaces for their pleasing visual appearance. Colour modification by steaming can help to modify the unattractive original colour or highly inhomogeneous colour to attractive brown colour (Tolvaj et al. 2009, 2010; Dzurenda 2013, 2017, 2018; Geffert et al. 2017; Dzurenda and Dudiak 2019, 2020).

Natural colour of black locust covers a wide hue range from greyish yellow to greenish brown. The high extractive content of black locust gives the possibility to create many brown tints up to chocolate-brown depending on the steaming parameters (Varga and Van der Zee 2008; Tolvaj et al.

2010; Dzurenda 2018). Beech is usually steamed to turn its initially white–grey colour into more attractive reddish. The colour difference between white and red heartwood of beech can be minimalised by steaming as well (Tolvaj et al. 2009; Geffert et al. 2017). Colour change of plantation poplar by steaming was investigated to obtain attractive colour suitable for various indoor applications (Banadics and Tolvaj 2019). Steaming was found to be a proper technique to turn the naturally unattractive colour of poplar wood to a pleasant brown. Steam was able to double the colour saturation, which is a significant result from the perspective of indoor wooden applications. The treatment increased both redness and yellowness values and reduced the lightness. Steaming properties of conifers (Scots pine, spruce, larch, sugi) have been investigated in the last decade (Tolvaj et al. 2012, 2019; Preklet et al. 2019). Depending on the applied steaming parameters, a variety of colours could be created from the initial yellowish to the modified light brown colour. These new colours were similar to the colours of aged indoor wooden constructions and furniture, thus providing a fully natural alternative to chemical surface colour modification.

Wood material is sensitive to weathering. The UV part of solar radiation is the main factor that induces wood photodegradation primarily through the decomposition of lignin. Photodegradation of natural wood is a widely studied phenomenon (Tolvaj and Faix 1995; Pandey 2005; Popescu et al. 2011; Zivkovic et al. 2014; Timar et al. 2016;

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Preklet et al. 2018; Varga et al. 2020). Nevertheless, only a few papers were found dealing with the photodegradation behaviour of steamed wood. The colour stability to photodegradation of poplar timber was investigated by Banadics et al. (2019). The results showed that steaming improved slightly the colour stability during photodegradation. Colour stability of steamed black locust, beech and spruce timber was investigated during short-term photodegradation (Varga et al. 2021). Black locust was the only species showing better colour stability after steaming. The effect of UV radiation on natural and steamed maple wood was presented in a conference paper (Dzurenda et al. 2020). The change in the lightness coordinate L^* was opposite for untreated and steamed wood. While untreated maple wood darkened, steamed maple wood faded. Todaro et al. (2015) showed that UV-C treatment caused irreversible changes in both chemical composition and morphology of steamed (130 °C) Turkey oak samples, via photooxidation and photodegradation processes. The hydrothermally treated wood surfaces exhibited better colour stability than untreated ones during UV-C irradiation.

The aim of this study was to investigate the chemical changes of steamed timber during UV irradiation to determine whether steaming is able to improve the UV stability of the investigated species.

2 Materials and methods

Black locust (*Robinia pseudoacacia* L.), beech (*Fagus sylvatica* L.), poplar (*Populus x euramericana* cv. *pannonia*) and spruce (*Picea abies* Karst.) specimens were used for the tests. Black locust and beech were chosen because these species are the most frequently steamed species in wood industry in Hungary so that their behaviour during UV irradiation could be of high importance from commercial point of view. Plantation poplar grows in large quantity and needs new application areas, whereas spruce was added for comparison of deciduous and conifer species. The specimen size was 30 × 12 × 5 mm (L × R × T). The radial surface was used for IR measurement. Initial moisture content of the specimens was between 9 and 10% before steaming. The steaming was carried out in an autoclave to keep the pressure generated by the steam. Wood specimens were placed in the autoclave with distilled water at the bottom for conditioning the air to maintain maximum relative humidity. The chosen steaming temperatures were 100 °C, 110 °C, and 120 °C, the steaming time was 2 days.

Steam-treated specimens were subjected to photodegradation together with the thermally untreated control specimens. UV light emitter mercury vapour lamp provided the light irradiation. The UV radiation was 80% of the total emissions (31% UV-A, 24% UV-B, and 25% UV-C). The total electric

power of the applied double mercury lamps was 800 W, and the distance between the specimens and the light source was 64 cm. The light power density was 76 J/m²s on the surface of the specimens. The irradiation chamber was set to 60 °C guaranteed ambient temperature conditions. The total irradiation time was 36 h. The irradiation was interrupted after 7, 16 and 36 h to measure the IR spectra.

The diffuse reflectance infrared spectrum of the samples was measured before and after treatments. Measurements were performed using an FTIR 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 at 3800 cm⁻¹ and at 1900 cm⁻¹ was carried out. The spectral intensities were calculated in Kubelka–Munk (K–M) units. The spectra were normalised to the band maximum around 1374 cm⁻¹. The intensity values of spectra were adjusted to 1.0 by this normalisation at 1374 cm⁻¹ wavenumber. 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 treated sample. In this case, absorption increase is represented by positive band while negative band represents absorption decrease. Details of IR data manipulations are described in a previous work (Csanady et al. 2015). Band assignment is presented in Table 1. Assignment of the individual bands was made using the difference IR spectra.

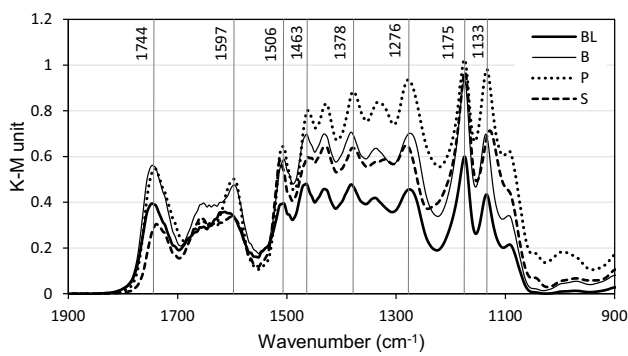
3 Results and discussion

The investigated species were chosen because of their diverse extractive content. Black locust has high extractive content covering 5–9% of total mass. The main component is dihydrorobinetin covering 2–5% of the total mass of wood (Molnar and Bariska 2002; Sanz et al. 2011). The total extractive content of beech is between 3 and 5%, comprised of a variety of hydrophilic (sugars, sugar acids, carboxylic acids, simple phenols and polyphenols) and lipophilic (fatty acid, fatty alcohols, sterols) compounds (Vek et al. 2015). The extractive content of poplar and spruce wood is the lowest (2–3%) among the investigated species. It is of high importance to be aware of the extractive content of wood because, on the one hand, mainly these chemical constituents determine the colour of each wood species, and, on the other hand, most of the extractives are sensitive to thermal treatment, thus giving the possibility to modify the wood colour by steaming, without any chemicals. Moreover, it is important to discover, whether steaming is able to modify the photodegradation properties of timber.

Figure 1 depicts the absorption spectra of the untreated specimens. The spectra of the four species are highly similar,

Table 1 Examined IR bands of wood (place of maximum) and band assignments (Tolvaj and Faix 1995; Huang et al. 2012; Csanady et al. 2015)

Wavenumber(cm^{-1})	Assignment
1770–1750	C=O stretching vibration of non-conjugated ketones and γ lactones
1750–1700	C=O stretching vibration of carboxyl groups and acetyl groups in xylans
1660–1650	conjugated C–O in quinines coupled with C=O stretching of various groups (flavones)
1604–1594	aromatic skeletal breathing with CO stretching (syringil lignin)
1512–1505	aromatic skeletal vibration (guaiacyl lignin)
1470–1460	C–H deformation in xylan
1380–1370	C–H deformation in cellulose and hemicellulose (Place of normalization)
1285–1275	$\text{C}_{\text{aryl}}\text{--O}$, guaiacyl ring breathing with CO stretching
1240–1230	C–O linkage in guaiacyl aromatic methoxyl groups and acetyl groups in xyloglucan
1180–1170	C–O–C stretching (asymm.) in cellulose and hemicelluloses
1140–1130	C–O–C stretching (symm.), arom. C–H i.p. deformation, glucose ring vibration

**Fig. 1** IR spectra of untreated black locust (BL), beech (B), poplar (P) and spruce (S)

showing peaks at the same wavenumbers. From the high number of peaks only those are assigned, which will be discussed in the present evaluation. Several overlapping absorption bands were found between the 1100–1500 cm^{-1} wavenumber range. The measured maxima of overlapping bands are usually not the real ones, small shifts are possible. (The difference spectrum gives more precise peak positions, because the changed bands are visible only). The only well-separated band between 1700 and 1800 cm^{-1} belongs to the absorption of unconjugated carbonyl groups. This is a composed band, including the absorption bands of the carbonyl groups, found in different molecules at various positions. The next band at 1597 cm^{-1} belongs to the aromatic ring vibrations of syringyl lignin.

This band is overlapped by the broad band of conjugated carbonyl groups between 1570 and 1700 cm^{-1} and the bound water also absorbs in this wavenumber range. Conjugated carbonyl groups are found mainly in extractives. Wood can contain a great variety of extractives in various amounts, depending on wood species (deciduous, coniferous) and tissue type (sapwood, heartwood), thus absorption changes

in this region only give information on the change of their amount but do not provide information on the type of the extractives.

The typical lignin band is visible at 1506 cm^{-1} . The next band at 1463 cm^{-1} is the superposition of the absorption for C–H deformation in lignin and C–H deformation in xylan. The peak at 1378 cm^{-1} belongs to the C–H deformation in cellulose. This band remains stable during photodegradation and is used for spectra normalisation and comparison. The peak at 1276 cm^{-1} represents the absorption of $\text{C}_{\text{aryl}}\text{--O}$, guaiacyl ring breathing with CO stretching. The two highest peaks represent the absorption of C–O–C stretching, both asymmetric and symmetric at 1175 and 1133 cm^{-1} , respectively.

The most sensitive chemical component of wood to photodegradation is the lignin macromolecule (Tolvaj and Faix 1995, Pandey 2005, Zborowska et al. 2015, Varga et al. 2017, Arpacı et al. 2021). The photons of UV radiation are able to degrade the aromatic bonds of lignin. The produced free radicals react with oxygen forming carbonyl groups (Tolvaj and Faix 1995; Pandey 2005; Jankowska et al. 2020; Li et al. 2020; Sandak et al. 2021).

Figure 2 presents the difference IR spectra of the investigated unsteamed species generated by 36-h UV irradiation. The greatest absorption decrease is visible at 1174 and 1138 cm^{-1} . The first decrease belongs to the asymmetric stretching of the ether bond in cellulose. The second decrease belongs to the symmetric stretching of the ether bond, the aromatic C–H deformation and to the glucose ring vibration. These absorption decreases indicate the ether bond splitting. Hemicelluloses have ether linkages in different positions. The determination of the exact positions of ether bond splitting needs further investigations using near infrared spectroscopy (NIR).

The negative band at 1174 cm^{-1} for spruce is not visible. The positive band with maximum at 1186 cm^{-1} overlaps and eliminates this negative band. The negative peak around

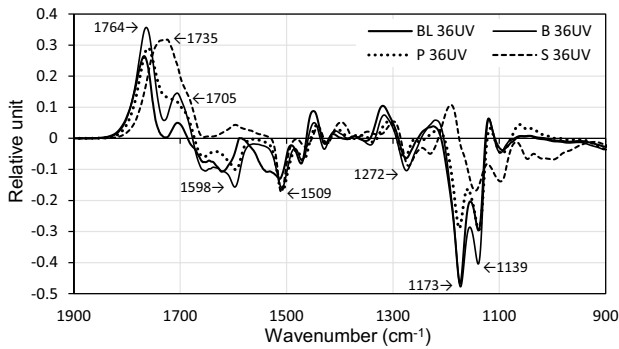


Fig. 2 Difference IR spectra of unsteamed black locust (BL), beech (B), poplar (P) and spruce (S) specimens generated by 36-h UV irradiation

1510 cm^{-1} belongs to the aromatic skeletal vibration of guaiacyl lignin. The exact places of this peak are 1512, 1510, 1509 and 1508 cm^{-1} for black locust, spruce, beech and poplar, respectively. This negative peak is detectable together with the absorption decrease of the guaiacyl ring breathing around 1270 cm^{-1} . Absorption decrease of syringyl lignin is visible around 1598 cm^{-1} . The exact places of the minima are at 1621, 1598 and 1596 cm^{-1} for black locust, beech, and poplar, respectively. The peak maximum, observed for black locust is not the real one. The neighbouring negative band overlaps with this lignin band and the superposition modifies the place of the maximum considerably. As softwoods contain insignificant amounts of syringyl lignin, the corresponding band is missing almost completely from the IR spectrum of spruce. Deciduous species presented an absorption decrease around 1650 cm^{-1} wavenumber. This region represents the absorption of conjugated carbonyl groups located mainly in extractives.

As a consequence of lignin degradation, the generated free radicals react with oxygen producing carbonyl groups and, in the end, a great variety of carbonyl compounds (Lanzalunga and Bietti 2000; Chang et al. 2014; Yu et al 2015; Li et al 2019; Mattonai et al. 2019). The absorption increase of unconjugated carbonyls is visible in the 1680–1820 cm^{-1} wavenumber range. For deciduous species the increase of two additional bands was observed at 1705 and at 1764 cm^{-1} wavenumbers. The band at 1764 cm^{-1} represents the absorption of CO stretching for unconjugated ketones and γ lactones generated by the oxidation after the splitting of the aromatic ring. The band at 1705 cm^{-1} represents the absorption of aliphatic carboxyl groups. These two bands were integrated into a broad band for spruce with a maximum at 1730 cm^{-1} .

Hemicelluloses are the most sensitive components of wood to thermal degradation. Deacetylation of hemicelluloses during steaming is caused by the cleavage of acetyl groups linked as an ester group to the hemicelluloses

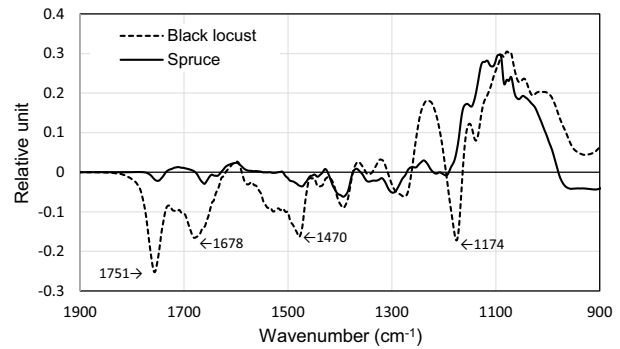


Fig. 3 Difference IR spectra of black locust and spruce specimens generated by two-day steaming at 100 °C

(Carrasco and Roy 1992; Tjeerdsma and Miltz 2005; Liu et al. 2016). Typical smell of acetic acid comes out from the steaming chamber, confirming deacetylation. Figure 3 represents the difference IR spectra of black locust and spruce generated by two-day steaming at 100 °C. The IR spectra clearly show that deacetylation of hemicelluloses is the main chemical change during steaming. The difference IR absorption spectra of black locust and spruce show great differences. The reason lies in the different hemicellulose content of black locust and spruce. The highly acetylated xylan is the main polyose component in hardwoods; however, glucose units in spruce are rarely acetylated. The absorption of carbonyl groups being in the acetyl unit decreased at 1751 cm^{-1} parallel with the absorption decrease of the ester bridge at 1174 cm^{-1} . These two main changes were accompanied by the absorption decreases at 1470 cm^{-1} (C–H deformation in xylan). All these absorption decreases clearly present the deacetylation effect of steaming. Spruce specimens showed only small absorption decrease at the above-mentioned wavenumbers showing the low deacetylation rate of hemicelluloses. There is a wide negative absorption band around 1678 cm^{-1} representing the degradation of extractives having conjugated carbonyl groups. A broad positive peak is visible between 950 and 1250 cm^{-1} . However, this peak does not represent real absorption increase. The Kubelka–Munk equation does not provide the absorption spectrum properly for intensive absorption bands if the surface roughness changes. This phenomenon elevates the difference spectrum intensities in this region. Swelling lifts up wood fibre edges during steaming, and these fibres never return to the original position during drying. The roughness increase modifies the light scattering adjusting the K–M values. A detailed discussion of this phenomenon can be found in a previous work of the authors (Tolvaj et al. 2011).

Photodegradation properties of steamed specimens are presented in Figs. 4, 5, 6, 7 and 8. Figure 4 shows the irradiation time dependence of the chemical changes in poplar specimens steamed at 110 °C. Although the lignin content

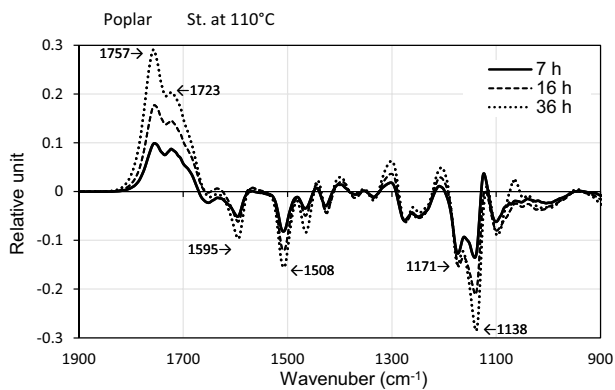


Fig. 4 Difference IR spectra of steamed (at 110 °C) poplar (P) specimens generated by 7, 16 and 36-h UV irradiation

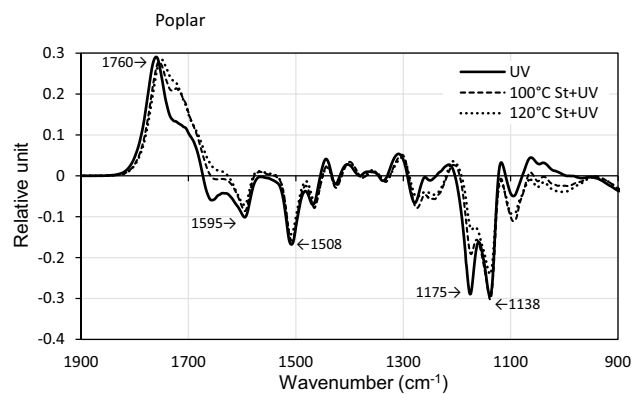


Fig. 7 Difference IR spectra of unsteamed and steamed (St) poplar specimens generated by 36-h UV irradiation

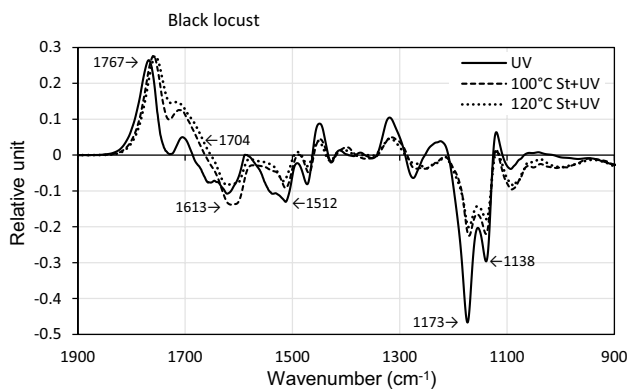


Fig. 5 Difference IR spectra of unsteamed and steamed (St) black locust specimens generated by 36-h UV irradiation

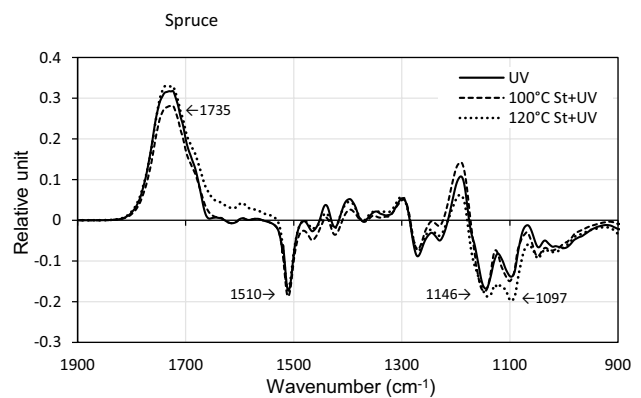


Fig. 8 Difference IR spectra of unsteamed and steamed (St) spruce specimens generated by 36-h UV irradiation

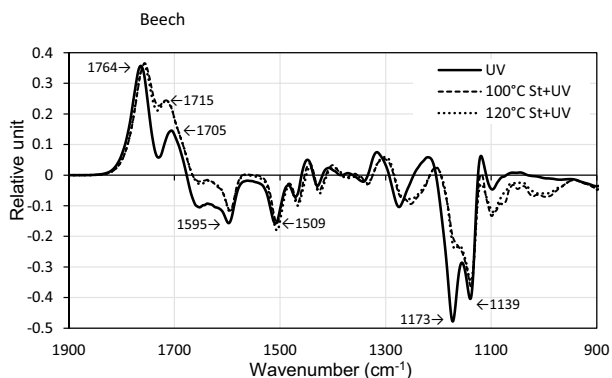


Fig. 6 Difference IR spectra of unsteamed and steamed (St) beech specimens generated by 36-h UV irradiation

decreased (both syringyl and guaiacyl) during UV irradiation, the absorption of guaiacyl ring breathing at 1270 cm^{-1} did not decrease after 7-h irradiation. The reason might be that an unidentified band has formed with increasing treatment time at the left side eliminating the decrease in

absorption at 1270 cm^{-1} . The absorption values of unconjugated carbonyl groups increased around 1710 and 1760 cm^{-1} wavenumbers by prolonged irradiation time. In general, the value of absorption changes were proportional to the UV irradiation time. The only exception was the absorption decrease of the ether bonds at 1174 cm^{-1} . Intensity decrease of this band was equal to that of the neighbouring band during the first 7 h of irradiation. The absorption intensity decrease of the band at 1174 cm^{-1} hardly changed after this 7-h period. The reason for this might be that the previous steaming generated great absorption decrease at 1174 cm^{-1} (Fig. 3 and Nemeth et al. 2016). Time dependence of the chemical changes was similar in the case of the other investigated deciduous species as well.

Figure 5 presents the difference IR spectra of unsteamed and steamed black locust specimens generated by 36-h UV irradiation. Steaming reduced the degradation rate of guaiacyl lignin absorbing at 1512 cm^{-1} . Steaming at 120 °C produced a slightly better lignin protection compared to the steaming at 100 °C . The photodegradation-induced

yellowing level of steamed black locust also indicated the protective effect of steaming (Varga et al. 2021). The spectra do not clearly show the photodegradation stability of syringyl lignin absorbing around 1600 cm^{-1} wavenumber. The conjugated carbonyl groups have absorption here as well, and the neighbouring bands overlap each other. Steaming reduced the rate of ether bond splitting (absorbing at 1174 cm^{-1}) compared to the unsteamed specimens. The reason might be that steaming produced ether bond splitting during this pre-treatment as well. The alteration of steaming temperature does not affect the intensity of this absorption band.

Two bands developed in the unconjugated carbonyl region. The band of CO stretching for unconjugated ketones and γ lactones is located at 1764 cm^{-1} . Pre-steaming did not modify the intensity of this band. Unsteamed specimens suffered similar absorption changes to steamed specimens. Only the place of the maximum shifted somewhat towards shorter wavenumbers due to the steaming. This maximum shift was produced by the growth of the adjacent overlapping band. The intensity change of the carbonyl band at 1705 cm^{-1} was modified by the steam pre-treatment. Steamed specimens showed greater absorption increase than the unsteamed one. Previous studies demonstrated that the absorption increase at 1705 cm^{-1} does not originate from the lignin degradation (Preklet et al. 2021a, b). Research papers demonstrated that photo-oxidation of cellulose and hemicelluloses results in the formation of aldehyde and ketone groups on carbon atoms C2 and C3 of the pyran or furan units, and just these carbonyls may contribute to the increase in absorbance in the unconjugated carbonyl region around wavenumber 1730 cm^{-1} (Müller et al. 2003; Reinprecht et al. 2018). Figure 3 shows that deacetylation of glucose rings was the only difference between the steamed and unsteamed black locust specimens. Consequently, the deacetylated glucose units must be more susceptible to photodegradation than the others. The reason that steamed specimens showed greater absorption increase than the unsteamed ones at 1705 cm^{-1} might be that deacetylated hemicelluloses underwent photo-oxidation during the UV treatment. This statement needs further chemical investigations. All of the investigated deciduous species showed this property except spruce (see also Fig. 8). This finding supports that deacetylated hemicelluloses play an important role in the photodegradation generated absorption increase at 1705 cm^{-1} .

Figures 6 and 7 show the difference IR spectra of unsteamed and steamed beech and poplar specimens, respectively. The changes were generated by 36-h UV irradiation. The IR absorption changes of beech and poplar were similar. Guaiacyl lignin absorbing at 1509 cm^{-1} produced similar degradation in case of both unsteamed and steamed

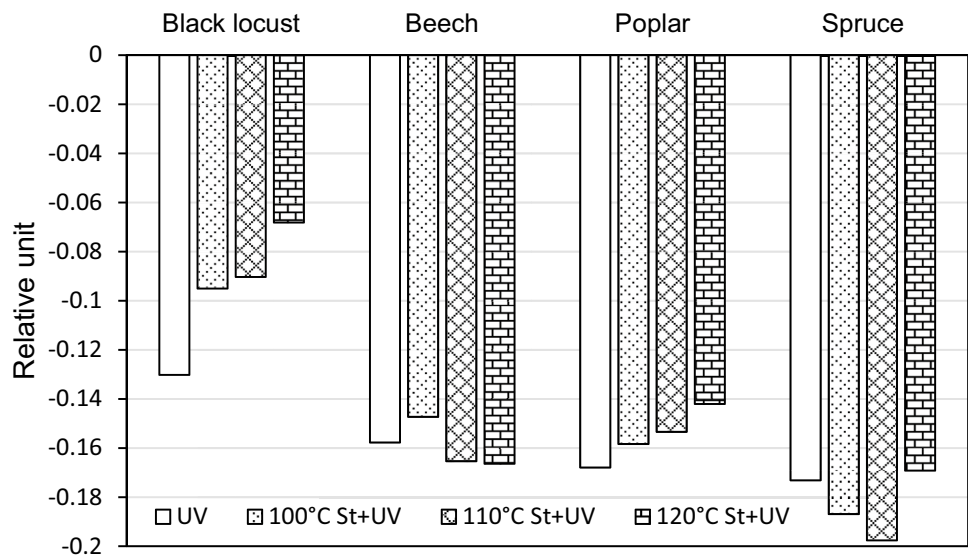
specimens. It means that steaming was unable to reduce the photodegradation sensitivity of lignin in beech and only slightly in poplar.

Syringyl lignin absorbing at 1598 cm^{-1} showed the same degradation properties as guaiacyl lignin. Unsteamed samples produced somewhat greater absorption decrease than steamed ones. The deviation was generated by the neighbouring overlapping band at 1650 cm^{-1} . Steaming reduced the rate of ether bond splitting absorbing at 1174 cm^{-1} compared to the unsteamed specimens, because the applied heat treatment already induced ether bond splitting (Fig. 3). The photo-oxidation of deacetylated hemicelluloses enlarged the absorption in the unconjugated carbonyl region at 1705 cm^{-1} for beech and poplar specimens as well.

Figure 8 presents the difference IR spectra of unsteamed and steamed spruce specimens generated by 36-h UV irradiation. Curves of unsteamed and steamed specimens are almost the same. This phenomenon shows that steaming does not modify the photodegradation properties of spruce timber. This finding confirms that the deacetylated hemicelluloses are partly responsible for the photodegradation of wood.

Figure 9 shows the absorption decrease of guaiacyl lignin of unsteamed and steamed species generated by 36-h UV irradiation. Degradation of lignin macromolecules is the main chemical alteration during the photodegradation of wood. The data of unsteamed species (empty columns) reveal that lignin was most stable in black locust compared to the other investigated species. A previous study demonstrated that extractives play an essential role in the photodegradation of wood, and the rate of wood degradation was lowered by the presence of extractives (Chang et al. 2010). The extractive content of black locust heartwood can reach 9%. Flavonoids make up 89% of the total extractive content. Within flavonoids, dihydrorobinetin is the main component covering 58% of total flavonoid content, followed by robinetin with 14% (Sanz et al. 2011). The UV light degrades the extractives followed by rapid oxidation of the degradation products. The modified chromophores act as a form of energy trap which slows down the photodegradation of lignin (Nemeth et al. 1992). Steaming widely modifies the extractives. This statement is supported by the intensive colour change during steaming. The modification rate increases with elevated temperature according to the Arrhenius law. Steam induced colour change demonstrates this modification of extractives. The modified extractives provide even higher protection for lignin. Steaming of black locust at 120 °C doubled (48%) the protection of lignin compared to the unsteamed specimens. As for the other investigated wood species, only poplar showed a 15% decrease in lignin degradation between the specimens, steamed at 120 °C and the untreated specimen. Beech and spruce species did not show the protective effect of the extractives for the steamed

Fig. 9 Absorption decrease of guaiacyl lignin of unsteamed and steamed (St) species generated by 36-h UV irradiation



specimens. In the case of black locust, the main extractive is dihydrorobinetin, which might play the main role in protecting lignin during UV irradiation. This phenomenon needs further chemical investigation.

Experimental results show that steaming was able to reduce the rate of UV degradation of lignin only in the case of black locust. In line with this finding, a previous study (Varga et al. 2021) demonstrated that steaming was able to increase the colour stability against UV radiation only of black locust. The current findings demonstrate that in the case of outdoor wooden applications, the steam generated attractive brown colour and the lignin in the timber requires effective protection using a proper finishing layer.

4 Conclusion

Untreated and steamed black locust, beech, poplar and spruce specimens were irradiated by UV emitter mercury lamp to determine the chemical changes generated by UV irradiation. The chemical changes were monitored by diffuse reflectance IR spectroscopy. Steaming deacetylated the hemicellulose molecules of hardwood species. Steamed spruce specimens showed the same photodegradation properties as the unsteamed ones. Steaming reduced the photodegradation sensitivity of lignin considerably in black locust only. Steamed hardwood specimens presented greater absorption increase at 1705 cm^{-1} than unsteamed ones. This increase refers to the photodegradation of hemicelluloses.

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Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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