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Wood Technology/Products

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Comparative investigation of chemical and structural properties of charred fir wood samples by Raman and FTIR spectroscopy as well as X-ray-micro-CT technology

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Abstract: Wood surface charring is a treatment method commonly employed to enhance weather protection and aesthetic appearance of building exteriors. This study aims to investigate the differences between two wood surface charring processes: the traditional Japanese method known as Yakisugi and an alternative charring technique industrially manufactured with a gas burner. The objective of the study was to assess whether a thicker layer after Yakisugi treatment has any advantages over a thinner layer after the alternative process. Vibrational spectroscopy techniques including UV resonance Raman (UVRR) and Fourier transform infrared (FTIR) spectroscopy, were utilized in conjunction with X-ray-micro-CT analysis. The findings revealed that ATR-FTIR spectroscopy detected the degradation of carbohydrates and changes in lignin within the charred surface, although both processes exhibited similar vibrational contributions. In contrast, UVRR spectroscopy provided insights into the carbonized layers, revealing spectral differences indicating variations in temperature during the charring processes. X-ray micro-CT analysis visually highlighted significant differences in the coal layers, suggesting distinct combustion profiles. Remarkably, the macrostructure of wood treated with Yakisugi remained intact despite a thicker charred layer compared to the alternative charring techniques. However, further investigations are required to assess the weather stability of the alternative charring method for a comprehensive understanding.

Keywords: surface charring; thermal degradation; wood modification; wood protection; Yakisugi

1 Introduction

The demand for renewable and recyclable materials has been heightened by the increasing focus on sustainable building practices. Wood is an excellent construction material for its sustainability, non-toxicity, relatively low cost, local availability and environmental friendliness for building components. Selecting wood products can contribute greatly to mitigating climate change. The prevalence of architectural designs that prioritise environmental sustainability is increasing, and researchers such as Čermak et al. (2019), Šeda et al. (2021), Machová et al. (2021), Kymäläinen et al. (2017, 2018, 2020, 2022), Ebner et al. (2021, 2022) have investigated the traditional technique of charring wood to improve its durability and its use in exterior applications. Consequently, adopting environmentally friendly techniques like surface charring shows to be highly advantageous for protection of wood for exterior, enabling the attainment of cost efficiency, contemporary design, ecological sustainability and cascading use.

Traditionally, the Yakisugi method involves tying three wooden boards together to form a triangular chimney (Ebner et al. 2022). As a starting energy, sawdust is wrapped in newspaper and set on fire. Due to the pulling effect of the chimney, the fire spreads quickly from the bottom to the top and the temperature rises rapidly (Ebner et al. 2021). Also, in addition to the Japanese Yakisugi process, other methods as for example: gas flame, an electric muffle furnace or a hot plate are employed to char wood surfaces (Kymäläinen et al. 2017, 2020). However, various processes take advantage of the principle that the cell wall components of wood change chemically at elevated temperatures (above 150 °C) (Sandermann and Augustin 1963). Based on the study by Runkel (1951), Fengel (1966) as well as Fengel and Przyklenk (1970), the effects of high temperatures on wood and its components were systematically investigated. Levoglucosan is considered the primary cleavage product in the thermal decomposition of cellulose (Sandermann and Augustin 1964). The degradation mechanisms of the polyoses are similar to those of the thermal degradation of cellulose (Sandermann and Augustin 1963). However, the polyoses exhibit lower thermal resistance compared to cellulose. The degradation reactions of lignin are based on cleavage of the lignin-polysaccharide bond and depolymerization of the molecules (Fengel and Przyklenk 1970). These effects can be observed in the region of the α- and β-aryl-ether bonds.

Shafizadeh (1984) divided the effects of temperature on wood into 2 phases, defining the threshold temperature of 300 °C. Below 300 °C, the displacement of water, the reduction of the degree of polymerization within the wood compounds, the formation of free radicals and carbonyl, carboxyl and hydroperoxide groups dominate, releasing carbon monoxide and carbon dioxide. Above a temperature of 300 °C, the decomposition mechanism includes the cleavage of the molecules, which yields sugar degradation products (e.g. furfural and levoglucosan) and volatile low molecular weight degradation products. Above 450 °C, volatile products no longer exist, and further bulk decomposition is due to the decomposition of the remaining char.

The major benefit of charring wood surfaces is generally linked to the reduced water absorption because charcoal is much more hydrophobic than untreated wood. However, less intense treatments leave amorphous polysaccharides intact, which interact with water. In contrast, excessive temperatures or time contributes to the formation of cracks on wood surfaces, which leads to increased water sorption (Kymäläinen et al. 2017).

Currently the focus of research primarily lies in the physical changes and the impact of treatment parameters on the moisture content of the wood or its water absorption (Čermák et al. 2019; Kymäläinen et al. 2017). Nevertheless, the chemical and structural properties of the charred wood surfaces remain unknown. The production process according to traditional Japanese methods is carried out manually, requiring experienced workers to perform the tasks (Ebner et al. 2021). On the other hand, the alternative method of achieving a gas-charred surface, more readily to industrial-scale applications, typically involves the use of a permanently installed machine. In this process, the boards are passed over a gas flame, and the charred layer thickness increases as the speed decreases. However, due to the high gas consumption involved in this process, the carbon layer is usually limited. On the central European market both variants are available and have a similar purchase price. The Yakisugi variant is labour intensive while the alternative requires high gas consumption. Therefore, in this study, the influence of two different charring processes on chemical compositions and structural differences is investigated by various complementary methods. The study employs advanced technologies such as UV Resonance Raman (UVRR), ATR-FTIR, micro-ATR and X-ray micro-CT to delve into the chemical composition and structural characteristics of the surface resulting from the combustion process. The primary objective is to ascertain whether a thicker layer of charcoal produced through the traditional Yakisugi method exhibits any notable distinctions compared to a thinner layer generated through an alternative, more industrial ready approach.

2 Materials and methods

2.1 Materials

Fir (*Abies Alba* L.) wood boards from an Upper Austrian lumber mill (Sägewerk Dax KG, Salzburg, Austria) were used for this study. The wood boards were in grading classes III to IV according to the Austrian Timber Trade Practices, pre-dried to an average moisture content of 12 %. The sawn wood boards of dimension 190 × 24 × 4000 mm^3 were treated.

2.2 Timber charring with the traditional Japanese method – Yakisugi (Yak)

Ebner et al. (2021) gave a detailed description about the one-sided surface charred process based on the traditional method by open fire. At the beginning of the process, three wood boards were tightened together using wet ropes, to create a triangle, to perform as a chimney. Then the reaction was started with an ignitor ball consisting of a handful oakwood shavings, which were wrapped in a newspaper. Measurements were conducted at various positions (0.5, 2.0, and 3.0 m) on the fir boards, starting from the chimney's origin and, revealed temperature readings of 300 °C within 67, 84, and 150 s, respectively (Ebner et al. 2021). Furthermore, the study demonstrated that after 300 s of treatment using the Yakisugi (Yak) method, the average temperatures reached approximately 740 °C, 770 °C, and 520 °C at different positions. After 300 s the process was finished by using water. These treatments were carried out in winter conditions (−1 to +4 °C).

2.3 Timber charring with an alternative method (Alt)

As an alternative method, the charring was made by hand with a flame blasting device, which is intended to represent the industrially charred production method. The selected wood boards of fir were moved under a gas burner (RoMax from Rothenberger Industrial, Germany) with a process velocity of 0.133 m/min and a distance between the wood surface and the gas burner of 0.03–0.05 m and that charred the top surface across the whole width of the boards. After the burning, the wood surface was extinguished with water to stop the burning process and to cool down the boards.

2.4 UV resonance Raman

These measurements were performed at the IUVS beamline of the Elettra Sincrotrone Trieste synchrotron radiation facility with an excitation wavelength of 266 nm, provided by a CryLas FQSS 266-Q2, Diode-Pumped Passively Q196 Switched Solid State Laser. Raman signal was collected in back-scattered geometry, analysed by a single pass of a Czerny-Turner spectrometer (Trivista 557, Princeton Instruments, 750 mm of focal length) equipped with a holographic grating at 1800 g/mm and detected using a CCD camera. The spectral resolution was 1.7 cm^{-1}/pixel.

For each sample, the cross section was inspected and spectra were collected for charcoal, wood and charcoal and wood border layer. Each acquisition lasted for 30 min (15 min scan × 2 scan). The final radiation power on the samples was kept at about 0.3 mW. Samples were analysed without any previous preparation through continuous spinning to avoid photo-damage effects.

2.5 ATR-FTIR spectroscopy

To obtain information of the chemical changes in the materials FTIR spectra were collected after the two carbonization processes. The surface on four samples after each treatment and untreated reference were analysed with a Perkin-Elmer Frontier FTIR spectrometer by using an ATR Miracle diamond crystal assessor. The samples were cut out manually with razor blade. The spectra were used in their unprocessed form, without undergoing any pre-processing steps, except for calculating the mean value of the measurements. The spectra used 32 scans with a resolution of 4 cm^{-1} in the wavenumber range between 4000 and 600 cm^{-1}

2.6 Micro-ATR spectroscopy

By using a micro-ATR 20x objective couplet to a microscope VIS-IR Bruker Hyperion 3000 and an interferometer Bruker Vertex 70v, spectra from a mapping area of 2 × 20 points were acquired by accumulating 64 scans with a spectral resolution of 4 cm^{-1} for one sample of each of the charring processes. Analysis was carried out on the cut surface starting from untreated wood to the charcoal layer. Each data point was 200 µm apart from the previous data point. Data were water vapor compensated using Opus 8.5 SP1 (Bruker Optics, Billerica, US) and then analysed using Quasar (https:\\quasar.codes) (Toplak et al. 2017, 2021).

2.7 X-ray-computed micro-tomography

The internal microarchitecture of charred wood (Alt and Yak) was investigated by the means of the X-ray Computed micro-Tomography (micro-CT) at Tomolab (Elettra Sincrotrone Trieste S.C.p.A.).

Acquisitions of one representative sample per carbonization process were performed in cone beam geometry with a micro-focus Hamamatsu source (40 kV, 200 µA, small focus) and a sCMOS PSL detector (2042 × 2039 pixel matrix, 31.2 µm equivalent pixel size). The source-to-detector distance was 495 mm, and the source-to-sample distance was 95 mm, corresponding to a geometrical magnification of 5.2. A set of 1800 projections in step and shoot mode were collected over a 360° rotation. The exposure time was fixed at 3 s.

Projections were flat-field corrected and pre-processed using a ring removal filter, a beam hardening correction filter and a Gaussian smoothing filter. The pre-processing and the reconstruction with FDK algorithm (Hamming filter) were performed by Nrecon software (v.1.7).

3 Results and discussion

The different charring processes were carried out successfully for the fir samples. Figure 1 shows a comparison between the end-grain cutting of samples after the Yakisugi (Yak) and an alternative (Alt) process. On the visual inspection, the charring wood surfaces presented different appearances depending on the different method used, and it was clear that the charred wood layer from the Yak process is thicker compared to the alternative method (Alt).

Based on the geometric shape of the produced wood chimney of the Yakisugi method, the charred layer becomes always thicker from the edges of the boards to the centre position. The average of the charred layer was 3.0 mm with a

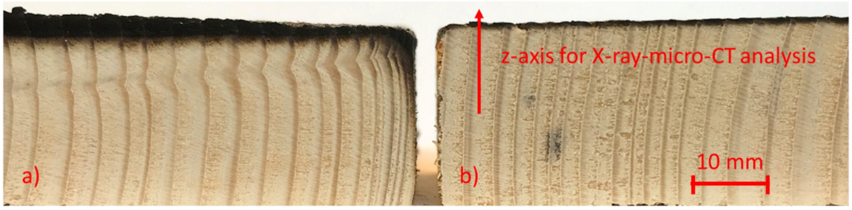

Figure 1: Visual inspection of treatment results on the end-grain cutting of fir boards: (a) one example of charred sample with the Yak method, (b) a sample after the Alt process.

standard deviation of 0.98 mm, whereas the coal layer from the alternative process showed an average of 0.4 mm with a standard deviation of 0.15 mm, and presented a more homogenic surface layer of the coal.

This is an interesting observation as the depth of the treatment has a great influence on the wood compositions and its structures and affects certain properties such as water absorption, cracking under weathering and thermal insulation (Sandberg et al. 2021). Hence, choosing the right treatment techniques could be a useful tool to fine-tune the properties of wood.

The results of the Fourier transform infrared (FTIR) spectroscopy showed that chemical changes can be observed due to the different charring processes (Figure 2). Moreover, the wavenumber, the material, the assigned functional groups and its corresponding reference are shown in Table 1 for the most important IR bands. The FTIR spectra obtained from the surfaces of the samples reveal characteristic alterations associated with fire or thermally treated wood. The pyrolysis layers of wood can be characterised by the functional groups of aromatic or aliphatic structures (Shafizadeh 1984).

However, the impact of these two processes is quite similar. The range between 1700 cm^{-1} and 800 cm^{-1} displays significant changes in the bands related to carbohydrates and lignin. A new band around 1700 cm^{-1} (C=O in phenolic esters) can be observed in the FTIR spectra (Bahng et al. 2011). The peak intensity of the absorbance bands at 1590 and 1270 cm^{-1} of the charred wood samples were attributed to C=O and C–O groups, respectively (Chen et al. 2016). The signal at 1510 cm^{-1} in the reference samples is no longer evident in both treated samples, which belongs to the stretching vibrations of the lignin aromatic ring (Faix 1991; Schnabel and Huber 2014). Instead, a distinct shift and broadening in the spectra shape can be observed between 1500 cm^{-1} and 1300 cm^{-1}, indicating changes in the lignin structure associated with phenolic ring and phenolic OH groups.

Additionally, the modified samples exhibit overlapping and disappearance of signals at 1150 cm^{-1} and 950 cm^{-1},

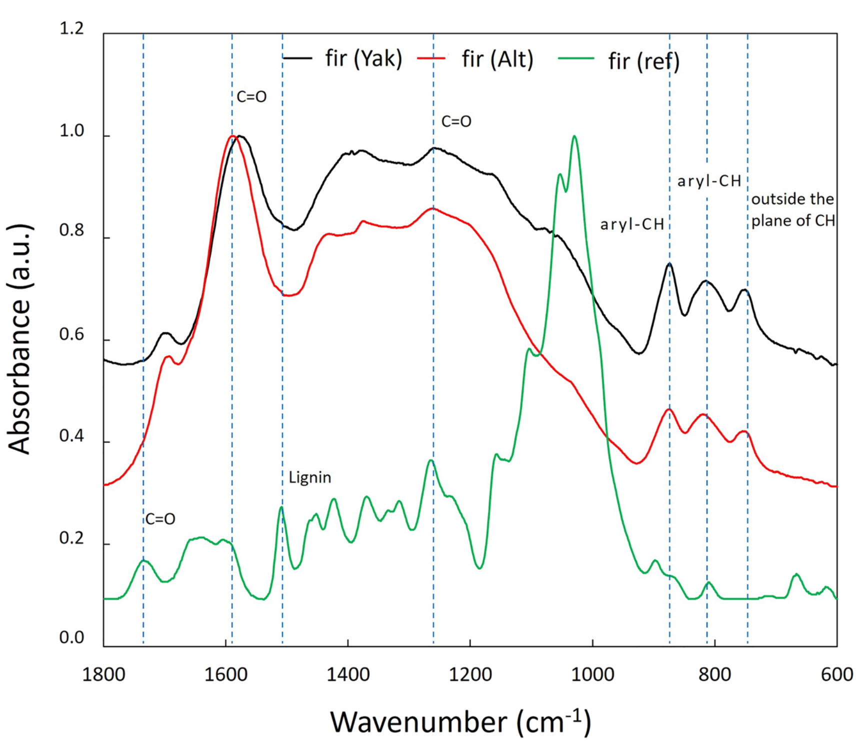

Figure 2: ATR FTIR spectra of untreated sample (fir (ref)) and two charred fir samples after using Yakisugi (fir (Yak)) and alternative method (fir (Alt)).

Table 1: Assignment of the important IR bands of natural wood and bio-coal.

Wavenumber (cm^{-1})	Materials	Functional group	References
1730	Carbohydrates	C=O stretching	Schwanninger et al. (2004)
1700	Pyrolyzed wood	Unconjugated C=O stretch, C=O in phenolic esters/ lactones	Bahng et al. (2011)
1590	Lignin	Aromatic skeletal vibrations	Faix (1991)
1510	Lignin	Aromatic skeletal vibration	Faix (1991)
1460	Lignin	C–H deformation, –CH_3 and –CH_2– of lignin	Faix (1991)
1270	Lignin	G ring plus C=O stretching	Faix (1991)
1230	Lignin, carbohydrates	C–C plus C–O plus C=O stretch,	Schwanninger et al. (2004)
1158	Cellulose	C–O– asymmetric valence vibration	Schwanninger et al. (2004)
1110	Cellulose	Glucose ring stretching vibration	Schwanninger et al. (2004)
1061	Cellulose, holocellulose	C–O valence vibration	Schwanninger et al. (2004)
869	Pyrolyzed wood	Wagging vibration of aromatic CH groups	Zuo et al. (2003)
799	Pyrolyzed wood	Wagging vibration of aromatic CH groups	Zuo et al. (2003)
745	Pyrolyzed wood	Deformation outside the plane of CH-groups	Rousset et al. (2010)

which correspond to strong signals in the reference samples and are related to C–C and C–O signals in carbohydrate components such as cellulose and hemicellulose (Kymäläinen et al. 2018). This observation supports the notion that during pyrolysis, carbohydrates undergo degradation.

Micro-attenuated total reflection (micro-ATR) analysis was conducted to investigate the spectral changes from the uncharred region to the charred layer, aiming to understand their progression. Figure 3 provides a comparison of the specimens treated with Yak and Alt method. In Figure 3a–c, the samples and the measured points are depicted, accompanied by a colour scale representing the integration value of the carbohydrate and cellulose composition within the range of 1175–917 cm^{-1}. Representative spectra from different acquisition points are shown in Figure 3b and c. The spectra acquired from the uncharred area are displayed in green, the mixed area in blue, and the charred area in red for each sample. For the samples obtained by the alternative method, where the charred layer is relatively thin, the progression could be analysed to a limited extent.

However, it is important to note that in both samples, the characteristic spectra of fir, indicative of carbohydrates and cellulose, are almost absent due to their degradation during the charring processes.

In Figure 4a and b the UVRR spectra recorded at the cross section for each sample are shown. Going from wood to char, a decrease in the band at ~1600 cm^{-1} is apparent. This band in wood spectra relates to the vibrations of the lignin aromatic rings, which have a strong resonance in the UVRRS (Elmay et al. 2015; Pandey and Vuorinen 2008). The redshift of 15 cm^{-1} and 11 cm^{-1} for Alt and Yak samples, respectively, can be linked to thermal treatments inducing depolymerization and condensation processes which result in poly aromatic structures in chars (Nieto-Delgado et al. 2021).

With pyrolysis, an overlapping of the two spectral components associated with hydrogen- and oxygen-rich amorphous carbon structures in chars can be observed (Guizani et al. 2017). As shown in the deconvoluted spectra in Figure 4c and d, both charred woods exhibit common features of carbons, commonly referred to as G (~1600 cm^{-1}) and D (~1400 cm^{-1}) peaks (Bormett et al. 1995; Schüpfer et al. 2021; Wang et al. 1990). The G peak derives from bond stretching of sp^2 atoms in aromatic rings and chains, while the D peak comes from breathing modes of sp^2 atoms in aromatic rings (Keown et al. 2007). UV radiation could excite a third peak called *T* around 1000 cm^{-1} in chars (Ferrari and Robertson 2001), due to the C–C sp^3 vibrations. Therefore, the absence of the *T* peak and the presence of the G and D peak in the UVRR spectra of both analysed samples suggest a high content of sp^2 hybridization of carbon atoms. Figure 4e–g present a summary of the spectral characteristics of the D and G bands, which enable a qualitative assessment of the charred wood. The G-band of the Yak sample appears slightly upshifted (~1600 cm^{-1}) and narrow compared to the alternative process sample (~1596 cm^{-1}). This suggests a first difference between the two chars related to the formation of larger, less distorted ring systems in the Yak sample (Ferrari and Robertson 2000). Hence, the D band is more downshifted and broader in the Yak than in the Alt sample (Figure 5e and f). This observation indicates the presence of more aromatic clusters, suggesting a higher temperature during the pyrolysis process (Smith et al. 2016). Finally, Figure 4g shows the intensity (band area) ratios between the D and G bands for both chars. The increased value for the Yak sample confirms the consuming and re-organization of more amorphous

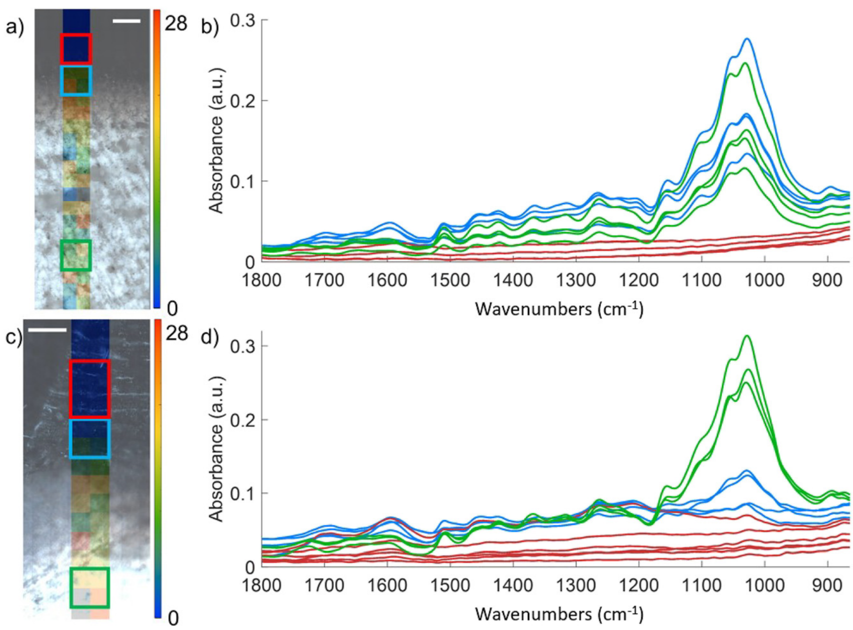

Figure 3: Micro-ATR analysis on specimens treated with alternative methods (a, b); specimens treated with Yakisugi method (c, d). Colour scale represents the intensity of the integration of 1175–917 cm^{-1} spectral region in arbitrary units. In coloured square (a, c): red, charred layer; green, untreated wood; blue, intermediate layer.

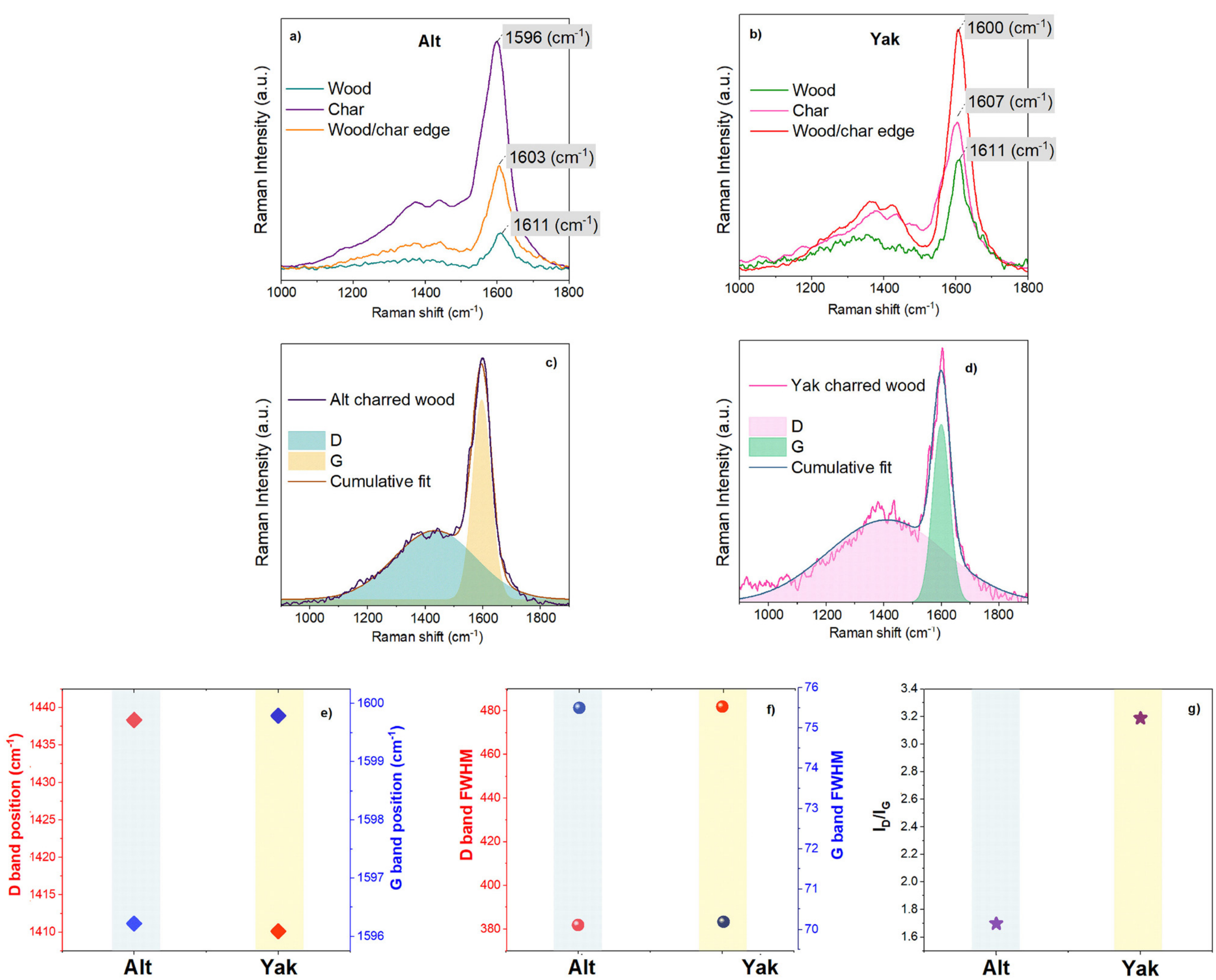

Figure 4: UVRR spectra recorded at the cross section for (a) Alt and (b) Yak samples; (c) and (d) the Gaussian deconvolution highlighting the components for charred woods spectra; (e) G and D band peak positions; (f) G and D bands FWHM; and (g) I_D/I_G values for Alt and Yak samples.

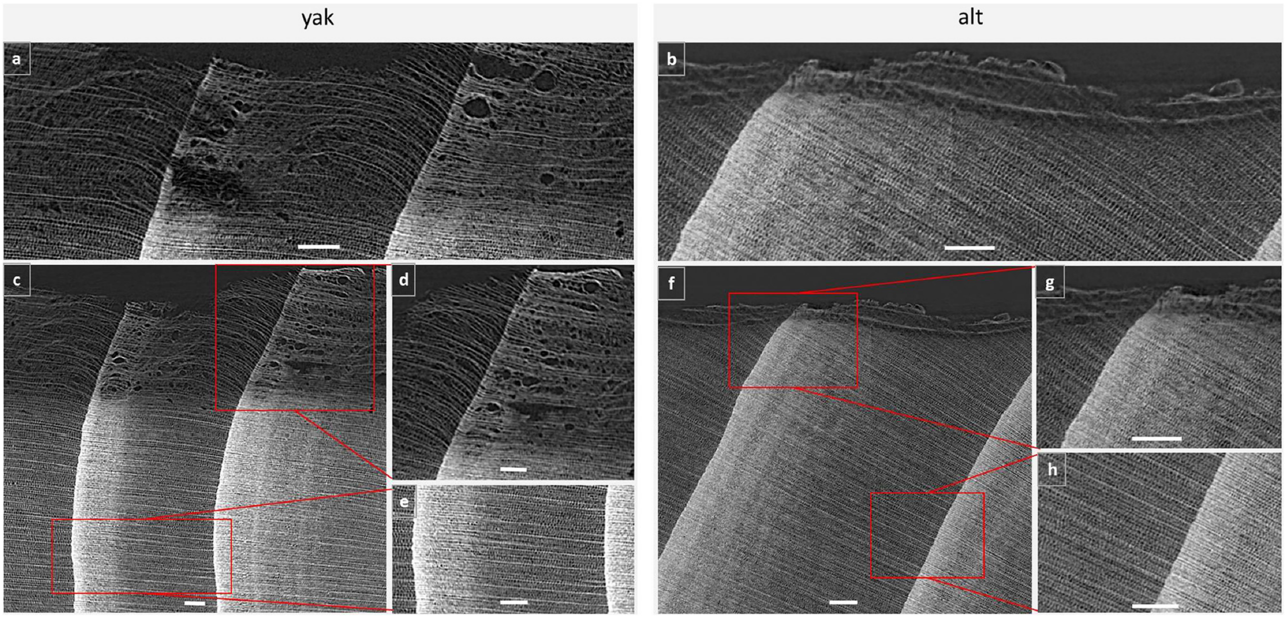

Figure 5: X-ray micro-CT slices (from 18 μm MinIP) virtually made from wood treated with two charring methods: (a) char layer of Yak; (b) char layer of Alt; (c) whole section with untreated and treated wood with Yak; (d) char layer of Yak; (e) untreated wood; (f) whole section with untreated and treated wood with Alt; (g) char layer of Alt; (h) untreated wood. The white scale bar corresponds to 1 mm.

structures as the wood has been possibly exposed to higher temperatures (McDonald-Wharry et al. 2013).

To understand how the two charring processes modify the internal structure of wood, X-ray computed microtomography was performed on both samples. To increase the contrast of low-density elements, minimum intensity projections (MinIP) were applied on 3 consecutive slices, each 6 μm thick, obtaining the selected section (total thickness 18 μm) reported in Figure 5.

Analysing the wood anatomy in the uncharred part in Figure 5, early wood is characterized by large cells and thin walls, while late wood exhibits a dense structure with small, compact cells. The higher cellular density of late wood imparts a brighter appearance in X-ray micro-CT slices.

In general, both charring processes result in cell wall rupture. However, the sample subjected to the alternative method exhibits the complete collapse of the internal structure of the wood. On the contrary, the Yakisugi process (Yak) provokes two outcomes: the development of substantial cavities in late wood and elongated voids in early wood (Figure 5a).

From Figure 5a and b the charred part of sample Alt is visibly reduced (in thickness and volume) compared to that of the sample Yak. On the other hand, the structure of sample treated with Yak method appears intact and less damaged, than sample treated with Alt method in terms preservation and visibility of wood macrostructure. In addition, the charred layer of Yakisugi method is significantly higher than the after alternative process.

The enhanced structural integrity observed in the Yakisugi method may be attributed to two key factors: the deeper penetration of heat facilitated by the chimney-like constructions, and the restricted availability of oxygen during the burning process. In contrast, when using a gas burner on a single surface, an unlimited supply of oxygen is accessible for combustion (Kymäläinen et al. 2017). This finding suggests that not only flame temperature and surface temperature play a crucial role, but also the specific temperature reaching the core of the board. This temperature distribution effectively maintains the macrostructure of the wood. However, it also promotes the degradation of polysaccharides and hemicellulose within the wood matrix, which are actively involved in water exchange reactions, while simultaneously preserving the wood skeleton. Consequently, it is imperative to carefully regulate and maintain the desired temperature while limiting the amount of oxygen present. This controlled approach promotes the thermal degradation of wood components instead of complete combustion and extensive degradation of the wood structure.

4 Conclusions

Various analytical methods were employed to explore and compare the chemical structure and structural characterization differences resulting from two potential methods of surface charring on fir wood and the differences between the traditional handcrafted technique and a modern method using a gas burner were evaluated.

From the charred samples, using ATR-FTIR analysis it was possible to observe three spectral contributions which most probably derived from the residual lignin. The spectral range of 1700 cm^{-1} to 800 cm^{-1} shows significant changes in carbohydrate and lignin bands in comparison to the reference samples. The modified samples also show overlapping and disappearing signals at 1150 cm^{-1} and 950 cm^{-1}, suggesting the degradation of carbohydrates during pyrolysis. Overall, the FTIR spectra of the two charred techniques showed minimal differences.

The UVRR study has helped to pin point chemical differences in the samples treated by the two distinctive methods. In particular, the possible lower temperature of the alternative production method may result in more disordered carbonaceous materials than those obtained by the Yakisugi method. The Yak samples demonstrate a higher presence of aromatic clusters, supporting the findings of the X-ray micro-CT study that the chimney allows for higher temperatures to be obtained within the board.

The investigation with X-ray micro-CT helped to understand how the two charring processes affect the morphological structure of the wood. The Yakisugi method enhanced structural integrity. The higher thickness may be attributed to deeper heat penetration facilitated by chimney-like constructions and restricted oxygen availability during burning, which the alternative method lacks. This controlled temperature distribution helps to maintain wood macrostructure and promotes thermal degradation of wood components while limiting combustion and extensive structure degradation. Although the Yak method shows higher thickness overall, the Alt method achieves a greater level of homogeneity throughout the sample.

The observed variations in the layers of charcoal strongly depend on the employed treatment process. Future investigations are necessary to explore the weather protection properties associated with the alternative method. Additionally, investigating the temperatures reached during the carbonization process and oxygen access may be crucial to determine their impact on the resulting carbonaceous materials and their suitability for practical applications.

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