Investigations on chemical changes of surface contact-charred Norway spruce (Picea abies L.) using ATR-FTIR spectroscopy and pH value measurements

Christian Leich
Eberswalde University for Sustainable Development
Alexander Pfriem
alexander.pfriem@hnee.de

Eberswalde University for Sustainable Development

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Abstract

Surface charring of wood has been used for many centuries to make wood more durable against biological pests (e.g. wood decaying fungi). The exact reasons for the increased durability of surface charred wood however remain more or less unclear. To obtain more information on possible reasons for the increased durability of surface charred wood, the chemical changes of surface contact-charred Norway spruce (*Picea abies* L.) got investigated by ATR-FTIR spectroscopy and pH value measurements. Temperatures of 300°C, 350°C and 400°C were used to carbonize the surface of specimens for a duration of ≈ 3 min. Measurements were conducted not only on the surface of the specimens but also on areas underneath the surface by removing material in steps. By that a “depth profile” of chemical changes could be created. Overall, the results of the applied methods seem to confirm the literature regarding the occurrence of different temperature zones in areas below the surface where different chemical changes occur. Concerning possible reasons for the increase of durability of charred wood the formation of phenolic compounds during pyrolysis could be determined. Those might play a key role inhibiting the infestation by wood decaying fungi.

Introduction

Building with wood presents a number of material-related challenges that need to be addressed to ensure a long service life. Many different methods of wood preservation have been developed to achieve this goal. One method that has been used for many centuries to make wood more durable against biological pests is surface charring (Sutter 1992). A traditional surface charring method that has received some attention in recent years is the Japanese method called “Yakisugi”. Several different scientific research approaches have mainly proven positive effects of surface charring on the durability of wood against wood decaying fungi (Leich 2024, Šeda et al. 2023, Kymäläinen et al. 2022, Machová et al. 2021, Akçay et al. 2020). It is assumed that the increased durability of charred wood against wood decaying fungi may be the result of 1) the loss of nutrients/the ”no recognition” of wood components by enzymes due to chemical changes (Kymäläinen et al. 2022, Machová et al. 2021); 2) changes of hygroscopic behaviour of wood (e.g. hydrophobic char layer, lower equilibrium moisture content) (Kymäläinen et al. 2022, Machová et al. 2021); 3) the formation of pyrolysis products that can act as fungicides or hinder wood decay (e.g. phenolic compounds like vanillin and syringaldehyde (Bi et al. 2024)) (Machová et al. 2021), but the migration of extractives to the surface during pyrolysis is also discussed (Kymäläinen et al. 2022). However, the exact reasons for the increased durability remain unclear, as the results of research differ also due to different test materials and methods.

A charred/carbonized surface can be achieved by various methods, either by setting the wood on fire (e.g. “Yakisugi” method) or by heating the wood surface with a (gas) flame or an electric hotplate. Depending on the chosen method the surface characteristics will vary.

Regardless of the surface charring method, the charring of wood results in several different chemical changes of the wood. Mainly the degradation of the wood components (cellulose, hemicellulose, lignin)

and the formation of various degradation products, the whole process is defined as pyrolysis. The extent of the chemical changes depends on various different factors such as specific material properties, but mainly the charring temperature.

When wood is exposed to high temperatures and begins to heat up, bound water will escape from the wood and the wood starts to dry on the surface. Depending on the chosen charring temperature, various chemical and structural changes take place on the surface of the wood as well as in areas “underneath” the surface as the material heats up. In this regard, Browne (1958), White & Dietenberger (2001) and Lowden & Hull (2013) defined several zones based on the temperature at which specific changes in wood chemistry occur (Fig. 1).

Due to the differences in the chemical composition of the main wood components, they have different thermal stability and form different degradation products during pyrolysis, depending on the charring temperature. The component with the lowest thermal stability is the hemicellulose due to its amorphous molecular structure and its different monomer units (Windeisen et al. 2007; Yang et al. 2007). The chemical changes and degradation of hemicellulose take place in a temperature range of 120°C – 315°C (Bartlett et al. 2018). The degradation products of hemicellulose during pyrolysis are mainly acetic acid due to deacetylation reactions (White & Dietenberger 2001) but also formaldehyde, carbon monoxide, hydrogen and other compounds (Browne 1958). The cellulose in wood is more thermally stable than the hemicellulose and undergoes chemical changes and decomposition in the temperature range of 240°C – 400°C (Bartlett et al. 2018). Degradation products of cellulose include levoglucosan, char, carbon dioxide, carbon monoxide and several other compounds such as formaldehyde, acetone, formic acid, acetic acid (Browne 1958). The most thermally stable component of the wood is the lignin with a temperature range of 110°C – 500°C, where changes due to pyrolysis occur (Bartlett et al. 2018). The degradation products of lignin are char and mainly aromatic compounds such as phenols, xylenols and guajacols (Browne 1958). Other products of lignin during pyrolysis can be carbon dioxide, hydrocarbons, organic acids and methanol (Browne 1958). Overall, most of the pyrolysis products mentioned escape from the wood in gaseous form.

A central product that forms during pyrolysis of wood is charcoal, which is a stable polyaromatic compound that is formed from the organic wood components through restructuring, whereby hydrogen and oxygen are lost (Pyle et al. 2015). The intensity of the pyrolysis significantly influences the degree of condensation and the ratio of aromatic rings to other functional groups. A higher pyrolysis intensity results in more condensation reactions and a higher proportion of aromatic structures (Pyle et al. 2015).

As pyrolysis of wood is a surface treatment, the question arises as to how these chemical changes occur in the depth of the treatment zone. In a previous article (Kampe & Pfriem 2018), the fundamental aspects of wood pyrolysis and its effects on the chemical composition of wood were discussed, including FTIR measurements that highlighted key chemical changes. This study builds upon those findings by specifically investigating the depth of chemical changes in pyrolyzed wood.

The aim of this work is therefore to investigate if and how these chemical changes in pyrolyzed wood can be determined along the thickness direction. To this end, layer-by-layer determination of pH should provide information on the presence of acid degradation products and in which of the pyrolysis zones these products are particularly present. On the other hand, a depth profile will be generated by layer-by-layer removal and surface chemical changes will be determined by ATR-FTIR spectroscopy.

Materials and methods

Material

The wood from three different Norway spruce (*Picea abies* L.) trees was used to cut specimen in equal numbers. Two specimen sizes were used for the tests. For ATR-FTIR spectroscopy specimens with the dimensions 20 x 20 x 20 mm³ were used, while the specimens for the pH value measurements had the dimensions 50 x 20 x 20 mm³.

Charring process/pyrolysis

The surface charring of all the specimens was carried out on a prototype contact-charring machine by Möhringer Anlagenbau GmbH, which uses an electric hotplate for charring. The specimens were surface charred at temperatures of either 300°C, 350°C or 400°C. The specimens for ATR-FTIR spectroscopy were charred on all surfaces, and the specimens for pH value measurements were charred on the tangential and radial surfaces only. The charring process of each specimen surface lasted for about 3 min.

ATR-FTIR spectroscopy

The ATR-FTIR spectroscopy spectra of the specimen were recorded directly on the specimen using a Nicolet iS5 FTIR spectrometer (Thermo Scientific Inc., USA) with an ATR (attenuated total reflectance) sampling accessory with a ZnSe crystal. Each recorded spectrum consists of 64 scans (32 background scans, 32 sample scans) with a range of 4000 cm^{-1} – 600 cm^{-1}. The resolution is at 4.0 cm^{-1} with an accuracy of 0.5 cm^{-1}.

In total 6 specimen of each test series (reference, charred at 300°C, 350°C, 400°C) were used for the spectral recording. The recording was always conducted in the centre of a radial surface at a defined point. Between spectral recordings, material was removed from each specimen in defined steps using the sliding microtome Leica SM2010 R (Leica Biosystems GmbH, Germany). The chosen steps of material removal measured from the surface (0 mm) were − 0.5 mm, -1 mm, -1.5 mm, -2 mm, -3 mm and for the 400°C test series specimens additionally − 4 mm and − 5 mm. By that procedure chemical changes due to the charring process could be observed in almost the same annual rings. After recording the spectra of all 6 specimens, means of the spectra with the same objective from the same test series

were generated by using an integrated feature of the OMNIC 9 software (Thermo Fischer Inc., USA). The analysis of the spectra focused on the “fingerprint area” between 1800 cm^{-1} – 800 cm^{-1}.

pH value measurements

For pH value measurements material was removed from 12 charred specimens in defined steps from the radial and tangential surfaces, also using the sliding microtome Leica SM2010 R (Leica Biosystems GmbH, Germany). The chosen shaving fractions were 0 to -0.5 mm, -0.5 to -1 mm, -1 to -1.5 mm, -1.5 to -2 mm, -2 to -3 mm and – 3 to -5 mm (all measured from the specimen surface). The different fractions of shavings were collected separately. Shavings from 12 uncharred reference specimens were also collected but not in different fractions. To investigate chemical changes in the wood by pH value changes, 4 g of each shaving fraction were extracted with 80 ml of distilled water for 24 hours. The extraction took place at room temperature and the beakers got sealed with cling film during the extraction. After this time the extracts were filtered to remove the shavings and the pH values were measured using the pH meter inoLab pH 7110 and the measuring probe SenTix 81 (Xylem Analytics Inc., Germany).

Results and discussion

ATR-FTIR spectroscopy

The results of the ATR-FTIR spectroscopy are depicted in Fig. 2, pictures of the specimen from each test series after the steps of material removal are depicted in table 1.

Table 1: Pictures of the charred specimen after each step of material removal

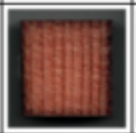
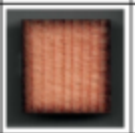
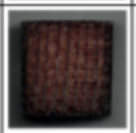
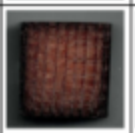
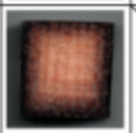
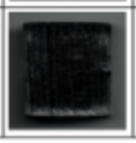
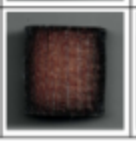
		Material removal (measured from the specimen surface)								Reference
		0 mm	-0.5 mm	-1 mm	-1.5 mm	-2 mm	-3 mm	-4 mm	-5 mm	
Test series	2 – 300 °C									
	3 – 350 °C									
	4 – 400 °C									

Figure 2 shows that the most severe chemical changes of the spruce specimen in each test series occurred on the surface (0 mm spectrum), where the biggest differences between the spectra of the charred specimens and the reference spectrum can be observed. It is also apparent that a higher

charring temperature leads to more severe changes in the spectrum. For example, no clear peaks can be recognised in the 0 mm spectrum of the 400°C test series, which means that all the main components of the wood have been chemically changed/degraded and charcoal has formed. On the contrary, in the 0 mm spectrum of the 300°C test series peaks corresponding with the reference spectrum can be identified. It can also be seen that a higher charring temperature not only led to more severe chemical changes on the specimen's surface, but also in areas underneath. Chemical changes in the material below the surface were observed to varying degrees in all test series depending on the charring temperature. However, these changes vanish with increasing distance from the surface in all test series until the spectra of the charred and the reference specimens are almost identical, meaning that no significant chemical differences between the two could be observed by

ATR-FTIR spectroscopy. After a material removal of 2 mm from the specimen's surface, no significant differences between the charred specimens and the references could be determined even at increased charring temperatures, although colour differences to the references were visible (e.g. table 1).

If the results of the test series are compared, similarities in the shape of the different spectra of the test series become visible. For example, between the 0 mm spectrum of test series 2, the − 0.5 mm spectrum of test series 3 and the − 1 mm spectrum of test series 4 or the 0 mm spectrum of test series 3 and the − 0.5 mm spectrum of test series 4. These observations indicate that similar chemical changes occurred in the specimens of all test series as a result of a similar temperature level, but depending on the charring temperature at different depths. The similarities between the 0 mm (test series 2), -0.5 mm (test series 3) and − 1 mm (test series 4) spectra, for example, can be interpreted to mean that in this case a temperature of approximately 300°C caused the chemical changes. These observations correspond to the model of the different temperature zones that occur when wood is heated (e.g. Browne 1958).

Apart from differences in the general appearance of the spectra between the individual test series, characteristic differences between the spectra of the charred specimens and their references can be determined in all test series.

At wavenumbers of about 1600 cm^{-1} and 1205 cm^{-1} there are peaks that appear in some spectra of the charred specimens in contrast to the references and then disappear in deeper, non-charred regions. The increased peak at 1600 cm^{-1} in the spectra of charred specimens can be attributed to vibrations of C = C groups in aromatic rings of aromatic compounds and charcoal formed during the pyrolysis of wood (Dias jr. et al. 2019). As described by Browne (1958) these aromatic compounds may be the degradation products of lignin during pyrolysis. This seems plausible, as a prominent peak at 1508 cm^{-1} attributed to lignin (Ganne-Chédeville et al. 2021; Hofmann et al. 2022; Horikawa et al. 2019) is attenuated in the spectra of charred specimens, while the 1600 cm^{-1} peak is more prominent. With increasing distance from the surface, the peak at 1600 cm^{-1} progressively vanishes and the peak at 1508 cm^{-1} progressively increases until they both match the reference spectrum. The peak at 1205 cm^{-1} in spectra of charred specimens can be attributed to phenolic compounds (Socrates 2001; Kymäläinen et al. 2018). Therefore, this peak might also indicate the presence of aromatic compounds, which are degradation

products of lignin. Like the peak at 1600 cm^{-1}, the peak at 1205 cm^{-1} disappears with increasing distance from the surface, and the appearance of the spectra of charred specimens and references matches better and better.

Peaks that appear in the reference spectrum but not in the spectra of charred specimen with severe degradation related changes, are found, for example at wavenumbers of approx. 1730 cm^{-1} and 1230 cm^{-1}. The peak at 1730 cm^{-1} appears as a result of IR-active C = O groups and can be associated with the hemicellulose of the wood (Ganne-Chédeville et al. 2021; Horikawa et al. 2019; Popescu et al. 2013). The peak at 1230 cm^{-1} can be partly linked with hemicellulose but also with lignin due to various groups vibrations (Ganne-Chédeville et al. 2021; Hofmann et al. 2022; Horikawa et al. 2019; Popescu et al. 2013). The disappearance of these peaks in the spectra of charred areas or their appearance only in areas with low temperature exposure below the surface may be related to the degradation of the less thermally stable hemicellulose.

pH value measurements

The results of the pH value measurements are shown in Table 2.

Table 2
Measured pH values of the extracts from the different shaving fractions of the test series

		Shaving fraction					
		1	**2**	**3**	**4**	**5**	**6**
		0 to -0.5 mm	*-0.5 to -1 mm*	*-1 to -1.5 mm*	*-1.5 to -2 mm*	*-2 to -3 mm*	*-3 to -5 mm*
Test series	**Reference**	4,89					
	2–300°C	5,12	4,75	4,59	4,63	4,74	4,91
	3–350°C	4,60	4,96	4,78	4,65	4,61	4,72
	4–400°C	3,89	4,98	4,89	4,76	4,65	4,65

The observed pH value of the extract of the uncharred specimens was 4,89 and is the reference value for the interpretation of possible chemical changes in the charred specimens as a result of pyrolysis. This value is consistent with the values published in the literature for cold-water extracts of *Picea abies* L. (Fengel & Wegner 2003). The charring process led to several changes of the pH value of the extracts from the shaving fractions of the charred specimens. Interestingly, all extracts of the shaving fractions from the surfaces of the specimens showed very different pH values in the test series. For example, the extract of test series 2 has the most neutral character of all extracts measured, while the extract of test series 3 is slightly more acidic than the reference and the extract of test series 4 has the lowest pH value of all extracts, although charcoal formed on the specimens surface in test series 3 and 4. The lower pH

values of test series 3 and 4 may not indicate the formation of acidic degradation products, but rather the accumulation of these products from the underlying areas, as the evacuation of the products could be hindered by the hotplate during the charring process. This assumption seems plausible if one considers the pH values of the extracts from the shavings of fraction 2 from test series 3 and 4, which are higher than those of the surface extracts and also the reference. These higher pH values of the extracts could indicate the zone in which strong pyrolysis of the wood takes place, from which degradation products evaporate. The lower pH values of the following fractions below this zone of pyrolysis compared to the reference could be due to acidic degradation products, especially of the hemicelluloses. Another possible explanation could be the evacuation of degradation products in front of the actual pyrolysis zone as described in literature (Park 2008; Bartlett et al. 2018).

Similar trends with regard to the overall changes were also observed in the pH value measurements in all test series as in the ATR-FTIR spectroscopy. Comparable changes occurred in the test series in different areas below the surface depending on the charring temperature. For example, almost similar pH values were measured below the “pyrolysis zone” of the specimen for the extracts of fraction 2 (test series 2), fraction 3 (test series 3) and fraction 4 (test series 4). This seems to continue for the next fractions, which means that a higher charring temperature leads to similar chemical changes, but ‘further away’ from the surface. However, only in test series 2 could an increasing equalisation of the pH value to the reference value be observed, but test series 3 and 4 show a similar trend as well. Overall, the pH value measurements seem to confirm the development of different zones in the wood during pyrolysis, as does the ATR-FTIR spectroscopy.

However, the pH value measurements indicate chemical changes in areas further away from the surface than ATR-FTIR spectroscopy. For example, the pH values in all test series may indicate the presence of acidic degradation products more than 2 mm below the surface. Whether these differences are the result of the degradation of hemicelluloses or the evacuation of its degradation products in front of the pyrolysis zone remains unclear.

Conclusion

The results of both methods, ATR-FTIR spectroscopy and pH value measurements, show that contact charring with a hotplate, though a surface treatment leads to chemical changes of the material underneath the surface too. Depending on the charring temperature, changes occur at varying depths within the wood. Similarities between the test series with different charring temperatures could be observed with both methods regarding chemical changes of Norway spruce (*Picea abies* L.). These similarities indicate the appearance of different temperature zones during pyrolysis, as described in literature. The appearance of certain peaks in the spectra of charred specimens in contrast to the reference spectrum, can be associated with the formation of aromatic/phenolic compounds in certain areas of the specimen during pyrolysis. The results also show that the applied methods for the ATR-FTIR spectroscopy and pH value measurements can be used to detect chemical changes of wood underneath the material surface.

However, further research is needed to obtain more information regarding the comparability of the temperature zones of wood with different charring temperatures and the specific chemical changes in these zones. Future studies should explore the long-term effects of various charring temperatures on wood durability and resistance to biological degradation. Additionally, a combined approach with qualitative chemical analysis methods, such as GC-MS, may provide a more comprehensive understanding of the degradation processes involved, contributing to the development of more sustainable wood preservation methods.

Declarations

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Author Contribution: Christian Leich contribute to the conception and design of the work; the acquisition, analysis, and interpretation of data and writing the manuscript.Alexander Pfriem contribute to the conception and design of the work, interpretation of data and writing the manuscript.

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Figures

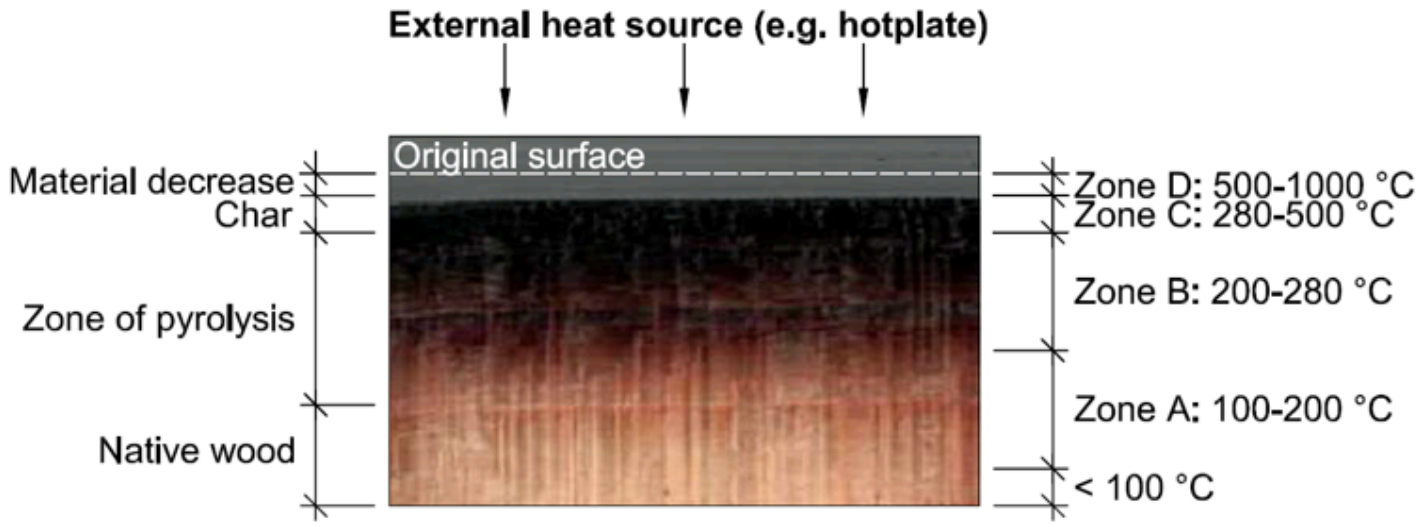

Figure 1

Temperature zones in wood during pyrolysis according to Ebner et al. 2023

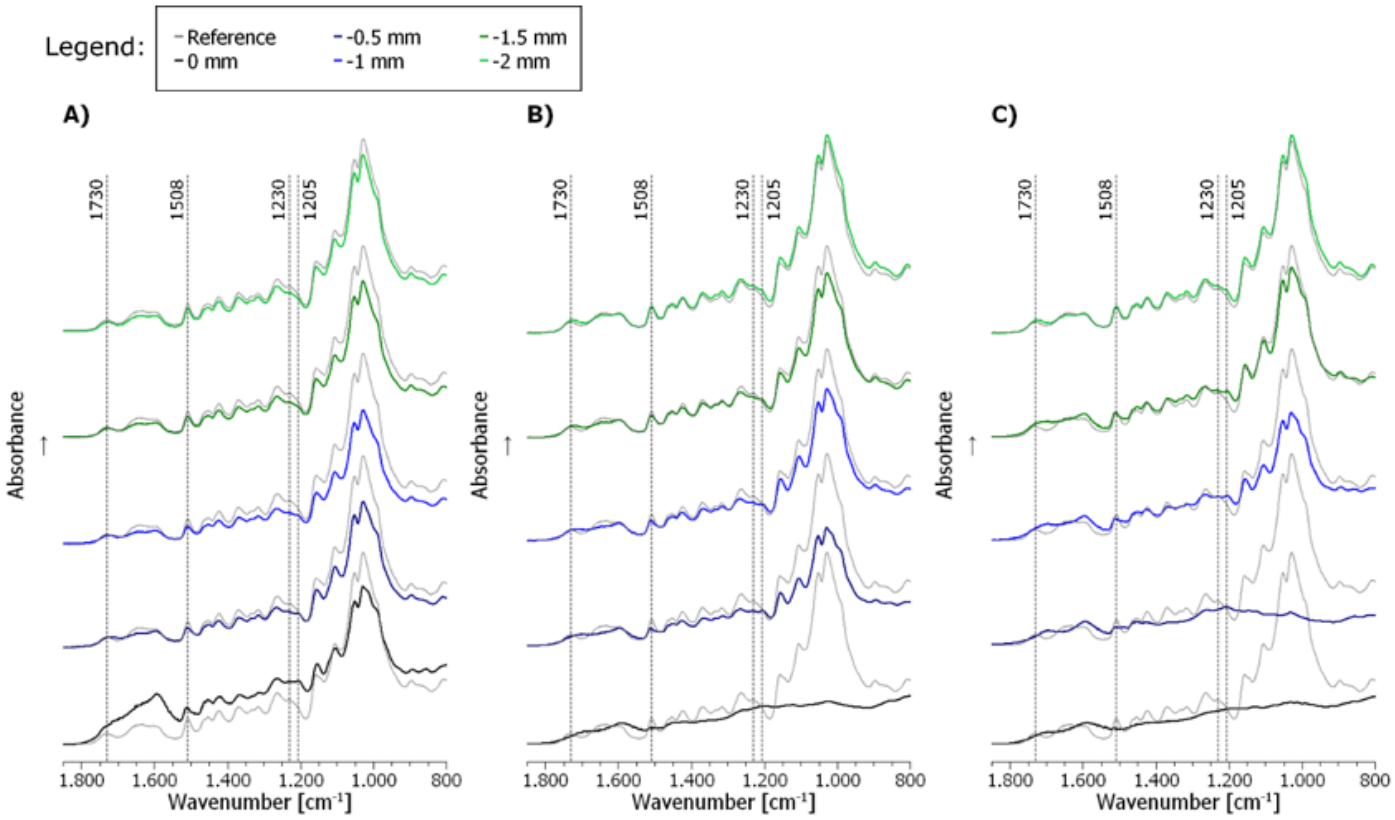

Figure 2

ATR-FTIR spectra of the charred Norway spruce specimens for the different steps of material removal, A) Test series 2 – 300 °C; B) Test series 3 – 350 °C; C) Test series 4 – 400 °C (n of each test series = 6)