

CHEMICAL CHANGES DURING WOOD CUTTING

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(RECEIVED FEBRUARY 2025)

ABSTRACT

Scots pine (*Pinus sylvestris*) and poplar (*Populus euramericana*) species were chosen for the experiments. Sawdust was made by a circular saw with a rare tooth spacing for producing bigger particles beside the fine fractions. Particles were separated by a vibration separator with the sieve sizes 1 mm, 0.63mm, 0.315 mm, 0.1 mm and 0.063 mm. Infrared reflectance spectra of the particles were measured to determine the chemical changes generated by the cutting procedure. The experimental results showed that the grinding of wood resulted in bond rupture for all ether bond types appearing in the infrared spectrum. The number of conjugated and unconjugated carbonyl groups in the wood was significantly reduced during the cutting procedure. After bond breaks, oxidation and recombination processes occurred generating compounds similar to the aromatic structure of lignin, which were shown by increases in absorption at 1514, 1271 and 1231 cm^{-1} .

KEYWORDS: Infrared spectrum, particle size, ether bridge, carbonyl groups.

INTRODUCTION

There are several types of wood cutting methods in wood industry. These methods separate the wood into different pieces depending on the need. Cutting methods break the cellulose-lignin matrix of wood by splitting chemical bonds. The infrared (IR) spectrum analysis method is an excellent analytical solution for following changes in chemical bonds. This analysis is widely used method in wood research to determine the alterations generated by different treatments. Chemical changes during photodegradation are one of deeply studied phenomenon by IR spectrum measurement (Tolvaj & Faix 1995, Colom et al. 2002, Pandey 2005, Kataoka et al. 2007, Chang et al. 2010, Huang et al. 2012, Cogulet et al. 2016, Varga et al. 2020, Tolvaj 2024). Thermal degradation of wood is also a heavily investigated topics studied by IR spectrum analysis (Noupponen et al. 2003, Tjeerdsma and Militz 2005, Kocaefer

et al. 2008, Esteves et al. 2013, Fabiyi& Ogunleye. 2015,Liu et al. 2016,Timar et al. 2016, Xin et al. 2017, Shi et al. 2024).

As wood is an opaque material, the direct absorbance measurement is only possible to use very thin samples. In practise, reflectance spectrum measurement coupled with mathematical transformation can deliver the absorbance spectrum more easily. It must keep in mind that absorbance spectrum generated by these processes is not always identical with the real absorbance spectrum. Because of the indirect measurement and mathematical manipulations, the calculated spectrum can contain anomalies. These anomalies appear mostly where the absorption intensity is high. This is the absorption region of ether bridges around 1000 cm^{-1} wavenumber for wood (Zavarin et al. 1990, Anderson et al. 1991, Tolvaj et al. 2011, Varga et al. 2020, Tolvaj 2022).The reflectance intensities highly depend on the surface quality of the sample. Roughness change modifies the reflectance intensity and it appears in the calculated absorbance spectrum as well (Zavarin et al. 1990, Faix & Böttcher 1992; Pandey and Theagrajan 1997,Bejo et al. 2019, Tolvaj 2024).

Chemical changes that occur during wood milling are a scarcely studied phenomenon.Faix & Böttcher (1992) investigated the effect of particle size of milled wood mixed inKBr powder by using DRIFT technique. It was found that the intensity of the carbohydrate band around 1100 cm^{-1} was strongly influenced by both particle size and wood concentration in KBr powder. Müller et al. (2009) studied the chemical changes in wood during particle- and medium density fibreboard production by FTIR-ATR technique. It was found that particles displayed higher absorbance in bands at $3336, 1738, 1649, 1032\text{ cm}^{-1}$ wavenumbers than wood, suggesting alteration in free OH-groups. These bands were also higher in fibres than in wood.

The main objective of this study was to discover the chemical changes during cutting of wood into small particles. The diffuse reflectance infrared spectroscopy was applied.

MATERIALS AND METHODS

There have been chosen poplar (*Populus euramericana*) and Scots pine (*Pinus sylvestris*) for the experiment, both of them were collected in the local sawmill in Sopron in West Hungary. The moisture content of the original wood was between 15-20% and a circular saw with a rare tooth spacing for producing bigger particles beside the fine fraction was used. Collected sawdust contains the full fraction of particles and this was separated by means of vibration separator with the sieve sizes 1 mm, 0.63mm, 0.315 mm, 0.1 mm and 0.063 mm. Samples were stored in laboratory conditions for several days to equalize and homogenize the moisture content which were about 12% and were stored in a hermetically sealed boxes until the measurements.

The diffuse reflectance infrared Fourier transformed (DRIFT) spectrum of the samples was measured. Measurements were carried out using an IR spectrophotometer (JASCO FT/IR 6300). The resolution was 4 cm^{-1} and 50 scans were obtained and averaged. The background spectrum was gained against an aluminium plate. The reflectance (R) intensities were measured, and the absorbance spectral intensities were calculated by $\text{Log}(1/R)$ function. Solid

wood block (30x10x5 mm) was used as control. Two-point baseline correction at 3800 and 1900 cm^{-1} was carried out. The intensity of spectra was normalised to the band maximum at 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 due its high intensity, central position and strong stability. This normalisation eliminates most of deviations between the spectra generated by the surface roughness differences. As a consequence, the absorbance spectra are given in relative units. The difference spectrum was calculated by subtracting the absorbance data of a solid wood block from the data of milled sample. In this case the positive band of difference spectrum represents absorption increase while a negative band represents absorption decrease. Details of IR data manipulations and the band assignment are described in a recently published book (Tolvaj 2024).

RESULTS AND DISCUSSION

Milling of wood separates particles, breaking several chemical bonds, as it is confirmed by our following experiments. These chemical changes can be monitored by IR spectrum measurement. The easiest way to present the changes is to compare the relative absorbance spectra of milled wood and solid wood. Fig. 1 presents the relative absorbance spectra of solid wood (Control) and wood powder with particle size between 0.31-0.63 mm. Comparison of the spectra shows clearly the absorption decrease of bands at 1740 and 1650 cm^{-1} representing the reduction of unconjugated and conjugated carbonyl groups, respectively. Small absorption decreases and increases are visible in the whole presented wavenumber region. There is an unusual intensity difference in the 900-1200 cm^{-1} wavenumber region. It cannot be a real absorption increase (generated by milling) in such a large wavenumber interval. This distortion was generated by the surface texture differences (including roughness) between solid wood and wooden powder. This anomaly appears often if the surface roughness is changing during a treatment. Photodegradation is an excellent example presenting this anomaly (Tolvaj et al. 2011, Bejo et al. 2019, Varga et al. 2020, Tolvaj 2024).

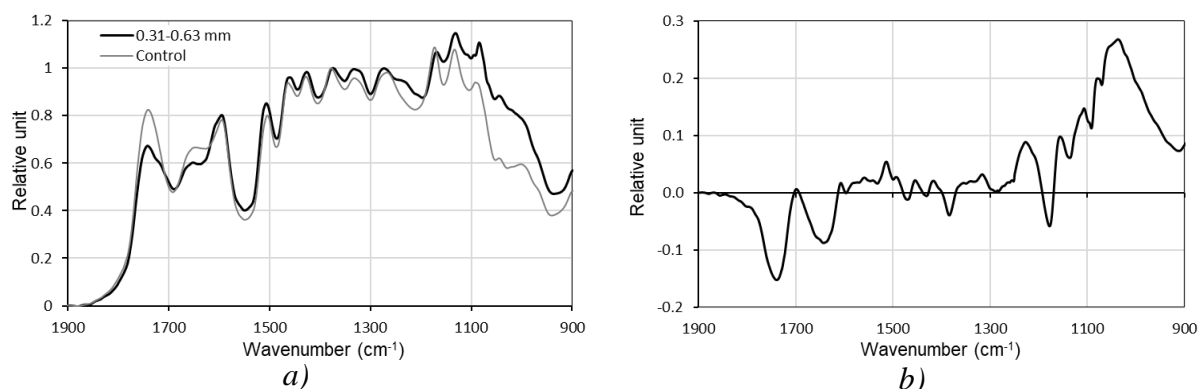


Fig. 1: Absorbance spectra of poplar solid wood (control) and powder in diameter between 0.31 and 0.63 mm (a), and the difference spectrum (b).

Difference spectrum represents more clearly the changes than the comparison of two spectra on top of each other. Fig. 1b shows the difference spectrum of poplar generated by milling. The negative bands at 1740 and 1650 cm^{-1} represent the reduction of unconjugated and conjugated carbonyl groups, respectively. There is small absorption increase at 1508 cm^{-1} .

It is clearly visible that the difference spectrum in the 900-1250 cm^{-1} region is abnormal. Four valleys are visible on the side of a broad positive band. These valleys are located exactly on the absorption positions of ether linkages being different positions in the cellulose-lignin matrix of wood. This broad positive band is not a real absorption band, but it is an abnormal peak. The broad positive band is the stacking up of an anomalous band and the difference spectrum.

To get the real difference spectrum, we must omit this broad band. After calculating the shape of the anomalous band, simple subtraction can eliminate it. Corrected difference spectrum was determined by subtracting the values of anomalous band from the values of measured difference spectrum. Fig. 2 shows the anomalous band, the original difference spectrum and the corrected difference spectrum. The corrected difference spectrum shows perfectly the real absorption decreases.

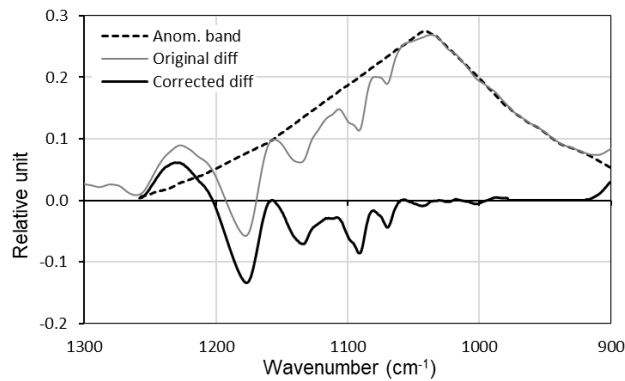


Fig. 2: Difference absorbance spectra of poplar powder in diameter between 0.31 and 0.63 mm presenting the abnormal positive band and the corrected difference spectrum.

The shape of anomalous band was calculated for both investigated species and for all particle size assortment. It was found that the shape of anomalous band was identical in all cases for both species, but the intensities were different. Fig. 3 shows the anomalous band intensities of all different particle size assortment for Scots pine powders.

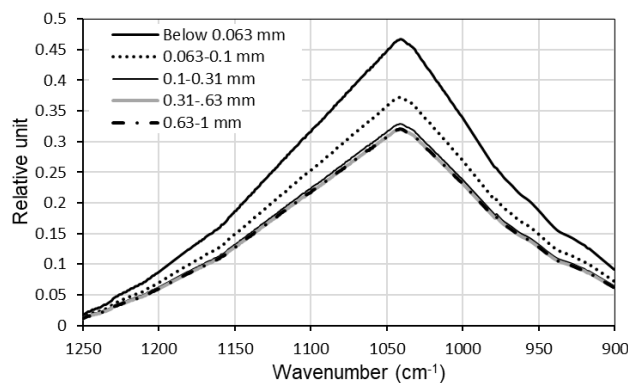


Fig. 3: Intensity distribution of the abnormal positive band depending on the particle size for Scots pine.

The smallest particles generated the greatest anomalous band intensities. Increasing particle size reduced the anomalous band values. The particle diameter above 0.1 mm did not modify the intensities of anomalous band. This finding shows that the reason of anomalous band generation is not only the roughness alteration. However, the roughness variations can modify the values of the anomalous band. To discover the reason of the above described anomaly needs further investigations.

After elimination of anomalous band, the difference spectra showed correctly the absorbance changes. Figs. 4 and 5 present the selected difference spectra for poplar and Scots pine species, respectively. Figures show mainly negative bands (absorption decreases). It is not surprising because milling splits the chemical linkages between molecules and within a molecule as well. Difference spectra of the investigated two species are similar. Alterations can be seen in the band intensities only. There are two intensive absorption decreases in the carbonyl region. The number of both unconjugated and conjugated carbonyl groups decreased represented by the absorption decreases at 1740 and 1644 cm^{-1} wavenumbers, respectively. If we compare the carbonyl bands with the ether bands around 1100 cm^{-1} , it is clearly seen that the carbonyl bands are much wider than the ether bands. The reason for this is that the carbonyl bands are complex, appearing as the sum of some adjacent bands. Decrease of the unconjugated carbonyl band is similar for both investigated species (Figs. 5 and 6). In contrast, the absorption decrease in conjugated carbonyl region shows deviations. Poplar samples present one main band but Scots pine samples introduce three individual bands. Conjugated carbonyls can be found in lignin and mainly in extractives. The reason of deviations is that poplar is pure in extractives, in contrast, Scots pine includes much more and different types of extractives than poplar.

The absorption region of ether bonds presents four absorption decreases at 1070, 1091, 1134 and 1176 cm^{-1} . These peak positions are the same for both poplar and Scots pine samples. The first absorption decrease belongs to the C-O stretching mainly from C(3)-O(3)H in cellulose. The second band belongs to the C-O-C ether linkage. The third absorption decrease represents the symmetric stretching of ether bond in cellulose and hemicelluloses, the aromatic C-H deformation, and the glucose ring vibration. The fourth decrease fits to the asymmetric stretching of ether bond in cellulose and hemicelluloses. The greatest absorption decrease at 1176 cm^{-1} shows the decomposition of cellulose and hemicelluloses.

It is not surprising that the split of ether linkages is one of the main changes during milling of wood. All cellulose, hemicelluloses and lignin molecules and their connections contain ether bridges. Moreover, the bonding energy of C-O linkage ($5.7 \cdot 10^{-19}$ Joule) is one of the smallest among the bonding energies existing in wood. Only the bonding energy of C-C linkage is smaller slightly ($5.5 \cdot 10^{-19}$ Joule).

There is an intensive positive band at 1514 cm^{-1} for both investigated species. This is the absorption region of aromatic ring vibrations arising from lignin. This band is much higher for Scots pine than for poplar. It is highly questionable that the lignin content of the samples increased during milling. The reason could be that the milling generated free radicals react with oxygen creating substances having similar structure to lignin.

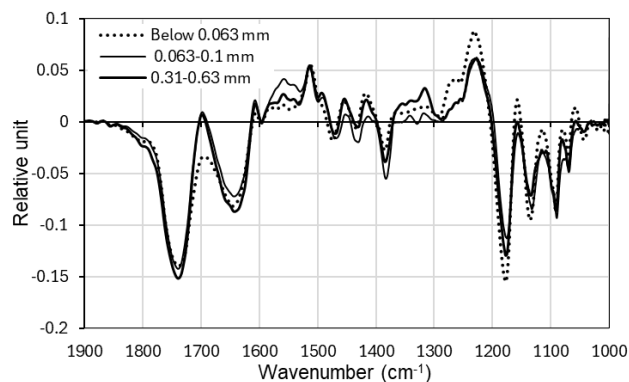


Fig. 4: Difference absorbance spectra of poplar powder with different diameters (fingerprint region).

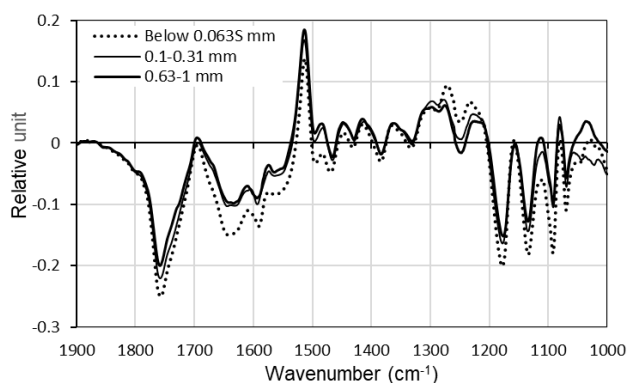


Fig. 5: Difference absorbance spectra of Scots pine powder with different diameters (fingerprint region).

Earlier works (Forsthuber et al. 2013, Tjeerdsma et al. 1998, Boonstra and Tjeerdsma 2006, Chen et al. 2012, Kacikova et al. 2013, Kacik et al. 2023) reported absorption increase due to thermal treatment in the 1508 cm^{-1} region, as consequence of the splitting of aliphatic side chains, the cleavage of $\beta\text{-O-4}$ linkages in the lignin structure, followed by the condensation reactions. At the same time, the increase of the intensity for this band suggests the degradation of amorphous carbohydrates (especially hemicelluloses) and increases the number of lignin-type molecules (Boonstra and Tjeerdsma 2006, Esteves et al. 2008, Popescu et al. 2013, Timar et al. 2016, Liu et al. 2017).

Previous study of photodegradation present that the absorption peak of aromatic ring vibration in lignin locates at 1507 and 1510 cm^{-1} for poplar and Scots pine, respectively (Varga et al. 2017). This finding also supports that the absorption increase at 1514 cm^{-1} does not originate from real lignin.

There are two positive bands at 1231 and 1271 cm^{-1} . The first absorption increase belongs to the C–O linkage in guaiacyl aromatic methoxyl groups and acetyl groups in xyloglucan. The second band represents the absorption of $\text{C}_{\text{aryl}}\text{-O}$ guaiacyl ring breathing with CO stretching. These two absorptions increases show arise in the number of lignin-type molecules, strengthening the assumption regarding the reason of absorption increase at 1514 cm^{-1} .

The absorption increases at 1514 , 1271 and 1231 cm^{-1} do not exclude the degradation of the aromatic structure in lignin during the milling of wood. It can happen that both absorptions decrease and increase also occurred and the increase was more dominant. The

behaviour of syringyl lignin resolves this uncertainty. The aromatic skeletal breathing of syringyl lignin absorbs around 1697 cm^{-1} . Poplar samples did not present any change around 1697 cm^{-1} showing that the aromatic structure of syringyl unit remained intact during milling. This finding suggests that the basic unit of guaiacyl lignin was also unbroken during milling. This statement does not exclude the breaking of the connection among the lignin basic units during milling. There are some small positive and negative bands in the $1300\text{-}1500\text{ cm}^{-1}$ region. Meanings of these bands are questionable. Most of them might be artificial, generated by small baseline shifts.

Fig. 6 shows the absorption changes in the carbonyl-methyl absorption region ($2800\text{-}3800\text{ cm}^{-1}$). There are visible broad positive and negative bands in the OH region. The maximum of the absorption decrease is located around 3636 cm^{-1} . This negative band presents the absorption decrease of the intramolecular hydrogen bond in the phenolic group in lignin. This intensity loss can also be interpreted as the rupture of intramolecular OH bonds of cellulose (Tolvaj & Faix 1995). The absorption increase around 3340 cm^{-1} represents the increase of intermolecular and intramolecular hydrogen bonds (Popescu et al. 2013).

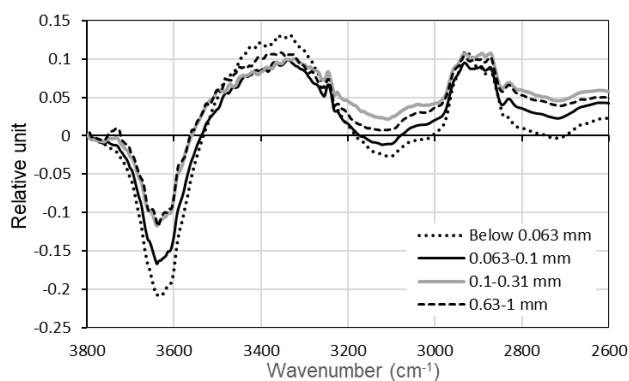


Fig. 6: Difference absorbance spectra of Scots pine powder with different diameters (Carbonyl-methyl region).

The absorption increases in the $2870\text{-}2950\text{ cm}^{-1}$ region represent the increase of symmetric and asymmetric CH stretching in methyl and methylene groups. There are small baseline shifts in both side of the absorption region of methyl and methylene groups.

CONCLUSIONS

Poplar and Scots pine wood was milled and the infrared spectra of the different fractions (between 0.063 and 1 mm) were analysed to determine the chemical changes. The difference spectrum method was used to reveal the breaking of chemical bonds. The calculated difference spectrum contained an intense abnormal positive band around 1050 cm^{-1} wavenumber, elevating the true bands. The shape of this broad band was the same for all fractions of both wood species, but the intensities were different. After removing the abnormal band, the difference spectrum presented the real changes.

The test results showed that the grinding of wood resulted in bond rupture for all ether bond types appearing in the infrared spectrum. The number of conjugated and unconjugated

carbonyl groups in the wood was significantly reduced during milling. During the oxidation and recombination processes which followed the bond breaks, compounds similar to the aromatic structure of lignin were formed, which were shown by increases in absorption at 1514, 1271 and 1231 cm^{-1} .

ACKNOWLEDGEMENT

This article was made in frame of the project TKP2021-NKTA-43 which has been implemented with the support provided by the Ministry of Culture and Innovation of Hungary from the National Research, Development and Innovation Fund, financed under the TKP2021-NKTA funding scheme.

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