THE EFFECT OF TENSION WOOD ON THE SELECTED PHYSICAL PROPERTIES AND CHEMICAL COMPOSITION OF BEECH WOOD (FAGUS SYLVATICA L.)

Tatiana Vilkovská – Ivan Klement – Eva Výbohová

ABSTRACT

The paper is focused on the comparison of selected properties of the tension and normal wood. Beech is wood with high frequency of defects such as red false heartwood, reaction wood (tension wood), dote and so forth. The quality of beech wood is determined according to the structure and properties. The tension wood is considered an important wood defect because it causes negative alterations in solid wood quality and limits an industrial utilization of the wood. Tension wood content in our research was evaluated by the initial longitudinal warping and the woolly appearance of surface. The wood turning blanks of normal and tension wood were selected with a thickness of approximately 50 mm, width of 55 mm, and length of 460 mm. Quantity of 32 woodturning blanks were determined for moisture content and 16 samples for density from two groups tension [TW] and normal wood [NW]. Fourier transform-infrared (FTIR) spectroscopy measurements were carried out using a Nicolet iS10 FTIR spectrometer equipped with Smart iTR attenuated total reflectance (ATR) sampling accessory with diamond crystal (Thermo Fisher Scientific). A resolution of 4 cm\(^{-1}\) and 32 scans per sample was used. Higher variations in average moisture content were in the samples of tension. No significant statistical differences were found between density of tension and normal wood samples measured in fresh state. The cellulose in tension wood has higher degree of crystallinity than in normal wood.

Key words: tension wood, normal wood, FTIR, moisture content, chemical composition of tension wood.

INTRODUCTION

Tension wood has different anatomical and also chemical characteristics than normal (opposite) wood. The tension wood occurs largely in beech wood. KÜDELA and ČUNDERLÍK (2012) stated 14 up to 21 % ratio of tension wood in beech wood. Due to tension wood occurrence, the consequences are shown in form of deformations, increased portion of waste, and decreased quality of final products (KLEMENT, VILKOVSKÁ 2016). KÜDELA and ČUNDERLÍK (2012), YAMAMOTO et al. (2005) studied an influence of tension wood on material moisstness. Authors stated that increased portion of cellulose in tension wood, where by weaker bonding between G-layer and S\(_2\) layer bigger swelling of other layers occurs and therefore, another sorption sites are created, as the main reason of its possible higher moisture content (MC). CHAFE (1990), ARGANBRIGHT et al. (1970),
Coutand et al. (2004) studied the difference in density in fresh state of tension and normal wood. According to Tarmian et al. (2012), Pilate et al. (2004), density of tension wood is about 5 up to 10 % higher than normal wood measured in fresh state. The density in ADS does not need to be different but based on made analysis, the density of tension wood is slightly higher (Kúdela and Čunderlík 2012; Barański et al. 2017, Dzelka et al. 2015, 2016, Dzurenda, Delisky 2012).

Washusen et al. (2001), Clair et al. (2001, 2003) observed that variability in density in oven-dry method is considerable in differently developed tension wood. Different microscopic and sub-microscopic structure influences physical and mechanical properties to a large extent. Clair et al. (2006) described the tension wood chemical composition and claimed that the tension wood is mainly composed of high portion of crystalline cellulose. According to Mattheck and Kubler (1995), due to the composition of the cell wall and its micro and submicroscopic structure, the cell wall can be established as a reinforced matrix, which is mainly composed of polymers and where microfibrils angle gives needed stiffness to increased crystalline cellulose content. The difference in density is conditioned by percentage of G-layer. Jourez et al. (2001) observed the difference in poplar wood (Populus nigra L.). They concluded that increased density is not based only on cell wall thickness but also on cell count and diameter in tension wood.

The physical and mechanical properties of polymers are profoundly dependent on the degree of crystallinity (Mo et al. 1994, Geffertová 2016 et al., Kačík et al. 2016). Kačíková (1997) studied crystallinity of tension and opposite wood and observed the higher portion of crystalline cellulose in tension wood. The measured infrared spectra of cellulose and calculated ratios of absorbance at wavenumbers A1108/1091 cm⁻¹ and A1430/1043 cm⁻¹ are in Tab. 1. It can be concluded that absorbance ratios of cellulose infrared spectra are higher in tension wood than in normal wood. It follows that cellulose crystallinity in tension wood is higher than in opposite. Chemical analysis showed higher percentage of cellulose in tension wood 8–33 % and lower percentage of lignin 19–26 % and pentosanes 16–22 %. Tension wood is characterized by higher average degree of polymerization (DP) of cellulose (Kačíková 1997). Tension wood with highly developed G-layer contains lower percentage of pentosanes and about 10 % more glucoses than normal wood. The more developed the tension wood, the less lignified the G-layer (Kúdela and Čunderlík 2012). Differences in the chemical composition of reaction (tension) and normal wood may be reflected in the chemical process of the raw material. Based on the cited work Kačíková (1997) reported that a lower content of lignin in the material results in a rapid delignification, reducing the time of the pulping and in a lower content of residual lignin in the obtained pulp. The main objective of the research is focused on comparison of selected physical properties and chemical composition of the tension and normal wood.

Tab. 1 Estimation of cellulosic crystallinity (Kačíková 1997).

<table>
<thead>
<tr>
<th>Wavenumbers [cm⁻¹]</th>
<th>TW₁</th>
<th>NW₁</th>
<th>TW₂</th>
<th>NW₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 1108/1091</td>
<td>1.379</td>
<td>1.0552</td>
<td>1.0562</td>
<td>1.0571</td>
</tr>
<tr>
<td>A 1430/1403</td>
<td>1.0392</td>
<td>1.0362</td>
<td>1.0723</td>
<td>1.0708</td>
</tr>
<tr>
<td>A 1459/1403</td>
<td>1.0081</td>
<td>1.0053</td>
<td>1.0159</td>
<td>1.0136</td>
</tr>
</tbody>
</table>
**MATERIAL AND METHOD**

Beech wood (*Fagus sylvatica* L.) was used for experimental measurements. Samples were chosen from beech logs with the size of 400 mm in diameter and 2,000 mm in length from Kronotimber Ltd. (*Lehota pod Vtáčnikom, Slovakia*). It is most produced diameter in this company. Logs without visible defects such as red false heartwood and dote were selected. The tension wood content were evaluated by the initial longitudinal warping and the woolly appearance of surface (Fig. 1).

![Fig. 1 Initial longitudinal warping and the woolly appearance surface of tested timbers.](image)

The test timbers of normal and tension wood were selected with a thickness of approximately 50 mm, width of 55 mm, and length of 460 mm. There were 32 test timbers used in the dry kiln from two groups Tension [TW] and Normal [NW] wood.

From the selected test timber, samples were cut to 20 mm in length (Fig. 1) and prepared for measured moisture content [MC].

The gravimetric method was used to determine the moisture content (MC). The moisture content of the wood samples was determined before and after drying as well as after the steaming process. The moisture content was calculated using Eq. 1,

\[ MC = \frac{m_w - m_0}{m_0} \times 100[\%] \]  

(1)

Where: \( m_w \) is *the weight of moisture sample* (g) and \( m_0 \) is *the weight of oven-dry sample* (g).

Density in the fresh state was determined on every second sample of the tension timber and normal (16 samples) according to EN 49 0108. The measurement was carried out under laboratory conditions. Density in fresh state was calculated using Eq. 2,

\[ \rho_w = \frac{m_w}{V_w} \text{ [kg.m}^{-3}\text{]} \]  

(2)

Where: \( m_w \) is *the weight of moisture sample in fresh state* (kg) and \( V_w \) is *the volume of moisture sample in fresh state* (m³).

Fourier-transform infrared spectroscopy (FTIR) is very useful analytical method for analysing the structure of wood constituents. Attenuated total reflectance (ATR) is non-destructive method enabling to observe the differences in wood chemical composition, and structure of wood constituents at different sample sites. These facts made us use the ATR-FTIR to compare the chemical structure of tension and opposite wood.

Fourier transform-infrared (FTIR) spectroscopy measurements were carried out using a Nicolet iS10 FTIR spectrometer equipped with Smart iTR attenuated total reflectance.
(ATR) sampling accessory with diamond crystal (Thermo Fisher Scientific). Spectra were measured in the wavenumber range from 4,000 up to 650 cm\(^{-1}\). A resolution of 4 cm\(^{-1}\) and 32 scans per sample was used. Six measurements were performed per sample (Fig.2). Spectra were evaluated using the OMNIC 8.0 software (Thermo Fisher Scientific). From obtained spectra, the average spectra for each sample were created and evaluated.

Normalization was performed on the absorption band at 1,033 cm\(^{-1}\). Measured FTIR spectra were used to estimation, of chemical composition and cellulose crystallinity in tension and normal wood.

Following ratios were used for cellulose crystallinity estimation:

- \(\frac{A_{1370}}{A_{2900}}\) (TCI) – total crystallinity index (O’CONNOR et al. 1958)
- \(\frac{A_{1424}}{A_{898}}\) (LOI) – lateral order index (O’CONNOR et al. 1958)
- \(\frac{A_{1335}}{A_{1315}}\) (COLOM et al. 2003)

Fig. 2 a.) Samples for FTIR analyses, b.) Apparatus Nicolet iS 10 (Thermo Fisher Scientific).

RESULTS AND DISCUSSION

Mean values of moisture content and elemental statistical characteristics for tension and normal samples are shown in Tab. 2. Moisture content range from 41.7–69.5 % for normal wood samples and from 43.6–81.6 % in tension wood samples, respectively. The ranges had higher values in tension wood (TW). Equally, the average final moisture content was about 0.52 % higher in tension wood, what can be considered a negligible difference in practice (production conditions). Analysis of MC by basic statistical characteristics two-factor t-test confirmed that there were no considerable statistical changes between measured differences between tension and normal wood.

<table>
<thead>
<tr>
<th></th>
<th>NW</th>
<th>TW</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>58.161</td>
<td>58.686</td>
</tr>
<tr>
<td>Variance</td>
<td>48.223</td>
<td>92.715</td>
</tr>
<tr>
<td>Observations</td>
<td>32000</td>
<td>32000</td>
</tr>
<tr>
<td>Hypothesized Mean Difference</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>df</td>
<td>56000</td>
<td></td>
</tr>
<tr>
<td>t Stat</td>
<td>-0.250</td>
<td></td>
</tr>
<tr>
<td>P(T\leq t) one-tail</td>
<td>0.402</td>
<td></td>
</tr>
<tr>
<td>t Critical one-tail</td>
<td>1.673</td>
<td></td>
</tr>
<tr>
<td>P(T\leq t) two-tail</td>
<td>0.803</td>
<td></td>
</tr>
<tr>
<td>t Critical two-tail</td>
<td>2.003</td>
<td></td>
</tr>
</tbody>
</table>

Tab. 2 t-Test Two-Sample for Variances of Moisture content (NW and TW).
The dependence of MC of two compared samples groups, tension and normal, were evaluated by inductive statistics using the probability value (p < 0.05). In both cases, p-value exceeded by 0.05 and therefore, there was no remarkable difference between compared samples, what is confirmed by Fig. 3 also showing non-significant difference. The present observations are consistent with those of POLETTO et al. (2012), VILKOVSKÁ et al. (2016), TARMIAN et al. (2012), which reported that tension wood can have higher MC values in some cases. Contrarily, DAVIS et al. (2002) observed lower values of MC of tension wood in comparison with normal wood.

Fig. 3 The dependence of MC of two compared samples groups (NW and TW).

Based on the research studies of KŮDELA and ČUNDERLÍK (2012), authors measured higher MC of tension wood, where they considered structure of tension wood, its chemical composition, as well as binding between G-layer and S2 layer as the cause. It is necessary to point out that tension wood has slightly higher value of fibre saturation point. The value of tension wood is 31.0–33.1 % and for normal wood is 28–30.06 %. Eventually, according to our measurements and measurements of other authors, we can conclude that MC of tension wood does not have to be considerable higher and everything depends on proportion and development of tension wood in the material. The mean value of density ρw and elemental statistical characteristics of tension and normal samples are in Tab. 3. Density in fresh state at definite MC varied in range 957–972 kg·m⁻³ and 970–996 kg·m⁻³ of tension and normal wood, respectively. The densities in the fresh state were measured at moisture content of approx. 78 %. Differences between tension and normal wood were measured; however, higher values of density tension and normal samples were reported. The differences were also measured by authors TARMIAN et al. (2012) and PILATE et al. (2004), who stated higher density value about 5–10 %. Our findings did not confirm higher density in fresh state and final measurements rather were of the opposite nature. JOUREZ et al. (2001) stated that the density can depend on ratio of cells in tension wood. PLACET et al. (2007), ARAKI et al. (1983) stated that portion of tension wood depends mainly on G-layer in material and its quantitative and qualitative representation.

Some other studies measured the change in density for a gradual change in severity of tension wood. Severity was expressed as the percentage of gelatinous layers in the wall (which is of course only applicable to species which have a G-layer) or was measured using release growth-strain measurement. Where the amount of G-layer was measured, a positive
correlation was sometimes found between density and tension wood fibre percentage (KROLL et al. 1992; WASHUSEN et al. 2001; CLAIR et al. 2001) although ARGANBRIGHT et al. (1970) showed no correlation between density of TW and NW.

Tab. 3 t-Test: Two-Sample Assuming Unequal Variances for Density in fresh state (NW and TW).

<table>
<thead>
<tr>
<th>Samples</th>
<th>NW</th>
<th>TW</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>977.82</td>
<td>970.78</td>
</tr>
<tr>
<td>Variance</td>
<td>442.17</td>
<td>259.94</td>
</tr>
<tr>
<td>Observations</td>
<td>16.00</td>
<td>16.00</td>
</tr>
<tr>
<td>Hypothesized Mean Difference</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>df</td>
<td>28.00</td>
<td></td>
</tr>
<tr>
<td>t Stat</td>
<td>1.06</td>
<td></td>
</tr>
<tr>
<td>P(T≤t) one-tail</td>
<td>0.15</td>
<td></td>
</tr>
<tr>
<td>t Critical one-tail</td>
<td>1.70</td>
<td></td>
</tr>
<tr>
<td>P(T≤t) two-tail</td>
<td>0.30</td>
<td></td>
</tr>
<tr>
<td>t Critical two-tail</td>
<td>2.05</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 4 The dependence of density of fresh state of two compared sample groups (NW and TW).

Figure 5 shows the ATR-FTIR spectra of tension and normal wood samples. Marked absorption bands were used for estimating the cellulose crystallinity and comparison of chemical composition. They are assigned to functional groups, respectively fragments of molecules as follows (COLOM et al. 2003; HON et al. 2001; PANDEY, PITMAN 2003, GEFFERT et al. 2017): 3,344 cm⁻¹ for OH stretching; 2,900 cm⁻¹ for C–H stretching in –CH₂ and CH₃; 1,734 cm⁻¹ for C=O stretching of acetyl or carboxylic acid; 1,504 cm⁻¹ for C=C stretching of the aromatic ring in lignin; 1,424 cm⁻¹ for C–H deformation in lignin and carbohydrates; 1,370 cm⁻¹ for C–H deformation in carbohydrates; 1,335 cm⁻¹ for OH in plane bending in cellulose; 1,316 cm⁻¹ for CH₂ wagging in cellulose; 1,236 cm⁻¹ for syringyl ring and C–O stretch in lignin and xylan; 1,033 cm⁻¹ for C-O vibration in polysaccharides and 898 cm⁻¹ for C–H deformation in cellulose.

Differences in intensities and shapes of absorption bands can be observed by comparison of measured FTIR spectra resulting from differences in chemical compounds and their different structure in tension and normal wood (Tab. 4). Characteristic absorption band of hemicelluloses is band at wavenumber 1,734 cm⁻¹, which result in carboxyl group vibrations of xylan and band at 1,236 cm⁻¹, which assigned to CO in xylan. The intensity of
absorption band in tension wood samples is lower at both wavenumbers, which suggests a lower content of xylan type hemicelluloses in these samples.

The ratio of the lignin and polysaccharides content in wood is reflected in the use of the ratio of absorbances $A_{1504}/A_{1369}$ (PANDEY, PITMAN 2003). In both cases the ratio is lower in samples of the tension timber.

Fig. 5 FTIR spectra of normal (NW) and tension (TW) beech wood.

Tab. 4 Values of absorbance at selected wavenumbers used for comparison of chemical composition of tension and normal wood samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$A_{1734}$</th>
<th>$A_{1236}$</th>
<th>$A_{1504}/A_{1369}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NW₁</td>
<td>0.311</td>
<td>0.330</td>
<td>1.19</td>
</tr>
<tr>
<td>TW₁</td>
<td>0.292</td>
<td>0.314</td>
<td>1.12</td>
</tr>
<tr>
<td>NW₂</td>
<td>0.293</td>
<td>0.326</td>
<td>1.17</td>
</tr>
<tr>
<td>TW₂</td>
<td>0.257</td>
<td>0.284</td>
<td>1.15</td>
</tr>
</tbody>
</table>

The results of the FTIR analysis are consistent with the results of chemical analysis of tension and normal wood, published by KAČÍKOVÁ (1997).

In the cited paper the author presents a lower proportion of lignin and pentosane and a higher percentage of $\alpha$-cellulose and cellulose Kürschner-Hoffer in the tension timber as compared to a normal wood. FTIR spectral results can be compared to the content in amorphous and crystallised I cellulose, respectively, the lower value of absorbance ratio $A_{1335}/A_{1315}$ signifies a higher crystallinity (COLOM et al. 2003). In addition to this ratio, in order to estimate the crystallinity of the tension and the normal wood we calculate other two parameters: a TCI and LOI. The TCI is proportional to the overall degree of order in the cellulose; the LOI represents the ordered regions of cellulose perpendicular to the chain direction (POLETTO et al. 2012; ŠIROKÝ et al. 2010).

Tab. 5 Absorbance ratios for comparison of cellulose crystallinity in tension and normal wood samples.

<table>
<thead>
<tr>
<th>Absorbance ratio</th>
<th>NW₁</th>
<th>TW₁</th>
<th>NW₂</th>
<th>TW₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>TCI ($A_{1730}/A_{2900}$)</td>
<td>0.726</td>
<td>0.765</td>
<td>0.772</td>
<td>0.777</td>
</tr>
<tr>
<td>LOI ($A_{1422}/A_{808}$)</td>
<td>1.417</td>
<td>1.816</td>
<td>1.655</td>
<td>1.698</td>
</tr>
<tr>
<td>$A_{1335}/A_{1315}$</td>
<td>0.985</td>
<td>0.614</td>
<td>0.492</td>
<td>0.388</td>
</tr>
</tbody>
</table>
The values obtained for all three followed parameters (Tab. 5) indicate that higher proportion of crystalline cellulose than in normal is in the tension wood. The present observations are consistent with the results of the research presented by Kačíková (1997).

CONCLUSIONS

Considering the high proportion of beech wood in our country, the way of its use is nowadays the subject of quality. The quality of beech wood is determined based on structure and properties of wood, frequency of defects in this material.

Tension wood affects the technological properties of wooden materials since it has different physical, anatomical, and chemical characteristics in comparison with normal wood. In this study a significant difference in the selected physical properties (density and moisture) was not confirmed.

On the basis of the measurements, we can conclude:

- The tension specimen showed bigger variances of MC mean values. However, measured differences were assessed by statistical analysis as insignificant.
- The differences in the MC values between compared groups (0.52 %) can be considered negligible.
- No significant statistical differences were found between density of tension and normal samples measured in fresh state.
- Lower ratio of lignin to polysaccharides and lower amount of hemicelluloses compared to the normal wood is in the tension wood. It can be supposed that tension wood has positive effect on delignification process.
- The cellulose in tension wood has higher degree of crystallinity than in normal wood.

LITERATURE


ACKNOWLEDGEMENTS

The research is carried out thanks to funding from VEGA Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic No. 1/0395/16.

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