Structural Differences between Reaction Wood and Opposite Wood with Different Drying Temperatures

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Reaction wood is characterized by having different anatomical and chemical features than normal wood. The different composition of cell walls, the higher quantitative proportion of thick-wall fiber cells, diameter, and the abundance of vessels have remarkable effects on reaction wood's physical and mechanical properties. Reaction wood has fewer vascular cells. In addition, it has a smaller lumen diameter, which results in reduced permeability. Therefore, reaction wood is more difficult to dry at a certain moisture content. The differences in the drying times of the reaction wood and the normal wood were largest at a temperature of 60 °C and durations greater than 30 h, and the reaction wood dried more slowly. At a temperature of 120 °C, the differences in drying time were minimalized, and drying end times were almost identical. The expected negative effect of higher temperature on the morphology of reaction wood and opposition wood was not confirmed.

Keywords: G-layer; Reaction wood; Tension wood; Morphology of tension wood; Drying tension wood

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INTRODUCTION

Beech wood (*Fagus sylvatica* L.) is the most common wood species in the Slovak Republic (Reinprecht 2016; Vilkovský and Čunderlík 2017). Reaction beech wood (tension wood) has different anatomical and chemical characteristics from normal or opposite wood. Tension wood is a defect in the structure of wood that develops during tree growth in the area of the trunk that is stressed by tension forces from different external factors (Okuyama *et al.* 1994; Kúdela and Čunderlík 2012). Reaction wood is a common defect in beech wood. Kúdela and Čunderlík (2012) reported a 14% to 21% proportion of reaction wood in beech wood. Due to the presence of reaction wood, deformations, higher proportions of waste, and lower final product yield occur. In fact, the difference in the drying rate curves of reaction wood and opposite wood gradually decreases when the drying process progresses to the bound water domain. The analysis of mass diffusivity and density in beech indicates that the diffusion of bound water is relatively easy in tension wood, which is consistent with the structure of the G-layer. The differences are noticeable in the microscopic structure of wood (Vilkovská *et al.* 2018). The different composition of cell walls and the higher quantitative proportion of thick-wall fiber cells, diameter, and the abundance of vessels have noticeable effects on the physical and mechanical properties of reaction wood (Meloche *et al.* 2006). Table 1 shows reported values of anatomical characteristics of tension and normal beech wood (Čunderlík and Hudec 2002).

Čunderlík and Hudec (2002) confirmed the presence of differences in vascular porosity of beech wood in different annual rings. The annual ring of normal wood had a vascular porosity of 30.3%, and the annual ring with a high proportion of reaction wood

had a vascular porosity of 19.9%, which is almost 33% lower. This difference in the vascular porosities of normal and reaction wood considerably affected the permeability of beech wood in individual zones. The difference in the composition of the cell wall was particularly remarkable.

Table 1. Anatomical Characteristics of Tension Wood and Normal Wood (*Fagus sylvatica* L.) Čunderlík and Hudec (2002)

	Diameter of Vessels (µm)	Diameter Bordered Pits (µm)	Quantity of Vessels (mm^2)
Tension Wood	31.7 ± 5.1	3.5 to 4.3 ± 0.8	51
Normal Wood	55.2 ± 15.8	4.9 to 6 ± 1.1	98

Figure 1 shows the differences in the cell walls of normal wood and reaction wood. The secondary cell wall of normal wood consists of S_1, S_2, and S_3 layers, whereas the S_3 layer is replaced by a gelatinous layer (G-layer) in reaction wood. The G-layer is thicker than the S_2 layer.

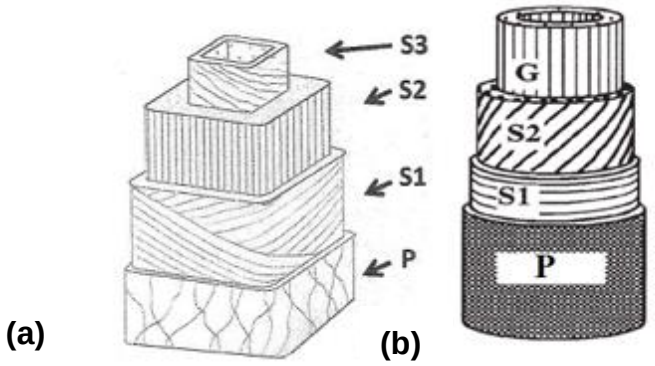

Fig. 1. The cell wall structure of normal wood (a) and tension wood (b) (Clair *et al.* 2006)

The G-layer has been the subject of number of studies (Norberg and Meier 1966; Araki *et al.* 1983; Clair *et al.* 2003, 2006; Kampf *et al.* 2017). These authors compared the chemical composition of normal wood with the chemical composition of the G-layer. Their results revealed that the G-layer is composed of a high portion of crystalline cellulose and it is non-lignified (Norberg and Meier 1966; Clair *et al.* 2003). Due to the parallel orientation of cellulosic microfibrils (0° to 5°) with the cell axis and the absence of lignin, it is highly predisposed to high transverse shrinkage. This shrinkage in the drying process results in the separation of the G-layer from the outer layers of S_1 and S_2. Norberg and Meier (1966) report that the quality and quantity of the G-layer has considerable influence on reaction wood properties. Their analysis of the chemical composition of reaction wood found that tension wood consists mainly of crystalline cellulose (Norberg and Meier 1966). In addition, Čunderlík *et al.* (1995) stated that the composition of the cell wall and its microscopic and submicroscopic structure make it a strengthening matrix that is composed mostly of polymers, and the angle of microfibrils provides the necessary strength to increase the crystalline cellulose content. The shrinkage and swelling of tension wood is more prominent in the longitudinal direction, which results in higher values of longitudinal contraction Čunderlík *et al.* (1995).

Further, Norberg and Meier (1966) and Placet *et al.* (2006) investigated the higher longitudinal shrinkage of reaction wood, and their aim was to search for more pronounced differences in shrinkage in layers other than the G-layer. A more detailed explanation of

tension wood's higher shrinkage in the longitudinal direction was reported by Čunderlík (1997) and Fang *et al.* (2008). The observation and analysis of the cell wall layer composition revealed that the G-layer is characterized by high transverse shrinkage. Because of this shrinkage and the weak bonds between the G and S_2 layers, the G-layer separates from the S_2 layer. This separation allows the S_1 layer and the S_2 layer to shrink more in the transverse direction. The thickness of the G-layer in cell walls varies. Fang *et al.* (2008) found that the cell lumen size of reaction wood increased due to drying, but lumen size decreased for normal wood (Fig. 2).

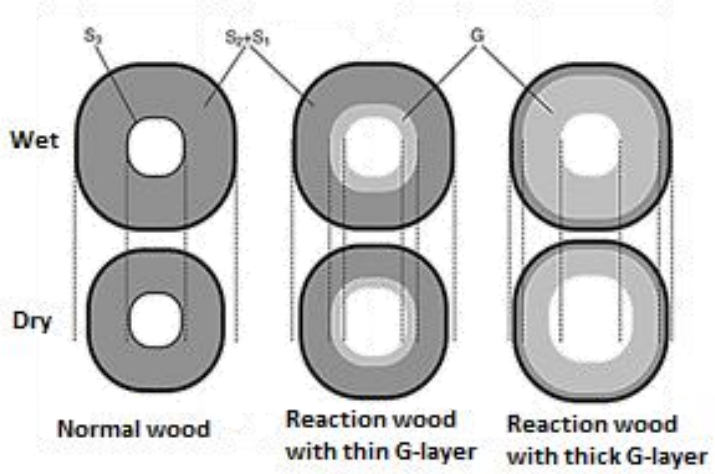

Fig. 2. The shrinkage patterns of normal and reaction wood (Fang *et al.* 2008)

Both the longitudinal shrinkage and volumetric shrinkage of reaction wood is higher. However, volumetric shrinkage is less sizable than shrinkage in the longitudinal direction. The nonlignified G-layer leads to easily deformable cell walls, which results in lower compression strength for the tension reaction wood (Placet *et al.* 2006; Fang *et al.* 2008).

The aim of this paper was to analyze the influence of temperature 60 and 120 °C on the structural differences of reaction and opposite wood.

EXPERIMENTAL

Materials

Beech logs were selected from the Michalková – Burzovo (550 m above sea level) forests of the University Forest Enterprise of the Technical University in Zvolen, Slovakia. The selection of suitable logs containing reaction wood was made by considering log curvature, the presence of exocentrically situated pith, the use of chemical reagents, and the pearlescent coloring of the cross-section after drying.

The selected beech logs had a diameter of 45 ± 1 cm at the wide end and a length of 2 m. The beech logs did not have red false heartwood or other visible defects that could negatively affect the measurement results.

Four beech logs were used for measurement. In the laboratory experiments, it was necessary to maintain the greatest match of properties between the two types of beech wood (reaction and opposite) compared. After identifying the reaction wood zone, two groups of samples containing reaction wood (R_1 and R_2) and two groups of samples containing opposite wood (O_1 and O_2) were cut from the zone (Fig. 3).

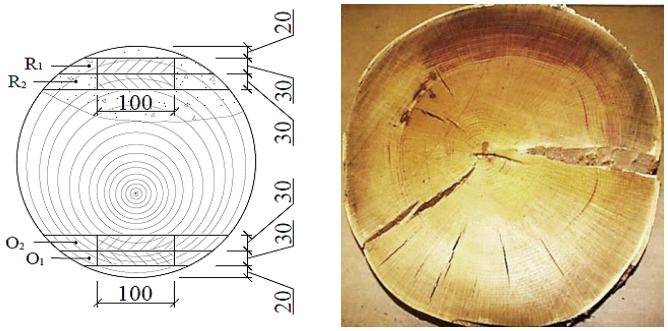

Fig. 3. The selection process of two groups of samples containing reaction wood (R_1 and R_2) and two groups containing opposite wood (O_1 and O_2); the dimensions of the drying samples were 100 mm × 300 mm × 30 mm (w × l × t)

Opposite wood was located on the side opposite to the reaction wood on the cut discs (Fig. 3). The samples had a thickness of 30 mm, a width of 100 mm, and a length of 400 mm. The fronts of the samples were treated with a chemical reagent (modified chloro-zinc-iodine chemical reagent) from both sides by brush to allow clearer identification of the reaction wood (red color) in the sample (Fig. 4). Subsequently, the zone treated with the chemical reagent was cut 50 mm from each side before the drying process.

Fig. 4. The front of the samples were treated with chemical reagents

The samples were dried in a Memmert HCP 108 laboratory kiln (Memmert GmbH + Co. KG, Schwabach, Germany). The drying was conducted at temperatures of 60 °C and 120 °C. Additional information about the drying mode is shown in Table 2. A constant flow velocity of the drying medium (2 ± 0.2 m · s^{-1}) was used for drying. Warm-air drying process was used by constant drying conditions above and under FSP temperature 60 °C and relative humidity 91 ± 0.5%.

The process of high temperature drying was divided into two phases. The first phase was heating, where the drying temperature was raised to 90 °C and the relative humidity was 94 ± 0.5%. These parameters were maintained in the first phase of the drying process until the moisture content in the samples did not decrease below the FSP (free water domain) approximately 30%. After decreasing the moisture content below the fiber saturation point in both samples, the temperature of the dry bulb increased up to 120 °C and the relative humidity was without regulation (bound water domain). The last part of drying schedule was cooling to approximately 20 °C. The moisture content (MC) was measurements on all the samples, both before and after the drying process, according the norm STN EN 49 0103. The average final moisture content was 10% ± 2%.

The oven-dry density was determined for every sample with reaction wood and opposite wood, and measurement was performed under laboratory conditions. The total number of samples was 32 pieces. The oven-dry density was calculated according to STN 490 108 (1993). The preparation of samples for scanning electron microscopy was as follows; the 1×1×1 cm wood blocks from both opposite and reaction wood were immersed in water at 20 ° C for 7 days to soften the wood. In order to obtain a smooth section, the

cross section was subsequently cut with a GSL1-microtome (Eidg. Forschungsanstalt WSL, Birmensdorf, Switzerland). Prepared samples were subsequently vacuum dried dusted with gold on a Q150R ES sputter (Quorum Technologies Ltd., Laughton, England). The prepared surface of the preparation was observed using a MIRA 3 electron microscope (Tescan Orsay Holding, Brno, Czech Republic). A secondary electron detector was used and an acceleration voltage of 15 kV, a working distance of 8 mm, and a spot size of 4 nm were set. The exported images have not been modified in any way. Samples were taken at 50 μm and 10 μm.

RESULTS AND DISCUSSION

The parameters of drying modes, average density, and moisture content are shown in Table 2, which also shows the values of drying time with different drying modes and samples.

Table 2. Parameter of Drying Modes (*relative humidity, temperature, and drying time*), MC and Density

Samples	Drying Mode (°C)	φ (%)	$\varnothing\ \rho_0$ (kg·m^{-3})	MC Before (%)	MC After (%)	τ (h)
Reaction	60	91	668	81.6	12.00	200
Opposite	60		662	82.7	10.44	170
Reaction	120	94	659	80.3	9.77	165
Opposite	120		649	76.9	8.47	163

MC: Moisture content; τ: drying time; φ: relative humidity; $\varnothing\ \rho_0$: average oven-dry density

The differences in drying times of the reaction wood and the opposite wood were most substantial at a temperature of 60 °C. The difference was approximately 30 h, and the reaction wood dried more slowly. At 120 °C, the differences were equalized, and the drying end times were almost identical. One possible explanation could be that the nanostructure of the G-layer, which is mesoporous (contains pores between 2 nm and 50 nm in size), allowed the migration of bound water more easily at 120 °C. Different density values were measured in the oven dry-state of the reaction wood samples, and the values for reaction wood were higher than opposite wood samples. Variability of density in dry state can be caused by different quantitative and qualitative representations of thick-walled fibers in the gelatinous layer. Based on the results of Čunderlík (1997), the microscopic and chemical structure of the cell wall of tension wood is responsible for the differences in physical properties to those of opposite wood. The differences were also measured by Tarmian *et al.* (2012) and Pilate *et al.* (2004), who reported higher density values of approximately 5% to 10%.

A 50 μm sample of reaction wood at 60 °C is shown in Fig. 5. Reaction wood is typically composed of high fiber cells with a thick G-layer ratio, and more irregular peripheral and perpendicular cracks appear in the cell wall. The cells lumen is irregular and smaller, and cracks in the compound middle lamella are less visible in the reaction wood.

The results, which were in concordance with Čunderlík and Hudec (2002), showed that the non-lignified G-layer was likely to have deformable cell walls, which reduced the compressive tensile strength of reaction wood and irregular lumen. Microscopic observations found that reaction wood had fewer vascular cells. The smaller lumen

diameter of reaction wood led to reduced permeability. Therefore, reaction wood is more difficult to dry at a certain moisture content.

This was also confirmed by the results of drying (Table 2), which found that the difference in the drying of reaction wood and opposite wood was most remarkable at 60 °C. The cause of slower drying could have been the low porosity of the wood. The longer drying time of the tension wood during free water removal may have been associated with the specific bordered pith structure in the cell walls and its smaller diameters. At a 120 °C drying temperature, differences were less remarkable and almost equal.

The results suggest that bound water diffusion is relatively easy in the G-layer of tension wood. This could be due to the chemical composition of this layer, which consists mostly of cellulose and contains almost no lignin (Gardiner *et al.* 2014). The present observations are consistent with those of Vilkovská *et al.* (2018), who reported that the tension wood had a lower ratio of lignin to polysaccharides and a lower amount of hemicellulose than opposite (normal) wood. The cellulose in tension wood had a higher degree of crystallinity than that in normal wood.

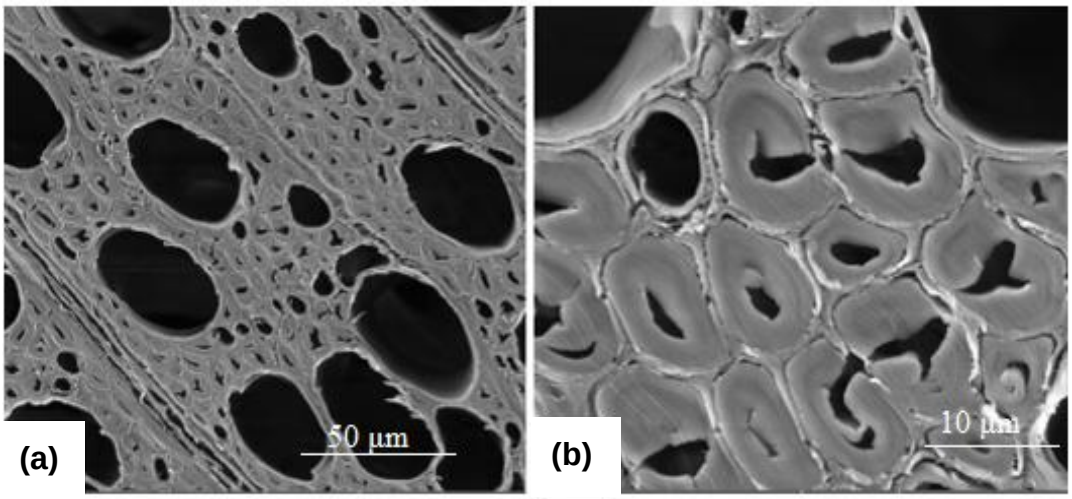

Fig. 5. Reaction wood at temperature 60 °C: 50 µm (a) and 10 µm (b)

The results of this study provide support for other findings on this topic (Norberg and Meier 1966; Araki *et al.* 1983; Clair *et al.* 2003, 2006). These authors claim that due to the almost parallel orientation of the cellulose microfibrils (0° to 5°) with the fibrous cell axis and the absence of lignin, reaction wood is expected to undergo large transversal shrinkages, which are cause of tearing the G-layer from the layer S_2 in the drying process. By comparing images of reaction wood and opposite wood at a temperature of 60 °C and an identical scale of 50 µm, opposite wood (Fig. 5a) was found to have fewer fibrous cells (Norberg and Meier 1966; Araki *et al.* 1983; Clair *et al.* 2003, 2006).

Compared to the reaction wood, opposite wood had more vascular cells, which led to better liquid permeability and faster reaching of the required final moisture. Results are also in agreement with Tarmian *et al.* (2012), who reported that the proportion of vessels in reaction wood is 21.8% and that of opposite wood is 32.9%. A temperature of 60 °C caused small drying cracks, but the cell wall structure was not excessively damaged. Figure 6b shows the opposite wood at a 10 µm scale, and thinner cell walls and larger lumens can be seen. The cracks are oriented mainly through the bordered pits.

A temperature of 120 °C was also used to compare the morphology of the reaction wood and opposite wood. Microscopic preparations of reaction wood are shown in Fig. 7. The reaction wood released drying stresses into the cell wall and G-layer but not into the composite middle lamella. The G-layer tore perpendicular to the perimeter of the cell,

which was reflected in the irregular shape of the lumen. The negative effect of the higher temperature was not confirmed.

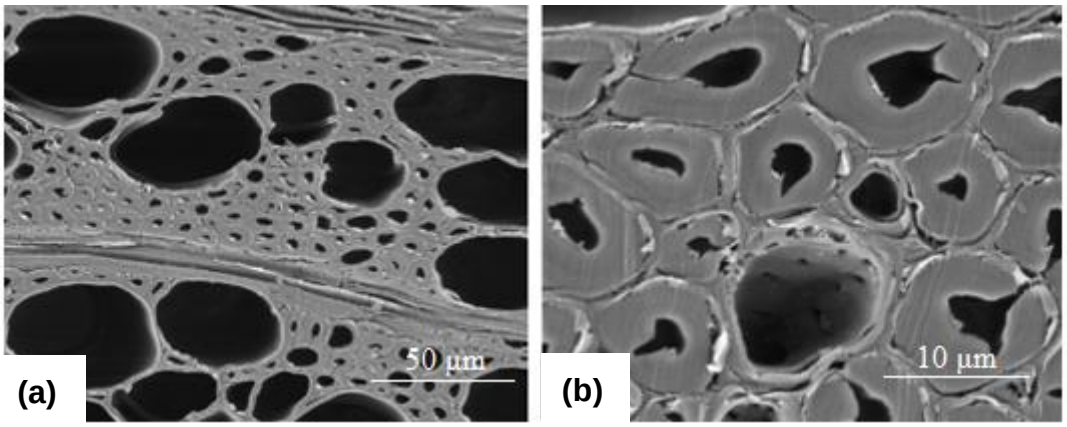

Fig. 6. Opposite wood at temperature 60 °C: 50 µm (a) and 10 µm (b)

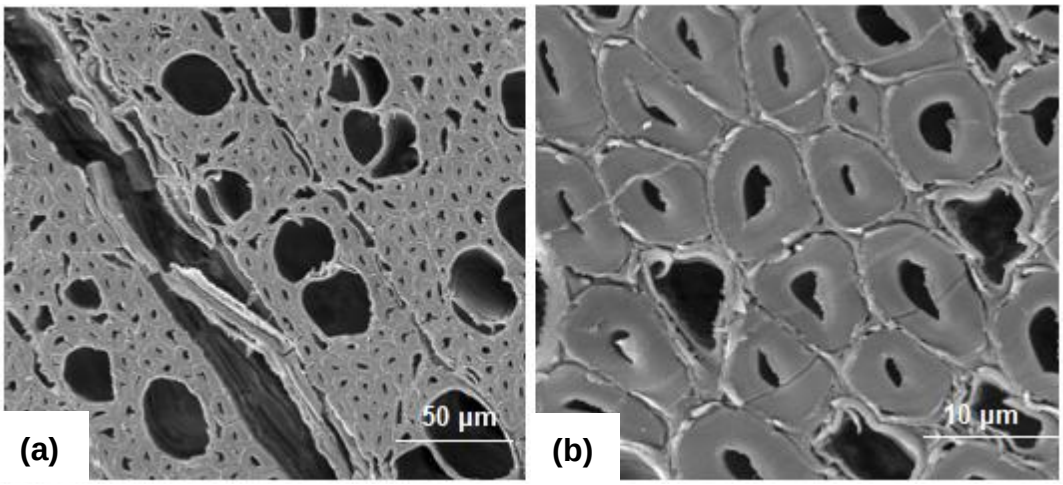

Fig. 7. Reaction wood at temperature 120 °C: 50 µm (a) and 10 µm (b)

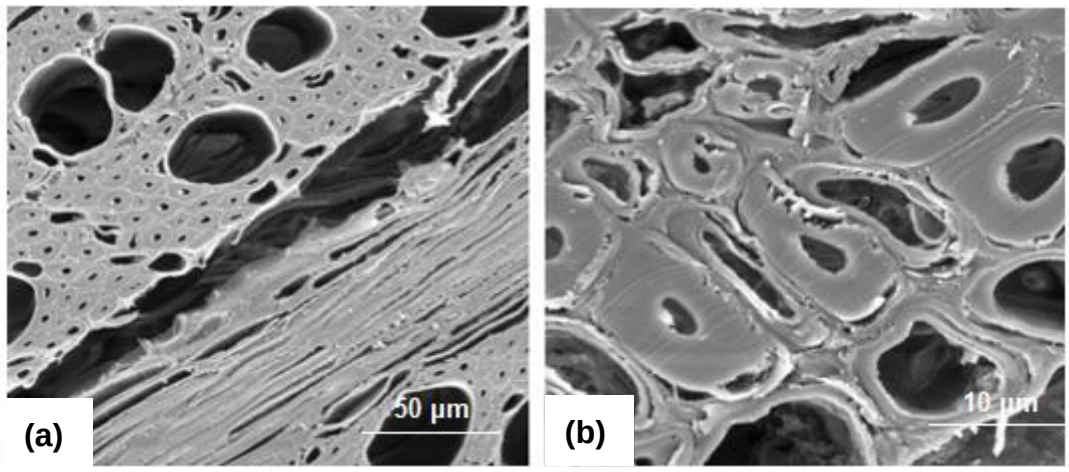

Fig. 8. Opposite wood at temperature 120 °C: 50 µm (a) and 10 µm (b)

One possible explanation is that the G-layer shown in Fig. 7 was poorly developed. Okuyama *et al*. (1994), Fang *et al.* (2008), Vilkovská *et al.* (2018), and Kučerová *et al.*

(2019) stated that the weakly developed reaction wood G-layer is less thick than the strongly developed reaction wood. Another characteristic of weakly developed reaction wood is that the G-layer is found only in some fibrous cells. The present observations are consistent with those of Vilkovská *et al.* (2018) and Gardiner *et al.* (2014), who reported that the thickness of the individual layers of the secondary wall varies from the middle to the ends of the fibrous cells as follows G-layer and layer S_2 become thinner, but the thickness of layer S_1 does not change. When analyzing the opposite wood (Fig. 8a, Fig. 8b), drying cracks were observed predominantly in the compound middle lamella at a drying temperature of 120 °C. At 60 °C, these cracks were less pronounced.

CONCLUSIONS

1. Higher density values were observed in the oven-dry state for the reaction wood samples. Variability in density in a dry state can be caused by different quantitative and qualitative representations of thick-walled fibers with a gelatinous layer.
2. The differences in the drying time of the reaction wood and the opposite wood were most noticeable at a temperature of 60 °C. The difference in drying time was 30 h and the reaction wood dried more slowly. The differences were equalized, and the drying end times were almost identical at 120 °C.
3. At 120 °C, the reaction wood did not release the drying stresses into the compound middle lamella, but it did release them into the cell wall and the G-layer. The G-layer tore perpendicularly to the perimeter of the cell, which was reflected in the irregular shape of the lumen.
4. Opposite wood cell lumens retained their oval shape at temperatures above 100 °C. The quality of the reaction wood was maintained.
5. The negative effect of higher temperature on the morphology of reaction wood and opposition wood was not confirmed.

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