

# Properties of Modified Wood According to Treatment Technology and Thermo-Vacuum Process for Birch (*Betula pendula* Roth) Veneers

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Thermally modified birch (*Betula pendula* Roth) veneers that had been subjected to wood treatment technology (WTT) or thermo vacuum (TV) processes were compared in this study. After modification of veneers in the range of temperatures from 160 °C to 218 °C and times from 0.5 h to 3 h, the color, mass loss, density, tensile strength, hygroscopicity, and decay resistance against brown rot fungus *Coniophora puteana* were determined. Treatment regimes with the greatest mass loss were at 217 °C for 3.0 h in TV (7.8%) and 160 °C for 0.8 h in the WTT (6.7%). As expected, wood mass loss correlated well with moisture exclusion efficiency (MEE) in all relative humidity (RH) environments ( $r = 0.95$  to  $0.99$ ). Strength loss in the WTT was considerable compared to the TV process (57% and 40%, respectively). The resistance against brown rot fungus was moderate with a mass loss of 12% to 33%. Among the investigated samples, the regime 217/3.0/TV showed the best resistance against brown rot fungus and acceptable other properties.

**Keywords:** Birch; Thermal modification; Wood treatment technology; Thermo vacuum treatment; Decay resistance

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## INTRODUCTION

With an increasing awareness of global climate change and greenhouse gas emissions, among which CO<sub>2</sub> is the crucial one, there is increasing interest in wood materials that can accumulate and store carbon dioxide. The longer the wood material can be used, the longer it stores carbon; therefore several techniques to prolong the life span of wood materials have been developed (Hildebrandt *et al.* 2017).

Plywood is an engineered cross-glued wood material made from peeled veneers. World production of plywood reached 107.4 million m<sup>3</sup> in 2017 (Raute 2017). The properties of plywood are mainly determined by the characteristics of veneers such as wood species and technological features (Gilbert *et al.* 2017). Veneer swells in a humid environment and shrinks when it is drying, which can provoke internal stresses in plywood and also its delamination.

Wood thermal treatment at temperatures of 160 °C to 240 °C is the most acceptable industrial method for wood modification. The wood thermal modification increases the dimensional stability of veneer by increasing its hydrophobicity. Chemical changes in wood due to the thermal modification depend on the duration of treatment and temperature.

Wood species, temperature, presence of oxygen in a reaction environment, and process duration are the main factors that determine the properties of the modified wood (Esteves and Pereira 2009; Sandberg *et al.* 2017). The thermal modification processes are mainly carried out in a dry environment, in inert gas, or a moist environment (Sandberg and Kutnar 2016; Sandberg *et al.* 2017). There are several commercial treatments, among which Thermowood® is the most popular (Shi *et al.* 2007). WTT is classified as a closed method that operates at lower temperatures (Irbe *et al.* 2017).

Organic acids formed from hemicellulose during thermal treatment cause hydrolysis of wood cells and wall carbohydrates, as well as lignin regrouping that affects chemical and physical properties of wood (Gérardin 2016). If these decomposition products are promptly evacuated by vacuum, the process lowers wood degradation (Candelier *et al.* 2013), especially in the case of thick veneers (Sandak *et al.* 2016). Several commercial processes use vacuum, such as Silvapro® (Rep *et al.* 2012), SmartHeat® (Van Acker *et al.* 2011), and Termovuoto® (Ferrari *et al.* 2013).

Silver birch is a widely used wood species in Latvia for plywood and furniture manufacturing as well as a raw material for the pulp and paper industry; it has limited biological durability and dimensional stability. By decreasing the drawbacks, the usage of silver birch could be widened, and thermal treatment is a methodology that increases biological durability and dimensional stability. Silver birch has been treated in WTT process (Grinins 2016) but has not been widely investigated in the thermo-vacuum (TV) process.

The objective of the present research is the comparison of thermally modified (*Betula pendula* Roth) veneers treated in the WTT and TV processes.

## EXPERIMENTAL

### Materials and Methods

The experiments were carried out in the Laboratory of Wood Drying and Thermal Treatment at the BioEconomy Institute CNR IBE, San Michele all'Adige, Italy (TV modification and moisture exclusion efficiency), the Laboratory of Wood Biodegradation and Protection of the Latvian State Institute of Wood Chemistry (WTT, microscopy and decay tests), and the Department of Wood Processing in LLU. Experiments were conducted with rotary-cut birch veneers from JSC "Latvijas Finieris," Riga, Latvia.

#### *Wood treatment technology (WTT)*

Thermal modification of birch veneers with the dimensions of 1000 mm × 350 mm × 1.5 mm was conducted with wood treatment technology under the previously determined optimal regime of 160 °C, 0.8 h (160/0.8/WTT), and 500 kPa to 900 kPa pressure in water vapor environments in packs of 10 sheets per each. In total 60 samples were treated. The process classified as a moist, closed thermal treatment technology was described in detail previously by Grinins *et al.* (2016).

#### *Thermo vacuum technology (TV)*

Thermo vacuum technology (TV) can be classified as a dry, open thermal treatment under vacuum (Allegretti *et al.* 2012; Sandak *et al.* 2015). Rotary – cut 600 mm × 600 mm × 1.5 mm birch veneers were treated in four experimental regimes (Table 1) under 25 kPa pressure, although this process was modified so that veneers were treated under a

convective heat regime between aluminum plates in packs of 6 to 10 layers, ~ 40 samples per regime in total.

**Table 1.** Experimental Regimes

Regime	Temperature (°C)	Treatment Time (h)	Treatment Type
160/0.8/WTT	160	0.8	WTT
204/2.0/TV	204	2.0	TV
214/2.0/TV	214	2.0	TV
217/3.0/TV	217	3.0	TV
218/0.5/TV	218	0.5	TV

The WTT experimental regime (160/0.8/WTT) was previously found as optimal for birch veneers regarding decay resistance (Irbe *et al.* 2017) and other properties (Grinins 2016). TV regimes were chosen to repeat mass loss of 160/0.8/WTT (214/2.0/TV), as well as to have ~2% more (217/3.0/TV) and 2% (204/2.0/TV) less mass loss.

## Laboratory Characterization

### Mass loss

Mass loss was determined by weighing each sample before and after the treatment, and it was calculated according to Eq. 1,

$$ML = \frac{m_m - m_o}{m_o} \cdot 100\% \quad (1)$$

where  $m_o$  is the mass of oven-dry untreated wood samples (kg) and  $m_m$  is the mass of oven-dry modified wood samples (kg).

### Density

Density was determined according to ISO 13062–2 (2014). Standard deviation and coefficient of variation were calculated for the analysis of the results.

### Color

A MicroFlash 200D portable spectrophotometer (DataColor, Lawrenceville, NJ, USA) suitable for direct determination of CIE  $L^*a^*b^*$  color coordinates according to ISO/CIE 11664–4 (2008) was used for measurement over an 18 mm diameter spot with a standard light source D65 and an observer angle of 10°. Color was also measured with a Minolta CM-2500d spectrophotometer with D65 light source and d/8 measuring geometry and 10° standard observer (Konica Minolta, Tokyo, Japan). Each of the 30 samples was measured 3 times at the same spot before and after the heat treatment.

The total color change  $\Delta E$  between treated and untreated samples was calculated according to Eq. 2,

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (2)$$

where  $L^*$  indicates the lightness in the range from black (0) to white (100) and  $a^*$  and  $b^*$  define the position in the green-red and blue-yellow axis respectively.

### EMC and MEE

Equilibrium moisture content was measured on samples with dimensions of 40 x 20 mm according to ISO 13061–1 (2014). One sample was cut from each of the treated and

untreated veneers (a total of 20 samples for each regime) and conditioned in a climatic chamber at a temperature of 20 °C and 30%, 65%, and 80% relative humidity (RH).

The Moisture Exclusion Efficiency (MEE) of samples equilibrated at each condition was estimated by Eq. 3,

$$MEE = \frac{EMC_{NT} - EMC_{HT}}{EMC_{NT}} \quad (3)$$

where  $EMC_{NT}$  (%) is the EMC of untreated reference samples and  $EMC_{HT}$  (%) is the EMC of treated samples.

The MEE value expressed the relative variation of EMC of treated wood equilibrated at RH 30%, 65%, and 80% (MEE = 0% indicated no EMC variation; MEE = 100% indicated an EMC value of 0%).

### *Tensile strength*

Tensile strength of veneers parallel to the grain was determined according to GOST 20800–75 (1976). Twenty random samples with the dimensions of: width 20 mm; length (grain direction) 200 mm; thickness 1.5 mm were cut from both treated and untreated veneers. To prevent the samples from slipping out of the clamps, pieces of plywood with the dimensions 20 mm × 50 mm × 4.5 mm were glued on both sides of the veneers and on both ends of samples using one-component polyurethane glue. Afterwards, the samples were conditioned at  $20 \pm 3$  °C and  $65 \pm 5\%$  relative humidity. A tensile strength test was conducted with the INSTRON 5967 device (Instron, Norwood, MA, USA) with a constant speed of approximately 1 mm per minute to obtain a rupture of samples in  $60 \pm 30$  s.

### *Decay resistance*

Decay resistance was determined according to European Prestandard ENV 12038 (2002) using brown rot fungus *Coniophora puteana* BAM Ebw 15. The fungus was cultivated on a medium which contained 5% malt extract concentrate and 2% Fluka agar. 10 samples of each regime with the dimensions of 50 mm × 25 mm × 1.5 mm were aseptically placed on 3 mm steel supports in Petri dishes on fungal mycelium and incubated at  $22 \pm 2$  °C and  $70 \pm 5\%$  RH for 6 weeks. After cultivation, the samples were removed from the culture vessels, brushed free of mycelium, and oven dried at  $103 \pm 2$  °C. Percentage weight loss (WL) of the samples was the measure for the extent of fungal degradation.

### *Microscopy*

Light microscopy (LM) was performed on 10 samples from thermally modified and unmodified birch veneers with the dimensions of 20 mm × 20 mm × 1.5 mm. Before microscopy, the samples were soaked for 24 h in distilled water to soften the wood structure and make it sliceable. Wood sample cuts (15 µm to 30 µm) were obtained with a razor blade. Microscopy was made with a Leica DMLB light microscope (Leica Microsystems, Mannheim, Germany) at 400× magnifications. The images were taken with a Leica DFC490 video camera, using Image-Pro Plus 6.3 software for picture analysis (Media Cybernetics, Inc., Silver Spring, MD, USA).

### *Statistics*

To determine a statistically significant difference among groups of data, one-factor ANOVA with a confidence level of 0.05 was considered.

The CORREL function in MS EXCEL was used to estimate how two or more variables were related to another.

## RESULTS AND DISCUSSION

### Mass Loss

Mass loss is the main indicator of the intensity of thermal modification. Mass loss is caused mainly by the degradation of hemicellulose due to its lower degree of polymerization and higher reactivity because of its amorphous structure (Hill 2006; Gérardin 2016). The average mass loss for each regime can be seen in Fig. 1.

The TV modification in a dry environment was less severe than in a moist environment of the WTT process. The 160/0.8/WTT regime had twice the loss of mass of the 204/2.0/TV regime despite 1.2 h shorter modification duration and 44 °C lower treatment temperature. This indicated the prevalence of hydrolysis processes in the wood thermal decomposition. Water and acetic acid formed by wood decomposition were easily evacuated from thin veneer by vacuum. The dependence of mass loss from wood species, process environment, temperature, and heat impact duration in wood thermal modification was ascertained by several authors and is well-known (Esteves and Pereira 2009; Xu *et al.* 2019).

Veneers' mass loss in TV increased with modification temperature and duration, and differences between 217/3.0/TV and 218/0.5/TV emphasized the importance of the duration.

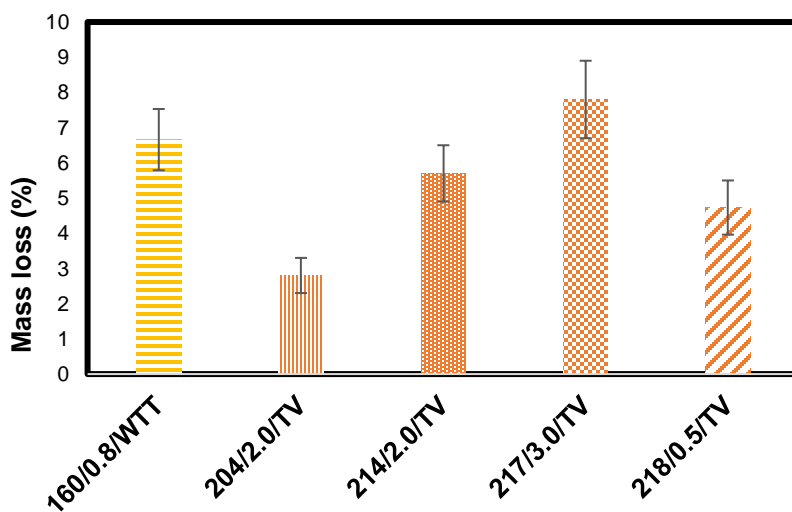


Fig. 1. Mass loss after thermal modification of birch veneer samples (STDEV error bars)

According to Grinins (2016) concerning the mass loss for birch veneers, 160/0.8/WTT was 6.3%, which is the same as that observed with treated veneers at 6.7%. If the mass and dimensions of the sample increase, for example for birch planks, mass loss at the same regime 160/0.8/WTT becomes considerably greater – reaching 16% (Biziks *et al.* (2016) and it is important for plywood production if thicker veneers are used. According to Chaouch *et al.* (2013), mass loss occurs mainly due to deacetylation of strongly acetylated glucuronxylan which causes liberation of acetic acid, which then catalyzes depolymerization of less – ordered carbohydrates.

## Density

The density of the investigated samples is shown in Table 2. The density of untreated birch wood was  $598 \pm 42 \text{ kg/m}^3$  and coincided with the previously observed density of  $568 \text{ kg/m}^3$  (Ruponen *et al.* 2015).

**Table 2.** Average, Standard Deviation, and Variation of Veneer Density

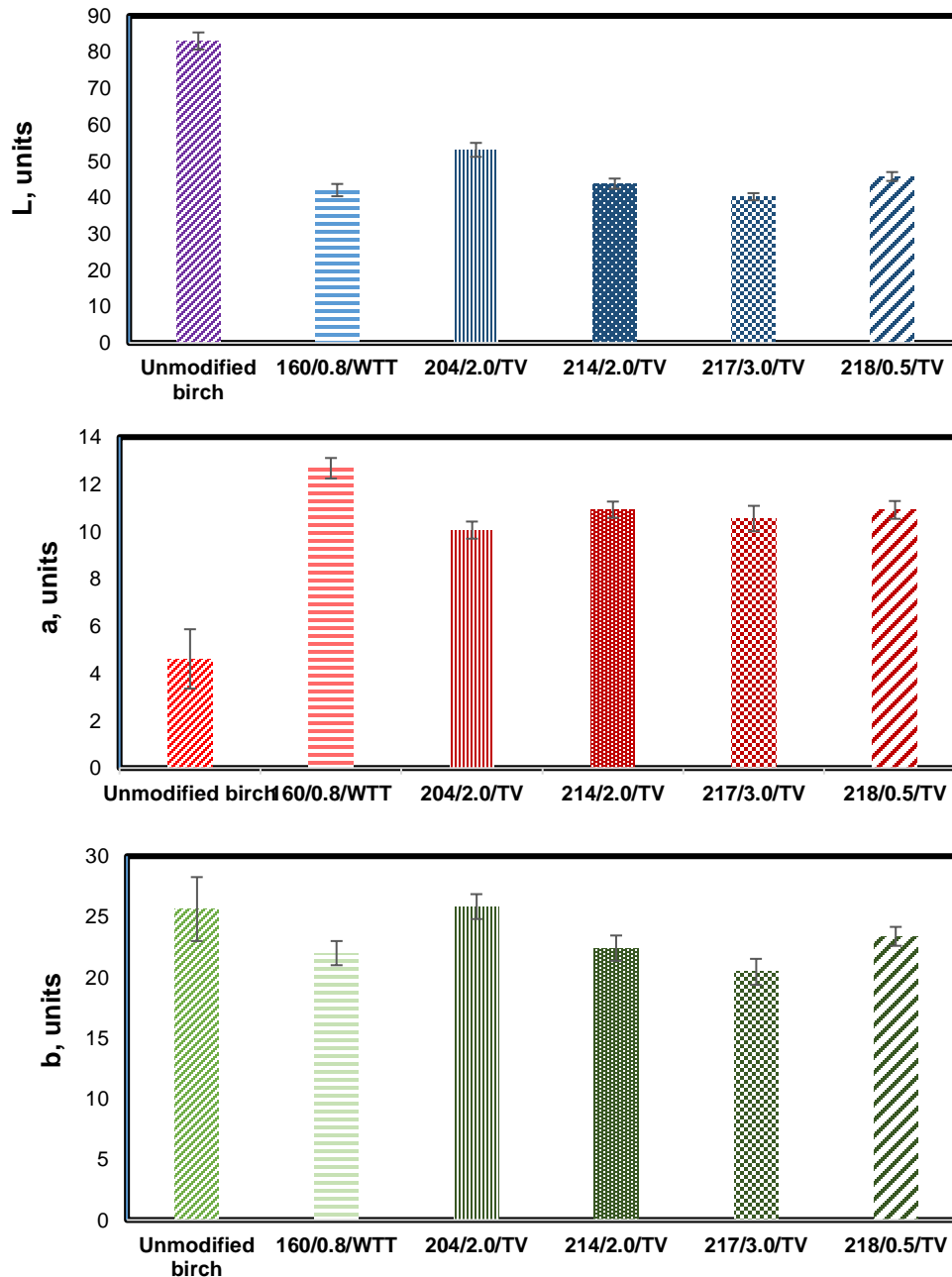
Sample	Average Density ( $\text{kg/m}^3$ )	Standard Deviation ( $\text{kg/m}^3$ )	Variation (%)
Unmodified	598	42.0	7.1
160/0.8/WTT	583	43.0	7.4
204/2.0/TV	584	34.9	6.0
214/2.0/TV	621	48.7	7.8
217/3.0/TV	574	38.6	6.7
218/0.5/TV	615	35.2	5.7

Using one-factor ANOVA analysis,  $F = 5.38 > F_{\text{crit.}} = 2.24$  ( $p = 0.0001$ ), and therefore the treatment affected veneer density above its natural variation. The variation between samples did not exceed 20% and was considered acceptable. Sandberg *et al.* (2013) stated that the density of wood during thermal modification decreases from 5% to 15%, thus additionally affecting its strength. Kotilainen (2000) also implies that thermally modified wood has a lower density than untreated wood and deviation is high.

## Color

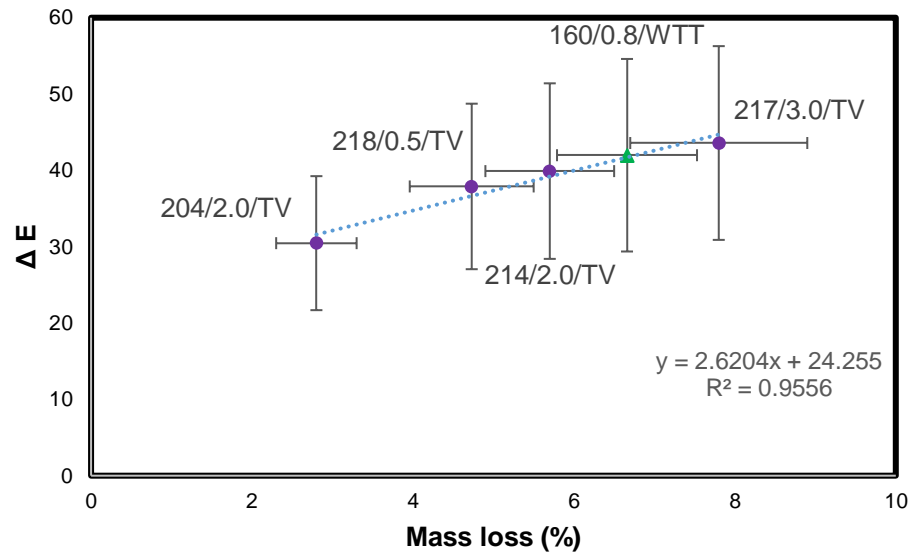
During the modification process, wood became darker and the variation depended on the treatment temperature. As shown in Fig. 2,  $L^*$  decreased on average by half after thermal treatment and reached 42 to 53 units, which coincided with the findings by Lovrič *et al.* (2014). The mildest treatment regime 204/2.0/TV had the highest  $L^*$  value among the treated samples. The modified veneers' surface color  $a^*$  component was several times greater than for unmodified ones. After modification, veneers became more reddish, especially the 160/0.8/WTT veneers. As cellulose and hemicellulose do not absorb visible light (Hon and Minemura 2001), these changes implied an alteration of phenolic structures in lignin and extractives during the thermal modification process. Veneers' surface color  $b^*$  component slightly decreased with the increase in the severity of the thermo-vacuum process and in WTT – treated veneers.

The total color changes ( $\Delta E$ ) depending of the mass loss can be seen in Fig. 3. Visually visible changes were considered if  $\Delta E$  was greater than 3.5 units (Mokrzycki and Tatol 2011). Hemicellulose was the component that was damaged most during heat treatment. Consequently, the relative content of lignin in heat-treated wood increased accordingly, which might explain darker colors (Lovrič *et al.* 2014). Brischke *et al.* (2007) described that the sum of lightness parameter and yellow-blue axis parameter ( $L^*+b^*$ ) correlate with treatment regime for beech wood ( $R^2=0.951$ ). The total color changes in this research correlated ( $r = 0.98$ ) well with mass loss, and the most severe was the regime 217/3.0/TV, followed by 160/0.8/WTT. As it is observed, color changes did not occur as a result of increase in temperature. Also, the treatment duration and treatment method were substantial. The results can be seen in Fig. 3.



**Fig. 2.** Color coordinates  $L^*$ ,  $a^*$ , and  $b^*$  of untreated and treated wood samples at different process conditions (STDEV error bars)

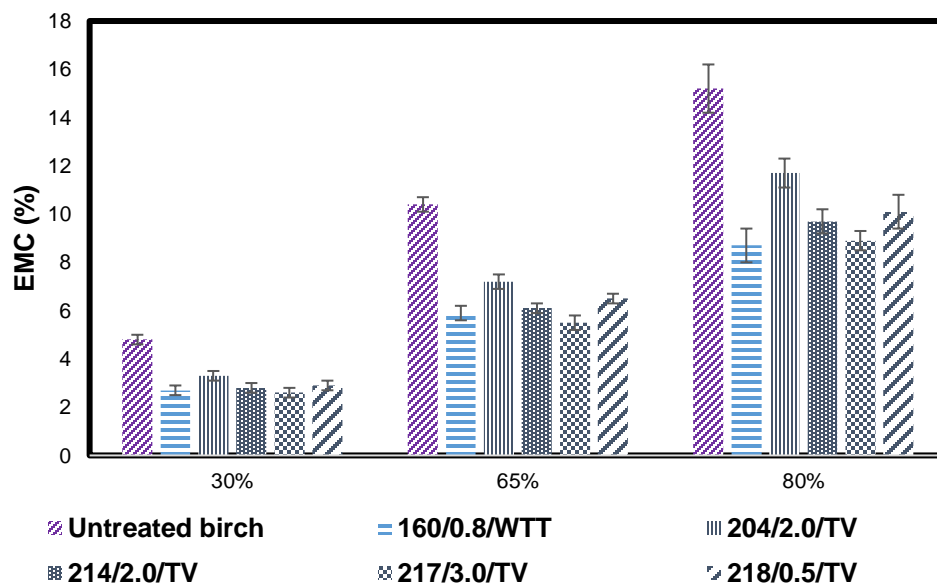
Total color changes in VT and WTT process treated veneers are considerably larger than observed by Barcik *et al.* (2015) – in the Termowood® process. For birch treated at 160 °C  $\Delta E$  was 1.16, and at 210 °C it was 2.56. Using the birch thermal modification under saturated 160 °C and superheated 185 °C steam, Torniainen *et al.* (2011) showed that  $L^*$  decreased from 80.92 to 52.72 and 55.48 accordingly, which coincides with the findings of this research.



**Fig. 3.** Total color change ( $\Delta E$ ) correlation with mass loss during thermal modification

### Equilibrium Moisture Content (EMC) and Moisture Exclusion Efficiency (MEE)

As shown in Fig. 4, the treated wood had a lower EMC compared to wood that was not treated. The veneers treated at 204/2.0/TV were the most hygroscopic at all RH. The least hygroscopic materials were 160/0.8/WTT and 217/3.0/TV. It is commonly known that hydrothermally-treated wood becomes less hygroscopic (Mirzaei *et al.* 2017). Water uptake of wood is reduced by the heat-treatment process (Hytinen *et al.* 2010), since hemicellulose and cellulose are the main wood components responsible for decay and hygroscopicity of wood (Li *et al.* 2017).



**Fig. 4.** The equilibrium moisture content at a different relative humidity of untreated and treated birch veneer samples (STDEV error bars)



The moisture content of heat-treated wood was about half compared to that of untreated wood. Because of the loss of hygroscopic hemicellulose sugars and their conversion to less hygroscopic furan-based polymers, predominantly furfural and hydroxymethylfurfural, during heat treatment, the equilibrium moisture content was reduced to about half the value of untreated wood (Jämsä *et al.* 2000; Sandberg *et al.* 2013), which coincided with the results in Fig. 4.

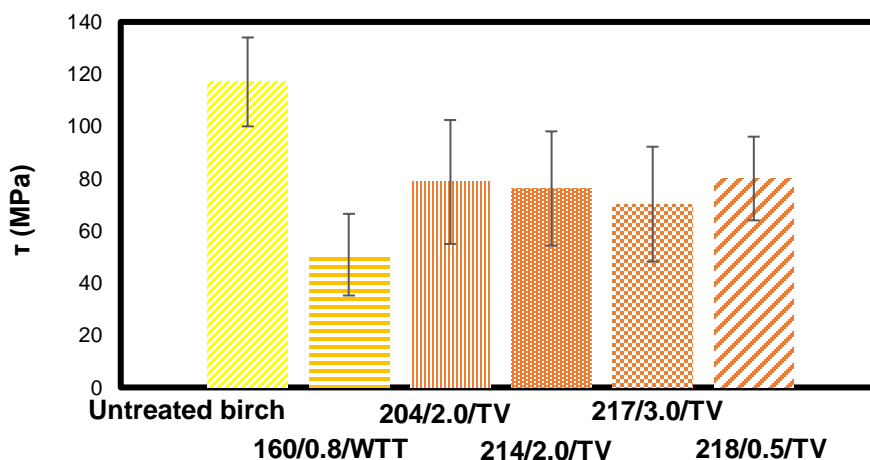
The MEE characterized the increase in the modified veneers' hydrophobicity. The results can be seen in Table 3. 217/3.0/TV was the most hydrophobic due to the more severe treatment conditions among all TV treatments. MEE correlated well with mass loss at RH 30% ( $r=0.99$ ), at RH 65% ( $r=0.99$ ) and at RH 80% ( $r=0.95$ ).

**Table 3.** MEE of Process Conditions at Different Relative Humidity

Treatments	RH 30%	RH 65%	RH 80%
160/0.8/WTT	-42.8%	-43.4%	-42.9%
204/2.0/TV	-31.3%	-30.7%	-23.5%
214/2.0/TV	-41.3%	-41.8%	-36.5%
217/3.0/TV	-45.6%	-47.0%	-41.3%
218/0.5/TV	-38.8%	-37.8%	-34.0%

### Tensile Strength

The results of tensile strength properties along fibers are reported in Fig. 5. The data had considerably high standard deviations, but the mean values of tensile strength were compared to the values for untreated birch, and there was no remarkable difference, such as 117 MPa in this research compared to 125.5 MPa according to Grinins (2016), although this was over 20% higher than 75 MPa as shown in literature (Volynsky 2009).



**Fig. 5.** Tensile strength along fibers of untreated and treated birch veneers (STDEV error bars)

The state of the wood cell wall directly affected the veneers' strength properties. As a result of the degradation of hemicelluloses, the wood becomes brittle and rigid, which indicates the important role that hemicelluloses have in imparting viscoelastic properties to wood (Hill 2006). After the thermal modification of birch veneers in the TV process, the tensile strength along the fibers decreased from 117 MPa to approximately 76 MPa apart

from treatment regime. In the WTT process, the tensile strength decreased to approximately 51 MPa. Strength loss in the WTT process was considerably greater than in the TV process. The tensile strength fairly correlated with mass loss ( $r = 0.57$ ), and for the TV-treated samples it was notable ( $r = 0.86$ ). As shown in Fig. 6, the WTT-treated veneers differed from the TV – treated veneers.

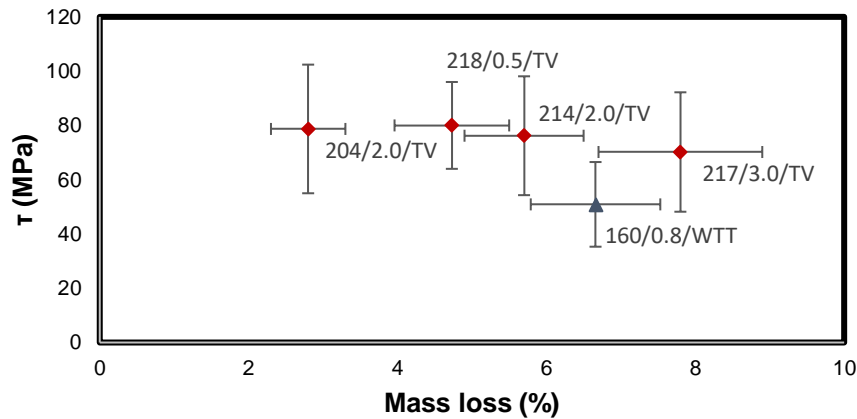


Fig. 6. Mass loss correlation of tensile strength of treated birch veneer samples (STDEV error bars)

### Decay Resistance

Figure 7 shows that none of the veneers could be considered durable according to the ENV 12038 (2002). Weight loss for all the samples exceeded 3%. The 217/3.0/TV veneers were the most durable, with weight loss of  $12.1 \pm 3.4\%$ . The 160/0.8/WTT and 204/2.0/TV treatments did not provide any improvements in durability compared to untreated birch veneers.

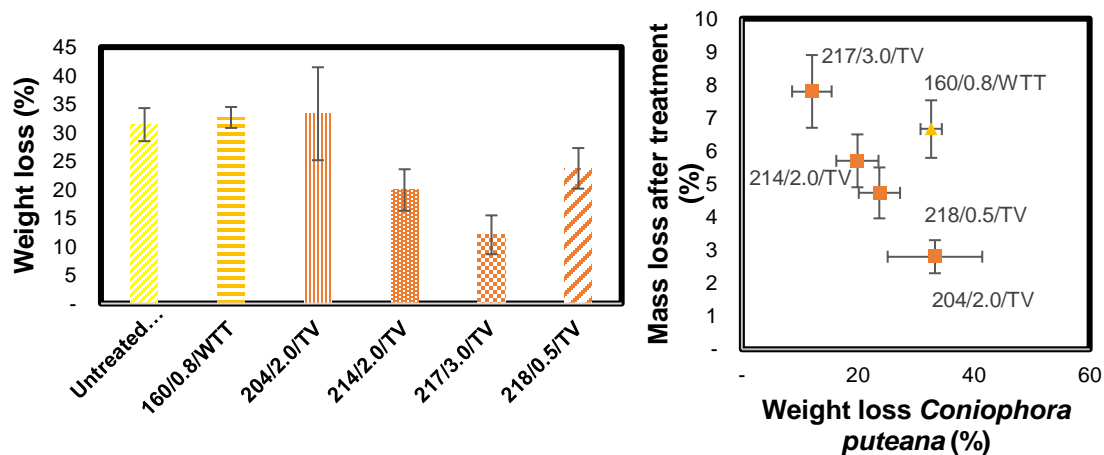
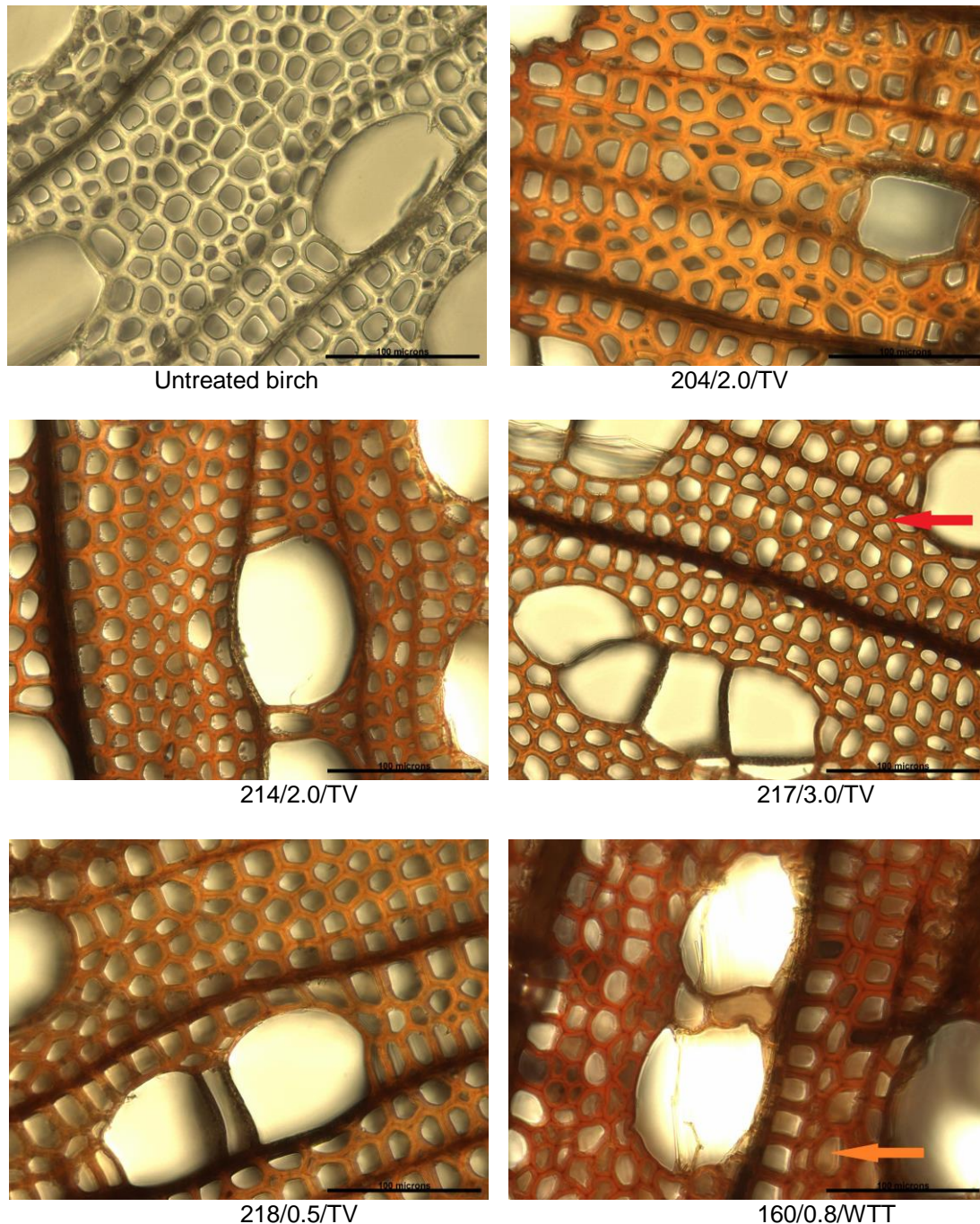


Fig. 7. Weight loss after fungus *Coniophora puteana* and its correlation with a mass loss after thermal treatment (STDEV error bars)

The TV treated veneers showed a high correlation between mass loss after thermal treatment and durability ( $r = 0.97$ ). Although 160/0.8/WTT had relatively high mass loss after thermal treatment, its durability was comparable with untreated samples with  $33 \pm 2\%$  weight loss, which coincided with  $31 \pm 13\%$  weight loss from the previous findings (Grinins *et al.* 2016).

## Microscopy

The color changes were observed to light brown in all TV regimes and dark brown in WTT process in comparison with untreated light silver birch sample (Fig. 2). The treated birch was the most affected by treatment processes 217/3.0/TV – cell walls became thinner – red arrow and 160/0.8/WTT cell walls became fluffy or sharp less – orange arrow, as shown in Fig. 8.



**Fig. 8.** Cross-section before and after thermal treatment, LM, x400. Bar = 100 µm

The shrinkage of fiber cell walls was observed in both treatment regimes. The thermally modified samples, especially for treatment regimes 217/3.0/TV and 160/0.8/WTT, were more brittle than their untreated counterparts. This coincides with the findings by Ahmed *et al.* (2013) determining that increased brittleness due to the modification process has an impact on tensile strength of treated samples (Fig. 5). During thermal modification, middle lamella did not disappear because it consisted mainly of lignin (70% to 90%), which was a more thermally stable wood component. Birch is diffuse-porous wood and has sparse apotracheal parenchyma (Schoch *et al.* 2004). Besides, regime 160/0.8/WTT caused deformation of the vessels and fibers due to the increased pressure and moist environment during modification. The cellulose framework shrank, and as a result of that the fibers obtained an oval form in the cross-section (Biziks *et al.* 2013).

## CONCLUSIONS

1. The TV thermal modification process was more suitable for birch (*Betula pendula* Roth) veneer treatment than the WTT process both in terms of maintaining strength properties and providing durability.
2. Among investigated samples, the regime 217/3.0/TV showed the best resistance against brown rot and acceptable other physical – chemical properties and tensile strength.
3. Treatment regimes with the highest mass loss were 217/3.0/TV (7.8%) and 160/0.8/WTT (6.7 %). Mass loss correlated well with the MEE in all the RH environments ( $r = 0.95$  to  $0.99$ ).
4. Veneer color depended on the treatment temperature and time. As expected, veneers became darker during the thermal modification,  $L^*$  value decreased twice, and  $a^*$  value increased. Total color change  $\Delta E$  correlated ( $r = 0.98$ ) with the mass loss after the thermal modification.
5. The TV treated veneers showed a high correlation between mass loss after the thermal treatment and durability against brown rot fungus *Coniophora puteana* ( $r = 0.97$ ).

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