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Properties of *Populus* genus veneers thermally modified by two modification methods: wood treatment technology and vacuum-thermal treatment

A. Meija[1,*], I. Irbe[2], A. Morozovs[1] and U. Spulle[1]

[1]Latvia University of Life Sciences and Technologies, Forest Faculty, Department of Wood Processing, Liela street 2, LV3001 Jelgava, Latvia
[2]Latvia State Institute of Wood Chemistry, Laboratory of Wood Biodegradation and Protection, Dzerbenes street 27, LV1006 Riga, Latvia
[*]Correspondence: meija.anete@gmail.com

Abstract. Due to environmental concerns the use of wood materials is becoming more extensive and is causing wood supply shortage, therefore the use of *Populus* genus wood species with a short rotation period is vital. *Populus* genus species wood has several shortcomings - it is not durable, has low density and is hygroscopic. Thermal modification is a technology that can be used to improve the situation. In this study aspen (*Populus tremula* L.) was thermally treated using the Wood Treatment Technology (WTT) device for 50 min at 160 °C (50–160 WTT) and poplar (*Populus x canadensis* Moench) was vacuum-treated (VT) 120 min at 204 °C (120–204 VT), 120 min/ 214 °C (120–214 VT), 180 min 217 °C (180–217 VT) and 30 min 218 °C (30–218 VT). Mass loss (ML), colour change, density, tensile strength along the fibres, moisture exclusion efficiency and weight loss (WL) after brown rot fungus *Coniophora puteana* were determined and also light microscopy images were taken. Aspen veneers showed a ML of 5.3% between 120–214 VT (6.2%) and 30–218 VT (4.6%) treatment that coincided with the same mass loss in aspen boards cited in the literature. The highest ML was 8.7% calculated from 180–217 VT, while the lowest ML was 2.9% computed from 120–204 VT. The total colour change ΔE was 44, where lightness parameter L provided the greatest impact that was reduced twice after modification. Tensile strength reduced by 47% in the WTT process and had ~29% reduction in the VT process. The WL after fungus *C. puteana* was 33% at 50–160 WTT. After VT treatment, WL was 0–2.4%. 120–214 VT and 180–217 VT poplar veneers were the most suitable for plywood production.

Key words: aspen, poplar, thermal modification, decay resistance.

INTRODUCTION

Due to the constantly growing eco-awareness around the world, wood products are increasingly being utilized in indoor and outdoor applications (Li et al., 2017). One way of reducing the emission of carbon dioxide is to use a larger portion of wood products and to increase the lifetime of these products so that carbon is stored over a longer period (Sandberg et al., 2013). Climate change should be considered, since it has strong negative effects on boreal timber supply (Brecka et al., 2018).

Plywood is an engineered wood material, which is widely used. Its production reached 107.4 million cubic meters in 2017 (Raute, 2017) and has a growing tendency to be used for different purposes. Traditionally plywood in Latvia is made from birch wood (Meija-Feldmane et al., 2020), but, due to climate changes, population growth and urbanization and infrastructure development, the potential area for forests might decrease that causes risk of a potential birch wood supply shortage. Therefore, it is crucial to investigate other species suitable for plywood production. Poplar tree species, including all their wide varieties, are largely cultivated in the world as a fast-growing energy crop (Todaro et al., 2017) and it may open the opportunities for the forest industry to widen Populus spp wood usage (Brecka et al., 2018). Aspen (*Populus tremula* L.) is usually the raw material of choice for the production of matches and interior materials (Biziks et al., 2015) in Latvia and it covers 3% from the species growing in Latvia's forests (Mezataksacija, 2016). It is timber with the highest potential from the point of view of the national economy in Latvia (Iejavs et al., 2018). Poplar (*Populus* × *euramericana*) is a fast-growing species with a short rotation period of 10 to 20 years and it is commonly used in veneer and plywood production, as a cellulose material, for lumber production, as biofuel, and in the production of packaging material (Lovrić et al., 2014) and therefore it has been extensively investigated (Aydin et al., 2006; Bulcke et al., 2012; Denes & Lang, 2013).

The material that is used for plywood production, as well as the adhesives used and manufacturing technology strongly affect the end-product quality (Tymyk et al., 2013). Poplar plywood tends to warp greatly with the absorption of moisture. This is one of the problems presented by such plywood that may be caused by its high hygroscopicity (Murata et al., 2013). Generally, shrinkage and swelling create the biggest problems in manufacturing of wood construction and carpentry elements (Spulle et al., 2018).

One of the environmentally friendly methods to improve the dimensional stability and biodurability of wood is its thermal modification (Zanuttini et al., 2019). Heat treatment process changes the composition of wood (Hyttinen et al., 2010). Chemical composition alterations are result of dehydration, hydrolysis, oxidation, decarboxylation and transglycosylation, and the wood becomes less hygroscopic (Kocaefe et al., 2008). In 2007, Jones predicted that in 5 years there would be thermally modified veneer supply in the market. Thermal wood treatment is the most commercialized and the most investigated method for modifying wood composition (Willems et al., 2013). Thermally modified veneers can also be used for decorative purposes indoors as a laminated layer for board materials (Wang et al., 2018).

Although poplar veneers had been previously investigated, there was no direct comparison between Wood Thermal Technology (WTT) and vacuum-thermo (VT) processes. According to Hill (2006) WTT is a closed and wet process, while VT is an open and dry process. WTT process operate at 140–160 °C, 7 bar pressure, while VT modifies wood at reduced pressure and higher temperatures (0. 25 bar, 204–218 °C).

In this paper, the properties of two *Populus* genus veneers treated with two treatment technologies, the VT modification and the WTT were compared to choose the most suitable for plywood production.

MATERIALS AND METHODS

Samples

The experiments were conducted with rotary-cut veneers from aspen (*Populus tremula* L.) and poplar (*Populus x canadensis* Moench) with moisture content of 7.5–8.1%. The samples were obtained directly from the production unit - peeling and wet veneer sorting line after drying and dry veneer sorting process, without visual wood defects with regular annual rings not wider than 2 mm.

Thermal modification

Thermal modification of 290×290×1.5 mm aspen veneers was conducted using Wood Treatment Technology under the previously determined optimal regime of 50 min/160 °C (50–160 WTT) 5–9 bar pressure in water vapour environments in packs of 10 sheets per each. The process applied was described in detail previously by Grīniņš (Grinins et al., 2016).

Rotary-cut 600×600×1.5 mm poplar veneers were treated at four experimental regimes: 120 min/204 °C (120–204 VT), 120 min/214 °C (120–214 VT), 180 min/217 °C (180–217 VT), 30 min/218 °C (30–218 VT) in VT process under 250 mbar pressure in dry environment, which was described in more detail by (Sandak et al., 2015) although this process was modified – veneers were treated under convective heat regime, between aluminium plates in packs of 3 to 12 pieces per pack, which was similar to the processes used in manufacturing.

Laboratory Characterisation

Mass loss (ML)

ML was determined by weighing each sample before and after the treatment and it was calculated according to Hill (2006).

Colour

A MicroFlash 200D portable spectrophotometer (DataColor Int, Lawrenceville,

USA), suitable for direct determination of the CIE L*a*b* colour coordinates according to ISO 11664–4 (2008) was used for the measurement over an 18 mm diameter spot with a standard light source D65 and an observer angle of 10°. Colour was also measured with minolta CM-2500d spectrometer with D65 light source and d/8 measuring geometry and 10° standard observer. Each sample was measured 3 times at the same spot before and after the modification (30 samples per regime in total). The total colour change ΔEab was the difference between the colour before and after the modification, it was calculated according to the suggestions of Technical report of the Calorimetry (International Commission on Illumination, 2004).

Tensile strength

Tensile strength was determined according to the standard GOST 20800-75 (GOST, 1976). After the thermal treatment, 20 random samples were cut both from treated and untreated veneers. The size of these samples was 20×200×1.5 mm. To avoid sample slipping out of the clamps, plywood pieces with the dimensions of 20×50×4.5 mm were glued on both sides of the veneers and both ends of samples, using a one component polyurethane glue. Afterwards, the samples were conditioned at

20 ± 3 °C and at a relative humidity of 65 ± 5%. A tensile strength test was performed with the INSTRON 5500 device with a constant speed (~1mm min^{-1}) to obtain a rupture of the samples in 60 ± 30 s.

Density

Density was measured by weighing the 30 samples of each treatment and measuring their dimensions. Calculation of density was done according to the standard ISO 13061-2 (ISO, 2014). To analyse the results standard deviation and coefficient of variation were calculated.

Moisture exclusion efficiency (MEE)

Twenty samples were weighed after each environment condition and afterwards an oven-dry mass was calculated according to the standard ISO 13061-1 (ISO, 2014).

Then samples were equilibrated in chambers at 20 °C with RH 30%, 65% and 85% and mass changes were measured. The MEE of samples equilibrated at each condition was estimated according to Hill (2006). The equilibrium moisture content was the MC that was constant at the corresponding relative humidity and temperature of the surrounding environment.

The MEE of samples equilibrated at each condition was estimated by Eq. 1:

$$MEE = \frac{EMC_{NT} - EMC_{HT}}{EMC_{NT}} \quad (1)$$

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

Decay resistance

Decay resistance was determined according to modified 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. Six samples with the dimensions of 50×25×1.5 mm were aseptically placed on 3 mm steel supports in Petri dishes on fungal mycelium and incubated at 22 ± 2 °C and 70 ± 5% RH for 6 weeks. After cultivation, the samples were removed from the culture vessels, brushed free of mycelium and oven-dried at 103 ± 2 °C. The weight loss (WL) of the samples expressed as a percentage was the measure for the extent of fungal degradation.

Statistics

To find out whether a statistically significant difference exists between two groups of data, an unpaired two-tailed t-test with a confidence level of 0.05 was used.

The correlation was the measure of how two or more variables were related to one another. To estimate that, the CORREL function in MS EXCEL was used, where the correlation coefficient r was used to determine the relationship between the two properties.

RESULTS AND DISCUSSION

Mass loss (ML)

ML is a property that allows one to compare different processes that had different temperatures, different treatment times and treatment environments. Heat treatment of wood in the WTT and VT processes differs in water vapours content in the reaction environment, and with hydrolysis reactions prevalence in the first one. Fig. 1 shows that aspen veneers treated in the 50–160 WTT had a ML 5.3% between 120–214 VT and 30–218 VT treatment that coincided with 5% mass loss for aspen boards 60 min 160 °C WTT process (Biziks et al., 2015). The highest ML 8.7% was observed in 180–217 VT with the longest treatment time that was close to ML 9%, obtained in VT process 120–210 °C treated poplar plywood (Zanuttini et al., 2019).

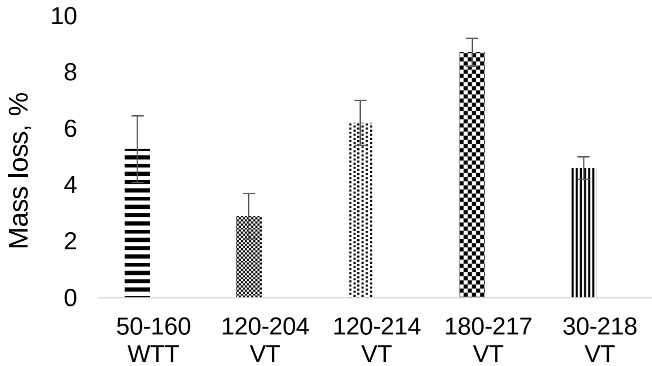

Figure 1. The ML after different thermal treatments of veneer samples.

Colour

The colour of the material is a result of selective light absorbance by the conjugated double bond chemical systems with chromophore groups, which are present in lignin and also in certain sort of extractives in natural wood (Nemeth et al., 2016). Structural change of lignin during thermal modification is mainly responsible for colour change (Kim et al., 2014). Lightness parameter L* was the one that changed the most during thermal modification as showed in Table 1.

Table 1. Colour of untreated and thermally modified veneers.

	L	a	b	ΔE
Untreated aspen	87.6 ± 1.6	1.4 ± 0.4	19.2 ± 1.0	x
Untreated poplar	89.5 ± 2.5	0.8 ± 1.4	18.4 ± 1.2	x
50–160 WTT	46.6 ± 2.2	13.7 ± 0.6	27.4 ± 1.8	43.5 ± 12.9
120–204 VT	52.9 ± 1.2	11.4 ± 0.2	26.8 ± 0.7	39.0 ± 11.1
120–214 VT	43.2 ± 1.5	11.0 ± 0.2	21.8 ± 0.9	47.5 ± 8.5
180–217 VT	40.4 ± 1.2	10.1 ± 0.2	25.8 ± 1.0	50.0 ± 9.2
30–218 VT	44.4 ± 1.7	11.3 ± 0.3	22.9 ± 1.1	46.6 ± 8.4

Zanuttini et al. (2019) states that lightness parameter L is 85 ± 2 for untreated poplar and 41 ± 1.4 for thermally treated aspen samples, which coincides with the findings in this research. Lightness parameter L has close correlation with mass loss (r = 0.90).

Total colour change ΔE values showed that thermally modified veneer colours are considered as different colours (Allegretti et al., 2009), which could be an advantage for customers who prefer a tropical-like appearance of wood.

Density

Density changes during the thermal modification of *Populus deltoides* were reported by (Mirzaei et al., 2017), implying density increase with an increase in temperature. The results of the present study show that density after thermal modification was not affected in both species (Table 2) which agreed with Chaouch et al.(2010a) that species of lower density presented better stability to thermo-degradation than species with higher density.

Table 2. Density of untreated and thermally treated veneers

	Density	stdev
Untreated aspen	442	58
50–160 WTT	420	54
Untreated poplar	300	29
120–204 VT	295	25
120–214 VT	289	35
180–217 VT	312	35
30–218 VT	309	18

Density of untreated aspen was 442 ± 58 kg m^{-3} which was slightly lower than 524 kg m^{-3} (Biziks et al., 2015), but it coincided with Li et al. (2017) 400 kg m^{-3} *Populus* spp. Density of untreated poplar was 300 ± 29 kg m^{-3}, which was less than *Populus nigra* (Chaouch et al., 2010b) 437 kg m^{-3} and poplar (Willems et al., 2013) 437 kg m^{-3}. Minor differences were expected, hence density depends not only on the species but also on growth conditions.

Tensile strength

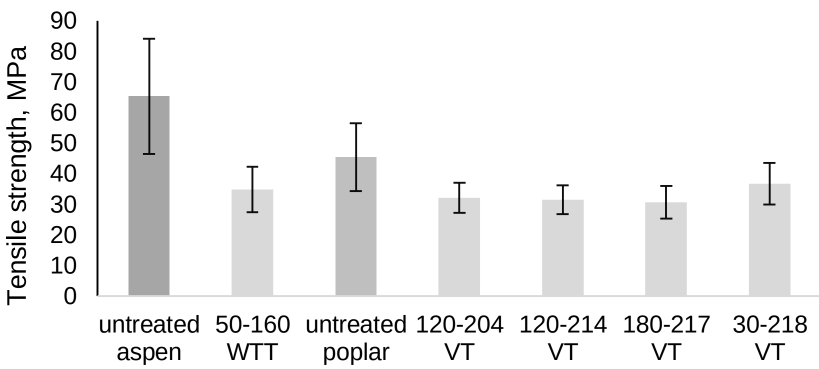

Figure 2. The tensile strength of veneer samples.

The tensile strength (Fig. 2) along the fibres for unmodified aspen of 65 MPa was slightly higher than 60 MPa, reported in the literature (Volinsky, 2009). Aspen veneers, treated in the WTT process reduced tensile strength from 65 to 35 MPa, which was statistically significant ($p = 2.81 \cdot 10^{-7}$). After modification in the VT process, the tensile strength of poplar veneers reduced from 45 to ~34 MPa. The difference between tensile strength of untreated and VT treated poplar veneers was statistically significant

$p = 2.24 \cdot 10^{-5}$ (120–204 VT), $p = 2.19 \cdot 10^{-5}$ (120–214 VT), $p = 0.003$ (180–317 VT), $p = 0.006$ (30–218 VT) and it was insignificantly affected by a treatment regime, except with 30–218 VT regime, which had significant ($p = 0.01–0.02$) difference compared to the other VT regimes. Although the remaining tensile strength was comparable in MPa, the strength decrease was considerably higher (47%) in WTT process than (29%) the decrease in VT process due to low molecular acids that were formed during thermal treatment and catalysed wood decomposition reactions if not removed in the closed WTT process.

Comparing the density and tensile strength changes after modification, the major difference in 50–160 WTT process could be observed. Both properties showed an average correlation (r = 0.64).

Moisture exclusion efficiency (MEE)

Moisture is a critical parameter for nearly all properties of wood, and one of the ways to provide better dimensional stability and decay resistance is wood thermal treatment (Thybring et al., 2018). MEE results are shown in Table 3, which very well correlate with ML (r = 0.95) at RH 30% and RH 65%, at RH 80% it is slightly lower (r = 0.71). Hemicelluloses are the major contributors to the hygroscopicity of the plant fibres. Removal of hemicelluloses decreases hygroscopicity (Kocaefe et al., 2008), although Willems et al. (2013) stated that hemicelluloses were not removed, but dehydrated to furans leading to the subsequent formation of carbonaceous condensation products within the wood structure. The more intense the treatment regime, the less hygroscopic the obtained material. Li et al. (2017) and Mirzaei et al. (2017) stated that the hygroscopicity of poplar wood decreased with increasing temperature in the heat treatment process. Thybring et al. (2018) implied that MEE above 40% was a threshold for decay resistance, 50–160 WTT regime was an exception, although MEE RH80% was 40.4%, WL after fungus *Coniophora puteana* was 33% (Fig. 3).

Table 3. Moisture exclusion efficiency after thermal modification in different relative humidity (RH) environments

	MEE RH 30%	MEE RH 65%	MEE RH 80%
50–160 WTT	-38,9%	-39,4%	-40,4%
120–204 VT	-37,5%	-32,9%	-23,0%
120–214 VT	-43,2%	-42,1%	-31,4%
180–217 VT	-46,6%	-44,1%	-38,4%
30–218 VT	-39,9%	-38,2%	-31,1%

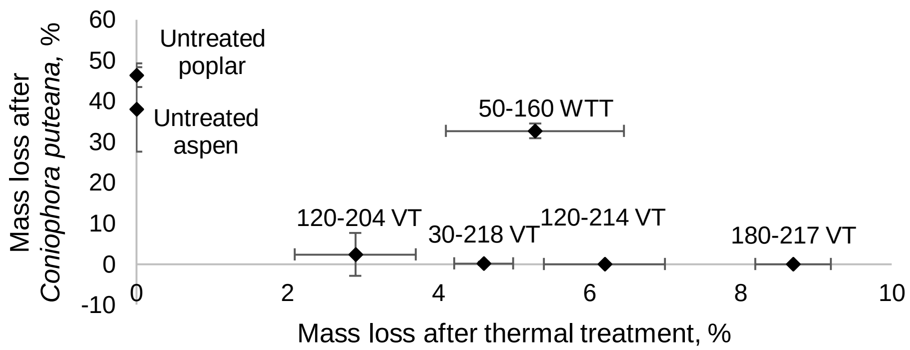

Figure 3. The WL after fungus *C. puteana* and its correlation with the ML after thermal treatment.

Decay resistance

86% of wood construction damage in Latvia is caused by brown rot (Irbe, 2008). Decay resistance and ML correlation can be seen in Fig. 3. WL caused by brown rot fungus *Coniophora puteana* for untreated aspen samples was 46 ± 3 %, 50–160 WTT treatment decreased WL till 33 ± 2%. According to Janberga et al. (2013) 60 min 160 WTT aspen boards showed WL 7 ± 4% while WL for untreated aspen was 50 ± 3%. Veneer samples had a higher surface to volume ratio that could affect the WL by brown rot fungus.

The WL for untreated poplar was 38 ± 10%, which was close to 24% of *Populus nigra* (Chaouch et al., 2013). After the VT treatment, mass loss was 0–2.4%, which was an insignificant decrease. Poplar veneers showed significantly better decay resistance compared to commonly used silver birch veneers treated under the same regime (Meija-Feldmane et al., 2020.).

The only regime that differs is 50–160 WTT where the relatively high ML of 5.3% after modification resulted in 33% WL after brown rot fungi. Poplar (*Populus* spp) plywood glued with urea-melamine-formaldehyde adhesive treated for 120 min at 210 °C WL after *Coniophora puteana* is 0.1% (Zanuttini et al., 2019) which was comparable with 0.0% 120–214 VT regime.

CONCLUSIONS

VT treated poplar veneers show better properties, compared to the WTT process treated aspen due to the less severe conditions during the treatment.

All the veneers can be used for decorative purposes (as outer lamination layers) as they become significantly darker during the thermal modification.

Aspen treated in 50–160 WTT is not suitable for plywood production as it has no relevant improvement regarding decay resistance against *Coniophora puteana*, but it has tensile strength reduction by 47%.

120–214 VT and 180–217 VT poplar veneers were the most suitable for plywood production due to the achieved WL 0% after degradation by brown rot fungus *Coniophora puteana* and 32% tensile strength loss along the fibre.

To have a better comparison between both treatment technologies, the same species of veneers should be used.

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