

# Combining Thermo-Hydro-Mechanical and Phenol-Resin Impregnation Treatments: Potential for High-Density Poplar Flooring

Matthew Schwarzkopf

## Abstract

Thermo-hydro-mechanical (THM) treatments can be used to improve certain properties of underutilized wood species, especially those with low densities. These treatments densify the wood by softening the cell walls using heat, pressure, and moisture but are subject to set-recovery (recovery of compressive deformation) when exposed to humidity. Phenol-based resins have been successful in mitigating this issue when impregnated into the wood. This work explores the use of a new phenol-based resin combined with a THM treatment to limit set-recovery and produce products with the potential for flooring applications. Scratch resistance, hardness, and glue-line shear strength were used to assess the performance. The THM treatment and impregnated phenol resin used in this study increased the scratch resistance, density, and hardness of natural poplar wood and created satisfactory bonding conditions for flooring purposes. With optimization of THM parameters and resin solids content, the resulting product could provide a high-quality flooring material used alone or as a thin laminate from a low-density species like poplar.

European policymakers and research funding in Europe have recently been pushing for reducing the dependency of European countries on imports of raw materials (e.g., proteins, energy, wood, etc.). Part of the strategy is to increase the utilization of all raw material that currently exists. A large number of poplar (*Populus* spp.) clones have been introduced in Europe (and elsewhere) for paper fiber supply but are considered relatively low grade and are not used in higher-value applications. Wood modification techniques have been used to help valorize underutilized wood materials and increase their performance with respect to durability, mechanical characteristics, and new forms and functions desired by consumers and designers alike (Sandberg et al. 2017). One such modification technique is thermo-hydro-mechanical (THM) treatment. THM treatments use heat, water, and mechanical pressure to compress and densify the wood material. This treatment results in increased density, hardness, abrasion resistance, and some strength properties. During the THM process, the wood cell is softened and relaxed by heat and moisture and compressed, resulting in densification without fracturing the cell walls. The densified wood will maintain its shape if it is cooled under pressure. One of the largest challenges facing this modification technique is dimensional stability. If this treated wood is exposed to high levels of moisture or water, it can partially revert to its original dimensions. This behavior is known as set-recovery. Impregnating the wood

microstructure with various curing resins is one technique that has been proposed to increase dimensional stability and reduce set-recovery. The most success has been found with aqueous, phenol-based resins that are impregnated via vacuum treatment into the wood structure. After impregnation, a drying step is applied and followed by THM treatment (Stamm and Seborg 1941; Stamm 1959; Hill 2006; Gabrielli and Kamke 2008, 2010). During the THM treatment, the phenol molecules impart a plasticizing effect on the wood that further reduces cracking in the cell walls (Franke et al. 2016). Once these resins reach a certain temperature, they polymerize throughout the microstructure, improving the dimensional stability. While this approach has found success in the past, there are few commercial products available using this technique. Both THM treatments and phenol resins have been used in the past as wood modification methods to improve either mechanical properties or dimensional stability. This study investigated

The author is Researcher, InnoRenew CoE, Izola, Slovenia, and Assistant Professor, Andrej Marušič Inst., Univ. of Primorska, Koper, Slovenia (matthew.schwarzkopf@innorenew.eu [corresponding author]). This paper was received for publication in January 2020. Article no. 20-00002.

©Forest Products Society 2020.

Forest Prod. J. 70(2):147–150.

doi:10.13073/FPJ-D-20-00002

coupling these techniques to capture the benefits from both. As a trial application, the potential as flooring was explored using a newly developed phenol-based industrial resin and THM process (Kantner et al. 2019). The objective of this study was to explore the potential use of this resin in densified wood flooring or other high-wear applications using poplar.

## Materials and Methods

The general approach of this study was to assess scratch resistance, hardness, and adhesive bond shear strength to gain insight into the viability of using this THM and resin treatment for flooring products.

### Wood specimens

Wood specimens used for THM treatment were produced using *Populus* spp. (poplar). Poplar is diffuse porous, which allows for even densification and impregnation of resin throughout the thickness of the specimens. Wood samples with flatsawn ring orientations were selected and milled to the nominal dimensions of 150 mm in length (L), 150 mm in width (T), and 20 mm in thicknesses (R). After cutting, samples were conditioned at 20°C and 65 percent relative humidity for 1 week. In total, 16 samples were prepared for impregnation.

### Resin impregnation

The aqueous, low-molecular-weight phenol-formaldehyde resin was supplied by Metadynea Austria GmbH (Krems an der Donau, Austria). A 40 percent solids content solution was prepared by mixing the resin with water. After conditioning, wood samples were submerged in the resin solution and vacuum impregnated at 0.001 bar for 1 hour. After impregnation, excess resin was wiped from the samples and dried overnight in ambient conditions. Samples were then dried at 60°C in an oven for 48 hours to remove water from within the panels.

### THM treatment

After drying, impregnated specimens were densified in a hydraulic, 30-ton-capacity “Perfect” LZT-UK-30-L model hot press (Langzauner GmbH, Lambrecht, Austria) equipped with a water-cooling system. To achieve a target thickness of 10 mm, steel hard stops were used (Fig. 1). Two panels were pressed simultaneously within the press. Aluminum foil was placed on the top and bottom to protect the platens from excess resin. Pressing parameters are presented in Table 1. The temperature of 170°C is chosen to target softening of hemicelluloses and lignin in the wood, while the increase in temperature to 200°C for 2 minutes targets the degradation of hygroscopic components of wood



Figure 1.—Left: Thermo-hydro-mechanical and phenol-resin-treated poplar panel; right: unmodified poplar.

Table 1.—Pressing parameters for densification.

Parameter	Value
Upper platen temp. (°C)	170
Lower platen temp. (°C)	170
Closing rate (mm s <sup>-1</sup> )	10
Closing pressure (MPa)	10
Heated holding time (s)	180
Heat treatment upper platen temp. (°C)	200
Heat treatment lower platen temp. (°C)	200
Heat treatment holding time (s)	120
Upper platen cooling temp. (°C)	60
Lower platen cooling temp. (°C)	60

after compression, especially hemicellulose. This has been found to improve the stabilization of densified wood (Kutnar and Šernek 2007). This approach allows compression without fracturing the cell wall and improved stability.

To assess the level of densification achieved, dimensions and weight of specimens were measured before and after THM treatment. Densification ratio was calculated using oven-dry density of the samples compared with the density after THM treatment.

### Specimen preparation

Test specimens were then cut from the THM-treated panels. Scratch and hardness tests used the same specimens with dimensions of 70 by 70 mm.

Two specimen groups were prepared for shear strength. Group 1 was a densified panel adhered to a hardwood plywood panel parallel to the grain. Group 2 was a densified panel adhered to another densified panel parallel to the grain. To ensure that the surfaces were joined well, prior to gluing, the panels were sanded using drum (80 grit) and hand (150 and 220 grit) sanding methods. A D3 polyvinyl acetate (PVA) adhesive, Synturit Universal 33 PLUS (Synthesa, Vienna, Austria), and layup parameters were provided by Metadynea Austria GmbH (Krems an der Donau, Austria). A spread rate of 150 g/m<sup>3</sup> was used, and the specimens were cured in a cold press under 1.72 MPa (250 psi) for 1 hour and left for 24 hours before further processing. Specimens were then cut into final test specimen dimensions with a shear test area of 30 mm<sup>2</sup> and a 10-mm overhang on either side.

### Mechanical testing

Specimen hardness and shear strength were assessed using a 100-kN-capacity AllroundLine model universal test machine (Zwick-Roell, Ulm, Germany). Hardness testing was performed following the hardness modulus test outlined in American Society for Testing and Materials (ASTM) D1037 (ASTM 1999) and methods for testing thin composites in Lewis (1968). As recommended, an extra densified specimen was used as a backing material. A loading rate of 6 mm/min was used. From this test, hardness modulus was derived from the straight-line portion of the load penetration curve. The equivalent Janka ball hardness value was obtained by dividing the hardness modulus value by a factor of 5.4. This factor is based on imperial units, and all necessary conversions were made to calculate the equivalent Janka ball hardness in newtons. Shear strength was assessed using the test standard DIN 52366 (Deutsches

Institut für Normung 1979) as guidance. A loading rate of 2 mm/min was used.

Scratch resistance was assessed using an Erichsen (Hemer, Germany) Scratch Harness Tester 413 following ISO 4586-2 (International Organization for Standards 2018). This assessment was chosen as it is used for high-pressure laminates and parquet flooring, which resemble the densified product produced in this study. The test method uses a 90° diamond test tip with a 90-µm radius. The scratch resistance value reported is the force required to create a visible score mark.

## Results

Results of the THM treatment are presented in Table 2. The THM treatment resulted in poplar specimens with an increased density from 0.37 to 1.03 g/cm<sup>3</sup> (density ratio of 2.78). If we consider the maximum density possible as the wood cell wall density (~1.54 g/cm<sup>3</sup>), there is still room for further densification. However, at these levels, cracking and damage are much more likely to occur. While not at this theoretical level of density, this final density is quite high compared with other THM treatments (0.79 g/cm<sup>3</sup>) applied to poplar (Kutnar et al. 2008) but similar to those found in THM studies with other wood species, like beech (*Fagus sylvatica*) (1.12 g/cm<sup>3</sup>; Schwarzkopf et al. 2017). This large increase in density is beneficial to some mechanical properties (stiffness, hardness, etc.) but does bring some aspects that should be taken into consideration. For example, it is known that the elevated temperature and steam conditions of the compression environment influence the set-recovery of compression deformation and that wood with higher levels of densification show the highest potential for set-recovery (Kutnar et al. 2009, Kutnar and Kamke 2012). This should be considered when designing products, and a lower density ratio may be desirable for applications involving water. The dark red-brown color observed in Figure 1 comes from the cured phenol resin.

Results of mechanical testing are presented in Table 3. As expected, the equivalent Janka hardness value of the THM-treated specimens is very high compared to untreated poplar (~1,735 to 2,400 N) or other, more dense hardwoods, like white oak (*Quercus alba*), which has a hardness value of 6,050 N (Forest Products Laboratory 2010, Meier 2020). This hardness level is also quite high compared to other densified poplar in which the highest density achieved using poplar was 0.79 g/cm<sup>3</sup> (Kutnar et al. 2008).

The mean scratch resistance was found to be 2.49 N, which is significantly increased from natural wood with no THM treatments or phenol impregnation. Keskin et al. (2010) measured the scratch resistance of several wood species including poplar and oak and found values of 0.49 and 1.04, respectively.

The highest mean shear strength was found in Group 1 at 7.68 MPa, in which densified wood was adhered to a beech plywood panel. The higher mean strength in Group 1 versus Group 2 was attributed to the combination of two high-density surfaces with little void space in Group 2, inhibiting the development of a strong bonding interphase. The values for both groups are similar to those found in a study conducted by Blanchet et al. (2003), who reported a strength value of 4.3 MPa for PVA glue lines. In another study by Wang et al. (2017), oak surface layers were bonded to poplar plywood using a polyurethane reactive adhesive. Shear strength of their samples was near 3.0 MPa. Fang et al. (2012) also studied the use of THM treatments (without impregnated resin) on sugar maple (*Acer saccharum*) veneers for use in engineered wood flooring. They observed shear strength values of 13.1 MPa when gluing two densified pieces together. While many test factors (e.g., species, THM parameters, etc.) vary, it is possible that the use of impregnated phenol resins decreases the bond strength. However, shear strength values were still higher than uncompressed sugar maple bond lines used in flooring applications.

## Conclusions

The THM treatment and impregnated phenol resin used in this study increased the scratch resistance, density, and hardness of natural poplar wood and created satisfactory bonding conditions for flooring purposes. The high levels of density and hardness may not be needed in practice, and more efficient flooring products could be created with modifications to the THM treatment targeting lower densities. These modifications may also allow for better bonding characteristics. Additionally, a lower solids level in the impregnated resin could also help lead to more realistic densities while maintaining dimensional stability and performance. With these optimizations, a high-quality flooring material used alone as a massive piece or as an upper laminate in engineered wood flooring could be produced from a low-density species like poplar. By using thinner sections of material in a laminate configuration,

Table 2.—Results of thermo-hydro-mechanical densification treatment. Data are means with standard deviations shown in parentheses.

Initial thickness (mm)	Ovendry density (g cm <sup>-3</sup> )	Compressed thickness (mm)	Compressed density (g cm <sup>-3</sup> )	Densification ratio
19.94 (0.16)	0.37 (0.05)	10.10 (0.54)	1.03 (0.12)	2.78 (0.16)

Table 3.—Mechanical test data of impregnated, thermo-hydro-mechanical-treated specimens. Data are means with standard deviations shown in parentheses.

Test specimen	Shear strength (MPa)	Scratch (N)	Modulus of hardness (MPa)	Equivalent Janka ball hardness (N)	n
Scratch/hardness	—	2.49 (0.68)	233 (135)	27,920 (16,190)	16
Shear Group 1	7.68 (2.63)	—	—	—	36
Shear Group 2	5.68 (2.19)	—	—	—	24

resin costs could be reduced while still providing the same performance.

### Acknowledgments

The author gratefully acknowledges the European Commission for funding the InnoRenew CoE project (Grant Agreement #739574) under the Horizon2020 Widespread-Teaming program and the Republic of Slovenia (investment funding of the Republic of Slovenia and the European Union of the European regional Development Fund). The author would also like to graciously thank the Metadynea company for their great knowledge, donation of the resin used in this study, and general support.

### Literature Cited

- American Society for Testing and Materials (ASTM). 1999. Standard test methods for evaluating properties of wood-base fiber and particle panel materials. ASTM D1037-99. ASTM, West Conshohocken, Pennsylvania.
- Blanchet, P., R. Ceauregard, A. Erb, and M. Lefebvre. 2003. Comparative study of four adhesives used as binder in engineered wood parquet flooring. *Forest Prod. J.* 53(1):89–93.
- Deutsches Institut für Normung. 1979. Testing of wood: Determination of ultimate shearing stress parallel to grain. DIN 52187. Deutsches Institut für Normung, Berlin.
- Fang, C. H., P. Blanchet, A. Cloutier, and C. Barbuta. 2012. Engineered wood flooring with a densified surface layer for heavy-duty use. *BioResources* 7(4):5843–5854.
- Forest Products Laboratory. 2010. Wood Handbook: Wood as an Engineering Material. US Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, Wisconsin.
- Franke, T., C. Lenz, N. Herold, and A. Pfriem. 2016. Investigations on phenol/formaldehyde impregnation of beech veneer for improved plasticization and form stability. In: WCTE 2016—World Conference on Timber Engineering, J. Eberhardsteiner, W. Winter, A. Fadai, and M. Pöll (Eds.), August 22–25, 2016, Vienna, Austria; TU Verlag, Vienna. 8 pp.
- Gabrielli, C. and F. A. Kamke. 2008. Treatment of chemically modified wood with VTC process to improve dimensional stability. *Forest Prod. J.* 58(12):82–86.
- Gabrielli, C. P. and F. A. Kamke. 2010. Phenol-formaldehyde impregnation of densified wood for improved dimensional stability. *Wood Sci. Technol.* 44(1):95–104.
- Hill, C. A. S. 2006. Wood Modification: Chemical, Thermal and Other Processes. John Wiley & Sons, New York.
- International Organization for Standards (ISO). 2018. High-pressure decorative laminates (HPL, DPDL)—Sheets based on thermosetting resins (usually called laminates)—Part 2: Determination of properties. ISO 4586-2. ISO, Geneva.
- Kantner, W., T. Zich, M. J. Schwarzkopf, M. D. Burnard, M. Mikuljan, and A. Kutnar. 2019. Method for Preparation of Densified Wood Article. Metadynea Austria GmbH, Vienna.
- Keskin, H., M. Atar, S. Korkut, and A. Tekin. 2010. Scratch resistance of cellulosic, synthetic, polyurethane, waterborne, and acid-hardening varnishes used on woods. *Ind. Crops Prod.* 31(2):219–224.
- Kutnar, A. and F. A. Kamke. 2012. Influence of temperature and steam environment on set recovery of compressive deformation of wood. *Wood Sci. Technol.* 46(5):953–964.
- Kutnar, A., F. A. Kamke, and M. Šernek. 2008. The mechanical properties of densified VTC wood relevant for structural composites. *Eur. J. Wood Wood Prod.* 66(6):439–446.
- Kutnar, A., F. A. Kamke, and M. Šernek. 2009. Density profile and morphology of viscoelastic thermal compressed wood. *Wood Sci. Technol.* 43(1–2):57–68.
- Kutnar, A. and M. Šernek. 2007. Densification of wood. *Zb. gozdarstva Lesar.* 82:53–62.
- Lewis, W. C. 1968. Hardness modulus as an alternative measure of hardness to the standard Janka ball for wood and wood-base materials. Research Note FPL-RN-0189. US Department of Agriculture, Forest Service, Forest Products Laboratory, Madison, Wisconsin.
- Meier, E. 2020. Poplar. In: Wood Database. <https://www.wood-database.com/poplar>. Accessed January 16, 2020.
- Sandberg, D., A. Kutnar, and G. Mantanis. 2017. Wood modification technologies—A review. *iForest* 10(6):895–908.
- Schwarzkopf, M., M. Burnard, G. Martínez Pastur, L. Monelos, and A. Kutnar. 2017. Performance of three-layer composites with densified surface layers of *Nothofagus pumilio* and *N. antarctica* from Southern Patagonian forests. *Wood Mater. Sci. Eng.* 13(5):1–11.
- Stamm, A. 1959. The dimensional stability of wood. *Forest Prod. J.* 9(10):375–381.
- Stamm, A. J. and R. M. Seborg. 1941. Resin-treated, compressed wood. *Trans. Am. Inst. Chem. Eng.* 37385–37397.
- Wang, H., L. Huang, P. Cao, F. Ji, G. Yang, X. Guo, and R. Li. 2017. Investigation of shear strength of engineered wood flooring bonded with PUR by response surface methodology. *BioResources* 12(2):3656–3665.