Drewno 2022, Vol. 65, No. 209
DOI: 10.12841/wood.1644-3985.380.01

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EFFECT OF THERMAL MODIFICATION ON SOME PHYSICAL AND MECHANICAL PROPERTIES OF YELLOW POPLAR (*Liriodendron tulipifera*)

Thermal modification is a treatment method used to improve some properties of wood in order to expand its range of usage and extend its lifetime. Although it generally causes a worsening of mechanical properties, some of them can change due especially to lower equilibrium moisture content when compared with untreated wood. Therefore, we wanted to compare some properties of thermally modified and untreated samples having either the same moisture contents or those of the area of use. For this purpose, we tested untreated and thermally modified (at 180° C for 3 hours) yellow poplar (Liriodendron tulipifera) solid wood samples. We investigated density, swelling and shrinkage ratios, and anisotropy of shrinkage as physical properties, and compression and bending strength, modulus of elasticity and impact bending as mechanical properties, according to the relevant ISO standards. The results showed that thermal modification increased the dimensional stability while having a negative impact on the mechanical properties (except modulus of elasticity) at the same moisture content, as indicated in the literature. However, thermally modified samples with moisture content as in the area of use exhibited improved properties (except in the case of impact bending).

Keywords: thermal modification, yellow poplar, dimensional stability, mechanical properties, usage area

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Introduction

Wood is utilized in many areas of use due to its various favorable properties. However, climatic conditions such as rain, sunlight, etc. pose a risk of dimensional change, decaying, discoloration, etc., especially in case of outdoor usage.

Many methods of impregnation and modification have been developed to extend the service life of wood. In particular, thermal modification (heat treatment) has been proposed as a natural and additive-free method leading to good aesthetic properties.

There are many methods using air, steam, oil or inert gases, different combinations of temperature and duration, etc. [Giebeler 1983; Sailer et al. 2000; Esteves et al. 2007]. One of these is the ThermoWood method, developed by VTT, in which the wood material is heated at 180 °C for stability and at 212 °C for durability, being protected with steam [Mayes and Oksanen 2002]. All thermal modification methods generally improve dimensional stability and durability properties, while negatively influencing some mechanical properties. For example, Salca and Hiziroglu [2014] reported lower equilibrium moisture content, density and contact angles for wettability, which are also important advantages of thermally modified wood, citing Candan et al. [2012] and Yildiz et al. [2011]. The method generally improves resistance to biological deterioration [Metsa-Kortelainen et al. 2006]. Bekhta and Niemz [2003], Mitsui et al. [2004] and many other researchers have reported that treated samples become darker with an increase in treatment time and temperature. Giebeler [1983] found a reduction in the modulus of rupture of wood by 20% to 50% after thermal treatment at 180 °C to 200 °C. Although the effect is generally similar, the impact ratios of the method depend on the tree species, as Giebeler [1983] and others have indicated. For example, Möttönen et al. [2015] found decrements with different ratios for birch and aspen species, while Godinho et al. [2021] found different decrement ratios for ash, iroko, Scots pine and spruce. Despite the negative impact on mechanical properties, the thermal modification method is becoming more popular, adding value to the wood of certain species, particularly fast-growing species.

One fast-growing species is yellow poplar (or tulip-poplar, *Liriodendron tulipifera* L.), which can reach sizes of 50 m (160 ft) in height and 2.5 m (8 ft) in trunk diameter. The sapwood is white, while the heartwood is commonly tan but varies in color, ranging from yellow (especially in trees of rapid growth) or tan to greenish brown. On exposure to air and light, the surface darkens. Although it is among the commercially important hardwoods in the United States, it ranks in the lower one-third of the range of many mechanical properties. The initial shrinkage is relatively large, but the wood stays in place well after drying. Lumber is by far the largest use, and secondary lumber is cut mostly for furniture, interior finishing, core stock for veneered furniture panels and plywood, and dimension

stock [Buchanan and Dickey 1960; Vick 1985; Beck 1990; Brito et al. 2018; WoodDatabase 2020]. Properties of yellow poplar are summarized in Table 1 [Ross 2010].

There are several existing studies of the thermal modification of yellow poplar. Ülker et al. [2018] investigated roughness, color and hardness changes with thermal modification of yellow poplar and two other species. After treatment at 130 °C, 160 °C and 190 °C for 8 hours, they found significant decrements of lightness, discoloration, surface roughness and hardness values for yellow poplar. Brito et al. [2018] found significant darkening, blueness and reddening after thermal modification, and the changes increased with temperature increments. They explained the change as resulting from degradation in the hemicelluloses, citing Sundqvist and Morén [2002] and Mitsui et al. [2004]. Salca and Hiziroglu [2014] investigated the effects of thermal modification (at 120 °C and 190 °C for 3 and 6 hours) on the hardness and surface quality of yellow poplar and three other tree species. The results showed that there was no significance difference for hardness. On the other hand, all tree species had smoother surfaces, especially after modification at 190 °C for 6 h. Esteves and Pereira [2009] reported that thermal modification generally decreases the mechanical strength. However, modulus of elasticity (MOE) can increase in softer treatments and decrease in more severe treatments. Rahimi et al. [2019] found a decrement in MOE, while compression strength was similar for untreated samples and for a modification process involving the presence of both steam and hot-compressed water and relatively low temperatures (100 °C and 140 °C).

Table 1. Some properties of yellow poplar [Ross 2010]

Shrinkage (%) (green to oven-dry)	Radial	Tangential	Volumetric
	4.6	8.2	12.7
	Green		Air-dry (12%)
Specific gravity	0.40		0.42
Compression parallel to grain (MPa)	18.3		38.2
Modulus of rupture (MPa)	41.0		70.0
Modulus of elasticity (MPa)	8400.0		10900.0

Although there have been publications concerning some physical properties, there is little information about the effect of thermal modification on the mechanical properties of yellow poplar. Therefore, we researched papers about other hardwood species, and found reports of decrements for impact bending [Korkut et al. 2008; Gaff et al. 2019], static bending strength [Poncsak et al. 2006; Shi et al. 2007], and compression strength [Unsal and Ayrilmis 2005; Korkut et al. 2012].

The aim of this study was to observe the effects of the thermal modification process on some physical and mechanical properties of yellow poplar wood. In particular, we wanted to compare usage area performances of treated and untreated materials. Additionally, we wished to evaluate the stability of this species in terms of the tangential-to-radial shrinkage ratio (T/R ratio), because this serves to measure the uniformity of the shrinkage, and a low T/R ratio is considered to be desirable. As a general rule of thumb, for most species the T/R ratio is about 2, and it can range from just over 1 to nearly 3 [Cockrell 1974; WoodDatabase 2020]. It can vary depending on the species, reaction wood, or modification methods. For example, Cockrell [1974] found a T/R ratio of 1 for compression wood of the giant sequoia (*Sequoia gigantea* (Lindl.) Decne.), compared with 1.6–2.1 for normal wood. Almeida et al. [2009] found a decrement in T/R ratio with an increment in thermal modification temperature and the inclusion of nitrogen in the atmosphere, for three *Eucalyptus* species.

Materials and methods

We obtained yellow poplar (*Liriodendron tulipifera*) planks from a commercial company. There were two groups of samples: untreated (control) and treated (Fig. 1). The treated planks were modified at 180 °C for 3 hours to gain stability in an industrial thermal modification kiln (Thermo-S) according to the ThermoWood principle, developed by VTT (Finland). We cut the planks to the sample sizes of related standard test methods. All samples were from heartwood and contained no defects.

Fig. 1. Samples for measurement of physical properties (darker: treated)

Table 2 shows the tests, related standards and sample numbers. Before the tests, we conditioned the samples at 20 °C and 65% relative humidity to obtain air-dry conditions. In addition to the physical tests, we calculated the anisotropy of shrinkage using the proportion of tangential to radial shrinkage (T/R ratio).

All mechanical and density standards have a formula for adjusting the results to 12% moisture content. However, only ISO 13061-17 [2017] suggests taking a correction factor from a national standard. The last valid national standard [TS 2595 1977] suggested using 0.05 as a correction factor for the difference of the moisture content from 12%. We calculated all coefficients of variation for the mechanical properties, and they were within the limits suggested by Ross [2010]. We performed a t-test to verify differences of all averages among groups, with a 95% confidence level ($p < 0.05$). We used IBM SPSS software for statistical analyses.

Table 2. Design of experiment

	Tests (abbreviation & unit)	Number of samples		Standard
		Control	Treated	
Physical	Oven-dry Density (D-OD, g/cm^3)	44	39	ISO 13061-2 [2014]
	Air-dry Density (D-AD, g/cm^3)	44	39	
	Moisture Content (MC, %)	44	39	ISO 13061-1 [2014]
	Shrinkage (Sh, %)	20	19	ISO 13061--13, 14, 15 & 16 [2016]
	Swelling (Sw, %)	24	20	
	Anisotropy of shrinkage (T/R ratio, %/%)	20	19	Tangential Sh / Radial Sh
Mechanical	Compression strength parallel to grain (Cs, MPa)	28	30	ISO 13061-17 [2017]
	Bending strength (MOR, MPa)	25	26	ISO 13061-3 [2014]
	Modulus of elasticity in bending (MOE, MPa)	25	26	ISO 13061-4 [2014]
	Impact bending (IB, kJ/m^2)	30	30	ISO 13061-10 [2017]

Results and discussion

A comparison of the results for physical properties is given in Table 3, and for mechanical properties in Table 4.

Although the samples were conditioned in air-dry conditions (20 °C and 65% RH, implying 12% EMC), the moisture content of the control samples was 11.30% (± 0.41). The reason for the lower EMC values may be the phenomenon known as hysteresis, as Brito et al. [2018] indicated in relation to kiln-dried samples. On the other hand, the EMC values of treated samples were found to be 4.94% (± 0.41). Brito et al. [2018] reported that the reason for the decrease in EMC was that cross-linkage between lignin and polymers decreases the hygroscopicity and improves the dimensional stability. Additionally, they explained it by the degradation of hemicelluloses and the amorphous region of cellulose, contributing to an increase in the degree of crystallinity of the polymer, citing Esteves and Pereira [2009].

Table 3. Comparison of physical properties

Tests			Control (C)	Treated (T)	Comparison
Air-dry equilibrium moisture content (EMC, %)			11.30 (0,41)[a]	4.94 (0.41)[b]	C > T
Shrinkage**	EMC -> 0%	Radial (%)	2.17 (0.22)[a]	1.75 (0.26)[b]	C > T
		Tangential (%)	2.15 (0.37)[a]	1.75 (0.20)[b]	C > T
		Volumetric (%)	4.25 (0.36)[a]	3.51 (0.36)[b]	C > T
	FSP -> 0% (Total)	Radial (%)	4.91 (0.40)[a]	3.56 (0.57)[b]	C > T
		Tangential (%)	7.21 (0.46)[a]	4.71 (0.47)[b]	C > T
		Volumetric (%)	11.83 (0.45)[a]	8.08 (0.65)[b]	C > T
	T/R (%/%) - (FSP -> 0%)		1.56 (0.09)[a]	1.34 (0.17)[b]	C > T
Swelling**	EMC -> FSP	Radial (%)	3.08 (0.41)[a]	2.24 (0.30)[b]	C > T
		Tangential (%)	6.01 (0.45)[a]	3.75 (0.64)[b]	C > T
		Volumetric (%)	9.33 (0.42)[a]	6.16 (0.80)[b]	C > T
	0% -> FSP (Total)	Radial (%)	5.84 (0.48)[a]	3.99 (0.42)[b]	C > T
		Tangential (%)	9.81 (0.47)[a]	5.62 (0.69)[b]	C > T
		Volumetric (%)	15.16 (0.54)[a]	9.60 (0.98)[b]	C > T
	Density (g/cm^3)	Oven-dry	0.47 (0.01)[a]	0.44 (0.01)[b]	C > T
		Air-dry	0.50 (0.03)[a]	0.45 (0.01)[b]	C > T
		Air-dry (adjusted to 12%)	0.51 (0.01)[a]	0.48 (0.01)[b]	C > T

* The same letter (and ~ in the comparison column) in each row shows that there is no significant difference ($p < 0.05$). FSP: Fiber Saturation Point.

** ISO standards do not consider the parallel-to-grain dimension for the calculation of volumetric shrinkage and swelling.

The results showed that all shrinkage and swelling values for the treated samples were lower than for the untreated samples. Moreover, the shrinkage ratios of treated samples were lower when based on EMC, although the differences between FSP and EMC were higher due to treated samples having lower EMC. The volumetric shrinkage ratios of untreated samples were similar, while the ratios of treated samples were lower when both steam and hot-compressed water were present in the modification process [Rahimi et al. 2019]. The difference may be related to the lower treatment temperatures. Total volumetric shrinkage ratios of untreated samples ranked as "medium", while after treatment the ranking decreased to "low", according to the classification of As et al. [2016]. The T/R ratio, serving as an indicator of shrinkage homogeneity, significantly decreased after treatment. These results confirm that thermal modification decreases the T/R ratio, as Almeida et al. [2009] indicated for *Eucalyptus*. Although the differences were statistically significant, the decrement was relatively low. Nevertheless, it can be concluded that the treated wood exhibits more stable behavior in usage; in other words, the treated material can substantially preserve its shape after swelling or shrinkage. Thermally modified yellow poplar is currently sold as "dimensionally stable" or having "superior stability" [Brito et al. 2018; Northland Forest Products 2018; Atlanta Hardwoods 2018]. The results of this study support those definitions.

We found significant density decrement in the treated samples. Rahimi et al. [2019] found that the oven-dry specific gravities of all samples were between 0.49 and 0.50. They did not find significant change after hydrothermal treatment using lower temperatures (100 °C and 140 °C < 180 °C). The decrement found in our study may indicate the degradation of some components and the separation of volatile components and extractives at higher temperatures. The decrement may also indicate other mass loss apart from volatile components and extractives. However, we suggest using other techniques, such as Todorović et al. [2020] indicated would be superior because of influencing factors.

Table 4. Comparison of mechanical properties*

Mechanical tests at given moisture content (MC) (%)		Control (C)	Treated (T)	Comparison
IB	Adjusted MC	54.92 (3.92)^a	38.25 (6.87)^b	C > T
(kJ/m^2)	Different MC	57.89 (3.92)^a	47.07 (8.83)^b	C > T
Cs	Adjusted MC	38.79 (2.48)^a	35.50 (3.77)^b	C > T
(MPa)	Different MC	46.75 (2.95)^a	54.19 (5.58)^b	C < T
MOR	Adjusted MC	77.50 (4.02)^a	63.56 (5.48)^b	C > T
(MPa)	Different MC	84.55 (5.16)^a	87.90 (7.79)^a	C ≈ T
MOE	Adjusted MC	9.73 (0.56)^a	9.98 (0.75)^a	C ≈ T
(MPa)	Different MC	10130 (590)^a	11370 (870)^b	C < T

* The same letter (and ≈ sign) in each row shows that there is no significant difference ($p < 0.05$).

The modulus of elasticity (MOE) values of our untreated samples were lower than those reported by both Faust et al. [1990] and Rahimi et al. [2019], but were similar to those of Ross [2010]. Faust et al. [1990] found a lower modulus of rupture (MOR; 48 MPa) for untreated samples. However, they studied structural--sized lumbers (5 cm × 10 cm × 365 cm), which might have defects and decreased MOR. With these dimensions, span distance and annual ring thickness, which are important for fast-growing species, may have affected the MOE and MOR results. Rahimi et al. [2019] found increments in MOE for hydrothermal treatments, especially in hot-compressed water at 100 °C and in steam at 140 °C, while we did not find significant differences for MOE. This may result from increased cross-linking of the lignin polymer network [Boonstra et al. 2007]. The MOR, compression strength and impact bending (IB) values of the treated samples significantly decreased. The reason may be the degradation of wood polymers when subjected to temperatures above 155 °C [Singh and Sivanandan 2014], resulting in more brittle or fragile samples.

As seen in Table 4, thermal modification decreased all mechanical properties at the same moisture level (except MOE). Many studies indicate the negative impact of thermal modification on mechanical properties. However, researchers equalize the moisture contents using correction factors to enable comparison when investigating the effects of thermal modification. Additionally, we wished to compare the samples with moisture contents for the area of usage. The results showed that all mechanical properties improved (except IB); moreover, they were better than for the untreated control samples when we considered samples as in the area of usage. The main reason is likely to be the lower EMC of treated samples (11.30% and 4.94%), as Boonstra et al. [2007] indicated.

Conclusions

The results of this study showed that thermal modification with the described process conditions increased the stability of yellow poplar heartwood, while the values of density and mechanical properties (except MOE) decreased. Especially a lower equilibrium moisture content had a positive effect when the conditions of the area of usage were considered. These differences show that researchers can additionally compare results without adjusting the moisture content when evaluating performance in a given area of usage.

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List of standards

ISO 13061–1:2014 Physical and mechanical properties of wood – test methods for small clear wood specimens – Part 1: Determination of moisture content for physical and mechanical tests

ISO 13061–2:2014 Physical and mechanical properties of wood – test methods for small clear wood specimens – Part 2: Determination of density for physical and mechanical tests

ISO 13061–3:2014 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 3: Determination of ultimate strength in static bending

ISO 13061–4:2014 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 4: Determination of modulus of elasticity in static bending

ISO 13061–10:2017 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 10: Determination of impact bending strength

ISO 13061–13:2016 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 13: Determination of radial and tangential shrinkage

ISO 13061–14:2016 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 14: Determination of volumetric shrinkage

ISO 13061–15:2016 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 15: Determination of radial and tangential swelling

ISO 13061–16:2016 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 16: Determination of volumetric swelling. Physical and mechanical properties of wood – test methods for small clear wood specimens – Part 1: Determination of moisture content for physical and mechanical tests

ISO 13061–17:2017 Physical and mechanical properties of wood – Test methods for small clear wood specimens – Part 17: Determination of ultimate stress in compression parallel to grain

TS 2595:1977 Paper – Determination of bursting strength

Submission date: 24.12.2020

Online publication date: 31.07.2022