COLOR CHANGES, EMC, AND BIOLOGICAL RESISTANCE OF THERMALLY MODIFIED YELLOW POPLAR

A. F. Brito

Agronomist Engineer
Department of Forest Science
School of Agriculture, São Paulo State University (UNESP)
P.O. Box 237
Botucatu, São Paulo, Brazil
E-mail: alinefernanda03@yahoo.com.br

F. W. Calonego

Forest Engineer
Department of Forest Science
School of Agriculture, São Paulo State University (UNESP)
P.O. Box 237
Botucatu, São Paulo, Brazil
E-mail: fwcalonego@ig.com.br

*B. H. Bond**†

Professor
Department of Sustainable Biomaterials
Brooks Forest Products Center, Virginia Tech
1650 Ramble Road
Blacksburg, VA 24061
E-mail: bbond@vt.edu

E. T. D. Severo

Adjunct Professor
Department of Forest Science
School of Agriculture, São Paulo State University (UNESP)
P.O. Box 237
Botucatu, São Paulo, Brazil
E-mail: severo@fca.unesp.br

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Abstract. *Liriodendron tulipifera* L., known as yellow poplar, are fast-growing trees, make up about 17% of commercially available hardwood in North America, and are generally used in furniture, doors, and millwork. The wood is used mostly where it would be hidden or painted. The value of yellow poplar is less than that of other hardwoods with more pronounced grain and color. This study evaluated the effect of various levels of thermal treatment on color and resistance to decay fungus and termites of yellow poplar wood. Boards (28.57 mm thickness × 150 mm width × 3.65 m length) were taken from a sawmill and thermally modified at temperatures of 180°C, 200°C, and 220°C. A summary of the findings were that thermal treatment caused: 1) darkening and reddening of yellow poplar wood; 2) a reduction of up to 51.4% in EMC when exposed to 21°C and 65% relative humidity, and 3) a significant increase in wood decay resistance against decay fungus *Pycnoporus sanguineus*. Treatment at 220°C resulted in a change in American Society of Testing Materialswood decay resistance class from slightly resistant to highly resistant; and no significant changes in resistance against the dry-wood termites *Cryptotermes brevis* were found.

Keywords: *Pycnoporus sanguineus*, *Cryptoermes brevis*, *Liriodendron tulipifera*, thermal modification, colorimetric values.

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* Corresponding author
† SWST member

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INTRODUCTION

Yellow poplar (*Liriodendron tulipifera* L.) is a fast-growing hardwood species that is abundant in North America and it ranks in the lower third in the range of commercially important hardwoods in the United States for density, mechanical strength and low biological resistance (Alden 1995). The wood is considered easy to machine and generally used for furniture, millwork, veneer, and pallets. The sapwood is white and the heartwood usually tan. The color of the wood is of some importance because of its influence on the commercial value (Camargos and Gonçalez 2001; Meints et al 2017) and may be considered distinctly yellow (Buchanan and Dickey 1960).

Yellow poplar has not been used for exterior applications because of its low decay and insect resistance. For example, brown-rot fungus *Gloeophyllum trabeum* and white-rot fungus *Trametes versicolor* caused weight losses of 68.5% (Shi et al 2007) and 61.85% (Schirp and Wolcott 2005), respectively. When exposed to subterranean termites *Reticulitermes flavipes*, high visual attack and a weight loss of 13.4% was noted (Shi et al 2007).

Thermal modification techniques provide a significant opportunity to both increase the value and open new markets for yellow poplar lumber. Thermal treatments expose timber to temperatures approaching 200°C for several hours, changing the chemical composition (Esteves and Pereira 2009). Thermal modification has opened new market opportunities for species such as yellow poplar for siding, flooring, and doors and is considered to be a fast-growing market (Espinoza et al 2015).

Thermal modification reduces the EMC of wood and the availability of nutrients (hemicelluloses) to fungi. Cross-linking occurs between the lignin and the cellulose polymer, making recognition by fungi difficult (Esteves and Pereira 2009). It also results in new molecules that act as fungicides (Mburu et al 2007) and possibly the production of compounds toxic to termites (Pessoa et al 2006; Mburu et al 2007).

The only published research on the thermal modification of yellow poplar assesses the hardness and surface quality. Salca and Hiziroglu (2014) determined that when yellow poplar was treated at 190°C for 6 h the Janka hardness decreased 7.9%, whereas the surface quality was enhanced, potentially adding value to the wood for its use in furniture manufacturing. Currently, thermally modified yellow poplar is being sold as "highly durable" or with superior stability and "exceptional resistance to weather and fungi-related deterioration," and "dimensionally stable" or having "superior stability" (Northland Forest Products 2018 and Atlanta Hardwoods 2018).

Little information currently exists in the literature regarding the impact of thermal modification on color changes and biological resistance of yellow poplar wood. Thus, the objective of this study was to evaluate the effects of thermal treatment on the color, EMC, and the resistance to decay fungus and termites for this specie. Although not evaluated in this report, it should also be noted that although many of the aforementioned studies on thermally modified wood found reduced hygroscopity and improved decay resistance, many also noted reduced mechanical properties.

MATERIALS AND METHODS

Wood samples were obtained from six 30- to 45-yr-old *L. tulipifera* L. (Magnoliaceae) trees, harvested from a naturally grown forest, located within 65 miles of Hillsville, VA. The trees were felled, sectioned into logs, and cut into flat-sawn boards with 28.57 mm thickness × 150 mm width × 3.65 m length. The boards were then dried to 8% MC. One dried 3.65-m-long board each from six trees was planed to 25-mm thickness and cut into four samples measuring 0.60 m in length free of cracks and knots. From each board, one sample was kept in its original condition (untreated wood), and the other three samples were used for the thermal modification treatments. Specimens from the four samples cut from each board were then cut to test for determination of color, EMC, and decay resistance.

Thermal Treatment

Three different thermal modification treatments were applied using an electric oven with a programmable controller. The first treatment started at an initial temperature of 100°C over a period of 14 h and then the temperature was increased (1.34°C/min) up to 180°C and maintained over a period of 2.5 h (Severo and Calonego 2011). The same procedure was performed at a final temperature of 200°C and then 220°C. Thermal treatments were conducted with exposure to atmospheric air. After thermal treatment, the wood pieces were allowed to cool naturally until they reached 30°C. Samples were then stored in a conditioning chamber until they equilibrated to a 12% EMC.

Specimens were cut from all the samples (untreated and thermally modified) according to the methods described in IPT-1157 (1980), ASTM E-308 (1999), and ASTM D-2017 (2008) for colorimetric characterization and decay and termite resistance tests. All specimens were cut approximately 40 mm from the pith.

Determination of Color

The International Commission on Illumination L *A *B* color space was used to evaluate the effect of thermal treatment on the color change of yellow poplar wood. We used six samples, one obtained from each of the six boards in each of the four groups (control and three thermally modified wood), for a total of 24 samples. The specimens measured 25 × 25 × 200 mm (tangential, radial, and longitudinal direction, respectively). The color of various points on the surface of each sample was measured using a reflectance spectrophotometer with 11-mm diameter field of view and 8° optical geometry angle. The equipment was calibrated with illuminant D65 (daylight) and an observer angle of 10°. The colorimetric parameters L* (brightness), a* (coordinate of the green-red axis), b* (coordinate of the blue-yellow axis), and E* (color) were determined as recommended by ASTM E-308 (1999). The color was measured at three points (each point with 95-mm^2 area) on the surface of each specimen for a total measured area of 285 mm^2.

Determination of EMC

Wood samples prepared for decay resistance were also used to evaluate the effect of thermal treatment on the EMC. Untreated and thermally modified wood samples were dried at 103 ± 2°C until they reached 0% MC. Subsequently, the samples were placed in a climatic chamber adjusted to 21°C and 65% RH until they reached EMC. The samples were weighed and the results were used to determine differences in moisture behavior.

Accelerated Laboratory Tests of Decay Resistance

The methodology outlined in ASTM D-2017 (2008) was used to evaluate the effect of thermal treatment on the decay resistance of yellow poplar wood. Wood samples were prepared to 25 × 25 × 9 mm (tangential, radial, and longitudinal direction, respectively) dimensions. Although the standard requires only six test blocks, we used a total of 18 obtained from the three test blocks per treatment for each of the six specimens from the four boards, totaling 72 test blocks.

The test blocks were dried at 103 ± 2°C to a constant weight and then acclimated to 12% MC. The test blocks were then placed in the culture bottles, with the cross-section face down on the feeder strip. The cultures of *Pycnoporus sanguineus* fungus were incubated in the dark at 26.7 ± 1°C and 70 ± 4% RH for 12 wk. *P. sanguines* is a common white-rot fungus in Brazil and has been used for several other thermally modified wood studies (Andrade et al 2012). The percent weight loss in the individual test block was then calculated to provide a measure of the relative decay susceptibility or, inversely, the decay resistance of the yellow poplar wood.

Laboratory Evaluation for Resistance to Termites

The IPT-1157 (1980) methodology was used to evaluate the effect of thermal treatment on termite resistance. Wood samples were prepared to

3 × 23 × 70 mm (radial, tangential, and longitudinal direction, respectively). Eight test blocks were obtained from each of the six boards to characterize each of the treatments, totaling 32 test blocks.

The test blocks were dried at 103 ± 2°C to a constant weight and reweighed. Two test blocks were each placed in Petri dishes, and a polyvinyl chloride tube measuring 2 inches in height and 1.5 inches in diameter was placed on the cross-section face of test blocks (superficial area of 32.2 cm^2), restricting the area of termite attack of the wood to 11.4 cm^2. Forty (39 workers and one soldier) dry-wood termites *Cryptotermes brevis* were added to each of the previously prepared containers. All containers were incubated in the dark at 27 ± 2°C and 70 ± 2% RH for 45 d.

The mortality rate of termites and damage was visually rated (0, no attack; 1, light attack; 2, moderate attack; 3, accentuated attack; and 4, heavy attack) and the number of holes and the percent weight loss in the test blocks was then evaluated. Comparisons of both the number of holes and weight loss between the thermally modified wood and the control (untreated) were then calculated and expressed as a percent difference.

Statistical Analysis

Color changes and differences in decay and termite resistance were evaluated using a Kolmogorov–Smirnov's normality test at 5% significance. Variables had normal distribution, except fungal and termite weight losses, the number of holes, and the visual damage ratings to termites.

A parametric one-way analysis of variance at 5% significance was then performed taking into account the thermal treatment (four levels), as well as Tukey's test at 5% significance for the comparison of the means in colorimetric parameters, EMC before and after test fungi, and termite mortality rate. A nonparametric Kruskal–Wallis test was performed taking into account the thermal treatment, as well as Dunn's test at 5% significance for the comparison of the means for weight losses, number of holes, and visual damage ratings.

RESULTS AND DISCUSSION

Color Changes from Thermal Modification

The brightness (L*), coordinate green-red (a*), coordinate blue-yellow (b*), and color (E*) values and coefficient of variation for each treatment level are presented in Fig 1. The respective colorimetric properties of untreated yellow poplar wood were 70.03, 3.98, 22.66, and 73.74. The wood can be classified as greyish-white because of its high brightness and strong presence of yellow pigment because of high b* value. Our results are similar to those of Buchanan and Dickey (1960), who stated that the color of yellow poplar may be distinctly yellow and by Alden (1995), who related that the sapwood is white and the heartwood usually tan.

Thermal treatment resulted in significant darkening (ΔL* up to −37.66), blueness (Δb* up to −13.19), and reddening (Δa* up to 1.98) in the wood with increasing thermal treatment temperature when compared with the untreated wood (Fig 1). Darkening and reddening of light-colored wood with thermal treatment is common. Sundqvist and Morén (2002), concluded that *Betula pubescens* wood also darkens and reddens after thermal treatment, as well as those reported by Bekhta and Niemz (2003), who showed that *Picea abies* wood when thermally modified at 200°C for 24 h presents less brightness (ΔL* −56.24) and 53 times more red and blue than untreated wood.

The colorimetric changes that occur during thermal modification have been explained by Sundqvist and Morén (2002) and Mitsui et al (2004) as resulting from the degradation in the hemicelluloses. Chemical variations that vary by thermal treatment can also influence color variation (Severo et al 2012, 2016).

EMC

The EMC of untreated wood (Table 1) averaged 10.11% when acclimatized at 21°C and 65% RH. The MC of wood was lower than usual (12%)

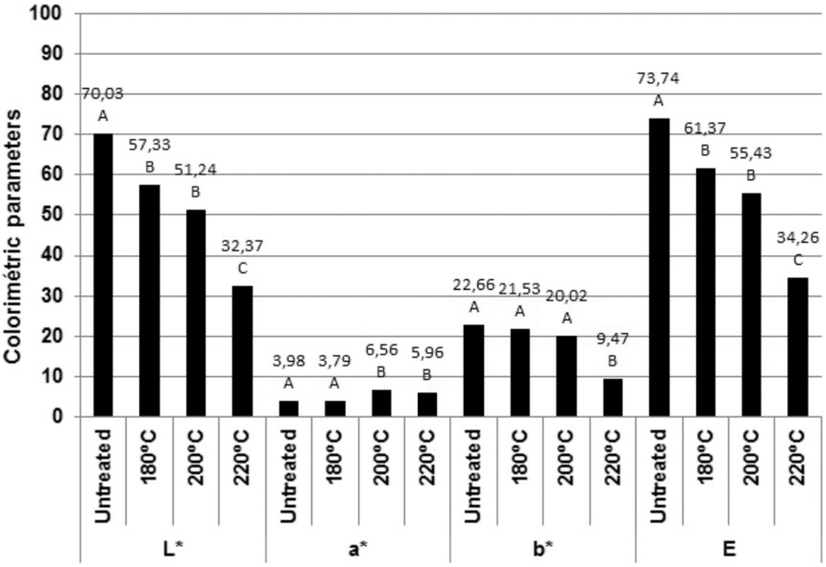

Figure 1. Effect of thermal modification on changes in color of *Liriodendron tulipifera*, where the same letter(s) do not differ from one another by Tukey's test at (α = 0.05).

because of the phenomenon known as hysteresis. The EMC of samples decreased with the temperature used for each thermal modification, with the greatest reduction occurring for wood treated at 220°C. Our results are similar to those reported for other thermally modified wood species by Bekhta and Niemz (2003), Metsä-Kortelainen et al (2006), Mburu et al (2007), and Severo et al (2012, 2016), who showed that an increase in the length of time and temperature during thermal treatment decreased the EMC of *P. abies*, *Pinus sylvestris* and *P. abies*, *Grevillea robusta*, *Pinus elliottii* var. *elliottii*, and *Hevea brasiliensis* wood.

The decrease in EMC has been explained by the degradation of the hemicelluloses and the amorphous region of cellulose, thereby contributing to the increase in the degree of crystallinity of this polymer (Esteves and Pereira 2009). We believe that cross-linkage between lignin and polymers occurs because of the thermal degradation of the wood, which is responsible for the decrease in the hygroscopicity and improvement of the dimensional stability.

Table 1. Effect of thermal modification on the biological durability of yellow poplar against *Pycnoporus sanguineous* using ASTM−017 soil block test.

	Treatment temperature			
Property	Untreated	180°C	200°C	220°C
Initial EMC (%)	10.11 a (4.88)	8.47 b (10.02)	7.30 c (10.46)	4.95 d (12.34)
Percent change from untreated		−16.22	−27.79	−51.04
Final EMC (%)	91.21 a (30.85)	87.45 a (20.79)	87.81 a (22.51)	52.13 b (20.52)
Percent change from untreated		−4.12	−3.73	−42.85
Weight loss (%)	44.68 a (52.18)	37.41 a (24.00)	34.14 a (31.20)	8.59 b (58.43)
Percent change from untreated		−16.27	−23.59	−80.77

Where positive numbers indicate an increase and negative numbers indicate a decrease; different letters indicate a significant difference by Tukey's and Dunn's tests at 5% significance between thermal treatment, to EMC and weight loss, respectively; same letters indicate no significant difference.

Decay Resistance of Thermally Modified Wood

Average weight loss for untreated wood samples exposed to the white-rot fungus was 44.68% (Table 1). Based on these results, yellow poplar wood can be classified according to ASTM D-2017 (2008) in the slightly resistant or nonresistant to decay class. Similar results were found by Schirp and Wolcott (2005), who concluded that yellow poplar wood exposed to white-rot fungus *T. versicolor* experienced a weight loss of 61.85%, as well as those reported by Shi et al (2007), who showed that this kind of wood presents weight loss of 68.5% after 12 wk in the accelerated test decay with brown-rot fungus *G. trabeum.*

There was a continual improvement in the decay resistance with increasing thermal treatment temperature, with the highest temperature showing the greatest improvement to decay resistance, 80.77% (Table 1). Our results are similar to those reported for other thermally modified hardwood species (Esteves and Pereira 2009).

The resistance to fungi comes from changes in the chemical composition of wood, making hemicelluloses unavailable to the fungi, the production of new molecules that may act as fungicides, reduction in MC, and the cross-linking between lignin and the polymer from the thermally degraded residual carbohydrates. (Esteves and Pereira 2009). Most importantly, heat treatment increased yellow poplar wood's resistance to fungi degradation. Thermally modifying wood at 220°C resulted in changing the decay resistance classification from slightly resistant (weight loss 45% or more) to highly resistant, one in which the weight loss due to decay varies between 0% and 10%. Thus, the yellow poplar wood when thermally modified at 220°C presents itself as a suitable product for use in environments with favorable conditions for the development of fungi, thus providing a potentially new market for lumber from this species.

Termite Resistance to Thermally Modified Wood

Average weight losses, average numbers of holes, and visual damage rating from untreated yellow poplar wood were 0.15, 0.38, and 1.25 (light at moderate attack), whereas the mortality rate of termites after test for resistance to *C. brevis* was 55.63% (Table 2). The weight loss in samples of wood studied may seem small because of the degradation capacity of dry-wood termites; however, although each pair of samples has a 32.2-cm^2 contact surface area, the area of termite attack was restricted to 11.4 cm^2. Similar visual damage ratings to termites were found by Gonçalves et al (2013), who studied the termite resistance of several different tropical hardwood species and concluded that they present low degradation by termites *C. brevis* with damage values between 0.53 and 1.87. Based on these results, untreated yellow poplar wood can be classified according to IPT-1157 (1980) in the class of light at moderate attack to dry-wood termites.

Thermally modified samples produced no significant improvement in the termites' resistance of yellow poplar wood (Table 2). Untreated and thermally modified wood when degraded by termites showed a weight loss not statistically different between 0.15% and 0.99%, whereas the average number of holes varied between 0.38 and 1.50. The thermal treatments did not cause

Table 2. Effect of thermal modification on the biological durability of yellow poplar against *Cryptotermes brevis* using IPT-1157 evaluation of termite resistance.

	Treatment temperature			
Properties	Untreated	180°C	200°C	220°C
Average weight loss (%) and CV	0.15 a (130.69)	0.27 a (94.39)	0.57 a (75.95)	0.99 a (98.47)
Average number of holes and CV	0.38 a (138.01)	0.63 a (169.71)	0.75 a (118.19)	1.50 a (87.29)
Average damage rating and CV	1.25 b (37.03)	3.25 a (35.85)	2.63 ab (45.25)	3.00 ab (25.20)
Average mortality rate of termites (%) and CV	55.63 a (13.75)	54.38 a (10.64)	52.50 a (12.98)	56.88 a (19.76)

Where CV, is the coefficient of variation; different letters indicate a significant difference by Tukey's and Dunn's tests at 5% significance; same letters indicate no significant difference.

changes in the visual damage rating in the test blocks of wood. The mortality rate of termites was not significantly different between untreated and thermally modified wood, which varied between 52.50% and 56.88%.

Similar results were obtained by Shi et al (2007), who tested the resistance of several different species, including yellow poplar, that were thermally modified to subterranean termites *R. flavipes*, and concluded that the treatment cannot significantly reduce the weight loss. These results are also consistent with those obtained by Oliver-Villanueva et al (2013), who concluded that there was a not change in the attack of thermally modified *Fagus sylvatica* wood by termites *Reticulitermes banyulensis*. However, our results differ from those obtained by Pessoa et al (2006) and Mburu et al (2007), who concluded that the thermal modification at 220°C for 24 h and at 250°C for 7 h increases the resistance to termites of *Eucalyptus grandis* and *G. robusta* wood, respectively, because of the possible creation of compounds toxic to termites. Thus, the thermal modification at 180-220°C in yellow poplar wood does not provide against drywood termites; however, there was a no significant increase in the visual damage rating with the increase of treatment temperature.

SUMMARY

Yellow poplar presents high brightness (L* of 70.03) and strong presence of yellow pigment (b* of 13.11). The thermal modification caused the darkening (ΔL^* of up to -37.66) and reddening (Δa^* of up to 2.58) of wood and a reduction of up to 51.4% in the EMC when exposed to 21°C and 65% RH. The decay resistance of yellow poplar against *P. sanguineus* increased with thermal treatment. The treatment at 220°C resulted in a change in ASTM D-2017 (2008) wood decay resistance class from slightly resistant to highly resistant but no significant changes in wood resistance against termites *C. brevis*.

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