Mold and Decay Resistance of Thermally Modified Douglas-Fir Heartwood

Li Yan
Jeffrey J. Morrell

Abstract

Most thermal modification studies have attempted to improve the durability of sapwood; however, it may be more fruitful to improve the durability of heartwood that already has some inherent durability. The effects of pretreatments with boron or glycerol coupled with thermal modification on resistance to mold growth and fungal degradation were examined for Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) heartwood, which is classified as moderately durable. Pretreatment with boron had marked effects on resistance to both mold and decay fungi, but the results were consistent with the well-known effects of this fungicide. Pretreatment with glycerol appeared to reduce mold resistance and had no effect on degradation by decay fungi. Thermal modification enhanced mold resistance with increasing temperature but had only a slight effect on decay resistance. These results suggest that thermal modification does not enhance performance to levels that meet North American durability requirements for aboveground, exterior exposures.

A variety of methods have been developed for improving the decay resistance of wood without the application of biocides (Kamdem et al. 2002, Chang and Chang 2006, Li et al. 2011). Among the more promising is thermal modification wherein wood is heated for varying periods of time, sometimes in the absence of oxygen, to alter the wood chemistry and reduce the risk of biological degradation. Thermal modification is not a new process, having originally been developed for modifying wood color to make certain species more marketable (Brischke et al. 2007, Esteves and Pereira 2009); however, its potential effects on the lignocellulosic matrix, notably its effects on hemicelluloses, make it potentially useful for reducing susceptibility of softwoods to biodegradation (Kamdem et al. 2002, Weiland and Guyonnet 2003). Hemicelluloses are believed to be among the first polymer components attacked in the degradation process, and modifying these components to make them less available might be an interesting solution to the vexing problem of protecting wood without the introduction of supplemental chemicals (Zabel and Morrell 1992).

The potential for thermal modification has spawned an extensive research effort, primarily centered in Europe, to enhance the properties of species such as Scots pine (*Pinus sylvestris* L.) and spruce (*Picea abies* L.; Militz 2002, Andersson et al. 2005, Borrega and Kärenlampi 2008, Korkut et al. 2008, Pfriem et al. 2010). More recently, these efforts have broadened to include a number of *Eucalyptus* species as well as other pines. While thermal modification continues to show promise, the degree of protection remains limited, and these materials do not appear to be suitable for exposure in higher decay hazard environments that would characterize most applications for conventionally treated wood in North America.

One possible approach for improving the prospects of using thermal modification would be to apply the process to a species that already has some degree of durability. Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) is found throughout the western United States and has heartwood that is moderately durable (Scheffer and Morrell 1998, Taylor et al. 2002, US Department of Agriculture 2010). Douglas-fir performs reasonably well when not in contact with soil owing to the presence of high quantities of dihydroquercetin in the heartwood (Scheffer and Cowling 1966, Hillis 1987). The wood of this species, however, is generally not recommended for outdoor exposure without supplemental treatments. Thermal modification might be useful for enhancing the moderate durability of this material, allowing it to be used under conditions more conducive to decay without supplemental treatment. Thermal modification

The authors are, respectively, Associate Professor, Northwest A & F Univ., Yangling, People's Republic of China (ylnefu@126.com); and Professor, Dept. of Wood Sci. & Engineering, Oregon State Univ., Corvallis (jeff.morrell@oregonstate.edu [corresponding author]). This paper was received for publication in August 2014. Article no. 14-00085.
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might also be enhanced by pretreatments. For example, pretreatment with glycerol may alter the modification process to improve durability (Yan et al. 2010). Similarly, boron is a well-known biocide with exceptionally low toxicity to nontarget organisms and a long history of use (Carr 1959). Its inclusion in the treatment process might enhance the protective effects of thermal modification.

The purpose of this study was to assess the potential for using thermal modification, alone or in combination with boron or glycerol pretreatment, to enhance the resistance of Douglas-fir heartwood to attack by mold and decay fungi.

Materials and Methods

Thermal modification

Douglas-fir heartwood lumber was cut into 19-mm blocks for decay tests or 12.5 by 75 by 100-mm-long wafers for mold tests. Material of a given size was thoroughly mixed before being allocated to 1 of 30 treatment groups, each containing 120 blocks and 60 mold test wafers. Ten groups received no pretreatment, 10 groups were dipped in 10 percent disodium octaborate tetrahydrate to produce a loading of 0.556 percent (wt/wt) boric acid equivalent basis (pH 8.3), and 10 groups were vacuum treated with 20 percent glycerol (20 min of vacuum at 800 Pa). Boron was evaluated for its potential to provide supplemental insect and fungal resistance to the thermally modified wood, while glycerol was evaluated for its ability to accelerate the thermal modification process.

The boron-treated blocks and wafers were stored wet for 28 days at 5°C under nondrying conditions to allow the boron to become more evenly distributed in the wood. The samples were then air-dried and finally oven-dried (65°C). Glycerol-treated blocks and wafers were weighed after treatment to determine uptake (average glycerol uptake was 147% by weight) before being allowed to air-dry.

The wood in a given pretreatment group was then wrapped in foil to limit oxygen access before being subjected to heating to 160°C, 180°C, or 200°C for 2, 4, or 6 hours. One set of each pretreatment group received no thermal treatment. After cooling, the samples were conditioned to constant weight at 23°C and 65 percent relative humidity. Each treatment was replicated on 12 blocks and six wafers. The samples were weighed after treatment to determine the effects of the various heating regimes on mass loss.

Resistance to fungal mold

Two mold boxes built according to the specifications stated in American Wood Protection Association (AWPA) Standard E24 were used for all testing (AWPA 2012b). Each box contained water in the bottom with moist soil on a mesh rack above the water. The soil was inoculated with a suspension of spores and mycelium of appropriate mold fungi and incubated for 2 weeks before the wafers were added. The fungi used in this test were *Aspergillus niger* Van Tiegh, *Alternaria alternata* (Fr.) Keissler, and *Penicillium citrinum* Thom.

The wafers were sprayed lightly with a freshly prepared suspension of the same fungi and suspended on rods across the top of the box so that the wide faces were in an upright position. Southern pine (*Pinus* sp.) sapwood wafers were included in the test to serve as mold-susceptible controls. Mold coverage was rated at 2, 4, and 6 weeks on a scale of 0 to 5 as follows:

0 No visible growth.
1 Mold covering up to 10 percent of surfaces providing growth is not so intense or colored as to obscure more than 5 percent of surfaces.
2 Mold covering between 10 and 30 percent of surfaces providing growth is not so intense or colored as to obscure more than 10 percent of surfaces.
3 Mold covering between 30 and 70 percent of surfaces providing growth is not so intense or colored as to obscure more than 30 percent of surfaces.
4 Mold covering greater that 70 percent of surfaces providing growth is not so intense or colored as to obscure more than 70 percent of surfaces.
5 Mold covering 100 percent of surfaces or with less than 100 percent coverage and intense or colored growth obscuring greater than 70 percent of surfaces.

The results were summarized and the data subjected to an analysis of variance.

Resistance to fungal decay

Decay resistance was assessed following the procedures described in AWPA Standard E10 (AWPA 2012a). The blocks were oven-dried (65°C) and weighed (nearest 0.001 g). The blocks were soaked with water for 30 minutes before being placed in plastic bags and sterilized by exposure to 2.5 mrad of ionizing radiation from a cobalt-60 source.

Decay chambers were prepared by half-filling 454-mL French square bottles with moist forest loam and placing a western hemlock (*Tsuga heterophylla* (Raf.) Sarg) feeder strip on the soil surface. The bottles were then loosely capped and autoclaved for 45 minutes at 121°C. After cooling, the bottles were inoculated with 3-mm-diameter malt agar disks cut from the actively growing edges of the test fungi cultures. The fungi evaluated in these procedures were *Gloeophyllum trabeum* (Pers. ex. Fr.) Murr. (Isolate Madison 617) or *Postia placenta* (Fr.) M. Larsen et Lombard (Isolate Mad 698). Both of these fungi produce brown rot. White rot fungi were not evaluated because they tend to have very limited activity against Douglas-fir heartwood (J.J.M., unpublished data, 2010). The agar plugs were placed on the edges of the wood feeder strips, and then the jars were loosely capped (to allow air exchange) and incubated until the feeder strip was thoroughly covered with fungal mycelium. The sterile test blocks were then placed on the surfaces of the feeder strips, and the bottles were loosely capped and incubated at 28°C for 12 weeks. Each treatment condition was evaluated on six blocks.

At the end of the incubation period, the blocks were removed and scraped clean of adhering mycelium. The blocks were then oven-dried (65°C) and weighed. The difference between the initial and the final ovendry weight was used as a measure for the decay resistance of each material.

The ASTM International Standard D2017 (ASTM International 2001) uses weight loss in a soil block test as a measure of durability and lists the criteria for various decay resistance classes as follows:

0 to 10 percent	Highly resistant
11 to 24 percent	Resistant
25 to 44 percent	Moderately resistant
>45 percent	Slightly or nonresistant

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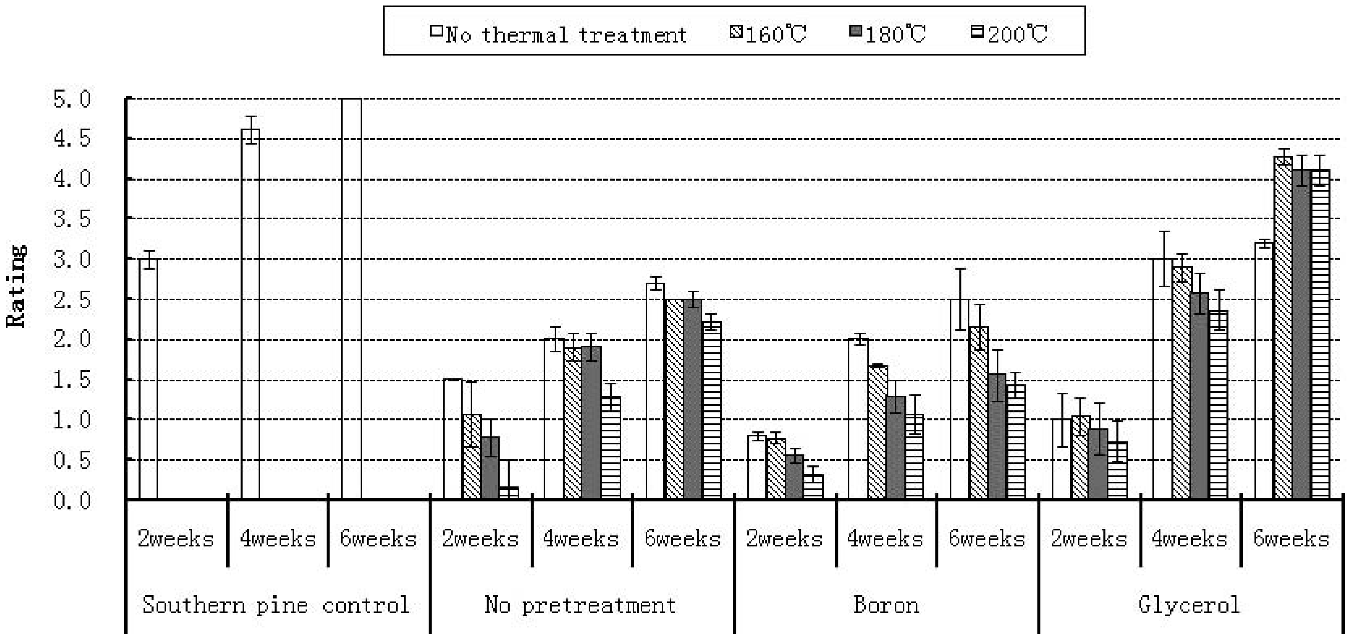

Figure 1.—Effect of combinations of pretreatment with boron or glycerol and thermal modification on resistance of Douglas-fir heartwood wafers to mold colonization in an American Wood Protection Association (AWPA) E24 mold box test (AWPA 2012b). Error bars represent one standard deviation.

The results from the current test were compared against the ASTM categories to determine if pretreatment or thermal modification affected the resistance category. The results were subjected to an analysis of variance to determine if treatment affected resistance to mold or decay fungi ($\alpha = 0.05$)

Results and Discussion

Resistance of thermally modified wood to mold attack

Mold damage increased steadily over the 6-week test period on nontreated southern pine included as a positive control, indicating that conditions were suitable for aggressive fungal growth (Fig. 1). Mold ratings were consistently lower on nontreated Douglas-fir heartwood than on southern pine but also steadily increased over the 6-week incubation period. Mold ratings were slightly lower for samples pretreated with boron but not thermally modified, while samples pretreated with glycerol but not thermally modified eventually had higher mold ratings than samples not subjected to pretreatment. Mold ratings of pretreated samples differed significantly from those of samples not subjected to pretreatment ($P \leq 0.001$). Heating temperature also had a significant effect on mold ratings ($P = 0.001$), while heating time had no significant effect on mold ratings ($P = 0.126$). For this reason, mold ratings for the samples subjected to the same pretreatment and thermal modification temperature were combined for graphical presentation.

Mold ratings on samples pretreated with glycerol were initially lower than those on the controls; however, ratings increased more rapidly on glycerol-treated samples over the next 4 weeks. There also appeared to be little effect of thermal modification temperature on mold susceptibility of glycerol-treated samples. The reason for the seemingly stimulatory effect of glycerol is unclear, although this compound is a component in some fungal media and might have a stimulatory effect on fungal growth, as evidenced by the use of glycerol derived from biodiesel production as a nutrient source for various fungi (Athalye et al. 2009). Glycerol also had a curious effect on sample moisture content. All of the samples were exposed at their ovendry condition following thermal modification. While samples without pretreatment and those pretreated with boron had similar moisture contents at the end of the 6-week exposure, samples pretreated with glycerol had much higher moisture contents that might have created more suitable conditions for fungal growth (Fig. 2).

The results indicate that thermal modification had little consistent ability to limit mold growth on Douglas-fir heartwood. Heartwood should generally be more resistant to fungal colonization than sapwood, and the higher mold ratings on southern pine controls illustrate this difference.

Effect of thermal modification on decay resistance

Weight losses of Douglas-fir heartwood controls (no pretreatment) averaged 35 percent for *P. placenta* (Fig. 3) and 30 percent for *G. trabeum* (Fig. 4). These values are a bit lower than would be found with nontreated southern pine sapwood, which would normally be greater than 40 percent for exposure to either fungus; however, these values were consistent for woods classified as moderately durable and indicated that both fungi were capable of aggressive wood degradation. The mass losses were also consistent with classification of Douglas-fir heartwood as moderately decay resistant (ASTM International 2001).

Weight losses for pretreated samples tended to differ significantly from those for samples without pretreatment ($P = 0.001$). Weight losses for samples pretreated with boron tended to be extremely low for both fungi, reflecting the well-known ability of boron to protect wood against decay

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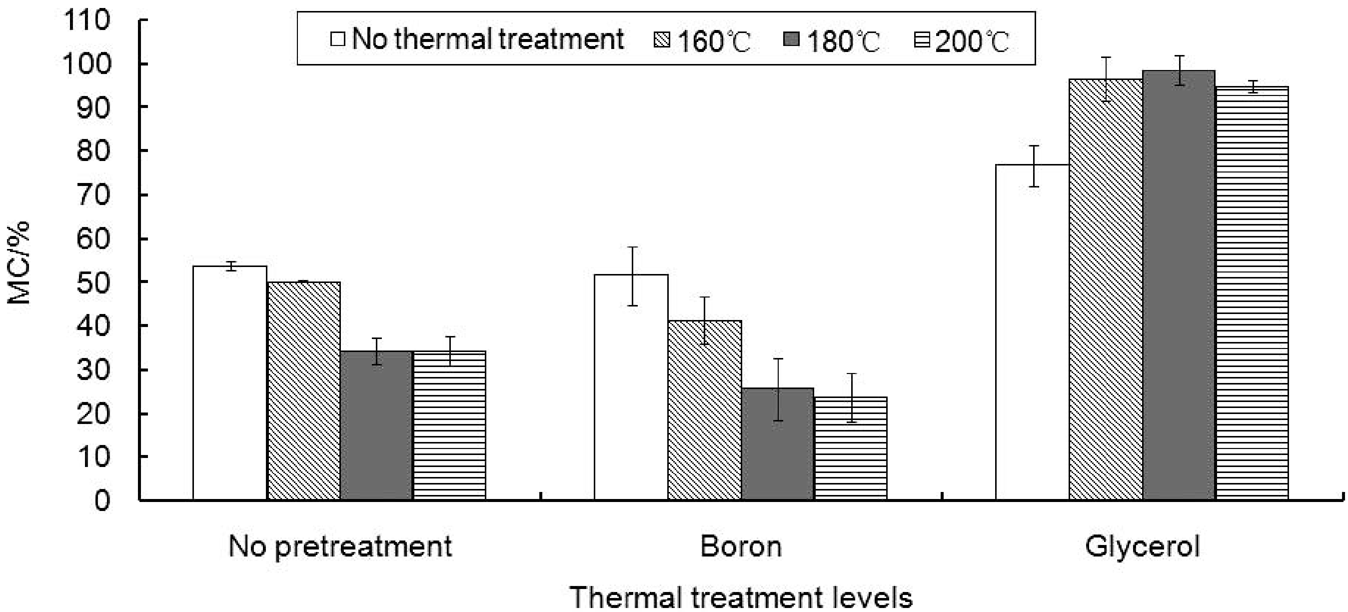

Figure 2.—Moisture contents at the end of a 6-week exposure of wafers exposed directly or pretreated with boron or glycerol and thermally modified before exposure to mold fungi in an American Wood Protection Association (AWPA) E24 mold box test (AWPA 2012b). Error bars represent one standard deviation.

fungi. Weight losses for boron-pretreated samples subjected to thermal modification tended to be similar to those found for the nonthermally modified blocks, suggesting that the process had no negative effects on boron performance. Weight losses for samples pretreated with glycerol tended to be similar to those for the samples without pretreatment, suggesting that glycerol pretreatment had no effect on decay resistance. Thermal modification for 2 or 4 hours at any of the three temperatures tested did not appear to have any effect on weight losses for samples without pretreatment and those pretreated with glycerol. Samples heated for 6 hours at either 180°C or 200°C did appear to be slightly more resistant to attack by *P. placenta,* while this effect only appeared for samples heated at 200°C when exposed to *G. trabeum*. In all cases, however, weight losses were only slightly below 25 percent, suggesting that thermal modification had only a marginal effect on durability of Douglas-fir.

The original intent of this research was to explore the potential for enhancing the durability of a species whose wood already had some natural resistance to decay. Most previous research examined the potential for improving the

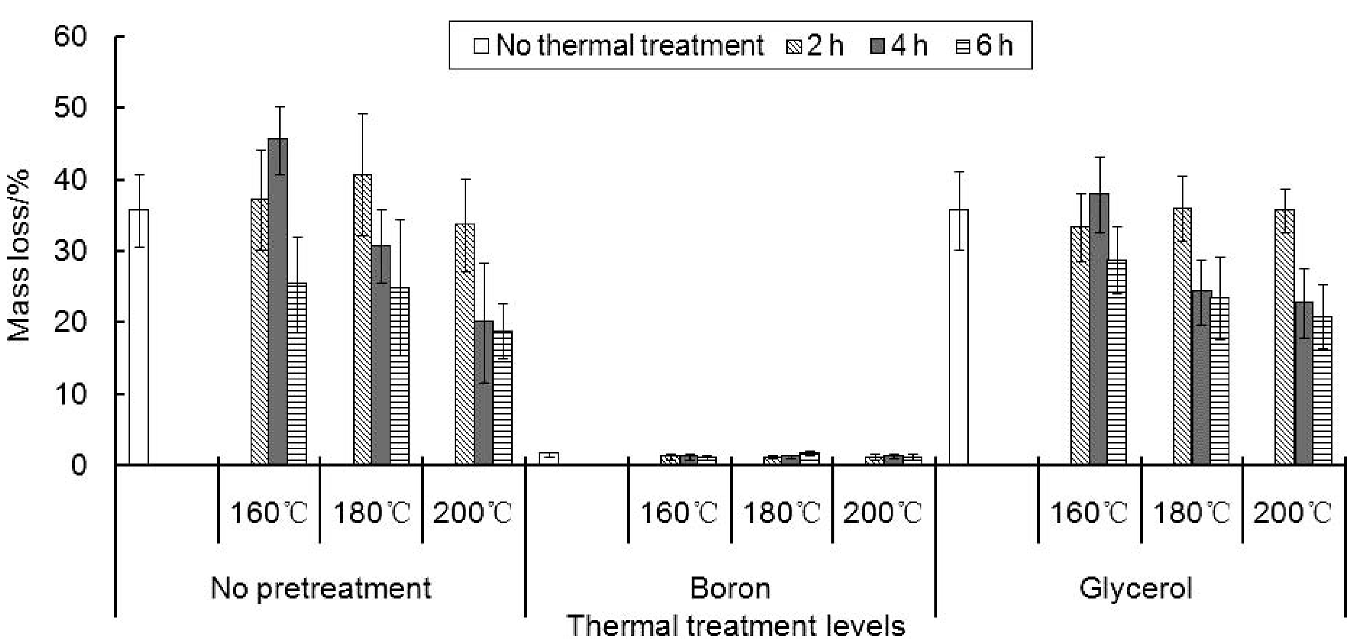

Figure 3.—Resistance of Douglas-fir heartwood blocks subjected to various combinations of pretreatment and thermal modification before exposure to Postia placenta *in an American Wood Protection Association (AWPA) E10 soil block test (AWPA 2012a). Error bars represent one standard deviation.*

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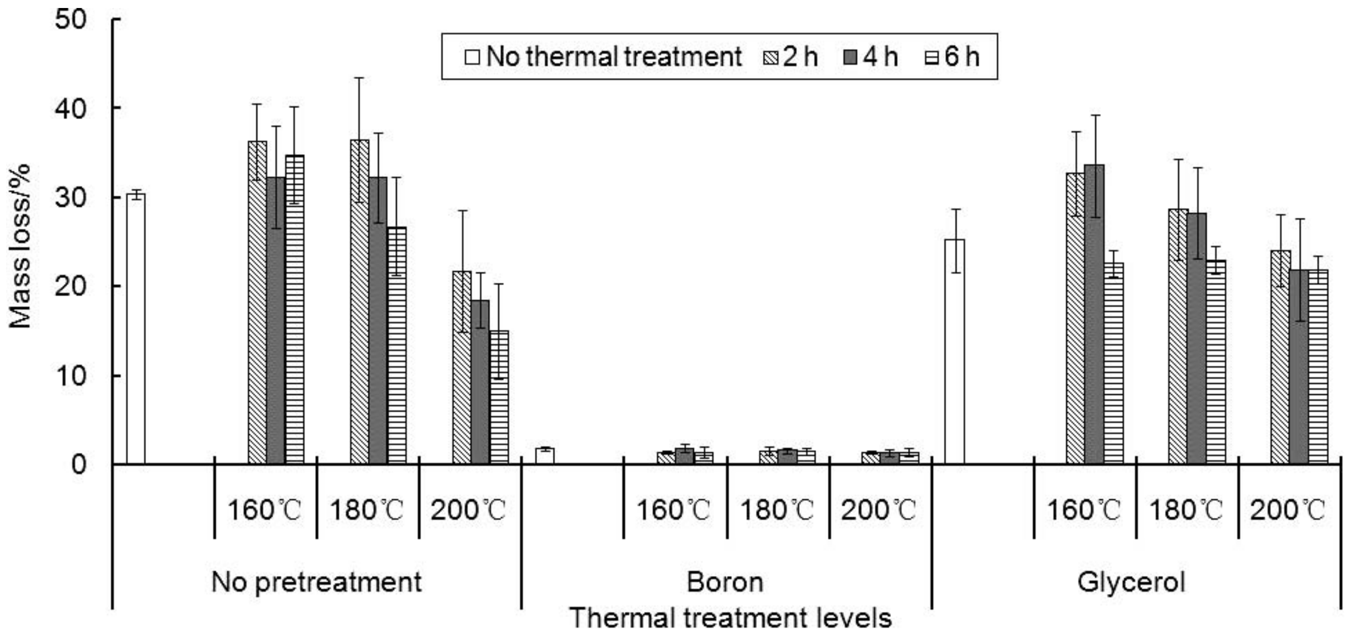

Figure 4.—Resistance of Douglas-fir heartwood blocks subjected to various combinations of pretreatment and thermal modification before exposure to Gloeophyllum trabeum *in an American Wood Protection Association (AWPA) E10 soil block test (AWPA 2012a). Error bars represent one standard deviation.*

performance of sapwood, which has little inherent resistance to degradation. While a number of European studies suggest that thermal modification improves decay resistance to the level where these products should perform well when not in contact with soil, these results have not translated into acceptance in North America. The methodologies used in North America and Europe differ, and these differences may account for the contrasting conclusions. For example, Vidrine et al. (2007) evaluated the decay resistance of thermally modified ponderosa pine (*Pinus ponderosa* L.) and yellow poplar (*Leriodendron tulipifera* L.) sapwood using soil block tests and concluded that these materials lacked the decay resistance required to meet North American standards for use in window frames. This exposure would be considered less severe than that of a wood deck. The North American soil block test tends to produce much higher mass losses than the corresponding agar block test. Thus, the differing criteria for acceptance may render this material less acceptable in North America.

While pretreatment with boron did enhance the decay resistance of Douglas-fir heartwood, these effects were consistent with those associated with the activity of boron alone and appeared to have been little affected by subsequent thermal modification. Thermal modification was associated with slight decreases in weight losses for some pretreatment–fungal combinations, but the differences were small and not likely to be biologically meaningful. Thus, thermal modification, with or without pretreatment, does not appear to improve decay resistance to the point where these materials could be used in most exterior exposures.

Conclusions

Pretreatment with boron consistently improved mold and decay resistance of Douglas-fir heartwood, while pretreatment with glycerol appeared to enhance mold susceptibility of the same material. Thermal modification affected both mold and decay resistance, but the differences were slight and not at levels that would allow these materials to be used under conditions suitable for fungal attack.

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Literature Cited

American Wood Protection Association (AWPA). 2012a. Standard method of testing wood preservatives by laboratory soil-block cultures. AWPA E10-12. *In:* AWPA Annual Book of Standards. AWPA, Birmingham, Alabama. pp. 448–458.

American Wood Protection Association (AWPA). 2012b. Standard method of evaluating the resistance of woods product surfaces to mold growth. AWPA E24-12. *In:* AWPA Annual Book of Standards. AWPA, Birmingham, Alabama. pp. 506–510.

Andersson, S., R. Serimaa, T. Väänänen, T. Paakkari, S. Jämsä, and P. Viitaniemi. 2005. X-ray scattering studies of thermally modified Scots pine (*Pinus sylvestris* L.). *Holzforschung* 59(4):422–427.

ASTM International. 2001. Standard method of accelerated laboratory test of natural decay resistance of woods. ASTM D2017. *In:* ASTM Annual Book of Standards, Volume 4.10: Wood. ASTM International, West Conshohocken, Pennsylvania. pp. 322–326.

Athalye, S. K., R. A. Garcia, and Z. Wen. 2009. Use of biodiesel-derived crude glycerol for producing eicosapentaenoic acid (EPA) by the fungus *Pythium irregulare*. *J. Agric. Food Chem*. 57:2739–2744.

Borrega, M. and P. P. Kärenlampi. 2008. Mechanical behavior of heat-treated spruce (*Picea abies*) wood at constant moisture content and ambient humidity. *Holz Roh- Werkst*. 66(1):63–69.

Brischke, C., C. R. Welzbacher, K. Brandt, and A. O. Rapp. 2007. Quality control of thermally modified timber: Interrelationship between heat treatment intensities and CIE L*a*b* color data on homogenized wood samples. *Holzforschung* 61(1):19–22.

Carr, D. R. 1959. Boron as a wood preservative. *In:* Record of the Annual Convention of the British Wood Preservers Association, Cambridge University, London. pp. 105–135.

Chang, H. T. and S. T. Chang. 2006. Modification of wood with isopropyl glycidyl ether and its effects on decay resistance and light stability. *Bioresour. Technol*. 97(11):1265–1271.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2024-12-26

Esteves, B. M. and H. M. Pereira. 2009. Wood modification by heat treatment: A review. *BioResources* 4(1):370–404.

Hillis, W. E. 1987. Heartwood and Tree Exudates. Springer-Verlag, Berlin. 268 pp.

Kamdem, D., A. Pizzi, and A. Jermannaud. 2002. Durability of heat-treated wood. *Holz Roh- Werkst*. 60(1):1–6.

Korkut, S., M. Akgül, and T. Dündar. 2008. The effects of heat treatment on some technological properties of Scots pine (*Pinus sylvestris* L.) wood. *Bioresour. Technol*. 99(6):1861–1868.

Li, Y., X. Dong, Y. Liu, J. Li, and F. Wang. 2011. Improvement of decay resistance of wood via combination treatment on wood cell wall: Swell-bonding with maleic anhydride and graft copolymerization with glycidyl methacrylate and methyl methacrylate. *Int. Biodeterior. Biodegrad*. 65(7):1087–1094.

Militz, H. 2002. Thermal treatment of wood: European processes and their background. IRG/WP 02-40241. International Research Group on Wood Preservation, Stockholm.

Pfriem, A., M. Zauer, and A. Wagenführ. 2010. Alteration of the unsteady sorption behavior of maple (*Acer pseudoplatanus* L.) and spruce (*Picea abies* (L.) Karst.) due to thermal modification. *Holzforschung* 64(2):235–241.

Scheffer, T. C. and E. B. Cowling. 1966. Natural resistance of wood to microbial deterioration. *Annu. Rev. Phytopathol*. 4:147–170.

Scheffer, T. C. and J. J. Morrell. 1998. Natural durability of wood: A worldwide checklist of species. Research Contribution 22. Forest Research Laboratory, Oregon State University, Corvallis. 58 pp.

Taylor, A. M., B. L. Gartner, and J. J. Morrell. 2002. Heartwood formation and natural durability: A review. *Wood Fiber Sci*. 34(4):587–611.

US Department of Agriculture. 2010. Wood Handbook: Wood as an Engineering Material. FPL-GTR-190. USDA Forest Service, Forest Products Laboratory, Madison, Wisconsin. 508 pp.

Vidrine, C., C. Freitag, J. Nicholson, and J. J. Morrell. 2007. Effects of heat treatments on decay resistance and material properties of ponderosa pine and yellow poplar. IRG/WP/07-40374. International Research Group on Wood Protection, Stockholm.

Weiland, J. J. and R. Guyonnet. 2003. Study of chemical modifications and fungi degradation of thermally modified wood using DRIT spectroscopy. *Holz Roh- Werkst*. 61(3):216–220.

Yan, L., J. Z. Cao, X. Y. Zhou, W. Gao, and G. J. Zhao. 2010. Deformation, fixation, mechanical properties and chemical analysis of compressed *Populus cathayana* pretreated by glycerin. *Forestry Stud. China* 12(4): 213–217.

Zabel, R. A. and J. J. Morrell. 1992. Wood Microbiology. Academic Press, San Diego, California. 474 pp.

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