TECHNICAL NOTE

Heat Modification of Wood: Chemical Properties and Resistance to Mold and Decay Fungi

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Abstract

The resistance of heat-modified sugi (*Cryptomeria japonica* D.) sapwood against mold and decay fungi was evaluated under laboratory conditions. Wood specimens were subjected to heat treatment at 180°C for 2 and 4 hours and at 220°C for 2 hours. Changes in lignin and polysaccharide weight percent composition, solubility, and pH of heat-modified specimens were determined. Treatment at 220°C for 2 hours decreased wood polysaccharide content. Heat treatment slightly increased resistance against *Rhizopus javanicus* and *Gliocladium* (*Trichoderma*) *virens,* but not against *Aspergillus niger*. The highest temperature used increased resistance to decay by the white-rot fungus *Trametes versicolor,* but mass losses with the brown-rot fungus *Fomitopsis palustris* were higher than those with *T. versicolor*.

Wood, a natural biological material, is one of the most important renewable natural polymers, and it is used for a number of purposes in both indoor and outdoor applications. However, wood can be biologically degraded by a variety of organisms, from fungi to insects. Heat modification has long been known to increase the dimensional stability by reducing the hydroscopicity, and it also enhances the biological resistance of wood to decay fungi. The dimensional stability results from the chemical modification of wood cell components (Kamdem et al. 2002), and this likely also increases the resistance of wood to fungal attack (Tjeerdsma et al. 1998, 2002; Kamdem et al. 1999, 2002; Weiland and Guyonnet 2003; Boonstra et al. 2007).

Studies vary on the decay resistance of heat-modified wood in ground contact. Welzbacher and Rapp (2005) have questioned whether the decay resistance of heat-modified wood in ground contact meets the requirements of European Hazard Class 4 EN 350-1 (European Committee for Standardization [CEN] 1994a). Some researchers have shown that heat-modified wood is not suitable for ground-contact applications (Jämsä and Viitaniemi 1998, Kamdem et al. 1999, Wienhaus 1999, Rapp et al. 2000, Doi et al. 2004). In contrast, Boonstra and Doelman (1999) classified heat-modified wood as ''very durable'' to ''durable'' based on EN 350-2 standards (CEN 1994b). Welzbacher and Rapp (2005) reported that heat treatment increased the durability of several types of Scots pine (*Pinus sylvestris* L.) sapwood specimens in ground contact.

Heat treatment at high temperatures and for long durations may increase the resistance of wood to specific types of fungal decay organisms as a result of chemical changes in wood structure. However, the resistance of heat-modified wood to decay may vary according to process temperature and time, wood species, type of fungal decay organisms to which the wood is exposed, and conditions under which the wood is used (in contact with the ground or aboveground). In laboratory decay tests, it was shown that heat treatment of wood at 180°C for 4 hours did not have any effect on resistance against the brown-rot fungus (*Fomitopsis palustris*) but slightly improved resistance against the white-rot fungus (*Trametes versicolor*; Kartal 2006).

In this study, we evaluated the resistance of heat-modified sugi (*Cryptomeria japonica* D.) sapwood against fungal degradation and mold growth. Mold and decay resistance tests were performed under laboratory conditions. Changes in lignin and hemicellulose content during heat treatment were determined by chemical analysis.

Materials and Methods

Two types of wood specimens were used: (1) 3 by 28 by 34 mm (tangential [T] by radial [R] by longitudinal [L]) for mold resistance tests, and (2) 5 by 10 by 50 mm (T by R by L) for decay resistance tests. The specimens were free of

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knots and visible concentration of resins and showed no visible evidence of infection by mold, stain, or wood-destroying fungi. All samples were prepared from sapwood portions of sugi.

Heat modification of wood specimens was performed at either 180°C (2 or 4 h) or 220°C (2 h) using a temperature-controlled laboratory oven. The modification process was carried out under normal atmosphere conditions without any nitrogen gas or steam.

For mold resistance tests, the specimens were soaked in distilled water under vacuum to obtain a high moisture content. Three mold fungi, *Aspergillus niger* ASN 5131, *Rhizopus javanicus* RHN 32, and *Gliocladium* (*Trichoderma*) *virens* GLV 9645, were inoculated on 2 percent malt extract agar (20 g of malt extract and 20 g of Bacto agar in 1 liter of distilled water) in petri dishes at 23°C for 3 weeks before inoculation of the specimens. All fungal cultures were obtained from the Research Institute for Sustainable Humanosphere (RISH), Kyoto University, Kyoto, Japan. Each petri dish was flooded with 20 mL of sterile distilled water and the surface of fungal growth in the petri dish was rubbed with a transfer pipette in order to loosen fungal spores and hyphal fragments. Two specimens were placed on glass rods in a glass petri dish (100 mm in diameter, 20 mm in height). To maintain high humidity in the petri dishes during the test period, five layers of filter paper were placed on the bottom of each dish. The papers were wetted with distilled water until free water appeared. The petri dishes including the specimens were then autoclaved at 121°C and 15 psi for 20 minutes, and 5 mL of inoculum was removed with the transfer pipette and applied to the radial surface and cross sections of the specimens. The inoculated petri dishes were placed in polyethylene bags to prevent drying and incubated at 25°C and 70 to 80 percent relative humidity. At the end of the test periods (1, 2, 4, and 6 wk), each specimen was visually rated for degree of discoloration. The incidence of fungal coverage on wood specimens was determined using a 6-point scale that corresponded to the percentage of mold cover (Pabuccu 2007).

The decay resistance of sugi wood was evaluated by inserting wood specimens directly into petri dishes inoculated with basidiomycetes fungi. Before decay testing, all wood specimens were conditioned at 20°C and 65 percent relative humidity for 2 weeks. Two basidiomycetes, brown-rot fungus [*Fomitopsis* (*Tyromyces*) *palustris* (Berk. et Curt) Gilbn. & Ryv.; FFPRI 0507] and white-rot fungus [*Trametes* (*Coriolus*) *versicolor* (L.:Fr.) Pilat.; FFPRI 1030], were inoculated separately on 2 percent malt extract agar in petri dishes for 3 weeks at 23°C before placement of the specimens into the dishes. All cultures were obtained from RISH. The wood specimens were autoclaved at 121°C and 15 psi for 20 minutes for sterilization, and then the specimens were placed into the inoculated petri dishes. Four specimens were placed in each dish and unheated specimens served as controls. After a 10-week incubation period, the specimens were taken out and reweighed to calculate mass loss.

For chemical analyses, wood specimens from each treatment group were ground and screened from 40 to 100 mesh (0.420- to 0.149-mm standard sieve opening). Klason lignin content in the wood specimens was determined using 72 percent sulfuric acid and 40 percent hydrobromic acid (Runkel and Wilke 1951). Klason lignin values were corrected for ash content gravimetrically following incubation of lignin at 575°C for less than 3 hours. Carbohydrate content of the hydrolysates was determined by anion exchange high performance liquid chromatography using pulsed amperometric detection (Davis 1998). Wood sugars were quantified using an internal standard method, and the results were reported in terms of percentage of original sample mass.

Cyclohexane and ethanol, 1 percent NaOH, and hot water solubility were determined based on TAPPI test methods (Technical Association of the Pulp and Paper Industry [TAPPI] 1988a, 1988b, 1992) by grinding wood specimens in accordance with TAPPI test methods (TAPPI 1984).

For pH measurements, sawdust samples were prepared by grinding wood specimens to pass through a 40-mesh screen. One gram of the prepared sawdust was then soaked in 30 mL of distilled water at 26°C for 24 hours. The test revealed that the 24-hour soaking process was sufficient to stabilize pH of the water/wood sawdust.

Results and Discussion

Table 1 shows the Klason lignin content and polysaccharide composition of the wood specimens. Klason lignin content of untreated specimens was 35.7 percent. Klason lignin content of specimens treated at 180°C for 2 hours was slightly decreased, whereas that of specimens treated at 220°C for 2 hours was increased 5.5 percent. The apparent increase in the amount of Klason lignin was probably due to the removal of hemicelluloses during thermal degradation. The increase in lignin content does not imply the formation of lignin during the process but rather the reduction of other wood components (Kamdem et al. 2002). Lignin content increases above 140°C to 150°C even though it is the most stable component of wood. It is also known that lignin content increases above 170°C based on the assumption that polycondensates form and are added to the original lignin complex (Beall and Eickner 1970). Haw and Schultz (1985) also suggested that during heat treatment, especially in an oxidative atmosphere as in our study, carbohydrates can form furfurals when condensed to form a polymer which, under Klason analyses, shows up as a pseudo-lignin. Pseudo-lignin might be the result of a repolymerization of polysaccharide degradation products during thermal treatments (Jakobsons et al. 1995). In addition to the effect of temperature, thermal degradation of lignin varies by wood species and is affected by exposure time and heating medium (Fengel and Wegener 1984).

Treatment at 220°C for 2 hours caused remarkable decreases in polysaccharide composition compared with the slight changes observed after treatment at 180°C for 2 and 4 hours (Tables 1 and 2). Treatment at 220°C for 2 hours caused severe degradation in hemicellulose content of wood, represented by arabinose, galactose, xylose, and mannose, by nearly 41 percent compared with untreated wood. High polymerization degree and crystalline structure of cellulose make its decomposition even more difficult than hemicelluloses. Treatment at 220°C for 2 hours caused remarkable degradation, represented by the loss of glucose content by 6.52 percent. Because polyoses are branched and substituted monomeric units with an amorphous structure, they are much more susceptible to heat degradation than are other polymeric cell wall components (Windeisen et al. 2007). It was also found that treatment at 66°C for 560 days decreased arabinose, galactose, xylose, and mannose content of southern pine by 50 percent or more compared

Table 1.—Chemical composition of heat-modified and untreated sugi sapwood specimens.

Specimen	Klason lignin (%)	Arabinose (%)	Galactose (%)	Glucose (%)	Xylose (%)	Mannose (%)
Untreated	35.66	0.87	1.13	42.46	4.64	7.24
180°C/2 h	35.44	0.79	1.17	42.26	4.65	7.53
180°C/4 h	36.31	0.80	1.11	42.10	4.51	7.07
220°C/2 h	37.64	0.23	0.54	39.69	2.55	4.93

Table 2.—Percent changes in chemical composition of heat-modified sugi sapwood based on untreated control specimens.[a]

Specimen	Klason lignin	Arabinose	Galactose	Glucose	Xylose	Mannose
180°C/2 h	−0.62	−9.03	+2.91	−0.48	+0.12	+3.94
180°C/4 h	+1.82	−8.55	−2.50	−0.84	−2.88	−2.39
220°C/2 h	+5.54	−73.51	−52.19	−6.52	−44.99	−31.95

[a] − = negative values showing percent decreases; + = positive values showing percent increases based on the values of untreated wood.

with that of untreated wood (Sweet and Winandy 1999). The degradation of hemicelluloses and alpha-cellulose most closely follows that of wood; the lower degradation temperature of wood compared with that of lignin is due to hemicelluloses in the wood (LeVan and Winandy 1990). Our results revealed that chemical changes during heat treatments are highly temperature dependent. It is well known that cellulose decomposition can be accelerated in the presence of water, acids, and oxygen (LeVan and Winandy 1990). As the temperature increases, additional dehydration and oxidation reactions occur. Heating in air causes oxidation of the hydroxyl groups resulting in an increase of carbonyl and subsequently of carboxyl groups (Fengel and Wegener 1984). It is clearly seen in Table 2 that cellulose, which was represented by glucose, decreased 6.52 percent in the treatment at 220°C for 2 hours compared with untreated wood. However, glucose shows only 0.84 percent decrease in the treatment at 180°C for 4 hours.

Tables 3 and 4 show the effect of heat modification on the solubility of wood components. Process temperature had a greater effect on solubility than did process time. Cyclohexane/ethanol (CHE)–extractable content of wood is a measure of waxes, fats, salts, resins, and nonvolatile hydrocarbons. Hot water extraction removes inorganic compounds, gums, sugars, and starches. Low-molecular-weight carbohydrates are extracted by 1 percent NaOH solution, and hot water solubility indicates the extent of degradation by heat, light, oxidation, and fungal decay. As the wood degrades, the percentage of soluble pyrolytic products increase. Increases in 1 percent NaOH solubility of heat-modified wood at 220°C for 2 hours were likely caused by degradation of lower-molecular-weight carbohydrates.

Table 3 also shows the effect of heat modification on wood pH. The increased acidity of heat-modified wood may be assumed to be caused by the release of organic acids (Fengel and Wegener 1984, Tjeerdsma et al. 1998, Garrote et al. 1999, Yilgor et al. 2001). Beall and Eickner (1970) stated that below 200°C, formic and acetic acids are produced and dehydration of sorbed water is complete.

Table 5 shows average ratings for mold growth on the specimens. The mold fungi were found on the surfaces of both heat-modified and untreated specimens. Moderate mold growth was found on specimens subjected to the fungi *R. javanicus* and *G. virens* compared with *A. niger*. Although the growth of *A. niger* on heat-modified specimens was slightly slower than on untreated specimens during the first 4-week exposure, both treated and untreated specimens were covered by this fungus by the end of the test. Heat-modified specimens showed slightly better resistance against the other mold fungi evaluated.

Table 3.—Solubility and pH values of heat-modified and untreated sugi sapwood specimens.[a]

	Solubility (%)				
Specimen	CHE	Ethanol	1% NaOH	Hot water	Wood pH
Untreated	1.36	0.49	7.23	2.78	3.14
180°C/2 h	1.52	0.80	10.20	2.89	2.56
180°C/4 h	1.81	0.97	11.70	3.08	2.53
220°C/2 h	2.28	1.28	17.11	3.70	2.46

[a] Each value is the average of duplicate specimens.

Table 4.—Percent changes in solubility of heat-modified sugi sapwood based on untreated control specimens.

	Solubility			
Specimen	CHE	Ethanol	1% NaOH	Hot water
180°C/2 h	+11.76	+63.26	+41.00	+3.96
180°C/4 h	+33.09	+97.96	+61.83	+10.79
220°C/2 h	+67.65	+161.22	+136.65	+33.09

Table 5.—Ratings for mold coverage (%) of untreated and heat-modified sugi sapwood specimens.

Specimen	Week[a] 1	2	4	6
A. niger				
Untreated	4.3 (0.4) A	5.0 (0.4) A	5.0 (0.3) A	5.0 (0.0) A
180°C/2 h	2.3 (0.2) B	3.3 (0.3) B	4.3 (0.3) B	5.0 (0.0) A
180°C/4 h	2.0 (0.2) B	2.7 (0.3) B	5.0 (0.4) A	5.0 (0.0) A
220°C/2 h	1.7 (0.1) B	3.0 (0.0) B	4.3 (0.2) B	5.0 (0.0) A
R. javanicus				
Untreated	1.0 (0.0) A	2.3 (0.2) B	3.7 (0.2) A	4.7 (0.3) A
180°C/2 h	1.0 (0.0) A	3.0 (0.1) A	3.7 (0.2) A	4.7 (0.4) A
180°C/4 h	1.0 (0.0) A	2.3 (0.3) B	2.7 (0.4) B	2.8 (0.2) B
220°C/2 h	1.0 (0.0) A	1.3 (0.3) C	2.0 (0.0) BC	3.0 (0.4) B
G. virens				
Untreated	2.7 (0.3) A	3.3 (0.5) A	4.0 (0.4) A	4.3 (0.4) A
180°C/2 h	2.0 (0.1) AB	2.0 (0.2) B	2.5 (0.3) B	3.0 (0.2) B
180°C/4 h	1.0 (0.0) B	1.3 (0.1) C	1.3 (0.2) C	3.0 (0.2) B
220°C/2 h	1.0 (0.0) B	1.0 (0.1) C	2.0 (0.1) B	3.0 (0.2) B

[a] Each value represents an average of 12 specimens. Values in parentheses are standard deviations. Within a column, values followed by the same letters are not statistically different according to the Duncan's multiple range test ($P \leq 0.05$). Ratings for mold coverage: 0 = 0 percent, 1 = 0 to 5 percent, 2 = 6 to 25 percent, 3 = 26 to 50 percent, 4 = 51 to 75 percent, 5 = 76 to 100 percent.

Mass losses in malt-agar plate tests with basidiomycetes fungi are given in Table 6. In tests with brown- and white-rot fungi, mass losses decreased with an increase in test temperature and duration. The white-rot fungus *T. versicolor* caused considerably less mass loss than did the brown-rot fungus *F. palustris*; for specimens subjected to 220°C for 2 hours, loss in mass was only 4 percent. Doi et al. (2004) found that PLATO treatment did not increase the resistance of wood against *F. palustris* and *T. versicolor* under laboratory conditions. They observed similar trends in termite resistance tests using *Reticulitermes speratus*. Kamdem et al. (2002) showed that heat treatment did not sufficiently increase the decay resistance of wood for use in ground contact. Tjeerdsma et al. (2002) showed that two-stage heat treatments wood and dry-heat treatments improved the resistance of wood against *C. puteana* and *T. versicolor*.

In tests on various species of wood, Welzbacher and Rapp (2005) found that heat treatment significantly increased the durability of Scots pine sapwood in ground contact for up to 4 years. In a previous study, Kartal (2006) showed that heat treatment at 180°C for 2 and 4 hours was apparently ineffective against decay by *F. palustris*. However, heat-modified specimens exposed to *T. versicolor* had much lower mass loss than untreated specimens. That study also showed that heat treatment increased the susceptibility of specimens to termites.

Table 6.—Mass loss of untreated and heat-modified sugi sapwood specimens after exposure to basidiomycetes.

Specimen	Mass loss (%)[a] *F. palustris*	*T. versicolor*
No heat	54.3 (1.2) A	41.4 (2.2) A
180°C/2 h	50.2 (2.1) A	20.1 (1.4) B
180°C/4 h	42.8 (1.3) AB	15.6 (0.9) BC
220°C/2 h	35.1 (2.0) B	4.1 (0.09) C

[a] Each value is the average of 12 specimens. Values in parentheses are standard deviations. Within a column, values followed by the same letters are not statistically different according to the Duncan's multiple range test ($P \leq 0.05$).

Conclusions

Heat modification of sugi sapwood improved its resistance to decay by *T. versicolor* due to changes in chemical structure. Heat treatment did not increase resistance of wood specimens to *A. niger*; however, slight improvements were seen when *R. javanicus* and *G. virens* were used in the tests. Degradation products of hemicelluloses such as sugar monomers may have affected the resistance of heat-modified wood against mold fungi.

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