J. Korean Wood Sci. Technol. 2017, 45(2): 195~201 pISSN: 1017-0715 eISSN: 2233-7180
https://doi.org/10.5658/WOOD.2017.45.2.195

Original Article

Evaluation of Two Species of Soft Wood Decay Resistance for Heat-Treated Wood Using the Catalyst (H_2SO_4)[1]

Kyung-Rok Won[2] · Nam-Euy Hong[3] · Su-Young Jung[4] · Byung-Ro Kim[5] · Hee-Seop Byeon[2,†]

ABSTRACT

This study was conducted to evaluate the resistance of heat-treated wood using the catalyst to decay caused by fungi for sapwood and heartwood of two tree species, Korean red pine (*Pinus densiflora*) and Japanese larch (*Larix kaempferi*), respectively. Wood samples were immersed for 10 min in sulfuric acid (7.5%) and then heat-treated at 130℃ for 90 min. *Fomitopsis palustris*, a brown-rot fungus, was used to examine the decay resistance of Korean red pine and Japanese larch wood. Weight and density of wood from the all conditions increased after heat treatment using the catalyst. Weight loss after decay resistance test was also dropped with a heat treatment. The lowest weight loss indicated at heat-treated heartwood of Japanese larch. Heat treatment using the catalyst effectively increased the resistance of wood to decay caused by fungi.

Keywords : heat-treated wood using catalyst, sulfuric acid, decay resistance

1. INTRODUCTION

Wood is a natural, renewable, and sustainable material widely used for building and construction because of its specific strength and aesthetic appeal (Wang *et al.*, 2014). These wooden materials can be used in many ways, such as to build log homes, interior framing, and wood buildings. However, wood is a complex of cellulose, hemicellulose, and lignin and is susceptible to degradation due to microbial agents, which may cause significant losses of weight and strength. Protecting wood from such bio-deterioration has long been a challenge for wood researcher (Jain *et al.*, 2011). The principal problems of bio-deterioration are related to fungi (Nagaveni *et al.*, 2011). In most cases, wood must be properly protected to avoid weathering-induced deterioration during its outdoor use. Some wood preservatives are well known to protect the wood from wood-degrading organisms, but cause environmental pollu-

1 Date Received January 13, 2017, Date Accepted March 1, 2017
2 College of Agriculture & Life Science, IALS, Gyeongsang National University, Jinju 52828, Republic of Korea
3 Department of Development, Samyoung Industrial, Gimhae 51-62, Korea
4 Southern Forest Resources Research Center, National Institute of Forest Science, Jinju 52817, Republic of Korea
5 Department of Wood and Paper Science, College of Agriculture, Life and Environment Science, Chungbuk National University, Cheongju 28644, Republic of Korea
† Corresponding author: Hee-Seop Byeon (e-mail: hsbyeon@gnu.ac.kr)

tion as they contain harmful substance.

Thermal treatment is an alternative to chemical treatment for wood preservation, which has sometimes been used to improve timber quality (Awoyemi and Jones, 2011; Poncsak *et al*., 2006). This heat treatment of wood is an environmentally friendly wood preservation technique. Wood is heated to temperatures above 200℃ depending on the wood species and the required wood properties (Kocaefe *et al*., 2010). Concurrently, heat treatment of temperatures above 200℃ adversely affects the mechanical properties of wood and weight, and density (Yildiz *et al*., 2006). In order to prevent both bio-deterioration and adverse effects of heat treatment, a new heat treatment method using a catalyst, sulfuric acid was developed. With the existing methods of heat treatment, wood is degraded, dimensional stability is enhanced, equilibrium moisture content is lowered, and color darkens and biological durability is increased (Sini *et al*., 2006). Changes in the chemical and physical properties of wood are caused by the high temperatures of heat treatment. Heat treatment using a catalyst results in small changes in the physical and mechanical properties of wood, as processing occurs at the relatively low temperature of 130 ℃. The method also consumes less energy than non-catalyst-based methods. Furthermore, heat treatment using a catalyst enhances decay resistance at the wood surface because of charcoal coatings that result from the treatment.

In this study, the effects of heat-treated wood using the catalyst to decay caused by fungi, wood from four conditions, sapwood and heartwood of Korean red pine and Japanese larch wood were investigated, respectively.

2. MATREIALS and METHODS

2.1. Materials

The sapwood and heartwood of Korean red pine (*Pinus densiflora* Sieb. et Zucc.) and Japanese larch (*Larix kaempferi* (Lamb.) Carr.) dried at room temperature were used. Samples of 20 × 20 × 20 mm for the decay resistance test were cut from each of the dried wood for the two species. Ten samples from each of the two species were used for each condition. Samples were maintained for six weeks at a constant temperature and humidity (20℃ ± 1℃, 65% RH ± 2%).

2.2. Heat treatment using the catalyst

Sulfuric acid was used as the catalyst for the heat treatment at concentration of 7.5%. The wood samples were immersed in the sulfuric acid concentration (7.5%) for 10 min. The heat treatment of the samples was performed at the maximum temperature of 130℃ maintained for 90 min in a dry oven (Muffle furnace, JeioTech OF-300, Korea). After heat treatment, the wood samples were washed in distilled water to remove the residual sulfuric acid. Samples were again maintained for 3 weeks at a constant temperature and humidity (20 ± 1℃, RH 65 ± 2%). Changes in weight, density, and decay resistance of the samples were measured.

2.3. Weight and air-dried density

The weight of before and after heat treatment using the catalyst for each specimen was measured by electronic scale. And measurement of density in heat-treated wood was carried out according to KS F 2198 (air-dried density) standard in constant temperature and humidity room (temperature 20 ± 1℃, relative humidity 65 ± 2%).

2.4. Decay resistance test

The brown-rot fungus, *Fomitopsis palustris* (Berkeley et Curtis), was used to examine the decay resistance of sapwood and heartwood Korean red pine and Japanese larch. The potato dextrose agar medium (Becton, Dickinson and Company sparks, MD 21152) was sterilized at 121℃ for 30 min and poured 50 mℓ into plant culture dish (100 × 40 mm). Then the fungi for the decay resistance test were inoculated on to potato dextrose agar medium. The specimens were decayed for 60 days in constant temperature (26 ± 2℃) after decay operation.

Observation for decay resistance was conducted, and photographs were taken, every week after inoculation. After the decay resistance test, weight loss were measured for the each condition.

Weight loss ratio (P_{wl}) was calculated according to the equation (1):

$$P_{wl} = \frac{W_1 - W_2}{W_1} \times 100(\%) \quad \cdots\cdots (1)$$

where W_1 is weight before decay resistance test, W_2 is weight after decay resistance test.

2.5. Observation of heat-treated wood using the catalyst with scanning electron microscopy (SEM)

Samples used for scanning electron microscopy (SEM) were cut into 2 mm (T) × 10 mm (R) × 1 mm (L) pieces after heat-treatment with sulfuric acid and the decay resistance test. To prepare for SEM, samples were freeze-dried and gold-coated prior to examination with a Hitachi S-2400 SEM. SEM images were obtained at accelerating voltage 5 kV at 500 magnification.

3. RESULTS and DISCUSSION

3.1. Weight changes of heat-treated wood using the catalyst

Table 1 shows the mean values for weight and air-dried density of sapwood and heartwood from two species before and after heat treatment using the catalyst (H_2SO_4).

The weight changes were indicated in heat-treated wood using the catalyst. The change percentages of weight of Korean red pine wood in heat conditions at heat-treated sapwood and heat-treated heart wood decreased -4.98 and -4.52%, respectively. And change percentages of weight of Japanese larch wood at the same condition also decreased -1.26% and -3.17%, respectively. Won *et al*., (2015) reported that decreased weight of Korean red pine wood at heating conditions of 180℃ for 12 h

Table 1. Weight and density changes of heat-treated wood using the catalyst

Species	Treatment conditions		Weight (g)			Density (g/cm³)		
			Before	After	P_w (%)[2)]	Before	After	P_D (%)[3)]
Pinus densiflora	Sap wood	Control	4.31 (0.62)	4.31 (0.62)	0 a [1)]	0.54 (0.07)	0.54 (0.07)	0 a
		Heat-treated wood	4.42 (0.68)	4.20 (0.55)	-4.98 b	0.55 (0.08)	0.52 (0.07)	-5.45 b
	Heart wood	Control	4.45 (0.52)	4.45 (0.52)	0 a	0.56 (0.07)	0.56 (0.07)	0 a
		Heat-treated wood	4.42 (0.40)	4.22 (0.42)	-4.52 b	0.56 (0.05)	0.53 (0.05)	-5.36 b
Larix kaempferi	Sap wood	Control	3.93 (0.38)	3.93 (0.38)	0 a	0.49 (0.05)	0.49 (0.05)	0 a
		Heat-treated wood	3.96 (0.36)	3.91 (0.36)	-1.26 b	0.49 (0.05)	0.48 (0.05)	-2.04 b
	Heart wood	Control	4.39 (0.51)	4.39 (0.51)	0 a	0.55 (0.07)	0.55 (0.07)	0 a
		Heat-treated wood	4.42 (0.51)	4.28 (0.62)	-3.17 b	0.55 (0.06)	0.53 (0.08)	-3.64 b

[1)] Means with the same letter are not significantly different at a P-value of 0.05 according to Duncan's new multiple range test.
[2)] P_W is percentage of weight change after heat treatment,
[3)] P_D is percentage of density change after heat treatment,
Parenthesis is standard deviation. Control data are the same as the previous paper (Won *et al.*, 2016).

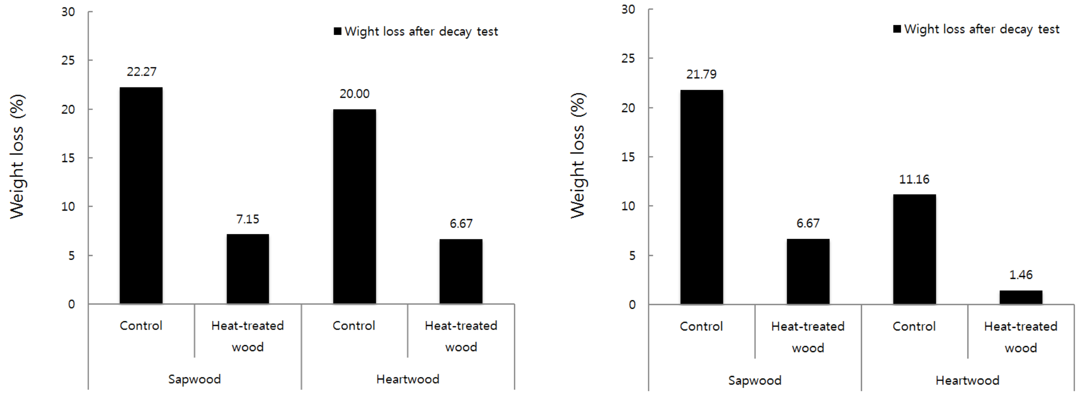
and 24 h, and of 210℃ for 3h and 6h, of 9.18, 11.90, 11.11, and 17.46%, respectively. The weight of Japanese larch wood at the same condition also decreased by 9.99, 11.44, 11.41, and 20.39%, respectively. However, heat treatment with a catalyst caused small changes in the physical and mechanical properties owing to processing taking place at the relatively low temperature of 130℃.

3.2. Density changes of heat-treated wood using the catalyst

The density changes were indicated in heat-treated wood using the catalyst. The change percentages of density of Korean red pine wood in heat conditions at heat-treated sapwood and heat-treated heart wood decreased -5.45 and -5.36%, respectively. And change percentages of density of Japanese larch wood at the same condition also decreased -2.04% and -3.64%, respectively. For both species, weight and density after heat treatment were decreased. The decreasing percentages of weight and density were higher in Korean red pine wood using the catalyst than in Japanese larch wood using the catalyst.

3.3. Decay resistance

Table 2 shows the characteristics of decay resistance for sapwood and heartwood from two species after heat treatment using the catalyst. Coverage by fungal hyphae was very slow in



Pinus densifora *Larix kaempferi*

Fig. 1 Weight loss of specimen of control, heat-treated wood, and wood heat-treated using the catalyst after decay resistance test.

heat-treated wood using the catalyst until 10 days. These results of naked-eye observation indicated that decay resistance was higher in heat-treated wood than in the control. Similar trends were observed in both Korean red pine and Japanese larch wood.

3.4. Weight loss after decay resistance test

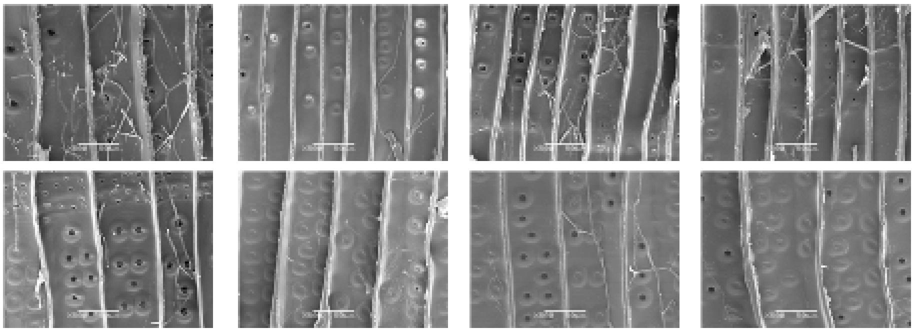
Fig. 1. The average values of weight loss for sapwood and heartwood of two species after the decay resistance test (20 × 20 × 20 mm).

The weight loss of Korean red pine wood after the decay resistance test at heat conditions of the sapwood control, heat-treated sapwood, and control, and heat-treated heartwood decreased to 22.27, 7.15, 20.00, and 6.67%, respectively. The weight loss of Japanese larch wood after decay resistance test under the same conditions also decreased to 21.79, 6.67, 11.16, and 1.46%, respectively. Weight loss after the decay resistance test also dropped with the heat treatment.

Further, heat treatment using a catalyst enhances decay resistance at the wood surface because of charcoal coatings that result from the treatment. In this process, the color of the specimen is blackened and the weight and density are decreased to cause a heat deterioration. The weight losses were higher in the Korean red pine wood than in the Japanese larch wood. Also, the lowest weight loss was identified as the heat-treated Japanese larch heartwood. Generally Japanese larch is known as a high decay resistance species. This can be explained by the higher decay resistance of Japanese larch wood based on the influence of phenols of extractives and the bigger size of heartwood ratio than those of Korean red pine wood; the influence of phenolics on wood decay resistance was demonstrated for Japanese larch heart wood (Gierlinger *et al*., 2004).

Pinus densifora

Larix kaempferi



Control | Heat-treated wood | Control | Heat-treated wood

Sapwood (Radial surfaces) | Heartwood (Radial surfaces)

Fig. 2. SEM micrographs (500×) of Korean red pine and Japanese larch for radial section (500×) in control of sapwood, heat-treated sapwood, control of heartwood, and heat-treated heartwood.

3.5. Anatomical observation by scanning electron microscope

Fig. 2. shows SEM micrographs of control wood and heat-treated wood (96 h) using the catalyst of Korean red pine and Japanese larch. The heat-treated sapwood and heartwood revealed higher anti-fungal activities in SEM images. Padhiar and Alvert (2011) reported this possibility with evidence from anatomical studies. The presence of hyphae decreased in all samples after heat treatment using the catalyst (Fig. 2). A crack was observed along the pit border of the bordered pit in the control and heat-treated wood. This phenomenon is known to be caused by the decomposition of the hyphae cell wall. Additionally, the fungi penetrated the wood through the pit apertures.

4. CONCLUSION

This study was conducted to confirm the ability to enhance the decay resistance of sapwood and heartwood after heat treatment using the catalyst. The weight and density of wood from the two species decreased after the heat treatment using the catalyst. Further, the rate of coverage by fungal hyphae reduced for the heat-treated heartwood when the sulfuric acid was used. Weight loss after the decay resistance test also decreased after the heat treatment. The weight losses were lower in heartwood than those in sapwood. In particular, weight loss after fungal decay was maintained to a greater extent in the heat-treated heartwood. There was a decrease in the observation of hyphae after heat treatment using the catalyst. Therefore, heat treatment using the catalyst effectively increased the resistance of wood to decay caused by fungi.

ACKNOWLEDGEMENT

This study was carried out with the support of 'Research center for ecological restoration in damaged land(Project No. S211214L020410)' provided

by Korea Forest Service and also the brown-rot fungus, *Fomitopsis palustris*, were provided by Korea Forest Research Institute.

REFERENCES

Awoyemi L., Jones, I.P. 2011. Anatomical explanations for the changes in properties of western red cedar (*Thuja plicata*) wood during heat treatment. Wood Science and Technolgy 45: 261～267.

Gierlinger N., Jacques D., Grabner M., Wimmer R., Schwanninger M., Rozenberg P., Paques L.E. 2004. Colour of larch heartwood and relationships to extractives and brown-rot decay resistance. Trees 18: 102～108.

Jain S.H., Nagaveni H.C., Vijayalakshmi G. 2011. Effect of leaf and bark extracts of *Cleistanthus collinus* (Benth. & Hook) and *Prosopis juliflora* (Sw.) DC in combination with inorganic compounds against wood decay fungi. Journal of the Indian Academy of Wood Science 8(2): 198～200.

Kocaefe D., Poncsak S., Tang J. 2010. Effect of heat treatment on the mechanical properties of North American jack pine: thermogravimetric study. Journal of materials science 45: 681～687.

KS F 2198. 2011. Determination of density and specific gravity of wood (in Korean). Korea Standards Association, Seoul Korea.

Nagaveni H.C., Vijayalakshmi G., Sundararaj R. 2011. Decay resistance of some imported timbers against brown and white-rot fungi. Journal of the Indian Academy of Wood Science 8(2): 190～192.

Poncsak S., Kocaefe D., Bouazara M., Pichette, A. 2006. Effect of high temperature treatment on the mechanical properties of birch. Wood Science and Technology 40(8): 647～663.

Sini M.K., Toni A., Pertti V. 2006. The water absorption of sapwood and heartwood of Scots pine and Norway spruce heat-treated at 170℃, 190℃, 210℃ and 230℃. Holz als Roh- und Werkstoff 64: 192～197.

Wang X., Liu S., Chang H., Liu J. 2014. Sol-gel deposition of TiO_2 nanocoatings on wood surfaces with enhanced hydrophobicity and photostability. Society of Wood Science and Technology 46(1): 109～117.

Won K.R., Hong N.E., Kwon S.S., Byeon H.S. 2015. Effects of heating temperature and time on the physical properties of heat-treated wood. Journal of Agriculture & Life Science 49(6): 1～8.

Won K.R., Hong N.E., Park H.M., Byeon H.S. 2016. Evaluation of sapwood and heartwood decay resistance after immersion-treatment with pyroligneous liquor. Journal of the Korean Wood Science and Technology 49(6): 1～8.

Yildiz S., Gezer E.D., Yildiz U.C. 2006. Mechanical and chemical behavior of spruce wood modified by heat. Build Environ 41: 1762～1766.