Full Length Research Paper

Preliminary evaluation of boron release and biological resistance of wood treated with disodium octoborate tetrahydrate (DOT) and a water-repellent compound

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Wood specimens were vacuum-treated with disodium octoborate tetrahydrate (DOT) and a commercial water repellent compound. Leachates sampled from the leaching cycles for 10 days and extracts from treated wood specimens were analyzed for boron content. Treated wood specimens were exposed to wood degrading fungi in Petri dishes. Wood specimens were also subjected to termite resistance tests by using the subterranean termites. Treatments with the water repellent compound resulted in nearly 50% less boron leaching at 0.5 and 1% DOT concentration levels in comparison with DOT-only treated specimens. More boron release was seen in the specimens treated with 0.1% DOT and the water repellent compound when compared to either 0.5 or 1% DOT + water repellent compound treatments. Wood specimens treated with the compound and 0.5% DOT or more concentrations were well protected from fungal attack even after leaching course. All unleached specimens showed perfect protection against termites; however, mass losses in control specimens and leached specimens were nearly 30%. After treatments with 0.5 and 1% DOT and the compound, nearly 50% of total boron remained in these specimens after leaching course increased termite resistance of wood. The termite mortalities were in accordance with the mass losses that occurred in the specimens.

Key words: Dipropylene glycol monomethylether, boron release, water repellent, decay resistance, termite resistance.

INTRODUCTION

Considerable work has been done on modification of wood by treatments with suitable polymers and waterborne polymer systems capable of increasing water repellency and dimensional stabilization of wood. One of the main advantages of chemical wood modifications is to make the wood durable without any biocides; however, modified wood might still be susceptible to biodegradation in both indoor and outdoor applications. Combinations of polymer and monomer systems with various biocides have been found to have potential for reducing biocide leaching rates, enhancing the properties of wood and increasing biological resistance against wood degrading fungi, termites and insects (Takashi, 1996; Johnson and Rowell, 1988; Dauvergne et al., 2000; Kartal et al., 2004).

Following acetylation, isocynate treatment or dimethylol dihydroxy ethylene urea (DMDHEU) treatments, it is possible to react these chemicals with wood cell wall polymers causing a strong cell wall bulking. Treatment of wood with vinyl polymers is advantageous for both preservation and water repellency. Treatment of wood by the polymerization of various monomer and monomer systems contributes to dimensional stability and strength properties of wood (Yalinkilic et al., 1998; Solpan and

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Abbreviations: DOT, disodium octoborate tetrahydrate; RH, relative humidity; MMA, methyl methacrylate; AGE, allyl glycidyl ether; MEA, malt extract agar; BAE, boric acid equivalent.

Guven, 1999a, b, c). Extensive research has been conducted with varying anhydrides and monomers (Imamura and Nishimoto, 1986; Johnson and Rowell, 1988; Beckers et al., 1994; Takashi, 1996; Forster et al., 1997; Suttie et al., 1997; Timar et al., 1999; Devi et al., 2003), methyl methacrylate (MMA), allyl glycidyl ether (AGE), textile finishing agents such as DMDHEU and several different water repellents such as organo-silicon compounds, oils, etc., to limit biocide release from treated wood and increase decay and termite resistance of biocide-treated and chemically modified wood (Verma et al., 2005; Xie et al., 2005, 2008). Yalinkilic et al. (1998) used styrene and methyl methacrylate (MMA) to bulk the cell lumens to prevent easy water-boron contact for decreasing boron leaching from wood. Treatment of wood with monomers can be best achieved by proper selection of consalidant materials. The structures of cellulose, lignin and hemicelluloses, main components of cell wall, have found to be in accordance with ally glycidyl ether (AGE) as a potential monomer for the conservation and consolidation of wood (Solpan and Guven 1998, 1999a, b, c; Kartal et al., 2004).

The current paper evaluated a commercial water repellent compound used mainly in the textile industry to decrease boron leaching from treated wood and increase decay and termite resistance of treated wood.

MATERIALS AND METHODS

Wood specimens

Wood specimens, 20 (radial) by 20 (tangential) by 10 (longitudinal) mm, were cut from sapwood portions of Scots pine wood (*Pinus sylvetsris* L.). Before treatments, all wood specimens were conditioned at 20 °C and 65% relative humidity (RH) for two weeks. The specimens were free of knots and a visible concentration of resins, and showed no visible evidence of infection by mold, stain or wood-degrading fungi.

Treatments

There were two different processes for preservative treatments: double treatments and single treatments (Table 1). In double treatments, wood specimens were first treated with DOT at either 0.1, 0.5 or 1% (% m/v) concentrations. The specimens were then dried at 60 °C for 24 h and then reconditioned at 20 °C and 65 % relative humidity (RH) for one day and treated with a commercial water and oil repellent compound called FORGUARD M[®] (Figure 1). FORGUARD M[®] is a chemical resin emulsion which provides water and oil repellency to cotton-polyester synthetic mixtures. The compound contains 3.6% dipropylene glycol monomethylether, 12% solids and 78.4% water in its formulation. It readily dissolves in cold water, shows weak cationic characteristics and its pH is 3 to 5.

In single treatments, DOT was mixed with the compound yielding either 0.1, 0.5 or 1% (% m/v) DOT concentrations. In both treatments, treatment cycle consisted of a 40 min vacuum (-88 KPa absolute pressure) in a treatment desiccator. After all treatments, the specimens were blotted dry and reweighed to determine the uptake chemical retention. All treated specimens were then reconditioned at 20°C and 65% RH for one day and then dried at 60°C for one day before leaching process.

Leaching

The leaching process was conducted according to Japanese Industrial Standard (JIS) K 1571 (JIS 2004), the process involved immersing wood specimens in deionized water, stirring with a magnetic stirrer (400 - 450 rpm) at 27 °C for 8 h followed by drying at 60 °C for 16 h. This cycle was repeated 10 times. After each leaching cycle, the water was renewed with fresh deionized water to a ratio of 10 volumes of water to 1 volume of wood.

Boron analyses

The sample preparation for boron analyses was similar to the American Wood Preservers' Association (AWPA) A2-98 standard method (AWPA, 1999). The specimens were ground to pass through a 40-mesh screen in the Wiley mill, oven-dried, and 1.5 g of ground wood was weighed to the nearest 0.001 g into a 250 ml flask. For each treatment group, two specimens were ground and analyzed. One hundred milliliter (100 μ I) of deionized water was added to the flask containing the ground wood. The flask was placed in a water bath at 90 to 95 °C for 60 min with agitation every 15 min. After cooling, the contents in the flask were filtered through Whatman No. 4 filter paper, rinsed 3 times with 20 ml of hot deionized water, and diluted to 200 ml in a volumetric flask.

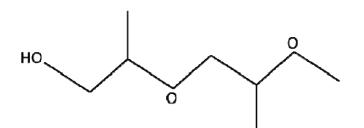
Both leachates sampled from the leaching cycles for 10 days and extracts from the treated wood were analyzed with an inductively coupled plasma (ICP) spectrometry (ICP-S 1000III Shimadzu Co., Ltd., Japan). The percentage reduction of boron in the specimens was calculated based on the initial amount of boron in the specimens.

Decay resistance tests

Decay resistance of treated wood specimens was evaluated by inserting the specimens directly into Petri dishes inoculated with Basidiomycetes fungi. Before decay testing, all wood specimens were dried at 60°C for 3 days. Two Basidiomycetes, brown-rot fungus, Tyromyces (Fomitopsis) palustris (Berk. et Curt) Gilbn. &Ryv. (FFPRI 0507) and white-rot fungus, Trametes (Coriolus) versicolor (L.:Fr.) Pilat. (FFPRI 1030) were inoculated on 2% malt extract agar (MÉA) in Petri dishes separately for 3 weeks at 23°C before placement of the specimens into the dishes. Wood specimens were autoclaved at 121°C, 15 psi for 20 min for sterilization and then placed into the inoculated Petri dishes. One specimen was placed in each dish and unheated specimens served as controls. Five specimens were used for each concentration and each fungus. After a 12-week incubation period in a temperature and humidity-controlled chamber at 26°C and 65% RH, wood specimens were taken out, re-dried at 60 °C for 3 days and reweighed to calculate mass losses based on the weights of the specimens before and after decay resistance tests.

Termite resistance tests

Untreated and treated specimens were exposed to the subterranean termites, *Coptotermes formosanus* Shiraki, according to the JIS K 1571 standard method (JIS 2004). An acrylic cylinder (80 mm in diameter, 60 mm in height) whose lower end was sealed with a 5 mm thick hard plaster (GC New Plastone, Dental Stone, GC Dental Industrial Corp., Tokyo, Japan) was used as a container. A test specimen was placed at the centre of the plaster bottom of the test container. A total of 150 worker termites collected from a laboratory colony of RISH, Kyoto University were introduced into each test container together with 15 termite soldiers. Five wood specimens per treatment were assayed against the termites. The assembled



CH₃(OC₃H₆)₂OH - Dipropylene glycol monomethyl ether

Figure 1. Chemical formula of dipropylene glycol monomethylether.

containers were set on damp cotton pads to supply water to the specimens and kept at 28 °C and >85 % RH in darkness for three weeks. The mass losses of the specimens due to termite attack were calculated based on the differences in the initial and final oven-dry (60 °C, 3 days) weights of the specimens after cleaning off the debris from the termite attack.

Data analysis

For statistical analysis, Duncan's multiple range test (p < 0.05) was used to evaluate the differences among mass losses in the decay and termite resistance tests.

RESULTS AND DISCUSSION

Table 1 gives DOT retention levels in the specimens before and after 10-day-leaching course. Figure 2 gives boron leaching as ppm from treated wood specimens during leaching course for 10 days. Percentage boron released from specimens is given in Figure 3. All boron was leached out from DOT-only treated wood specimens; however, less boron release was seen in the specimens containing water repellent compound. For 0.5 and 1% DOT concentration levels, percentage boron release in double treatments was lower than that in single treatments, suggesting that stronger chemical bonding of the compound might have occurred in wood. Nearly 50% of boron remained in the specimens treated in double process after 10-day leaching. Less boron concentration resulted in more boron leaching from wood specimens treated with water repellent compound.

Decay resistance tests results are shown in Table 2. All untreated control and disodium octoborate tetrahydrate (DOT) only treated wood specimens after leaching (without treatment with the water repellent compound) showed considerably higher mass losses when compared to unleached specimens. Treatment with the water repellent compound resulted in less mass losses after leaching due to less DOT leaching from respective specimens. In general, double treatments caused decreased mass losses for both *T. palustris* and *C. versicolor* since DOT retention levels in double treatments were

higher than those in single treatments. Mass losses in the specimens subjected to laboratory termite resistance tests for 3 weeks and termite mortalities occurred during the tests (Table 2). Untreated control specimens showed nearly 30% mass losses; however, mass losses in all unleached and leached specimens decreased after treatment with water repellent compound. The water repellent compound solution itself also caused decreases in mass losses. Disodium octoborate tetrahydrate only treated and leached specimens were not resistant against termites due to excessive boron release from specimens during leaching. As DOT concentration increased in these treatments, mass losses decreased after leaching. Treatment with the compound resulted in considerably less mass losses in the specimens because of less boron leaching from wood specimens treated with water repellent compound. In 1% DOT treatments, even leached specimens showed substantially low mass losses after treatment with water repellent compound. In general, double treatments resulted in lower mass losses when compared to single treatments depending on less boron release during leaching. Termite mortalities also conformed to the mass losses that occurred in the specimens. For commercial use against termites, retention in excess of 4.5 kg m⁻³ boric acid equivalent (BAE) is recommended. In the UK, a minimal cross-sectional retention of 1.8 kgm⁻³ BAE is recommended; however, in the United States, lumber is treated with disodium octoborate tetrahydrate, in that borate retention of 2.7 kg m⁻³ BAE is required to control fungi, beetles and native termites, and 4.5 kg m-³ BAE is needed to eliminate Formosan subterranean termites (Lloyd, 1997). In our study, treatment with water repellent compound in single and double treatments resulted in 2.8 and 3.8 kg m⁻³ DOT retention, respectively, after leaching. Treatments with 0.1% DOT and water repellent compound also showed low mass losses due to probably toxic effect of the compound to termites since mass losses in treated specimens with only the compound was about 8%. Termite mortality might be also an evidence for this since the mortality was 100% for unleached specimens treated with the compound only.

Table 1. Wood specimens, treatments and DOT retention levels before and after leaching.

Treatments	Leaching	DOT retention (kg m ⁻³) by ICP			
Untreated control	-	-			
0.1% DOT	Leached	0.00			
0.1% DOT	Unleached	0. 76 (0.03)			
0.1% DOT + water repellent compound (single treatment)	Leached	0.13 (0.01)			
0.1% DOT + water repellent compound (single treatment)	Unleached	0.56 (0.05)			
0.1% DOT + drying + water repellent compound (double treatment)	Leached	0.10 (0.03)			
0.1% DOT + drying + water repellent compound (double treatment)	Unleached	0.77 (0.05)			
0.5% DOT	Leached	0.00			
0.5% DOT	Unleached	3.96 (0.11)			
0.5% DOT + water repellent compound (single treatment)	Leached	1.00 (0.27)			
0.5% DOT + water repellent compound (single treatment)	Unleached	2.75 (0.28)			
0.5% DOT + drying + water repellent compound (double treatment)	Leached	1.85 (0.13)			
0.5% DOT + drying + water repellent compound (double treatment)	Unleached	3.95 (0.18)			
1% DOT	Leached	0.00			
1% DOT	Unleached	7.77 (0.31)			
1% DOT + water repellent compound (single treatment)	Leached	2.83 (0.27)			
1% DOT + water repellent compound (single treatment)	Unleached	6.20 (0.54)			
1% DOT + drying + water repellent compound (double treatment)	Leached	3.77 (0.19)			
1% DOT + drying + water repellent compound (double treatment)	Unleached	7.45 (0.29)			

Values in parentheses are standard deviations.

Conclusion

In this study, we evaluated the effects of a commercial water and oil repellent compound on boron release from treated wood and termite resistance of wood under laboratory conditions. Treatments with the compound helped the wood specimens retain about 3 kg m⁻³ DOT after leaching process during 1% DOT treatments. Previous studies have shown that termite resistance usually requires a retention level of more than 1 kg m⁻³ BAE in wood; however, a retention level of less than 1 kg m⁻³ is needed to protect against fungal wood decay. In our study, the specimens treated with 0.5 and 1% DOT and water repellent compound showed good resistance against the fungi tested and subterranean termites as a

result of retained boron after leaching process. Further studies are in progress for wettability and dimensional stability tests and better understanding of chemistry of the compound to fix the boron into the wood.

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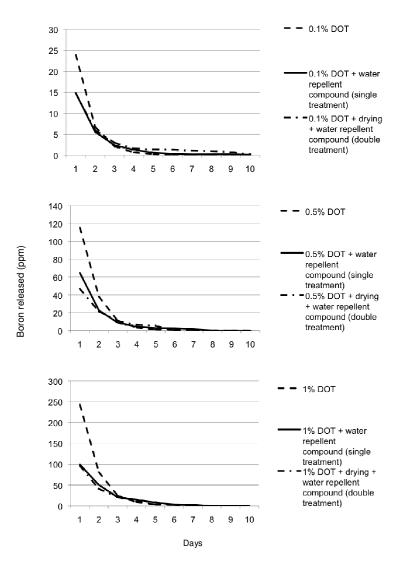


Figure 2. Boron released from treated wood specimens.

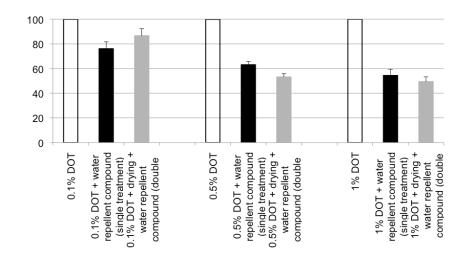


Figure 3. Percentage of boron released from treated wood specimens.

Boron released (%)

Table 2. Mass losses in decay and termite resistance tests and termite mortalities in termite resistance tests^a.

Treatments	Leaching	Mass loss in decay resistance tests (%)				Mass loss in termite resistance tests (%)		Termite mortality (%)
		T. palustris		C. versicolor		C. formosanus		
		Average	S.D.	Average	S.D.	Average	S.D.	Average
Untreated control	-	15.08A	7.19	13.40AB	2.47	29.33A	1.23	0
0.1% DOT	Leached	7.75D	3.23	18.34A	6.05	24.67AB	2.45	0
0.1% DOT	-	2.01EF	1.38	2.25D	0.99	11.34B	1.90	5
0.1% DOT + water repellent compound (single treatment)	Leached	14.85A	1.91	10.08B	1.33	12.27B	1.14	0
0.1% DOT + water repellent compound (single treatment)	-	8.64C	2.73	6.53C	1.38	3.52D	0.87	100
0.1% DOT + drying + water repellent compound (double treatment)	Leached	9.68C	5.90	2.32D	0.46	9.70BC	0.45	0
0.1% DOT + drying + water repellent compound (double treatment)	-	3.98E	0.43	2.16D	0.35	2.07E	0.77	100
0.5% DOT	Leached	11.42AB	4.00	17.10A	5.86	20.98AB	3.21	5
0.5% DOT	-	3.32E	0.78	0.46E	0.65	8.23BC	1.67	70
0.5% DOT + water repellent compound (single treatment)	Leached	4.36E	1.07	3.95D	1.16	7.13C	0.55	20
0.5% DOT + water repellent compound (single treatment)	-	3.23E	0.88	2.84D	0.78	4.52D	2.34	100
0.5% DOT + drying + water repellent compound (double treatment)	Leached	3.03E	0.37	2.21D	0.70	4.41D	1.09	100
0.5% DOT + drying + water repellent compound (double treatment)	-	1.46F	0.21	2.08D	0.51	2.37E	0.89	100
1% DOT	Leached	6.22D	3.96	20.83A	6.26	21.78AB	2.98	5
1% DOT	-	0.64F	0.32	0.91E	0.59	2.24E	0.11	95
1% DOT + water repellent compound (single treatment)	Leached	2.42EF	0.34	2.32D	0.39	2.92E	0.00	100
1% DOT + water repellent compound (single treatment)	-	1.56F	1.29	1.60DE	0.17	1.05E	2.34	100
1% DOT + drying + water repellent compound (double treatment)	Leached	3.57E	0.75	1.99DE	0.43	2.34E	0.23	100
1% DOT + drying + water repellent compound (double treatment)	-	2.69EF	0.59	1.78DE	0.15	1.94E	0.22	100

^a Each value represents the average of 12 and 5 specimens for decay and termite resistance tests, respectively. The same letters in each column indicate that there is no statistical difference between the specimens according to Duncan's multiple range test (p ≤ 0.05).

REFERENCES

- AWPA (1999). Standard methods for analysis of waterborne preservatives and fire-retardant formulations. American Wood Preservers' Association A2-98. Birmingham, Alabama.
- Beckers EPJ, Militz H, Stevens M (1994). Resistance of acetylated wood to Basidiomycetes, soft rot and blue stain. International Research Group on Wood Preservation, Stockholm, Sweden Doc. IRG/WP 94-40021.
- Dauvergne ET, Soulounganga P, Gerardin P, Loubiinoux B (2000). Glycerol/glyoxal: A new boron fixation system for wood preservation and dimensional stabilization. Holzforsch. 54: 123-126.
- Devi RR, Ali I, Maji TK (2003). Chemical modification of rubber wood with styrene in combination with a crosslinker: effect on dimensional stability and strength property. Bioresour. Technol. 88: 185-188.
- Forster SC, Hale MD, Williams G (1997). Efficacy of anhydrides as wood protecting chemicals. International Research Group on Wood Preservation, Stockholm, Sweden Doc. IRG/WP 97-30162.
- Imamura Y, Nishimoto K (1986). Resistance of acetylated wood to attack by subterranean termites. Wood Res. 72: 37-44.
- JIS (2004). Test methods for determining the effectiveness of wood preservatives and their performance requirements (in Japanese). Japanese Standard Association K 1571.
- Johnson BR, Rowell RM (1988) Resistance of chemically modified wood to marine borers. Mater Org. 23(2): 147-156.
- Kartal, SN, Yoshimura T, Imamura Y (2004). Improvement of boron leachability from disodium octoborate tetrahydrate (DOT)-treated wood by in situ copolymerization of allyl glycidyl ether (AGE) with methyl methacrylate (MMA). Int. Biodeterior. Biodegrad. 53(2): 111-117.
- Lloyd JD (1997). International status of borate preservative systems. In: Proceedings of the Second International Conference on Wood Protection with Diffusible Preservatives and Pesticides. Proceedings No. 7284, Forest Products Society, Madison, WI pp. 45-54.
- Solpan D, Guven O (1998). Comparison of the dimensional stabilities of oak and cedar wood preserved by in situ copolymerization of ally glycidyl ether with acrylonitrile and methyl methacrylate. Die Angewande Makromolekulare Chemie. 259: 33-37.
- Solpan D, Guven O (1999a). Preparation and properties of some wood/(co)polymer composites. Die Angewande Makromolekulare Chemie. 269: 30-35.
- Solpan D, Guven O (1999b). Improvement of mechanical stability of beech wood by radiation-induced in situ copolymerization of ally glycidyl ether with acrylonitrile and methyl methacrylate. J. Appl. Polym. Sci. 71: 1515-1523.

- Solpan D, Guven O (1999c). Preservation of beech and spruce wood by allyl alcohol-based copolymers. Radiat. Phys. Chem. 54: 583-591.
- Suttie ED, Hill CAS, Jones D, Orsler RJ (1997). Assessing the bioresistance conferred to solid wood by chemical modification. International Research Group on Wood Preservation, Stockholm, Sweden Doc. IRG/WP 97-40099.
- Takashi M (1996). Biological properties of chemically modified wood. In: Hon S (ed). Chemical modification of lignocellulosic materials, Marcell Dekker Inc, New York, pp. 331-359.
- Timar MC, Pitman A, Mihai MD (1999). Biological resistance of chemically modified aspen composites. Int Biodeterior Biodegrad. 43: 181-187.
- Verma P, Mai C, Krause A, Militz H (2005). Studies on the resistance of DMDHEU treated wood against white-rot and brown rot fungi. International Research Group on Wood Protection, Stockholm, Sweden Doc. IRG/WP/05-10566.
- Xie Y, Krause A, Mai C, Militz H, Richter K, Urban K, Evans PD (2005). Weathering of wood modified with the N-methylol compound 1,3dimethylol-4,5-dihydroxyethyleneurea. Polym Degrade Stabil. 89(2): 189–199.
- Xie Y, Krause A, Militz H, Mai C (2008). Weathering of uncoated and coated wood treated with methylated 1,3-dimethylol-4,5dihydroxyethyleneurea (MDMDHEU). Holz Roh Werkst. 66: 455– 464.
- Yalinkilic MK, Tsunoda K, Takahashi M, Gezer ED, Dwianto W, Nemoto H (1998). Enhancement of biological and physical properties of wood by boric acid-vinyl monomer combination treatment. Holzforsch. 52: 667-672.