Fiberboards treated with N'-N-(1, 8-Naphthalyl) hydroxylamine (NHA-Na), borax, and boric acid

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Abstract

This paper reports on the physical and mechanical properties and decay and termite resistance of fiberboard panels made from pine and beech treated with N'-N-(1, 8-Naphthalyl) hydroxylamine sodium salt (NHA-Na), borax, and boric acid at varying loadings. The panels were manufactured using 10 percent urea-formaldehyde resin and 1 percent NH_4Cl. Mechanical and physical tests demonstrated no statistically significant reduction in modulus of rupture of specimens treated with borax + NHA-Na compared to untreated control specimens. Similar results were obtained for modulus of elasticity for all treatments at concentrations of 1 percent and lower. Internal bond values of treated specimens were somewhat reduced in comparison with values of control specimens, but higher than DIN EN 622-5 standards for medium density fiberboard. Treated specimens showed increased resistance against the decay fungi *Fomitopsis palustris* and *Trametes versicolor* and the Formosan subterranean termite *Coptotermes formosanus* Shiraki. For individual treatments, specimens treated with borax and boric acid showed lower weight loss compared to specimens treated with NHA-Na alone. The possible synergy of borax + NHA-Na resulted in low water absorption, high modulus of elasticity, and increased resistance to decay and termites.

Wood-based composites are susceptible to biological attack because the bulk of the material is usually wood (Curling and Murphy 1999, Morrell 2002). It is possible to increase the resistance of composite products to biodeterioration without using a preservative by using durable wood species for furnish, especially in applications with low to moderate decay hazard. However, wood-based composites need protection in applications with severe decay and termite hazard (Barnes and Amburgey 1993, Evans et al. 2000, Kartal and Green 2003). Adhesives and additives such as preservatives play an important role in maintaining the integrity of wood-based composites (Evans et al. 1997, Akbulut et al. 2000, Kartal and Green 2003). The addition of biocides in composite production helps increase decay resistance, but poses potential problems (Barnes and Amburgey 1993). The biocides must be compatible with resins and other additives in terms of their effect on mechanical and physical properties and still be effective in protecting wood from fungi and insects.

Borates are environmentally benign wood preservatives that can be used to increase the resistance of composites to biodegradation. However, most borates are water-soluble and susceptible to leaching (Barnes and Amburgey 1993, Murphy et al. 1993, Laks and Manning 1997, Tsunoda et al. 2002). Laks and Palardy (1990) showed that addition of zinc borate to flakeboards caused some decrease in mechanical and physical properties as borate content increased. On the other hand, Tsunoda et al. (2002)

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Forest Prod. J. 54(10):59-64.

Table 1. — Preservative contents of fiberboard panels.

Fiberboard number	Preservative	Target preservative concentration[a]	Preservative content [b]	
			Target	Actual
		(%)	- - - - - - - - - - (kg/m^3) - - - - - - - - - -	
1	Control	--	--	--
2	NHA-Na	1	8.40	7.98
3	NHA-Na	0.5	4.20	4.10
4	NHA-Na	0.1	0.84	0.80
5	Borax	3	25.20	23.30
6	Borax	1	8.40	7.80
7	Borax	0.5	4.20	3.90
8	Borax	0.1	0.84	0.90
9	Boric acid	3	25.20	22.10
10	Boric acid	1	8.40	8.20
11	Boric acid	0.5	4.20	4.11
12	Boric acid	0.1	0.84	0.83
13	Borax + NHA-Na	0.1 + 0.1	0.84 + 0.84	0.81 + 0.80

[a] Based on ovendry furnish weight and BAE for borax and boric acid.
[b] Based on BAE for borax and boric acid.

found no significant loss in the mechanical and physical properties of fiberboard treated with zinc borate.

Originally evaluated as a water-soluble calcium-precipitating agent, N'-N-(1, 8-Naphthalyl) hydroxylamine sodium salt (NHA-Na) has been thought to disrupt normal calcium cycling in fungal hyphae, rendering the agent either fungicidal or fungistatic (Sobota et al. 1988). The selective precipitation of calcium by NHA-Na at several concentrations has been shown to protect southern yellow pine from fungal decay and termite damage in laboratory tests (Green et al. 1996a,1996b,1997a,1997b,2000; Crawford and Green 1999). Field tests have also shown that pressure treatment with 1 percent NHA-Na can protect southern yellow pine to the same extent as does chromated copper arsenate type C (CCA-C) for 38 months in environments with high decay hazard indices (Green et al. 1997a,1997b; Crawford and Green 1999). Precipitation of boron in wood via NHA-Na treatments seems to have the potential to reduce boron leachability. In addition, the combination of boron and NHA-Na in wood seems to have a synergistic effect against wood-degrading organisms (Kartal and Green 2002; Kartal and Imamura 2003,200_).

In this study, we evaluated the mechanical and physical properties and biological resistance of fiberboard panels incorporated with NHA-Na, borax, and/or boric acid. The objectives were to determine the compatibility of NHA-Na with resins in terms of mechanical and physical properties of the composites and to compare the properties of fiberboard panels treated with NHA-Na to that of fiberboard panels treated with borax or boric acid.

Materials and methods

Manufacturing of fiberboard panels

The fiberboard panels were manufactured in the Wood Mechanics and Technology Laboratory, Forestry Faculty, Istanbul University. Pine (*Pinus nigra* Arnold *var. pallasiana*) and beech (*Fagus orientalis* Lipsky) chips were obtained from a commercial MDF plant in Gebze, Turkey. The chip furnish (50:50 blend of pine and beech) was thermoplasticized in an Asplund Defibrator (Metso Corp., Finland) using 7.6 kg/cm^2 steam pressure at 178ºC for 5 minutes. The resulting fiber was used to manufacture 26 fiberboard panels. N'-N-(1,8-Naphthalyl) hydroxylamine sodium salt (NHA-Na) ($C_{12}H_6NNaO_3$) (Aldrich Chemical Company, Inc., Milwaukee, Wisconsin), disodium tetraborate decahydrate (borax) ($Na_2B_4O_7{\cdot}10\ H_2O$) (Merck KgaA, Darmstadt, Germany), boric acid ($H_3\ BO_3$) (Merck KgaA, Darmstadt, Germany), or borax + NHA-Na were added to the furnish. Target loadings (based on ovendry furnish weight) were 1, 0.5, and 0.1 percent for NHA-Na; 3, 1, 0.5, and 0.1 percent boric acid equivalent (BAE) for borax and boric acid; and 0.1 + 0.1 percent BAE for borax + NHA-Na (**Table 1**). Treated and untreated (control) fiber was blended with 10 percent urea-formaldehyde resin (ovendry solid wood basis) and 1 percent NH_4Cl (ovendry solid wood basis) as hardener. The resin had a solids content of 55 percent. Wax was not used in the manufacture. Fiber mats at 10 percent moisture content were hot-pressed at 150ºC and 3.5 N/mm^2 for 7 minutes. After cooling, the panels were trimmed to a final size of 500 by 500 by 10 mm. The panels were then subjected to volatilization to remove free formaldehyde and low molecular weight resin compounds.

Analysis of preservative contents

The samples were prepared by a method similar to the AWPA A2-98 standard method (AWPA 1999). For each fiberboard panel, two 20- by 20- by 210-mm samples were ground and analyzed. The samples were ground to pass through a 40-mesh screen in a Wiley mill and ovendried; 1.5 g of ground sample was weighed in a 250-mL flask to the nearest 0.001 g. De-ionized water (100 mL) was added to the flask containing the ground sample. The flask was placed in a water bath at 90° to 95°C for 60 minutes, with agitation every 15 minutes. After cooling, the contents in the flask were filtered through Whatman #4 filter paper (Fisher Scientific, Pittsburgh, Pennsylvania), rinsed three times with 20 mL hot de-ionized water, and diluted to 200 mL in a volumetric flask. Extracts from fiberboard samples were analyzed for boron with an ICP sequential plasma spectrometer (ICP-S

Table 2. — Physical and mechanical properties of fiberboard specimens.[a]

Preservative	Target preservative content	Air-dry density	MOR	MOE	Internal bond strength	Thickness swell	Water absorption
	(%)	(g/cm^3)	(MPa)	(GPa)	(MPa)	---------- (%) ----------	
Control	--	0.85 (0.06)	23.55 (3.80)	1.70 (0.29)	1.34 (0.12)	23.87 (3.39)	56.25 (8.52)
NHA-Na	1	0.86 (0.05)	19.36 (3.10)	1.62 (0.13)	0.80 (0.16) **	36.17 (5.62) **	69.90 (10.47) **
	0.5	0.86 (0.08)	19.53 (4.11)	1.73 (0.40)	0.85 (0.21) **	34.11 (3.47) **	70.51(10.16) **
	0.1	0.84 (0.05)	20.22 (3.40)	1.81 (0.27)	1.01 (0.20) **	31.51 (3.71) **	54.38 (4.79)
Borax	3	0.84 (0.02)	20.81 (4.28)	1.12 (0.30) **	1.19 (0.06)	30.62 (5.70) **	62.73 (8.50)
	1	0.85 (0.05)	22.88 (4.04)	1.74 (0.36)	1.01 (0.17) **	29.78 (2.73) *	66.44 (7.26)
	0.5	0.88 (0.04)	20.91 (3.24)	1.59 (0.39)	1.28 (0.12)	29.07 (2.83)	62.41 (8.20)
	0.1	0.95 (0.03) **	29.58 (3.31) **	2.62 (0.49) **	1.69 (0.41) **	28.79 (4.57)	47.92 (7.06)
Boric acid	3	0.74 (0.04) **	9.04 (1.67) **	0.68 (0.13) **	0.61 (0.08) **	49.07 (2.66) **	110.41 (10.61) **
	1	0.85 (0.08)	17.69 (3.24) **	1.27 (0.32)	1.14 (0.19)	38.55 (5.85) **	72.82 (9.39) **
	0.5	0.86 (0.06)	18.69 (3.83) *	1.67 (0.40)	1.03 (0.20) **	33.32 (5.85) **	72.50 (10.61) **
	0.1	0.86 (0.07)	22.49 (2.87)	1.77 (0.32)	1.23 (0.32)	29.44 (4.17) *	56.74 (8.47)
Borax + NHA-Na	0.1 + 0.1	0.85 (0.08)	19.53 (4.11)	2.09 (0.52)	1.02 (0.13) **	33.05 (3.35) **	55.96 (6.02)

[a] Values in parentheses are standard deviations ($n = 10$). Asterisks denote significant difference compared with untreated control fiberboard specimens: * $p = 0.05$; ** $p = 0.01$.

1000III, Shimadzu Co. Ltd., Japan). An ultraviolet spectrophotometer (Hitachi U-2001, Hitachi Ltd., Japan) was used to obtain NHA-Na absorbance values at 340 nm (Kartal and Green 2002).

Evaluation of physical and mechanical properties

Fiberboard panels were cut into 250- by 50- by 10-mm specimens. Prior to testing, the specimens were conditioned at 65 percent relative humidity (RH) and 20°C for 4 weeks to reach 12 percent equilibrium moisture content. Three-point static bending modulus of rupture (MOR), modulus of elasticity (MOE), and internal bond strength were performed in conformance with EN standards (TSE 1996,1997). Thickness swell and water absorption measurements were made by immersing specimens in water in a horizontal position for 24 hours at ambient temperature in accordance with the EN 317 (TSE 1996) and ISO 819 (ISO 1975) standards, respectively.

For physical and mechanical properties, all multiple comparisons were first subjected to an analysis of variance (ANOVA) and significant differences between mean values of control and treated specimens were determined using Duncan's Multiple-Range Test.

Fungal bioassay

A laboratory decay test was conducted according to modified Japan Wood Preserving Association (JWPA) Standard 3 (JWPA 1992) using the brown-rot fungus *Fomitopsis palustris* (Berk. et Curt) Gilbn. & Ryn. and the white-rot fungus *Trametes versicolor* (L. ex Fr.) Quel. Both fungal cultures were obtained from the Laboratory of Deterioration Control, Wood Research Institute, Kyoto University, Japan. Fiberboard specimens, 20- by 20- by 10-mm, were ovendried at 60ºC in a laboratory oven until they reached constant weight. The specimens were then sterilized with gaseous ethylene oxide after measuring ovendried weight. Three specimens from the same board were placed in a glass jar containing 250 g quartz sand and 80 mL nutrient solution and then inoculated with liquid fungal culture. The jars were incubated at 27ºC and 80 percent RH for 12 weeks. Nine replicates were tested for each decay fungus and fiberboard panel. Weight loss was determined on the basis of the difference in initial dry weight and final dry weight of the specimen after surface debris from fungal attack was brushed away.

Termite bioassay

Termite tests were conducted according to JWPA Standard 11(1) (JWPA 1992). Fiberboard specimens, 20- by 20- by 10-mm, were ovendried at 60ºC until they reached constant weight and placed in the center of the 5-mm-thick hard plaster breeding container (8 cm diameter, 6 cm long). A total of 165 *Coptotermes formosanus* Shiraki termites (150 workers and 15 soldiers) were introduced into each container. The assembled containers were put on damp cotton pads to supply water to the specimens and kept at 28ºC and >80 percent RH for 3 weeks in a dark conditioning room. Termite mortality was determined regularly. Weight loss was determined on the basis of the difference in initial dry weight and final dry weight after surface debris from the termite attack was brushed away. Five replications were made for each board type.

Results and discussion

Physical and mechanical properties

Physical and mechanical properties of fiberboard specimens are shown in **Table 2**. Each value is an average of 10 specimens. Air-dry density values ranged between 0.84 and 0.88 g/cm^3, with the exception of values for specimens treated with 0.1 pecent borax (0.95 g/cm^3) and 3 percent boric acid (0.74 g/cm^3). Density plays an important role in influencing both physical and mechanical properties of wood and wood-based materials. In this study, the same processing conditions were chosen rather than varying the process to achieve equal board densities. The density values of the fiberboards tested met the requirements of EN 622-5 (TSE 1997) and EN 323 (TSE 1996); that is, a minimum of 0.65 g/cm^3 for 9- to 12-mm-thick boards.

The MOR and MOE values indicated that NHA-Na and borax treatments did not have adverse effects on bending strength. Specimens treated with NHA-Na and borax, regardless of loading level, showed no significant loss in

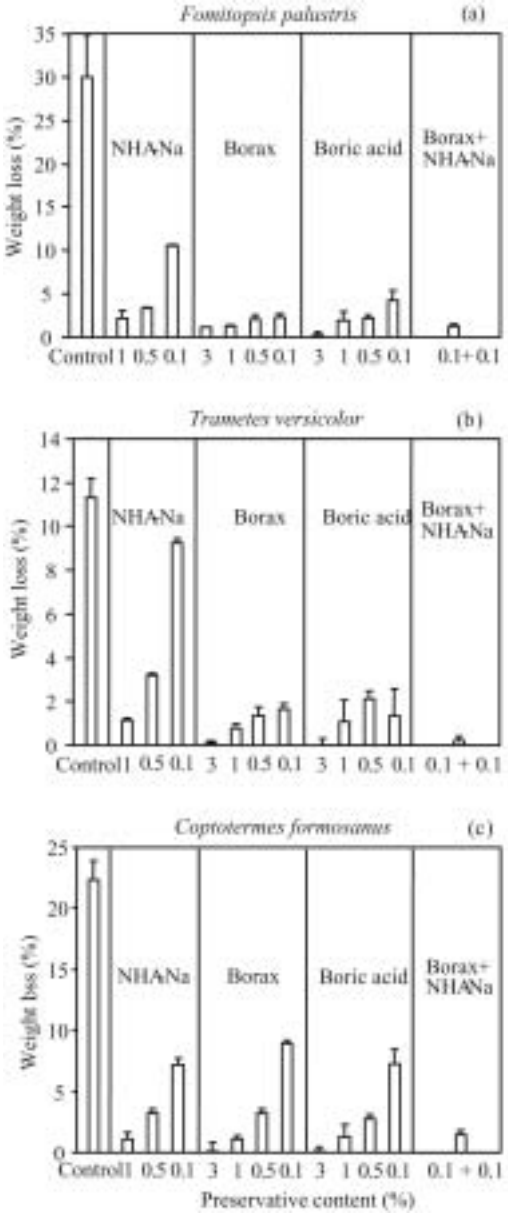

Figure 1. — Weight loss in fiberboard specimens after exposure to fungi and termites: (a) 12-week exposure to brown-rot fungus F. palustris; (b) 12-week exposure to white-rot fungus T. versicolor; and (c) 3-week exposure to Formosan subterranean termite C. formosanus.

MOE compared to control specimens. The MOE values of specimens treated with 0.1 percent borax or boric acid were higher than those of untreated control specimens and specimens treated with other loadings of borax or boric acid or other treatments. In addition, the MOR of 0.1 percent borax-treated specimens was higher than that of control specimens; the air-dry density of those specimens, however, was relatively higher than that of other specimens.

A decrease in average internal bond values, caused by possible interaction with the chemicals used, was observed in all treated fiberboard specimens with the exception of those treated with 0.1 percent borax. The internal bond values also decreased as boron and NHA-Na loading increased. However, internal bond values for all specimens exceeded the minimum property requirement of 0.60 MPa based on EN 622-5 standards.

In the 24-hour soaking tests, treated fiberboard specimens had higher average thickness swell and water absorption values compared to that of control specimens, suggesting that the chemicals adversely influenced the hygroscopic properties of the panels. At the lowest borax and NHA-Na loading levels and for the borax + NHA-Na treatment, water absorption values of treated specimens were lower than that of control specimens. Thickness swell, in turn, increased with higher preservative loading. Thickness swell and water absorption values were higher than those specified in the EN 622-5 and EN 317 standards, which could be attributed to the fact that wax was not used in manufacture.

The physical and mechanical properties of specimens treated with borax + NHA-Na may suggest that this mixture is compatible with the urea-formaldehyde resin. Our results are consistent with those of previous studies on wood composites treated with boron compounds. Water-soluble boron compounds may have little or no effect on the bonding performance of urea-formaldehyde resin; however, these chemicals do have a negative effect on the bonding performance of phenol-formaldehyde resin (Laks and Manning 1995, 1997; Tsunoda et al. 2002).

Decay and termite resistance

Average weight losses of fiberboard specimens exposed to decay fungi for 12 weeks are given in **Figures 1a and 1b.** All weight losses of treated boards were below 10 percent. For all specimens, *Fomitopsis palustris* caused more weight loss than did *Trametes versicolor.* The greatest weight losses were observed in control specimens. On the other hand, visual inspections revealed that all treated specimens were almost free from attack by both fungi. These results are consistent with those of a previous study by Tsunoda et al. (2002) for the same organisms and zinc borate as a preservative, suggesting that 0.25 percent BAE concentration is a threshold level for the same fungi. Murphy et al. (1993) found that several wood composites protected with vapor boron at a retention of 0.5 percent BAE were resistant to brown- and white-rot fungi. Our results indicated that at 0.1 percent BAE, borax treatment decreased weight loss to below 3 percent for both fungal exposures. Specimens treated with boric acid, however, lost slightly more weight when exposed to *F. palustris* compared with *T. versicolor.*

The calcium-precipitating agent NHA-Na has been shown to protect southern yellow pine from fungal decay and termite attack (Green et al. 1997b,

2000,2002). In the study reported here, specimens treated with NHA-Na experienced slightly higher weight loss compared with specimens treated with boron compounds. The performance of specimens treated with 0.1 percent BAE borax + 0.1 percent NHA-Na was better than that of specimens treated with 1 percent borax or 1 percent boric acid. These results may suggest that borax + NHA-Na has a synergistic effect against the brown- and white-rot fungi used in this study. Kartal and Imamura (2003) also showed that NHA-Na and boron compounds play a synergistic role in solid wood against *F. palustris* and *T. versicolor* decay fungi and the Formosan subterranean termite *C. formosanus.* NHA-Na and several boron compounds, including boric acid, disodium octaborate tetrahydrate, and calcium borate, also had inhibitory effects *in vitro* on the growth of these fungi.

Termite mortality was greater in all treated fiberboard specimens compared with control specimens, which suggests that the treatment chemicals improved resistance to termites. Termite mortality increased as preservative concentration increased and exceeded 90 percent in all treated specimens. Average termite mortality for control specimens was only 15.15 percent. Average weight loss of treated specimens measured by the termite bioassays is shown in **Figure 1c**. Average weight loss of control specimens was 22 percent. Weight loss of treated specimens decreased as preservative concentration increased. Weight loss over 7 percent occurred at the 1 percent concentration level of all preservatives. These results are similar to those of previous studies. Tsunoda (2001) and Tsunoda et al. (2002) showed that below the 0.5 percent concentration, specimens treated with zinc borate and boric acid were attacked by *C. formosanus*. Specimens treated with 0.1 percent BAE borax + 0.1 percent NHA-Na showed lower weight loss compared to specimens treated with NHA-Na, borax, and boric acid alone.

Conclusions

Our results suggest that incorporating borax + NHA-Na in fiberboards during manufacture does not have a negative effect on physical and mechanical properties. Fiberboards treated with 0.1 percent BAE borax + 0.1 percent NHA- Na had similar MOE and water absorption compared to that of control specimens. Treatment with borax + NHA-Na also improved decay and termite resistance compared with treatment with NHA-Na, borax, and boric acid alone. In addition, borax + NHA-Na has a synergistic effect on both fungal decay and termite attack compared to borax and boric acid at concentrations above 1 percent. Compared to boron alone, the lower leachability of NHA-Na may be beneficial for combined treatment of wood composites in areas of severe decay and termite hazard. Leaching tests and field trials are needed to determine the amount of depletion of boron as well as decay and termite resistance of fiberboard panels treated with NHA-Na and boron compounds.

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