USE OF BIOCIDES AND SOY OIL IN PRESERVATIVE TREATMENT OF STRUCTURAL FLAKEBOARD

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Abstract. Many uses of structural wood composite panels require preservative treatment to increase decay resistance. The most cost-effective way to treat structural flakeboard is done during manufacturing, but it is difficult to accomplish because of incompatibility between adhesive resins and preservatives. The objective of this study was to evaluate physical properties and decay resistance of flakeboard made with phenolic wood adhesive resins blended with biocides dissolved in soybean oil. The blended phenolic adhesive resins contained equal parts of iodo-propynyl butyl carbamate, propiconazole, and tebuconazole. Hybrid poplar flakeboards were made at the combined biocide retention levels of 0, 0.51, 0.81, and 1.63 kg/m^3. Results indicated that the strength and dimensional stability properties of flakeboard were not affected by the in-process preservative treatment. The biocides were stable and maintained their efficacy against decay after pressing boards at 200°C for 7 min. Boards treated with 1.63 kg/m^3 biocides sustained 2.5% to 5.0% weight loss after exposing to the brown-rot fungus (*Postia placenta*) for 12 wk compared with over 27% weight loss of nontreated boards.

Keywords: Flakeboard, preservative treatment, biocides, soy oil, brown-rot decay.

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INTRODUCTION

Fungal decay of oriented strandboard (OSB) for house construction may sometimes be a major problem if the material is allowed to get wet. Thus, preservative treatment of OSB panels is highly desirable to ensure long service. There are a number of ways to manufacture preservative-treated OSB, including treating wood furnishes prior to board manufacturing and incorporating preservatives during board manufacturing. Preservatives also can be administered after the panels are manufactured, but treating manufactured panels with waterborne formulations often results in swelling that compromises physical properties, while treating manufactured panels with oil-borne formulations is too costly. Recent research has shown that treating manufactured panels with supercritical fluids as preservative carrier solvents (Acad et al 1997; Oberdorfer et al 2004) and vapor phase treatment with borate esters (Murphy 1994) may overcome these problems.

As pointed out by Laks and Palardy (1993), mixing preservatives with adhesive resins or with wax emulsions is the most convenient way of manufacturing treated panels. However, many water-soluble preservative salts are incompatible with phenol–formaldehyde (PF) resins (Laks et al 1988; Vick et al 1990), resulting in poor adhesive bonds. Schmidt and Gertjejansen (1988) found that mixing powdered fungicide azaconazole with polymeric methyl diphenyl diisocynate (pMDI) adversely affected adhesive bond strength of flakeboard, but the powdered fungicide did not interfere with adhesive bonding when it was blended with PF resin. To reduce interactions between preservative and PF resin, Knudson and Gnatowski (1989) mixed zinc borate powder with powdered PF to make flakeboard and found the resulting flakeboard had acceptable physical properties. However, in the "dry" systems where dry preservative chemicals are mixed with adhesive resins or applied onto wood furnishes already coated with adhesive resins, it may affect efficacy of the preservatives. For example, Laks and Palardy (1993) found that flakeboard had poor decay resistance when chlorothalonil powder was applied onto wood flakes sprayed with pMDI. They attributed lack of protection of the flakeboard against fungal attack to immobility of chlorothalonil particles on the flake surfaces. Nevertheless, Vidrine et al (2009) reported that aspen strandboards bonded with PF resin and in-process treated with 10 dry chemicals, including zinc borate, copper ammonium acetate, copper diamine carbomate, micronized copper carbonate, and isothiazolone, performed well against mold and decay fungi and that the in-process preservative treatment did not adversely affect physical properties of aspen strandboards (Vidrine et al 2008).

Hybrid poplar flakeboard was successfully made with phenolic adhesive resins blended with a commercial preservative formulation containing three biocides in mineral spirit and soybean oil (Kuo et al 2006). The treated flakeboard had excellent physical properties and performed well against brown-rot decay but the resin/preservative blends generated high levels of undesirable mineral spirit vapor during pressing. In this study, biocide dissolved in soybean oil alone was blended with phenolic adhesive resins to fabricated flakeboard and the treated boards were evaluated for decay resistance and physical properties.

MATERIALS AND METHODS

Biocides and Adhesive Resins

A commercial wood preservative (Woodlife 111 4:1 Concentrate, Kop-Coat, Pittsburgh, PA) containing equal parts of 3-iodo-2-propynyl butyl carbamate (IPBC), propiconazole, tebuconazole, and paraffin in mineral spirit was used as the source of biocides. The commercial preservative solution was pan-dried at 100°C for 3 da to evaporate mineral spirit, obtaining a dark waxy paste without evident mineral spirit odor. No effort was made to analyze the effect of thermal exposure on biocide composition.

Flakeboards were made with three different phenolic adhesive resins. Resin 1 was a neat PF resin synthesized at 2.1 F/P molar ratio and catalyzed with 0.4 mol NaOH. The resulting neat PF resin was approximately 200 cps (Brookfield) in viscosity at 25°C and between 50 and 53% solid content. Resin 2 was prepared by blending 85% of the above laboratory-made PF resin and 15% soy flour hydrolysate, based on solids, at 50°C for 30 min just before board-making. The soy flour hydrolysate was obtained by alkaline hydrolysis of soybean flour (8% NaOH based on dry weight of soy flour) at 140°C for 2.0 h followed by evaporating excess water to approximately 36-38% solids. Resin 3 was a PF-crosslinked soy resin prepared by crosslinking soy flour hydrolysate with a PF crosslinking agent. The PF crosslinking agent was synthesized at 2.4 mol formaldehyde to 1 mol phenol catalyzed with 0.1 mol NaOH at 70°C for 1 h followed by heating at 95°C for 45 min. The PF crosslinking agent was about 50 cps viscosity at 25°C and 55% solids. The final resin containing 60% soy hydrolysate and 40% PF crosslinking agent was formulated at 70°C for 30 min. The PF-crosslinked soy resin contained 42-45% solids and was 9.5 in pH and ranging from 1000 to 1200 cps in viscosity.

Blending Biocides and Adhesive Resins

Crude soy oil was used as the biocide solvent in the amount of 15 g per 100 g of wet resin when blending. Crude soy soil was heated to 60°C to dissolve a specific amount of the waxy biocide paste. Target treating levels of combined biocides (IPBC, propiconazole, and tebuconazole) were 0 (without soy oil), 0.51, 0.81, and 1.63 kg/m^3. During blending, adhesive resins were heated to 60°C followed by adding soy oil containing specific amounts of preservative under strong stirring for 20 min. The resulting resins/preservative blends were dark-colored emulsions. Amounts and compositions of the three types of resin/preservative blends for making one three-board patch at 7% resin solids are listed as follows: PF/preservative blend: 504 g PF (50% solids) + 0 g or 76 g soy oil with preservative = 504 g or 580 g; PF/soy/preservative blend: 428 g PF (50% solids) + 102 g soy hydrolysate (37% solids) + 0 g or 80 g soy oil with preservative = 530 g or 610 g; and PF-crosslinked soy resin/preservative blend: 183 g PF (55% solids) + 409 g soy hydrolysate (37% solids) + 0 g or 89 g soy oil with preservative = 592 g or 681 g.

Flakeboard Fabrication and Testing

Flakeboards were made with juvenile hybrid poplar flakes 0.52 mm in thickness, 2-1.2 cm in width, and 6.35 cm in length. Flakes were dried to 4% MC prior to board making. Three replicates of three-board batches were made for each biocide retention level in the following conditions: resin solids = 7% without wax based on weight of dry flakes; target density = 0.70 g/c.c; flake orientation = random; board size = 38.1 × 38.1 × 1.27 cm; press temperature = 200°C; press time = 7 min. With 3 replicate patches, 9 boards of each treatment group were fabricated, obtaining a total of 99 test boards. Boards were conditioned in the laboratory (approximately 60% RH in early summer) for 7 da before trimming to 35.6 × 35.6 cm followed by determination of board density. Boards were evaluated for modulus of rupture (MOR), modulus of elasticity (MOE), internal bond strength (IB), and percent thickness swell (TS) according to ASTM D 1037 standard methods (American Society for Testing and Methods 1996a). Two static bending, six IB and two each cold TS and boiled TS samples were tested for each board. Initial measurements for thickness swell samples were obtained after the samples were conditioned at 65% RH at 20°C for 2 da.

Soilblock Culture Tests

Specimens 1.9 cm × 1.9 cm × 1.27 cm (board thickness) in dimension were used for decay resistance tests according to the ASTM D 1413-99 standard method (American Society of Testing and Materials 1996b). Four test specimens were randomly cut from each board, and a total of 36 test specimens from each treatment group were subjected to soilblock culture test without leaching. Percent weight loss was used as a measure of brown-rot decay resistance. Test specimens were oven-dried overnight at 100°C initially to record oven dry weights followed by autoclaving at 125°C and 0.14 MPa steam pressure for

20 min and exposed to the brown-rot fungus *Postia placenta* (FPL 698/ATCC 11538) for 12 wk. At the end of the fungal incubation period, specimens were cleaned and oven-dried overnight and percent weight loss was calculated based on the initial and final oven-dry weights.

Statistical Analysis

The General Linear Methods procedure of SAS (SAS Institute Inc 1987) was used to analyze the effects of resin type and preservative retention on flakeboard properties and decay resistance. The Scheffe's test in the General Linear Methods was used to conduct the contrast test of multiple means.

RESULTS AND DISCUSSION

The chemical composition of the biocides might have changed during pan-drying of the commercial preservative due to evaporation of the biocides, especially for paraffin and IPBC having melting points below 60°C. The pen-drying procedure was done to obtain biocides for this study. Since the main objective of this study was to evaluate a different preservative delivery system in flakeboard making, neither changes in biocide composition nor changes in the efficacy of these biocides as a result of pan-drying to remove mineral spirit were investigated. The biocide paste could be easily dissolved in soy oil at 60°C. When blending adhesive resins with biocides in soy oil, gelling or precipitation was not observed during or after blending. The resulting homogenous resin blends were dark in color and no apparent phase separation was observed after 1 h of blending. Blending resins with soy oil dissolved biocides reduced resin viscosity, especially with the PF-crosslinked soy resin which had high viscosity when formulated at solid content greater than 45%. Reduced resin viscosity and additional resin volume after blending facilitated better resin spray during flakeboard fabrication.

Physical properties of flakeboard are shown in Table 1, and statistical analysis of factors influencing board properties is summarized in Table 2.

Table 1. Physical properties and decay resistance of flakeboard[a] bonded with various resins and containing different amounts of preservatives.

Resin[b] type	Preservative (kg/m^3)	Density (g/cm^3)	MOR (MPa)	MOE (MPa)	IB (MPa)	Soak[c] (%TS)	Boil[d] (%TS)	Decay[e] (%WL)
PF	0	0.68 (0.02)[f]	34.85 (3.74)	4447 (337.8)	1.13 (0.14)	5.04 (0.78)	24.96 (1.81)	26.33 (9.86)
	0.51	0.70 (0.01)	37.16 (3.56)	4599 (593.0)	1.22 (0.08)	7.69 (2.39)	22.70 (2.19)	7.15 (3.02) a[g]
	0.81	0.70 (0.02)	34.39 (2.83)	4206 (268.9)	1.00 (0.03)	8.34 (1.61)	26.68 (1.80)	5.51 (1.82) b
	1.63	0.69 (0.02)	34.58 (2.52)	4544 (337.8)	1.05 (0.06)	7.60 (1.54)	27.81 (1.90)	5.06 (2.75) b
Average		0.69	35.25 b	4447 b	1.10 a	7.17 b	25.54 b	
Soy–PF	0.51	0.68 (0.02)	32.27 (1.47)	4592 (310.3)	0.90 (0.08)	11.40 (4.26)	28.64 (1.64)	4.58 (2.48) a
blend	0.81	0.67 (0.04)	32.61 (2.16)	4399 (626.4)	0.94 (0.08)	10.57 (4.92)	29.98 (3.17)	4.35 (2.37) ab
	1.63	0.68 (0.02)	32.30 (1.95)	4399 (510.2)	0.99 (0.03)	12.80 (2.14)	27.63 (2.08)	3.43 (1.65) b
Average		0.68	32.39 c	4463 b	0.94 b	11.59 a	28.75 b	
Soy/PF	0	0.71 (0.02)	36.30 (5.24)	4875 (303.4)	0.92 (0.10)	8.37 (1.88)	37.10 (7.43)	28.48 (10.83)
	0.51	0.69 (0.03)	36.00 (5.73)	4902 (855.0)	0.81 (0.08)	5.83 (1.50)	42.81 (11.80)	6.85 (3.55) a
	0.81	0.71 (0.02)	39.88 (4.51)	5185 (517.1)	0.91 (0.10)	6.49 (1.16)	39.88 (4.50)	2.76 (1.40) b
	1.63	0.70 (0.01)	42.65 (4.51)	5302 (482.6)	0.89 (0.08)	6.43 (1.28)	41.25 (7.90)	2.57 (1.58) c
Average		0.71	38.71 a	5066 a	0.88 c	6.78 b	40.26 a	

[a] Flakeboard made with juvenile hybrid popular flakes bonded with 7% resin solids and pressed to 1.27 mm in thickness at 200°C for 7 min.
[b] PF, commercial phenol–formaldehyde resin; Soy–PF blend, 85 w% commercial PF resin blended with 15 w% soy flour hydrolyzate; Soy/PF, 60% soy flour hydrolyzate crosslinked with 40 w% PF resin.
[c] Percent thickness swell after soaking samples in cold water for 24 h.
[d] Percent thickness swell after boiling samples in water for 2 h.
[e] Percent weight loss after exposure to brown-rot fungus *Postia placenta* for 12 wk.
[f] Numbers in parentheses are standard deviations.
[g] Means with different letters are significantly different at the 5% level.
MOR, modulus of rupture; MOE, modulus of elasticity; IB, internal bond strength; TS, thickness swell; WL, weight loss.

Table 2. Statistical analysis of factors influencing flakeboard physical properties and decay resistance.

Factors	MOR	MOE	IB	2-h boil TS	Decay resistance
			Pr>F		
Resins	<0.0001	<0.0001	<0.0001	<0.0001	0.0014
Preservatives	0.0503	0.6808	0.5937	0.8198	0.0004
Resins × preservatives	0.0223	0.2688	0.0001	0.4727	0.1498

MOR, modulus of rupture; MOE, modulus of elasticity; IB, internal bond strength; TS, thickness swell.

MOR ranged from 35.2 to 38.7 MPa, MOE from 4447 to 5066 MPa, and IB from 0.88 to 1.10 MPa. The test boards meet the minimum requirements of 29.0 MPa MOR and 0.345 MPa IB in the CSA standard (Canadian Standards Association 1993) for OSB, but the MOE is below the minimum requirement of 5500 MPa because flakes in test boards were randomly oriented. Statistical analysis showed that physical properties of aspen flakeboard were strongly influenced by adhesive resin types. Boards bonded with PF resin (resin 1) had the best physical properties followed by soy/PF blend resin (resin 2) and PF-crosslinked soy resin (resin 3). For example, IB of PF-bonded boards was 20% higher (1.10 MPa vs 0.88 Mpa) and 2-h-boiled TS was 36% lower (25.54% vs 40.26%) than boards bonded with PF-crosslinked soy resin (Figs 1 and 2). However, MOR and MOE of boards bonded with PF-crosslinked soy resin were greater than boards bonded with PF resin (Fig 3), which may be attributed to higher board density (0.71 g/c.c vs 0.69 g/mL). Statistical analysis also showed that flakeboard properties were not influenced by preservative treatments,

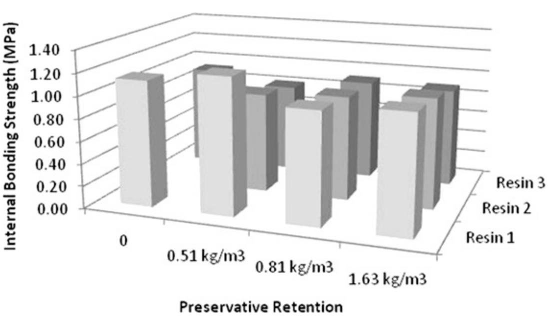

Figure 1. Effects of resin type and preservative retention on internal bonding strength (IB) of aspen flakeboard. (resin 1 = phenol–formaldehyde resin [PF]; resin 2 = PF [85]/ soy hydrolysate [15] blend resin; resin 3 = PF-crosslinked soy resin).

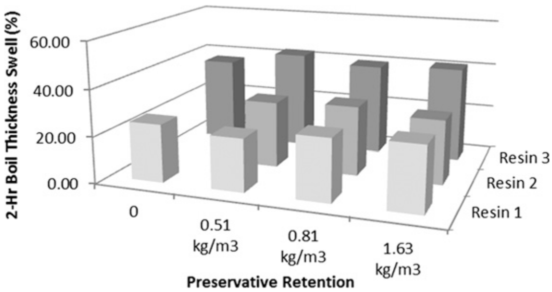

Figure 2. Effects of resin type and preservative retention on 2-h boil thickness swell of aspen flakeboard.

suggesting neither soy oil nor biocides interfered with adhesive bond strengths and curing of adhesive resins. Soy oil, like linseed oil and tung oil, is a drying oil, which does not contribute much vapor pressure during hot pressing of boards to cause internal blows.

Results of decay resistance test of all experimental flakeboards are presented in Table 1 and Fig 4. After exposing to brown-rot fungus *Postia placenta* for 12 wk, samples from boards bonded with PF resin and PF-crosslinked soy resin boards without biocides sustained 26.33% and 28.48% weight loss, respectively. Under the same brown-rot fungal exposure conditions, untreated poplar

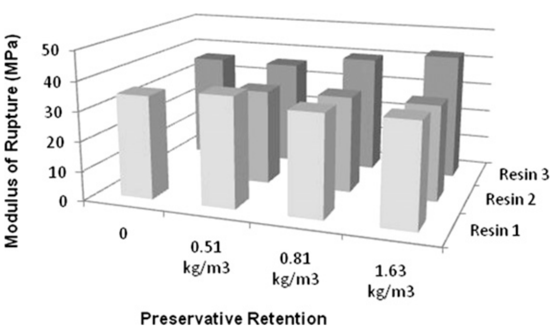

Figure 3. Effects of resin type and preservative retention on bending strength (MOR, modulus of rupture) of aspen flakeboard.

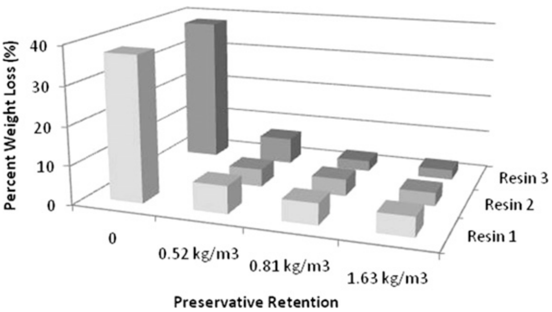

Figure 4. Effects of resin type and preservative retention on brown-rot decay resistance of aspen flakeboard.

solid wood normally would sustain more than 40% weight loss (Kuo et al 1988). Because of high density and due to resin protection of some cell wall materials, the untreated flakeboard samples were somewhat more decay-resistant than untreated aspen wood. In addition, pressing of flakeboard at 200°C also may increase decay resistance resulting from thermal degradation of hemicelluloses and lignin toxic to fungi (Hakkou et al 2006). The biocides evidently remained effective after hot pressing at 200°C for 7 min. Table 1 and Fig 4 also show that percent weight loss of flakeboard samples decreased with increasing biocide retention level. Boards bonded with PF resin containing 0.51, 0.81, and 1.63 kg/m^3 biocide sustained 7.15, 5.51, and 5.06% weight loss, respectively. The corresponding percent weight loss for boards bonded with PF/soy blend resin and PF-crosslinked soy resin were 4.58, 4.35, and 3.43% and 6.85, 2.76, and 2.57%. At the same biocide retention, there is a general trend that PF-bonded boards sustained more weight loss followed by boards bonded with PF/soy blend and PF-crosslinked soy resins. Variance analysis indicated that as single factors, both preservative retention level and resin type significantly affected decay resistance and that there also was significant interactions between resin type and preservative retention. The significant effect of resin type and its interaction with preservative retention on flakeboard decay resistance is believed to be attributed to total volume of each resin type for spraying. During resin application, there were more PF/soy blends and PF-crosslinked soy resins (5.2% and 17.4%, respectively) to spray than PF resin, which contributed to more uniform distribution of resin and biocide.

The precise threshold retention of biocide in flakeboard treatment was not investigated. At a loading level of 1.63 kg/m^3, the average weight loss of board samples bonded with PF, PF/soy blend, and PF-crosslinked soy resin was 5.06, 3.43, and 2.57%, respectively. Low weight losses indicate that flakeboard treated at 1.63 kg/m^3 biocide retention provided sufficient protection against brown-rot decay. The system of delivering biocide together with adhesive resin used in this study seems more effective than the dry powder systems reported by Schmidt and Gertjejansen (1988) and Laks and Palardy (1993). The wet delivery system used in this study may have the advantage of uniform distribution and mobility of the preservatives in flakeboard. Some biocides may penetrate into the cell walls during hot pressing and thus provide more effective protection against fungal decay.

CONCLUSIONS

In the production of treated flakeboard, it is most cost-effective to administer preservatives during the process of panel manufacturing. In-process treatment of flakeboard is challenging because of incompatibility between certain wood preservatives and adhesive resins. Deleterious preservative–resin interactions can be reduced or avoided by incorporating dry preservative into adhesive resin, but such dry systems limit preservative mobility to obtain full efficacy. In the present study, the wet delivery method in flakeboard making was investigated, in which biocides were dissolved in soy oil and blended with phenolic adhesive resins to manufacture aspen flakeboard. No precipitation or gelling was observed when blending phenolic resins with biocides dissolved in soy oil, and physical properties of flakeboard were not adversely affected by the preservative treatment. Brown-rot decay resistance of flakeboard increased with increasing retention of biocides and boards containing 1.63 kg/m^3 biocides sustained only less than 5% weight loss after exposing to brown-rot fungus *Postia placenta* for 12 wk. The method of preparing biocides for this study by evaporating the solvent (mineral spirit) from the commercial preservative is not an appropriate procedure for the production of treated flakeboard. However, this study demonstrated that the wet preservative delivery method for treating flakeboard obtained satisfactory results.

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