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Performance Characteristics of Borate Fatty Acid Formulations as Mold Inhibitors

Robert D. Coleman
Vina W. Yang
Carol A. Clausen

Abstract

The combination of boric acid (BA) or disodium octaborate tetrahydrate (DOT) and a fatty acid (FA) such as heptanoic, octanoic, and nonanoic acids (C7–C9) is an effective treatment solution for protecting wood structures against mold. BA or DOT alone have substantial potency against insects and decay fungi, but have negligible or no mold inhibitor activity. However, boric acid or DOT combined with a fatty acid either as a dip or vacuum treatment appears to act synergistically to inhibit mold growth. Southern Pine vacuum-treated with BA (or DOT)/FA appears to retain much or all of its inhibitor properties after extensive leaching with water. Boric acid/FA and DOT/FA formulations applied as dip solutions had similar efficacy against the test fungi, but greater efficacy as a mold inhibitor was achieved when DOT was formulated with C7, C8, or C9 FA and used as a vacuum treatment.

Keywords: fatty acid, boric acid, disodium octaborate tetrahydrate, mold fungi, fungicide

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Robert D. Coleman, President
Summerdale, Inc., Verona, Wisconsin

Vina W. Yang, Microbiologist
Carol A. Clausen, Supervisory Research Microbiologist
Forest Products Laboratory, Madison, Wisconsin

Introduction

Current insecticides and fungicides used to protect wood from biodeterioration are effective but may be toxic and unsuitable for use in modern homes, especially those sealed and insulated to reduce the exchange of hot or cold air from the outside. Another concern to human health in modern homes is exposure to mold (Quarles 2008). Spores from mold fungi can be particularly problematic, not only as human and animal allergens, but also because of their recalcitrance to chemical remediation. Among the three primary wood infestations (termite attack, mold, and decay fungi), spores from mold fungi appear to be the most resistant to chemical treatments; hence, mold spores are more difficult to suppress and control (Clausen and Yang 2004).

Earlier work revealed that aliphatic mono-carboxyl compounds such as fatty acids are active against a broad spectrum of fungal pathogens (Kabara and others 1972; Walters and others 2004). Lauric acid, a saturated C12 fatty acid (Kabara and others 1972), and linoleic and linolenic acids, unsaturated C18 fatty acids (Walters and others 2004), had antifungal properties.

Lower molecular weight aliphatic, saturated fatty acids, such as pentanoic, hexanoic, heptanoic, octanoic, nonanoic, and decanoic acids (C5–C10, respectively) have also been studied for control of numerous fungi (Coleman 2010a,b; 2011) including mold and decay fungi (Schmidt 1982; Coleman and Clausen 2009; Clausen and others 2010; Coleman and others 2010a,b). Formulations, at various application rates, have been tested with and without selected adjuvants such as organic acids (L-lactic and glycolic acid). Although exclusive use of organic acids as broad-spectrum biocides has not been effective for long-term control of mold and other fungi, their potential as synergists with certain fatty acids may result in the development of broad-spectrum, environmentally compatible fungicides.

This study evaluated the efficacy of multifunctional fatty acid/boric acid (or disodium octaborate tetrahydrate (DOT)) combinations against common mold species on wood. Antimicrobial formulations having multiple mechanisms of action with greater formulation potency and improved retention characteristics would be considerably more efficacious. The fatty acid/boric acid (or DOT) combination may provide cost effective and safe performance required by the construction industry and the public's environmental and health concerns. The research was conducted under a joint venture agreement between Summerdale, Inc., and Forest Products Laboratory (FPL).

Materials and Methods

Chemicals

Various combinations of the following chemicals were evaluated: Boric acid and DOT (U.S. Borax, Inc., RioTinto Minerals, Greenwood Village, CO, USA), octanoic acid, nonanoic acid and capric acid (Emery Oleochemicals, Cincinnati, OH, USA), hexanoic acid (Acros Organics, Geel, Belgium), heptanoic acid (Sigma-Aldrich, St. Louis, MO, USA), glycerol (U.S. Glycerin, Kalamazoo, MI, USA) , L (+) lactic acid (Purac FCC, Lincolnshire, IL, USA) , mineral oil (Fisher Scientific, Pittsburgh, PA, USA), and PE 1198LA, a phosphate ester emulsifier (Huntsman Chemical, The Woodlands, TX, USA).

Treatment

Kiln-dried Southern Pine (SP) specimens (7 by 20 mm cross section by 7 cm long) were soaked overnight in deionized (DI) water to elevate moisture content prior to dip or vacuum treatment. Groups of specimens were dip-treated for ~30 s in each chemical formulation and held overnight in a closed container according to the ASTM standard test method D4445-10 (ASTM 2010) or ASTM D3273-12 (ASTM 2012) prior to exposure to test fungi. Vacuum treatments were conducted according to a modification of AWPA T1-07 (2007). Briefly, specimens were submerged in treatment solution and held under vacuum (–172 kPa) for 70 min. Vacuum-treated specimens were air-dried and conditioned at 27° C and 70% RH to a constant weight before exposing them to test fungi.

Mold and Sapstain Fungi and Inoculum Preparation

Mold fungi, *Aspergillus niger* 2.242, *Penicillium chrysogenum* PH02, *Alternaria alternata,* and *Trichoderma atroviride* ATCC 20476, were grown on 2% malt agar and individual spore suspensions prepared by washing the surface of a 2-wk-old culture of each fungus with 10 mL

of sterile DI water according to ASTM standard D4445-10 (ASTM 2010). Spore suspensions were transferred to a spray bottle and diluted to 100 mL with DI water to yield approximately 10^7 spores mL^{-1}. The spray bottle was adjusted to deliver 1 mL inoculum/spray. The sapstain fungus, *Aureobasidium pullulans*, was grown on 2% potato dextrose agar and an individual spore suspension was prepared from a 2-wk-old culture according to ASTM D4445-10.

Chemical Leaching

Leaching procedures were similar to AWPA E11-06 standard method (AWPA 2009). After conditioning, treated specimens were placed into a 500-mL glass container, submerged in 100 mL of DI water, and subjected to mild agitation for a total of 14 days, and with a complete water change after 6 h, and at 1, 2, 4, 6, 8, 10, 12, and 14 days. Leached and unleached specimens were exposed to mold and sapstain testing.

Mold and Sapstain Test

Twelve random replicate treated specimens were arranged over four layers of blotting paper saturated with 30-mL DI water and a polyethylene mesh spacer in sterile disposable Petri dishes (150 by 25 mm) (B-D Falcon, Los Angeles, CA, USA). Untreated specimens dipped in DI water served as controls. Specimens were sprayed with 1 mL of individual mold or sapstain spore inoculum, sealed in polyethylene bags to prevent drying, and incubated at 27 °C and 70% RH for 8 to 12 wk.

Results

Certain fatty acids and boric acid (or DOT), in selected combinations, appear to be more effective as mold inhibitors than the fatty acid alone. Since mold inhibition of leached SP matched that of unleached SP, sufficient boric acid (DOT)/FA appeared to be retained in vacuum-treated, leached SP. The efficacy of DOT as a means to improve mold-inhibiting activity of an octanoic acid (C8) formulation is shown in Table 1. Specifically, DOT at two rates (0.25%, 2.00%, g/v) in aqueous solutions were compared to water alone as the carrier solution for the C8 formulation. Although both DOT carrier solutions alone (treatments 4, 5) had an absence of mold protection, DOT combined with the C8 formulation (treatments 2, 3) improved mold-inhibiting activity slightly over using water as the carrier.

Boric acid (0.25%, g/v) also appeared to be synergistic with fatty acids in dip treatments (Table 2). Boric acid alone (treatment 5) was ineffective as a mold inhibitor when compared to water control (treatment 6). However, when the boric acid solution was compared to water as the carrier solution and amended with either a C8 or C9 formulation, boric acid had increased efficacy against test fungi. This finding suggested that the increase in efficacy was synergistic rather than additive. Protection from mold growth after exposure to a C8- or C9-based treatment without boric acid was essentially equivalent. Fatty acid (C8/C10) mold-inhibiting formulations with and without DOT were compared at two application rates (3%, 12%, v/v). Dip treatments inclusive of DOT (treatments 3, 4) were more effective than treatments without DOT at both rates (Table 3).

Vacuum treatments of SP using fatty-acid-based boric acid formulations were effective at all three application rates (6%, 12%, and 18%, v/v). This was true for both C9 and C8/C10 where nearly complete control of mold growth was observed at 4, 8, and 12 wk (Table 4).

Mold test results for specimens that were vacuum-treated with fatty acids and boric acid (or DOT), followed by water leaching, suggested that certain fatty acid species were retained in SP and performed better as mold inhibitors than other species (Table 5). In particular, C8 (treatments 3, 8) and C9 (treatments 4, 9) were more effective. Overall, DOT-fatty acids were more efficacious than formulations containing boric acid. An absence of mold growth was evident at 4–12 wk for DOT/C7 – C9 (treatments 7–9).

Table 6 revealed inhibition of mold growth after dip applications with the same formulations and rate tested in vacuum treatments (Table 5). Overall dip treatments, including boric acid and DOT, had similar levels of mold inhibition. However, C7 formulations (treatments 2, 7) were more effective and resulted in control of mold growth through 12 wk. The other fatty acid species (C6, C8, C9, C8/C10) reduced mold by more than 90% at 4 and 8 wk; quite possibly, these fatty acid formulations could be reformulated to match the fungicide activity of C7.

To investigate fatty acid/borate inhibition of sapstain (Table 7), a C8/C10-based preparation was supplemented with DOT at various concentrations (2%, 4% and 6%, g/v). At 12%, v/v, all formulations (treatments 1–4) decreased growth of the sapstain fungus compared to the control (treatment 5). Formulations containing 4% and 6% DOT (treatments 3–4) completely inhibited sapstain at 12 wk.

Discussion and Conclusions

DOT is used worldwide for treating wood (Lloyd and Manning 1995). In the United States, Australia, and South Africa, borates such as DOT are commonly used for treating timber-framed constructions. This natural mineral has a record of human and aquatic safety, is very inexpensive, and does not corrode metal (Schultz and Nicholas 2003). DOT is relatively soluble in water and thus has good penetration of all major structural lumber species in the United States. Moreover, DOT treatments are compliant with major building codes and in general are noncorrosive to most metal nails, screws and fasteners.

Boric acid and DOT leaching can occur with exposure to water, thus restricting the bulk of current treatments of

boron compounds to indoor construction. As a result, treated lumber exposed to rainfall (leaching conditions) over time loses substantial amounts of boric acid or DOT, reducing its effectiveness as a preservative. Although DOT has demonstrated capability in combating insects and decay fungi (Drysdale 1994), it has very limited ability to inhibit mold (Barnes and others 1989). Five percent DOT was unable to substantially inhibit mold fungi (Clausen and Yang 2003). Current and past efforts to fix boric acid and DOT within the wood structure often result in a loss of activity.

The fatty acid/boric acid (DOT) combination, upon extensive leaching, was substantially retained in treated wood. Sufficient amounts of fatty acid/ boric acid (DOT), as active ingredients for control of wood mold, remain in treated wood after repeated and prolonged exposure to water. Leached wood containing the fatty acid/boric acid (or DOT) combination had substantially the same fungicidal activity as an unleached wood sample suggesting that the formulation is suitable for limited UC2 and UC3A exposure. Finally, the fatty acid/boric acid (DOT) combinations exhibited synergistic efficacy against test fungi.

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Table 1. Mold inhibition in SP dip-treated with disodium octaborate tetrahydrate and C8 fatty acid

Treatment group	Formulation	Concentration (%)	Carrier	Rating[a] (12 wk)	FPLSD[b] *P*@0.05
1	C8[c]	6.00	Water	0.83	a
2	C8	6.00	DOT (0.25%)	0.58	a
3	C8	6.00	DOT (0.25%)	0	a
4	DOT	0.25	Water	3.40	b
5	DOT	2.00	Water	3.33	b
6	Control	—	Water	3.00	b

[a]Average mold growth based on a rating system where 0 is no growth and 1, 2, 3, 4, and 5 represent 20%, 40%, 60%, 80%, and 100% growth, respectively.
[b]FPLSD: Fisher's protected least significant difference; same letters are not significant at $P < 0.05$.
[c]50% C8, 20% phosphate ester (PE) 1198, 10% L-lactic acid, 20% mineral oil.

Table 2. Mold inhibition on SP dip-treated with boric acid and C8 or C9 fatty acids

Treatment group	Formulation	Concentration (%)	Carrier	Rating[a] (12 wk)	FPLSD[b] *P*@0.05
1	C8[c]	6.00	Water	0.90	b
2	C8	6.00	Boric acid (0.25%)	0.33	b
3	C9[d]	6.00	Water	0.80	b
4	C9	6.00	Boric acid (0.25%)	0.33	b
5	Boric acid	0.25	Water	3.91	a
6	Control	—	Water	3.50	a

[a]Average mold growth based on a rating system where 0 is no growth and 1, 2, 3, 4, and 5 represent 20%, 40%, 60%, 80%, and 100% growth, respectively.
[b]FPLSD: Fisher's protected least significant difference; same letters are not significant at $P < 0.05$.
[c]50% C8, 20% phosphate ester (PE) 1198, 10% L-lactic acid, 20% mineral oil.
[d]50% C8, 20% PE 1198, 10% L-lactic acid, 20% mineral oil.

Table 3. Mold inhibition of SP dip-treated with C8/C10-based formulations with and without DOT

Treatment group	Aqueous formulation	Concentration (% v/v)	Rating[a] (12 wk)
1	C8/C10[b]	3.0	1.33
2	C8/C10	12.0	0.33
3	C8/C10 plus DOT[c]	3.0	0.58
4	C8/C10 plus DOT	12.0	0.25
5	Control, water	—	2.00

[a]Average mold growth based on a rating system where 0 is no growth and 1, 2, 3, 4, and 5 represent 20%, 40%, 60%, 80%, and 100% growth, respectively.
[b]50% C8/C10, 20% phosphate ester (PE) 1198LA, 30% mineral oil.
[c]50% C8/C10, 20% PE 1198LA, 10% mineral oil, 4% DOT, 16% glycerol.

Table 4. Mold inhibition of SP vacuum-treated with varying concentrations of C9 or C8/C10-boric acid formulations

Treatment group	Formulation	Concentration (% v/v)	Mold rating[b] 4 wk	8 wk	12 wk
1	C9[c]	6.0	0	0	0
2	C9	12.0	0	0	0
3	C9	18.0	0	0	0
4	C8/C10[d]	6.0	0	0	0
5	C8/C10	12.0	0	0	0.08
6	C8/C10	18.0	0	0	0
7	Control, water	—	3.00	3.75	3.92

[a]Vacuum treatment at –172 kPa for 70 min.
[b]Average mold growth based on a rating system where 0 is no growth and 1, 2, 3, 4, and 5 represent 20%, 40%, 60%, 80%, and 100% growth, respectively.
[c]50% C9, 20% phosphate ester (PE) 1198LA, 6% boric acid, 24% glycerol.
[d]50% C8/C10, 20% PE 1198LA, 6% boric acid, 24% glycerol.

Table 5. Fatty acids + boric acid (or DOT) as mold inhibitors on SP vacuum-treated[a] and then leached in water, replicate trials A and B

Treatment group	Aqueous formulation (18% v/v)	Mold rating[b] 4 wk Trial A[c]	Trial B[c]	Avg	8 wk Trial A	Trial B	Avg	12 wk Trial A	Trial B	Avg
1	50% C6[d]	0.83	0.16	0.48	3.00	0.66	1.83	3.66	2.60	3.13
2	50% C7[d]	0.16	0	0.08	0.50	0	0.25	2.33	0.16	1.25
3	50% C8[d]	0	0.16	0.08	0	0.16	0.08	1.00	1.00	1.00
4	50% C9[d]	0	0	0	0	0	0	0.16	0.50	0.33
5	50% C8/C10[d]	0.16	0	0.08	0.66	0.16	0.41	1.33	1.66	1.49
6	50% C6[e]	0.16	0	0.08	0.16	0	0.08	1.33	1.33	1.33
7	50% C7[e]	0	0	0	0	0	0	0	0	0
8	50% C8[e]	0	0	0	0	0	0	0	0	0
9	50% C9[e]	0	0	0	0	0	0	0	0	0
10	50% C8/C10[e]	0	0	0	0	0	0	0.32	0	0.16
11	Control, water	1.00	1.16	1.08	2.50	2.00	2.25	2.66	2.50	2.61

[a] Vacuum treatments at –172 kPa for 70 min.
[b] Average mold growth based on a rating system where 0 is no growth and 1, 2, 3, 4, and 5 represent 20%, 40%, 60%, 80%, and 100% growth, respectively.
[c] $n = 12$.
[d] Additional formulation components 20% phosphate ester (PE) 1198LA, 6% boric acid, 24% glycerol.
[e] Additional formulation components 20% phosphate ester (PE) 1198LA, 30% boric acid, 24% glycerol.

Table 6. Comparison of fatty acids combined with boric acid (or DOT) as mold inhibitor on dip-treated Southern Pine, replicate trials A and B

Treatment group	Aqueous formulation (18% v/v)	Mold rating[a]								
		4 wk			8 wk			12 wk		
		Trial A[b]	Trial B[b]	Avg	Trial A	Trial B	Avg	Trial A	Trial B	Avg
1	50% C6[c]	0.33	0	0.16	0.50	0	0.25	0.32	0	0.16
2	50% C7[c]	0	0	0	0	0	0	0	0	0
3	50% C8[c]	0.16	0	0.08	0.16	0	0.08	0.16	0	0.08
4	50% C9[c]	0.16	0	0.08	0.32	0	0.16	0.32	0.16	024
5	50% C8/C10 [c]	0.66	0	0.33	0.66	0	0.33	1.00	0	0.50
6	50% C6[d]	0	0.16	0.08	0	0.32	0.16	0	0.32	0.16
7	50% C7[d]	0	0	0	0	0.16	0.08	0	0	0
8	50% C8[d]	0.16	0	0.08	0.16	016	0.16	1.00	0	0.50
9	50% C9[d]	0.16	0	0.08	0.16	0.16	0.16	0.33	0.16	0.24
10	50% C68/C10[d]	0.16	0.16	0.16	0.32	0.32	0.32	0.66	0.83	0.75
11	Control, water	2.50	2.50	2.50	3.16	2.83	3.00	3.16	3.00	3.08

[a]Average mold growth based on a rating system where 0 is no growth and 1, 2, 3, 4, and 5 represent 20%, 40%, 60%, 80%, and 100% growth, respectively.
[b] $n = 12$.
[c] Additional formulation components: 20% phosphate ester (PE) 1198LA, 6% boric acid, 24% glycerol.
[d]Additional formulation components: 20% PE 1198LA, 6% DOT, 24% glycerol.

Table 7. Inhibition of *Aureobasidium pullulans* on SP dip-treated with C8/C10 fatty acid formulations with and without disodium octaborate tetrahydrate

Treatment group	Aqueous formulation	Concentration (% v/v)	Rating[a] (12 wk)
1	50% C8/C10, 20% phosphate ester (PE) 1198LA, 30% mineral oil	12	0.25
2	50% C8/C10, 20% PE 1198LA, 20% mineral oil, 2% DOT, 8% glycerol	12	0.16
3	50% C8/C10, 20% PE 1198LA, 10% mineral oil, 4% DOT, 16% glycerol	12	0
4	50% C8/C10, 20% PE 1198LA, 6% DOT, 24% glycerol	12	0
5	Control, water	—	2.25

[a]Average mold growth based on a rating system where 0 is no growth and 1, 2, 3, 4, and 5 represent 20%, 40%, 60%, 80%, and 100% growth, respectively.