Chapter 23

New Environmentally-Benign Concepts in Wood Protection: The Combination of Organic Biocides and Non-Biocidal Additives

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The principal wood preservative for residential use is currently a broad-spectrum biocide which contains As, Cr and Cu. However, the use of totally organic wood preservative systems in certain applications or localities may be required in the future. This will greatly increase the price of treated wood. The combination of organic biocides with non-biocidal additives to give enhanced efficacy is one possible means to lower the cost of future wood preservative systems. Possible non-biocidal additives which could be mixed with organic biocides include water repellents, antioxidants, and chelators for specific elements, including metals required by wood-decaying fungi such as iron or manganese. Selected laboratory and field exposure results obtained by combining organic biocides with one or more non-biocidal additives are presented in this chapter. Future wood preservative systems, either based on this chapter's concept or other possible techniques, will likely be used for relatively specific applications rather than today's one broad-spectrum preservative. This will require users of treated wood to be more educated in the future.

INTRODUCTION

Wood is a natural and renewable resource used extensively in home construction, decks, fences, utility poles, etc. Unfortunately, if sufficient moisture is present lumber, poles, and wood composites can be attacked by many organisms, principally brown-rot fungi and termites. In the U.S. alone the wood damaged yearly in residential structures by fungi and insects amounts to about $500,000,000 per year, and the labor involved further increases this to about $5 billion per year (Amburgey, Miss. State Univ., personal communication). Fortunately, wood can be treated with biocides to prevent damage caused by fungi or insects. Use of treated wood not only helps homeowners save money but also conserves our forests.

The major wood preservative used today in the U.S. is water-borne chromated copper arsenate (CCA). Based on wood volume, about 80% of all treated wood in the U.S. is preserved with CCA *(1)*, and CCA is -by far - the major preservative used to treat lumber for residential construction, the major market for treated wood. However, there are some concerns with CCA including a public perception of possible arsenic exposure, leaching of the metal oxides and the question of the ultimate disposal of CCA-treated wood. Thus, use of CCA-treated lumber will be restricted starting by 2004 to only non-residential applications. An alternative is borate-treated lumber *(2-4)* which is commercially available in Hawaii and provides cost-effective and benign protection in certain applications. However, borate use is limited to non-leaching applications.

Alternative, second generation wood preservatives for residential applications are copper-organic mixtures *(2)*, such as ammoniacal copper quat (ACQ) and copper azole mixtures, with or without boron (CBA, CA), and both ACQ and CA are commercially available. Although copper is not as toxic as arsenic or chromium accumulation of copper does have some negative environmental effects, especially in aquatic systems, and disposal of any metal-treated wood product may be expensive and difficult in the future. Thus, copper-organic mixtures may also face future restrictions in the U.S. Indeed, several countries in Europe are already moving towards totally organic wood preservative systems. Consequently, third-generation wood preservative(s) for residential use, based solely on non-metallic biocides *(2)* or a fixed borate system, need to be developed. A number of organic biocides are already commercially available as agrochemicals and have been examined as potential wood preservatives *(5)*. The relatively high cost of these organic biocides (mostly $15 - 25/lb versus $1.50/lb for CCA), environmental regulations, and public concerns with bioactive compounds will undoubtably result in efforts to minimize the amount of biocide used. In addition to effectively and economically protecting wood against a wide variety of wood decaying organisms, alternative preservative systems must have good weathering and UV protection properties, minimal leaching of the active compound, and be noncorrosive to metal fasteners. Finally, water-based emulsion formulations for the organic biocides (2) will likely need to be developed.

Two potential methods for reducing the level of a biocide, and thus the cost and environmental impact of treated wood, are to combine two or more biocides (fungicides and/or insecticides) to give a synergistic mixture [covered in Leightley's chp.], or to combine biocides and non-biocidal additives to give increased efficacy. This second option is the objective of this chapter. Specifically, we are examining non-biocidal additives which by themselves offer little or no protection but when combined with a biocide give wood greater and/or broader protection against fungi and/or termites than achieved with only the biocide. While many of the compounds discussed in this chapter are ineffective or require unreasonably high levels when used alone, when combined with a biocide the mixture may provide increased and/or broader efficacy and improved economics. A second advantage of this approach is that a synergistic mixture may be patentable.

NON-BIOCIDAL ADDITIVES EXAMINED

For fungi to survive and grow in wood they need to: 1) Be able to live in and colonize the woody substrate; 2) generate the enzymes and reagents necessary to initially disrupt and break down lignocellulose; 3) have free water in the lumen so that the enzymes and small reagents can diffuse from the fungal mycelium to the cell wall and then penetrate and partially degrade the lignocellulose; and, 4) digest the partially degraded wood components as a food source. For example, adding a fungicidal compound can prevent fungi from living in treated wood; this is the approach currently used to preserve wood.

Alternatively, non biocidal compounds might affect one or more of the above steps. For example, it may be possible to tie up essential minerals, elements, or compounds such as amino acids needed by fungi to colonize and grow in wood. Thus, one control method would be to prevent fungi from obtaining essential elements such as N, P, or Ca. Positive results have already been reported using only the calcium chelating compound Na-*N'*N-napthaloylhydroxamine (NHA, sodium salt) as a wood preserving agent *(6,7).* [Alternate mechanisms, beside calcium chelation, might also explain NHA's effectiveness against termites and fungi]. In another example, researchers have long examined methods to make wood more hydrophobic so that lumber used in an above-ground application such as decking would absorb less water. This approach includes the commercially available and relatively inexpensive wax emulsion formulations *(2,8).* An alternate but expensive approach is to add monomeric compounds to wood which are then polymerized *in situ* to make wood hydrophobic *(8)*. These compounds might also covalently bond with, and thus alter, the chemical structure of the wood components such that the fungal enzymes would no longer be capable of degrading the woody material, thus inhibiting both steps 3 and 4 above. While this approach has been extensively studied in the laboratory, in discussing this and other novel wood preservation approaches Suttie - in a British understatement - noted that "The scaling-up of laboratory techniques to the pilot plant is not without problems" *(8)*. Alternatively, to degrade wood fungi need to first disrupt and open up the cell wall. Fungi accomplish this by generating compounds which diffuse into the cell wall and generate reactive radicals which then disrupt the wood lignocellulose *(9-12)* [also see chps. by Aust, Enoki, Messner, and Goodell]. Consequently, the presence of free radical scavengers (antioxidants) in the cell wall would protect the cell wall from becoming more porous *(9,13)* [and may also help protect an organic biocide from being biodegraded]. An alternative and potentially elegant method would be to identify an additive which would disrupt the initial generation of the pre-radical oxidant in the acidic region of the fungal mycelium, and thus prevent the wood cell wall from being perturbed while possibly also causing the oxidant to form a radical near the fungus. Finally, metals such as Fe or Mn are well known to be involved in fungal degradation mechanisms, either as part of an enzymatic system or as a free metal. Thus, addition of appropriate metal chelators might prevent the metals from being available to the fungi *(8,9,14)*. Other mechanisms, besides chelating essential metals, might also explain the enhanced efficacy obtained when a biocide and metal chelator are combined, e.g. *(15)*.

In reviewing the above potential additives, it is worthwhile to examine the properties of the heartwood extractives in naturally durable woods. The fungicidal properties of the vast majority of extractives have been found to be mediocre - at best - when compared to commercial biocides *(16)*. However, the various phenolic extractives are well known to be excellent antioxidants *(17-19)*, and many of these phenolics also have metal chelating properties *(19)*. Thus, the combination of a biocide, antioxidant and/or metal chelator might simply mimic nature's approach to make wood durable. For example, the combination of various antioxidants and/or metal chelators increases the efficacy of a wide variety of organic biocides *(14,20)*. Furthermore, gallic acid derivatives, derived from the tannic acids found in heartwood, enhances the efficacy of the relatively expensive biocide propiconazole *(14)*. Finally, many extractives, such as the terpenoids, are hydrophobic. The high level of water-repelling rosin in SYP lightered wood makes this wood extremely durable, even in ground contact *(21)*. Adding extremely high levels of wax alone (about 26 pcf) to ground contact stakes mimics this and increases their average life to about 19 years as compared to less than 3 years for untreated stakes *(22)*. While adding such high wax levels is unrealistic, we believe that the co-addition of smaller amounts of a wax along with a biocide and other possible additives might improve the biocide's efficacy so that relatively low biocide levels could protect wood. Indeed, the combination of a biocide and water repellent is already used in window joinery.

While less is known about the mechanisms by which termites and their symbiotic microorganisms degrade the holocellulose in wood, many of the above additives may also control termites. For example, the heartwood of naturally decay-resistant woods are also usually resistant to termites and, thus, the combination of biocides, antioxidants and/or metal chelators may be successful in protecting wood against both termites and fungi. SYP lightered wood, such as found in old pine stumps, has both decay and termite resistance, and the stakes with very high levels of wax described above were also resistant to termites even though paraffin wax has no termiticidal properties.

LABORATORY AND FIELD TEST RESULTS

Initial Considerations

The development of wood preservative systems requires considerable time and expense. This is especially true with systems comprising two or more components, where the number of tests to be run is much greater than for single component systems. While the most realistic test of a system's efficacy is an outdoor exposure study, the extensive time required [years] and the multitude of potential combinations makes initial screening using outdoor testing unpractical. Thus, initial evaluation requires selection of an appropriate laboratory decay method to rapidly test the many different blends **and greatly reduce the number of the potential systems - hopefully without "tossing the baby out with the bath water".** That is, initial testing should be conducted quickly and provide data which explicitly separates promising from inferior systems.

The selection of an appropriate laboratory test(s) is more difficult than generally realized. The most common laboratory decay test used in wood preservation studies in the U.S. is the AWPA E10 12-week incubation soil block test, but this test may not necessarily be the optimal screening assay and also takes 16 or more weeks in total to perform. For example, when developing a new system for above-ground use the presence of minerals in the soil media may overwhelm certain additives and give a negative result which would not necessarily be observed during actual outdoor tests, as shown below. Another example is water repellents; minimizing the amount of water adsorbed by wood will obviously reduce the decay potential. However, wood blocks are typically first steam sterilized and, therefore, already have sufficient moisture for decay to occur at the start of the laboratory soil-block test. Finally, the soil-block test is unrealistically harsh and is initiated by actively growing fungal mycelium. In contrast, outdoor above-ground tests are initiated by delicate spores. Other bioactivity laboratory tests include the fungus cellar *(23,24)*, agar-block and agar-plate tests (25); each test has particular advantages and limitations. In this chapter we describe results obtained using soil- and agar-block tests using compression strength loss to measure extent of fungal degradation after 5-6 weeks of incubation.

Biocide/Antioxidant/Metal Chelator Combinations

As discussed above, heartwood extractives have biocidal, antioxidant and metal chelating properties, all of which may influence natural durability. Consequently, we have combined various commercial organic biocides with different antioxidants and/or metal chelators *(13,14,20)*. In laboratory tests, an antioxidant or metal chelator alone often had little or no protective effect *(12,26)* but when combined with a biocide enhanced or synergistic efficacy can be observed. For example, the antioxidant propyl gallate at 2% levels provided no protection to southern yellow pine (SYP) exposed to the brown-rot fungus Gloeophyllum trabeum for 5 weeks in the soil-block test and, similarly, no protection is observed with 3% of the metal chelator EDTA (Figure 1). [The concentrations given in this and other experiments are the active ingredients (% a.i.) used to treat the wood by a full-cell process for laboratory or outdoor exposure tests]. The biocide propiconazole, at treating solution concentrations up to 0.12% a.i., provided essentially no protection, and no benefit was observed when the same treating concentrations of propiconazole were combined with either 2% propyl galllate or 3% EDTA. However, when all three components are present increased efficacy at all four biocide levels is readily apparent. The results obtained here and previously with relatively low levels of propyl gallate *(14)*, compared to the higher levels of other antioxidants used such as 4 to 5% butylated hydroxytoluene (BHT), is intriguing. It is possible that the good results in Table 1 are due to the dual antioxidant and metal chelating properties of gallate derivatives (14); other mechanisms might also account for the promising results.

We mentioned above that the particular laboratory test used can affect the results and thus the conclusions. For example, when aspen sapwood is exposed to the white-rot fungus Trametes versicolor for 6 weeks in the agar-block test, 3% EDTA alone was surprisingly effective (1 00% strength loss for untreated samples vs. only 5.4% loss for aspen samples treated with 3% EDTA). However, in the soil-block test EDTA alone offers little protection (Fig. 1). Other researchers have also reported that a metal chelator alone offers little protection to wood when a soil media test is used but is very effective in an agar media test *(14,26)*. Presumably, the large amounts of minerals in a soil-containing test can quickly overwhelm and inactivate a metal chelator, while in agar media (which has relatively low mineral levels) a metal chelator alone may be quite effective at protecting wood. Similarly, when testing antioxidants better results are often obtained with an agar-block than soil-block test, perhaps because of the different levels of radicals produced *(13)* with the different substrates *(27)*. Finally, the substrate used also has an effect. For example, a fungicide's effect on protecting cotton cellulose against a fungus is not comparable with the results obtained using SYP wood *(28)*.

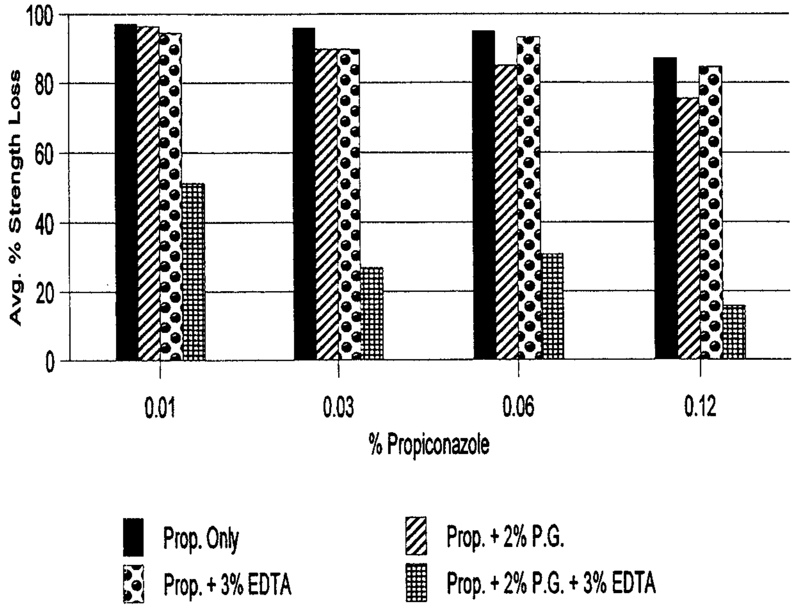

Figure 1. Efficacy of a biocide (propiconazole), antioxidant (propyl gallate, P.G.), and metal chelator (EDTA) systems. The soil block test was run using southern yellow pine sapwood with the brown-rot fungus G. trabeum and 5 weeks of incubation. The average strength loss with only 2% propyl gallate was 93.4%, 3% EDTA alone had 98.5% strength loss and untreated controls had an average of 98 and 96% strength loss.

The combination of the antioxidant BHT with the commercial biocide chlorothalonil also gave enhanced efficacy in ground-contact field tests conducted at two sites. [The Dorman Lake plot is located in northeast MS near Mississippi State Univ., has a heavy clay, poorly-drained soil and is in a high (Zone 4) deterioration zone. The Saucier test plot is located in the Harrison National Forest near the town of Saucier, MS, and has a sandy loam, well-drained soil and is in a severe (Zone 5) deterioration zone. Since this site is near the Gulf Coast it has a relatively mild winter and wet summer as compared to the Dorman Lake site]. For example, better protection was observed against both fungi and termites when BHT was combined with the chlorothalonil as compared to chlorothalonil alone (Table 1). It is interesting that 4% BHT alone provided some protection to wood in outdoor exposure as compared to untreated controls, even after 33 months of exposure. By contrast, in the soil block test 5% BHT alone gave no protection after four weeks of incubation (13). Thus, laboratory decay tests are unrealistically harsh but data is obtained within weeks as compared to the years needed for outdoor exposure tests. We use laboratory decay tests to quickly determine if a particular combination is synergistic, and outdoor exposure to judge the efficacy of a particular system under "real life" conditions.

'Table 1. Average decay and termite ratings for SYP field stakes treated with chlorothalonil (CTN) alone, or a mixture of CTN and BHT, after 33 months of exposure at two field test sites.

Treatment	Average Retention, kg m^{-3}	Dorman Lake		Saucier	
		Decay	Termite	Decay	Termite
0.15% CTN	0.74	7.4	7.2	7.4	7.9
0.15% CTN/2%BHT	0.72/9.5	9.8	9.8	9.7	7.3
0.15% CTN/4%BHT	0.70/18.8	9.8	9.9	9.5	8.4
0.30% CTN	1.47	8.8	8.9	8.4	7.8
0.30% CTN/2%BHT	1.54/10.1	9.9	9.9	9.9	9.6
0.30% CTN/4%BHT	1.41/18.8	10	9.9	9.4	8.7
0.50% CTN	2.42	10	10	8.0	8.0
0.50% CTN/2%BHT	2.41/9.6	10	10	10	9.9
0.50% CTN/4%BHT	2.43/19.5	10	10	10	9.9
4% BHT	19.4	9.4	9.4	4.9	5.5
Controls	–	0.7	0.4	0	0

Average of 10 stakes per treatment per site. A "10" rating is no attack, "9" trace of attack, etc., as per AWPA Standard E7-93.

Water Repellents

The addition of a water repellent with a biocide not only improves the decay resistance of wood in above-ground applications (Table 2), including samples treated with only a water repellent, but also greatly improves the weathering and dimensional stability of exposed lumber *(2)*. Furthermore, paraffinic waxes may be the most cost-effective additive for improving durability (A. Preston, CSI, personal communication) and are environmentally benign. Consequently, various companies already have commercialized water-repellent systems for applications such as CCA-treated decking, and formulations for ground-contact applications may be available in the future. Other researchers *(29)* have also examined the combination of a water repellent and metal chelator to protect wood against decay and mold fungi.

Table 2. Average decay ratings of above-ground L-joint samples, with and without a co-added water repellant, after four years of exposure at CSI's Hilo, HI test site.

Treatment, % a.i. (DDAC:Na Omadine)	Average Decay Rating[a]	
	without water repellent	with water repellent[b]
0.1 : 0.02	3.4	7.7
0.2 : 0.05	4.7	6.6
0.4 : 0.1	4.9	9.1
0.6 : 0.15	8.6	9.9
0.8 : 0.2	9.5	10.0
Controls	5.3	7.7[c]

[a] Average of 10 replicates, with a "10" being no attack, etc.

[b] Water repellent was 5% palmitic acid, 3% butyl amine, and 3% butyl carbitol dissolved in water, with the biocides co-added.

[c] These control samples were treated with only the water repellent.

NHA Systems

The polycyclic organic compound NHA as its sodium salt chelates calcium, and has been examined as a possible stand-alone wood preservative *(6,7,26).* [As mentioned earlier, however, NHA might protect wood by other mechanisms]. When used at a level below the threshold [~0.5% a.i.], the protonated form of NHA alone provides no protection against the brown-rot fungus *G. trabeum* in the soil-block test (Fig. 2), or the white-rot fungus *Irpex lacteus* in the agar-block test (data not shown). When low levels of NHA are combined with low levels of the biocides DDAC, propiconazole, or 3-iodo-2-propynyl butyl carbamate (IPBC), synergism is clearly evident with IPBC against *G. trabeum* (Fig. 2) and synergism is likely against *I. lacteus.* However, no synergism was observed when NHA was combined with DDAC or propiconazole in these wood decay tests.

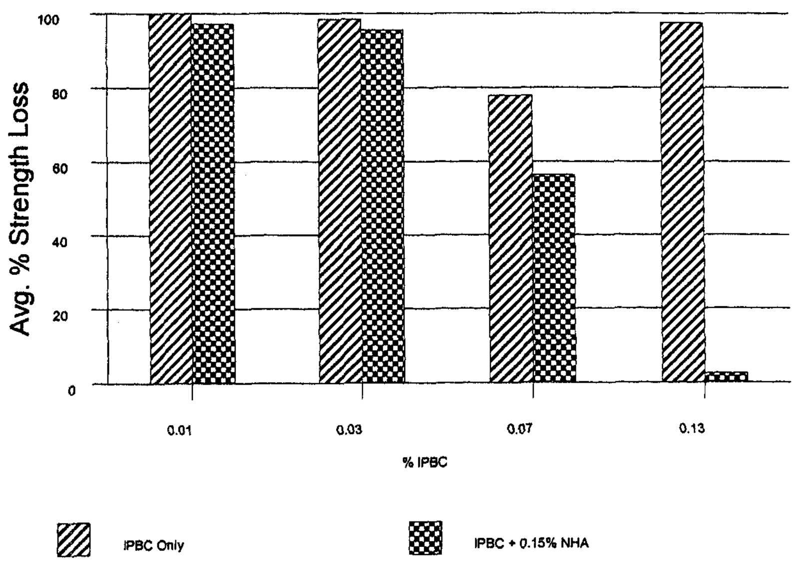

Figure 2. Synergism between IPBC and NHA, in the soil-block test using the brown-rot fungus G. trabeum for 5 weeks of incubation and SYP sapwood, with 5 replicates per set. The untreated controls lost an average of 97.9 and 97.6% strength, 0.15% NHA alone had a 97.5% loss, and the sodium salt form of NHA had 94.4 and 97.8% average strength loss at 0.15 and 0.30% concentration, respectively.

A preliminary synergism test against termites was also examined using NHA combined with DDAC or disodium octaborate tetrahydrate. However, no synergism was observed with either biocide.

CONCLUDING REMARKS

Future wood preservatives for certain applications or localities may be required to use non-metallic (organic) biocides, which will greatly increase the cost of treated wood. The combination of one or more organic biocide(s) with various non-biocidal additives might be one method to reduce the cost. Possible additives might include water repellants, free-radical scavengers (antioxidants), and chelators for specific metals required by the wood-destroying fungus and/or termites. Development of future totally organic wood preservative systems, based on ideas from this chapter or other techniques, will require extensive time and effort. It is extremely unlikely that under harsh conditions these systems will prove to be as effective as CCA in protecting wood. Furthermore, these future systems may be limited to specific applications, such as above-ground-use only. Thus, consumers will need to be aware of any limitations inherent in the forthcoming non-metallic targeted wood preservatives.

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