

CONTROL OF BROWN STAIN IN SUGAR PINE WITH ENVIRONMENTALLY ACCEPTABLE CHEMICALS¹

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ABSTRACT

Because of the hazards in using sodium azide for controlling brown stain, a less hazardous chemical was sought. Phosphoric acid was found to be the most successful treatment of the chemicals screened. A sufficient concentration of an iron chelating agent, in conjunction with lowered pH, resulted in a reduction in brown stain. Antioxidants were found to be ineffective.

Keywords: Kiln brown stain, chemical control, sodium azide, phosphoric acid, chelating agents, antioxidants, *Pinus lambertiana*.

INTRODUCTION

The staining of lumber has been recognized as a problem for at least a hundred years. Active study of the causes of staining—chemical and microbiological—had to wait until “ferments” were recognized, the action of enzymes understood, and fungi were identified in the stained lumber. Bailey (1910) discussed the discoloration of sapwood and sap stain. He cited blue stain caused by fungi and the yellow- to rust-colored stain caused by oxidation. He stated that hot, humid, summer weather promoted the latter stain, which developed in the wood rays and parenchyma cells. He demonstrated that an oxidizing enzyme was present by testing with guaiacum, and then he inhibited the enzyme’s action and the appearance of brown stain by placing the boards in boiling water.

Hubert (1926) recognized three types of stain: “brown sap-stain” found in the sapwood of logs, which did not progress during seasoning; “yard brown stain” found in the sap and heartwood, which developed during yard seasoning; and “kiln brown stain” found in sapwood, which developed during kiln-drying. He considered the latter to result from the translocation of materials from the interior of a board which then concentrated at the point of evaporation of water at the surface. Stout (1950) indicated that the most critical time of stain development was during the solid piling before drying. Millett (1952) surveyed and summarized the previous studies. Modification of the kiln schedule was used as a means of stain control; however, this required special handling, lengthened drying time, and did not always prevent brown stain.

Brown stain was best controlled by treating sugar pine and eastern white pine with a buffered sodium azide dip (Stutz 1959; Stutz et al. 1961). The staining process was found to be a result of an enzymatic reaction involving the action of

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peroxidases on tannins and phlobaphenes and dependent upon the moisture content of the wood, the length of time it is exposed to air and the temperature (Millett 1952; Stutz 1959). To reduce the cost of treatment and also the health hazard involved with sodium azide, sodium fluoride was successfully used as a dip in the prevention of brown stain in eastern white pine (Cech 1966; Catterick and Gillies 1966). However, this treatment required rubber gloves and washing facilities for the workmen in addition to careful ventilation to prevent hay fever-like effects (Catterick and Gillies 1966).

Sodium azide is hazardous to workers. Although it has been used medically to reduce blood pressure, excessive doses may cause profound hypotension (Stecher 1960). Stutz (1959) considered it a toxic reagent and stressed the use of rubber gloves, adequate ventilation, and caution. Cech (1966) found the buffered sodium azide solution expensive, highly toxic, and very corrosive.

Any method of inhibiting reactions that involve enzymes, as the brown staining of sugar pine does, is potentially hazardous to man because of possible interrelationship with the many enzyme systems involved in human life. The enzyme peroxidase, which has been implicated in brown stain formation (Millett 1952; Stutz 1959), is inhibited by hydrogen cyanide, hydrogen sulfide, dithionite, and sodium azide, all of which combine with the ferric iron of the hematin group (Bonner 1950) and are hazardous to humans. Peroxidase, found in plants, leucocytes, and milk, belongs to the hydroperoxidases, a group of hemiproteins that includes catalase (Mahler and Cordes 1966). Catalase is found widely distributed in human tissues such as liver and red blood cells and in various microorganisms (Neilands and Stumpf 1955). Both enzymes are inhibited by the same chemicals. Therefore, many inhibitors of peroxidase are too toxic to man and the environment to be used safely.

Recently, fairly good control of brown stain was reported using ammoniacal zinc oxide (Shields et al. 1973) and a variety of alkaline salts (Hulme 1975) as replacements for sodium azide or sodium fluoride. The present research reports results of experiments on additional approaches to brown stain control involving substances considered far less dangerous to humans and the environment than sodium azide.

Brown stain is considered to be a two-step chemical process initiated by an enzymatic reaction involving peroxidase during the solid stacking of the lumber. In the second step, the product of enzyme action is oxidized or polymerized during drying to produce the brown stain found throughout the boards (Millett 1952; Stutz 1959). We hypothesized that these reactions should be controllable with either an antioxidant, an antiperoxidant, or any treatment able to denature the enzymes involved. The criteria used in selecting chemicals and treatments were as follows: 1) soluble or emulsifiable in water, 2) an acid or basic pH, 3) nonstaining in the concentration used, 4) stable in the treatment tank and on the treated boards up to 21 days, 5) not promoting microbiological growth, 6) not a human health hazard in the concentrations used or by cumulation, 7) not inactivated by iron, and 8) nonflammable. The additional criterion of ability to chelate ferric ions was considered desirable but not a necessary trait of every treatment.

We began with a list of antioxidants found in the *Encyclopedia of Chemical Technology* (Standen 1963). Many antioxidants are classified as chemical preservatives and come under the jurisdiction of the Federal Food, Drug, and Cosmetic

Act when they are present in food carried in interstate commerce. Such classification was considered documentation that they did not constitute a health hazard. Their characteristics and common names were obtained from the *Merck Index* (Stecher 1960). Many antioxidants proved to be insoluble in water and had to be used with emulsifying agents.

Brown stain might also be controlled by inactivating the peroxidase. Because this enzyme has a heme group as part of its structure (Spector 1956), iron chelating agents were tried. The sawing process itself supplies trace quantities of iron through erosion and corrosion of the blades, which might affect the treatment chemicals, therefore providing another reason for adding chelating agents to the treatments.

Certain acids, such as citric and ascorbic, are naturally occurring substances capable of chelating some metals, such as copper and iron, which play a catalytic role in autoxidation. Ascorbic acid is frequently recommended as a treatment whenever a brown stain develops in plant matter. However, ascorbic acid, while it does prevent the browning of fruit, chelates copper not iron, and might promote the growth of fungi and thus was not considered further. Citric acid chelates iron and was included in the study despite the possibility that it might serve as a carbon source for stain fungi (Cochrane 1958). Phosphoric acid was selected for its chelating ability. LABTONE,² HEAMO-SOL, and trisodium phosphate were used as treatments because of their ability to digest protein (the enzyme peroxidase) and to thoroughly wet and wash the surface of wood. None of the above treatments was considered as hazardous as sodium azide in the concentrations used. During the three experiments, thirteen chemicals were surveyed, and process parameters were examined, such as treatment chemical, the amount of time between sawing and treatment, and the concentration of the treatment chemical, for a total of twenty-five treatment combinations.

The storage conditions after the treatments and before drying (the period where the peroxidases would be expected to initiate the staining reaction) were deliberately made as severe as possible to insure maximum testing of the treatments.

EXPERIMENT 1

Methods

Two, 16 ft, 5/4, Shop and Better grade, random width, untreated, sugar pine (*Pinus lambertiana* Dougl.) sapwood boards were obtained for preliminary testing and procedure development. They were cut from fresh logs (log age 3 months or less) and from different logs. During transportation to the laboratory, they were wrapped in plastic to prevent drying. Each board was cut into sixteen 1-ft specimens, numbered always on the same end so that the length of the board could be reconstructed, and end-coated. Because of the distance from the mill, the sectioning, end-coating, and treatment were not begun until 24 h after milling and were completed 24 h later. The pH of specimen surfaces was determined, just after sectioning, using HYDRION PENCILS³ and a HELDIGE-TRUOG RE-

² Trade names and registered trademarks are indicated by capital letters. Such names are provided for identification and clarity only and no specific recommendation or exclusion of competing products is intended.

³ Arthur H. Thomas Co., Philadelphia, PA 19105.

TABLE 1. Degree of brown stain in Experiment 1.¹

Treatment	Rating ²
1. Control ³	++
2. HAEMO-SOL (3%)	-
3. LABTONE (1%)	+
4. LABTONE (5%)	0
5. trisodium phosphate (1%)	+
6. phosphoric acid (2%)	+
7. phosphoric acid (4%)	0
8. citric acid (3%)	0

¹ Combined ratings of boards from both storage times after kiln-drying.

² - Discolored but not brown stain.

0 None—no stain on unsurfaced wood.

± Very light—stain removed by planing.

+ Light—stain not removed by planing.

++ Moderate—dark brown stain on less than half of the planed surface.

+++ Heavy—dark chocolate brown stain covering most of planed surface.

³ Not dipped.

ACTION KIT.⁴ Tap water was used in preparing all of the treating solutions. Each specimen was submerged and agitated in the treatment bath, in a plastic pan, for one minute. The treatments are listed in Table 1. Four specimens, two from each board, were used for each treatment. The specimens were then solid-piled by treatment and stored in a closed shed at an average temperature of 60 F. All of the specimens from one board were removed from storage after seven days, stickered and kiln-dried to a 10% moisture content. The kiln schedules used were those of Arganbright's study of drying characteristics and typical of commercial California practice (Arganbright 1972). During a 120-h schedule, the dry bulb temperature rose from 125 to 170 F and the wet bulb from 110 to 120 F. This was followed by an 8-h conditioning period during which the wet bulb was set to 180 F while the heating lines were closed. Finally, the specimens were visually rated for brown stain. This process was repeated on the specimens from the second board after 14 days of solid-piled storage.

Results

The results of Experiment 1 are given in Table 1. The pH of the control specimens right after sectioning was between 4.5 and 5.0. The specimens had very little stain after storage and developed only light to moderate stain after kiln drying. No blue stain occurred. Trisodium phosphate did not protect as well as the 5% concentration of LABTONE and was dropped from testing. Results of the HEAMO-SOL and LABTONE were so similar that only LABTONE was continued. Citric acid, LABTONE, and phosphoric acid at its higher concentration, were selected to be repeated in Experiment 2. Further testing would be conducted on heartwood or heart-sap boundary boards as these were found to exhibit the heaviest brown stain (Arganbright 1972). It was also decided to increase the severity of the storage conditions for Experiment 2, as the storage temperature was considered to be too low, and to shorten the length of time between milling and treatment.

⁴ The Ben Meadows Co., Atlanta, GA 30366.

EXPERIMENT 2

Methods

Four, untreated, 16-ft, 5/4, Shop and Better grade, random width, sugar pine boards containing the heart-sap boundary were used to test the effectiveness of antioxidants, chelating agents, and the effective treatments from Experiment 1. Phosphoric acid, Gardian Chemical's solubilizers "M" and "G," and sodium hydroxide were used to keep some of the compounds in solution. The treatments in which they were used are as follows: Gardian Chemical's VOIDOX—1% with solubilizer "M" (at 2.5% concentration) (pH 8); VOIDOX—1% with solubilizer "G" (at 2.5% concentration) (pH 6); 8-hydroxyquinoline—3% with 0.2% phosphoric acid; Shell's IONOL CP-40 (a registered trade name for BHT [butylated hydroxytoluene])—1% with solubilizer "G" (at 2.5% concentration); and Goodrich's CARBOSET 514—5% with enough 1N sodium hydroxide to keep the solution at pH 8. Solutions prepared at higher temperatures were used at room temperature. The procedure was the same as in Experiment 1 with the following exceptions: 1) the boards were end-coated as soon as they were cut into lengths; 2) the eight controls (two from each board) were dipped into tap water for one minute to stimulate treatment; 3) 12 treatments were run within 30 h of milling and three were duplicated 24 h later for a total of 15 treatments; 4) specimens from the different treatments were separated by plastic and then all were completely wrapped in plastic and placed in an environment of controlled humidity and temperature (91% and 80 F); 5) the three later treatments (Table 2, numbers 13, 14 and 15) were also wrapped in plastic and then placed in a closed shed for 14 days; 6) the first 12 treatments were inspected for stain and mold after 7 days; 7) all specimens remained stacked for 14 days before kiln-drying (see Experiment 1, Methods); 8) after drying and conditioning, the worst face was chosen and $\frac{1}{32}$ of an inch was removed from the surface of half the length of each specimen with a jointer; and 9) the smoothed surfaces were rated for brown stain. The treatments used are listed in Table 2.

Results

The pH of the untreated specimens, as determined by the HELLIGE-TROUG REACTION KIT, was 5.0 in the heartwood and 6.0 in the sapwood. The eleven treatments and the controls that were stored in the humidity chamber (Treatments 1–12) were inspected after seven days (Table 2). There was a heavy growth of mold on the sapwood of five out of the eight controls. The specimens treated with citric acid and the resin CARBOSET 514 also had heavy mold growth on the sapwood. The chelating agents, 8-hydroxyquinoline and 8-hydroxyquinoline sulfate, had heavy iron staining but did not mold. The treatments, TDPA, VOIDOX—pH 6, phosphoric acid, and LABTONE, had only a slight amount of mold, while VOIDOX—pH 8 and propyl 3,4,5-trihydroxybenzoate (propyl gallate) were clear and bright. There was no evidence then of brown stain. Treatments 13, 14 and 15 were not inspected at this time. Inspection after 14 days of storage noted no further change in any of the specimens.

The kiln-drying and conditioning resulted in the development of brown stain of varied severity in all specimens (Table 2). Blue stain was found associated with heavier brown stain. The treatments that protected against brown stain also

TABLE 2. Degree of staining in Experiment 2.

Treatment	Discoloration present before kiln-drying			Brown stain rating ¹ after kiln-drying
	Blue stain ²	Mold ²	Chemical	
1. Control ³	X	X		+++
2. LABTONE (5%)		slight		++
3. Phosphoric acid (4%)		slight		++
4. Citric acid (3%)	X	X		+++
5. 8-hydroxyquinoline (3%)			iron	±
6. 8-hydroxyquinoline sulfate (3%)			iron	±
7. VOIDOX (1%-pH 8)				++
8. VOIDOX (1%-pH 6)		slight		++
9. Propyl 3,4,5-trihydroxybenzoate (3%) (propyl gallate)			lt. green	+++
10. 3,3'-thiodipropionic acid (3.7%) (TDPA)	X	slight		++
11. Butylated hydroxytoluene (1%) (BHT)				++
12. CARBOSET (5%)				++
13. Repeat of #10 ⁴		X		++
14. Repeat of #6 ⁴				++
15. Repeat of #2 ⁴				++

¹ - Discolored but not brown stain.

0 None—no stain on unsurfaced wood.

± Very light—stain removed by planing.

+ Light—stain not removed by planing.

++ Moderate—dark brown stain on less than half of the planed surface.

+++ Heavy—dark chocolate brown stain covering most of planed surface.

² X = present after storage and before drying, amount not rated.

³ Dipped in tap water.

⁴ Treated 24 hours later.

excluded blue stain. The chelating agents, 8-hydroxyquinoline and 8-hydroxyquinoline sulfate, of treatments 5 and 6 gave the best protection. Treatment 14, 8-hydroxyquinoline sulfate, while using the same chemical as 6, was sensitive to the length of time before treatment; the storage conditions were not as severe but the additional time before treatment resulted in considerably more brown stain.

EXPERIMENT 3

Methods

The boards used were similar in grade and dimension to those used in the prior experiment except that they were 6/4 rather than 5/4. The most promising treatments from Experiment 2 were repeated in Experiment 3 together with one additional antioxidant. The concentrations of the previous treatments were increased and the storage conditions made as severe as possible. The previous treatment time of one minute was used for all except treatment 6, where it was lengthened to 2½ min. The length of time from milling to treatment was shortened to between 10 and 12 h. Aluminum flake was added to marine varnish used as the end coating. The specimens were wrapped in plastic by treatment and then as a whole, as in Experiment 2, and placed in a controlled environment of 80 F and 91% relative humidity for 14 days without inspection. At the end of this period, the specimens were removed, unwrapped, and rated for stains. They were then

TABLE 3. Degree of staining in Experiment 3.

Treatment	Brown stain rating	Other chemical discoloration present after kiln-drying
1. Control ²	+++	
2. LABTONE (10%)	+++	
3. Phosphoric acid (10%)	0	
4. 8-hydroxyquinoline (6%)	±	blue-black
5. 8-hydroxyquinoline sulfate (6%)	±	blue-black
6. 8-hydroxyquinoline (6%) ³	±	blue-black
7. Butylated hydroxytoluene (10%)	+++	
8. Ethoxyquin (3.3%)	+++	lt. orange

¹ - Discolored but not brown stain.

0 None—no stain on unsurfaced wood.

± Very light—stain removed by planing.

+ Light—stain not removed by planing.

++ Moderate—dark brown stain on less than half of the planed surface.

+++ Heavy—dark chocolate brown stain covering most of planed surface.

² Dipped in tap water.

³ Dipped for 2½ minutes.

stickered, kiln-dried, conditioned, surfaced on the jointer, and rated as in Experiment 2.

The treatments are given in Table 3. The solubility of Treatment 4 was improved with 1.0% phosphoric acid. Treatment 8 was Monsanto's SANTOQUIN, the antioxidant ethoxyquin. None of the treatments discolored the specimens after dipping even though the BHT solution was a milky white and the ETHOXYQUIN solution an opaque brown.

Results

The pH of the untreated specimens as determined by the HELLIGE-TROUG REACTION KIT was 4, and by the HYDRION PENCILS 3–4. After 14 days of solid-piled storage, the condition of the specimens was as follows: 1) there was no mold or blue stain; 2) LABTONE, phosphoric acid, and specimens were clean and bright; 3) BHT specimens were clean, and slightly orange, but still acceptable; and 4) the specimens treated with 8-hydroxyquinoline and 8-hydroxyquinoline sulfate were heavily iron-stained a dark blue-black. The aluminum end-coating, while effective, was not very satisfactory here as it rubbed off and may also have affected some of the treatments.

The condition of the specimens after kiln drying and conditioning (Table 3) was as follows: 1) The chelating agents had iron and blue stain present. However, they gave acceptable protection against brown stain, better than the present sodium azide treatment.⁵ 2) The antioxidants and LABTONE failed to protect from brown stain. 3) The specimens treated with phosphoric acid were totally free of blue or brown stain before and after shallow planing. 4) None of the treatments discolored the wood enough to be considered objectionable after surfacing.

⁵ A sodium azide treatment was run, in the laboratory, concurrently with this experiment on boards obtained from the same logs at the same source (Arganbright 1972). This treatment served as our sodium azide reference standard.

DISCUSSION

Brown stain in sugar pine was found to vary greatly in appearance. The stains appeared as spots, streaks, or total browning of the whole board with shade variations from light to dark brown. Contrary to the results of earlier studies, the heaviest discoloration was found in heartwood or at the heart-sap boundary.

Although the sample size was small, i.e., the number of specimens per treatment, the following trends were indicated: 1) The heaviest brown stain developed in the heartwood, or heart-sap boundary. 2) Blue stain was not significant and probably was suppressed by most of the more successful brown-stain treatments. 3) Antioxidants failed to control brown stain under our test conditions. 4) Iron chelating agents gave better results than the sodium azide treatment applied to boards obtained from the same source at the same time (Arganbright 1972). 5) The pH of the treating solution had to be below pH 5 to have a stain reducing effect, within the pH range incorporated in this study. 6) A multiple effect treatment gave the best results (e.g., one that had a low pH, sufficient concentration of the treating agent and iron chelation for the inactivation of peroxidase). Phosphoric acid had these properties when used in sufficient concentrations.

Kiln brown stain was not controlled with antioxidants such as BHT, ETHOXY-QUIN, and propyl gallate, probably because many of these antioxidants were designed to function in a small, closed environment, such as a cracker or cereal box, for a limited length of time. Even in *much* higher concentrations than those used to preserve foods (reached with the aid of additives and/or emulsifiers) the antioxidants tested were ineffective. Lundberg (1962) found that extremely high concentrations of the antioxidants BHT (included in this study), BHA, DPPD and NDGA (much higher than those allowed by law in foods) caused toxic effects in rats, chicks, and rabbits.

From our measurements, it was noted that the pH of the lumber increased the longer the milled boards were held before treatment. Brown stain also increased in severity and frequency with the length of time the boards were held solid-piled after milling before kiln-drying. Brown stain appeared to be controlled by keeping the pH of the board surfaces low during this period. In Experiment 3 the phosphoric acid treatment completely prevented staining.

The next best results were from those treatments that required an acid medium for solubility or those which themselves were acidic in solution. The chelating agents 8-hydroxyquinoline and 8-hydroxyquinoline sulfate showed good results at 3 and 6% concentrations. Both functioned in an acid medium since 0.2% phosphoric acid (Experiment 2) and 1.0% phosphoric acid (Experiment 3) were used to increase the solubility of 8-hydroxyquinoline and the sulfate in water produced an acid solution. Arganbright (1972), in similar research on toxicants, also found that acidic formulations controlled brown stain more effectively than basic treatments. The worst results in the present study were from treatments which had basic pH's, e.g., LABTONE or CARBOSET 514. The tap water was slightly acidic (pH 5.5) but this was not a low enough pH to reduce staining.

Apparently reduced pH is not alone sufficient to completely control staining, but must be combined with the inactivation of the peroxidase enzyme to be effective. However, the opposite also was true in that LABTONE, presumed to be active as an antiperoxidase, did not effectively control brown stain at pH 10. A 10% concentration of phosphoric acid (Experiment 3) was sufficient to prevent

staining, whereas 4% (Experiment 2) was too low. This may indicate that phosphoric acid, an iron chelating agent, was in a high enough concentration to inactivate the peroxidase. In Experiment 1 the length of time from milling to treatment probably allowed the boards to dry enough to mute the development of brown stain. That, along with the facts that there was only sapwood present and the storage conditions apparently were not very severe, allowed the 4% phosphoric acid to prevent staining in Experiment 1.

Hulme's work (1975) agrees with our conclusions that the concentration of the active agent and the pH of the treating solution are both important factors in stain control. Hulme's treatments between pH 7 and 10 were ineffective or minimally effective in preventing brown stain. In this study, LABTONE, an anti-peroxidase, even in high concentrations, had no effect at pH 10. Hulme had mixed results at pH 10 with one treatment; the 2% sodium carbonate and sodium bicarbonate (1:1) concentration was ineffective while the 5% concentration worked well. Hulme's results at pH 11.5 were also mixed and directly proportional to the concentration of the treating agent, sodium carbonate. Hulme's results and the trends indicated in this study suggest that pH's at either end of the scale may help control brown stain but only in conjunction with adequate concentrations of the treating agent.

CONCLUSIONS

It appears that iron chelating agents controlled brown stain more effectively than antioxidants or sodium azide. In using treating agents less toxic than sodium azide, either a very low or very high pH was necessary for an effective treatment. However, while the pH of the treating solution was important, the concentration of the treating agent was more significant. Phosphoric acid, 8-hydroxyquinoline and 8-hydroxyquinoline sulfate controlled brown stain in this study; however, the economics and mill feasibility of these treatments were not determined.

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