

Effect of Grafted UV Stabilizers on Wood Surface Erosion and Clear Coating Performance

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Synopsis

The critical element for a durable exterior clear coating on wood is photochemical stability of the wood surface beneath the coating. If this interface is not stabilized, even a durable coating will delaminate due to photoinduced wood degradation. A new method for stabilizing the wood surface was developed and evaluated. HEBP [2-hydroxy-4-(2,3-epoxypropoxy)benzophenone] was grafted to western redcedar (*Thuja plicata*). HEBP contains an ultraviolet (UV) stabilizer moiety that improved the xenon arc accelerated weathering performance of western redcedar (WRC). The weathering performance of the HEBP-modified WRC was compared with WRC treated with an unbound UV stabilizer of similar type and with untreated controls. The grafted stabilizer reduced the erosion rate (weathering) of unfinished WRC, and, as a pretreatment under clear finishes (spar varnish and exterior grade polyurethane), the stabilized surfaces improved coating performance and color retention.

INTRODUCTION

Many contemporary building exteriors incorporate the natural color and grain of wood siding. To preserve the natural appearance, wood must be protected against weathering. This weathering or photochemical degradation at the wood's surface causes a color change within a few days, and longer exposure erodes the surface. Although *solid* wood erodes slowly (0.25–0.5 in./century¹), and can create an attractive driftwood grey appearance, plywood shows the effects of weathering more quickly because of lathe checks and the thin surface veneer. If unprotected, complete failure of the surface veneer can occur within 10 years. With approximately 1/2 billion square ft of plywood siding used annually in the United States,² the importance of extending the life of this product goes beyond just aesthetic considerations. Longer service life of plywood siding relates directly to conservation of our national forest resource. The research presented here outlines a new approach for protecting wood surfaces against photodegradation.

Environmental effects on wood siding include solar radiation, water, oxygen, pollutants, and mechanical abrasion. While all of these can affect weathering, the ultraviolet (UV) portion of solar radiation causes the most damage. The degradation mechanism involves photoinitiated free radical generation followed by oxidation of the lignin.^{3–8} Early stages of lignin oxidation can be observed because of the formation of highly colored quinones and cyclohexadienones.^{4,5} Demethoxylation and other cleavage reactions occur along with this oxidation and lead to the destruction of lignin, the natural adhesive in wood. As the lignin is degraded, the individual wood fibrils are eroded away from the surface of wind, rain, and mechanical abrasion. Since the UV light penetrates only 75 μm into

wood,⁹ stabilizing this surface to UV light should reduce photodegradation and subsequent erosion.

Paint offers the most effective method for preventing weathering of wood. Paint pigments block or reflect UV light. If paint is removed from wood after many years outdoor exposure, the wood appears newly cut. But, since paint does not permit the color and grain of wood to show, the use of semitransparent stains has become popular. Although not as effective in blocking UV light as paint, stains allow the wood grain to show. Clear finishes, such as varnishes, contain no pigment and achieve a "natural look" since most of the natural color and grain of the wood can be seen. Clear finishes, however, are UV translucent and expose wood to intense solar radiation that permits rapid surface degradation of the wood.

In assessing the mechanism of clear-coating failure, it is often difficult to distinguish between wood failure (delamination) and coating failure (embrittlement, crazing, cracking, and erosion of the polymer film). Both are likely to occur with traditional alkyd and urethane varnishes. Most previous research has emphasized improvements in the coating rather than a pretreatment of the wood.

Past methods for stabilizing clear coatings have emphasized three concepts: first, incorporating UV stabilizers in clear finishes¹⁰⁻¹⁴; second, using UV transparent coatings¹⁵; and, third, stabilizing the wood surface with inorganic salts.^{15,16} UV stabilizers in the finish (method 1) extended the coating life, but did not produce long-term protection. The UV transparent coating (method 2) was not degraded but allowed rapid degradation of the wood's surface and thus delaminated. Both these methods failed to stabilize the critical wood-coating interface. Use of inorganic salts (method 3) differs from the previous methods in that the emphasis is on modification of the wood to enhance coating performance. Of the inorganic salts, aqueous chromium trioxide proved most effective. A simple dip or brush application of a 5% aqueous solution reduced erosion of uncoated wood¹⁶ and extended the life of coated wood when used as a pretreatment.^{15,16} Environmental concerns with chromic acid (aqueous chromium trioxide) and the green color of the photoreduction product [Cr(III)] impair its usefulness.¹⁶⁻¹⁸

The mechanism by which chromium trioxide affects wood properties and coating performance has been under investigation in our laboratories^{16,19} and by Pizzi.^{20,21} Although the mechanism has not been fully defined, chromium forms a leach-resistant complex at the wood's surface and, if coated with a finish, remains at the coating-wood interface. The observation that chromium was an effective pretreatment led me to investigate chemical bonded (grafted) organic UV stabilizers to wood as a pretreatment for clear finishes. This grafting would assure permanence and proximity of the stabilizer with the wood. This proximity is necessary in stabilizers that quench photoreactions since the limit for energy transfer is less than 10 nm for resonance transfer and less than 1.5 nm for exchange energy transfer.²² Unbound organic UV stabilizers are leachable and, if coated with a solvent-based finish, can partition into the organic coating. Thus, they are removed from the wood-coating interface.

Rowell and Gutzmer²³ established procedures for chemically modifying wood with epoxides and isocyanates, and grafted monomers of propylene and butylene oxide to hydroxyls in lignin and cellulose. The same reaction should be possible

TABLE I
Treatment Conditions and Statistical Data

Specimen no.	Treatment conditions ^a	Statistical data		
		y intercept	Slope ($\mu\text{m}/\text{h}$)	R^2
1	Pressure, 2% HEBP, dioxane	-71	0.28	0.74
1c ^b	Control (end-matched, untreated)	-75	0.36	0.78
2 ^c	Pressure, no HEBP dioxane (conditioned control)	-29	0.28	0.89
2c	Control (end-matched, untreated)	0	0.25	0.90
3	Pressure, 2% HEBP, dioxane	-4	0.16	0.62
3c	Control (end-matched, untreated)	-25	0.29	0.84
4	Pressure, 4% HEBP, dioxane	-16	0.22	0.60
4c	Control (end-matched, untreated)	-38	0.32	0.78
5	Pressure, 2% HEBP, acetone	-68	0.28	0.87
5c	Control (end-matched, untreated)	-41	0.35	0.72
6 ^c	Pressure, no HEBP acetone	-82	0.40	0.95
6c	Control (end-matched, untreated)	-30	0.30	0.94
7	Dip, 5% DHBP, acetone	-37	0.25	0.87
7c	Control (end-matched, untreated)	-0.2	0.26	0.96

^a The treating conditions are more fully explained in the experimental section. The brief description is to help identify the various specimens. The formatted information gives pressure vs. dip, stabilizer and concentration, and solvent.

^b The "c" designates an untreated end-matched control.

^c Specimens 2 and 6 are "conditioned controls" (i.e., pressure treated without having the UV stabilizer grafted).

with UV stabilizers having an epoxide moiety. Epoxide and UV stabilizer functionality are found in 2-hydroxy-4-(2,3-epoxypropoxy)benzophenone (HEBP). Molecules containing the *o*-hydroxybenzophenone moiety (the UV stabilizer) inhibit UV degradation in synthetic polymers.^{13,24,25} Thus HEBP contains a UV stabilizing group and a glycidyl ether (epoxide) that can react with wood hydroxyls as did propylene and butylene oxides.²³ Although the use of organic solvents and the size of this molecule would limit its penetration into the wood, surface reactions seemed feasible. (HEBP is insoluble in water. A water-soluble chemical would penetrate better because water swells wood more than the organic solvents used in this work.) As noted previously, UV degradation of wood is a surface phenomenon. Therefore, only surface modification should be necessary to effect improved performance.

The objective of this research was to determine if a UV stabilizer chemically bound to wood could retard or prevent UV-induced degradation of the treated wood surface. This chemically bound stabilizer was compared with, first, the same stabilizing group not chemically bound and, second, untreated controls.

EXPERIMENTAL

Methods

The experiments evaluated treatment conditions, solvent effects, HEBP concentration, and chemically bound versus unbound stabilizer. Table I lists seven treatments that addressed these parameters. Each treatment (three replicate specimens) was compared with an end-matched untreated control

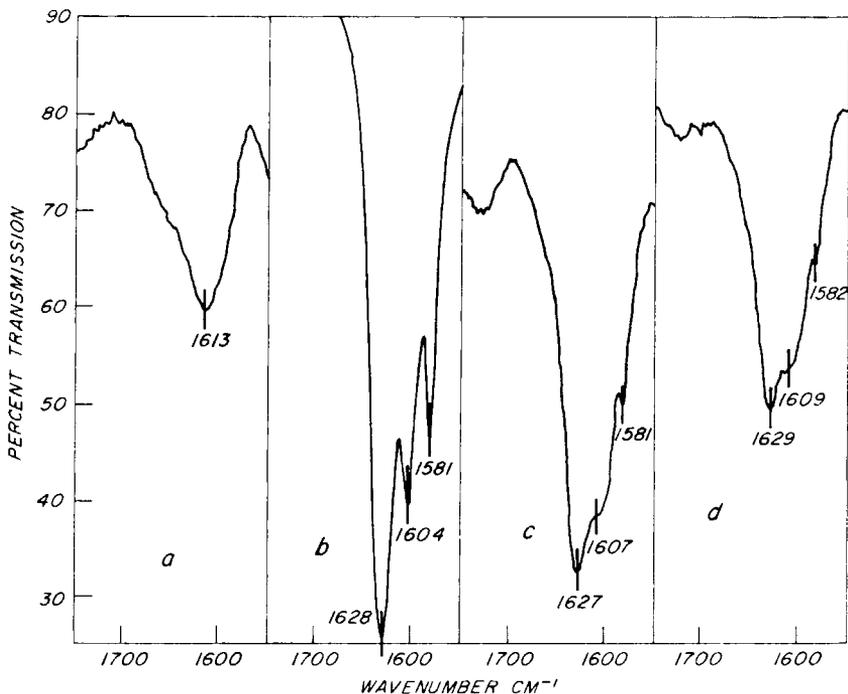


Fig. 1. IR carbonyl absorptions of wood, HEBP, and modified wood: (a) Western redcedar (WRC); (b) HEBP; (c) HEBP-modified WRC before leaching with acetone; (d) HEBP-modified after leaching with acetone.

designated with a *c* suffix. Specimens in treatments 2 and 6 were prepared in the same way as the specimens in treatments 1, 3, 4, and 5, but the UV stabilizer (HEBP) was not included. The specimens from treatments 2 and 6 establish a base from which to judge the performance of HEBP and will be referred to as “conditioned controls” to differentiate them from the end-matched untreated controls (*#c*). Comparison of treatments 1–4 vs. 5 and 6 addresses solvent effects, and treatment 4 identifies the HEBP concentration effect. Treatment 7, dip treatment with DHBP (2,4-dihydroxybenzophenone), contrasts the stabilizer moiety absorbed at the surface with those chemically grafted to the surface.

Erosion of treated and control specimens was measured at 400, 600, 800, and 1000 h accelerated weathering based on light exposure only. Measurements prior to 400 h were inconsistent and difficult to obtain. Each measurement consisted of five observations that were averaged prior to statistical analysis. (Only three observations were obtained for one of the specimens from treatment 3.) Measurement of the same growth rings each time minimized variation and led to consistent erosion data on a particular specimen.

Materials

All chemicals were reagent grade. Epichlorohydrin and triethyl amine were distilled prior to use, and all solvents were dried over 4 Å molecular sieves. DHBP (2,4-dihydroxybenzophenone) was used as received. HEBP was prepared

from DHBP according to Maňásek et al.,²⁶ recrystallized twice from 95% ethanol, and melted at 97.5°C to 99°C (lit. 92–95°C).

Infrared (IR) and mass spectroscopy data were obtained from a Beckman IR-12 Infrared Spectrophotometer and a Finnigan GCMS 4500. IR absorption at 3450, 1628, 1604, 1581, 1355, 1267, 1233, 1118, 910, 864, 702, and 631 cm^{-1} , and mass spectroscopy fragmentation (numbers in parentheses are percent) of 271(15), 270(100), 269(91), 213(16), 193(15), 136(18), 105(15), and 77(14) were obtained. Data were not collected below mass 50, and only peaks greater than 13% of most intense peak are listed.

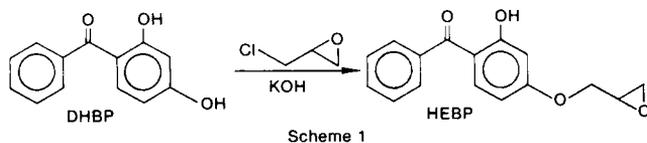
All specimens were cut from vertical-grain western redcedar (*Thuja plicata* Donn ex D. Don) having about 40 growth rings per inch. Strips 1-in. (radial) by $\frac{5}{16}$ in. (tangential) were abrasive planed with 100-grit paper to $\frac{1}{4}$ in. tangential. Specimens and their end-matched controls ($1 \times 1 \times \frac{1}{4}$ in.) were cut from the $1 \times \frac{1}{4}$ in. strips and stored in a dark room at 80°F (26.7°C)/65% relative humidity prior to chemical treatment. After oven-drying at 105°C for 24 h, specimens were pressure treated with HEBP for 22 h at 150 psi (1034 KPa) and 120°C, and oven-dried 6 h (105°C) following treatment. Treating solutions were nominally 2% HEBP and 0.05% triethylamine (TEA) catalyst in either 1,4-dioxane or acetone, except as noted in Table I. DHBP specimens were prepared by dip-treating edge-sealed specimens 1 min in 5.3% DHBP acetone solution followed by air drying. All other specimens were edge sealed with aluminum flake pigmented varnish prior to accelerated weathering. Accelerated weathering equipment and cycle were the same as used previously by Feist¹⁶ (20 h xenon arc light followed by 4 h water spray in the dark). The erosion measurement procedure used was identical to that developed by Feist and Mraz.¹

Commercial spar varnish and exterior grade polyurethane were applied by brush to the wood surface. Average spreading rates were 182 ft^2/gal and 273 ft^2/gal for coats 1 and 2 of the polyurethane, and 373 ft^2/gal and 459 ft^2/gal for coats 1 and 2 of spar varnish.

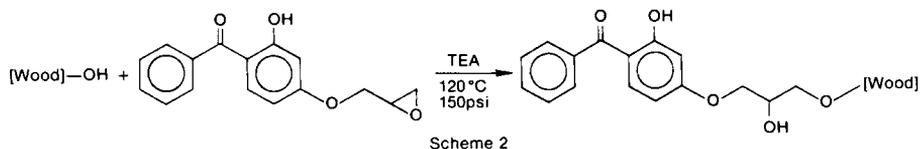
RESULTS AND DISCUSSION

Grafting of HEBP to Wood

The UV stabilizer was prepared as shown in Scheme 1 according to Maňásek et al.²⁶



Note that the product is *para*-substituted. *Ortho* substitution would destroy the *o*-hydroxybenzophenone moiety and render the compound useless as a UV stabilizer. The *para*-substituted glycidyl ether made possible the chemical reaction of the stabilizer with wood hydroxyls as illustrated in Scheme 2:



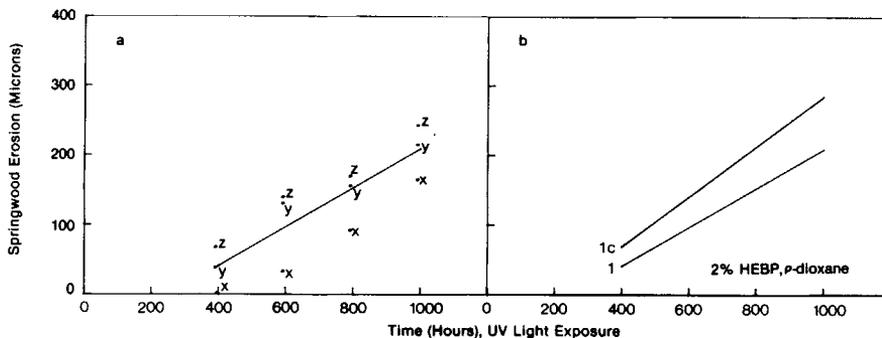


Fig. 2. Springwood erosion vs. time. Figure 2(a) shows the average springwood erosion for the three observations (x , y , and z) of treatment 1 and the best least squares fit for the 12 data points. Figure 2(b) shows the same line as Figure 2(a) (1) and the end-matched control (1c) for treatment 1.

This reaction is similar to the previously discussed work by Rowell et al.⁸

The reaction of HEBP with wood should involve only the epoxide function as shown in Scheme 2. The IR spectrum of HEBP [Fig. 1(b)] has characteristic carbonyl absorptions at 1628, 1604, and 1581 cm^{-1} . Comparison of Figure 1(a) (IR of unmodified wood) with Figure 1(c) (IR of modified wood) shows these characteristic carbonyl absorptions superimposed on the broad absorption due to wood. These spectra indicate the UV stabilizing group (*o*-hydroxy ketone) is not involved in the reaction at the surface and is therefore available to function as a UV stabilizer. Extraction of treated wood with acetone, methylene chloride, and water had no effect on the IR spectra [Fig. 1(d)]. Although the spectra prove that the reaction involves only the epoxide group, the exact reaction cannot be determined from IR data. Homopolymerization of the epoxide forming an insoluble polymer could explain the data. To differentiate between these two possibilities, solubility of the polymer in wood was compared with HEBP-polymerized neat. The homopolymer exhibited slight solubility in acetone and methylene chloride. This behavior was not observed with treated wood. Scanning electron microscopy (SEM) revealed homopolymer formation in some areas of the wood, but not in others. These differences in surface morphology had little effect on overall performance. Based on the solubility, performance, and SEM data, the reaction at the wood surface probably involved both grafting and homopolymerization. The grafting is the most crucial since this assures that oligomers and linear polymer will remain at the surface.

Grafting HEBP to wood resulted in almost no change in weight of specimens, and therefore percent grafting could not be calculated. The pickup of HEBP was of the same magnitude as weight loss due to leaching and was quite small. The modified wood had rather small amounts of UV stabilizer at the surface of the wood.

Erosion Measurements

The primary method for evaluating UV stabilized wood involved springwood erosion measurements. Specimens were measured at specified intervals of xenon arc accelerated weathering. Improved surface stability due to the grafted UV

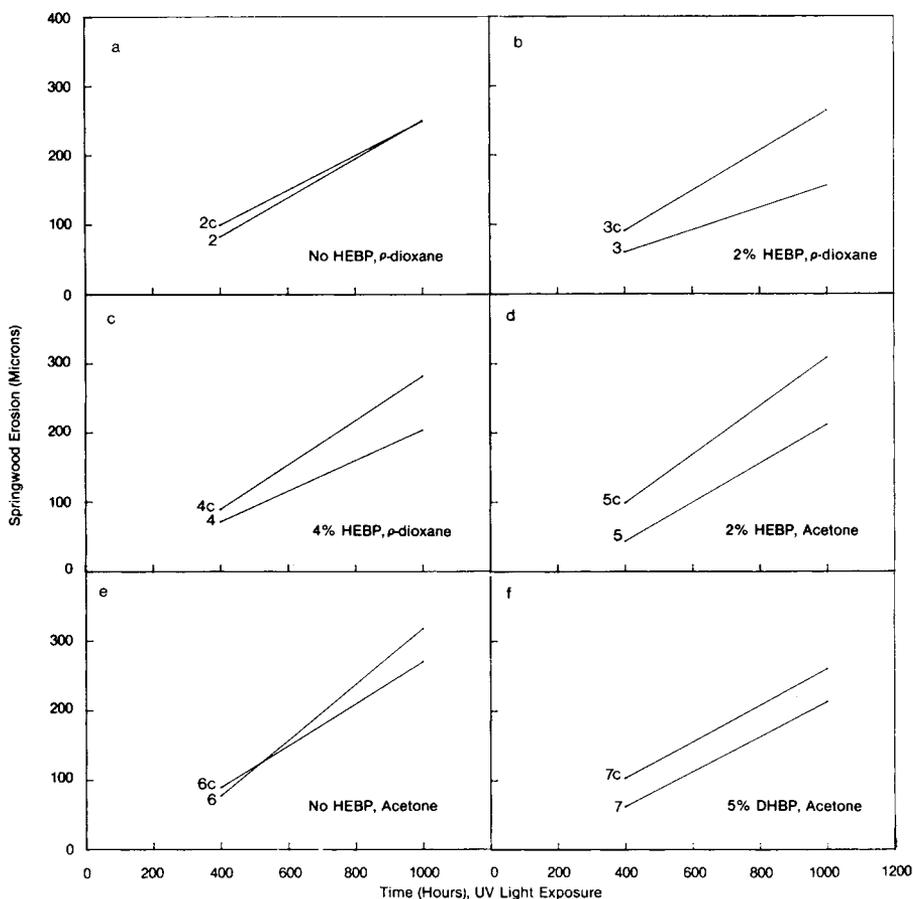


Fig. 3. Springwood erosion vs. time. a-f represent the erosion rates in treatments 2-7, respectively. Each part shows the performance of the treated specimen vs. its end-matched control.

stabilizer was manifest as slower springwood erosion. Protection of the top half of each specimen during accelerated weathering established a datum for these measurements. Statistical analysis of erosion data of the seven treatments indicated a linear relationship between erosion and time during the 400-1000-h weathering interval. Least squares fit of the average erosion data gave the linear parameter estimates listed in Table I.

Erosion performance was evaluated using two criteria. First, the difference in rate of erosion (slope) between specimens and matched controls indicated erosion retardation by the treatment. These slopes are listed in Table I and show significant differences for some treatments. Second, the erosion difference between values at 400 h suggests erosion inhibition. Ideally, the erosion trends should be extrapolated back to the x -axis to separate inhibition from retardation since difference in slope affects the intercept. In addition to the complication due to slope, early stages of erosion were difficult to measure, and those that were obtained suggested nonlinear behavior. Thus the effects of erosion treatment should not be extrapolated prior to 400 h. However, if the slopes are equal, these threshold values permit qualitative estimates of inhibition.

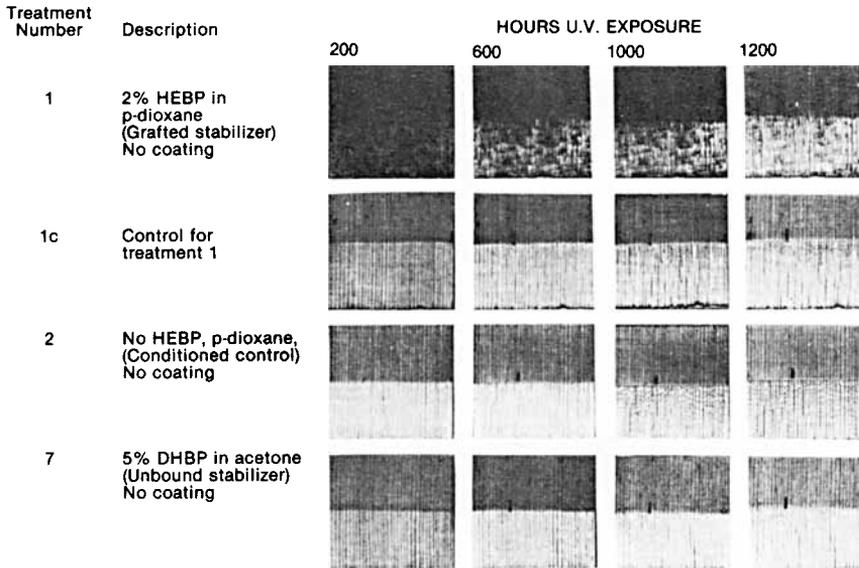


Fig. 4. Erosion performance of HEBP-treated western redcedar (1) vs. an end-matched control (1c), conditioned control (2), and DHBP (7). The end-matched controls for treatments 2 and 7 were similar to 1c and are not shown. The top half of each specimen was protected during weathering.

Figure 2 illustrates the average erosion values for treatment 1 specimens and controls at each time and the line based on the regression analysis. The R^2 (correlation coefficients) values listed in Table I undoubtedly reflect the large natural variation in wood rather than the evaluation procedure or treatment. Excellent correlations were obtained from individual observations. For example, the correlation coefficient for observation x in Figure 2(a) was 0.98 and illustrates consistent erosion within a particular growth ring and consistent evaluation procedures. Similar spread in data among the controls indicated that the inherent variability of wood is more important than treatment variability.

All HEBP-treated specimens (treatments 1, 3, 4, and 5) had reduced rates of erosion (smaller slope) compared with the end-matched controls (Table I). Figure 2(b) illustrates the average erosion data from each of the specimens and controls from treatment 1 and a representation of the best least squares fit for each set of data. Figure 3 illustrates the other data in Table I. Comparison of treatments 1, 3, and 5 shows no solvent dependence between acetone and *p*-dioxane. The variation among *p*-dioxane-treated specimens is greater than the difference between treatment 1 and 5 (*p*-dioxane and acetone). The differences at 400-h exposure suggested some inhibition, but, as mentioned previously, this may be an artifact of rate difference. The performance of treatment 4 (increased HEBP concentration) shown in Figure 3(c) indicated no significant improvement with increased concentration of treating solution compared with treatments 3 and 5. Low HEBP concentration seems adequate to achieve improved surface stability.

Conditioned controls [treatments 2 and 6, Figs. 3(a) and 3(e)] showed little difference at 400 h compared with untreated controls (treatments 2c and 6c). The slope (Table 1), however, indicated faster erosion of conditioned controls.

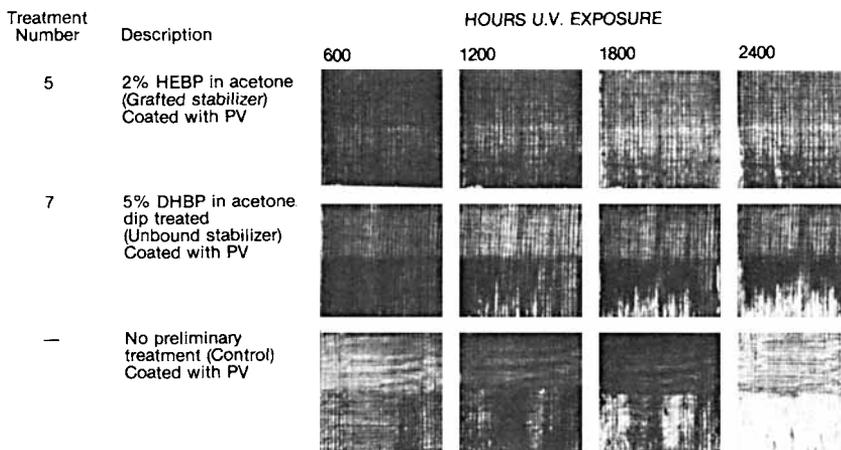


Fig. 5. Performance of exterior grade polyurethane varnish (PV) over HEBP- and DHBP-treated and untreated western redcedar at 600, 1200, 1800, and 2400 h of xenon arc UV exposure. The top half of each specimen was protected during weathering.

The treating conditions (120°C for 22 h in a basic solvent) probably caused wood degradation that gave rise to faster erosion. Considering the negative effects of treating conditions, the improvement due to the UV stabilizer (treatment 1, 3, 4, and 5) may be more significant than expressed in Figures 2 and 3. No attempt was made to quantify this difference since, in future work, less drastic conditions will be used to graft the stabilizer.

The performance of the unbound UV stabilizer [Fig. 3(f)] indicated no retardation. The slopes from the control and unbound stabilizer are almost equal. The difference at 400 h suggested inhibition early in the test, and visual inspection confirmed the loss of the stabilizer by 200 h. Compare treatment 7 with treatment 1 in Figure 4. The stabilizer was effective for a short time, but, once leached out, the treated wood eroded the same as the control. Thus bound HEBP not only inhibited initial erosion but also slowed the rate of erosion.

In this study, chromic acid-treated wood was not included. Chromic acid-treated specimens could have established a reference for added evaluation of the seven treatments (Table I). In subsequent work, I evaluated treated specimens from the same boards of western redcedar as used in this work. The treated wood eroded at 0.16 μ /h and their end matched controls eroded at 0.31 μ /h. Feist¹⁶ reported erosion rates of 0.04–0.16 μ /h and showed that the rate is dependent on chromium concentration, species, and even the piece of wood. Matched controls are essential for proper evaluation. Even with specimens cut from the same boards, the erosion rates (0.16 and 0.31 μ /h) should not be rigorously compared with the rates in Table I. The intensity of xenon arc radiation changes with time and varies slightly with different bulbs. On a qualitative basis, however, the performance of HEBP-treated wood compares favorably with chromic acid-treated wood (for example, specimen numbers 3 and 3c in Table I).

The erosion specimens were evaluated for color retention and photographed in black and white and color at 200-h intervals up to 1200 h accelerated weathering. Figure 4 is a composite made from typical samples of these photographs. The composite includes a single specimen from three different treatments (1,

2, and 7), and the end-matched control from treatment 1. The other controls were similar and are not shown. The specimen from treatment 1 and its end-matched control (1c) can be compared at 200, 600, 1000, and 1200 h of UV exposure. The top half of each specimen was protected during weathering. Even from the black and white photograph in Figure 4, it is obvious that the HEBP-grafted wood performed better than the other specimens. In fact, little difference is apparent among the controls, conditioned controls, and unbound stabilizer specimens. The photographs reinforce the erosion data. The other specimens from the treatments shown in Figure 4 were similar. Specimens treated in acetone showed slightly less color retention; however, all specimens having grafted UV stabilizer showed excellent color retention up to 1000 h compared with unbound DHBP, untreated, and conditioned controls. The color stability due to grafted HEBP was lost by 1200-h xenon arc accelerated weathering. The treatment lacked long-term durability because the chemically modified surface eventually eroded. As long as the surface contained grafted HEBP, erosion was slowed thus proving enhanced surface stability, and this stabilized substrate should improve performance of clear coatings.

Performance of Clear Coatings

The second method used to evaluate the UV stabilizer performance on wood involved monitoring the performance of two types of clear coating (a heavy-duty varnish and a polyurethane finish). These coatings were applied over the same pretreatments used in the erosion experiments [that is, HEBP (#5) and DHBP (#7)]. An untreated control was also coated. As mentioned previously, the object was to compare the chemical-bound HEBP with unbound DHBP and an untreated control. Improvements in substrate should be manifest as improved coating performance.

The coated specimens were photographed for color retention and coating performance. Figure 5 shows a black and white composite of typical specimens coated with exterior grade polyurethane varnish at 600, 1200, 1800, and 2400 h accelerated weathering. The spar varnish performed similarly and is not shown. The importance of chemical bonding can be seen at 1200 h exposure (Fig. 5). The unbound DHBP and unmodified control show streaking and dark areas. The light-colored streaks indicate delamination of the film caused by failure at the interface. This type of failure is visible over large portions of number 7 (DHBP) and the control at 1800 h. Even at 2400 h, the HEBP-treated wood resisted widespread delamination. The grafted stabilizer improved the wood-coating interface and extended the life of the coating. At 2400 h, the color of the exposed grafted wood was almost the same as the protected portion.

CONCLUSIONS

The results of this study prove that a UV stabilizer [2-hydroxy-4-(2,3 epoxy-propoxy)benzophenone] can be chemically bound to the surface of western redcedar and that the grafted stabilizer slows weathering. The chemically bound system exhibited superior performance over both untreated wood and the same stabilizing moiety unbound. The improvements included reduced erosion rates

of uncoated wood, prolonged clear-coating life, and improved wood color retention both with and without a clear coating.

The positive trends shown by these preliminary results have been expanded to include accelerated outdoor weathering on modified southern pine and redwood. Other UV stabilizers, which require less severe reaction conditions to graft to wood, and those which exhibit greater stabilizing efficiency such as hindered amines and benzotriazoles, are currently under investigation. Future research will also investigate combinations of stabilizers and antioxidants to evaluate synergistic effects.

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