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Performance of wood treated with prospective organic surface protectants upon outdoor exposure: FTIR spectroscopic analysis of weathered surfaces

Abstract: Visual appeal of wood is as important as its structural integrity in outdoor applications. Discoloration and checking of wood favor the utilization of alternative materials for outdoor cladding and decking. Lignin depolymerization is one of the critical processes leading to weathering. In the present paper, the potential of different classes of surface protection agents has been assessed on loblolly pine (*Pinus taeda*) impregnated with water repellents, organic UV inhibitors, and an organic biocide. The treated samples were exposed to predetermined doses of solar radiation in Eastern Oregon, and the chemical changes occurring on the surfaces were evaluated by FTIR spectroscopy. After 1-year exposure, lignin loss was complete on all surfaces; however, some treatments provided longer term protection than others. Organic UV light inhibitors were most effective in this regard. A petroleum-based water repellent with a melting point of 54–58°C also provided protection against lignin degradation.

Keywords: 4,5-dichloro-2-n-octyl-3-isothiazolinone (DCOIT), chromic acid, FTIR spectroscopy, hindered amine light stabilizer (HAL), hydroxyphenyl bezotriazole, isothiazolone, lignin, loblolly pine, *Pinus taeda*, UV stabilizers, water repellents, wax, weathering, wood surfaces

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Introduction

The attractiveness of wood in outdoor cladding, decking, and structural applications is considerably reduced by graying and checking (weathering). This leads to the use of other building materials – such as metal, stone, or synthetic polymers – which do not discolor or crack with time (Truini 1996; Principia Partners 2001; Evans et al. 2002; Williams 2005).

The underlying chemical reactions of weathering were extensively studied worldwide and at the United States Forest Products Laboratory in the 1980s (Feist and Hon 1984; Hon and Feist 1986). In short, self-sustaining bleaching reactions are initiated through the absorption of sunlight and the organic free radicals that arise (Hon 1981). Atmospheric oxygen and free radicals form hydroperoxide, which is capable of destroying the covalent bonds in lignins and hemicelluloses (Hon et al. 1982). These chemical reactions were shown to directly affect surface appearance, resulting in discoloration with increasing exposure time (Pandey 2005). Cellulose remains after the degradation products are removed by rain, and this imparts a grayish surface appearance (Browne and Simonson 1957; Kalnins 1966). More specifically, lignin degradation in the compound middle lamella (CML) reduces interfiber bonding, leading to defibrillation of the wood. As the process advances, the weakened surfaces are further eroded by wind and rain resulting in the formation of checks, raised grain, and reduced wood cross section (Sell and Feist 1986; Williams 2005).

Black and Mraz (1974) investigated a large number of chemicals for protecting wood surfaces from weathering without obscuring the natural pattern, color, and texture of the wood. Chromium-based chemicals, particularly chromic acid, were the most effective in preventing weathering (Feist and Ellis 1978). As revealed by FTIR spectroscopy, lignin was chemically modified providing a great resistance against UV radiation (Pandey and Khali 1998). Unfortunately, the hazardous nature of chromic acid has

prevented its commercial use for this application. Iron oxide-based chemicals and grafting of UV absorbers onto the wood surface were also promising, but none of these compounds completely prevented surface degradation (Evans et al. 1992; Ntshilele et al. 1994; Chou et al. 2007). The development of effective and environmentally benign protective agents is still one of the greatest challenges in wood research as pointed out in recent publications, for example, by Verma et al. (2009), Namyslo and Kaufmann (2009), Robinson and Laks (2010), Pilgård et al. (2010), Lee and Cooper (2010), Chirkova et al. (2011), Freitag et al. (2011), Schultz and Nicholas (2011), and Pankras and Cooper (2012).

The surface durability of wood that is modified by heat treatment, acetylation, or by other means is promising (Chang and Chang 2001; Evans et al. 2002; Termiz et al. 2006; Stanzl-Tschegg et al. 2009; Windeisen et al. 2009; Pfriem et al. 2010; Thygesen et al. 2010). Although these technologies are being commercialized, not all timber species are suitable, and the technology remains expensive compared to the conventional wood impregnation techniques.

There is still a need to improve the protection of wood surfaces from the combined action of sunlight, oxygen, and water (weathering). Wax-based water repellents have been shown to reduce check formation in southern pine as a result of reduced moisture cycling (Cui and Zahora 2000; Zahora 2000). Wood preservatives have also been shown to have an effect on the weathering performance of wood surfaces. Yet, these results have not been related to chemical changes in the wood polymers.

Ammonium copper quaternaries and chromate copper arsenate improve color retention (Termiz et al. 2005). Wood preservatives containing dialkyl quaternary compounds have also been shown to lead to surface defiberization when exposed to artificial weathering (Jin et al. 1991). Treating *Pinus elliotii* with copper-amine partially diminishes/discoloration but does not prevent lignin degradation (Zhang et al. 2009). The effects of the more recently developed organic wood preservatives – such as 4,5 dichloro-2-octyl-2H isothiazol-3-one (DCOIT) – on weathering have not been evaluated, and the majority of the weathering studies conducted, thus far, have only been carried out under controlled laboratory conditions negating the effects of air pollution and biological agents.

Thus, the present paper is aiming at the comparison of chemical changes that occurred in wood treated with different wax-based water repellents and DCOIT with those in untreated, chromic acid-treated, and organic UV inhibitor-treated samples. The outdoor tests were performed

over a 1-year period in the western United States, and the results were checked mainly by FTIR analysis.

Materials and methods

Sample preparation

The characteristics of the protective materials (three wax-based water repellents, two organic UV stabilizers, and one organic biocide) are presented in Table 1. Clear, flat sawn loblolly pine sapwood (*Pinus taeda*) samples (10×85×155 mm³, rad.×tang.×long.) were prepared from nominal 25×100 mm² stocks by planing and crosscutting. One sample from each of the 10 parent boards was impregnated. Impregnation: 1. A 5-min initial vacuum at -3.3 kPa; 2. Pressure period of 10 min at 1034 kPa; 3. Final vacuum at -3.3 kPa. The samples were weighed before and after the treatment. The samples were reconditioned to 9% moisture content (MC) in a climate chamber (25°C, 50% RH). Chromic acid was painted on the surface and not vacuum/pressure impregnated. The net retentions of the solvents after impregnation were compared by means of Tukey's multiple comparison test in SAS version 4.1 software (SAS Institute, Cary, NC 2006, USA).

Outdoor exposure

The samples were end-sealed with a marine epoxy resin and mounted on aluminum supports so that the wood growth rings were oriented concave face upward. Each sample was fixed to the rack with an M 4.5 stainless steel screw in each corner. This manner of mounting the samples restrained the samples, preventing cupping and warping and, instead, resulting in the release of moisture-related stresses through check formation. In addition, this type of restraint simulated the typical installation methods used for such products as wooden cladding and decking. Six samples per treatment were exposed to weathering in the Eastern Oregon high-elevation desert, at a site near Klamath Falls (Lat: 42.22, Long: -121.78, elevation of 1250 m) on racks pitched at 45° pointing to the south. Solar irradiation data was collected daily from an AgriMet weather station operated by the Bureau of Reclamation and located at the site. Natural precipitation was augmented with nonchlorinated irrigation water between May and the end of October by spraying the samples for 15 min per day at sunrise. One sample per treatment was harvested as closely as possible to a targeted 454-MJ m² irradiation interval. This irradiation corresponds to the amount of solar energy received in the 2-week bracketing the summer solstice. The harvested samples were reconditioned to 9% MC and examined by FTIR spectroscopy.

FTIR spectroscopy

Small specimens (2×10×20 mm³) were removed from each sample with a carving knife. Three specimens from different earlywood (EW) and latewood (LW) bands were selected from each sample at random. The FTIR instruments operating in the ATR mode: 1. ThermoNicolet Avatar 370 (equipped with a Smart Performer ZnSe crystal, Madison, WI, USA) and 2. Perkin Elmer Spectrum One (Waltham, MA, USA) (equipped with a MIRacle, Pike Technologies Inc. 2009, ATR adapter fitted with a com-

| Type | Active agent | Compound and source | Effect | Retentions of solvents (kg m ⁻³) | |
|------------------------|---------------------------------------|-------------------------------|--|--|--------------------|
| | | | | Target | Found ^a |
| UV stabilizers | | | | | |
| A | Hydroxyphenyl benzotriazole | Tinvine 1130 ^b | Blocks UV-initiated reactions | 0.75 | 0.56 (0.53–0.59) |
| B | Hindered amine light stabilizer (HAL) | Tinvine 292 ^b | Free radical scavenger | 0.75 | 0.55 (0.52–0.59) |
| Oil in water emulsions | | | | | |
| A | Wax m.p. 34–38°C | Indrawax 097 ^c | Water repellent | 15.00 | 9.8 (8.7–10.8) |
| B | Wax m.p. 54–58°C | Indrawax 210 ^c | Water repellent | 15.00 | 11.1 (10.6–11.7) |
| C | Wax m.p. 65.5–75°C | Indrawax 582 ^c | Water repellent | 15.00 | 11.3 (10.3–12.3) |
| Biocide | | | | | |
| | DCOIT | Kathon 287 ^d | Inhibits mold + biological discoloration | 0.75 | 0.50 (0.4–0.5) |
| Controls | | | | | |
| Positive | Chromic acid | Thermo Fisher Scientific Inc. | Inhibits surface discoloration | 3% Solution ^e | – |
| Negative | Untreated | – | – | – | – |

Table 1 Compounds tested in the present study during outdoor weathering of loblolly pine sapwood.

^aIn parentheses: upper and lower confidence interval limits at $\alpha=0.05$. ^bCiba Specialty Chemicals Inc (Tarrytown, NY, USA). ^cIndustrial Raw Materials LLC (Plainview, NY, USA). ^dRohm and Hass (Philadelphia, PA, USA). ^eBrushed on the surface.

posite diamond crystal, Madison, WI, USA). The spectra were baseline corrected, and six spectra collected at different places were averaged to produce composite spectrum (Omnic 7.4 software, Thermo Scientific Inc. 2007, Madison, WI, USA), that represents the entire sample.

The spectra were normalized to the C-O stretching band of cellulose and hemicelluloses located at 1033 cm⁻¹. The carbonyl region was treated differently from the other regions. Here, the spectral range between 1750 and 1700 cm⁻¹ was normalized. Fourier self-deconvolution (Omnic v7.4) was applied to resolve overlapping peaks, peak fitted and quantification, allowing for the analysis of esters (1736 cm⁻¹), carboxylic acid (1726 cm⁻¹), and conjugated ketones (1716 cm⁻¹) (Fabiyl et al. 2011). The peak height of each peak, identified by self-deconvolution, was mathematically computed (after peak fitting) and expressed as % change (Figure 1).

Results and discussion

As Table 1 indicates, the net retentions of the two UV stabilizers did not differ statistically from each other ($\alpha=0.05$).

The retentions of the WAX_{34–38°C} were statistically lower (9.8 kg m⁻³) than that of the other two waxes with higher melting points (11.1–11.3 kg m⁻³). The target retentions were calculated on mean solution uptakes of 600 kg m⁻³, typical of the loblolly pine grown in the southeastern USA. The actual retentions were below the target retentions for all treatments. This systematic error was partly due to the variable permeability of the pine specimens observed in this study.

The actual irradiation doses received by the samples (Table 2) were within 7% of the target, except for period six, where samples were arbitrarily removed after approximately 1 year of exposure. The length of exposure time required to achieve a specific irradiation dose varied by season, increasing with the onset of winter and then decreasing again as summer arrived (Table 2). Surface discoloration occurred rapidly as predicted by Evans et al. (1996), while the low RH (Table 2) kept the biological

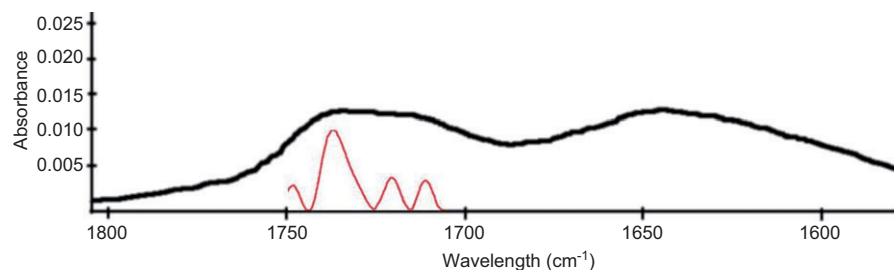


Figure 1 FTIR spectra of the carbonyl region (black) overlaid with the same spectra after self-deconvolution (red). Ester band at 1736 cm⁻¹, carboxylic acid at 1726 cm⁻¹, and conjugated ketone at 1716 cm⁻¹.

| | Exposure Time (days) | Irradiation dose (MJ m ⁻²) | Average RH (%) |
|--------|----------------------|--|----------------|
| Period | | | |
| 1 | 19 | 484 | 52 |
| 2 | 42 | 939 | 50 |
| 3 | 78 | 1363 | 58 |
| 4 | 152 | 1886 | 70 |
| 5 | 201 | 2337 | 72 |
| 6 | 357 | 6096 | 67 |

Table 2 Exposure time, irradiation dose, and average relative humidities (RHs) of loblolly pine samples exposed up to 357 days to weathering in Klamath Falls, OR.

discoloration of the surfaces to a minimum. The low RH combined with the daily water sprays allowed for the rapid development of checks in all of the materials (Schauwecker 2011). The checks were predominately found in the LW bands (Figure 2), and their number generally increased over time, while the check length and width remained uniform. This may be explained by the greater stiffness of the cell walls in the LW portion of the growth ring, when compared to the thinner cell walls in the EW.

FTIR spectroscopy of wood surfaces

The two FTIR spectrometers used were calibrated by comparing the spectra obtained from the untreated, nonexposed control sample. The ATR crystals of the two instruments were different, but the spectra of the two instruments are directly comparable (see also Schauwecker 2011) with the exception of the region at 1700–1550 cm⁻¹. The ATR_{ZnSe} crystal appears to be slightly less sensitive in this region than the ATR_{diamond} crystal. Nevertheless, both instruments clearly resolved the two shoulder bands in this area located between 1650 and 1600 cm⁻¹. Accordingly, the spectra obtained by the two instruments will not be differentiated in the discussion.

Weathering became noticeable primarily in the 2950–2850 cm⁻¹, 1735–1716 cm⁻¹, and 1660–1513 cm⁻¹ regions (Figure 3). The 2950–2850 cm⁻¹ region is associated with aliphatic groups (C-H stretch in methyl and methylene groups) that are typical for extractives (Table 3; Mayo et al. 2004; Fabiyi et al. 2011). Bands between 1735 and 1716 cm⁻¹ are due to carbonyl groups and originate from all major wood polymers and their degradation products. The deconvolution (peak fitting) helps identify these



Figure 2 Photographs showing progressive check and discoloration development on untreated control loblolly pine sample (a) 0 and the samples (b–g) for the indicated irradiation doses.

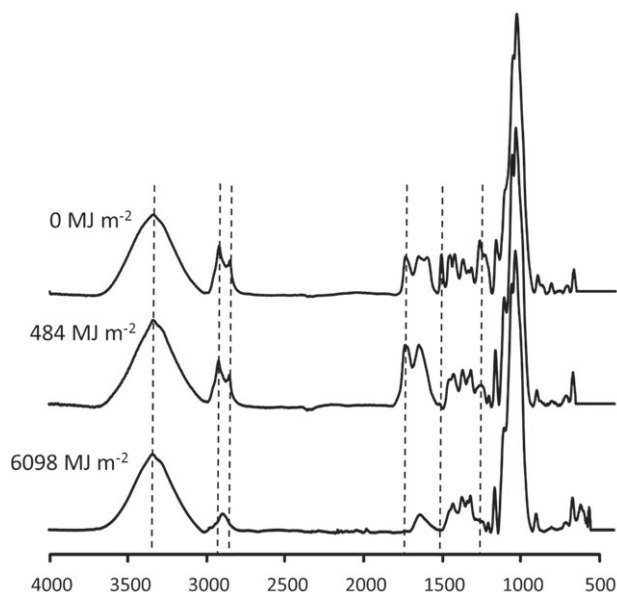


Figure 3 FTIR spectra of the untreated loblolly pine after exposure to different doses of solar radiation in Eastern Oregon. The bands of interest are with dotted lines.

peaks (Leary 1967; Anderson et al. 1991; Fabiyi et al. 2011). Three bands typical of lignin are located between 1660 and 1513 cm^{-1} with the primary aromatic vibration located at 1513 cm^{-1} (Table 3; Anderson et al. 1991; Tolvaj and Faix 1995; Stirling and Morris 2009; Schauwecker 2011). The lignin degradation/retention was calculated based on these band heights, which are most sensitive to photodegradation.

Samples treated with petroleum waxes

The aliphatic bands lost definition over time for all but one treatment, resulting in the eventual absence of the band located at 2850 cm^{-1} (Figure 3). However, a remnant band could be discerned at 2950 cm^{-1} even after 1 year of exposure. The untreated and chromic acid-treated controls lost the band at 2850 cm^{-1} within the first 2000 MJ m^{-2} of exposure, while the peak at 2950 cm^{-1} lost 80% of its initial height over the same exposure interval (Figures 4 and 5). The natural waxes and extractives were likely leached from the surface. The preservation of the aliphatic band was strongly influenced by the type of wax. The waxes with higher melting points (WAX_{hmp} , with longer molecular chains) provided greater protection than those with lower melting points (WAX_{imp}). Thus, WAX_{hmp} was less susceptible to oxidation and leaching of the degradation products. The samples treated with $\text{WAX}_{34-38^\circ\text{C}}$ lost the aliphatic bands within the first 2000

| Wavelength (cm^{-1}) | Functional group | Origin in wood |
|---------------------------------|--|--|
| 2950 and 2850 | Aliphatic groups, $\text{R-CH}_2\text{-CH}_2\text{-R}$ | Extractives |
| 1736 | Esters, RCO_2R | Hemicelluloses |
| 1726 | Carboxylic acid RCO_2H | Lignin degradation products, muconic acids |
| 1716 | Conjugated ketones, R-CO-R | Cellulose |
| 1660–1590 | Aromatic rings | Lignin |
| 1600 and 1466 | Ethers, R-O-R | Lignin |
| 1513 | Aromatic skeleton vibration C=C | Lignin |

Table 3 FTIR absorption band assignments corresponding to functional groups relevant to wood polymers.

MJ m^{-2} of irradiation in a manner similar to that observed with the controls. The wax treatment initially protected the aliphatic functionality, but this period was followed by a rapid degradation. A linear reduction in the aliphatic band at 2950 cm^{-1} was seen in the samples treated with $\text{WAX}_{54-58^\circ\text{C}}$. Similarly, the band at 2850 cm^{-1} initially decreased rapidly followed by a period of stabilization and then continued to lose band intensity. The results are interpreted that the wax protected the aliphatic compounds of wood before the wax itself was decomposed. The $\text{WAX}_{65.5-75^\circ\text{C}}$ completely prevented the bands at 2950 and 2850 cm^{-1} from degradation.

The wax-based systems provided little protection to the essential wood polymers but did prevent the accumulation of the degradation products on the surface. The ester functionality was lost quickly from the surface of the treated samples (Figure 4c). The WAX_{hmp} led to a more rapid loss of this functional group, mainly by the deacetylation of hemicelluloses (Fabiyi et al. 2011). Conjugated ketones, associated with cellulose, normally increased on the surface as the lignin and wax are degraded indicating cellulose enrichment (Kataoka and Kiguchi 2001). The enrichment of conjugated ketones was moderated and delayed by wax or chromic acid treatments (Figure 4d). Similarly, the carboxylic acid band, associated with the lignin degradation products was not enhanced during the weathering of the wax-treated samples (Leary 1967; Anderson et al. 1991). The aromatic bands at 1513 cm^{-1} and between 1660 and 1590 cm^{-1} were lost from all the treatments with the exception of the samples treated with the $\text{WAX}_{54-58^\circ\text{C}}$. This wax formulation seemed to prevent the aromatic ring opening or its removal otherwise (Figure 4e).

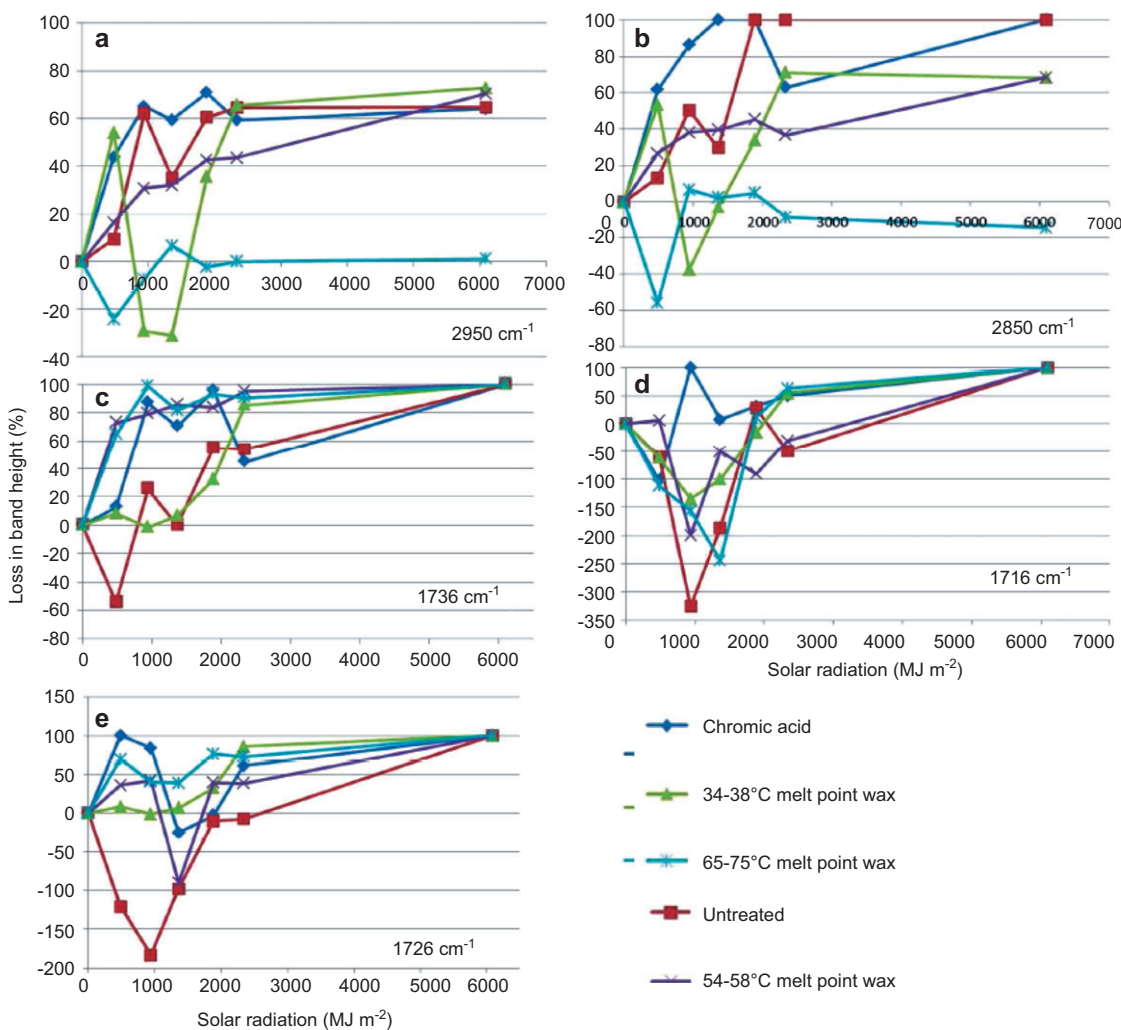


Figure 4 Changes in the FTIR band height of loblolly pine samples impregnated with different types of wax emulsions and exposed for set periods of irradiation doses in Eastern Oregon. (a) aliphatic band 2950 cm^{-1} , (b) aliphatic band 2850 cm^{-1} , (c) ester band 1736 cm^{-1} , (d) conjugated ketone band 1716 cm^{-1} , (e) carboxylic acid band 1726 cm^{-1} .

Organic UV inhibitors

The UV stabilizers neither slowed down the loss of aliphatic compounds nor did they prevent deacetylation of hemicelluloses. However, the UV stabilizer B (free radical scavenger) prevented the augmentation of carboxylic acids and conjugated ketones. This compound was especially effective in the early exposure period (Figure 5a and b). The UV stabilizer A with hydroxyphenyl benzotriazole as an active agent provided the best lignin protection among the formulations investigated (Figure 5c and d). This UV inhibitor reduced the rate of lignin loss, yet the depolymerization process did not stop; the relative abundance of cellulose on the surface and the production of carboxylic acids were increased. The effect of the free

radical scavenger (UV stabilizer B with hindered amine as active agent) was different: it mainly prevented the accumulation of the degradation products on the surface but did not slow down lignin degradation. Obviously, the combination of the organic UV inhibitors (A and B) has a synergistic effect for optimal protection.

Biocide as wood preservative

The treatment of loblolly pine with the isothiazolone (DCOIT) resulted in an accelerated loss of the aliphatic bands indicating an enhanced extractive removal. In addition, the peak deconvolution of the carbonyl region, highlighting the ester group, showed that the loss of the

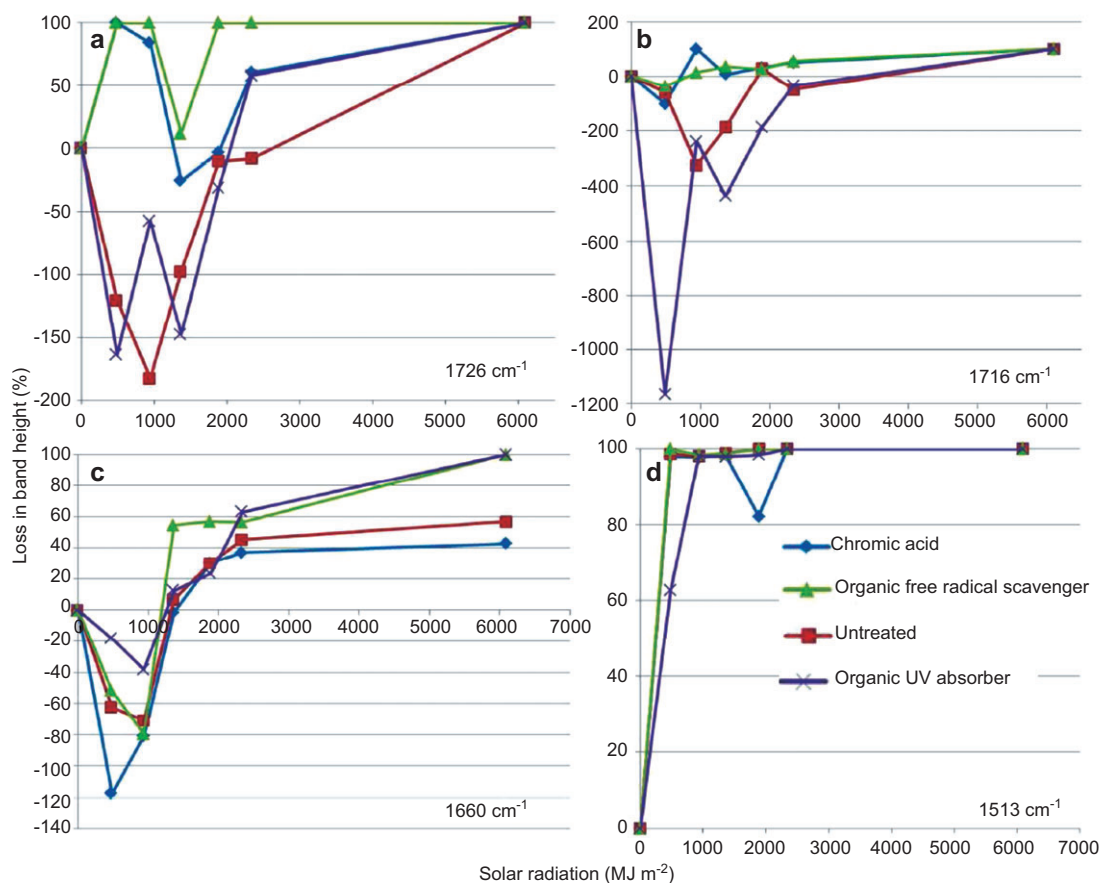


Figure 5 Changes in the FTIR band height of loblolly pine samples impregnated with different types of wax emulsions and exposed for set periods of irradiation doses in Eastern Oregon. (a) carboxylic acid band 1726 cm^{-1} , (b) conjugated ketone band 1716 cm^{-1} , (c) aromatic lignin band 1660 and 1590 cm^{-1} , (d) aromatic lignin band 1513 cm^{-1} .

acetyl groups from hemicelluloses was also accelerated compared to the untreated control. On the other hand, the biocide did not accelerate the accumulation of the lignin degradation products or the loss of lignin from the surface compared to the untreated control. This was surprising as the surface appeared bright white in color shortly after light exposure. Thus, the bleaching process prevented the formation of the brown tones normally associated with lignin degradation. In addition, mold growth inhibition on the surface by a preservative enhanced the white appearance of the resulting cellulose rich layer.

Conclusion

Rapid changes in the chemical composition of untreated wood surfaces occurred during outdoor exposure in Eastern Oregon. Treatment with different organic

compounds, notably UV inhibitors and some types of wax emulsions, retarded these chemical changes. Even with these treatments, lignin was lost from the sample surface after 1 year of outdoor exposure regardless of treatment, resulting in surface discoloration. Although some of these organic compounds applied at economically viable levels slowed the photodegradation of the wood surfaces, none of them completely prevented it. Thus, the mixtures of the compounds should be evaluated further. In addition, the chemical mechanisms of the interactions between the compounds and the wood matrix merit further consideration.

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