

## TREATMENTS TO MINIMIZE EXTRACTIVES STAIN IN WESTERN RED CEDAR

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Under certain conditions involving uneven exposure to weather, stains related to the extractives can reduce the aesthetic appeal of western red cedar in exterior applications such as fence boards, siding, and sidewall shingles. Selected chemical treatments were evaluated for their ability to inhibit the formation of extractives stain. DDACarbonate, alkyl amine oxide, and combinations thereof delayed extractives stain formation in an accelerated field test, with higher loadings having greater effect.

*Keywords:* Alkyl amine oxide; DDACarbonate; Discoloration; Extractives; Stain; *Thuja plicata*; Western red cedar; Wood products

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### INTRODUCTION

Western red cedar (*Thuja plicata* Donn) heartwood color varies from straw-colored to reddish-brown and pale chocolate-brown (Eades and Alexander 1934). The darker heartwood colors are believed to come from the condensation products of lignans, such as plicatic acid (Kai and Swan 1990; Johansson *et al.* 2000). When western red cedar is exposed to weathering, it slowly turns a silvery driftwood color. These colors are all generally considered to be visually appealing, and they contribute to western red cedar's value as an appearance-grade product.

There are several types of discolorations that can occur on western red cedar that may diminish its aesthetic appeal. Black stain fungi, such as *Aureobasidium pullulans* (de Bary) Arnaud, can colonize the surface of the weathered wood, turning the silvery surface to a dull grey (Chedgy *et al.* 2007). The use of improper fasteners can lead to the formation of grey or black iron stains, caused by the reaction of iron and phenolic extractives (Barton and MacDonald 1971). Extractives can also cause stains in the absence of metals. Many of western red cedar's heartwood extractives are water soluble. When wood is unevenly exposed to water, the wetting at one point and drying at another may redistribute these extractives. This leads to concentrated deposits of extractives that can polymerize, giving dark reddish-brown discolorations, often in an irregular line across a piece (Gardner 1965). This commonly occurs on sidewall shingles where water wicks up from the end grain and dries out through the surface. It also occurs in fences where the end grain of the fence board is held in a trough consisting of the bottom rail and battens, and the top of the board is shielded from wetting by the top rail and battens. This type of stain is commonly seen on recently installed structures and reduces the visual appeal of the product.

Coatings can prevent or obscure extractives redistribution staining. However, extractives can bleed through some wood coatings, leaving a “tannin” stain on the coating surface (Burke *et al.* 2010). Several additives and primers have been developed to address this problem (Kimerling and Bhatia 2004). Primary methods include metal-based pigments, modified functional groups, and amphiphilic block copolymers (Hart 2011). However, many of these compounds are opaque, leachable, or otherwise unsuited for application to uncoated wood. Another approach is to use chemicals to fix the extractives to the wood. Feist (1977) found improved stain resistance under coatings due to extractives fixation on wood surfaces treated with stannous chloride, zinc oxide-ammonia, chromium trioxide, acid copper chromate, and ammoniacal copper chromate. Non-metallic fixatives evaluated by Stirling and Morris (2010) without coatings were not able to reduce extractives stain. However, a carbon-based preservative reference treatment, included as an alternative method of enhancing durability, was associated with slower extractives stain formation (unpublished data). Preliminary laboratory studies indicated that didecyldimethylammonium carbonate/bicarbonate (DDACarbonate) and alkyl ammonium oxide may be able to prevent the formation of extractives stain on uncoated wood. The present work examines these chemicals for their ability to prevent extractives stain on uncoated wood in an accelerated field test. The method used differs fundamentally from standard methods that evaluate tannin stain resistance on coatings (e.g. ASTM D6686), since only uncoated wood was considered and it was longitudinal movement of extractives that was evaluated. Coating compositions containing polymeric quaternary ammonium groups have been previously reported to be resistant to extractives stain formation (Haag and Kline 1974).

## EXPERIMENTAL

### Materials

Seven nominal 1 x 4 inch (19 x 89 mm) kiln-dried second growth western red cedar outer heartwood boards were obtained from the University of British Columbia’s Malcolm Knapp Research Forest in Maple Ridge, British Columbia. DDACarbonate (Carboquat 250T®) and alkyl amine oxide (Barlox 12®) were obtained from Lonza (Fairlawn, NJ). Field test racks were constructed from untreated nominal 2 x 6 inch (38 x 140 mm) white spruce lumber (Tolko, High Level, Alberta).

### Methods

Western red cedar heartwood was cut into forty-two 300 x 90 x 19 mm test samples and randomly distributed into seven test groups, each containing six samples (Table 1). One end was sealed with three coats of a two part epoxy resin (Intergard 740, International Marine Coatings) to prevent end grain penetration. Samples allocated to treatment groups 2 to 7 were pressure-treated with the solutions indicated. The treatment consisted of a 30-minute vacuum at 95 kPa followed by two hours of pressure at 1034 kPa and a 15 minute vacuum at 95 kPa. After treatment, all samples were conditioned at 30°C for 24 hours followed by air drying. The pH of the wood surface was determined using pH indicator sticks applied to water drops placed on the surface of the boards.

A field test was designed to maximize extractives redistribution stain formation, based on observations of such stain on residential fences. The severe water trapping used in this test was intended to accelerate the formation of extractives stain. Samples were positioned vertically with the end-sealed tops placed underneath the wide face of a 2 by 6 inch (38 x 140 mm) board and the sample fastened to a 2 by 2 inch (38 x 38 mm) upper rear support board with stainless steel screws. The bottoms of the boards were placed into a shallow saw cut on the wide face of a 2 by 6 inch (38 x 140 mm) board. This created a rain water trap around the end grain of the sample. Samples were randomly placed in individual saw cuts separated from one another by approximately 30 mm to prevent extractives leaching from one sample and staining an adjacent sample.

**Table 1.** Treatment Groups for Extractives Staining Field Test

Treatment Group	Solution Strength (%)		Average Gauge Retention (kg/m <sup>3</sup> )	
	Alkyl amine oxide	DDACarbonate	Alkyl amine oxide	DDACarbonate
1	0	0	0	0
2	0.3	0	1.2	0
3	1.0	0	5.3	0
4	0	0.5	0	2.4
5	0	1.0	0	5.2
6	0.3	0.5	1.6	2.7
7	1.0	1.0	5.4	5.4

Samples were exposed from December 15, 2010 to July 18, 2011 at the field test site at FPInnovations – Wood Products Division in Vancouver, British Columbia. This site has an updated Scheffer Index of 50 (Morris and Wang 2008) and falls within the same moderate decay hazard zone as most North American cities. Racks, each containing twelve samples, had a southern exposure. Both north and south faces were evaluated for extractives stain formation.

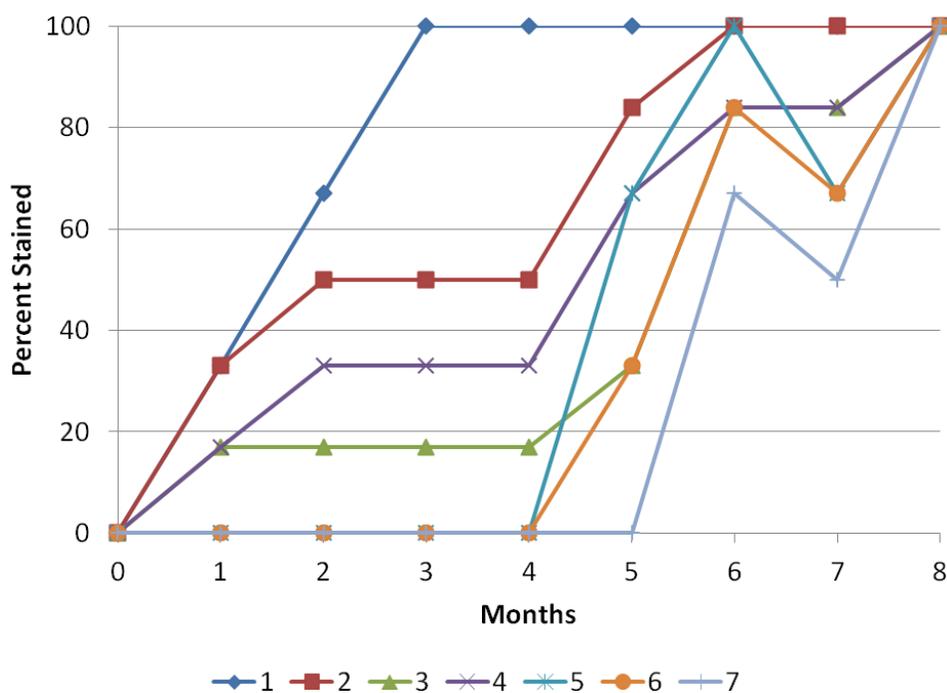
Two color measurements ( $L^*a^*b^*$ ) were made at set locations on each sample using a Konica Minolta CM-700d spectrophotometer, and photographs were taken prior to installation. After eight months of exposure, color measurements were taken at the same locations, which were unstained, as well as on the most stained parts of each board face. These data were used to calculate  $\Delta E^*$  values to indicate total color change (Equation 1).

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

## RESULTS AND DISCUSSION

The pH of all the samples before exposure ranged from 4.5 to 5. Treatment was not associated with a change in pH. Rainfall during the test amounted to 748 mm (based on data from Vancouver International Airport, approximately 10 km away). Extractives stain was found in all untreated samples after three months of exposure (Fig. 1). At this time, extractives stain was also observed in some of the samples treated with alkyl amine oxide at both concentrations, and in some of the samples treated with the low

concentration of DDACarbonate. The higher concentration of DDACarbonate and the lower concentration of DDACarbonate/alkyl amine oxide delayed the onset of stain by four months, and the higher concentration of DDACarbonate/alkyl amine oxide delayed stain onset by five months (Fig. 1). The decrease in stain observed between six and seven months was likely due to stain assessments carried out under wet conditions vs. dry conditions. After eight months, stain was present on all samples (Fig. 2). While a five-month increase in stain resistance does not appear to be a great improvement, the acceleration factor of this test is not known. Most applications would not have the highly efficient water trap used in this test. In addition, extractives stain resistance only needs to last long enough to prevent discoloration before significant weathering has occurred – perhaps one or two years. Combined with building designs that shed water, and perhaps end-sealing or coating, these treatments may be effective in reducing extractives stain for long enough to meet market demands for stain-free products.



**Fig. 1.** Percentage of samples with extractives stain in an accelerated field test

After eight months of exposure, unstained portions of the samples were lighter (higher  $L^*$ ), less red (lower  $a^*$ ), and less yellow (lower  $b^*$ ). Combined, this resulted in  $\Delta E^*$  values ranging from 11 in the untreated control (Treatment 1), up to 18 in Treatment 5 (Fig. 3). Difference in  $\Delta E^*$  between untreated samples (Treatment 1) and treated samples were statistically significant ( $p < 0.05$ ) for treatment groups 3, 4, 5, 6, and 7. The increased color change in treated samples may be indicative of more rapid weathering or less biological activity. Some untreated samples exhibited grey discolorations consistent with colonization by black stain fungi, such as *A. pullulans*. This was not present on treated samples and could have led to reduced lightening in the untreated samples.

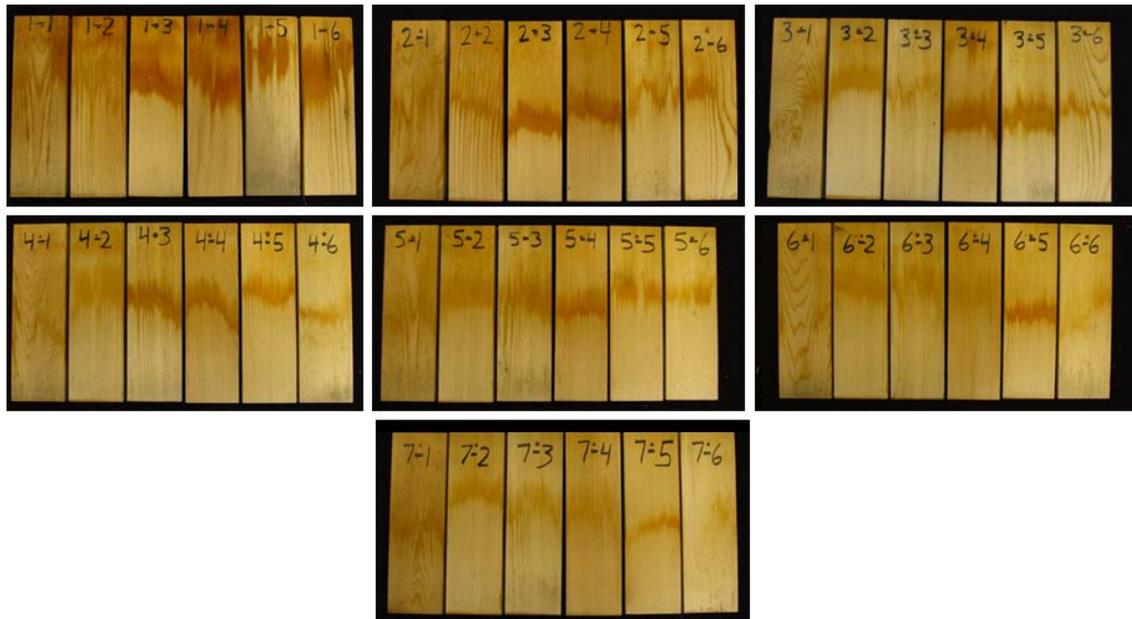


Fig. 2. Photographs of samples after eight months of exposure in an accelerated field test

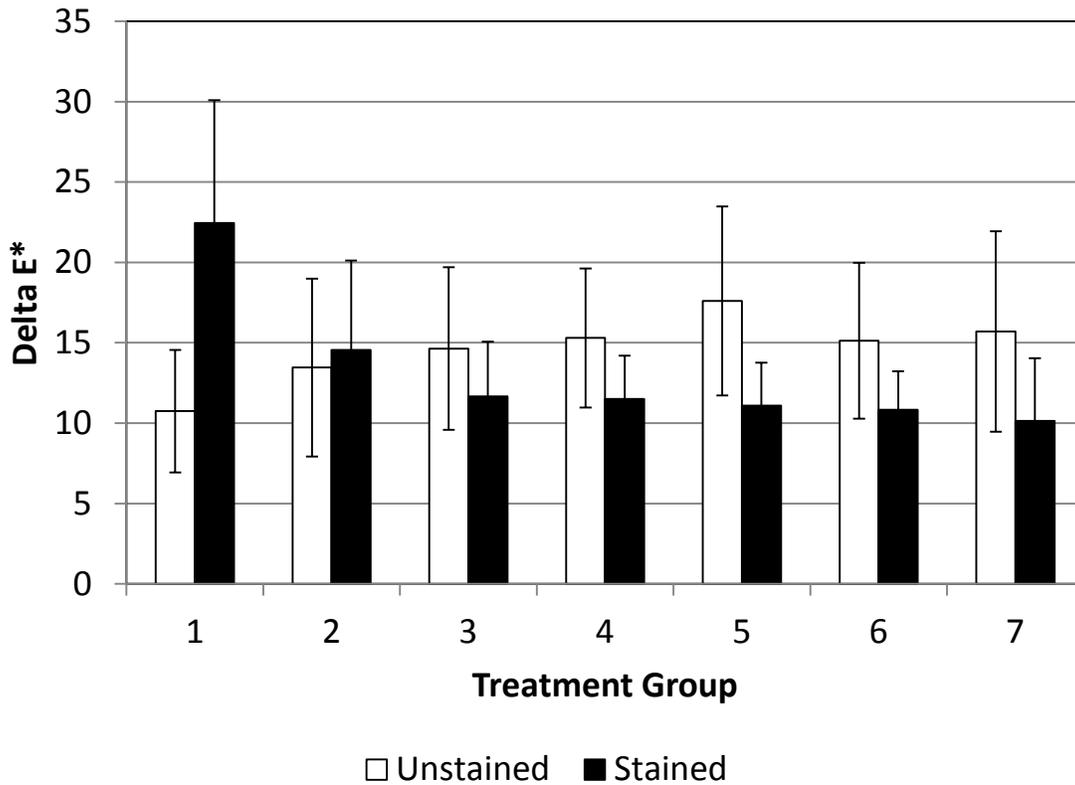


Fig. 3. Average color change in unstained and stained locations after eight months of field exposure (Error bars represent standard deviations, n = 6)

The stained portions of the samples were darker (lower  $L^*$ ), more red (higher  $a^*$ ), and more yellow (higher  $b^*$ ). This was the opposite of unstained sections and furthered the contrast between unstained and stained sections.  $\Delta E^*$  values for the untreated control averaged 22. The stain present on treated samples was less intense, ranging from 10 in Treatment 7 to 15 in Treatment 2. Difference in  $\Delta E^*$  between stained portions of untreated samples (Treatment 1) and stained portions of treated samples was statistically significant ( $p < 0.05$ ) for all treated groups. These data confirm visual observations of less intense staining in treated samples, and quantify the apparent dose response, as higher concentrations of chemicals were associated with less color change.

Quaternary ammonium cations have a strong affinity for the carboxylic acid and phenolic hydroxyl groups found in wood (Jin and Preston 1991). As a result they could interact with most of the known extractives in western red cedar that contain carboxylic acids (*e.g.* thujic acid), phenolic hydroxyl groups (*e.g.* thujaplicins), or both (*e.g.* plicatic acid). One of the risks of exposing wood treated with quaternary ammonium compounds, in the absence of UV resistant coatings or transition metals, is enhanced surface degradation of the wood (Jin *et al.* 1991). Western red cedar may be more resistant than other woods, since its extractives have been associated with improved weathering resistance of wood treated with DDACarbonate (Zhang 2003). Nevertheless, long-term field tests are needed to determine the weathering performance of western red cedar treated with DDACarbonate or alkyl amine oxide.

DDACarbonate and alkyl amine oxide are components of commercial wood preservatives (Walker 1996, 2003). In addition, alkyl amine oxides have been shown to enhance the penetration of DDACarbonate (Jiang 2008). The combination of western red cedar's natural durability and the preservative effects of DDAC treatment have also been associated with enhanced durability decay (Cabrera Orozco 2010). It may therefore be possible to produce a western red cedar product that is resistant to extractives redistribution stain and is also more durable.

## CONCLUSIONS

Treatment of western red cedar with DDACarbonate, alkyl amine oxide, and combinations thereof delay the formation of extractives stains and reduce their intensity.

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