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THE EFFECTS OF VARIOUS COATINGS ON WESTERN REDCEDAR EXTRACTIVE RETENTION

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Summary

A variety of extractives are believed to be responsible for the natural durability in western redcedar (WRC) and these can be lost through biological detoxification or leaching. A range of clear coatings were applied to WRC samples and artificially weathered with UV and water spray to test their ability to prevent the loss of extractives through leaching. Significant extractive loss occurred in the top millimetre of the weathered samples. Although extractive loss was significant in all samples, two finishes were able to provide some degree of protection: a waterborne polyurethane varnish and a water-based film-former with an alkyd varnish primer.

1. Introduction

The presence of extractives in the heartwood of western redcedar (WRC) is the most important factor contributing to the high natural durability for which it is prized. Loss of extractives results in reduced durability (Sowder, 1929). Toxicity of the extractives to decay fungi is considered to be the primary way in which extractives contribute to durability; however, they may also act as a mechanical barrier or by reducing wettability (Taylor *et al*., 2002). The thujaplicins are considered to be the major toxic constituents of WRC extractives (Rennerfelt, 1948), however thujic acid and plicatic acid also exhibit some fungicidal activity (Barton and MacDonald, 1971, Roff and Atkinson, 1954) and many extractives remain uncharacterized, both structurally and with respect to fungal toxicity. Although β- and γ-thujaplicin content does not always correlate with durability (DeBell *et al*., 1997), there is a concern that second growth WRC will be less durable because it has been reported to have reduced thujaplicin concentration (Nault, 1988). If second growth WRC has lower initial extractive content, then preventing extractive loss becomes even more important.

The loss of extractives, and resulting loss of durability, can be attributed to one or both of fungal detoxification and leaching. Colonisation of old growth trees by pioneer fungi has been shown to result in biodegradation of a broad range of extractives (Jin *et al*., 1988), and work is underway to determine whether such detoxification also occurs in wood in service (Lim *et al*., 2005). Leaching in service has been shown to result in loss of thujaplicins (Johnson and Cserjesi, 1980). It is expected that other water-soluble extractives, such as plicatic acid, would also be lost. There is thus a need for methods of enhancing extractive retention in WRC. In this report we investigate the use of coatings to prevent the leaching of extractives during artificial weathering.

Wood coatings can be grouped into two categories: film-forming and penetrating. They prevent extractive loss by blocking UV radiation, and preventing water and fungi from entering the wood. Paints are film-formers that can provide good protection; however, they do not let water escape, which can lead to blistering and peeling. Moreover paints are necessarily opaque and cover up wood's natural appearance. Varnishes are clear film-formers, but can fail rapidly from UV degradation of the underlying wood when used outdoors (Feist, 1983). Penetrating stains allow moisture to enter and exit the wood and do not form a film. Therefore they do not blister or peel (Williams and Feist, 1999). Penetrating stains often contain a water repellent, a moldicide and chemicals (pigments, UV-absorbers, hindered amine light stabilizers) that prevent UV degradation (Williams and Feist, 1999). Penetrating stains can be either oil- or water-borne. Although oil-borne stains are reported to have better performance (Morrell *et al*., 2001), increasing regulations on VOC emissions may make water-borne penetrating stains preferable. Finishing wood with a water repellent preservative is reported to significantly reduce extractive bleed (Williams and Feist, 1999a).

Coatings were selected that would not only prevent the loss of extractives, but would also be clear, so as not to detract from wood's natural appearance. A waterborne polyurethane varnish, penetrating oil and water repellent were selected from leading manufacturers, as well as a water-based film former and a water-based penetrating stain previously shown to perform better than others tested (Morris *et al*., 2004). In this accelerated experiment the effects of fungal degradation should be minimal (Groves *et al*., 2004); therefore performance will be indicative of the coating's ability to prevent leaching. The best coatings will prevent extractive loss while at the same time protecting the appearance of the wood and maintaining their integrity when weathered.

2. Methodology

Second-growth WRC outer heartwood, obtained from the Malcolm Knapp Research Forest, old-growth WRC outer heartwood grown on the Queen Charlotte Islands and Ponderosa pine from interior B.C. were cut into edge-grained 16 x 6.5 x 1 cm coupons. All wood samples were free of any visual decay or stains. Extractives were analyzed by HPLC according to the methods developed at Forintek (Daniels and Russell, 2006). Initial extractives content was determined from the average extractives content of 18 second-growth heartwood samples, 12 old-growth heartwood samples and 3 Ponderosa pine samples. All coupons were air dried and second-growth western redcedar heartwood samples were coated with the products listed in Table 1 in replicates of six.

Table 1: Coatings Applied to Second-Growth Western Redcedar

Number	Description	Number of Coats
F1	Waterborne polyurethane varnish	3
F2	Penetrating oil	3
F3	Organic solvent-based water repellent	3
F4	Water-based film former	2 - step 1, 1 - step 2
F5	Alkyd varnish + water-based film former	1 - alkyd varnish, 2 - step 2
F6	Water-based stain	3

The uptake of the coatings was measured on a dry weight basis (Table 2), since performance depends on the amount of solids left after drying, not on the amount of finish applied (Morrell *et al*., 2001). Coverage was consistent between coupons in the same treatment group but varied significantly between coatings. F1, F4 and F5 had a greater amount of dry solids applied than F2, F3 and F6.

Table 2: Average Dry Weight of Coatings on Test Face

Sample	Average Dry Solids Applied to Test Face (g/m^2)
F1	86 (10)[1]
F2	62 (6)
F3	41 (3)
F4	89 (6)
F5	97 (6)
F6	63 (8)

[1] Standard deviations are shown in parentheses

Coatings were allowed to dry for one week before being artificially weathered. An Atlas Weather-Ometer with a 65 Watt Xenon lamp with inner and outer borosilicate filters was used to weather the samples. Five coupons from each treatment group, plus untreated second-growth WRC heartwood, old-growth WRC heartwood and Ponderosa pine sapwood coupons were weathered at 40°C with 24 hour cycles of 8 hours UV, 8 hours UV plus water spray, and 8 hours only water spray (the sixth coupon remained un-weathered for comparison purposes). The average relative humidity was 63.2%, with a dry bulb of 30.2°C and a wet bulb of 22.5°C.

Coupons were monitored weekly for loss of colour and coating failure. After 1995 hours the coupons were removed and formally rated by the criteria listed in Table 3 on a scale from 1 (total failure) to 10 (perfect).

Table 3: Evaluation Methods

Evaluation		Method
Substrate condition		Subjective visual assessment
Water repellency		ASTM D 2921-98
Discoloration		Similar to ASTM D 3274-88
Finish	Flaking	ASTM D 772-86
	Erosion	ASTM D 662-93
	Cracking	ASTM D 661-93
General Rating		Average rating

Coupons were cut into three pieces and analyzed for extractives. The first 0.5 cm on either end was discarded to reduce the effects of any leaching through the end-grain. The next centimetre was ground to pass through a 20-mesh screen and used to estimate extractive content though the depth of the sample. The top millimetre of the remaining coupon was removed with a Precix 3600 router and used to estimate the extractive content in the weathered part of the sample (Chedgy *et al.*, 2005). Replicate samples were combined to provide enough sample for analysis.

3. Results and Discussion

The finish performance (Table 4, Figure 3) corresponded roughly to the average dry solids applied to the test face (Table 2). F1, F4 and F5 performed well, with average general ratings of 6.2, 7.0 and 8.4, respectively. F2 and F6 performed poorly, but better than untreated controls. The average general rating for F3 was the same as for the untreated control. F3 was also the only finish that was completely eroded; F1, F4 and F5 remained in excellent condition, while F2 and F6 exhibited some flaking and/or cracking. All uncoated samples performed poorly. All samples exhibited some surface discoloration. Mostly this consisted of uniform whitening; however, some of the F1 and F5 samples displayed patches of whitening surrounded by tobacco stain around breaches in the finish. The growth of black stain fungi on the back and ends of some of the coupons was first observed in the

Ponderosa pine coupons after 768 hours of weathering. After 1995 hours it was well established in all of the uncoated samples and on some of the coated samples.

Table 4: Finish and Substrate Ratings after Weathering

Sample	Substrate	Water repellency	Surface discoloration	Finish flaking	Finish erosion	Finish cracking	General rating
F1	10 (0)[1]	10 (0)	6.2 (1.1)	10 (0)		10 (0)	6.2 (1.1)
F2	7.6 (0.5)	7.0 (0)	3.0 (0)	8.8 (0.4)		2.4 (0.9)	3.0 (0)
F3	6.0 (0)	1.0 (0)	1.0 (0)		1.0 (0)		1.0 (0)
F4	10 (0)	10 (0)	7.0 (0.7)	10 (0)		10 (0)	7.0 (0.7)
F5	10 (0)	10 (0)	8.4 (0.9)	10 (0)		10 (0)	8.4 (0.9)
F6	10 (0)	2.6 (2.1)	2.2 (0.4)	10 (0)		8.8 (1.6)	2.2 (0.4)
Uncoated	4.8 (0.4)	1.0 (0)	1.0 (0)				1.0 (0)
OG WRC	5.8 (0.8)	1.0 (0)	1.4 (0.5)				1.4 (0.5)
P. pine	6.4 (0.5)	1.0 (0)	1.0 (0)				1.0 (0)

[1] Standard deviations are shown in parentheses

The average extractives content of the second growth and old growth WRC coupons before weathering are shown in Table 5. The old-growth heartwood sample contained significantly more plicatic acid, β-thujaplicin and methyl thujate than the second-growth sample. These data were consistent with typical WRC extractive profiles (Daniels and Russell, under review).

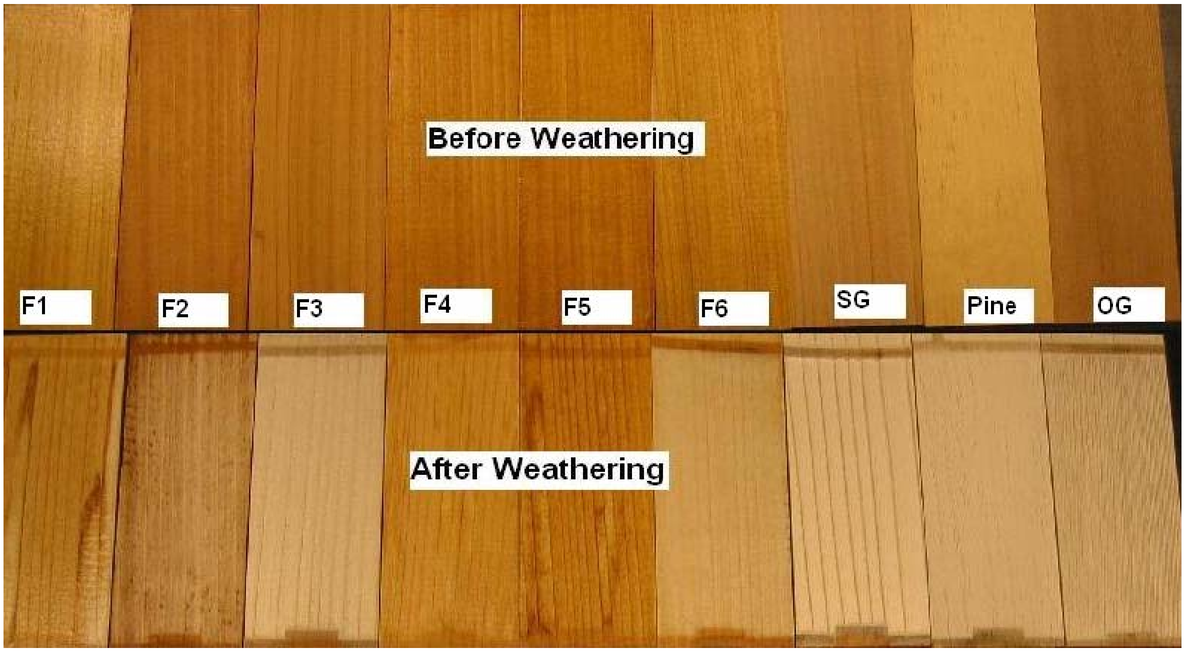

Figure 1: Photograph of Coupons before and after Artificial Weathering

For the whole cross section analysis (Table 6, Figures 2 and 3), the concentration of most extractives was not significantly affected by weathering since the weathered surface of the board did not represent enough of the coupon's volume to affect a change in the overall extractives content greater than the precision of the analytical method. However, γ-thujaplicin and thujic acid were significantly reduced in some samples. The loss of γ-thujaplicin is likely similar to β-thujaplicin, but since γ-thujaplicin was initially more abundant, losses were easier to observe and became statistically significant. The concentrations of β-thujaplicinol, plicatic acid and thujaplicatin methyl ether were significantly greater after weathering in some samples. This increase may be due to the breakdown of precursors or may simply reflect the resilience of these chemicals to weathering. The concentration of methyl thujate was significantly greater in all samples, largely because it was not detected in any un-weathered samples. This methyl thujate may be formed by methylation reactions with thujic acid. The old-growth samples exhibited significant losses of β- and γ-thujaplicin, β-thujaplicinol, thujic acid and methyl thujate. The pine samples included as weathering controls did not contain any of the quantified WRC extractives before or after weathering.

Table 5: Average Extractive Content in Samples before Artificial Weathering

WRC Heartwood	Plicatic acid	Thujaplicatin methyl ether	Gamma thujaplicin	Beta thujaplicin	Beta thujaplicinol	Thujic acid	Methyl thujate
Second growth	0.4 (0.3)	0.07 (0.05)	0.15 (0.03)	0.04 (0.02)	0.02 (0.02)	0.40 (0.06)	0 (0)
Old growth	1.1 (0.2)	0.07 (0.02)	0.15 (0.06)	0.13 (0.03)	0.08 (0.03)	0.49 (0.06)	0.08 (0.05)

To better estimate the effect of weathering on extractives concentration, the top millimetre of the coupons was removed and analyzed for extractives content (Table 7). Since only a small amount of wood was obtained from the weathered surface, the sawdust from the five replicate samples was pooled. Although the natural variation in the wood is represented in these samples, standard deviations could not be determined since only one composite sample was obtained. The error associated with the measurement of extractives in other samples provides a reasonable estimate of the error associated with these samples.

Table 6: Average Extractive Content in Sample Cross-Sections after Artificial Weathering

Sample	Plicatic acid	Thujaplicatin methyl ether	Gamma thujaplicin	Beta thujaplicin	Beta thujaplicinol	Thujic acid	Methyl thujate
F1	▨ 0.9 (0.5)	0.09 (0.04)	0.15 (0.07)	0.06 (0.05)	▨ 0.04 (0.02)	■ 0.3 (0.1)	▨ 0.02 (0.03)
F2	0.5 (0.6)	0.08 (0.02)	■ 0.12 (0.02)	0.06 (0.05)	0.03 (0.02)	■ 0.25 (0.07)	▨ 0.02 (0.04)
F3	0.6 (0.4)	0.09 (0.04)	■ 0.11 (0.05)	0.04 (0.02)	▨ 0.03 (0.01)	■ 0.25 (0.09)	▨ 0.03 (0.03)
F4	0.4 (0.4)	0.07 (0.02)	■ 0.10 (0.05)	0.05 (0.03)	0.03 (0.01)	■ 0.2 (0.1)	▨ 0.04 (0.03)
F5	▨ 0.7 (0.4)	0.10 (0.05)	0.13 (0.07)	0.05 (0.02)	▨ 0.05 (0.02)	■ 0.3 (0.1)	▨ 0.03 (0.03)
F6	0.6 (0.5)	▨ 0.11 (0.05)	■ 0.14 (0.08)	0.05 (0.03)	▨ 0.05 (0.04)	■ 0.3 (0.1)	▨ 0.03 (0.03)
Uncoated	▨ 0.8 (0.5)	0.09 (0.02)	■ 0.10 (0.04)	0.04 (0.05)	0.03 (0.03)	■ 0.2 (0.1)	▨ 0.01 (0.03)
OG WRC	1.0 (0.4)	0.06 (0.04)	■ 0.08 (0.07)	■ 0.09 (0.04)	■ 0.03 (0.02)	■ 0.2 (0.1)	■ 0.00 (0.01)

▨ Significant increase in concentration ($p < 0.05$, 1-tailed t-test)
■ Significant decrease in concentration ($p < 0.05$, 1-tailed t-test)

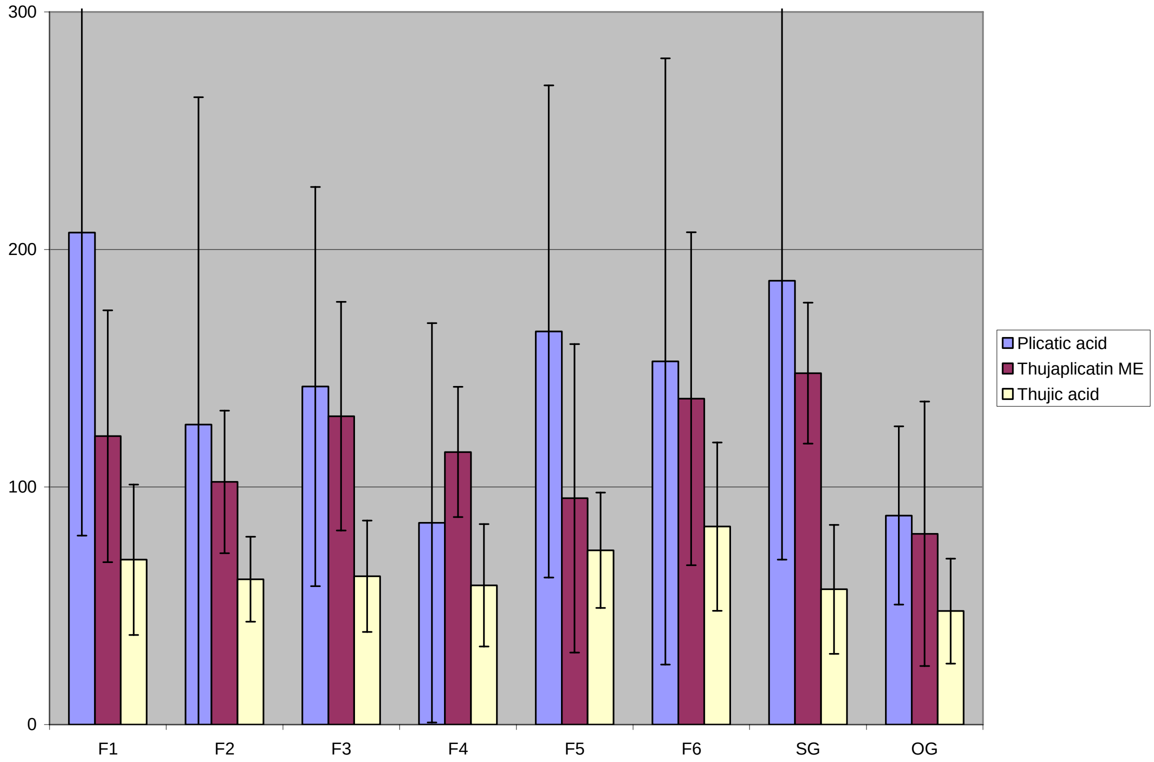

Figure 2: Percentage of Plicatic Acid, Thujaplicatin Methyl Ether and Thujic Acid Remaining in Cross-Sections of Western Redcedar Coupons after Artificial Weathering

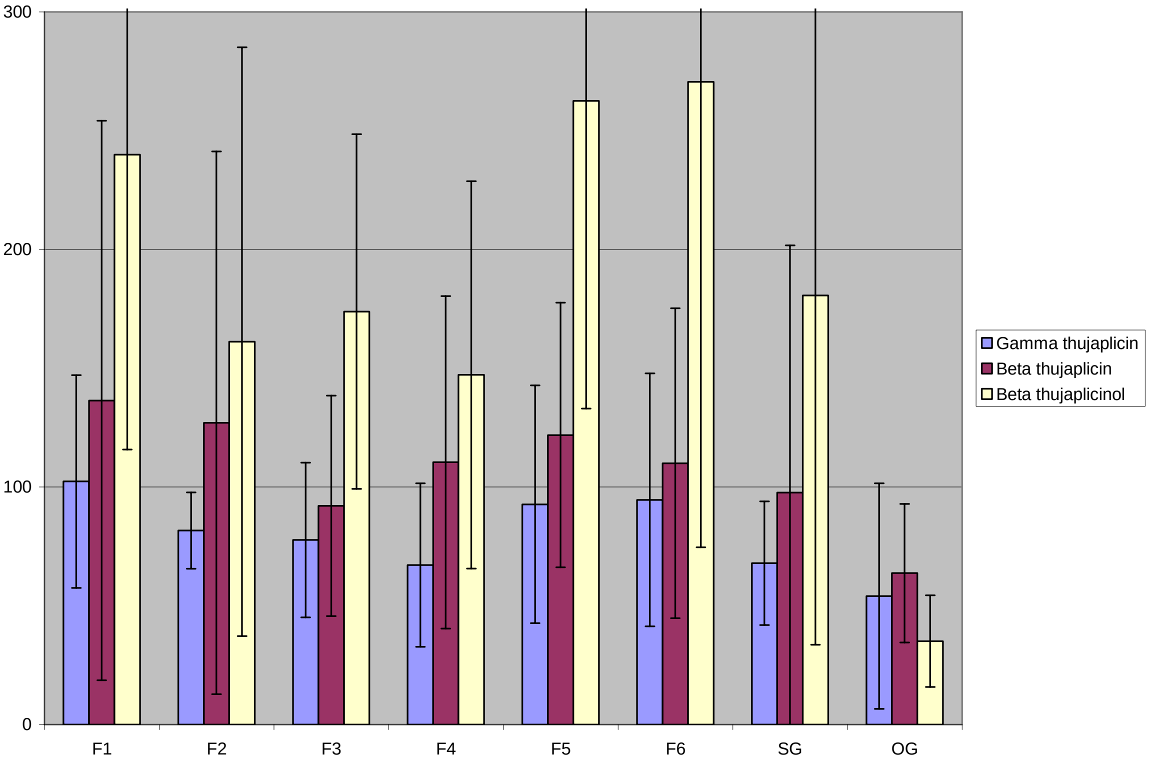

Figure 3: Percentage of Thujaplicins Remaining in Cross-Sections of Western Redcedar Coupons after Artificial Weathering

Table 7: Average Extractive Content in Top Millimetre of Samples after Artificial Weathering

Sample	Plicatic acid	Thujaplicatin methyl ether	Gamma thujaplicin	Beta thujaplicin	Beta thujaplicinol	Thujic acid	Methyl thujate
F1	0.22	0.05	0.04	0.01	0.03	0.11	0
F2	0.15	0.04	0.03	0.01	0.02	0.10	0
F3	0.10	0.02	0.02	0.01	0.01	0.06	0
F4	0.12	0.03	0.02	0.01	0.02	0.06	0
F5	0.21	0.06	0.04	0.02	0.03	0.08	0
F6	0.10	0.03	0.02	0.01	0.01	0.07	0.01
Uncoated	0.04	0.02	0.01	0	0.01	0.02	0
OG WRC	0.84	0.06	0.06	0.04	0.03	0.16	0.04
P. pine	0.01	0.01	0	0	0	0.01	0

Extractives content from the top millimetre of weathered and un-weathered samples were compared to estimate the extractives loss from the coupons (Figure 3). All extractives were lost from all groups, with the exception of β-thujaplicinol, which had highly variable extractive concentration and was more abundant after weathering in F1, F2 and F5. This was likely due to high natural variability and low concentrations. Large amounts of all other extractives were lost. Thujic acid and β- and γ-thujaplicin were most readily lost from all samples. Although most extractives were still lost, F1 and F5 were best able to prevent extractive losses. F2, F4 and F6 prevented the loss of some extractives, while F3 was only slightly better than the uncoated control.

Losses from the top millimetre of uncoated second-growth samples were very high for all extractives. Less than 10% of the initial γ-thujaplicin and no β-thujaplicin were detected. This is consistent with Chedgy *et al*. (2005) who found less than 10% of the original tropolones remained after artificial weathering with UV and water spray for 803 hours.

The old-growth control lost fewer extractives from the top millimetre than the second-growth control. Although the old-growth samples started with more plicatic acid and β-thujaplicin, initial extractive contents were similar for thujaplicatin methyl ether, thujic acid and γ-thujaplicin. Reduced losses in all of these extractives suggest that the old-growth WRC was better able to retain extractives during artificial weathering. Second-growth WRC coated with F5 retained a similar proportion of extractives to the old-growth WRC. The old-growth WRC samples had a much tighter grain than the second-growth samples, which may have helped to inhibit extractives loss in the old-growth samples. Groves *et al*. (2004) found no significant differences in the finishing properties of old- and second-growth western redcedar. Therefore, although the magnitude of effect may vary, the general ability of the test coatings to inhibit extractive loss with respect to one another should not be different for old- and second-growth WRC.

In many of the samples, the most abundant extractives after weathering, relative to un-weathered samples, were the lignans (plicatic acid and thujaplicatin methyl ether). Therefore in old, heavily weathered WRC samples, these more resilient extractives might play a greater role in durability.

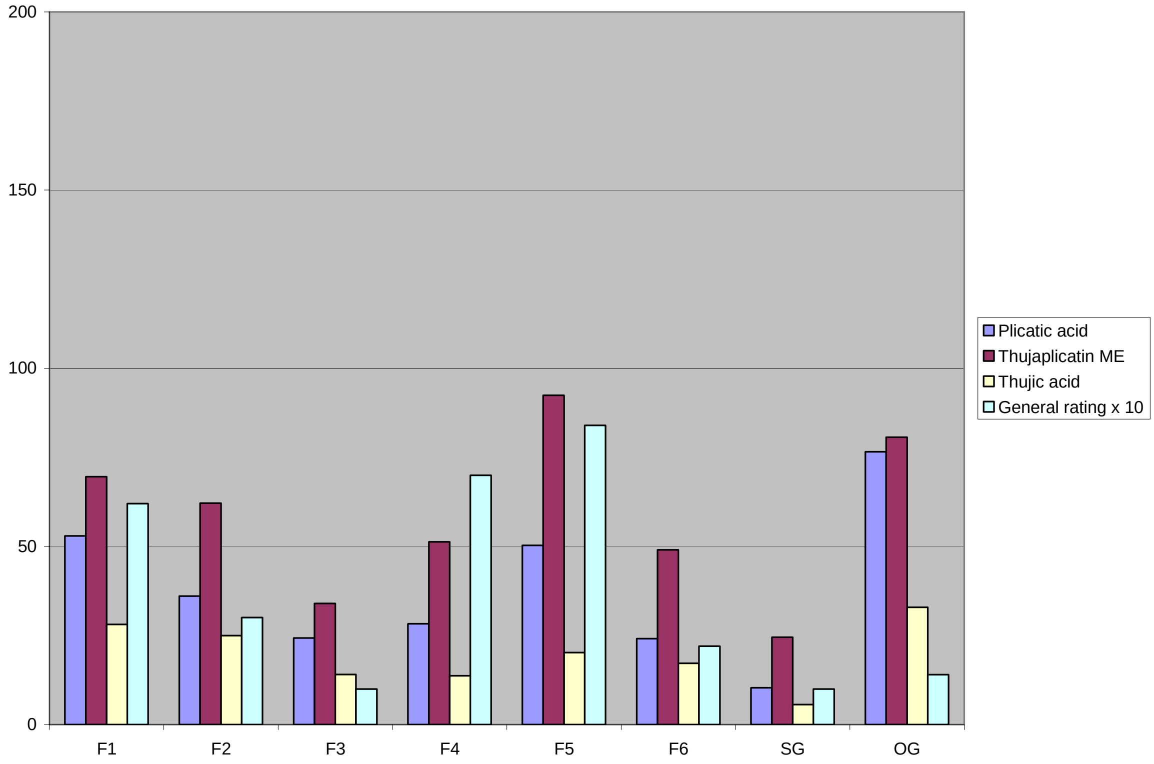

Figure 4: Percentage of Plicatic Acid, Thujaplicatin Methyl Ether and Thujic Acid Remaining in Top Millimetre of Western Redcedar Coupons after Artificial Weathering

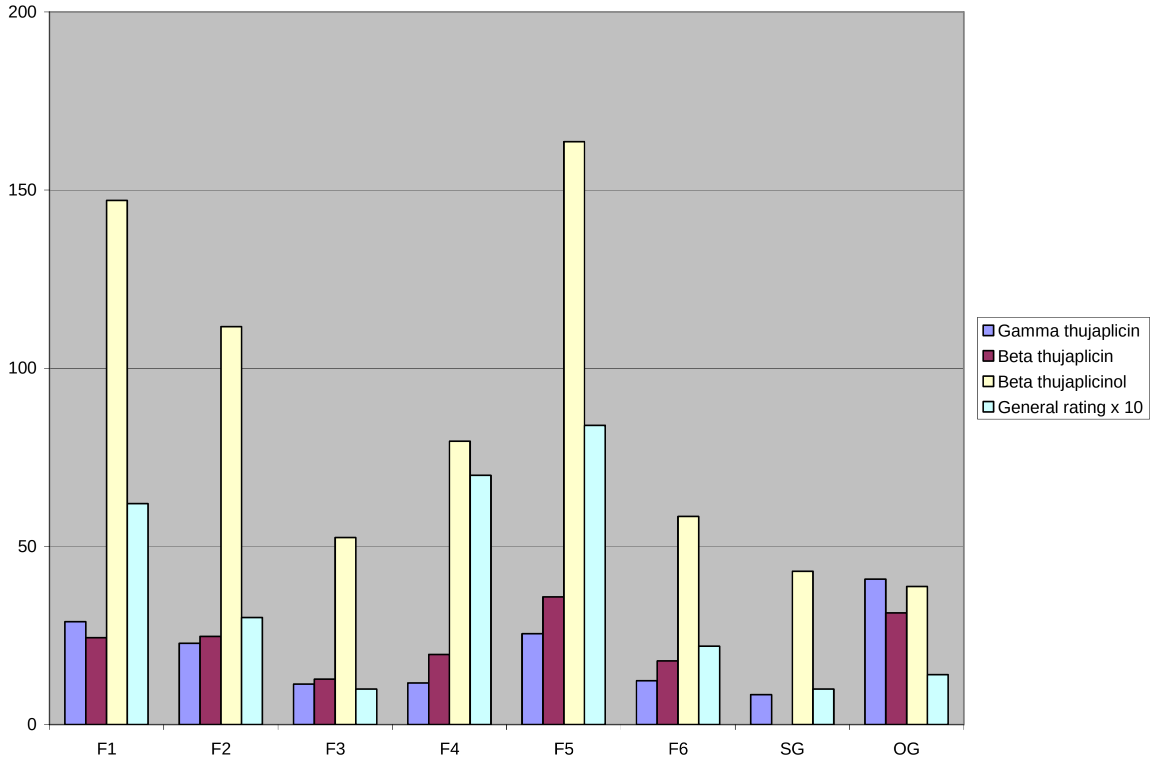

Figure 5: Percentage of Thujaplicins Remaining in Top Millimetre of Western Redcedar Coupons after Artificial Weathering

The general ratings of the coupons from Table 4 were plotted (scaled to percentages) in Figure 3 to show their relationship with extractive loss. The general ratings corresponded very well with the overall loss of extractives from the top millimetre of coupons from each group. This provides a useful indication of potential extractive loss. If the finish is failing, there will likely be a loss of extractives, which may lead to poorer durability. This underscores the importance of regular maintenance on clear finishes in order to enhance WRC's high natural durability.

Finishes provide aesthetic appeal to many WRC products and also help to inhibit the loss of extractives. Future work should evaluate the ability of coatings to prevent extractive loss in field tests. Combinations of biocides that target detoxifying fungi, and fixatives that bind the extractives to wood, should also be investigated.

4. Conclusions

Significant extractive loss occurred in the top millimetre of all samples after 1995 hours of artificial weathering with UV and water spray.

Finishes F1 and F5 were best able to prevent the loss of extractive due to leaching, and also had the highest appearance ratings.

Plicatic acid and thujaplicatin methyl ether were retained in higher proportions than the thujaplicins and thujic acid.

Old-growth samples lost a lower proportion of their extractives than second-growth samples.

5. Acknowledgements

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