U.S. Department of the Interior National Park Service

2016 Preservation Technology and Training Grant Federal Grant Number: P16AP00194

PROTECTIVE TREATMENTS FOR WESTERN REDCEDAR SHINGLE AND SHAKE ROOFS

REPORT

Washington State Parks & Recreation Commission

Prepared by

Suzana Radivojevic, PhD Principal Investigator Ligno Logic LLC suzana@lignologic.com 1 (541) 485-7991 Alex McMurry Historic Preservation Planner Washington State Parks Alex.McMurry@PARKS.WA.GOV 1 (360) 902-0930

April 15, 2018

Submitted to

Dr. Mary F. Striegel National Center for Preservation Technology and Training 645 University Parkway Natchitoches, LA 71457 1 (318) 356-7444

TABLE OF CONTENTS

Executive Summary	iii
Introduction	1
Background	1
Mechanisms of deterioration of wooden shingles and shakes	1
Protective treatments for shingle and shake roofs	2
Research objectives	3
Methods and Materials	4
Testing sites	4
Materials	6
Western redcedar shingles	6
Wood finishes	6
Test structures	8
Methods	10
Data collection	10
Water Repellency - laboratory testing	10
ATR-FTIR	11
Results and Discussion	11
Climate data	11
Field monitoring	13
Color	13
Graying	15
General condition of the finish	17
Surface growth	
Water repellency	
	20 29
AIR-FIIR	20
Conclusions	37
Acknowledgements	38
References	38
Appendix	42
••	

EXECUTIVE SUMMARY

This research study was initiated by Washington State Parks to investigate the potential of wood coatings to improve the durability and extend the service life, while maintaining the historical appearance of shingle and shake roofs. Washington State Parks oversees the maintenance and the preservation of approximately four hundred historic and modern roofs, mostly covered with locally available western redcedar (*Thuja plicata*) shingles and shakes that and rarely painted or coated with protective wood finishes. The study is motivated by the rising costs of maintaining and replacing historic roofs, combined with the declining quality of new western redcedar (WRC) stock and the lack of technical information on the performance of wood coatings for protection of WRC shingle and shake roofs.

Ten penetrating, non-film forming, traditional and contemporary, commercially available finishes were selected for the study based on predetermined set of criteria, including aesthetic appearance, service life, environmental and health risks, cost, and efficacy toward biotic and abiotic deterioration. The study encompasses the natural weathering exposure testing over the course of one year at three sites locations within the Washington State Parks, representative of major Pacific Northwest climate regions (Oceanic, Maritime and Arid climates). Field data on the performance of uncoated control and coated sample groups were collected after six and twelve months of outdoor exposure for color change, graying, surface erosion, microbial surface growth, fungal decay, water repellency, checking and cupping. Laboratory testing was performed to determine water permeability of wood coatings, and chemical changes resulting from natural weathering using ATR-FTIR.

The results demonstrate pronounced differences in the performance among test groups with respect to the climate conditions and coating formulations. The most prominent changes during the first six months of exposure were loss of color, graying, and appearance of microbial surface growth on tested shingles, with the onset of surface erosion, checking, and cupping over the course of the second six months of exposure. In general, all coatings performed equally well or better compared to uncoated groups. The strong climate effect was expressed as higher risk of UV photodegradation to uncoated wood at Arid site, and higher risk to degradation of coatings at Oceanic and Maritime sites characterized by harsh humid climates. The respective differences among individual coatings were governed by the formulation compositions. The overall best performance was observed for TWP 1500 oil-borne, and Sta Brite R water-borne formulations.

The study will be continued over the course of the second year of natural weathering exposure. to supplement long-term data on the performance of investigated coatings. The future findings will support further evaluation and consideration for their use by Washington State Parks.

INTRODUCTION

BACKGROUND

Wooden shingle and shake roofs have been one of the most common traditional roofing types in North America and continue to remain an important structural and esthetic feature on historic buildings from all periods (Sweetser, 1978). Shingles manufactured from naturally decayresistant heartwood of old growth timber stock have been reported to last more than 60 years (Park, 1989). The service life of new roofs is significantly shorter depending on the quality of shingles or shakes, roof design, installation and maintenance procedures, climate-related exposure conditions, and use of protective treatments (Niemiec and Brown, 2015).

The selection of wood species for the manufacturing of wood shakes and shingles has been traditionally based on their local availability and on the wood properties desirable in roofing material. The most commonly used wood species are those that have naturally durable heartwood, including white pine (*Pinus strobus*), white oak (*Quercus alba*), and eastern white cedar (*Thuja occidentalis*) in the North and North East, bald cypress (*Taxodium distichum*) in the South, and coastal redwood (*Sequoia sempervirens*) and western redcedar (*Thuja plicata*) in the West.

At present, western redcedar (WRC) remains one of the principal wood species utilized for the production of shingles and shakes for roofing and siding in North America. The distribution range of western redcedar extends from the Pacific Northwest along the Pacific Coast to Alaska. It is logged and manufactured mainly in Washington and Oregon in the U.S., and in British Columbia in Canada. A unique set of wood properties that makes WRC an outstanding shingle and shake material includes the natural durability of heartwood, straight grain, and low wood density responsible for exceptional thermal insulating properties and easy manufacturing. However, over the last few decades, the availability and quality of WRC shingle and shakes has been declining due to the inferior properties of second-growth timber, which is at present a main source of the material.

MECHANISMS OF DETERIORATION OF WOODEN SHINGLES AND SHAKES

The quality of shingles and shakes is critical for their longevity. Poor performance of shingles that contain sapwood (which is not decay resistant), flat grain (prone to cupping and curling), and wood defects (prone to checking) is well established (Dwyer et al., 2011). Premium-grade shingles are required to be absolutely free of wood defects and comprise only heartwood and vertical grain. The most common mechanisms of degradation that affect the appearance and lifespan of shingles and shakes are photodegradation, biological degradation, and dimensional changes.

Photodegradation is the main cause of weathering, a complex and successive process of surface degradation caused by solar radiation, water, and heat (Feist and Hon, 1984). Photodegradation is the process of chemical degradation of wood components caused primarily by the UV component of solar radiation. It is largely a surface phenomenon limited by the depth of penetration of UV radiation to approximately 75 um below the wood surface (Hon, 2001). Initial photodegradation of chromophores present in lignin, which accounts for 80-95% of the absorption of solar radiation by wood (Hon, 2001), leads to deposition of decomposition products that result in progressive color changes on the wood surface (Heinskill, 1994). In outdoor exposure, lignin degradation products and exposed cellulose fibers are subsequently removed from the surface through leaching by water and/or by the mechanical action of wind

(Derbyshire and Miller, 1981; Evans et al., 1993), leading to silvery-gray appearance and surface erosion at the final stages of weathering.

Biological deterioration of wood is caused by molds, lichens, mosses, and true wood decay fungi when the moisture and temperature conditions required for their growth are present. Molds, lichens and mosses develop only on the wood surface and are mostly of an aesthetic concern because they do not affect the strength properties and only impart the color to wood. Their prolonged presence on the wood surface can indicate, and in some instances provide, favorable moisture conditions for the colonization of true wood decay fungi. Wood decay fungi are responsible for the loss of strength and affect many physical properties of wood. Heartwood of western redcedar is naturally decay-resistant due to the presence of thujaplicins and other phenolic extractives.

Dimensional changes caused by the hygroscopic and anisotropic properties of wood comprise checking and splitting, and deformations such as cupping and curling. Checking and splitting of shingles and shakes is caused by the cyclic wetting and drying of wood in outdoor exposures. The rates of wetting and drying decrease from the exposed weathered surface toward the center of the piece, resulting in surface tensile stresses. Depending on the moisture gradient and the strength of wood perpendicular to the grain, weathered wood develops micro checks in the cell walls followed by the development of visible surface checks (Stamm and Loughborough, 1942; Schniewind, 1963). Cupping and curling are deformations caused by differential dimensional changes that are caused by the fluctuations in moisture content.

Environmental factors that affect the durability of wood shingles and shakes are those that: i) support the growth of biological decay organisms including molds, moss, algae, and decay fungi by creating favorable moisture conditions (i.e. precipitation); ii) promote surface weathering and erosion (i.e. UV/VIS solar radiation); and iii) create material stresses that lead to splitting or checking (i.e. temperature and moisture fluctuations) (Feist and Hon, 1984). The relationship between environmental factors and durability is illustrated by a service life of approximately 15 years for shingles or shakes exposed to high humidity in a coastal Pacific Northwest (PNW) environment, compared to approximately 25 years for shingles or shakes exposed to weathering at extremely dry sites in the eastern parts of Washington and Oregon.

PROTECTIVE TREATMENTS FOR SHINGLE AND SHAKE ROOFS

Historically, protective finishes have been used to increase the durability of shingles and shakes. Various formulations of finishes such as linseed oil / iron oxide mixture, or creosote / mineral pigment mixtures have been used in the past to improve water repellency and UV resistance of shingles and shakes (Sweetser, 1978). While opaque pigmented coatings, i.e. paints, are effective against UV photodegradation, clear coatings are particularly desirable due to their ability to enhance the aesthetic appearance of wood texture and color, especially on historically unpainted surfaces. Since 1950's, various formulations of water repellents and water repellent preservatives have been used for the surface coating of shingles (Park, 1989; Williams and Feist, 1999; Feist, 2006). Many of these formulations that were developed at the U.S. Forest Products Laboratory ceased to be used, following the ban on some of their main ingredients including the wood preservatives such as creosote and PCP (pentachlorophenol) (Browne, 1960).

Older formulations of surface finishes, generally described as oil-borne penetrating stains with or without pigments, biocides, UV absorbers, and water repellents, have a service life between 1-3 years (Niemiec and Brown, 2015; Williams et al., 1996). However, they have been the subject of major developments over the last decades in an effort to extend the service life and meet environmental regulations pertaining to VOC emissions and the use of organic oils, petrochemicals, and biocides. New generations of wood finishes, marketed for use on WRC

roofs, include waterborne formulations, "green" products from renewable resources (i.e. plant oils), and novel oil-borne formulations. These new formulations have improved characteristics

RESEARCH OBJECTIVES

This study is initiated by the need of Washington State Parks to investigate protective finishes to improve durability and preserve the historic appearance of shingle and shake roofs on park structures. Washington State Parks is responsible for the preservation of around 400 wood roofs on historic and modern buildings, typically made with shakes and shingles of western redcedar. Historically, surface finishes were randomly applied to western redcedar shingles and shakes produced from high-quality, naturally decay resistant, old-growth timber. However, the trend of declining quality of new shingles and shakes produced from the second-growth timber has resulted in a decrease in the service life of roofs and an increase in replacement and maintenance costs.

such as higher solids content, improved penetration, good environmental performance, and the

additional benefits of decreased flammability and ease of application.

To be considered for application on existing and new roofs, protective finishes need to be able to preserve the historic appearance of the shingle and shake roofs, not generate environmentally hazardous byproducts, have a service life of at least 3-5 years, be non-film forming, cost-effective and easy to apply, while having a broad efficacy towards deterioration mechanisms. This study tackles an existing research gap on comparative in-service performance of protective finishes for WRC shingle and shake roofs by furnishing test data that are either unavailable due to non-disclosure policies or inadequate for specific or harsh environmental exposures. The objective of this research is to generate scientifically supported technical information about the performance of pre-selected finishing formulations for western redcedar (WRC) shingles and shakes to be used by Washington State Parks and other entities for historic preservation projects. Performance of ten pre-selected penetrating, non-film forming, commercial finishes against biotic and abiotic deterioration of WRC shingles is studied in the lab and in natural weathering exposure testing, at three sites representing distinct climates and corresponding deterioration risks for WRC roofs in the Pacific Northwest. Field testing includes monitoring of weathering (i.e. color change, graving and erosion), incidence of microbial growth and fungal decay, water repellency (water spray / beading method), and dimensional and surface changes due to temperature and moisture fluctuations (i.e. checking, splitting, and cupping). Laboratory testing includes determination of water permeability of wood coatings and chemical characterization of surface changes of exposed shingles using attenuated total reflection Fourier-transform infrared (ATR-FTIR) spectroscopy (Colom et al., 2003). Properties of finishes and their performance with respect to environmental variables following the one year outdoor exposure testing will be analyzed as part of the three-year study to provide future guidance on the selection of appropriate coatings for specific environments.

The findings of this study will be applicable to shingle and shake roofs in other climate regions in North America that fall within a range of investigated climatic conditions. Considering that the roofs are the most exposed and most vulnerable building surfaces, the results of this study can guide the choice of protective treatments for other more durable or less exposed building elements of historic structures such as siding and decking, as well as for wood in other cultural resources.

METHODS AND MATERIALS

TESTING SITES

Natural weathering exposure tests (ASTM, 2013) were set up at three locations in Washington State Parks. The sites were selected as representative of three major climate regions in the Pacific Northwest: Oceanic – west of the Coastal Mountain Range, Arid – east of Cascade Mountain Range, and Temperate Oceanic (Maritime) – between the two mountain ranges (U.S. Department of Commerce, n.d.). Each site is characterized by a distinctive climate and unique set of corresponding environmental deterioration risks to WRC shingles and shakes. Oceanic exposure creates elevated wood moisture contents due to high precipitation and additional action of water spray, which increases the risk of microbial growth and decay. Cumulative action of high UV radiation, salt-water spray, and strong winds can promote excessive weathering and erosion rates. Arid exposure with low precipitation and generally high UV index places WRC shingles at above average risk of UV photodegradation and at low risk of biodegradation. Maritime exposure is characterized by high moisture and mild temperature conditions conducive to biodeterioration by mold, algae, moss, and decay fungi.

One testing site was chosen in each region respectively, based on climate-specific variables including average annual values for precipitation, temperature and UV index, and a climate index known as a "Schaeffer index" that represents aboveground decay potential for wood (Scheffer, 1971). Cape Disappointment State Park, Beacon Rock State Park, and Columbia Hills State Park, selected as sites for Oceanic, Maritime, and Arid exposures respectively, are characterized by historic climate data summarized in Table 1. The aerial images of each testing site location are shown in Figure 1 – 3. The locations of test structures are at: i) Cape Disappointment State Park: 46.2854, -124.070, elevation 6m; ii) Beacon Rock State Park: 45.6186, -122.030, elevation 11m; and iii) Columbia Hills State Park: 45.6453, -121.112, elevation 74m.

Climate region		Oceanic	Maritime	Arid
Testing sites		Cape Disappointment State Park, WA	Beacon Rock State Park, WA	Columbia Hills State Park, WA
Avg. annual Temp. ¹ (°F)	High	57	58	65
	Low	43	37	36
Avg. annual Precip. ¹ (in)		79	89	14
Avg. UV index ²	Winter	low	low	low
		1	1	1-2
	Summer	moderate-high	moderate-high	very high
		8	8	10
Schaeffer index ³		high	moderate	low
		~70	40-50	9-22

Table 1. Average annual weather conditions at the testing sites.

¹- Source: National Weather Service, U.S. NOAA; ²-Source: U.S. EPA; ³ - From: Scheffer (1971).



Figure 1. Testing site location at Cape Disappointment State Park for Oceanic exposure.



Figure 2. Testing site location at Beacon Rock State Park for Maritime exposure.



Figure 3. Testing site location at Columbia Hills State Park for Arid exposure.

MATERIALS

WESTERN REDCEDAR SHINGLES

Western redcedar shingles used for testing are No. 1 Blue label Five X (16" x 5/2"), Certigrade Red Cedar Shingles, 100% all clear, 100% heartwood, and 100% vertical grain. All shingles were trimmed to the exact width of 5" and inspected prior to treatment and installation. High variability between individual shingles was observed with respect to growth rate (number of tree rings per inch), slope of grain, and the roughness of the surface while otherwise being consistent with No. 1 Blue label Certigrade grade for western redcedar shingles. Since slope of grain and roughness can potentially affect the finish performance and durability of shingles, data sets for these two variables were included in the baseline data collection. At the time of coating application and installation, shingles were air-dry with moisture content of 12% on average.

WOOD FINISHES

Selection of finishes

Ten traditional and contemporary commercially available, clear, and lightly pigmented coatings were selected for testing after the review of compiled trade and scientific literature including Technical Data Sheets, Material Safety Data Sheets, testing results, and scientific publications. Selection of testing formulations was based on the evaluation of UV-protection and water-repellent properties, and for: being non-film forming, moisture-permeable, non-toxic, non-flammable, easy to apply, cost-effective, having long service life (as per manufacturer's warranty), and not releasing harmful byproducts during or following application. The following commercial products were selected and obtained from distributors:

1. Genuine Pine Tar 850 (Pine Tar World)

Pine tar has been traditionally used in Europe, especially Scandinavia, for the preservation of wooden buildings for many centuries and is still being used for historic buildings such as stave churches. Use of pine tar for the treatment of wooden roofs has been documented in the U.S. during the heightened production of pine tar for naval stores (Park, 1989). There has been a renewed interest in exploring pine tar as a bio-based and "traditional" alternative to coatings formulated with petrochemicals and biocides that pose potential human and environmental health risks.

Pine Tar coating is formulated with three components: pine tar (100%), gum turpentine (100%), and boiled linseed oil (100%), mixed at the volumetric ratio of 1:1:1 and applied at the ambient temperature immediately upon mixing. The finish has a dark appearance of pine tar.

Pine Tar (100%) comprises tall oil pitch, rosin acids composed of neutral fractions such as fatty alcohols and phytosterin, and a small amount of terpenes (Pine Tar World, 2014). Gum turpentine (100%) is used as a solvent and is a mixture of cyclic terpene hydrocarbons, predominantly α -Pinen (C₁₀H₁₆) (Pine Tar World, 2014a). Boiled linseed oil is supplied in pure form (Pine Tar World, 2014b).

The product is specified for general use on wood in exterior settings.

2. Chevron Shingle Oil (Chevron)

Chevron shingle oil is the only product that has been routinely used on the western redcedar shingle and shake roofs of historic buildings in Oregon and Washington.

The oil is a highly refined mixture of C15-C50 petroleum derived hydrocarbons (100%) (Alexis Oil Company, 2009).

3. Exterior Finish (Heritage Natural Finishes) (Clear)

This finish is penetrating oil that was originally developed specifically for large timbers and logs. It does not contain petroleum based products and heavy metal dryers. Exterior finish is recommended for shingle and shake roofs and all timber exposed to exterior conditions.

This finish is formulated with natural products such as linseed oil, tung oil, beeswax, pine rosin, orange oil, and undisclosed UV blocker and mildewcide additives for extended protection (Heritage Natural Finishes, 2015).

4. TWP 1500 (Natural) (Amteco)

TWP 1500 is an oil-borne penetrating semi-transparent stain formulated with mineral spirits, linseed oil, and a proprietary EPA registered biocide (Amteco, 2015). The biocide has no aquatic toxicity and does not leach from the dry finish, as per manufacturer's claims. The product has UV resistant and biocidal properties. This finish is recommended for shingle and shake roofs, log buildings, and all exterior wood. A recent study suggests outstanding performance of this finish on historic log buildings in the Grand Teton National Park (Magill, 2015).

5. Ultra Premium (Red Label) Penofin Penetrating Oil Finish (Transparent Cedar) (Penofin)

Penofin Ultra Premium Finish is a penetrating oil formulated with Brazilian rosewood oil and marketed as a new generation of plant-based oil-borne finishes. The finish is a complex mixture of petroleum based solvent, xylene, oleic acid, tall-oil, and tung oil among other components (Penofin, 2017). The finish is formulated with transoxide TiO₂ pigments, which provide 99% of UV protection, and IPBC (3-lodo-2propynylbutylcarbamate) as a biocide. Recommended applications extend to all wood in exterior settings.

6. U.V. Plus (Natural Cedar) (Messmer's)

This finish is a penetrating stain based on oragnosilane technology and is a oil-based formulation of linseed oil, alkyds, and cyclic siloxane D4 (octamethylcyclotetrasiloxane) (Messmers Inc., n.d.). It contains undisclosed fungicides and transparent UV absorbing iron oxide pigments. The finish is recommended for exterior wood.

7. Extreme Wood Stain (Crystal Clear) (DEFY)

This finish is a transparent water-borne acrylic resin, fortified with high content of UV protective trans-oxide nano particles (ZnO), a moldicide, and IPBC biocide (Saver Systems, 2015). It is recommended for wood siding, log homes, shakes and shingles, and any exterior wood application.

8. Waterproofer – Total Wood Protection (Natural) (Seal Once)

This product is a water-borne finish based on a proprietary sub micro polymer technology (New Image Coatings, 2015) that ensures reaction between wood and pre-polimerized polymer on a cellular level up to the depth of 3/8". The formulation protects against mold, mildew, cracking, warping, splitting, UV photodegradation, and fungal decay. It has a service life on shingles of at least six years, as per manufacturer's claims.

9. Log and Siding Formula (Clear) (Timber Pro)

This coating is a water-borne formulation of nano-size, bio-based plant oils, so called hydrooil (Timber Pro UV, 2016). It is recommended for exterior wood including shingles and shakes.

10. Sta Brite R (Cedar) (ISK)

Sta Brite R is a semi-transparent water-borne finish marketed as "specially formulated for lasting protection on new or restored cedar roofs." The composition is undisclosed; it contains trans-oxide pigments (TiO₂) as UV blockers and other natural pigments (Isk Biocides Inc., 2016).

Coating Application

Shingles were coated with each of the products according to manufacturer's instructions using brush application (Table 2) and left to air-dry 5-7 days before installation. All sides were coated with one single coat, and faces of shingles were coated with additional coats, per manufacturer's recommendations.

Table 2. Formulation	information for	r tested wood	finishes.
-----------------------------	-----------------	---------------	-----------

Number	Manufacturer	Product	Color	Formulation	Application
1	Pine Tar World	Genuine Pine Tar 850	Semi-transparent, Dark brown	Oil-based: Pine tar	One coat, 2nd coat on the face, 24 hours apart
2	Chevron	Chevron Shingle Oil	Clear	Oil-based: Petrolem	Two coats all sides
3	Heritage Natural Finishes	Exterior Finish	Clear	Oil-based: Plant oils	Two coats all sides, wet on wet
4	TWP / Amteco	TWP 1500	Clear (Natural 1530)	Oil-based: Petroleum, Plant oils	One coat, 2nd coat on the face
5	PENOFIN	Ultra Premium Penetrating Oil Finish (RED label)	Transparent Cedar	Oil-based: Petroleum, Plant oils	One coat, 2nd coat on the face, 2 hours apart
6	MESSMER'S	UV Plus / Natural	Natural Cedar	Oil-based: Plant Oil, Alkyd	One coat on all sides
7	DEFY	Extreme Wood Stain	Crystal Clear	Water-based: Transoxide nano particles	One coat, 2nd coat on the face, 20 minutes apart
8	Seal Once	Waterproofer - Total Wood Protection	Natural	Water-based: Nano prepolymerized polymer	One coat, 2nd coat on the face
9	Timber Pro	Log and Siding Formula	Clear	Water-based: Hydrooil	One coat, 2nd coat on the face, 24 hours apart
10	ISK	Sta Brite R	Cedar	Water-based:	One coat on all sides

TEST STRUCTURES

Test structures were built to resemble a gable roof, resting on the existing grade and elevated 2 feet from the ground, with two slopes angled at 45° facing southwest (SW) and northeast (NE) exposures that correspond to principal directions of weather effects in chosen geographic regions (Figure 4-Figure 6). The test structure at the Oceanic site is shaded from the trees on the NE side. The test structure at Maritime site has tall trees on the S and SE side and is located in forested setting. The Arid site does not have any vegetation or shade in the vicinity of the test structure.

The same layout of treated shingles was repeated on each slope, containing eleven treatment groups in vertical rows with a 1" gap between the groups. Each treatment group, as well as an untreated control group comprised five replicate samples on each exposure. Shingles were nailed with standard stainless steel ringshank roofing nails overlapping at half their length. The base structure was constructed of pressure treated 2 x 4 lumber, with 4 x 4 posts on pier blocks with galvanized brackets. The shingle nailers were utility grade cedar to prevent contact between tested products and wood preservative components in pressure treated lumber of the frame. Untreated shingles were used both as a "starter" course and for a ridge cap, simulating a typical assembly of this roofing type. Simpson ties were used for rafter connections.



Figure 4. Experimental set up at Cape Disappointment State Park. Oceanic exposure.



Figure 5. Experimental set up at Beacon Rock State Park. Maritime exposure.



Figure 6. Experimental set up at Columbia Hills State Park. Arid exposure.

METHODS

DATA COLLECTION

WRC shingles treated with selected finishes were subjected to natural weathering exposure tests modified from ASTM D1006 (ASTM, 2013). Test structures were set up during October 3-6, 2016.

Weather data were obtained from WSU AgWeatherNet, Washington State University (WSU AgWeatherNet, 2018) and the National Oceanic and Atmospheric Administration (NOAA) (NOAA, 2018) as monthly climate records for the closest weather station to each test site. Data sets were obtained for average temperature, average relative humidity, average wind direction, average wind speed, total precipitation, and total solar radiation

Data collection encompassed photographing, evaluations of color, graving, microbial surface growth, decay, checking / splitting, cupping / curling, water repellency, and general condition of the finish based on surface erosion. Initial data collection was performed at the time of the installation of test structures in October 2016, for all variables and for roughness and slope of grain. Data collection was repeated following 6 months and 12 months of natural weathering during April 6-14, 2017 and October 9-16, 2017 respectively. Surface color was assessed by visual evaluation of the color of coated shingles relative to unexposed control samples on the scale from 10 (no changes in initial color) to 6 (no initial color) (Morrell et al., 2001). Surface graving was assessed by visual evaluation of the graving of coated shingles relative to control unexposed samples on the scale from 10 (no changes in initial tone) to 6 (completely gray). Microbial surface growth (i.e. mold, mildew) was assessed visually and rated from 10 (no evidence of growth) to 6 (surface completely covered with growth). Decay assessment was done visually based on ratings of AWPA Standard E10 (AWPA, 2010) and rated from 10 (no evidence of decay) to 6 (complete decay and loss of structural integrity of wood). Checking and splitting was evaluated visually and rated on a scale 10 (no checking) to 6 (heavy checking and splitting). Cupping and curling was evaluated on a scale of 10 (no cupping or curling) to 6 (heavy cupping or curling). Water repellency in situ was assessed by evaluating water beading following the spraying of the shingle surface and rated from 10 (rounded water beads) to 6 (complete wetting) (Morrell et al., 2001). General condition of the finish was assessed based on the erosion of the surface and visually rated on a scale from 10 (intact) to 6 (no evidence of the finish). General condition of the wood surface on uncoated control group was evaluated based on the amount of weathering erosion that is indicated by the exposure of new wood under weathered gray surface and rated on a scale from 10 (intact) to 6 (completely eroded).

The initially proposed rating system on a scale of 1 to 10, was replaced by a scale of 6 to 10 because the scale based on 10 ratings was too sensitive to be used accurately with visual evaluation of investigated properties.

WATER REPELLENCY - LABORATORY TESTING

Determination of water repellent properties of coatings was performed in laboratory following the gravimetric test method modified from the ASTM D5401-03 standard (ASTM, 2013a). Water repellent properties of coatings are described by water absorption over 30 minutes of water immersion, and as the water repellent efficiency of coatings relative to uncoated controls (Temiz et al., 2006). Testing was performed for all ten investigated coatings and for an uncoated control group. Each of the eleven groups consisted of five replicate specimens. Shingles were coated following the application methods used for outdoor exposure testing, according to manufacturer's directions. All samples were conditioned at the temperature of 21 ± 3 °C and relative humidity of $36 \pm 5\%$ to establish uniform moisture content by daily monitoring of weight change until the difference between two successive weight measurements was less than 0.1 g

for each individual specimen. All samples were weighed and each group of five coated shingles was transferred to a separate container filled with up to 2" of distilled water. Shingles were allowed to float for 30 minutes and were turned after 15 minutes. Following the 30 min water immersion, shingles were removed from the container and the excess water was wiped off using paper towels. The specimens were reweighed using balance with accuracy of 0.01 g.

Water absorption (WA) was calculated for each specimen as follows (Eq 1):

 $WA = 100 \times [(M_2 - M_1) / M_1] (\%)$ (1)

where:

 M_1 = weight of the specimen before the water contact

M₂ = weight of the specimen after water contact

Water absorption for each coating and uncoated control is calculated as the mean WA for each five-specimen set and expressed in percentages.

(2)

Water repellent effectiveness of coatings was calculated for each coating as follows (Eq 2):

WRE = $100 \times [(WAC-WAT)/WAC]$

where:

WAC = average water absorption of control (uncoated control) group

WAT = average water absorption of coated group

ATR-FTIR

Chemical changes on the uncoated control and the coated shingles that resulted from the weathering exposure were investigated by ATR-FTIR. The FTIR spectra were obtained on a Thermo Scientific Nicolet 6700 FT-IR spectrometer fitted with a Smart iTR Attenuated Total Reflectance (ATR) accessory with a diamond crystal. Infrared spectra were collected in the wavelength range from 4000 cm⁻¹ to 400 cm⁻¹as an average of 32 scans at a spectral resolution of 2 cm⁻¹. The bands in the FTIR spectra were assigned based on data available in the literature.

Samples for ATR-FTIR analysis were collected from uncoated control, from coated unweathered shingles, and from one shingle for each treatment from the SW elevation for each exposure, following 12 months of weathering. Samples were extracted using razor blades to produce approximately 0.5 mm thick, 7 x 7 mm sections from the shingle surfaces.

RESULTS AND DISCUSSION

CLIMATE DATA

Weather data for investigated locations were obtained as monthly records from the National Oceanic and Atmospheric Administration (NOAA) (NOAA, 2018) for average temperature, mean maximum and minimum temperature, and total precipitation, and from the WSU AgWeatherNet, Washington State University (WSU AgWeatherNet ,2018) for average relative humidity, average wind direction, average wind speed, and total solar radiation. Data used for Cape Disappointment State Park (Oceanic exposure) were obtained from the Long Beach Experiment Station, WA US USC00454748 (NOAA), and WSU Long Beach (AgWeatherNet) weather stations located 4 miles north of the Cape Disappointment testing site. Data for Beacon Rock

State Park (Maritime exposure) were obtained from Bonneville Dam, OR US USC00350897 (NOAA), and Stevenson WSU (AgWeatherNet) weather stations located 4 miles to the East, and 8 miles to the West from the testing site, respectively. Data used for Columbia Hills State Park (Arid exposure) site were obtained from the Dallesport Airport, WA US USW00024219 (NOAA), and WSU Maryhill (AgWeatherNet) weather stations located 12 and 3.5 miles from the testing site respectively.

Monthly climate data were used to derive average values for the 1st phase of testing (from October 2016 to March 2017), the 2nd phase of testing (from April 2017 to September 2017), and for the cumulative 12 months between October 2016 and September 2017 (Table 3).

Table 3. Overview of climate data for test sites for the period of testing of one year. Data for Avg T, Mean T max, Mean T min, and Total Prec obtained from NOAA weather records (NOAA, 2018), and Avg RH, Avg Wind Direction, Avt Wind Speed and Total Solar Radiation were obtained from WSU AgWeatherNet (WSU AgWeatherNet, 2018).

Site climate	Phase	Time period	Avg T (°F)	Mean T max (°F)	Mean T min (°F)	Avg RH (%)	Avg Wind Direction (°)	Avg Wind Speed (mph)	Tot Prec (in)	Solar Radiation (MJ/m²)
OCEANIC	1 (1-6 months)	10/2016 - 3/2017	44.3	50.2	38.4	88.3	S - SW	3.3	83.77	
	2 (6-12 months)	3/2017 - 9/2017	55.7	60.4	49.8	84.8	SW-W	2.3	22.29	3269
	YEAR 1	10/2016 - 9/2017	50.0	55.3	44.1	86.2	S-SW-W	2.7	106.06	
MARITIME	1 (1-6 months)	10/2016 - 3/2017	41.9	46.6	37.2	85.6	N - NW	3.5	81.63	875
	2 (6-12 months)	3/2017 - 9/2017	62.8	73.4	52.2	66.2	SW-W	3.0	19.59	3394
	YEAR 1	10/2016 - 9/2017	52.4	60.0	44.7	75.9	N-NW-W-SW	3.2	101.22	
ARID	1 (1-6 months)	10/2016 - 3/2017	40.4	47.5	33.3	81.0	NW-W	4.8	12.28	1292
	2 (6-12 months)	3/2017 - 9/2017	67.8	81.2	54.4	49.5	SW-W	6.9	1.60	4194
	YEAR 1	10/2016 - 9/2017	54.1	64.3	43.8	65.3	S-SW-W	5.9	13.88	

Average annual temperature was highest at the Arid testing site at 54.1 \degree F, followed by 1.7 \degree F lower average annual temperature at the Maritime site (52.4 \degree F) and 4.1 \degree F lower at the Oceanic site (50.0 \degree F). A similar trend of decreasing average temperature between sites was observed for the second phase with 5 \degree F higher average at Arid than the Maritime site, and 7.1 \degree F higher average at Maritime than Oceanic site. In contrast, the average temperatures decreased from Oceanic to Maritime by 2.4 \degree F, and from Maritime to Arid site by 1.7 \degree F during the winter months of the 1st testing phase. The largest differences among sites were the mean maximum temperatures during second testing phase, which increased from 60.4 \degree F for Oceanic to 73.4 \degree F for Maritime and to 81.2 \degree F for Arid site.

The Oceanic and Maritime sites received high amounts of total precipitation of 106" and 101.2" respectively, with Oceanic site receiving on average 2" to 3" higher amount than Maritime site over the course of each testing phase and over the whole year. Most precipitation occurred during the 1st testing phase (winter) at all three locations. Average relative humidity was highest at the Oceanic site and decreased between the Oceanic, Maritime and Arid sites for each investigated time period. Arid site received only 12.3" of precipitation for the whole year, and only 1.6" during the spring and summer months of the 2nd testing phase.

Monthly UV index records were not available for investigated locations. The composition of the maximum amount of solar radiation of 1,000 W/m² available at the earth's surface on a clear day is approximately 5% UV (286–380 nm), 45% visible (380–780 nm), and 50% IR (780–3,000 nm) (Evans et al., 2002). Data for total solar radiation (Table 3) were used for approximations of

13

UV radiation at test sites. Solar radiation during the 2nd testing phase was considerably higher at the Arid site than at other two sites. The Maritime site received most shade from the surrounding vegetation, followed by the Oceanic site, while the Arid site did not have shade.

The Maritime and Arid sites also received heavy snow during the 1st test phase, however the weather records for snowfall were not available. The weather data and respective differences among all three locations are consistent with historic data (Table 1).

FIELD MONITORING

COLOR

Color change was one of the most significant outcomes of natural weathering exposure at all investigated sites and for all treatments, with the exception of TWP 1500 which sustained no color change at any sites and for Sta Brite R, which did not have color change at the Arid site (Figure 7). All other coated and uncoated control groups had significant loss of color at the end of the 1st phase, with further changes taking place for most groups over the 2nd phase of natural weathering exposure.



Figure 7. Average ratings and standard deviation (SD) for color change for individual test groups for Oceanic (O), Maritime (M), and Arid (A) exposures for SW and NE elevations.

Most rapid color loss was observed in untreated control groups (Figure 8.). They completely lost the natural color of western redcedar after six months of weathering (1st phase) at Arid exposure, followed by Maritime and Oceanic exposures where the color ratings were higher at the end of the 1st phase, but further decreased over the 2nd phase to near complete loss (Figure 7a).



Figure 8. Comparisons for color and graying between uncoated control shingles (white label) and unweathered control following 1st and 2nd outdoor exposure phases for NW and SE exposures at three testing sites.

Oil-borne coatings had differing rates of color loss; Chevron and Heritage had the lowest ratings of 6 by the end of the twelve-month exposure at Oceanic and Maritime sites, and somewhat higher ratings of 8 for Chevron and 7 for Heritage at Arid site (Figure 7 c, d). At all three sites, color ratings of Pine Tar treatments decreased over time, resulting in the final ratings after the 2nd phase of 6 at the Oceanic site, 7 at the Maritime site, and between 7 and 8 at the Arid site (Figure 7 b). Color change of Penofin and Messmer's treatments followed very similar trends, with progressive color change from 9 for the 1st phase to 7 for the 2nd phase at the Oceanic and Maritime sites (Figure 7 e, f). At Columbia Hills, the color ratings decreased to 9 over the 1st phase and did not change over the 2nd phase of natural exposure. No significant color changes were observed for TWP 1500 treatments for any test groups (Figure 7 g). It appears that the most severe climate effects on color retention occurred at the Oceanic site, followed by Maritime and Arid sites over both investigated periods: winter months of the 1st phase and summer months of the 2nd phase. No consistent differences between SW and NE elevations were observed for color change.

These results suggest different effects of climate on the mechanisms responsible for color loss in uncoated control groups, which were more sensitive at the dry Arid site when compared to groups coated with oil-borne coatings, which had more pronounced changes at the humid Oceanic and Maritime sites.

Color ratings of water-borne coatings also generally decreased over time. DEFY completely lost the initial color of the coating by the end of the 12-month exposure at the Oceanic and Maritime sites (rated 6), but after initial rapid color loss (rating 7) at the Arid site during 1st phase, no change was observed after the 2nd phase. Color of Seal Once was somewhat more stable than in DEFY, reaching average ratings of 7 for test groups at the Oceanic and Maritime sites and an average rating of 8 at the Arid site. Final ratings for Timber Pro were between 6 and 7 for the Oceanic and Maritime sites and 7 for the Arid site. Sta Brite R had the best color retention among water-borne coatings, with a progressive decrease to average rating showen 8 and 9 at the Oceanic site, 9 at the Maritime site, and no color loss (rating 10) at the Arid site (Figure 9). It appears that the effects of climate on color retention were similar and pronounced at the Oceanic and Maritime sites when compared to Arid site. This exposure difference was especially evident over the course of the 2nd phase, when little or no change in color of water-borne coatings was observed at the Arid site. In general, there were no differences in color change between the test groups on SW and NE elevations of test structures.



Figure 9. Test shingles coated with Sta Brite R at NW and SE elevations at three testing sites following the 1st and 2nd phase of outdoor exposure.

<u>GRAYING</u>

Mean ratings for graying of shingle surfaces are presented in Figure 10. In general, the ratings for graying in general follow similar trends to those observed for color change for investigated treatment groups. Graying of untreated control groups was most prominent at the Arid exposure, followed by Maritime and Oceanic exposures after the 1st phase, reaching low ratings of 6 and 7 at all sites by the end of the 2nd phase (Figure 10 a).

Groups coated with oil-borne coatings (Pine Tar, Chevron, and Heritage) performed similar to uncoated control groups, with final ratings of 6 and 7 at the Oceanic and Maritime exposures at the end of the 2nd phase (Figure 10 b, c, d). However, the extent of graying of these groups was lower at the Arid site when compared to the same coatings at the other two sites and the uncoated controls at the Arid site. Oil-borne Penofin and Messmer's had similar performances

with the most severe graying at the Maritime exposure, followed by Oceanic exposure and no apparent graying at Arid exposure following the 2nd phase, and with no apparent graying following the 1st phase at either site (Figure 10 e, f). TWP 1500 retained the maximum rating of 10, indicating no graying of the surface for any test groups on all three testing sites. TWP 1500 had the best performance of all investigated treatments (Figure 10 g).



Figure 10. Average ratings and standard deviation (SD) for graying of the shingle surface for individual test groups for Oceanic (O), Maritime (M), and Arid (A) exposures for SW and NE elevations.

Among the groups coated with water-borne coatings, best results were obtained for Sta Brite R groups, which exhibited low levels of graying at Oceanic and Maritime sites and no graying at Arid site (Figure 10 k). Seal Once also performed well at Arid site, with low average ratings of 9 on both elevations at the end of the 2nd phase and no change in graying between two phases; graying was more pronounced at Oceanic and Maritime sites and occurred mostly over the 2nd phase, reaching a final average rating of 7 (Figure 10 i). DEFY-coated groups reached the lowest average graying rates of 6 at Oceanic and Maritime sites and 7 at Arid site (Figure 10 h). For water-borne coating groups, most of the change in graying occurred over the 2nd phase at Oceanic and Maritime sites.

In general, all coatings had same or better ratings for graying than the uncoated control. Less graying of all treatment groups occurred at the Arid than at Oceanic and Maritime sites. Several treatment groups (Chevron, Heritage, Penofin, and Messmer's) had higher ratings after the 2nd

phase than after 1st phase at Arid site. This trend was been observed at the time of the 2nd data collection and confirmed based on the photographs, but the reasons for these trends are not clear.

GENERAL CONDITION OF THE FINISH

General condition of the finish was based on the erosion of the finish in coated samples and on the surface erosion of the uncoated control groups. Erosion was apparent on the uncoated control and coated wood surfaces (Figure 11) following the 2nd phase of outdoor exposure (April to October 2017), while no erosion was observed after the 1st phase (October 2016 to April 2017).



Figure 11. Surface erosion on uncoated control and Heritage and DEFY coated shingle. Maritime site after 12month outdoor exposure.

Surface erosion on uncoated control shingles resulted from weathering of wood caused by the combined action of UV radiation, precipitation, and wind (Schniewind, 1963). In contrast, erosion observed on samples coated with oil-borne coatings resulted from the failure of coatings caused by similar environmental factors to those responsible for weathering of wood that was, in some instances, followed by the weathering of unprotected wood. The erosion pattern was somewhat different on surfaces coated with water-borne coatings appearing to be caused by mechanical action.

Most extensive erosion was observed at the Maritime site. It affected, to varying extents, the untreated control and Chevron, Heritage, DEFY, Seal Once, and Sta Brite R treatments on both elevations. While the uncoated control shingles were almost completely eroded, coated groups had a mean rating of 9 and above (Figure 12). Erosion was not observed for Pine Tar, TWP 1500, Penofin, Messmer's, and Timber Pro treatments. At the Oceanic site, erosion was recorded only on untreated controls, Heritage, and Sta Brite R coated groups. At the Arid site, only untreated controls sustained erosion.

On all test sites and elevations, the highest amount of erosion was observed on uncoated control shingles. Among the test sites, the most severe effects in terms of the amount of erosion and the number of coatings that sustained damage was observed at the Maritime site with six affected test series, followed by the Oceanic site with three affected test series, and the Arid site with only the uncoated control group showing erosion. No consistent trends in the extent of surface erosion were apparent between SW and NE elevations at any sites.



Figure 12. Mean ratings and standard deviation (SD) for surface erosion for control (O), Chevron (C), Heritage (H), DEFY (D), Seal Once (S), and Sta Brite R (B) (first row of category labels on X axis) following 12-month outdoor weathering for southwest (S) and northeast (N) exposure (second row of category labels on X axis) at Oceanic (O), Maritime (M) and Arid (A) sites (third row of category labels on X axis).

SURFACE GROWTH

Surface growth was observed on some, but not all, treatments following the 1st and 2nd phase of outdoor exposure. Two general types of surface growth were distinguished. The first type appears to be black mold and was the most frequently observed form of surface growth. The second type was the green growth that is usually associated with algae and mildew and was only observed on uncoated control samples at the Maritime site (Figure 13).



Figure 13. Appearance of green growth on the surface of uncoated control shingles and of black mold on shingles coated with Pine Tar and Seal Once.

The results for all three test sites are presented in Figure 14. In many instances, the amount of surface growth decreased over the 2nd phase from the 1st phase. This can be explained by generally less favorable conditions for the biological growth during warm and dry summer months, depletion of soluble nutrients from wood and oils in coatings required for the mold growth, and by the effect of weathering which seemed to cause removal of mold along with

wood fibers from the surface. Decrease in the mold growth was especially noticeable for controls and oil-borne coatings such as Chevron, Heritage, Messmer's, and Penofin.

Among the three sites, most biological growth was observed at the Maritime site (Figure 14b). The least amount of surface growth was observed on test groups treated with oil-borne TWP 1500 and water-borne Sta Brite R, while the highest amount of surface growth was observed on untreated controls. With the exception of TWP 1500, all oil-borne coatings had considerably low ratings but no consistent differences were apparent. Among water-borne coatings the least amount of mold developed on shingles coated with Sta Brite R, followed by DEFY, Seal Once and Timber Pro. Over the second testing phase during the summer months, all samples coated with Timber Pro developed patchy black mold with a different appearance than that observed on other samples.

A decrease in the amount of surface growth over the second testing phase was especially apparent at the Oceanic site, where most coatings had only traces of surface growth following the 2nd phase of natural exposure. However, the Timber Pro and Seal Once coated groups developed patchy black mold pattern which was more pronounced on the Timber Pro than on the Seal Once coated shingles (Figure 13, Figure 15).

At the Arid site, no surface growth was observed on shingles coated with TWP 1500, DEFY, and Sta Brite R, and no surface growth was apparent on shingles coated with Penofin and Mesmer's following the 2nd testing phase. Shingles treated with Seal Once had a dense spotty appearance of black mold (Figure 13). Ratings for oil-borne coatings Pine Tar, Chevron, and Heritage were consistently lower following the 2nd testing phase at the Arid site.

There were no significant differences in the amount of surface growth between SW and NE elevations on either site.





Figure 14. Average values and SD for surface growth on three testing sites for uncoated control (O), Pine Tar (N), Chevron (C), Heritage (H), TWP 1500 (T), DEFY (D), Seal Once (S), Timber Pro (U), and Sta Brite R (B).



Figure 15. Surface growth on shingles coated with Timber Pro after 1st and 2nd testing phase respectively for three exposures.

SURFACE CHECKING

First occurrences of surface checking on tested shingles were observed after 12 months of outdoor exposure. Most checks appeared as hairline checks (Figure 16) of different lengths along the grain. The largest number of test groups that developed checks was observed at the Oceanic site (DEFY, Seal Once, Sta Brite R, Pine Tar, and Chevron), followed by the Maritime site, where checking was observed on test groups coated with DEFY and Sta Brite R, and the Arid site, where checking was observed on Timber Pro and Sta Brite R coated test groups (Figure 17). Many of the test groups had checking develop only on one out of five samples.



Figure 16. Examples of surface checking following 12-month outdoor exposure.

While no apparent trends were observed between SW and NE elevations or between individual coatings, the results suggest different performances between the groups of water-borne and oilborne coatings. At least one occurrence of checking was observed for all investigated waterborne coatings (DEFY, Seal Once, Timber Pro, and Sta Brite R). In total, 16 samples coated with water-borne coatings developed checks while only two samples coated with oil-borne coatings (Pine Tar and Chevron) developed checks. Among the water-borne coatings, samples coated with Sta Brite R were most prone to checking (6 samples), followed by Timber Pro (5

22

samples), DEFY (4 samples), and Seal Once (1 sample). In addition, only Sta Brite R had checking on at least one sample at all three testing sites. No checking was observed on control samples. The possible effect of surface roughness and slope of grain on the incidence of checking was also considered, but there was no correlation and the results are not shown here. The results suggest a higher tendency of shingles to develop checks when they are coated with water-borne coatings, rather than with oil-borne coatings or when uncoated.



Figure 17. Mean ratings and standard deviation for surface checking following 12-month outdoor exposure at Oceanic (O), Maritime (M) and Arid (A) site, for southwest (S) and northeast (N) exposure for DEFY (D), Seal Once (S), Sta Brite R (B) Pine Tar (N), Chevron (C), and Timber Pro (U).

CUPPING

Cupping was not observed on any samples following the 1st phase of outdoor exposure. However, almost all test groups had at least some samples that exhibited cupping following the 2nd phase, which occurred between April 2017 and October 2018 (Figure 18). The lowest observed rating for cupping was 8 for control samples and most coated samples had only a small amount of cupping rated at 9.

At the Oceanic and Maritime sites, all coated groups performed equally or better than the uncoated control groups and on average exhibited less cupping when compared to control groups (Figure 18). Cupping was also more prominent on SW exposures at both sites, based on an independent-samples t-test conducted to compare cupping at SW and NE elevation (Table 4). At 95% significance level, the difference at the Arid site was not significant, indicating no difference between the amount of cupping at two elevations. Difference in cupping for water-borne coating groups between SW and NE exposures was more pronounced than for oil-borne coatings and contributed most of the observed difference between the two exposures.





Figure 18. Mean ratings and standard deviation for cupping of shingles following 12-month outdoor exposure for a) Oceanic, b) Maritime, and c) Arid site, for southwest (SW) and northeast (NE) exposure for uncoated control (O), Pine Tar (N), Chevron (C), Heritage (H), TWP 1500 (T), Penofin (P), Messmer's (M), DEFY (D), Seal Once (S), Timber Pro (U), and Sta Brite R (B).

Sito		SW			NE		+(10)	n
Sile	Ν	Mean	SD	Ν	Mean	SD	(10)	μ
Oceanic	11	9.25	0.30	11	9.49	0.27	2.23	0.014
Maritime	11	9.22	0.21	11	9.56	0.25	2.23	0.005
Arid	11	9.42	0.40	11	9.55	0.20	2.23	0.224

Table 4. t-test comparing cupping at SW and NE exposure for three test sites.

Cupping was more prominent on SW than on NE exposures for water-borne coatings based on t-test at p<0.05 for all three sites. No significant differences were observed for oil-borne coatings.

No consistent effects of testing site and elevation were observed among individual coatings, although the analysis of the mean ratings for all samples with cupping from all three sites suggests that in general, oil-borne coatings had less cupping than water-borne coatings. TWP 1500 and Messmer's consistently had the least cupping regardless of the exposure conditions (Figure 19).



Figure 19. Mean cupping ratings and SD for samples from all test groups (three sites and two elevations). TWP 1500 (T), Messmer's (M), Pine Tar (N), Chevron (C), Heritage (H), Penofin (P), Timber Pro (U), DEFY (D), Sta Brite R (B), Seal Once (S), and Control (O).

WATER REPELLENCY

Results for water repellency of test groups based on field evaluation (Figure 20) are presented in Figure 21. There was very little variability in water repellent properties within each test group over the course of data collection. Uncoated control and Sta Brite R coated groups had no water beading on the shingle surface and were assigned the lowest rating of 6 at the time of installation with no subsequent change during the 12-month outdoor exposure.



Figure 20. Examples of water beading on shingles during the field testing.

Water repellency of oil-borne coatings decreased progressively to different extents over time: Pine Tar, Penofin, and Messmer's, which initially had maximum water repellency ratings, deteriorated to very low values of 6 or 7 over a 12-month outdoor exposure, with overall higher residual water repellent properties at the Arid site than at the Oceanic and Maritime sites. Water repellency of Chevron coating was initially 9 and decreased only slightly to 8 at all sites. For Heritage, water repellency decreased from the initial rating of 8 to the raing of 6 at the Oceanic site, the rating of 7 at the Maritime site, and remained between the ratings of 7 and 8 at the Arid site. TWP 1500 had the best retention of water repellency, which remained at initial maximum of 10 for all sites.



Figure 21. Average water repellency and SD for individual test groups based on on-site evaluation of water beading for SW and NE exposures for Oceanic (O), Maritime (M), and Arid (A) sites.

26

Among water-borne coatings, DEFY lost all of the initial highest-rated water repellency over the course of first 6 months at all sites. In contrast, Seal Once was rated as having no water repellent properties at the time of installation, but ratings progressively increased over the course of outdoor exposure to a maximum of 10. Timber Pro, which was rated 6 at the time of installation, retained the same rating at the Oceanic and Maritime sites and had an improved water repellency of 7 and 8 respectively at the Arid site following the 12 month outdoor exposure compared to initial water repellency. A possible explanation for the improvement of water repellency in Seal Once, and to a small extent in Timber Pro is likely related to ongoing polymerization of coating components to more hydrophobic forms over the course of outdoor exposure.

WATER REPELLENT PROPERTIES

Results for laboratory tests of water absorption and water repellent effectiveness of investigated coatings in shown in Figure 22, Figure 23, and Table 3. For coated groups, average water absorption over 30 minutes of water immersion ranged from 0.7% for TWP 1500 to 10.4% for Sta Brite R, compared to 23.5% for the control group. All coatings significantly reduced water absorption of WRC shingles compared to uncoated controls. Water repellent efficiency, which represents the ability of coating to prevent water absorption relative to uncoated controls, ranged from 55.8% for Sta Brite R to 97.1% for TWP 1500.



Figure 22. Average water absorption (%) and standard deviation (%) for coated shingles and controls following the laboratory testing. Uncoated control (O), Pine Tar (N), Chevron (C), Heritage (H), TWP 1500 (T), Penofin (P), Messmer's (M), DEFY (D), Seal Once (S), Timber Pro (U), and Sta Brite R (B).

Based on the water absorption and water repellent efficiencies, investigated coatings can be ranked as follows:

- 1. Highly water repellent coatings: TWP 1500
- 2. Moderately water repellent oil-borne coatings: Messmer's UV Plus, Penofin Ultra Premium, Genuine Pine Tar 850, Chevron Shingle Oil, and Heritage Exterior Finish
- 3. Moderately water repellent water-borne coatings: DEFY Extreme Wood Stain and Timber Pro Log and Siding Formula
- 4. Low water repellent water-borne coatings: Seal Once Total Wood Protection and Sta Brite R.

27



Figure 23. Water repellent efficiency relative to uncoated control samples for laboratory tested water absorption. Uncoated control (O), Pine Tar (N), Chevron (C), Heritage (H), TWP 1500 (T), Penofin (P), Messmer's (M), DEFY (D), Seal Once (S), Timber Pro (U), and Sta Brite R (B).

Figure 24 shows the comparison of water absorption determined by laboratory testing and ranking of water repellency determined in the field before the weathering exposure. As seen in the graph, overall there was a good correlation between laboratory and field results for all coatings with the exception of Timber Pro, which had low water absorption comparable to DEFY in laboratory testing but was rated at 6 as having no apparent water beading during field testing of water repellency.



Figure 24. Comparison of water absorption (%) determined by laboratory testing and ranking of water repellency determined in field before the weathering exposure. Uncoated control (O), Pine Tar (N), Chevron (C), Heritage (H), TWP 1500 (T), Penofin (P), Messmer's (M), DEFY (D), Seal Once (S), Timber Pro (U), and Sta Brite R (B).

ATR-FTIR

The ATR-FTIR spectra were collected from unweathered coated and uncoated control samples and from one sample for each treatment group from each site after 12 months of outdoor exposure. The spectra were grouped and plotted on the same graph for each treatment and presented in the Appendix with the exception of Timber Pro, for which the spectra appeared anomalous due to error during spectra collection. ATR-FTIR band assignment was based on the published literature (Table 5). Although the spectral region between 1800 cm⁻¹ and 800 cm⁻¹ is considered as a fingerprint region for wood, a broader 4000-700 cm⁻¹ range was used in the analyses to include possible spectral bands originating from the coatings. No attempt was made to identify the constituents of the coatings based on the FTIR spectra. The ATR-FTIR spectra provided qualitative, and in some instances comparative, information about chemical changes in wood and coatings that resulted from the outdoor weathering.

Table 5. FTIR band assignment based on the literature (Harrington et al., 1964; Faix et al	., 1991; Faix, 1992;
Pandey, 1999; Hon, 2001; Pandey and Pitman, 2003; and Müller et al., 2003).	

Literature (cm ⁻¹)	Literature (after weathering) (cm ⁻¹)	Compound or chemical group
3331		O-H stretching of bonded hydroxyl groups
2921	2886	C-H stretching vibration
2850	2886	C-H stretching vibration
1735	1721	C=O stretching in unconjugated ketone, carbonyl and ester groups
1732		C=O stretch in xylan
1646 (1641)	1630	Absorbed O-H and conj. C-O
1593 (1595)		Arom. skeletal vibrations and C+O stretching
1505 (1509)		Arom. skeletal vibrations
1460 (1453)		C-H deformation vibrations in lignin and xylan
1424 (1421)	1425	Arom. skeletal vibr. in lignin with C-H in plane deformation.
1370	1367	C-H def. in cellulose and hemicell.
1328		S ring and G ring condensed
1318 (1314)		C-H vibration in cellulose (CH ₂ wagging)
1267 (1262)	1279	C-O stretch lignin ring in G units (G- ring plus C=O stretch in lignin)
1235		C-O stretch in G units, C-C plus C-O plus C=O stretch
1228		C-C plus C-O plus C]O stretch
1157		C-O-C vibration in cellulose and hemicell.
1122		Arom. skeletal vibr. and C-O stretch
1106		O-H association band in cellulose and hemicelluloses
1046	1049	C-O stretch in cellulose and hemicelluloses
1031		C-O vibr. in cellulose and hemicell.
1026		C=O stretching vibration in cellulose, hemicelluloses and lignin
897 (895)		C-H def. in cellulose
859		C-H out-of-plane arom. in L
818		C-H out-of-plane arom. in L

Uncoated control: In the valence band area, the ATR-FTIR spectra (Figure A 1) exhibits a broad band at 3331 cm⁻¹, assigned to the O-H stretching vibration for the water molecules absorbed in wood, and a prominent twin band located between 3000 and 2800 cm⁻¹ (2923 and 2853 cm⁻¹) corresponding to the C-H stretching vibration and characteristic for aliphatic groups (C-H stretch of CH2 and CH3) in wood extractives (Fabiyi et al., 2011). These bands lost the definition over one year of weathering at all three sites, indicating decomposition of aliphatic wood extractives. The band at 1740 cm⁻¹, associated with carbonyl ester, shifted at 1721 cm⁻¹ and decreased for the Arid site and almost completely disappeared at the Oceanic and Maritime sites, suggesting deacetylation of hemicelluloses (Anderson et al.; 1991, Temiz et al., 2006; Lionetto et al., 2012) over the course of outdoor exposure. A band at 1600 cm⁻¹ (1594-1602 cm⁻¹) associated with conjugated C–O, primarily in lignin, lost the definition at Arid exposure, but disappeared completely in samples at Oceanic and Maritime exposures. A band located at 1508 cm⁻¹ (1510-1501 cm⁻¹) specific to aromatic skeletal vibrations and associated with lignin ether

linkages (Harrington et al., 1964) was completely lost after one year of exposure at all three sites. This band is considered a primary lignin band and is assigned to the total content of the lignin components (Faix, 1992). Significant reduction in the 1508 cm⁻¹ band has been observed following the UV photodegradation of wood (Evans et al., 2008) and has been consequently used in evaluation of photodegradation. A broad band around 1630 cm⁻¹ appeared in all weathered samples. Tolvaj and Faix (1995) suggested that the peak centered between 1660 and 1590 cm⁻¹ is associated with the aromatic stretching of lignin and the absorption of water by the cellulose. The band at 1452 cm⁻¹ (1450-1456 cm⁻¹) associated with C-H deformation vibration in lignin, disappeared in all three samples, and the band at 1421 cm⁻¹ (1417-1424 cm⁻¹) for aromatic skeletal vibration in lignin, decreased considerably in samples from the Arid and Maritime sites, and less in the sample from the Oceanic site. The band at 1363-1370 cm⁻¹ associated with C-H deformation in cellulose and hemicelluloses, decreased in all samples. Assignment of 1368-1370 cm⁻¹ band to the total cellulose and hemicellulose content has been used to evaluate the extent of degradation of wood components (Emandi et al., 2011). The band at 1264 cm⁻¹ (1264-1270 cm⁻¹) associated with vibrations of the quajacyl rings and stretching vibrations of the C-O bonds, disappeared in all samples. These differences, combined with the disappearance of 1600 cm⁻¹ band, confirmed that lignin underwent structural changes during outdoor exposure. The band at 1156 cm⁻¹ (1150-1156 cm⁻¹) associated with C–O–C in cellulose and hemicelluloses (xylan), intensified in weathered samples at all three locations. The characteristic bands for cellulose at 1106 cm⁻¹ did not change for the Oceanic and Maritime sites, but disappeared in the sample from the Arid site. Similarly, the intensity of the band at 1046 cm⁻¹ associated with C-O stretch, decreased for the Arid site, while the 1157 cm⁻¹ band associated with C-O-C vibration in cellulose and hemicelluloses, increased considerably over the course of weathering for all three sites.

The analysis of ATR-FTIR spectra suggests that the most important chemical changes induced by the outdoor weathering exposure on uncoated western redcedar are the loss of wood extractives and the degradation of lignin, affecting its content and composition in agreement with studies on photodegradation of wood (Teacă et al., 2013). Based on the changes in FTIR spectra, lignin was almost completely lost at all three sites and there were no apparent differences in the effect of photodegradation on lignin among all three sites.

The FTIR bands characteristic for carbohydrate component of wood were similar for the Oceanic and Maritime sites following the weathering. When compared to unweathered control, they were characterized by more prominent bands assigned to cellulose and hemicelluloses. This is consistent with a relative increase of carbohydrate content in wood following the photodegradation of lignin, and was more pronounced for Oceanic than Maritime exposure. Although the 1368-1370 cm⁻¹ band assigned to the total cellulose and hemicellulose content (Emandi et al., 2011) did not change in response to weathering for any of the exposures, a result that indicates no differences among sites, bands assigned to hemicellulose and cellulose lost definition in the sample from the Arid site. This indicates structural changes and depolimerization of these components in addition to photodegradation of lignin. The observed difference is consistent with higher UV radiation at the Arid than at the Oceanic and Maritime sites resulting in more severe photodegradation of all wood components.

Pine Tar coated western redcedar had stronger twin bands at 2923 cm⁻¹ and 2853 cm⁻¹ in the 3000-2880 cm⁻¹ region when compared to untreated controls (Figure A 2). This is contributed to the constituents of pine tar and linseed oil present in the formulation and these bands can be used for monitoring the changes in the coating itself (Derrick, 1989). Following the 12 months weathering exposure, the bands in the 3000-2880 cm⁻¹ region decreased considerably for Arid and somewhat more for Oceanic exposure, and disappeared completely for Maritime exposure. This suggests that the loss of coating components was highest at Maritime, followed by Oceanic

and Arid exposures. Second strong band characteristic for the Pine Tar treatment, observed at 1741 cm⁻¹, and associated with linseed oil (Derrick, 1989), decreased most at the Maritime site, followed by the Oceanic site, and decreased least at the Arid site. This band also shifted to lower wavenumbers for Arid (1708 cm⁻¹), followed by Oceanic (1715 cm⁻¹), and Maritime (1710 cm⁻¹) exposures. These results suggest that the most severe degradation of Pine Tar coating occurred at the Maritime site, followed by the Oceanic and Arid sites.

Similarly to the uncoated controls, the primary lignin band at 1507 cm⁻¹, which was not affected by the presence of Pine Tar treatment on wood, was lost in all exposures following the weathering. Photodegradation of lignin for all treatments is further confirmed by the decrease of the 1454 cm⁻¹ and 1263 cm⁻¹ bands.

The band at 1365 cm⁻¹ decreased for all three sites, indicating changes to carbohydrates. The characteristic bands for cellulose at 1106 cm⁻¹ decreased slightly for the Maritime site, but lost definition for the Oceanic and Arid sites. The intensity of the bands at 1046 cm⁻¹, associated with C-O-C stretch in cellulose and hemicelluloses, lost definition for the Oceanic and Arid sites but not for the Maritime site, while the 1157 cm⁻¹ band associated with C-O-C vibration in cellulose and hemicelluloses, did not change considerably after weathering for either site.

The comparative analysis of FTIR spectra for unweathered and weathered Pine Tar treatment suggests that the coating was most severely affected at Maritime exposure and a similar effect was observed at Oceanic exposure. In contrast, coating was least affected at Arid exposure, suggesting the sensitivity of pine tar to climate conditions characteristic for "humid" Oceanic and Maritime exposures. Corresponding changes in spectral bands for wood components are in good agreement with observed changes in coatings. While advanced lignin degradation occurred in all exposures, carbohydrate components were most severely affected at Arid exposure, although the mechanism responsible for these observations is not clear.

Chevron: The spectrum of Chevron-coated control had more prominent bands at 2921 cm⁻¹ and 2854 cm⁻¹ (-C-H stretching in aliphatic compounds) in comparison to uncoated western redcedar control, and two additional weak bands at 2937 cm⁻¹ and 2858 cm⁻¹ (Figure A 3). Following the 12 months weathering exposure, no significant change in the 3000-2880 cm⁻¹ band region was observed for any of the exposures, indicating an overall good condition of the coating. A strong band at 1713 cm⁻¹ and a weak band at 1636 cm⁻¹, developed for all three sites following the weathering exposure, possibly suggesting the chemical changes to the coating.

Similar to the uncoated controls, the primary lignin band at 1507 cm⁻¹ disappeared completely in all exposures following the weathering. The 1454 cm⁻¹ band, which was significantly stronger in Chevron-coated than uncoated control samples, decreased considerably for all three exposures, likely reflecting the changes in both, the coating and the wood. Two weaker bands at 1423 cm⁻¹ and 1261 cm⁻¹, characteristic for lignin, were lost following the weathering at all three exposures, suggesting photodegradation of lignin.

No significant response to weathering for cellulose and hemicellulose was observed for the 1368-1370 cm⁻¹ band, but 1156 cm⁻¹ band increased for all three exposures. Bands at 1100 cm⁻¹ and 1051 cm⁻¹ characteristic for cellulose, decreased for Maritime and Oceanic exposure and disappeared for Arid exposure.

The results suggest that the coating constituents discernable in the FTIR spectra were well preserved at all three exposures, with no significant exposure effects. At all three sites, lignin was degraded following the one-year outdoor exposure. Carbohydrate component was not significantly affected at any of the sites, although previously observed loss of bands in the 1150-850 cm⁻¹ spectral region at Arid exposure was also confirmed for the Chevron coating.

Heritage: The spectrum of a weathered sample coated with Heritage coating is not available for Maritime exposure, while other spectra are shown in Figure A 4. The spectrum of the Heritage-coated control sample had more prominent bands at 2921 and 2852 cm⁻¹ in the 3000-2880 cm⁻¹ band region (-C-H stretching in aliphatic compounds) when compared to western redcedar control, indicating contribution of aliphatic constituents from the coating to intensity of these bands. Following the 12 months weathering exposure, the twin bands at 2921 cm⁻¹ and 2852 cm⁻¹decreased significantly for both exposures, indicating the loss or chemical changes in the coating over the course of weathering. Another strong band characteristic of the coating at 1739 cm⁻¹, which was also observed in Pine Tar treatment and can be indicative of drying oils present in the coating formulation, decreased considerably at the Oceanic site and even more at the Arid site, suggesting their decomposition over the course of weathering.

The primary lignin band at 1507 cm⁻¹ disappeared completely for Oceanic exposure, but remained strong for Arid exposure. Other bands characteristic for lignin at 1454 cm⁻¹ and 1264 cm⁻¹ had similar response to the weathering exposure, confirming photodegradation of lignin at Oceanic exposure but not at Arid exposure.

A 1314 cm⁻¹ band assigned to cellulose increased after outdoor weathering at both sites, while the 1368 cm⁻¹ band did not change. The bands characteristic for cellulose and hemicellulose at 1157 cm⁻¹, 1051 cm⁻¹, 1027 cm⁻¹, and 897 cm⁻¹ decreased slightly at both sites.

While the results do not indicate significant differences in the effect of Oceanic and Arid weather exposure on the coating condition, it appears that the Oceanic exposure resulted in severe degradation and loss of lignin. In contrast, Heritage appears to provide a high level of protection against weathering of wood for Arid exposure.

Messmer's: Coated controls had a very similar spectrum to uncoated western redcedar controls, with the exception of a band shift from 1739 cm⁻¹ to 1729 cm⁻¹, another band shift from 1509 cm⁻¹ to 1496 cm⁻¹, and a weak band at 1605 cm⁻¹ (Figure A 5). Following the 12 months weathering exposure, the bands in the 3000-2880 cm⁻¹ region decreased but were not completely lost for all three sites, while these bands disappeared completely in the uncoated control sample. Band intensity at 1729 cm⁻¹ increased at all three sites, the most for Arid exposure, followed by Oceanic and Maritime exposures.

Bands characteristic for lignin at 1508 cm⁻¹, 1451 cm⁻¹, and 1423 cm⁻¹ were almost completely lost for all three exposures following the weathering. Bands characteristic for cellulose at 1157 cm⁻¹ developed for all for all three sites following the weathering and the 1367 cm⁻¹ band was not affected by weathering.

The results suggest that the coating condition was affected at all exposures as a result of outdoor weathering. Photodegradation of lignin occurred at all three exposures, with no significant differences among sites and no significant changes to carbohydrate complex were observed.

Penofin: Only unweathered control samples and weathered samples for Maritime and Arid exposures were analyzed by ATR-FTIR (Figure A 6). Samples coated with Penofin had very similar spectra to those coated with Messmer's. They differed from the uncoated control due to a prominent band at 1733 cm⁻¹ (shifted from 1739 cm⁻¹ in uncoated control), which seem characteristic for Penofin. Following the 12 months weathering exposure, the bands in the 3000-2880 cm⁻¹ region lost intensity at both sites.

Following the weathering at both sites, bands characteristic for lignin at 1508 cm⁻¹ and 1454 cm⁻¹ disappeared and the band at 1262 cm⁻¹ lost intensity, indicating photodegradation of lignin. Similar to Messmer's treatment, a small peak at 1168 cm⁻¹ present in control increased for both sites following the weathering. A band at 1157 cm⁻¹ characteristic for cellulose, developed at both sites following the weathering and a band at 1367 cm⁻¹ decreased slightly in weathered samples, indicating no significant changes in carbohydrate component.

The results suggest loss or decomposition of the coating at both exposures after 12 months of weathering and chemical changes in lignin.

TWP 1500: When compared to uncoated controls, coated controls had a more complex FTIR spectrum in the higher wavenumber 3100-2800 cm⁻¹ band region, with strong bands at 2916 cm⁻¹ and 2845 cm⁻¹ and two weak bands at 2953 cm⁻¹ and 2863 cm⁻¹ (Figure A 7). Following the 12 months weathering exposure, no significant change was observed for these bands. In the wood fingerprint region, TWP 1500 also had a strong band at 1746 cm⁻¹, which shifted to 1741-1712 cm⁻¹ and did not change during the weathering at any investigated exposure. Another band at 1644 cm⁻¹, characteristic for the TWP 1500 coating did not change over the course of weathering.

The primary lignin band at 1508 cm⁻¹ disappeared for all exposures, and the 1454 cm⁻¹ band was masked by the band at 1462 cm⁻¹ that originated from the coating. A 1262 cm⁻¹ band disappeared and a new band at around 1248 cm⁻¹ developed at all three sites, suggesting photodegradation of lignin. No significant changes were observed in spectral regions characteristic for carbohydrates.

While the FTIR spectrum of TWP 1500 suggests a complex product formulation, there seems to be little change in the coating composition over the course of outdoor weathering and no apparent differences between the three exposures. This is in agreement with other results of the outdoor weathering exposure monitoring. Although the characteristic bands for wood were partly masked by the coating-specific bands, it appears that lignin in TWP 1500-coated samples was affected by photodegradation.

DEFY: Coated controls had a more complex IR spectrum in the 3000-2800 cm⁻¹ wavenumber region when compared to uncoated controls, with bands at 2916 cm⁻¹ and 2845 cm⁻¹ and two weaker bands at 2953 cm⁻¹ and 2870 cm⁻¹ (Figure A 8). Following the 12 months weathering exposure, peaks in the 3000-2800 cm⁻¹ region decreased for the Arid site and did not change significantly for the two "humid" sites.

In the wood fingerprint region, the FTIR spectrum of DEFY was very similar to uncoated controls. It appears that following the weathering, the 1740 cm⁻¹ band associated with the coating, increased at all three sites, particularly at the Oceanic site.

The bands characteristic for lignin at 1508 cm⁻¹ disappeared at all three sites and a new band appeared at 1494 cm⁻¹, increasing in intensity from Arid to Maritime and Oceanic exposure. The band at 1452 cm⁻¹ associated with C–H deformation vibration in lignin, decreased considerably at the Arid site, did not change at the Maritime site, and increased at the Oceanic site. However, the 1454 cm⁻¹ band was lost at all exposures. The band at 1367 cm⁻¹ characteristic for cellulose shifted to higher wavenumbers but did not change significantly for any investigated exposure. The bands at 1106 cm⁻¹ and 1046 cm⁻¹ characteristic for cellulose did not change for Maritime exposure and decreased for Arid exposure. However, a broad band of high intensity appeared at the 1142 cm⁻¹ wavenumber. The band at 897 cm⁻¹ shifted to a higher wavenumber for all three exposures following the weathering.

The observed changes in FTIR spectra indicate significant changes in the composition of the coating. They also indicate that for all exposures, there were possible chemical reactions between the coating and wood components over the course of outdoor exposure, in particular for Oceanic exposure. However, due to the lack of information about the chemical composition of the DEFY formulation, the nature of these chemical changes is not understood.

Seal Once: Coated controls had a strong band at 2922 cm⁻¹ and additional weak bands at 2946 cm⁻¹, 2870 cm⁻¹, and 2850 cm⁻¹ (Figure A 9). Following the weathering exposure, bands in the 3000-2800 cm⁻¹ wavenumber region decreased for Maritime and Arid exposures significantly more than for Oceanic exposure. A band at 1726 cm⁻¹ that was observed similar to the spectrum of DEFY, broadened and decreased only slightly for the Arid site, followed by a more significant decrease at the Maritime and Oceanic sites.

A band characteristic for lignin at 1508 cm⁻¹ disappeared completely at the Oceanic and Maritime sites, and decreased significantly at the Arid site. The 1614 cm⁻¹ band disappeared and 1260 cm⁻¹ band decreased at all three sites, suggesting photodegradation of lignin. Spectral bands that are characteristic for carbohydrates did not change significantly.

Sta Brite R: Coated controls had strong bands at 2916 cm⁻¹ and 2848 cm⁻¹ and two weak bands at 2939 cm⁻¹ and 2859 cm⁻¹ (Figure A 10). In the wood fingerprint region, a band at 1726 cm⁻¹ instead of 1740 cm⁻¹ was observed similar to the spectrum of DEFY and Seal Once. The band at 1462 cm⁻¹ was more prominent than in uncoated controls.

Following the weathering exposure, bands in the 3000-2800 cm⁻¹ wavenumber region decreased for the Maritime site and disappeared at the Oceanic and Arid sites. The band at 1726 cm⁻¹ broadened and decreased only slightly for the Arid site, followed by a more significant decrease at the Maritime and Oceanic sites. A band at 1614 cm⁻¹ disappeared at all three sites. The band at 1508 cm⁻¹ disappeared completely at the Oceanic and Maritime sites, and decreased significantly at the Arid site. A bands at 1259 cm⁻¹ decreased at all three sites and a new band formed at 1200 cm⁻¹.

GENERAL DISCUSSION

The results of laboratory and outdoor exposure testing of selected coatings and uncoated western redcedar shingles confirm significant differences in performance among tested groups and suggest a strong effect of climate variables on observed changes.

Over the course of the 1st phase of outdoor weathering exposure, which lasted over the fall and winter period from October 2016 to March 2017, the most evident changes were: loss of color, increase in graying, appearance of surface growth (biofilm), notably mildew and unidentified black-appearing mold, and changes in water repellent properties. Surface erosion, checking, and cupping appeared only during the 2nd phase of testing over the spring and summer period, which was between April and October 2017. The results of outdoor weathering exposure field monitoring are further verified by ATR-FTIR spectroscopy.

Based on the results from monitoring color change and graying over the course of outdoor weathering exposure, complete loss of color accompanied by graying occurred on uncoated western redcedar shingles within a year of outdoor exposure at all testing locations. Somewhat higher rates of color loss and graying were observed at Arid exposure, characterized by the highest total solar radiation among sites, than at Oceanic and Maritime exposures. The color change is the most sensitive indicator for photodegradation of wood. Typically, initial photodegradation appears in the form of yellowing of the natural color of wood, due to chemical changes in chromophores present in lignin and deposition of colored decomposition products on the wood surface (Heinskill, 1994; Hon, 2001). Subsequently, lignin degradation products are removed from the surface through either leaching by water, or by the mechanical action of wind (Derbyshire and Miller, 1981; Evans et al., 1993), leaving exposed cellulose fibers that contribute a silvery gray appearance to the wood. The results of field monitoring are further confirmed by the respective responses of the FTIR spectra of uncoated wood to outdoor weathering exposure, suggesting surface delignification at all exposures, accompanied by

respective chemical changes in the carbohydrate wood constituents (cellulose and hemicelluloses) resulting from the weathering at the Arid site. Since the photodegradation is largely a surface phenomenon, limited by the depth of penetration of UV radiation to approximately 75 um below the wood surface (Hon, 2001), final stages of weathering are marked by the loss of the wood degradation products from the surface through erosion. Surface erosion is the final stage of weathering, which is a complex process initiated by UV radiation, and facilitated by a mechanical action of wind and precipitation. The highest level of erosion of uncoated control samples was observed at the Maritime site, which can be explained by the combined effect of high precipitation similar to the Oceanic site, and a higher average wind speed and total solar radiation than at the Oceanic site. The results suggest that although the Arid exposure has the highest risk for photodegradation governed by high UV radiation, the environmental factors such as precipitation and wind are critical for the degree of weathering, as indicated by erosion rates.

Clear transparent, or lightly pigmented, coatings, were selected in this study for their compatibility with the historic appearance of texture and color of traditionally unfinished western redcedar shingles. They are commonly formulated with additives aimed at improving their UV protection properties, although they are not as efficient as opaque film forming finishes. Most commonly used additives include UV absorbers (UVAs), which are colorless and have high absorption coefficients in the UV spectrum, HALS (hindered amine light stabilizers), usually used together with UVA, and mineral screening pigments (e.g. TiO₂, ZnO and iron oxides) which act as UV blockers (Aloui et al., 2004; Pospísil and Nespurek, 2000). In addition to providing UV protection to underlying wood substrate, these additives also protect the coating material itself. As a result, the color change in coated wood surfaces is often an indication of the overall coating condition and its ability to fulfill its other roles, such as providing water repellency (Chang and Chou, 1999). Since the detailed information about the composition of tested coating formulations in not available, the extent of photodegradation of coatings and wood is based on the same tests used for uncoated wood, including change of color, extent of graying, erosion, and FTIR spectra.

The color change and graying of Pine Tar treatment was most significant at the Oceanic site, followed by the Maritime site and then the Arid site. Based on the changes in FTIR spectra of aliphatic components in pine tar, it appears that the coating suffered the most damage at the Maritime site, followed by the Oceanic and Arid sites, indicating its sensitivity to harsh humid climates. However, this treatment was not efficient in preventing photodegradation of lignin at any exposure, and structural changes to carbohydrates were apparent at the Oceanic and Arid sites. Similar effects of climate exposure were also observed for transparent non-pigmented oilbased Chevron coating, with more pronounced changes at the humid Oceanic and Maritime sites than at the dry Arid site. The loss of color and increase in graying entirely reflects the changes in the chemical composition of lignin and carbohydrates confirmed by the ATR-FTIR findings. The coating seems to be well preserved at all sites based on the lack of response in FTIR bands characteristic for the coating.

Based on the FTIR spectra available for Oceanic and Arid exposure, for Heritage coating outdoor weathering exposure resulted in a decrease in the aliphatic content of the coating at both sites. The photodegradation of lignin, accompanied by chemical changes in carbohydrates, was evident in FTIR spectra for Oceanic exposure and confirmed by the complete loss of color and graying at this site. However, there was no significant chemical degradation of wood coated with Heritage was observed for the Arid site, although the initial color was mostly lost and the extent of graying was similar to Chevron treatment. Performances of lightly tinted oil-based Penofin and Messmer's coatings were similar, characterized by moderate loss of color and graying at the two humid sites and an overall good retention of color and slight graying at the

Arid site. The rate of loss of aliphatic components and the advanced photodegradation of lignin was similar for two coatings at all investigated sites, with no apparent differences observed between different exposures. Excellent color retention and absence of graying for TWP 1500 treatment in any of the three exposures was also confirmed by the lack of response in coating-specific FTIR bands. However, photodegradation of lignin in wood was indicated on the basis of FTIR spectra, although some of the bands that are characteristic for wood were partly masked by the coating-specific bands.

DEFY was the only tested non-pigmented formulation among water-based coating. Coated wood completely lost the color and attained a level of graying similar to the uncoated control group at the Oceanic and Maritime sites, with a slightly better color retention at the Arid site. It appears that the coating was not significantly lost over the course of outdoor weathering exposure based on the coating-specific FTIR bands. However, the spectra of weathered DEFY coated wood changed considerably following the weathering, especially for the Oceanic site. Due to the lack of information about the chemical composition of the DEFY formulation, no attempt was made to interpret these complex spectral changes. FTIR bands characteristic for lignin suggest photodegradation in weathered samples. Lightly pigmented Seal Once coating performed better than DEFY with respect to color retention and graving, exhibiting similar site effects with an overall better performance for the Arid site than for the Oceanic and Maritime sites. Timber Pro had a variable performance among three exposures and two tested elevations, but overall lost the color significantly at all the sites and had a fairly good resistance to graving at the Arid site. Sta Brite R had the best color retention and least graving among the tested water-based finishes, with a slight decrease in color and an increase in graving for Oceanic and Maritime exposures and no changes for Arid exposure. FTIR spectra indicate that photodegradation of lignin occurred despite the good visual appearance of the coating.

In terms of the aesthetic appearance, the most desirable coatings are those that resemble either the original or the weathered color of western redcedar. Heritage and Chevron coated shingles had the closest color to weathered control group after a 12 months exposure at the Maritime and Oceanic sites. The color of other oil-borne coatings Pine tar, Penofin and Messmer's shifted towards natural western redcedar color over the course of the 1st testing phase, and attained considerably darker tones when compared to weathered western redcedar at advanced stages of color loss after 12 months. TWP 1500 retained its original color over the whole year of outdoor exposure, closely resembling the color of unweathered western redcedar heartwood. Water-borne coating DEFY also weathered to a similar appearance as the weathered western redcedar when dry, but a milky white film appeared on the surface when the coating was wet (Figure 25).



Figure 25. Whitish appearance of wet DEFY coated shingles

It is suspected that the color of Timber Pro was affected by an incomplete curing of coating prior to installation and thus will not be discussed further. Sta Brite R and Seal Once coating formulations were "Clear" and "Natural" tones that did not match the color of western redcedar, but both coatings were well preserved and had an overall good retention of color. Upon closer inspection, water-borne coatings are less transparent than oil-borne coatings. They also appear to form a film-like deposit on the surface that developed micro checks after one year of outdoor exposure (Figure 26).

Color loss and graving of uncoated wood is mostly affected by the UV radiation. Therefore, more rapid weathering of uncoated control groups was observed on the exposed and dry Arid site. However, all coated groups had better color retention at the Arid site than at the other two sites, suggesting that they were successful in protecting wood against severe effects of UV radiation. The results of field monitoring suggest that the coatings were more prone to damage at the Oceanic and Maritime sites that had more precipitation. The mechanisms behind coating failures are beyond the objectives of this study. The information on the changes in the coating composition and condition could not be derived from the ATR-FTIR spectra, but the trends of color change suggest that coatings in general might have shorter service life at the two "humid" sites. Higher susceptibility to damage and eventual failure of coatings at "humid" sites is further indicated by a higher number of instances of surface erosion of coatings. The total number of samples with evident erosion was very low, often observed on only one specimen per group and only in a single location. Yet, it is important to note that the number of test groups with early signs of erosion was especially high at the Maritime site, followed by the Oceanic site for oilborne as well as water-borne coatings. In contrast, only uncoated control shingles had noticeable erosion at the Arid site. It is expected that the future monitoring will provide additional information on the performance of coatings at different exposures, and indicate whether winter or summer weather conditions are more conducive to these changes.



Figure 26. Micro checks on water-borne coatings.

Biological growth, which can include fungal (mildew), mold, algal or lichen growth, is limited to the wood surface. It does not impact strength properties of wood, but is considered important for the aesthetic appearance of roofs. Because most molds need a moisture content of about 20% to begin growth, their presence often indicates favorable moisture conditions for growth of wood decay fungi. Since western redcedar shingles are preferably made from naturally decay resistant heartwood, they are not prone to wood decay until final years of service life, when heartwood extractives are leached out by precipitation and favorable moisture conditions are present for longer periods of time. A biofilm, appearing as black mold, was the most common form of biological growth on all test sites for uncoated samples and oil-borne coatings after the 1st phase of testing, and decreased substantially or completely during the 2nd phase. Black or gray biofinish, which commonly develops on wood treated with vegetable oils such as linseed oil, is caused by the fungi *Aureobasidium pullulans* (van Nieuwenhuijzen et al., 2015) that is common in the Pacific Northwest. A study that investigated the effect of location on the coating

37

performance found a greater diversity of organisms growing on the panels in Oregon than in other humid locations in the U.S., including fungi, primarily *Cladosporium* spp. and Aureobasidium pullulans, green algae Neospongiococum, the blue-green cyanobacteria Gloeocapsa, and lichens (Colon et al., 2004). It is unclear whether the extent of mold growth was affected by the climate parameters (i.e. temperature and precipitation) or by the depletion of nutrients from the wood and / or coating surface. Location and climate can influence the microbiota on wood surfaces and seasonal climatic changes may also be important (Gaylarde et al., 2011). It has been shown that biological fouling on building surfaces is higher in the spring and fall (Hofbauer et al., 2006), as observed in this study for the first year of outdoor exposure monitoring. It is indicative that the only instance of mildew was observed at the Maritime site, which has high total precipitation, high relative humidity, and a microclimate characterized by more vegetation and more shade around the test structure. In general, biological growth was more frequent and abundant on uncoated control groups than on coated groups. This suggests that all investigated coatings contribute a level of protection, through either through the action of biocidal components or by controlling the moisture levels of wood. It is important to note that patchy black mold spots developed prominently on Timber Pro coated groups at the Oceanic and Maritime sites but not at the Arid site. It is possible that a higher tendency towards mold development is affected by incomplete curing of Timber Pro on these samples before installation. The best overall performance among tested coatings was observed for oil-borne TWP 1500 and water-borne Sta Brite R, which had the least amount of growth at all sites. No fungal decay was observed after the 1st year of outdoor exposure on any test specimens.

Checking and cupping developed on shingles over the course of the 2nd testing phase, affecting only a small number of samples and was generally more prominent for water-borne than for oilborne coatings. No checking was observed in uncoated control groups, which were at the same time slightly more prone to cupping than coated groups. Checking and cupping are affected by the water permeability of wood and the effects of cyclic changes in wetting and drying. The results suggest that oil-borne coatings performed better at limiting the water absorption and the development of drying stresses in coated shingles. This result is in agreement with their better water repellent properties, as determined in both field and laboratory tests. In contrast, waterborne coatings, which had poorer water repellent properties, were more prone to cupping and checking, and suffered from micro checking of the coated wood surface. There is a possibility that polymerization reactions between the coating and wood components within the wood matrix decrease the ability of wood to shrink and swell in response to changing moisture content. In combination with poor water repellent properties of water-borne finishes, this can lead to tensile stresses that result in checking. Continued monitoring of these changes over the 2nd year of outdoor weathering exposure testing will provide more information about the severity of checking, as affected by the coating formulation and climate effects.

CONCLUSIONS

The comparative study of the performance of ten coatings considered for the protection of western redcedar shingle and shakes was conducted under natural weathering exposure in three distinctive climate regions of Washington.

The data collected over one year of exposure confirmed a strong effect of climate variables, such as total precipitation and solar radiation, on the condition of coated and uncoated test groups with significant differences among investigated coatings. The most significant changes were observed in color, graying, surface microbial growth, and water repellency during the first

six months, with the onset of checking and cupping occurring during the second six months of outdoor exposure.

The climate conditions prevalent at Arid exposure are conducive to rapid UV photodegradation of uncoated western redcedar, as well as photodegradation of wood in coated shingles. However, following one year of natural weathering, the general condition of coatings was however better at Arid exposure than at humid Oceanic and Maritime exposures. The major effects of harsh climate conditions on coatings at the Oceanic and Maritime sites were a rapid loss of color, an increase of graying, and a higher incidence of surface erosion, checking, and cupping, and the amount of microbial growth.

The results of outdoor exposure monitoring were confirmed with ATR-FTIR spectroscopy, which provided further evidence of both photodegradation of wood components and chemical changes in coatings. Comparatively better water repellency was observed in oil-based than water-based coatings and uncoated specimens in both outdoor exposure testing and in laboratory tests. Lower water repellency was related to a higher incidence of checking and development of micro-checking in samples coated with water-based coatings. Among investigated coatings, TWP 1500 and Stabrite R had the best condition of coating, and in general, a better performance than other coatings at all three sites, with more variable performance observed for other coatings.

The results of this study highlight significant effects of natural weathering exposure on the condition of investigated coatings and indicate the most important deterioration mechanisms that may affect shingle and shake roofs in service in three climate regions. The continued monitoring over the course of the second year of outdoor exposure will provide more pertinent information about the potential of these coating treatments to protect western redcedar roofs and meet the criteria for their consideration by Washington State Parks.

ACKNOWLEDGEMENTS

The authors thank the National Center for Preservation Technology and Training for the financial support and administrative support of Washington State Parks & Recreation Commission that made this research possible. We thank the staff from the Cape Disappointment WA State Park, Beacon Rock WA State Park, and Columbia Hills State Park, in particular Evan Roberts, Rebekah Vessey, Heath Yeats, Andy Kallinen, and Lem Pratt for providing and securing testing sites and for their assistance during data collection, Dr. Jeffrey Morrell (Oregon State University) for his support and contribution to the experimental design, Dr. Dragica Jeremic (formerly Mississippi State University) for her suggestions to research proposal development, Dr. Casey Check (CAMCOR Polymer Characterization Laboratory, University of Oregon) for the assistance with ATR-FTIR study, and Clara Gorman (University of Oregon) for text editing.

REFERENCES

- 1. Alexis Oil Company. (2009). Material safety data sheet. Chevron Shingle Oil. Retrieved from http://pwngservices.com/wp-content/uploads/2013/07/Chevron-Shingle-Oil-MSDS.pdf
- 2. Aloui F., A. Ahajji, Y. Irmouli, B. George, B. Charrier, and A. Merlin. (2007). Inorganic UV absorbers for the photostabilisation of wood-clearcoating systems: comparison with organic UV absorbers. *Applied Surface Science*, 253:3737–3745.

- 3. Amteco. (2015). Safety data sheet. TWP 1500 Clear. Retrieved from https://sealandprotect.com/docs/TWP1500.pdf
- 4. Anderson, E.L., Z. Pawlak, N.L. Owen, and W.C. Feist. (1991). Infrared studies of wood weathering. Part I: Softwoods. *Journal of Applied Spectroscopy*, 45(4):641-647.
- 5. ASTM. (2013). ASTM D1006. Standard Practice for Conducting Exterior Exposure Tests of Paints on Wood. West Conshohocken, PA: ASTM International.
- 6. ASTM. (2013a). ASTM D5401. Standard Test Method for Evaluating Clear Water Repellent Coatings on Wood. West Conshohocken, PA: ASTM International.
- Browne, F.L. (1960). The preservative treatment and staining of shingles. FPL Report No. 761 (revised). Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory.
- 8. Chang, S. T., and P. L. Chou. (1999). Photo-discoloration of UV-curable acrylic coatings and the underlying wood. *Polymer Degradation and Stability*, 63:435-439.
- Colom, X., F. Carrillo, F. Nogués, and P. Garriga. (2003). Structural Analysis of Photodegraded Wood by Means of FTIR Spectroscopy. *Polymer Degradation and Stability*, 80:543–9.
- Colon, I., E.L. Kuusisto, and K. Hansen. (2004). Location affects performance of biocidecontaining paints. *Paint and Coating Industry*. Retrieved from <u>https://www.pcimag.com/articles/86718-location-affects-performance-of-biocide-containingpaints
 </u>
- Derbyshire H., and E.R. Miller. (1981). The photodegradation of wood during solar irradiation. Part 1: Effects on the structural integrity of thin wood strips. *Holz als Roh- und Werkstoff*, 39(8):341–350.
- 12. Derrick, M.R. (1989). Fourier transform infrared spectral analysis of natural resins used in furniture finishes. *Journal of the American Institute for Conservation* 28(1):43-56.
- Dwyer, J., T. Bonura, A. Nebelsick, S. Williams, and C.G. Hunt. (2011). Installation, Care, and Maintenance of Wood Shake and Shingle Roofs. General Technical Report FPL–GTR– 201. Madison, WI: USDA, Forest Service, Forest Products Laboratory.
- 14. Emandi, A.N.A., and C.I. Vasiliu. (2011). Quantitative Investigation of Wood Composition by Integrated FT-IR and Thermogravimetric Methods. *Cellulose Chemistry and Technology*, 45(9-10): 579-584.
- 15. Evans P.D., K.J. Schmalzl, and A.J. Michell. (1993). Rapid loss of lignin at wood surfaces during natural weathering. In: J.F. Kennedy, G.O. Phillips, and P.A. Williams (Eds.), *Cellulosics: pulp, fibre and environmental aspects* (pp. 335–340). Chichester: Ellis Horwood.
- Evans, P.D., K. Urban, and M.J.A. Chowdhury. (2008). Surface Checking of Wood Is Increased by Photodegradation Caused by Ultraviolet and Visible Light. *Wood Science and Technology*, 42(3):251–65.
- Evans, P.D., M. J. Chowdhury, B. Mathews, K. Schmalzl, S. Ayer, M. Kiguchi, and Y. Kataoka. (2002). Weathering and Surface Protection of Wood. In M. Kutz (Ed.), *Handbook of Environmental Degradation of Materials* (pp. 277-297). Norwich, NY: William Andrews Publishing.
- Fabiyi, J.S., A.G. McDonald, J.J. Morrell, and C. Freitag. (2011). Effects of wood species on durability and chemical changes of fungal decayed wood plastic composites. *Composites. Part A*, 42: 501–510.
- 19. Faix, O. (1992). Fourier transform infrared spectroscopy. In S.Y. Lin and C.W. Dence (Eds.), *Methods in Lignin Chemistry* (pp. 83–109). Berlin, Heidelberg, New York: Springer-Verlag.
- 20. Faix, O., J. Bremer, O. Schmidt, and T. Stevanovic. (1991). Monitoring of chemical changes in white-rot degraded beech wood by pyrolysis-gas chromatography and Fourier-transform infrared spectroscopy. *Journal of Analytical and Applied Pyrolysis*, 21, 147-162.

- Feist W.C., and D.N.-S. Hon. (1984). Chemistry of weathering and protection. In: Rowell R.M. (Ed.), *Chemistry of solid wood* (pp. 401–454). Washington, DC: American Chemical Society.
- 22. Feist, W.C. (2006). Exterior wood finishes. In: A.A. Tracton (Ed.), *Coatings Technology Handbook,* FL: Taylor and Francis Group.
- 23. Gaylarde, C.C., L.H.G. Morton, K. Loh, and M.A. Shirakawa. (2011). Biodeterioration of external architectural paint films A review. *International Biodeterioration & Biodegradation*, 65 (2011) 1189-1198.
- 24. Harrington, K.J., Higgins, H.G., and Michell, A.J. (1964). Infrared spectra of *Eucalyptus regnans* F. Muell and *Pinus radiata* D. Don. *Holzforschung*, 18(4):108–113.
- 25. Heinskill J. (1994). Möglichkeiten der UV-Stabilisierung farbloser Holz-Außenanstriche. *Farbe und Lack* 100:1011–1015.
- 26. Heritage Natural Finishes. (2015). Product info. Retrieved from http://www.heritagenaturalfinishes.com/SearchResults.asp?Cat=48
- 27. Hofbauer, W., C. Fitz, M. Krus, K. Sedlbauer, and K. Breuer. (2006). Prognoseverfahren zum biologischen Befall durch Algen, Pilze und Flechten an Bauteiloberflächen auf Basis bauphysikalischer und mikrobieller Untersuchungen [Assesment procedure for microbial growth of algae and mould on the basis of physical and biological investigations]. Hrsg.: Fraunhofer-Institut für Bauphysik elBP-, Holzkirchen; Bauforschung für die Praxis Band 77.
- 28. Hon, D.N.S. (2001). Weathering and photochemistry of wood. In: D.N.S. Hon and N. Shiraishi (Eds.), *Wood and Cellulosic Chemistry* (2nd ed.) (pp. 513-547). New York and Basel: Mercel Dekker.
- 29. ISK Biocides Inc. (2016). Safety data sheet. Sta Brite R Cedar. Retrieved from http://www.iskbiocides.com/uploads/ ROOT/File/Sta%20Brite%20R%20Cedar%20122%20 SDS.pdf
- 30. Lionetto, F., R. Del Sole, D. Cannoletta, G. Vasapollo, and A. Maffezzoli. (2012). Monitoring Wood Degradation during Weathering by Cellulose Crystallinity. *Materials*, 2012:1910-922.
- Magill, C. (2015). Performance Assessment and Evaluation of Hydrophobic and Ultraviolet Protective Treatments for Historic Log Structures. (Masters Thesis). University of Pennsylvania, Philadelphia, PA.
- 32. Messmers Inc. (n.d.) Material safety data sheet. UV Plus CMC. Retrieved from http://www.messmers.com/msds/MSDS-UV-PLUS-250.pdf.
- 33. Morrell, J.J., P.F. Schneider, and R.S. Williams. (2001). Protecting Wood Decks from Biodegradation and Weathering: Evaluation of Deck Finish Systems. *Forest Products Journal*, 51(11/12):27-32.
- 34. Müller, U., M. Rätzsch, M. Schwanninger, M. Steiner, M., and H. Zöbl. (2003). Yellowing and IR-changes of spruce wood as result of UV-irradiation. *Journal of Photochemistry and Photobiology B: Biology*, 69, 97-105.
- 35. New Image Coatings. (2015). Safety data sheet. Seal-Once Waterproofer. Retrieved from http://www.logfinish.com/wp-content/uploads/2014/03/Seal-Once MSDS.pdf
- 36. Niemiec, S.S., and T.D. Brown. (2015). Care and maintenance of wood shingle and shake roofs. In J.W. Pscheidt and C.M. Ocamb (Eds.), *Pacific Northwest Plant Disease Management Handbook*. Corvallis, OR: Oregon State University.
- 37. NOAA. (2018). Climate Data Online: Dataset Discovery. National Centers for Environmental Information, National Oceanic and Atmospheric Administration. Retrieved from https://www.ncdc.noaa.gov/cdo-web/datasets.
- 38. Pandey, K.K. (1999). A study of chemical structure of soft and hardwood and wood polymers by FTIR spectroscopy. *Journal of Applied Polymer Science*, 71, 1969-1975.

- 39. Pandey, K.K., and A.J. Pitman. (2003). FTIR studies of the changes in wood chemistry following decay by brown-rot and white-rot fungi. *International Biodeterioration & Biodegradation*, 52:151–160.
- 40. Park, S.C. (1989). Preservation Brief 19: The Repair and Replacement of Historic Wooden Shingle Roofs. Washington D.C.: National Park Service.
- 41. Penofin. (2017). Safety data sheet. Penofin 550 VOC Red Label Ultra Premium Formula. Retrieved from <u>http://www.penofin.com/pdf/msds/MSDS-red-label-550.pdf</u>
- 42. Pine Tar World. (2014). Safety data sheet. Genuine pine tar 850. Retrieved from http://www.pinetarworld.com/wp-content/uploads/2015/06/GENUINEPINETAR850.pdf
- 43. Pine Tar World. (2014a). Safety data sheet. Gum turpentine. Retrieved from http://www.pinetarworld.com/wp-content/uploads/2015/06/GUMTURPENTINE.pdf
- 44. Pine Tar World. (2014b). Safety data sheet. Linseed oil, boiled. Retrieved from http://www.pinetarworld.com/wp-content/uploads/2015/06/LINDSEEDOILBOILED.pdf
- 45. Pospísil J., and S. Nespurek. (2000). Photostabilization of coatings. Mechanisms and performance. *Progress in Polymer Science*, 25:1261–1335.
- 46. Saver Systems. (2015). Safety data sheet. Defy Extreme Wood Stain. Retrieved from https://saversystems.s3.amazonaws.com/product-data/defy-extreme-wood-stain-sds.pdf
- 47. Scheffer, T.C. (1971). A climate index for estimating potential for decay in wood structures above ground. *Forest Products Journal*, 21(10):25-31.
- 48. Schniewind, A.P. (1963). Mechanism of check formation. *Forest Products Journal*, 13(11):475–480.
- 49. Stamm, A.J., and Loughborough, W.K. (1942). Variations in shrinking and swelling of wood. *Transactions of the American Society of Mechanical Engineers*, 64:379–385.
- 50. Sweetser, S.M. (1978). Preservation Brief 4: Roofing For Historic Buildings. Washington D.C.: National Park Service.
- Teacă, C.A., D. Roşu, R. Bodîrlău, and L. Roşu. (2013). Structural Changes in Wood Under Artificial UV Light Irradiation Determined by FTIR Spectroscopy and Color Measurements–a Brief Review. *BioResources*, 8(1):1748-1507.
- 52. Temiz, A., N. Terziev, B. Jacobsen, and M. Eikenes. (2006). Weathering, Water Absorption, and Durability of Silicon, Acetylated, and Heat-Treated Wood. *Journal of Applied Polymer Science*, 102 (5): 4506–13.
- 53. Timber Pro UV. (2016). Safety data sheet. Log & Siding, Log & Siding Smooth and Deck & Fence Semi-Transparent and Semi-Solid. Retrieved from <u>https://timberprocoatingsusa.com/wp-content/uploads/2017/03/Wood-Stain-Semi-Trans-Semi-Solid-SDS-Copy.pdf</u>
- 54. Tolvaj, L., and O. Faix. (1995). Artificial ageing of wood monitored by DRIFT spectroscopy and CIE L*a*b* color measurements. *Holzforschung*, 49(5):397-404.
- 55. U.S. Department of Commerce. National Oceanic and Atmospheric Administration. Earth System Research Laboratory. (n.d.). Location of US Climate Divisions. Retrieved from http://www.esrl.noaa.gov/psd/data/usclimdivs/data/map.html.
- 56. van Nieuwenhuijzen, E.J., M.F. Sailer, L.R. Gobakke, O.C.G. Adan, P.J. Punt, and R.A. Samson. (2015). Detection of outdoor mould staining as biofinish on oil treated wood. *International Biodeterioration & Biodegradation*, 105: 215-227.
- 57. Williams, R., M. Knaebe, and W. Feist. (1996). Finishes for exterior wood. Technical report. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory.
- 58. Williams, R.S., and Feist, W.C. (1999). Water repellents and water- repellent preservatives for wood. Gen. Tech. Rep. FPL–GTR–109. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory.
- 59. WSU AgWeatherNet. (2018). Monthly data. Washington State University. Retrieved from http://weather.wsu.edu/?p=93150.





Figure A 1. FTIR spectra of uncoated western redcedar control before weathering and following the weathering at three exposures.



Figure A 2. FTIR spectra of Pine Tar coated wood before weathering (Control) and following the weathering at three exposures.



Figure A 3. FTIR spectra of Chevron coated wood before weathering (Control) and following the weathering at three exposures.



Figure A 4. FTIR spectra of Heritage coated wood before weathering (Control) and following the weathering at three exposures.



Figure A 5. FTIR spectra of Messmer's coated wood before weathering (Control) and following the weathering at three exposures.



Figure A 6. FTIR spectra of Penofin coated wood before weathering (Control) and following the weathering at Arid and Maritime exposures.



Figure A 7. FTIR spectra of TWP 1500 coated wood before weathering (Control) and following the weathering at three exposures.



Figure A 8. FTIR spectra of DEFY coated wood before weathering (Control) and following the weathering at three exposures.



Figure A 9. FTIR spectra of Seal Once coated wood before weathering (Control) and following the weathering at three exposures.



Figure A 10. FTIR spectra of Sta Brite R coated wood before weathering (Control) and following the weathering at three exposures.