# Coatability of Oil-thermal-treated Post-MPB Lodgepole Pine Sapwood

Prepared for


by

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# Executive Summary

Thermal treatments to improve the dimensional stability and durability of wood for exterior applications impart a pleasant dark brown colour but this rapidly fades to gray when exposed to weathering. A coating may solve this problem but adhesion to oil-thermal-treated wood may be an issue. The general objective of this research is to investigate the feasibility of coating oil-thermal-treated post-Mountain Pine Beetle (MPB) lodgepole pine for above-ground residential products such as siding. This is a continuation of previous research in 2006/07 on treating post-MPB lodgepole pine sapwood with oil-thermal treatment, also funded by FII. The current project focuses on surface modification and coating systems evaluation for this treated pine by laboratory tests, and initiating field tests for monitoring long-term coatings performance.

The project was carried out in collaboration with Dr. Paul Cooper of the University of Toronto, Dr. Phil Evans of the University of British Columbia, and Dr. Sam Williams of the Forest Products Laboratory of USDA. Based on the study carried out by FPInnovations–Forintek Division, Sikkens Cetol 123 and SuperNatural showed good adhesion on oil-thermal-treated pine, but the appearance of SuperNatural was preferable for the targeted applications. Hence, SuperNatural was selected for a long-term field test in Vancouver.

Based on the study undertaken by FPL, an aluminum isopropoxide sol-gel precursor was able to improve surface adhesion of the oil-thermal-treated wood for a water-borne finish, but did not improve the adhesion for solvent-borne finishes. The oil-thermal treatment did not appear to appreciably change the hardness or Young's modulus of the wood based on the nano-indentation measurements. It was also found that the oil-thermal-treated wood could be easily treated with hydroxymethylated resorcinol (HMR), a coupling agent for coating. Its efficacy on coatings performance is being evaluated using an outdoor exposure test.

Based on the University of Toronto's study, the oil-thermal treatment reduced the wettability of the wood to a number of solvents and had an adverse effect on coating curing and adhesion. Light sanding improved the wetting and resulted in improved adhesion. Among all the finishes evaluated, SuperNatural clear finish formed a hard coat with good adhesion.

The study by the University of British Columbia found that plasma treatment is able to remove oil from the surface of oil-thermal-treated pine, and increased its wettability as well as adhesion to coatings. Scanning electron microscopy, confocal profileometry, and Fourier transform infra-red spectroscopy also indicated that high-energy plasma treatment impacted wood structures, particularly around pits. The consequence of the plasma treatment on coatings performance is being studied with a weathering test.

Overall, the study showed that oil-thermal-treated blue-stained pine can be coated to improve weathering performance for exterior above-ground applications. It confirmed that sanding can improve the coatings performance. The effects of a coupling agent and plasma treatment on coatings performance are to be reported. Thermal modifications may provide a promising way to improve dimensional stability and also mask blue stain for post-MPB lodgepole pine. However, the potential bleeding of oil from wood with initially intense blue stain poses a major challenge for coating application and for developing residential appearance products from the post-MPB lodgepole pine using such an oil-thermal treatment. In that case, alternative thermal treatment processes, particularly using steam as the heating medium, could be considered.

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# 1 General Objectives

- Characterize the wood surface after oil-thermal treatment.
- Investigate the effects of surface modifications on coating-related wood surface characteristics.
- Select coating systems for oil-thermal-treated post-MPB lodgepole pine sapwood, targeting outdoor above-ground residential applications such as siding.
- Initiate field testing on long-term coatings performance of oil-thermal-treated post-MPB lodgepole pine sapwood.

# 2 Introduction

This project is a continuation of the FII-funded project in 2006/07: Hot Oil-Treated Siding Using Post-MPB Lodgepole Pine Sapwood (Wang 2007), based on the extensive research done at the University of Toronto (Wang and Cooper 2003; 2004; 2005a; 2005b). The previous research at the University of Toronto compared different heating media, including various vegetable oils, industrial thermal oils and slack wax. Soybean oil stood out as the best option because soybean oil-treated wood showed higher decay and mould resistance during laboratory tests and exterior weathering tests. This was likely due to polymerization of the unsaturated fatty acids of soybean oil during and after the treatment. Generally, vegetable oil is more thermally stable, cost-efficient and user-friendly than most mineral oils. The previous research also investigated the effects of treatment temperature and time on moisture, biological and mechanical resistance, and it was found that 220ºC was an optimal treatment temperature, with two hours as the best compromise for properties of treated wood between moisture resistance, biological durability and mechanical performance. It was found that after thermal-oil treatment at 220ºC for 2 hours, both the hygroscopicity reduction and dimensional stability improvement (two indices for wood moisture resistance) of spruce and fir were about 40%, and the mass loss was reduced from over 60% to 32% during laboratory decay tests against *Gloeophyllum trabeum*. Meanwhile, MOR and MOE were reduced by about 40% and 20% respectively, together with significant reductions in abrasion resistance and hardness. So thermal-treated wood is usually only recommended for above-ground non-structural uses where the reduced strength is less critical.

In the previous FII project all the treatment was completed with a one-meter long oil bath, and the process indicated that the oil-thermal treatment should be feasible on a larger scale without too many barriers. Oil-thermal treatment at 220ºC for 2 hours or even 1 hour was able to effectively mask the blue stain of post-MPB lodgepole pine sapwood, and turn the wood into relatively uniform golden brown colour. However, the treated pine showed unsatisfactory weathering performance during the artificial laboratory and natural outdoor weathering tests (Fig. 1). Hence, it was suggested in that project that special surface treatments, or appropriate coating systems, should be developed for oil-thermal-treated wood if it is to be targeted at above-ground residential products.

***Figure 1 Appearance changes of the siding in Vancouver after 14 months***
(From right to left, ACQ-treated pine, oil-thermal-treated pine treated for 2 hours, oil-thermal-treated pine treated for 1 hour, cedar siding, and untreated pine. Each treatment has two columns)

This project aims to characterize the pine surface after oil-thermal treatment, investigate the effects of different surface pre-treatments, select optimal coating systems, and initiate field testing for evaluating long-term coatings performance. There has been very little specific research on the coating of oil-thermal-treated wood or other types of thermal modified wood. Previous research by the lead author at the University of Toronto indicated that oil-thermal-treated wood can still hold coatings, and coatings were very effective against weathering for outdoor applications. The work most relevant to this project was done by Petric *et al.*, (2007), who investigated the coating of oil heat-treated Scots pine treated with rape seed oil. It demonstrated that the treated wood had fewer surface defects such as flaking, blistering, cracking and mould growth, but poorer adhesion for coatings compared to untreated wood. Among all the transparent, semitransparent and opaque alkyds or acrylics tested, the partially pigmented semitransparent stains gave encouraging results. In the research the treated pine was planed, but no sanding or any other pre-treatments before coating was tried. Nor was any transparent water-based polyurethane used.

In order to improve the surface characteristics for this seemingly water repellent wood surface, pH adjustment, sanding, coupling agent treatment, and plasma treatment were chosen as pre-treatment methods in this study, and the related research was undertaken by FPInnovations–Forintek Division (Forintek), the University of Toronto (U of T), the Forest Products Laboratory (FPL) of USDA, and the University of British Columbia (UBC), respectively. A wide range of coatings were included in the laboratory screening tests.

# 3 Background

Wood thermal modification has been of particular interest in terms of its ability to improve dimensional stability and biological durability. Compared with wood treatments with preservatives or other chemicals, thermal treatment has the environmental advantage of being pesticide-free. The treatment processes which have been industrialized in recent years include: Thermo Wood (or Premium wood) developed in Finland (Syrjänen and Kangas 2000; Syrjänen 2001; Jämsä 2001; Militz 2002a, 2002b; Welzbacher and Rapp 2002), the Retification process (New Option Wood) and Bois Perdure in France (Dirol and Guyonnet 1993; Vernois 2001; Militz 2002a, 2002b; Welzbacher and Rapp 2002; Jermannaud *et al.*, 2002), and the Plato process in the Netherlands (Tjeerdsma *et al.,* 1998a, 1998b, 2000; Militz and Tjeerdsma 2001; Boonstra *et al.,* 1998; Militz 2002a, 2002b; Welzbacher and Rapp 2002). With the exception of the Retification process which uses nitrogen gas, all of these processes use steam as the heating medium to exclude oxygen, and all require accurate control of high temperature treatment to improve targeted wood properties. The duration for the high-temperature treatment lasts a few hours or longer, depending on the processes, heating media, wood species, and treatment purposes. In Canada wood thermal treatment processes including Perdure have also become industrially available, mainly in Quebec and Ontario (Tremblay 2006; Tremblay 2007; Leitch 2007). The purpose of thermal treatments in Canada appears to be more focused on enhancement of color and dimensional stability of hardwoods and softwoods for interior use, with less intensive thermal treatment, rather than on durability improvement of softwoods for exterior applications, with more intensive thermal modification (Leitch 2007). Kiln-dried high-grade lumber is usually used for treatment in order to reduce the thermal treatment cost and improve the treated wood quality, especially for appearance use products. Mills in Quebec offer treatment service at a base price varying from $450 to $600/Mbf (Tremblay 2007), more than double the costs estimated in Europe (about $\$100/m^3$, Militz 2002a, 2002b).

In addition to the above industrialized processes, a different approach using oil as the heating medium has been extensively investigated in Germany (Sailer *et al.,* 2000a, 2000b; Rapp and Sailer 2001; Militz 2002a, 2002b; Welzbacher and Rapp 2002; Nunes *et al.,* 2006), and it is likely to be commercialized in the near future. The research on such a thermal treatment has also been contributed considerably by other organizations, particularly the University of Toronto (Wang and Cooper 2003; 2004; 2005a; 2005b; Spear *et al.,* 2006). It was demonstrated that the moisture resistance and biological durability of the treated wood not only benefit from the high-temperature treatment, but also from the shell formed by the penetration of the water-repellent oil. In addition, such a treatment creates the potential for other additives to be added into the oil to further enhance wood properties.

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# 5 General Materials Preparation

## 5.1 Sample Preparation and Oil-thermal Treatment

Wood used in this project was mainly kiln-dried 1 in. by 6 in. post-MPB lodgepole pine boards heavy to blue stain. Fifty pieces of 900 mm long samples from 25 boards were cut for oil-thermal treatment. The treatment was carried out using a one-metre long oil bath in the wood science lab at the University of Toronto, with fresh soybean oil as the heating medium. The wood was treated at 220ºC, with a total treatment time of 2.5 hours, followed by oven-conditioning at 100ºC for about 20 hours. Then the treated wood, together with untreated wood, was distributed to FPL, U of T, and Forintek for laboratory surface characterization and coating screening tests. Due to the special size requirements for the plasma treatment, UBC purchased 2 in. by 4 in. post-MPB blue-stained pine sapwood, and followed a similar thermal treatment using a smaller oil bath in their own lab.

## 5.2 Coating Selection

A range of coatings were selected for laboratory coating performance screening tests by the four collaborating organizations. The general principle behind the selection was to choose the best products on the market which were highly promising for such treated wood, based on the expertise of all the scientists involved in the project. Consideration was also given to coatings with different chemistry. Since the major function of a coating is to reduce weathering mainly caused by exposure to UV, one criterion used was that the coatings should not be totally transparent. But it was hoped that the natural grain and golden brown color after the thermal treatment could still show through the finish. Hence, it was mainly exterior semi-transparent coatings that were chosen for tests. A white acrylic paint was included as a control. As a result, seven coatings, including six water-borne or solvent-borne semitransparent products, with different resins including polyurethanes, were selected for tests by Forintek and U of T (Table 1). FPL added another two types of coatings including Amteco TWP 101 and Sansin Enviro St. Gold for a wider range. It was also agreed by the four parties to use the tape test as the major adhesion evaluation test, according to ASTM D 3359-1997: Standard Test Methods for Measuring Adhesion by Tape Test. The cross-cut and tape test kits used by Forintek and U of T were made by Precision Gage & Tools Company, purchased from Folio Instruments based in Ontario.

***Table 1 Selected coatings for the laboratory screening tests***

| Coating type | Carrier | Product | Company | Colour | Coating No. |
|---|---|---|---|---|---|
| Film-forming | Water-borne | White acrylics | Benjamin Moore | White, as control | 1 |
| | | SuperNatural semitransparent coating | Napier | Clear natural cedar | 2 |
| | Solvent-borne | Sikkens Cetol 123 | Sikkens | | 3 |
| Non-film coating | Water-borne | Natural Deck Oil Cedar | Napier | Cedar tone | 4 |
| | | Semi-transparent Wood Sealer and Finish | Behr | | 5 |
| | Solvent-borne | UV Plus | Messmers | Natural | 6 |
| | | Deck Scapes™ | Sherwin-Williams | | 7 |

# 6 pH Adjustment and Coating Tests by Forintek

## 6.1 Rationale for pH Adjustment

The basic rationale behind the pH adjustment for the coating substrate is that the natural wood surface is slightly acidic, and pH affects wood characteristics and also the subsequent coatings stability. Highly acidic or alkaline wood surfaces, particularly the former, could contribute to rapid coating failure. It was hypothesized that a simple pH adjustment might provide enhanced coating stability.

## 6.2 Materials and Methods

### 6.2.1 pH Adjustment and Coating Application

Both treated and untreated 1 in. by 6 in. wood was cut into 8 cm long samples, with replication of five for treated wood and three for untreated wood for the pH adjustment and the subsequent coating tests. Measurement methods of wood surface acidity were investigated, including using extraction and titration or simply using pH indicator paper. The latter was chosen for its simplicity and also because it was non-destructive. One drop of distilled water was applied to the dry wood surface and then pH was measured after equilibrium using indicator strips with appropriate pH ranges. Before the formal tests pH adjustment of wood surface was examined with different acids including glycolic acid and acetic acid, and bases including 2-amino-2-methyl-1-propanol (AMP) and ammonium hydroxide. It was decided to carry out the pH adjustment with 10% AMP and 2% AMP, plus distilled water (as a control), with a specified amount of liquid for all treatments, targeting a wood surface pH of 10 (basic), 7 (neutral) and 4.5 (natural), respectively. It was found that the liquid was sucked into the surface of untreated wood quickly, but it took several hours to penetrate into the treated wood due to its water repellent surface. After the wood surface dried, pH was measured on the two surfaces of all samples (Table 2). Coatings in Table 1 were applied to both faces of pH modified treated and untreated pine, with the number of coats following manufacturer's instructions in most cases. For SuperNatural, two coats of Step One and one coat of Step Two were applied. For Sikkens, one coat of Cetol 1, and two coats of Cetol 23 were applied. Two coats were applied for Napier Natural Deck Oil Cedar and Benjamin Moore white acrylics, and one coat for Deck Scapes™, Messmer's UV Plus and Behr.

***Table 2 Average pH values of wood surfaces after pH adjustment***

| | 10% AMP | 2% AMP | Distilled water |
|---|---|---|---|
| Untreated wood | 9.4 (0.4)* | 7.8 (1.0) | 4.6 (0.2) |
| Oil-thermal-treated wood | 9.1 (0.4) | 7.1 (1.2) | 3.9 (0.4) |

** Values in parentheses are standard deviations.*

### 6.2.2 Coating Adhesion Tests and Accelerated Weathering Test

After coatings cured on the wood surface, adhesion evaluation using the tape test was carried out on one surface of each sample. The other face of each sample was subjected to an artificial weathering program to study the effect of weathering on adhesion and coatings performance. It was evaluated using the tape test based on ASTM D 3359-1997, and the rating scale for adhesion is from 0 to 5, with 5 for the best adhesion.

The accelerated weathering test was done using an Atlas Weather-Ometer® (model Ci65A) equipped with a 6500 watt, Xenon arc UV lamp and borosilicate inner and outer filters. This light source irradiated samples with near equal sunlight exposure with a lower UV wavelength cut-off at about 290 nanometers. The weathering program consisted of two phases: in each 2-hour cycle, there was 102 minutes' light and 18 minutes' water spray. The purpose of such a program was to simulate the UV and moisture conditions most siding products experience under natural conditions. The total duration of the exposure was 500 hours.

## 6.3 Effects of pH Adjustment on Coatings Performance

Overall, the adhesion values were all in the same range for treated and untreated blue-stained pine (Tables 3 and 4). Weathering appeared to differentiate coatings performance. Based on the limited data in this study, the adhesion of Natural Deck Oil Cedar on either treated or untreated wood was reduced significantly after the artificial weathering, and before the exposure it showed the best adhesion on oil-thermal-treated pine. Overall, Sikkens Cetol 123 and SuperNatural had the highest adhesion after the artificial weathering, followed by Behr Premium Sealer, Messmer's UV Plus and Benjamin Moore Acrylics for treated pine.

Based on the methods used and limited tests in this project, pH adjustment did not demonstrate significant effects on coating performance for either oil-thermally treated or untreated blue-stained pine. Relatively speaking, for treated wood, Behr Premium Sealer, SuperNatural, Deck Scapes™ and Messmer's UV Plus showed in slight favour of treatment with 10% AMP, but the adhesion data varied among replicates. Considering the water-repellent surface after the oil-thermal modification, it was concluded that pH adjustment may not be highly effective for such treated products.

***Table 3 Adhesion ratings for oil-thermal-treated pine with different initial pH***

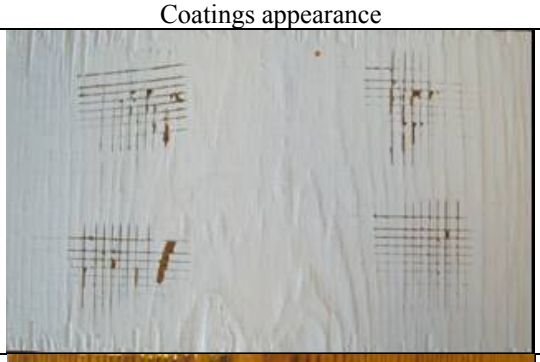
| Coatings | Ratings before weathering | | | Ratings after weathering | | |
|---|---|---|---|---|---|---|
| | 10% AMP | 2% AMP | None | 10% AMP | 2% AMP | None |
| Benjamin Moore Acrylics | 3 | 3 | 3 | 3 | 3 | 3 |
| SuperNatural | 4 | 3 | 3 | 4 | 3 | 4 |
| Sikkens Cetol 123 | 4 | 4 | 4 | 4 | 4 | 4 |
| Natural Deck Oil Cedar | 4 | 4 | 5 | 2 | 2 | 2 |
| Behr Premium Sealer | 4 | 4 | 4 | 4 | 3 | 3 |
| Messmer's UV Plus | 4 | 3 | 3 | 3 | 3 | 3 |
| Deck Scapes™ | 4 | 3 | 3 | 3 | 3 | 2 |

***Table 4 Adhesion ratings for untreated pine with different initial pH***

| Coatings | Ratings before weathering | | | Ratings after weathering | | |
|---|---|---|---|---|---|---|
| | 10% AMP | 2% AMP | None | 10% AMP | 2% AMP | None |
| Benjamin Moore Acrylics | 3 | 3 | 3 | 4 | 3 | 4 |
| SuperNatural | 3 | 4 | 5 | 4 | 3 | 3 |
| Sikkens Cetol 123 | 4 | 4 | 4 | 4 | 3 | 4 |
| Natural Deck Oil Cedar | 4 | 3 | 3 | 3 | 2 | 1 |
| Behr Premium Sealer | 4 | 2 | 3 | 3 | 3 | 3 |
| Messmer's UV Plus | 4 | 2 | 4 | 3 | 3 | 3 |
| Deck Scapes™ | 4 | 3 | 3 | 3 | 3 | 2 |

Considering adhesion along with the coatings appearance before and after the weathering test (Table 5), the opaque Benjamin Moore white acrylics could be peeled off the surface quite easily, indicating poor adhesion for the changed wood surface. Both Sikkens Cetol 123 and SuperNatural showed good adhesion on treated pine. However, the appearance of SuperNatural was preferable since it revealed the golden brown together with the intensified wood grain after the oil-thermal treatment, meanwhile the cedar tone of the product matched the treated wood color very well. By comparison, Sikkens was less transparent, and looked very yellow for the brown color of the treated wood. Among the remaining four coatings, all of them showed less satisfactory adhesion or weathering performance, and considerable erosion was found on wood surfaces coated with Natural Deck Oil Cedar, Messmer's UV Plus, and Behr Premium Sealer. Deck Scapes™ was also relatively opaque. It should be pointed out that the tape test may not provide a precise measurement of coating adhesion, and the laboratory artificial weathering test with limited exposure time is no substitute for field testing.

***Table 5 Coating appearance after weathering and adhesion testing***

| Coatings appearance | Coating name |
|---|---|
|  | Benjamin Moore Acrylics |
|  | SuperNatural |
|  | Sikkens Cetol 123 |
|  | Natural Deck Oil Cedar |
|  | Behr Premium Sealer |

| Coatings appearance | Coating name |
|---|---|
|  | Messmer's UV Plus |
|  | Deck Scapes™ |

During artificial weathering oil was observed bleeding from a few samples (Fig. 2), with or without coatings. It was noticed that pine samples with intense blue stain tended to absorb more oil during the oil-thermal treatment, and oil bleeding was also observed from the siding test of the previous project. This could pose a large challenge for coating application since coatings are not effective at preventing oil or resin bleeding. So it could create a major barrier for such oil-thermal treatment to be applied to post-MPB blue-stained lodgepole pine targeting above-ground residential appearance products.

***Figure 2 Oil bleeding from oil-thermal-treated pine presumably with initially intensive blue stain***

# 7 Conclusions

Based on the limited study done by Forintek, Sikken Cetol 123 and SuperNatural showed good adhesion on oil-thermal-treated pine, but the appearance of SuperNatural was preferable since it was more transparent, with its tone matching the color of the treated wood.

Thermal modifications may provide a promising way to improve dimensional stability and to mask blue stain for post-MPB lodgepole pine. However, the potential bleeding of oil from wood with initially intense blue stain poses a major challenge for coating application of oil-thermal-treated pine, and then for developing exterior use appearance products from post-MPB lodgepole pine using such an oil-thermal treatment.

# 8 Recommendations

Other thermal modifications without oil as the heating medium may be more suitable for post-MPB blue-stained lodgepole pine for masking discoloration and improving dimensional stability and durability, especially for pine with intense blue stain.

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# Appendix I

## Summary of Adhesion Test for Non-Weathered and Weathered Samples by Forintek

| Coating Name | pH Treatment | Pre-treatement | Non Weathered Adhesion Test (mean) | Weathered Adhesion Test (mean) |
|---|---|---|---|---|
| Benjamin Moore Acrylics | 10% AMP | None | 3 | 4 |
| Benjamin Moore Acrylics | 10% AMP | Oil | 3 | 3 |
| Benjamin Moore Acrylics | 2% AMP | None | 3 | 3 |
| Benjamin Moore Acrylics | 2% AMP | Oil | 3 | 3 |
| Benjamin Moore Acrylics | None | None | 3 | 4 |
| Benjamin Moore Acrylics | None | Oil | 3 | 3 |
| SuperNatural | 10% AMP | None | 3 | 4 |
| SuperNatural | 10% AMP | Oil | 4 | 4 |
| SuperNatural | 2% AMP | None | 4 | 3 |
| SuperNatural | 2% AMP | Oil | 3 | 3 |
| SuperNatural | None | None | 5 | 3 |
| SuperNatural | None | Oil | 3 | 4 |
| Sikkens Cetol 123 | 10% AMP | None | 4 | 4 |
| Sikkens Cetol 123 | 10% AMP | Oil | 4 | 4 |
| Sikkens Cetol 123 | 2% AMP | None | 4 | 3 |
| Sikkens Cetol 123 | 2% AMP | Oil | 4 | 4 |
| Sikkens Cetol 123 | None | None | 4 | 4 |
| Sikkens Cetol 123 | None | Oil | 4 | 4 |
| Natural Deck Oil Cedar | 10% AMP | None | 4 | 3 |
| Natural Deck Oil Cedar | 10% AMP | Oil | 4 | 2 |
| Natural Deck Oil Cedar | 2% AMP | None | 3 | 2 |
| Natural Deck Oil Cedar | 2% AMP | Oil | 4 | 2 |
| Natural Deck Oil Cedar | None | None | 3 | 1 |
| Natural Deck Oil Cedar | None | Oil | 5 | 2 |
| Behr Premium | 10% AMP | None | 4 | 3 |
| Behr Premium Sealer | 10% AMP | Oil | 4 | 4 |
| Behr Premium Sealer | 2% AMP | None | 2 | 3 |
| Behr Premium Sealer | 2% AMP | Oil | 4 | 3 |
| Behr Premium Sealer | None | None | 3 | 3 |
| Behr Premium Sealer | None | Oil | 4 | 3 |
| Messmer's UV Plus | 10% AMP | None | 4 | 3 |
| Messmer's UV Plus | 10% AMP | Oil | 4 | 3 |
| Messmer's UV Plus | 2% AMP | None | 2 | 3 |
| Messmer's UV Plus | 2% AMP | Oil | 3 | 3 |
| Messmer's UV Plus | None | None | 4 | 3 |
| Messmer's UV Plus | None | Oil | 3 | 3 |
| Deck Scapes™ | 10% AMP | None | 4 | 3 |
| Deck Scapes™ | 10% AMP | Oil | 4 | 3 |
| Deck Scapes™ | 2% AMP | None | 3 | 3 |
| Deck Scapes™ | 2% AMP | Oil | 3 | 3 |
| Deck Scapes™ | None | None | 3 | 2 |
| Deck Scapes™ | None | Oil | 3 | 2 |

# List of Reports Appended

Report from FPL: Coating of Oil-Thermal-Treated Post-MPB Lodgepole Pine

**Coating of Oil-Thermal Treated Post-MPB Lodgepole Pine**
Final Report 14 March 2008

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## Executive summary

The USDA Forest Service Forest Products Laboratory (FPL) collaborated with FPInnovations Forintek Division under a grant from Forestry Innovation Investment Ltd. to study the effect of surface treatments to improve adhesion of coatings to thermally treated wood. Other partners in the project included the University of British Columbia and the University of Toronto. Research at FPL had three components: sol-gel treatment of oil-thermally (henceforth also called "thermally treated") treated wood to improve the adhesion of finishes (Component 1), evaluation of wood cell wall using nano-indentation and atomic force microscopy to evaluate changes in wood properties caused by the thermal treatment (Component 2), and surface modification of thermally treated wood using hydroxymethylated resorcinol to improve adhesion of finishes (Component 3).

## Background

The mountain pine beetle (MPB) has infested widespread areas of North American forests, particularly in the Rocky Mountains (Canada and the USA). The infestation of the pine is usually fatal and leaves the sapwood of the tree discolored with blue stain. As the sapwood of lodgepole pine (*Pinus contorta* Dougl. ex Loud) is only about 2 inches thick, removal of one-inch thick boards from the outside of the log by sawing usually removes most of the stained wood. Although these boards are only a small part of the log, they are usually of good quality and could have high value for a variety of appearance grade applications. It has been found that oil-thermal treatment masks the stain and decreases water vapor uptake. The beetle-killed wood is also permeable and readily absorbs the oil used to thermally treat it. The oil gives the wood a brown color and improves the wood's water repellency. These attributes increase its value for many applications, but the color fades over several weeks when exposed to direct sunlight.

It is desirable to develop surface treatments and coatings systems to stabilize the brown color. Typical commercial coatings may not adhere well to oil-thermally treated wood, therefore it may be necessary to modify the wood's surface. It is crucial to identify the wood surface properties and characterize the interphase region between the oil-treated wood and the coatings. Various surface modifications may affect adhesion.

## Objectives

The FPL collaborated with FPInnovations Forintek Division under a grant from Forestry Innovation Investment Ltd. to study the effect of surface treatments to improve adhesion of coatings to thermally treated wood. The study included indoor and outdoor components and the indoor components were completed during April 1st 2007 to March 31st 2008. The outdoor component was started in October 2007 and will continue for several years. Subsequent reports will be forwarded on the outcome of the outdoor component as data is obtained. The objective of the studies conducted at the FPL were to modify the surface of thermally-treated wood using a sol-gel technique and evaluate the adhesion of several coatings

(component 1), evaluate the changes in cell was properties of the thermally-treated wood using nano-indentation (Component 2), and modify the thermally-treated wood with hydroxymethylated resorcinol (HMR) and evaluate the performance of several commercial coatings exposed outdoors (Component 3), The following was completed:

- Modified the surface of thermally-treated wood using sol-gel chemistry and evaluated the effect on adhesion of several commercial wood finishes
- Evaluated changes in cell-wall properties following oil-thermally modification using nano-indentation
- Modified the surface of thermally-treated wood with (HMR) and finished the boards with several commercial coatings (These boards were placed in outdoor exposure and will be evaluated over the next several years.)

# Component 1: Effect of sol-gel treatment on coating adhesion

The objective of this component was to evaluate the effect of alumina sol-gel treatment on adhesion of clear finishes to oil-treated MPB-impacted lodgepole pine.

**Experimental**

Sol-gel treatment

Hybrid inorganic/organic thin films have been used to modify the surface chemistry of wood. Such films were deposited on the wood substrates by the sol-gel process. The sol-gel process allows room-temperature deposition of hybrid inorganic–organic thin films on a wide range of substrates, including wood. With judicious choice of metaloxane precursors and deposition conditions, such thin films can be tailored to modify the coatability characteristics of oil-treated wood specimens.

Both oil-treated and control (without oil treatment) wood specimens were cut into 75 x 102 x 6 mm blocks, and divided into three sets of twelve specimens for each finish. As shown in Table 1, each set consisted of two alumina-treated, two heat-treated and two control specimens. Thus there were a total of 36 specimens, twelve of which were subjected to the sol-gel formulation, which contained an aluminum isopropoxide precursor. The other twenty-four specimens were subjected to heat treatment or used as controls.

**Table 1. Sample preparation scheme**

| Finish | Oil-treated | | | Control(without oil treatment) | | |
|---|---|---|---|---|---|---|
| | Alumina | Heat | Control | Alumina | Heat | Control |
| Minwax® Helmsman Spar Urethane | 2 | 2 | 2 | 2 | 2 | 2 |
| Zar® Ultra Exterior Polyurethane | 2 | 2 | 2 | 2 | 2 | 2 |
| Minwax® Water-based Polycrylic | 2 | 2 | 2 | 2 | 2 | 2 |

Prior to application of the clear finishes, oil-treated and control (without oil treatment) specimens were subjected to alumina sol-gel treatment by immersion for 24 h in an alumina sol followed by drying at 65ºC, and curing at 105ºC.

Each clear finish was applied in two coats, with the second coat applied after the first had been allowed to air-dry for 24 h. The amount of finish applied was determined by difference from weights of the finish container and brush applicator before and after each coat (Table 2). With only slight variation in the amount of finish applied to each specimen, it is assumed that the finish coating is of essentially the same thickness for each specimen.

**Table 2. Amount of finish applied to each specimen**

| Finish | Average Amount Applied (g) |
|---|---|
| Zar® Ultra Exterior Polyurethane | 2.1 ± 0.3 |
| Minwax® Helmsman Spar Urethane | 1.9 ± 0.2 |
| Minwax® Water-based Polycrylic | 2.3 ± 0.2 |

Adhesion of the finish to the substrate was evaluated using tape test method B of ASTM Standard D 3359-90. With this method, a lattice pattern with six cuts in each direction is made in the film on the substrate. Pressure-sensitive tape is applied over the lattice and then removed and placed on white paper for viewing under UV light. Adhesion was evaluated by comparing the adhesion pattern on the tape with illustrations and descriptions in the tape test method B, and scored on a five-point scale (Table 3).

**Table 3. Qualitative description of ASTM classifications**

| **ASTM Tape Test Method B Classification** | **Adhesion Strength** |
|---|---|
| 5 | Complete Adhesion |
| 4 | Very Good |
| 3 | Good |
| 2 | Poor |
| 1 | Very Poor |
| 0 | No Adhesion |

Three adhesion tape tests were conducted on oil-treated specimens, and three on control specimens. The initial three tape tests on oil treated specimens showed large variability; therefore three additional tape tests were conducted on these specimens.

**Results and discussion**

As shown in Figure1, the variability in adhesion strength of the clear finishes to the oil-treated specimens was about one unit and was about what we expected for this test. In spite of this variability some general trends in the adhesion characteristics of these finishes can be observed.

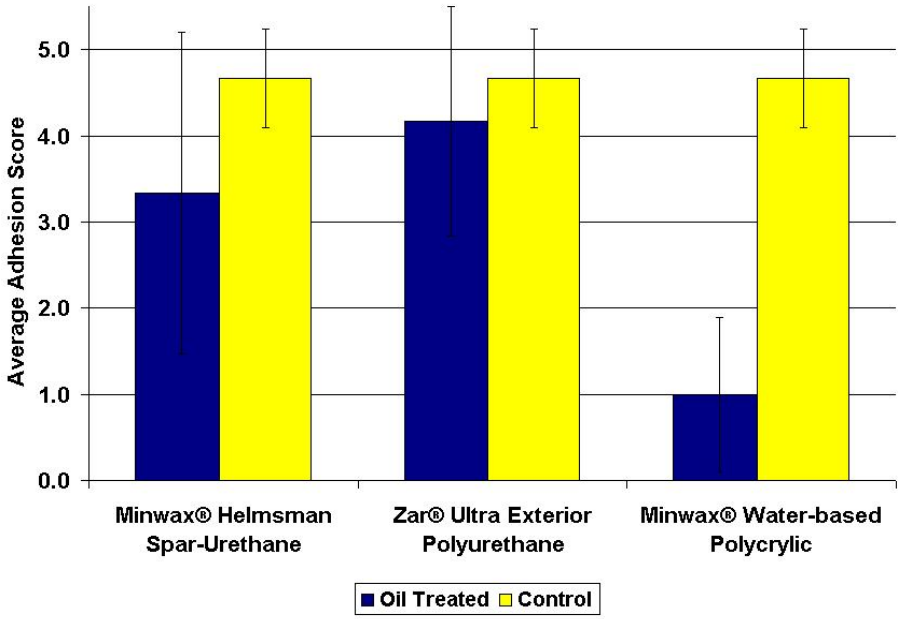


**Figure 1. Adhesion of clear finishes on oil-treated and control specimens prior to sol-gel treatment with alumina**

The solvent-borne polyurethane finishes (spar urethane and exterior polyurethane) appeared to have better adhesion to the oil-treated specimens compared to the water-borne Polycrylic finish. By comparison all three finishes showed very good adhesion to the control specimens that were not oil-treated. This would seem to suggest that the oil treatment weakens adhesion of the water-borne Polycrylic finish to MPB-impacted lodgepole pine, but has a much more limited effect on solvent-borne polyurethane finishes.

Alumina sol-gel treatment of the wood specimens resulted in dramatic changes in the adhesion behavior of the finishes on the oil-treated and control specimens. As shown in Figure 2 the solvent-borne spar urethane showed zero adhesion, while the exterior solvent-borne polyurethane showed very poor adhesion to the alumina-treated specimens, regardless of whether they were oil-treated or not. By comparison, alumina treatment noticeably improved the adhesion strength of the water-borne Polycrylic finish to both the oil-treated and control specimens.

In general it appears that alumina sol-gel treatment improved the adhesion strength of the water-borne Polycrylic finish to the oil treated MPB-impacted lodgepole pine, but degraded the adhesion strength of the solvent-borne polyurethane finishes.

It is worth noting that for both the oil-treated and control specimens, treatment with sol-gel alumina resulted in essentially the same adhesion properties. Both oil and control substrates, when treated with sol-gel alumina, exhibited poor adhesion for solvent-borne polyurethane finishes and very good adhesion for the water-borne Polycrylic finish. With both oil-treated and control substrates behaving similarly in this respect, it appears that the sol-gel alumina treatment plays a dominant role in determining finish

adhesion. This also suggests that the alumina sol-gel interacts with the oil-treated and control substrates in much the same way.

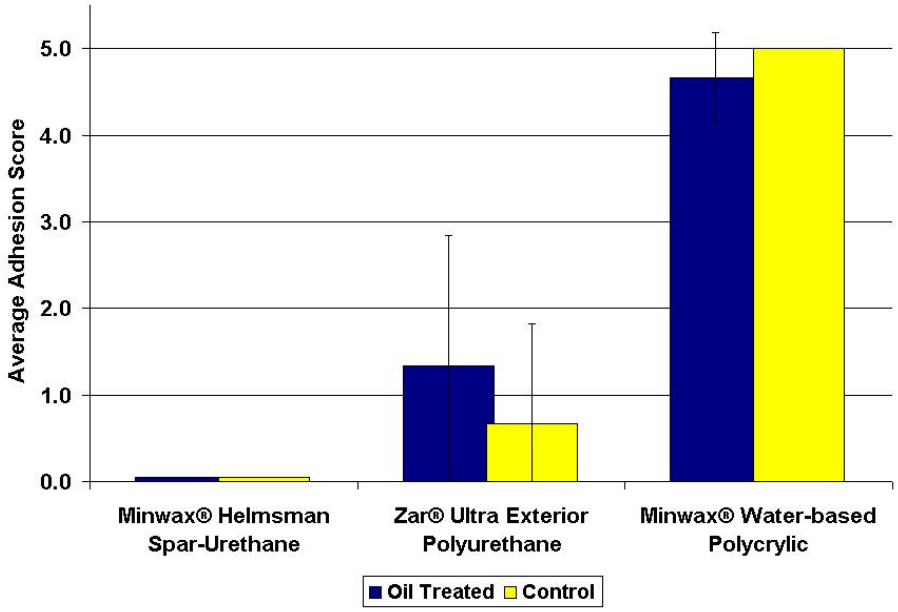


**Figure 2. Adhesion of clear finishes on oil-treated and control specimens after sol-gel treatment with alumina**

It was also interesting to investigate the effect of heat treatment on the adhesion of the clear finishes. Sol-gel alumina treatment requires exposure of the specimens to heat; therefore we wanted to ensure that the change in adhesion properties observed in the alumina treated substrates was wholly a result of the presence of alumina, and not exposure to heat.

Briefly, it appears that heat treatment of oil-treated and control specimens (Figure 3) had little effect on adhesion when compared to specimens, which were not exposed to heat treatment (Figure 2). Similar to the specimen set, which was not exposed to heat, the heat treated set exhibited relatively good adhesion with both the solvent-borne polyurethane finishes, but poor adhesion with the water-borne Polycrylic finish.

It seems that, within limits of experimental error, heat treatment does not affect the adhesion of commercial clear finishes as much as sol-gel treatment with alumina.

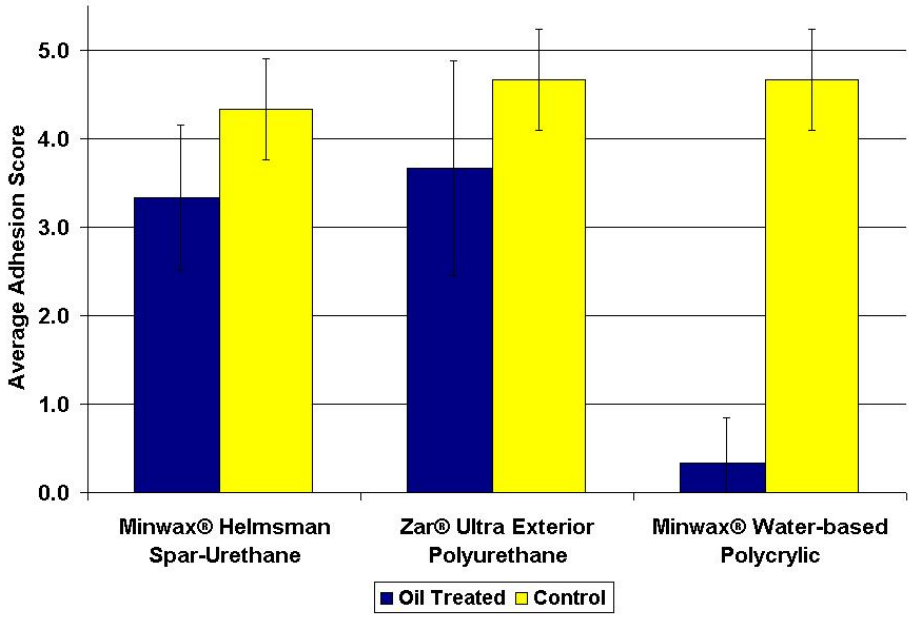


**Figure 3. Adhesion a of clear finishes on oil-treated and control specimens after conditioning for 6 h at 65ºC, followed by 24 h at 105ºC**

## Conclusions

Thermal-oil treatment of MPB-impacted lodgepole pine appears to have the effect of decreasing the adhesion strength of the commercial clear finishes examined in this study. Though the decrease in adhesion strength was relatively small for both solvent-borne polyurethane finishes, the decrease in adhesion strength for the water-borne Polycrylic finish was relatively large.

When oil-treated MPB-impacted lodgepole pine was treated with alumina sol-gel, adhesion for both solvent-borne polyurethane finishes was considerably degraded, while adhesion for the water-borne Polycrylic finish was greatly improved. These observations suggest that treatment with sol-gel alumina activates the oil-treated substrate surface to water-borne Polycrylic finish, and deactivates the surface to the solvent-borne spar-urethane and exterior polyurethane finishes. Consequently, if it is desired to apply a water-borne clear finish to oil treated MPB-impacted lodgepole pine, pretreatment with alumina sol-gel would be advantageous.

It should be noted that the study of the effect of heat treatment on adhesion of clear finishes to the oil-treated and control wood specimens was initially motivated by a desire to identify the cause of resin migration to the surface of some alumina-treated specimens (Figure 4). This surface resin bleed occurred on both oil-treated and control specimens after heat treatment. Similar surface resin bleed also occurred on both alumina-treated and alumina-free specimens. It appears that the resin bleeding is independent of both oil and alumina treatments, but dependent on heat exposure. With the extent of resin bleeding varying between similarly treated specimens, it seems that such bleeding may depend on the intrinsic concentration of resin in the wood at the time of specimen preparation. At this point it is unclear how this bleeding affects the alumina treatment, or if it will play an important role in the adhesion of commercial clear finishes to alumina-treated substrates.

Future work should include the study of the effect of sol-gel treatment on water repellence characteristics of thermally treated wood specimens.

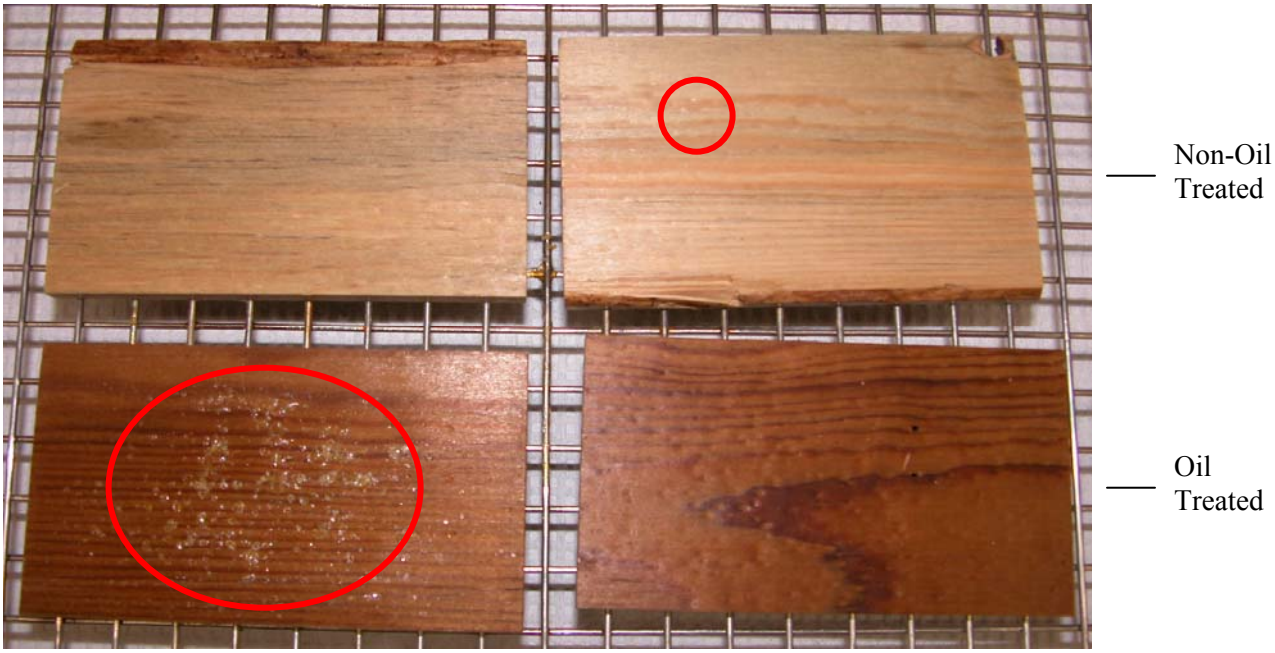


**Figure 4. Adhesion a of clear finishes on oil-treated and control specimens after conditioning for 6 h at 65ºC, followed by 24 h at 105ºC (resin beads circled in red)**

## Component 2: Mechanical Properties of Treated Wood Cell Walls Using Nano indentation

Nano-indentation was used to measure wood cell wall properties (hardness and Young's modulus) before and after thermal treatment. These mechanical properties were measured on a sub-micrometer scale, which should indicate the cell wall structures most affected by the treatment.

### Experimental

Sample Preparation

Small samples (about 1mm x 1 mm x 12 mm long) were removed from latewood rings. Two samples were removed from the same growth ring of the thermally treated wood; one sample (Specimen 2) was from a stained area, and the other (Specimen 3) from an unstained area. A sample (Specimen 1) was removed from a portion of the untreated wood, which was not affected by blue stain. The growth pattern of the untreated wood was much different than that of the treated wood. Specimen 1 was removed from the growth ring most comparable to that sampled for the treated wood. Figure 1 documents the locations that were sampled.

The toothpick-sized specimens were embedded in Spurr's epoxy, which was cured overnight at 70° C. The bottom of each specimen was machined flat and perpendicular to the cylindrical sides using a lathe. The upper portions were carved into a pyramid exposing a small portion of the transverse cross section of the wood samples. For nano-indentation a very smooth surface needs to be created which is parallel to the machined bottom of the embedment. This is accomplished by ultramicrotomy using a diamond knife. The microtomed surface was created by removing 1 to 2 µm of material in 100 nm slices to minimize damage to the wood.

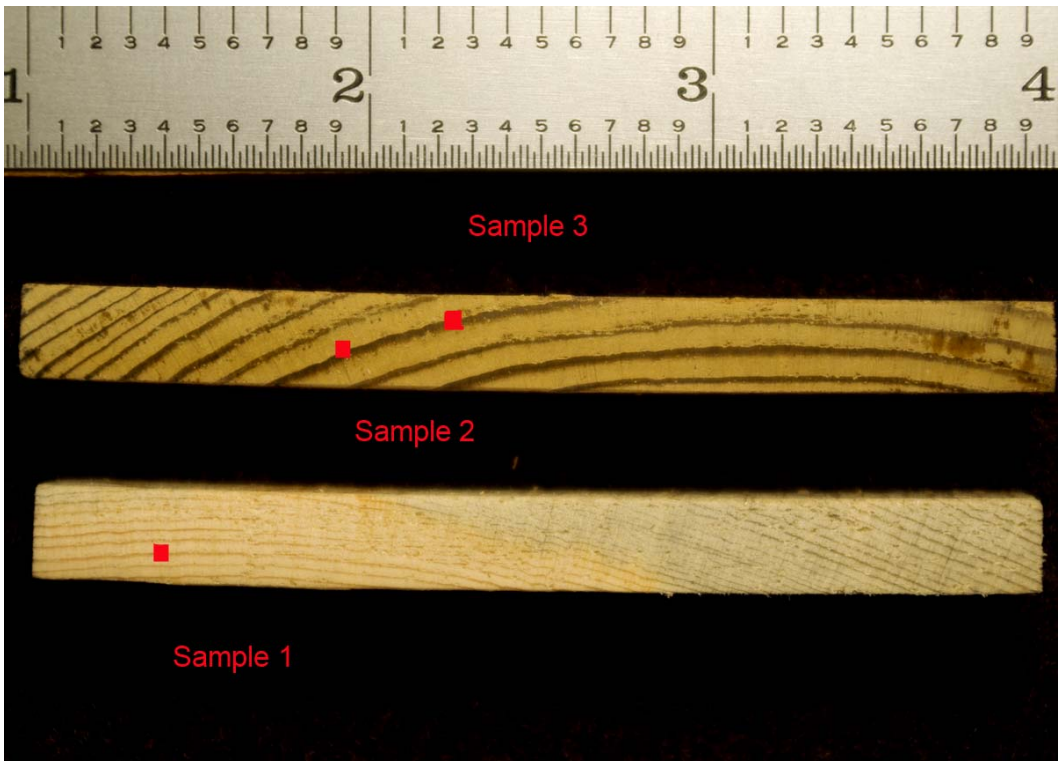


**Figure 1. Location of samples removed for this study**

During the preparation of the pyramidal ends prior to microtomy, it seemed that the treated samples were more plastic like and somewhat softer. While the indentation data clearly does not support this observation, measurements of the mechanical properties of the embedment epoxy and lignin rich middle lamella in each sample were made to determine if there were any differences among the samples. No differences were found.

Nanoindentation

The mechanical properties were determined by nanoindentation using a Hysitron TriboIndenter. A controlled force is applied to a diamond stylus with a shallow pyramidal tip (Berkovich tip) and the displacement is measured as the force is applied and removed. The hardness is determined from the maximum force applied and the projected area of the indentation. The area of the indentation is computed from an area function derived by controlled indentation of a fused silica standard. The modulus of elasticity is determined from the slope of the force-displacement plot as the stylus tip is withdrawn by relaxing the applied force.

Locations for measurement were selected by viewing the samples at the optical microscope station of the TriboIndenter. Figure 2 illustrates Specimen 1 and the areas where measurements were made. Each number indicates the location at which a series (4 to 10) of measurements were made. Most of the indentations were in the $S_2$ layer of the cell wall. Some measurements were made in the epoxy filling the lumens and the middle lamella between the cells.

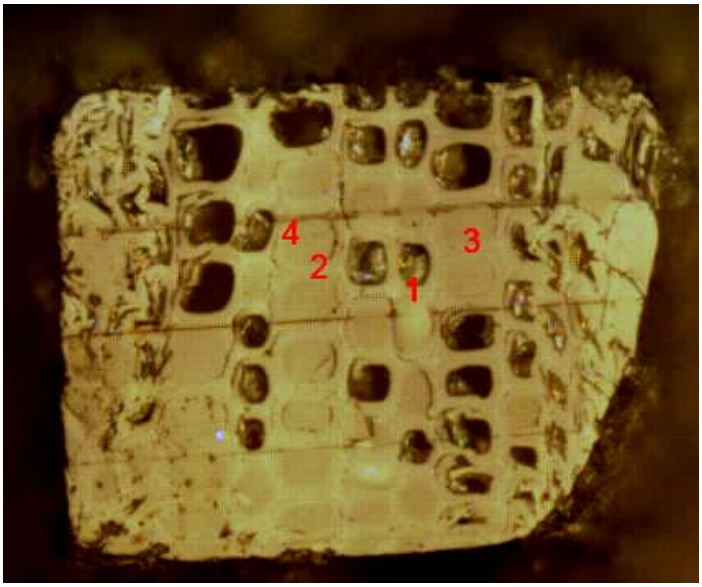


**Figure 2. Optical micrograph of the prepared surface of Sample 1**

At each selected area a topographical image was created by rastering the diamond tip over a 20 µm x 20 µm area while maintaining a very minimal force on the tip. Locations for indentation were selected from the topographical images. In each case the maximum indentation force was 600 µN (micronewtons). A representative force-displacement curve is shown in Figure 3. The tangent line on the right-hand side indicates the slope that determines the modulus of elasticity.

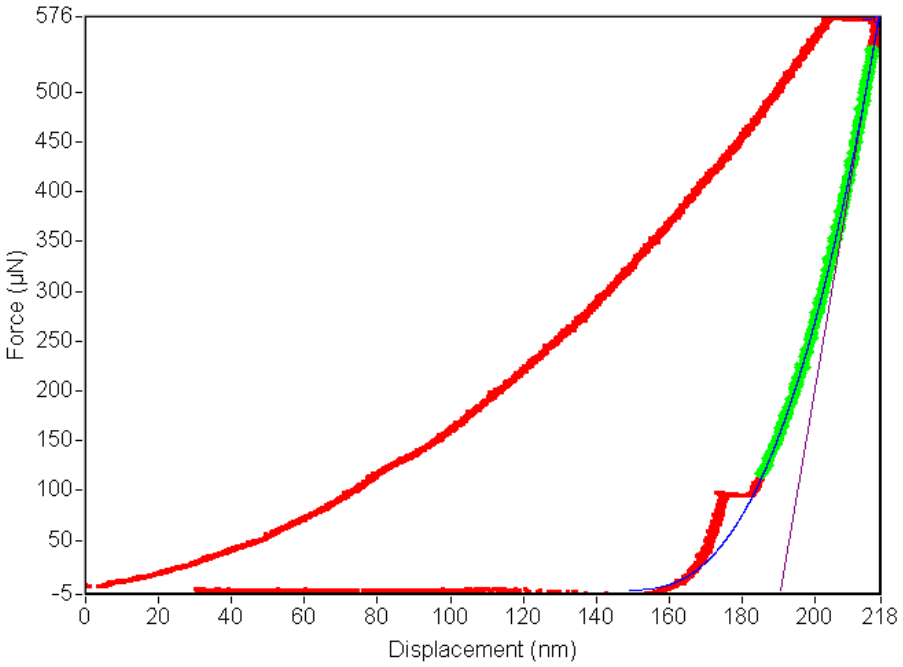


**Figure 3. Force-Displacement curve for a typical specimen**

After a series of indentations were completed, an atomic force micrograph (AFM) was prepared to document the location of the indents and the morphology of the site. Figure 4 is a typical AFM image. Six indents were placed in adjacent cell walls and four indents were placed in epoxy filled lumens. The indents in the epoxy are much larger, even though the same force was applied, because the epoxy is appreciably softer than the cell walls. The lateral ridges evident in the AFM image are knife marks caused by the edge of the diamond microtome knife.

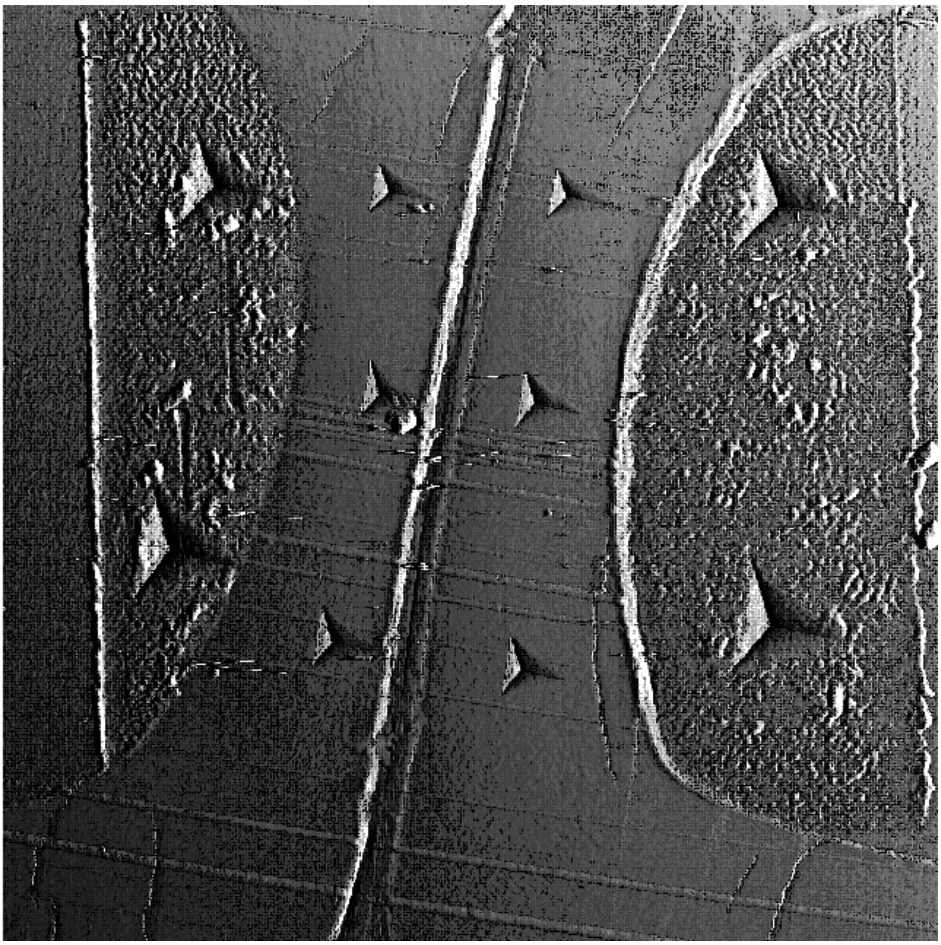

**Figure 4. Atomic force micrograph of Sample 3, Series 1. 25 µm x 25 µm**

**Results and Discussion**

The following mechanical properties were measured:

| Wood Cell Walls | $E_r$ (GPa) | Hardness (MPa) | number |
|---|---|---|---|
| Sample 1 (untreated) | 19.9 ± 1.4 | 656 ± 73 | 17 |
| Sample 2 (treated, stained) | 20.3 ± 2.3 | 714 ± 81 | 13 |
| Sample 3 (treated) | 23.0 ± 2.7 | 819 ± 86 | 15 |

| Middle Lamella | $E_r$ (GPa) | Hardness (MPa) | number |
|---|---|---|---|
| Sample 1 (untreated) | 10.7 ± 1.8 | 478 ± 61 | 4 |
| Sample 2 (treated, stained) | 11.3 ± 1.9 | 612 ± 184 | 5 |
| Sample 3 (treated) | 9.1 ± 0.4 | 721 ± 19 | 3 |

| Epoxy | $E_r$ (GPa) | Hardness (MPa) | number |
|---|---|---|---|
| Sample 1 (untreated) | 3.9 ± 0.4 | 205 ± 20 | 5 |
| Sample 2 (treated, stained) | 4.3 ± 0.1 | 176 ± 30 | 4 |
| Sample 3 (treated) | 3.9 ± 0.9 | 189 ± 27 | 4 |

Most of the variation in measurements reflects differences in properties from place to place. Smaller variation was found between measurements on a single wood cell. Although there was a small increase in hardness and modulus between the untreated sample (Specimen 1) and the stained thermally treated sample (Specimen 2) and also the unstained heat treated sample (Specimen 3), the differences fall within the range of variation. As stated in the introduction, there is no appreciable change in mechanical properties associated with this treatment.

The embedment epoxy has not been found to alter the wood properties to a significant extent. That is our experience in this laboratory and also has been reported elsewhere. The variation in the lignin (middle lamella) properties was not extensively sampled but does not suggest differences between the wood samples or differences related to treatment. The purpose of measuring these components was to explore the possibility of property change caused by preparation of the specimens.

None of these measurements were adjusted to account for sample compliance caused by proximity of different phases or cracks. These compliance effects are on the same order of variation as those observed between locations. A large number of measurements were made on all samples, reasonable locations were selected, and all samples were measured in the same manner, therefore the observation regarding differences in properties should be sound.

### Conclusion

The hardness and modulus of elasticity of cell walls of thermally treated lodgepole pine was measured by nano-indentation. No appreciable change in Young's modulus or hardness was found for the treated wood in comparison to an untreated wood.

## Component 3: Field study (HMR pretreatment)

### Surface modification

Hydroxymethylated resorcinol treatment—a coupling agent developed several years ago for use with wood adhesives has shown efficacy for bonding difficult substrates. The treatment is based on a brush application of an oligomer of hydroxymethylated resorcinol (HMR). HMR is prepared by reacting formaldehyde with resorcinol (1:1.5 mole ratio) at mildly alkaline aqueous conditions. In addition to other surface techniques such as sanding, pH adjustment, and plasma, a treatment with HMR on sanded and un-sanded wood might improve paint adhesion. The coupling agent can be applied by brush at ambient conditions and following a room temperature cure can be painted.

### Field studies

Boards (457 x 137 mm (18 x 4.5 inches)) were sectioned into four 102 x 137 mm areas for finishing with 25.4 x 137 mm areas left unfinished at each end. One half of the board (two sections) was treated with HMR. Each board had one finishes; Finishes were applied in primer and top coat or primer and two top coats according to manufacturers direction (Tables 2 and 3). Finishes 1-8 had three replicates; finish 9 had only two replicates.

Specimens were equilibrated to about 12% moisture content prior to finishing and finished in the laboratory under ambient conditions. HMR was prepared just prior to treating the boards and the primer was applied 5 days later. Subsequent coats were applied according to manufacturers direction, usually within several hours.

Tables 1 and 2 list finishes percent solids and Tables 3 show the application rates ($ft^2$/gal. and $m^2$/liter

All boards were placed on a test rack at outdoors near Madison Wisconsin vertically facing south (Figure 1).

**Table 1. Finish percent solids**

| Finish Applied | Wt./Gal. | % Solids |
|---|---|---|
| 1) Cetol 1 | 7.35 | 40.3 |
| Cetol 23 Plus | 7.45 | 53.4 |
| 2) Deck Scapes™ | 7.48 | 41.9 |
| 3) Messmer's U.V. Plus | 7.29 | 52.7 |
| 4) Behr Premium Sealer | 8.55 | 32.3 |
| 5) Amteco TWP 101 | 7.03 | 37.6 |
| 6) Natural Deck Oil Cedar | 8.46 | 13.3 |
| 7) Napier Supernatural Bl. | 8.67 | 38.9 |
| Supernatural U.V. Block | 8.76 | 55.6 |
| 8) Sansin Enviro St. Gold | 8.49 | 22.3 |
| 9) Benjamin Moore Lat.Pr. | 11.51 | 71.9 |
| Ben. Moore Ultra Lat. | 11.53 | 85.2 |

**Table 2. Finish application rates (Square feet/gallon)**

| Finish Applied | Untreated Wood | | Hot Oil Treated Wood | |
|---|---|---|---|---|
| | 1st | 2nd | 1st | 2nd |
| **(Coverage in Sq. Ft. / Gal.)** | | | | |
| 1) Cetol 1 | 100 | | 165 | |
| Cetol 23 Plus | 130 | 200 | 140 | 220 |
| 2) Deck Scapes™ | 115 | 150 | 190 | 195 |
| 3) Messmer's U.V. Plus | 70 | 130 | 170 | 275 |
| 4) Behr Premium Sealer | 125 | 150 | 195 | |
| 5) Amteco TWP 101 | 150 | 240 | 220 | 275 |
| 6) Natural Deck Oil Cedar | 120 | 140 | 195 | 185 |
| 7) Napier Supernatural Bl. | 125 | 215 | 180 | 210 |
| Supernatural U.V. Block | 145 | | 150 | |
| 8) Sansin Enviro St. Gold | 115 | 135 | 140 | 185 |
| 9) Benjamin Moore Lat.Pr. | 330 | | 370 | 200 |
| Ben. Moore Ultra Lat.Top | 265 | 425 | 385 | 450 |

**Table 3. Finish application rate (square meters/liter)**

| Finish Applied | Untreated Wood | | Hot Oil Treated Wood | |
|---|---|---|---|---|
| | 1st | 2nd | 1st | 2nd |
| **(Coverage in Sq. M / Liter)** | | | | |
| 1) Cetol 1 | 2.5 | | 4.1 | |
| Cetol 23 Plus | 3.2 | 4.9 | 4.4 | 5.4 |
| 2) Deck Scapes™ | 2.8 | 3.7 | 4.7 | 4.8 |
| 3) Messmer's U.V. Plus | 1.7 | 3.2 | 4.2 | 6.8 |
| 4) Behr Premium Sealer | 3.1 | 3.7 | 4.8 | 4.9 |
| 5) Amteco TWP 101 | 3.7 | 5.9 | 5.4 | 6.8 |
| 6) Natural Deck Oil Cedar | 3.0 | 3.4 | 4.8 | 4.5 |
| 7) Napier Supernatural Bl. | 3.1 | 5.3 | 4.4 | 5.2 |
| Supernatural U.V. Block | 3.6 | | 3.7 | |
| 8) Sansin Enviro St. Gold | 2.8 | 3.3 | 3.4 | 4.5 |
| 9) Benjamin Moore Lat.Pr. | 8.1 | | 9.1 | |
| Ben. Moore Ultra Lat.Top | 6.5 | 10.4 | 9.5 | 11.0 |

**Figure 5. Finished boards in outdoor exposure near Madison Wisconsin**

**Results and discussion**

All specimens were photographed and will be evaluated every six months during exposure. Evaluations will include substrate checking, discoloration, mildew, finish flaking, cracking, and erosion and general appearance.

## Summary, Components 1-3

Surface modification of thermally treated wood using an aluminum isopropoxide sol-gel precursor showed that the surface of thermally treated wood could be altered to improve the adhesion of a water borne finish, but did not improve the adhesion of solvent borne finishes. Thermal treatment did not appear to appreciably change the hardness or Young's modulus of the wood. Thermally treated wood could be easily treated with hydroxymethylated resorcinol (HMR). HMR has been shown to improve adhesion of wood, but the efficacy of this treatment to improve adhesion of finishes used in this study is pending results from outdoor exposure.

# Report from University of Toronto: Coating of Hot Oil-Treated Siding Using MPB Impacted Lodgepole Pine – Effect of Sanding on Coating Adhesion

# Coating of Hot Oil-Treated Siding Using MPB Impacted Lodgepole Pine – Effect of sanding on coating adhesion

**Paul Cooper and Tony Ung, University of Toronto**

## Introduction

Thermal analysis of wood in hot soybean oil not only modifies the chemistry of wood, rendering it more hydrophobic, but also leaves a surface film of partially cross-linked ("dried") soybean oil. Both of these effects may interfere with penetration and bonding of coatings in the wood surface. It was hypothesized that light sanding of the surface would reduce the second effect and improve the bonding quality of different finishes.

In this phase of the study, the surface properties of thermally treated MPB impacted lodgepole lumber was evaluated and the adhesion of test coatings evaluated.

## Background

Sanding is often a preferable method to renew weathered or aged wood surfaces prior to coating. Outdoor weathering reduces wood's surface energy, increasing contact angle, and increasing acidity. As a result the wood surface has reduced adhesion leading to poorer coatings performance (Grindl et al. 2004). Wood surface aging even happens indoors, and sanding is a very effective way not only to reduce roughness and lower relative quantity of coating for coverage, but also improve adhesion and coatings performance (Richter et al. 1995; Grindl et al. 2004; Sinn et al. 2004; de Moura and Hernandez 2005; 2006). The optimal grit size of sanding paper is around 100 (de Moura and Hernandez 2005; 2006). "

During thermal treatment, there are considerable changes in the chemical composition of wood. These changes result mostly in degradation of amorphous carbohydrates (Boonstra and Tjeerdsma 2006, Kamdem et al. 2002, Metsa-Kortelainen et al. 2006, Udaka and Furuno 2003) and consequently formation of acetic acid (Sundqvist et al. 2006, Tjeerdsma and Militz 2005), increase in cellulose crystallinity (Bhuiyan et al. 2000, Tejada et al. 1997, Udaka and Furuno 2003) and in apparent lignin content (Kamdem et al. 2002, Nuopponen et al. 2004). It is important to note that the hemicelluloses and cellulose are more prone to degradation than the lignin (Alen et al. 2002). Increase in wood acidity (Hodgin and Lee 2002, Kamdem et al. 2002) and decrease in extractive content (Kamdem et al. 2002, Nuopponen et al. 2003) are other reported changes.

The various chemical changes affect physical properties of wood such as increasing wettability (Hakkou et al. 2005, Mohammed et al. 2005, Petrissans et al. 2003); however, the effect on wettability when vegetable oil is used as the heat transfer fluid are not known. Thus, it is not clear what effect thermal treatment in soybean oil will have on wetting and adhesion of water based and oil based coatings.

It is hypothesized that sanding of the surface will improve coating wetting and bonding characteristics on soybean oil thermally modified wood, by removing surface oil.

**Methodology**

***1. Treatment***

Mountain pine beetle (MPB)-affected lodgepole pine boards were treated with soybean oil at 220ºC for 2 hours in our laboratory for the studies described below, as well as for studies conducted by other researchers.

***2. Surface properties***

**Measurement of wettability**

The contact angle was measured at 23°C using a goniometer by the sessile drop method. A 5-micrometer pipette was used to apply a drop of the probe liquid (the test solvent) manually. The contact angle was recorded five minutes after application. The probe liquids used for the test were, glycerol, ethylene glycol, formamide, glycerine, diiodoethane and water, representing a range of polarities, viscosities and acid and base characteristics.

To compare the effects of sanding on the surface properties, contact angles with the five probe liquids were measured after 5 minutes (20 replicates per treatment) for unmodified wood, modified but unsanded and modified and sanded samples.

***3. Coating adhesion***

Three pieces of soy oil thermally treated boards were prepared by lightly sanding one part of each board with 50grit sand paper (finer grit paper rapidly clogged with oil), then the whole board (sanded and not sanded areas) as well as untreated boards were coated with seven test finishes according to the manufacturers recommendations. The coatings tested were:

1. Supernatural Clear + UV finish with activator (Napier)
2. Low luster exterior acrylic latex with primer (Benjamin Moore)
3. Cetol 1 + Cetol 23 plus
4. Natural deck oil cedar tone
5. Weatherproofing wood sealer (Behr premium)
6. Deck scapes oil based transparent
7. Messmor's UV plus

The coated samples were allowed to cure at ambient temperature for 2 weeks before the adhesion test. A cross-cut kit by Precision Gage & Tools Company was used to evaluate the adhesion according to ASTM D 3359- 1997 : Standard Test Methods for Measuring Adhesion by Tape Test. American Society for Testing and Materials, Pennsylvania.

**Results and Discussion**

***1. Treatments***

Boards were heated in oil until the oil temperature reached 220°C, then held at that temperature for 2 hours. Samples were removed from the oil immediately to prevent cooling of the oil and excess absorption of oil into the permeable sapwood. As noted earlier, the treatment resulted in the wood becoming brown in colour with considerable masking of the blue stain in the wood.

***2. Surface Properties***

Contact angle measurements on surfaces of soy treated LPP for Water, Glycerol, Ethylene glycol, Diiodomethane and Formamide are shown in Table 1. For all probes, the contact angle was higher for the modified but unsanded samples than for the untreated wood or the modified and sanded wood. This indicates that the soybean oil on the wood surface reduced the wetting of all probes, and suggests that bonding of coatings might be impaired. The sanding treatment resulted in better wetting and produced results similar to those for the untreated wood.

**Table 1: Effect of sanding on contact angle of lodgepole pine thermally modified with soybean oil (average (S.D.) contact angle in degrees)**

| | Liquid probe | | | | |
|---|---|---|---|---|---|
| **Wood surface** | **Water** | **Ethylene glycol** | **Glycerine** | **Formamide** | **Di-iodomethane** |
| Unmodified | 47 (5.5) | 21 (9.2) | 45 (3.2) | 18 | 11 |
| Modified, Unsanded | 65 (6.0) | 39 (6.8) | 55 (5.3) | 36 (10) | 20 (7.4) |
| Modified, Sanded | 45 (10.0) | 16.5 (7.1) | 38 (7.6) | 0 | 16 (7.7) |

### *3. Coating adhesion*

The results for the grid peel test are shown in the Table 2 and the photos in the Appendix.
Coatings that produced a hard film (coatings 1, 2 and 3) could be reliably assessed by the tape method. The other coatings did not cure to a hard film and the soft coating or film was easily scratched off. However, these often showed high ratings by the tape test because the tape did not adhere well to the surface and the expected tear stress was not developed. Thus the ratings shown in the table below are not good indicators of adhesion for these coatings.

Of the coatings that produced hard films, Coating 1 had excellent film adhesion and coating 2 had good adhesion; coating 3 was not as well bonded. Thermal modification in soybean oil had an adverse effect on all coating's adhesion. For thermally modified samples, without sanding, coatings 4, 5 and 7 were the least affected by the oil treatment followed by coatings 1, 6, 2 and 3. The acrylic latex coating (#2) was the most adversely affected by the treatment. Lightly sanded samples had somewhat improved adhesion characteristics; however, adhesion of sanded samples was still worse than for untreated samples.

**Table 2: Grid peel test results (ASTM D3359)**

| **Coating** | **Peel off classification** | | | **Comments** |
|---|---|---|---|---|
| | **Untreated** | **Unsanded** | **Sanded** | |
| 1. Supernatural Clear | 5B,5B,5B,5B | 3B,4B,4B,3B | 3B,4B,4B,5B | Hard coat, good adhesion |
| 2. Benjamin Moore Acrylic latex with primer | 4B,4B,5B,5B | 2B,2B,3B,2B | 2B,2B,3B,3B | Hard coat, poor adhesion |
| 3. Cetol 1 + Cetol 23 plus | 3B,3B,4B,5B | 2B,3B,3B,1B | 3B,3B,5B,2B | Film forming, poor adhesion |
| 4. Natural deck oil cedar tone | 4B,4B,5B,5B | 4B,4B,4B,5B | 4B,4B,5B,5B | Soft coat, easily scratched off |
| 5. Behr premium wood sealer | 5B,5B,5B,5B | 4B,4B,4B,4B | 4B,4B,4B,5B | Soft coat, easily scratched off |
| 6. Deckscapes oil | 5B,5B,5B,5B | 3B,3B,3B,4B | 3B,3B,4B,5B | Film forming, soft coat |
| 7. Messmor's UV plus | 5B,5B,5B,5B | 4B,4B,4B,4B | 4B,4B,4B,5B | Soft coat, easily scratched off |

| Note | Classification | % Area Removed |
|---|---|---|
| | 5B | 0% |
| | 4B | <5% |
| | 3B | 5-15% |
| | 2B | 15-35% |
| | 1B | 35-65% |
| | 0B | Greater than 65% |

The best coatings on the soybean oil thermally modified samples, whether sanded or not, were the Natural deck oil (coating 4), followed by the Behr premium wood sealer (coating 5) and Supernatural Clear (coating 1). The Supernatural clear finish (coating 1) formed a hard coat with good adhesion. The Behr premium, Messmer UV and Natural deck oil are penetrating stains and did not form a hard finish and they could be easily scratched off. The soy oil appeared to have some interference on the curing of these finishes. The last three finishes i.e. Deckscapes, Cetol and Benjamin Moore all are film forming but with rather poor adhesion.

**Conclusions**

1. The soybean oil treatment reduced the wettability of the wood to a number of solvents and had an adverse effect on coating adhesion.
2. Light sanding improved the wetting properties and resulted in improved adhesion, although not as good as untreated wood.
3. Of the hard film forming coatings, the supernatural clear gave the best results.

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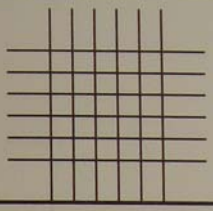
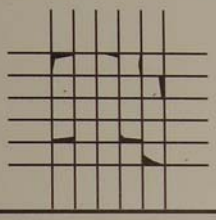
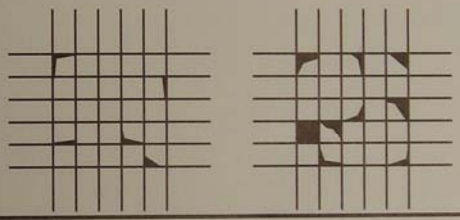
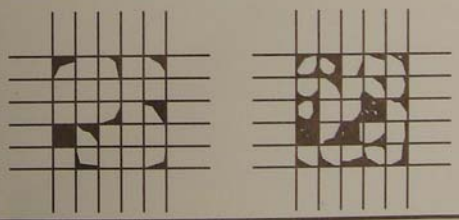
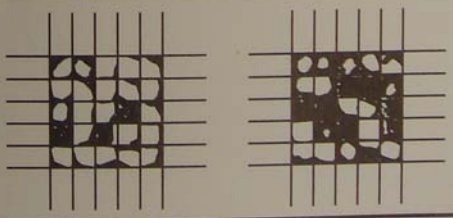
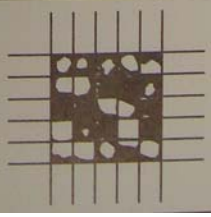
# APPENDIX

## Classification of adhesion and photos of coatings adhesion results

**Classification of Adhesion Test Results**

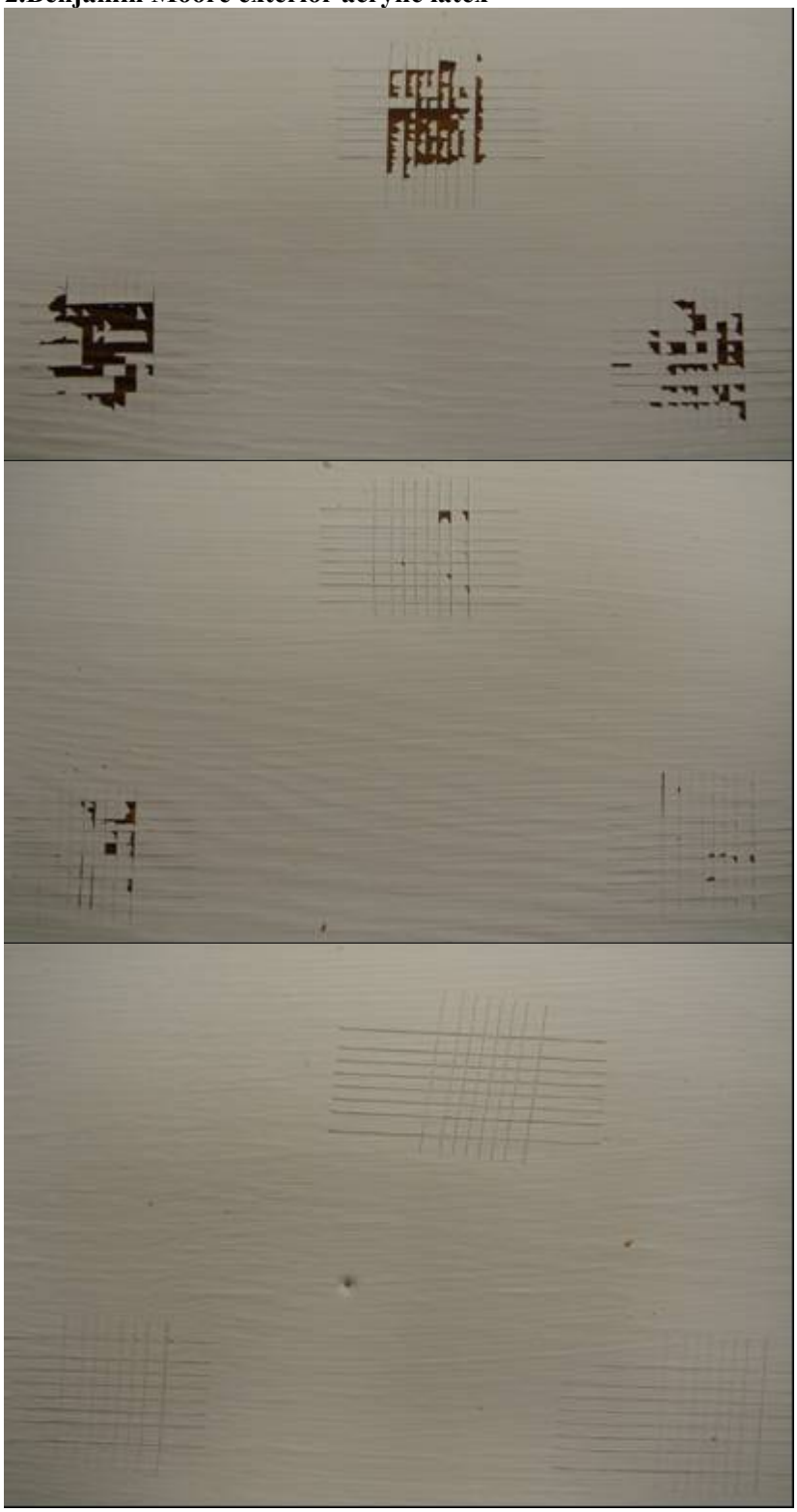
| Classification | Percent Area Removed | Surface of crosscut area from which flaking has occurred for six parallel cuts and adhesion range by percent |
|---|---|---|
| 5B | 0% None |  |
| 4B | Less than 5% |  |
| 3B | 5–15% |  |
| 2B | 15–35% |  |
| 1B | 35–65% |  |
| 0B | Greater than 65% |  |

**1.Supernatural (Napier)**

Sanded face

Untreated wood

**2.Benjamin Moore exterior acrylic latex**



Sanded face

Untreated wood

**3.Cetol 1 & Cetol 23 plus**

Sanded face

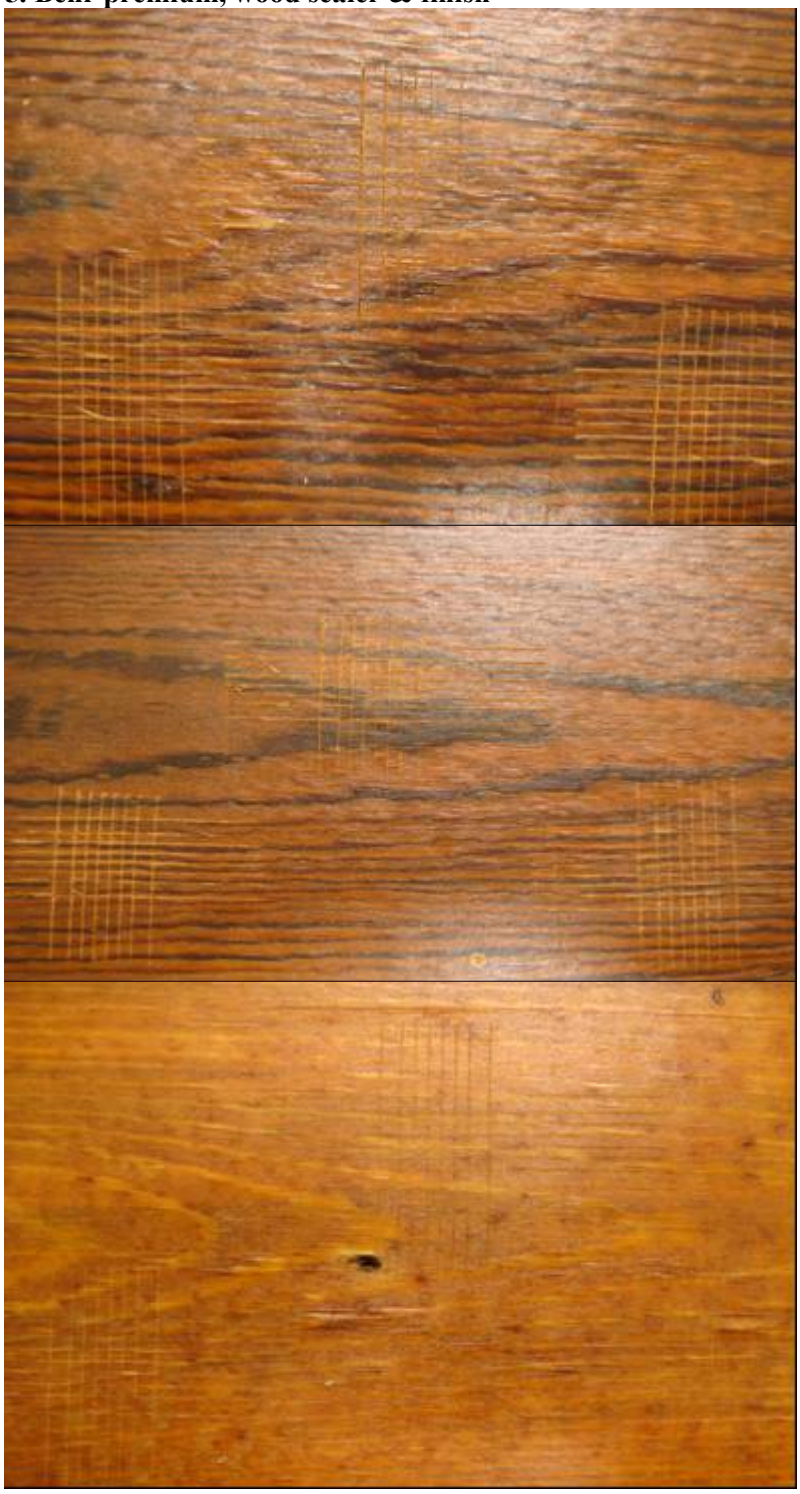
Untreated wood

**4.Natural Deck Oil**

Sanded face

Untreated wood

**5. Behr premium, wood sealer & finish**



Sanded face

Untreated wood

**6.Deckscapes Oil based Semi Transparent**

Sanded face

Untreated wood

**7.Messmer's UV plus (oil based)**

Sanded face

Untreated wood

# Report from University of British Columbia: Plasma Treatment of Oil-Modified MPB Wood

# Plasma Treatment of Oil-modified MPB Wood

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## 1.0 Introduction

Thermal modification increases the dimensional stability and decay resistance of pine, and changes its colour (from light yellow to brown). Blue stained pine from trees infested by the mountain pine beetle (MPB-wood) is less dimensionally stable than unstained wood, and the blue colour of sapwood is disliked by consumers (Byrne et al. 2006). Hence, thermal modification is seen as a simple way of improving two key properties of MPB wood. Thermal modification of MPB-wood with hot soybean oil has been shown to improve the wood's dimensional stability and mask the blue stain, but there is concern that the hydrophobicity of the oil modified wood may affect the adhesion and performance of coatings applied to the wood. The adhesion of coatings on hydrophobic substrates can be improved by modifying the material with plasma. For example, plasma treatments are used industrially to improve the adhesion of coatings on plastics, and plasma treatment using inorganic gases has been used to increase the wetting properties and adhesive bond strength of wood (Uehara and Jodai 1987, Sakata *et al.,* 1993, Podgorski *et al.,* 2000). This study examined the plasma treatment of hot-oil modified MPB-wood, and in particular whether the treatment could improve the adhesion and performance of coatings on the modified wood.

## 2.0 Materials and Methods

Initial experimentation examined the effect of plasma modification on the structure and chemical characteristics of hot-oil modified MPB wood. Subsequent experiments examined the effect of plasma modification on the surface energy and adhesion of finishes on hot-oil modified MPB wood. Finally, an experiment examined the effect of plasma modification on the performance of finishes on hot-oil modified MPB wood.

### 2.1 Thermal modification and plasma treatments

Wood was thermally modified by placing pre-weighed and conditioned samples in a oil-bath containing soybean oil at 220 ºC. Modified wood was removed from the oil bath after 2 hours and blotted on paper towels to remove excess oil. Hot-oil modified MPB wood and unmodified wood samples were treated

with plasma in a reactor that was designed to treat silicon wafers to produce clean, high energy, surfaces. One wood sample was placed in the chamber of the plasma reactor at a time, and a vacuum of 0.15 ± 0.01 torr was drawn. A valve was opened to allow water vapour from a glass reservoir into the chamber and the vacuum was redrawn. Radio frequency (R.F.) energy at 125 kHz was transmitted to the treatment chamber. The energy applied to the samples was varied from 5 kJ to 200 kJ, by changing the length of treatment. Samples that were subjected to vacuum acted as controls. After treatment the chamber was vented to atmosphere. Samples were removed from the chamber, taking care to avoid touching and contaminating their upper surfaces.

### 2.2 Scanning electron microscopy and confocal profileometry

Scanning electron microscopy was used to examine the effects of oil and plasma treatment on the structure of MPB-wood. Samples measuring 15 x 15 x 30 mm were cut from blue stained lumber. These blocks were soaked in water for 3 days and individual blocks were clamped in a small vice beneath the stage of a low power binocular microscope with their radial face uppermost. A sharp single-edged razor blade was then used to manually slice thin (20 to 30 μm) sections from the radial longitudinal face of each specimen until a clean, undamaged surface was obtained. Specimens were dried over silica gel at 20 ± 1$^{o}$C for 24 h. Four types of specimens were prepared using procedures described above: (1). Hot oil-treated MPB wood; (2). Hot oil-treated MPB wood that had been treated with 50 kJ of plasma (medium plasma); (3). Hot oil-treated MPB wood that had been treated with 200 kJ of plasma (very high plasma); (4). Untreated MPB wood, which acted as a control. Specimens were dried over silica gel at 20 ± 1$^{o}$C for 24 h and reduced in size to ~5 x 5 x 8 mm using a razor blade. They were then glued to separate aluminium stubs using Nylon nail polish as an adhesive. The stubs were sputter coated with an 8 nm layer of gold and they were then examined using a Hitachi S-2600 variable pressure scanning electron microscope at accelerating voltages of 5 to 6 kV. Secondary and back-scattered electron images of samples were obtained and saved as TIFF files.

Non-contact surface profileometry was used to probe the surface structure of hot-oil modified and plasma treaed MPB wood. One specimen measuring 38 x 89 x 220 mm was cut from five different pieces of dimensional lumber (2 x 4) containing MPB-affected wood. An area measuring (1.5 x 1.5 mm) was marked on the radial surface of each specimen and an AltiSurf 500 profileometer (probe No.2- 300μm) was used to image the wood in this area. Each specimen was modified in hot oil (as above) and the surfaces of the specimens were re-imaged. A sample measuring 3 x 15 x 30 mm and containing the scanned area was cut from each of the oil-modified specimens. These samples were then treated with plasma (200 kJ), and the surfaces of the samples were re-imaged. The software Papermap was used to produce topographical images of oil-modified and plasma treated wood surfaces.

## 2.3 Fourier transform infra-red spectroscopy

Fourier transform infra-red spectroscopy was used to examine the effect of plasma treatment on the surface chemical properties of hot-oil modified and plasma treated wood. This technique was better at picking up such changes than electron spectroscopy for chemical applications (ESCA). Six samples measuring 15 x 15 x 30 mm were cut from MPB wood. These blocks were divided into 2 groups. Two spots were marked on each block and FTIR spectra of the wood surfaces were obtained using a single bounce attenuated total reflectance accessory (PikeMiracle) attached to a Perkin Elmer Spectrum One spectrometer. The penetration of infra-red radiation into the wood sample was approximately 1.2 μm and each spectrum represented 16 accumulations at 8 $cm^{-1}$ resolution. Samples from the first group were treated with plasma and infra-red spectra of the marked areas were obtained. Samples from second group were thermally modified, as above and FTIR spectra of modified samples were obtained. Samples were then plasma treated and FTIR spectra of treated samples were obtained.

## 2.4 Surface contact angle and paint adhesion

Five pieces of blue-stained pine (2 x 4" x 8') with their growth rings oriented tangentially to their wide faces, were purchased from a big box store. One defect free sample measuring 38 x 89 x 220 mm was cut from each piece of lumber. These pieces were placed in a conditioning room at 20 ± 1 ºC and 65 ± 5% r.h. for 72 h and weighed. Each piece was separately heat-treated as described above. These pieces were then blotted on paper towels to remove excess oil, conditioned for 24 h, as above, and reweighed. Five samples measuring 3 x 38 x 60 mm were cut from the surface of each piece of heat-treated lumber. Three parallel grooves were then sawn into the surface of each sample to delimit areas measuring 15 x 38 mm. The samples were then subjected to the following treatments; (1). Vacuum treated control (Vac, 0 J); (2). Low energy plasma treatment (LP, 5 kJ); (3). Medium energy plasma treatment (MP, 50 kJ); (4). High energy plasma treatment (HP, 100 kJ); (5). Very high energy plasma (VHP, 200 kJ). The effects of plasma treatments on the contact angle of a water droplet applied to the surface of the hot-oil-modified plasma treated blue-stained pine was then assessed. Modified samples were placed on a platform, which was then adjusted so that it was level with a horizontal microscope containing a goniometer eyepiece. A 25 μL droplet was placed on the surface of the modified sample and the contact angle that the droplet made with the wood surface was measured within 10 seconds. A second measurement was then made. Droplets were wiped from the surface of treated specimens and the four areas within each specimen were brush coated with the following finishes according to manufacturers instructions; 1. Sikkens 123 (2 coats); (2). Coelan polyurethane boat finish (2 coats); (3). Supernatural water based coating (2 coats); (4). CIL acrylic primer (1 coat) and (5). CIL Dulux topcoat (1 coat). The specimens finished with the different coatings were

sawn from samples and kept in a conditioning room for 1 week. The dry adhesion of the coatings to oil-modified plasma treated wood was assessed using a tape test. After the test, all samples were scanned using a desk-to scanner and coating adhesion for each piece was rated in accordance with the standard classification scale. Analysis of variance was used to assess the effects of plasma treatment on contact angle, and treatment and coating on paint adhesion.

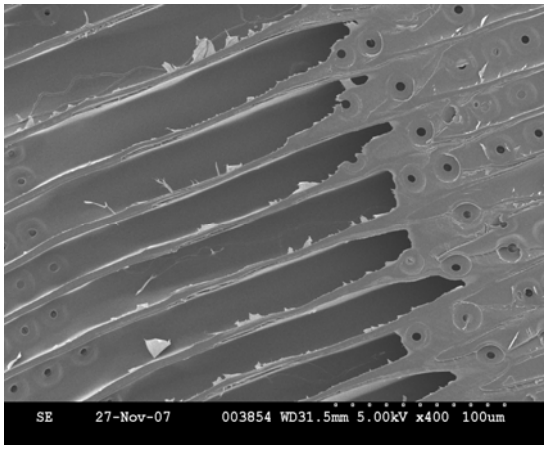
### 2.5 Performance of coatings on hot-oil modified plasma treated MPB wood

Five pieces of blue-stained pine (2 x 4" x 8') with their growth rings oriented tangentially to their wide faces, were purchased. Two defect free samples measuring 38 x 89 x 240 mm were cut from each piece of lumber. These pieces were placed in a conditioning room at 20 ± 1 ºC and 65 ± 5% r.h. for 72 h and weighed. Each piece was separately heat-treated as described above. Four samples measuring 3 x 55 x 89 mm were cut from the surface of each piece of heat-treated lumber. Four parallel grooves were then sawn into the surface of each sample to delimit areas measuring 17 x 55 mm. These areas were coded according to the experimental design. Wood samples were plasma treated with 100 kJ of energy. The five areas within each specimen were brush coated with the following finishes according to manufacturers instructions; (1). Exterior acrylic latex primer (1 coat) and paint (1 coat); (2). Natural deck oil (2 coats); (3). Natural wood finish (1 coat); (4). Weather proofing, wood sealer and finish (1 coat); (5). Premium oil-based deck stain (1 coat). Finished specimens were conditioned, attached to glass backing plates and exposed to the weather on a rack inclined at 45˚ to the vertical and facing south.

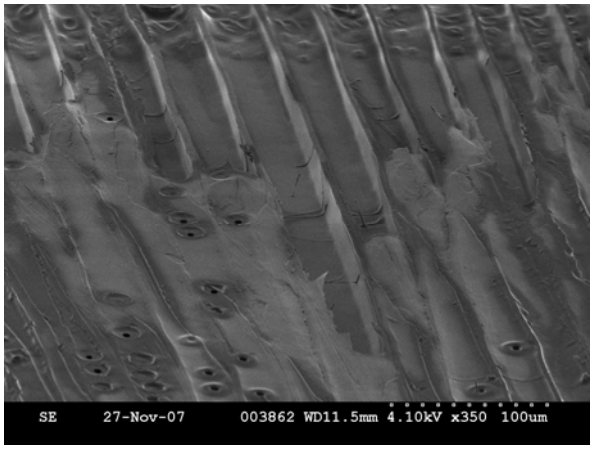
## 3.0 Results & Discussion

### 3.1 Scanning electron microscopy and confocal profileometry

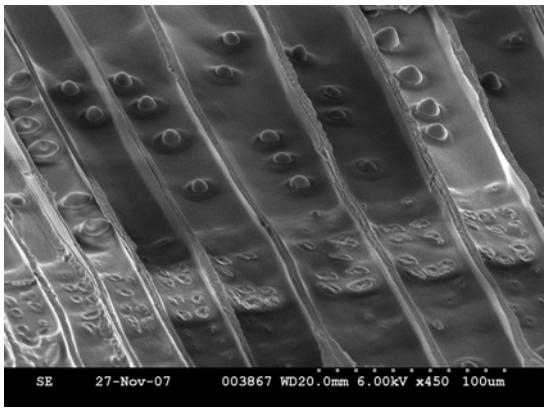
Scanning electron microscopy revealed that the oil treatment deposited oil within the wood structure (Fig. 1, compare a v b, below). Plasma treatment removed oil from the wood (see Fig 1c below). Plasma treatment also modified the structure of the pits in oil treated wood by causing pit membranes to balloon through pit openings (Fig. 1c). At high plasma treatment energies there was significant etching and removal of cell wall material (Fig. 1d-f).



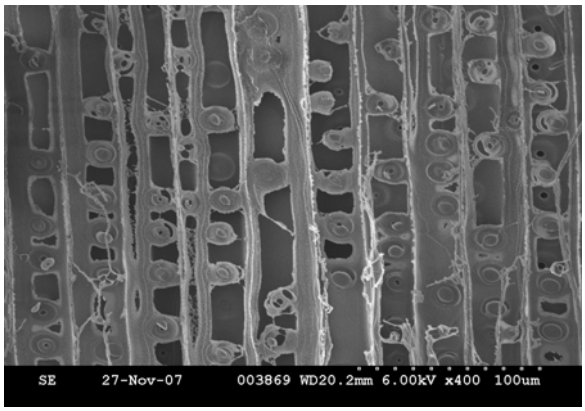

(a) Untreated MPB wood



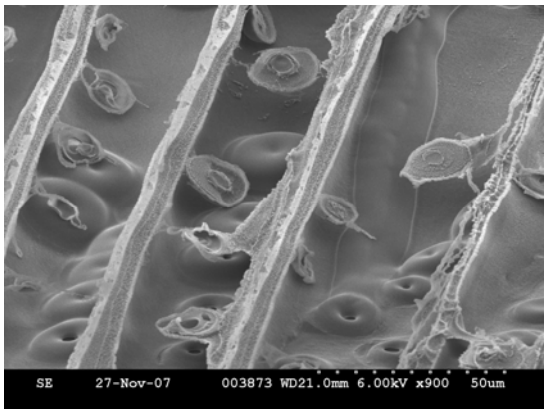

(b) Oil modified MPB wood



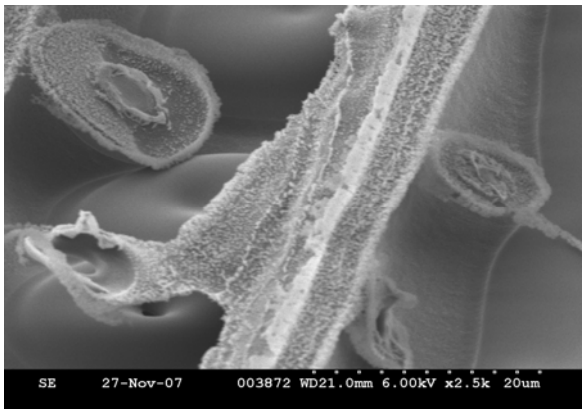

(c) Plasma treated oil modified MPB wood (50kJ)




(d) Plasma treated oil modified MPB wood (200kJ)



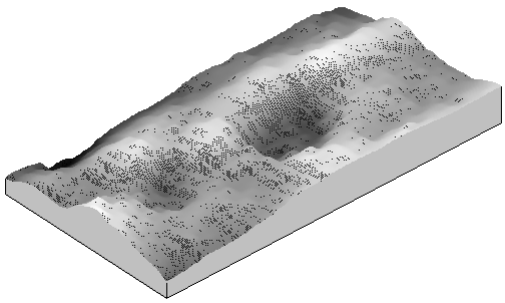

(e) Plasma treated oil modified MPB wood (200kJ)



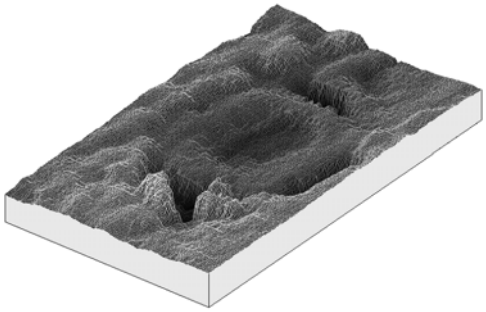

(f) Plasma treated oil modified MPB wood (200kJ)

*Figure 1. SEM photomicrographs of blue-stained wood before and after oil and plasma treatment*

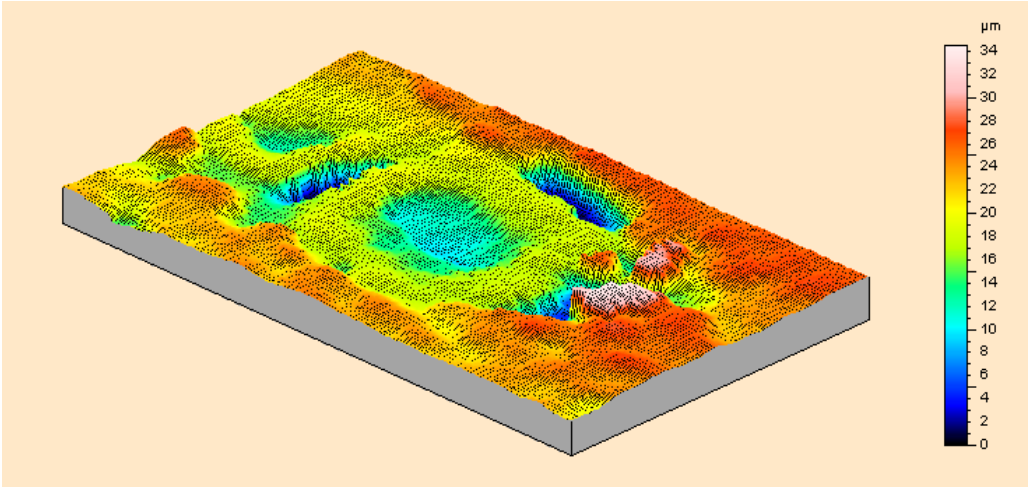
The erosion of pits by plasma was confirmed by scanning confocal profileometry. Figure 2 shows topographic images of a pit in hot-oil modified wood before (Fig. 2a) and after (Fig 2b-c) plasma treatment. The pit in wood that has not been plasma treated shows a raised border around the pit aperture, which appears as a depression in the topographic image (Fig 2a). After plasma treatment the border is etched away as well as the surrounding cell wall material. The torus and possibly the margo appear to be more resistant to etching than the surrounding cell wall material.



(a) Oil modified MPB wood



(b) Plasma treated oil modified MPB wood (200kJ)




(c) Plasma treated oil modified MPB wood (200kJ)

*Figure 2. Confocal surface topographic maps of the cell wall in hot oil modified MPB wood before and after plasma treatment*

## 3.2 Fourier transform infra-red spectroscopy

Changes in the chemical composition of plasma treated wood accord with observations of the effects of the treatments on the wood structure. FTIR spectra of plasma treated wood showed changes, suggestive of chemical modification of the wood matrix (Figure 3). The extent of such modification can be assessed

with reference to the spectra below for the unmodified (in black) and plasma modified wood (in blue). The most obvious differences between the spectrum of untreated wood and that for plasma treated wood were the weakening of peaks at wave numbers of 3350 $cm^{-1}$ (bonded OH stretching), 2925 $cm^{-1}$ (C-H stretching),1 650 $cm^{-1}$ (keto-carbonyl conjugated with benzene ring), 1261 $cm^{-1}$ (guaiacyl nuclei in lignin), 1057 $cm^{-1}$ (C-O stretching in cellulose and hemicellulose) and 660 $cm^{-1}$ (COH out-of-plane bending in cellulose).

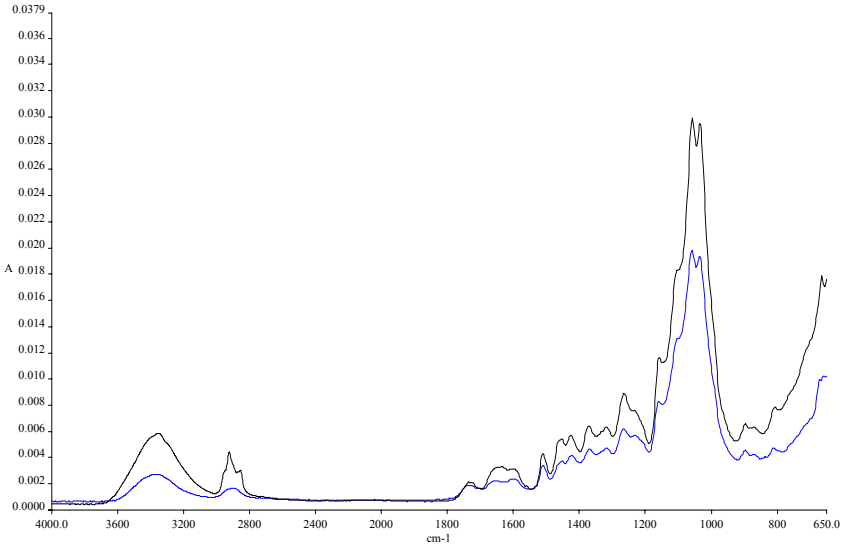



*Figure 3. FTIR spectra of MPB wood before (black) and after plasma modification (blue)*

The spectra of the samples treated with hot oil and then with plasma are shown in Figure 4. The spectrum for wood that has been modified with oil is shown in black and that for oil-treated wood that has been plasma treated is shown in blue. The oil treatment introduced two prominent peaks at wave numbers of 2800-3000 $cm^{-1}$ and 1700-1775 $cm^{-1}$ due to the presence of carbonyl and carboxylic acid groups in the oil. These two peaks are still prominent in the spectrum of wood that has been plasma treated, but there are changes in spectrum that indicate modification of the wood matrix.

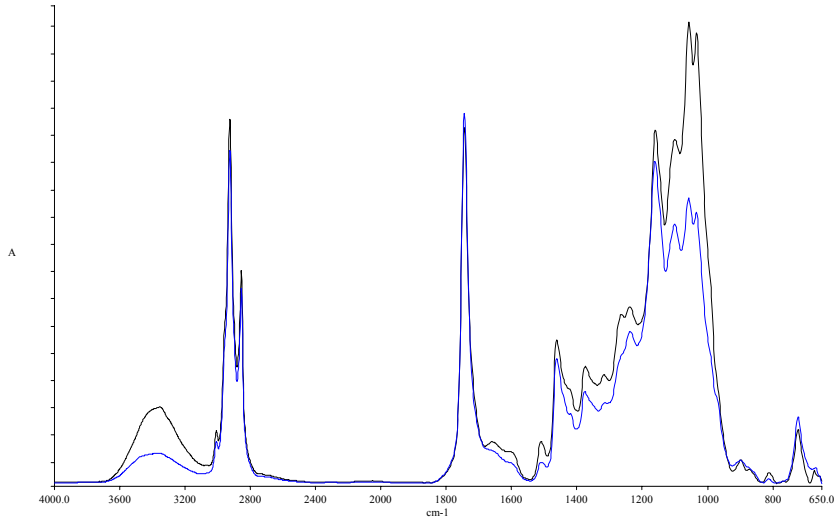



*Figure 4. FTIR spectra of oil-modified MPB wood before (black) and after plasma treatment (blue)*

### 3.3 Surface contact angle and paint adhesion and performance

Plasma treatment significantly increased the wettability of hot-oil-treated blue-stained pine. A significant difference was observed between the contact angles of water droplets applied to untreated and low energy treated samples and those that were treated with higher levels of plasma energy (Fig. 5).

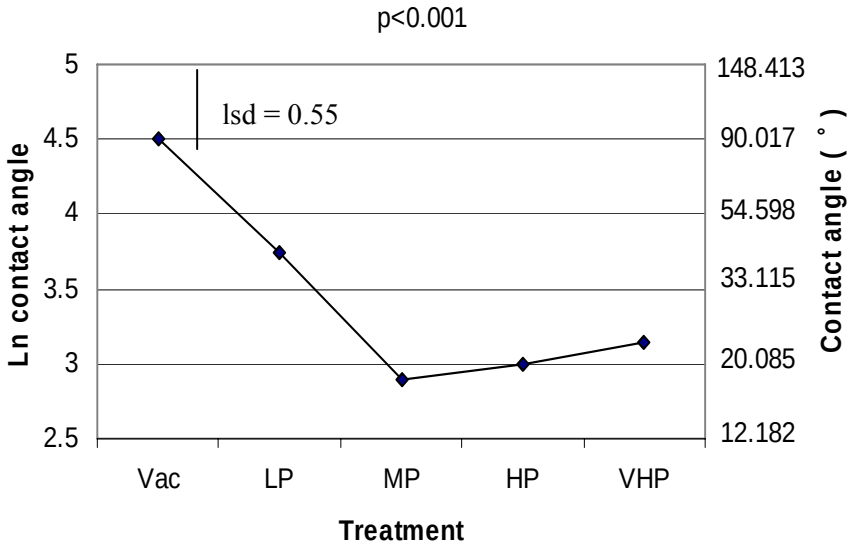



*Fig 5. The effect of plasma treatment on contact angle of hot-oil-treated blue-stained pine (Y1 axis is expressed on a logarithmic scale, Y2 axis is back transformed $e^x$ to compare results on the natural scale)*

The effect of different plasma treatment on the dry adhesion of coatings is shown in Fig.6. A significant difference was observed between the adhesion of coatings on untreated wood and wood treated with very high levels of plasma energy.

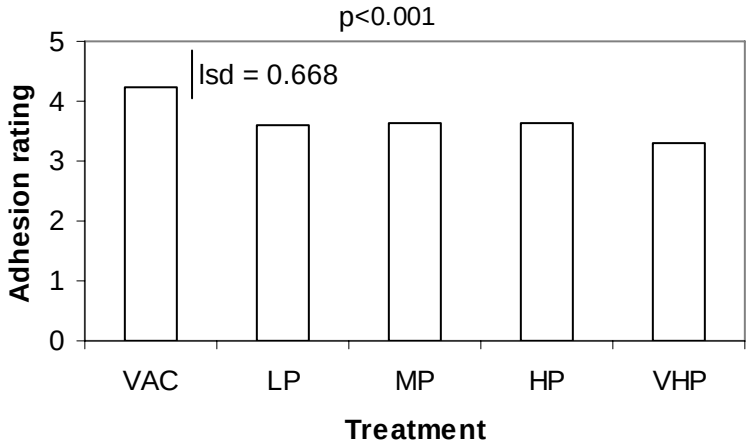



*Fig. 6. Effect of plasma treatment on dry adhesion of coatings on hot-oil-treated blue-stained pine (averaged across different coating types)*

Weathering tests are still in progress to determine if plasma treatment can enhance the performance of coatings on hot oil modified MPB wood.

Our results indicate that plasma treatment can significantly modify the structure of MPB wood and its chemical composition. The treatment removed oil from the upper surface of wood that has been thermally modified with hot oil. These changes may explain why plasma treatment increased the wettability of hot-oil modified wood and slightly improved the adhesion of finishes to modified wood. The increases in adhesion of the finishes to hot oil modified wood after plasma modification, however, were not dramatic and probably do not justify the use of the treatment prior to finishing. Furthermore, the plasma treatment used in this work required very high vacuum and long treatment times, and these aspects of the treatment would make it difficult to apply in practice. Low vacuum treatments are used, however, to improve the performance of coatings on plastics, as mentioned above, and there may be merit in investigating the use of these if weathering tests indicate that plasma treatment can improve the performance of coatings on hot-oil modified MPB wood.

## 4.0 Conclusions

Hot oil treatment of MPB wood deposits some oil within the wood. Plasma treatment is capable of removing such oil from the surface, but not the bulk of the wood. Nevertheless, plasma treatment significantly increased the wettability of the hot-oil modified wood and increased the adhesion of finishes to the wood. Plasma treatment also modified the surface structure and chemical composition of untreated and hot-oil modified wood. Scanning electron microscopy and confocal profileometry indicated that plasma was able to etch wood cell walls. Etching of cell wall material was particularly noticeable around bordered pits. FTIR spectroscopy showed that plasma was capable of etching most of woods chemical constituents. The plasma treatment used in this work required very high vacuum and long treatment times and these aspects of the treatment would make it difficult to apply in practice. Low vacuum treatments are used, however, to improve the performance of coatings on plastics, and there may be merit in investigating the use of these if weathering tests (that are in progress) indicate that plasma treatment can improve the performance of coatings on hot-oil modified MPB wood.

## 5.0 Acknowledgements

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## 6.0 References

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