THE ADHESION OF AN ACRYLIC PRIMER TO WEATHERED RADIATA PINE SURFACES

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ABSTRACT

Radiata pine (P. radiata) heartwood and sapwood were cut to produce flat-grain blocks in which the upper tangential surface consisted of either earlywood or latewood. Blocks were subjected to 6 weeks of weathering during an Australian summer and coated with an acrylic primer; and the adhesion of the primer to the different weathered and unweathered wood surfaces was assessed using a tape test. Primer adhesion decreased on earlywood as a result of weathering, but increased on latewood. Scanning electron microscopy (SEM) observations of wood/primer interfaces after tape testing suggested that the increase in primer adhesion on weathered latewood was due to greater penetration of the primer into relatively undegraded subsurface tracheids. There was little difference in the adhesion of the primer to heartwood or sapwood, but, as expected for unweathered surfaces, primer adhesion was greater on earlywood than on latewood. The practical implications of these findings for the coating of exterior wood are discussed briefly.

Keywords: Pinus radiata, weathering, acrylic primer, adhesion, earlywood, latewood, SEM.

INTRODUCTION

Paint adhesion to freshly sawn softwood surfaces is usually greater on earlywood (springwood) than on latewood (summerwood) because large earlywood cavities absorb more paint than those of latewood (Browne 1931). Lack of penetration of paint into latewood has been used to explain why painted softwood surfaces weather unevenly showing increased paint failure on latewood bands (Miller 1981). An exception to this rule of better paint adhesion to earlywood was observed by Williams et al. (1987). They applied an acrylic or alkyd primer to weathered, radial longitudinal, western red cedar (Thuja plicata D. Don) surfaces and then assessed the adhesion of the primers to the weathered wood using shear or tensile tests. Primer adhesion decreased as a result of weathering, but there was preferential failure of the alkyd primer on earlywood and greater retention of the primer on latewood (Williams et al. 1987). Similar trends for other wood species were noted subsequently (Williams et al. 1990), and it was also found that short periods of weathering increased the adhesion of an alkyd primer to three dense wood species, but had the opposite effect in two low density species. Failure of the acrylic primer on weathered earlywood and latewood was more uniform, and increases in adhesion as a result of weathering were generally not noted (Williams et al. 1987, 1990).
The aims of this research were twofold: to determine the extent to which weathering of earlywood and latewood surfaces alters the adhesion of a subsequently applied acrylic primer; second, using scanning electron microscopy of wood/primer interfaces before and after weathering, to rationalize changes (if any) in primer adhesion as a result of weathering.

MATERIALS AND METHODS

In the experiment described here, radiata pine (*Pinus radiata* D. Don) heartwood and sapwood were carefully cut to produce blocks in which one flat-grain (tangential) surface consisted of either earlywood or latewood. Blocks were subjected to 6 weeks' weathering during an Australian summer and coated with an acrylic primer. The adhesion of the primer to unweathered and weathered blocks for each surface type was then assessed using a tape test.

Preparation of wood specimens

The wood used in this study was obtained from a single 31-year-old radiata pine tree growing in Greenhill State Forest (New South Wales, Australia). The tree was felled and cross-cut to produce two logs, 500 cm in length and 45–55 cm in diameter. Heartwood on the transverse surfaces of the logs was distinguished using an *o*-anisidine spray test (Stalker 1971). Two boards containing both heartwood and sapwood were cut from each log at the heartwood-sapwood boundary using a chain saw. Boards were planed to a cross-sectional size of approximately 80 x 40 mm (tangential x radial), and air-dried under cover for 6 weeks. Heartwood and sapwood specimens with a cross-sectional size of 25 x 12 mm (tangential x radial) were cut from the boards using a bandsaw. Specimens were stored in a conditioning room at 20 ± 1°C and 65 ± 5% relative humidity for one week, re-planed to a cross-sectional size of 23 x 10 mm (tangential x radial) and cross-cut to produce specimens 23 mm in length. Heartwood and sapwood specimens in which one upper surface consisted mainly of earlywood or latewood were then selected and sanded using abrasive paper ("MIRKA 120—Jepua Finland") to obtain surfaces consisting only of earlywood or latewood. Finally, all sample blocks were sequentially hand-sanded using three types of abrasive paper of decreasing grit size (80, 180, and 360 "Norton—no-fil adalox A239—P360") to ensure that all testing surfaces were relatively smooth and uniform. A total of 80 specimens was used in the experiment, [2 (log 1 or 2) x 2 (sapwood vs. heartwood) x 2 (earlywood vs. latewood) x 2 (weathered vs. unweathered) x 5 (replicates)].

Weathering, painting, and adhesion testing

Forty specimens were exposed outdoors for 6 weeks in a randomized complete block design facing the equator (north), and at an angle of 45° to the horizontal, in Canberra, Australia, during the summer (January–February) of 1993. Information regarding the meteorological conditions encountered during the exposure trial was obtained from measurements made in Canberra by the Australian Department of Science and Technology, Bureau of Meteorology (Table 1). The weather station used to make these measurements is located 0.5 km from the exposure site. A similar number of controls was kept in a conditioning room (as above) for the duration of the exposure trial. After exposure, specimens were removed from the weathering rack, washed with distilled water, brushed using a soft brush to remove dust, stain, or dirt and then kept in a conditioning room (as above) for 4 weeks.

Table 1. Weather conditions during the 6-week exposure trial.

<table>
<thead>
<tr>
<th>Week</th>
<th>Temperature (°C)</th>
<th>Rainfall (mm)</th>
<th>Sunshine (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Max</td>
<td>Min</td>
<td></td>
</tr>
<tr>
<td>Week 1</td>
<td>30.8</td>
<td>14.8</td>
<td>5.0</td>
</tr>
<tr>
<td>Week 2</td>
<td>28.2</td>
<td>16.2</td>
<td>4.1</td>
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<tr>
<td>Week 3</td>
<td>28.4</td>
<td>14.7</td>
<td>10.8</td>
</tr>
<tr>
<td>Week 4</td>
<td>30.6</td>
<td>15.4</td>
<td>0.2</td>
</tr>
<tr>
<td>Week 5</td>
<td>25.4</td>
<td>14.7</td>
<td>10.2</td>
</tr>
<tr>
<td>Week 6</td>
<td>23.5</td>
<td>11.8</td>
<td>22.0</td>
</tr>
</tbody>
</table>

All figures are means except rainfall, which is recorded as a total.
prior to painting. In order to assess the amount of primer applied to each wood block, all specimens were weighed after conditioning. One coat of a white exterior acrylic primer (ICI Dulux Weathershield, Dulux Australia, McNaughton Road Clayton, Vic. 3168, Australia) was brushed onto the weathered surfaces of exposed specimens and to the prepared upper surface of unexposed controls in accord with paint manufacturers' instructions. After curing for 3 weeks in a conditioning room, painted specimens were reweighed and a tape test (ASTM 1976) was used to assess the adhesion of the acrylic primer to weathered and unweathered blocks. Painted specimens were held firmly in a jig and then six cross-cuts, 1 mm apart, were made manually on the painted surface using a scalpel and a steel ruler. Detached flakes or ribbons of primer were removed from painted surfaces with a soft brush. A strip of fiber-reinforced cellulose acetate pressure-sensitive tape (Sellotape “Stylus 801 Filament”), 20 mm wide and 60 mm long, was then placed over the cut surface of blocks. The tape was manually smoothed, and pressure was applied using a rubber eraser to ensure good contact between the tape and the paint film. After 1 day the tape was removed from the painted surface by rapidly pulling it off, back upon itself, at an angle as close to 180° as possible (ASTM 1976). Quantification of the area of paint removed from specimens was carried out using image analysis. Images of painted and tested wood surfaces were obtained using a color CCD TV camera (Panasonic WV-CL 350) and recorded onto video tape. Images were then fed into a personal computer (Macintosh II) and converted to a digital form using a digitizing program (Quick Image 24 Mass Microsystems, California). Wood beneath areas of paint that were detached from specimens during tape testing appeared darker than painted surfaces and this enabled the area of paint removed from each specimen to be quantified using an image analysis program (Image, National Institute of Health, USA). The total area delimited by the cross-hatch was 36 mm², and the area of paint removed from this area, and not outside it, was measured for each specimen.

The appropriate analysis of variance (ANOVA) on the area of primer removed after tape testing was carried out to assess the effect of wood source (log 1 or 2), wood type (heartwood or sapwood), wood surface type (earlywood or latewood), and weathering on primer adhesion. In order for data to conform to the underlying assumptions of ANOVA, i.e., normality with constant variance, they were converted to natural logarithms prior to analysis. The areas of primer removed after tape testing are plotted graphically in Fig. 1 as natural logarithms. For comparison of the effect of wood surface type (earlywood or latewood) and weathering on primer adhesion, a bar representing the least significant difference (p < 0.05) is presented in Fig. 1.

Scanning electron microscopy

After tape testing, representative sample blocks were clamped in a small vise, with the transverse face uppermost, and viewed under a stereomicroscope at low magnification (× 10–20). A hand-held, single-edged razor blade (Magnuson Injector type) was then used to cut a clean face at the interface between the primer and earlywood or latewood. A match-stick-sized sample containing the cut surface was split from the sample block and dried at atmospheric pressure over silica gel for 12 h. Dried samples were attached to aluminium stubs using nylon finger-nail varnish and sputter-coated with a 10-nm layer of gold. A Cambridge Instruments S360 scanning electron microscope fitted with a high-brightness lanthanum hexaboride (La₁₂) electron source was used to examine the paint/wood interface. Images obtained from back-scattered electrons provided more detail of the penetration, and interaction of the primer with the wood substrate and therefore are reproduced in prefer-

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2 Trade names are for identification purposes only and do not imply endorsement or otherwise of any products mentioned in the text.
ence to secondary electron images. There was little difference in the interaction of the primer with heartwood or sapwood surfaces, but images of paint/wood interfaces from sapwood specimens were clearer and therefore are reproduced in preference to those from heartwood specimens. All images were photographically recorded on Kodak PXP 220 film.

RESULTS AND DISCUSSION

Analysis of variance indicated a significant effect ($p < 0.001$) of weathering on primer adhesion, but there was no significant effect ($p > 0.05$) of wood source (wood blocks obtained from logs 1 or 2) on adhesion. The adhesion of the acrylic primer to unweathered and weathered surfaces was assessed using a tape test in which the area of finish removed by the tape gives a relative guide to the adhesion of the primer to the underlying wood (ASTM 1976). Figure 1 shows the areas (expressed as natural logarithms) of acrylic primer removed during tape testing of weathered and unweathered radiata pine earlywood and latewood for both heartwood and sapwood. As there was no significant effect of wood source on primer adhesion, results at this level were aggregated, and the points in Fig. 1 therefore represent means obtained by testing 10 specimens ($5$ replicates $\times 2$ [log 1 and 2]). Overall there was no significant effect ($p > 0.05$) of wood type (heartwood or sapwood) on primer adhesion, but there was a significant interactive effect ($p < 0.01$) of wood type and surface type on adhesion. This occurred because primer adhesion was lower on earlywood in heartwood than in sapwood whereas for latewood, primer adhesion was higher in heartwood compared to sapwood (Fig. 1). The areas of primer removed from unweathered earlywood were smaller than those removed from unweathered latewood, indicating, as others have noted (Browne 1931; Miller 1981), greater adhesion of the finish on earlywood.

After weathering, the adhesion of the finish decreased significantly on earlywood and again this accords with previous studies (Ash- ton 1967; Desai 1967; Boxall 1977; Underhaug et al. 1983; Williams et al. 1987, 1990; Williams and Feist 1993; Evans et al. 1996) which have shown that paints and other finishes applied to weathered wood surfaces show reduced adhesion. However, the adhesion of the primer on weathered latewood was greater than on unweathered latewood (Fig. 1). Increased adhesion on weathered latewood was observed for both heartwood and sapwood specimens, but increases were more pronounced for the former where adhesion on weathered latewood exceeded that on unweathered earlywood (Fig. 1). Weathering increased the absorption of primer on both earlywood and latewood (Table 2), and therefore differences in the absorption of paint between surface types probably cannot be used to explain why weathering increased adhesion on latewood, but had the opposite effect for earlywood. Some indication of why adhesion increased on latewood after weathering can be obtained by examination of the interfaces be-
between the acrylic primer and the weathered and unweathered latewood. On unweathered latewood there was intimate contact between the acrylic primer and the wood, and generally the primer did not penetrate greatly beyond the wood surface (Fig. 2a). In contrast, on weathered latewood there was greater penetration of the primer into subsurface tracheids (Fig. 2b, c). There was some evidence of penetration of the primer into the wood cell wall, via weathering checks (Fig. 2d). Deeper penetration of the primer into weathered wood also occurred via degraded rays (Fig. 2b, c). Greater penetration of the primer into weathered latewood could have increased the adhesion of the primer by allowing the formation of mechanical bonds with subsurface latewood tracheids. Weathering slightly increased the penetration of the primer into earlywood (compare Fig. 2e with 2f), but cell walls in weathered earlywood were thin and lacked integrity (Fig. 2f). Hence any positive effect of increased primer penetration in earlywood on primer adhesion was probably offset by more severe weathering degradation of earlywood tracheids.

As mentioned above, Williams et al. (1987, 1990) in studies of the adhesion of primers to weathered radial longitudinal surfaces noted preferential failure of an alkyd primer on weathered earlywood compared to latewood. It was suggested that such a reversal of the normal trend (for freshly cut wood) of better adhesion of paints to earlywood could have occurred as a result of two effects. First, earlywood is more rapidly degraded during weathering than latewood and this would tend to reduce adhesion to a greater extent in earlywood. Second, increased penetration of the alkyd primer into weathered latewood might allow for increased mechanical bonding to subsurface tracheids, which would be less degraded as a result of weathering. Derbyshire et al. (1996) suggested that the findings of Williams et al. (1990) lent support to their concept that short periods of weathering might increase the surface strength properties of wood. While the possibility that strength increases due to weathering might have played a part in increasing the adhesion of the acrylic primer to latewood cannot be discounted, it seems more likely from scanning electron microscopy that increases in adhesion occurred due to greater penetration of the primer into weathered latewood, resulting in the formation of mechanical bonds with subsurface latewood tracheids which, in contrast to earlywood, were less degraded by weathering. Our observations therefore, support the aforementioned suggestions of Williams et al. (1990).

This study has demonstrated that wood surface structure and weathering interact strongly to influence the adhesion of an acrylic primer to wood surfaces. Degradation of earlywood surfaces during weathering clearly reduced primer adhesion, but the results for radiata pine latewood, in which primer adhesion increased as a result of weathering, suggest that if finishes can penetrate beyond the weathered surface layers to bond to relatively undegraded subsurface tracheids, then the deleterious effects of weathering on the adhesion and performance of finishes may be ameliorated. It is well known that because of their greater ability to penetrate into wood, alkyd primers adhere to and perform better on weathered wood than acrylic primers. However, it may be possible to formulate acrylic primers which, while retaining such beneficial characteristics as water emulsifiability and ease of painting, are more compatible with weathered wood surfaces. Further work is needed to develop such primers, particularly since wood is often unavoidably exposed to the weather prior to priming.

The results of this study suggest that the adhesion of acrylic finishes on flat-sawn surfaces could be improved by increasing the penetration of the finish into the latewood. Emulsified resin particles in acrylic paints are usually spherical with a diameter of 1–3 μm (Jewell 1993) and are therefore too large to penetrate cell-wall capillaries in wood which may be up to 45 nm in diameter (Rudman 1965). However, they are able to penetrate cell lumens, microscopic checks, and other larger pathways within the wood structure. Wood modification techniques, for example roller
Fig. 2. Back-scattered SEM images of primer/wood interfaces; a. unweathered latewood showing little penetration of the primer beyond the wood surface; b. weathered latewood showing penetration of the primer beyond the wood surface; c. weathered latewood showing penetration of the primer beyond the wood surface via rays (arrowed) and tracheid lumens; d. split-screen image of weathered latewood showing penetration of the primer into tracheid walls (arrowed) via weathering checks (right-hand image = ×4); e. unweathered earlywood showing some penetration of the primer beyond the wood surface; f. weathered earlywood showing some penetration of the primer beyond the wood surface; note degraded cell walls (arrowed).
compression treatments (Cech and Huffman 1970) and steaming or enzyme treatments (Siau 1984), can facilitate the development or enlargement of such microscopic voids and recently it has been shown that a bacterial pre-treatment increased the penetration of an alkyd finish into radiata pine (Singh et al. 1996). Such techniques may increase the penetration of acrylic finishes into wood, and latewood in particular, with benefits for improved coating adhesion and performance; but further research is required to confirm this.

CONCLUSIONS

Short periods of weathering of radiata pine wood may increase or decrease the adhesion of a subsequently applied acrylic primer. On latewood the development of surface weathering checks in tracheids, rays, and wood cell walls increases the penetration of the primer into the wood, which has beneficial effects on adhesion. Weathering does not greatly increase the penetration of primer into earlywood; and because earlywood tracheids are more severely degraded as a result of weathering, primer adhesion decreases. Normally in the case of unweathered wood, paint adhesion is greater on earlywood than on latewood; but the effect of short periods of weathering (before application of the finish) is to reverse this trend resulting in better adhesion of the finish on latewood compared to earlywood.

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REFERENCES