strength and durability of one-part polyurethane ADHESIVE BONDS to wood

ABSTRACT

One-part polyurethane wood adhesives comprise a new class of general purpose consumer products. Manufacturers' claims of waterproof bonds brought many inquiries to the Forest Products Laboratory (FPL) from users constructing aircraft, boats, lawn furniture, and other laminated materials for outdoor use. Although FPL has technical information on several types of polyurethane and isocyanate-based adhesives for wood, no information was available on this new class of adhesives. Four commercial polyurethane adhesives, along with a resorcinol-formaldehyde adhesive to represent a standard of performance, were subjected to a series of industry-accepted tests that assess varying levels of bond strength and durability. In bonds to yellow birch and Douglas-fir, the polyurethanes did not differ significantly from each other in their performance; as a group, though, their dry shear strengths showed that they were significantly stronger than the resorcinol. Dry wood failures by the polyurethanes were high and did not differ significantly from the resorcinol. After three water-saturating procedures, wet shear strengths of polyurethanes and the resorcinol were statistically comparable. Wet wood failures, however, were very low among polyurethanes, which is a sharp contrast to the high wood failure by the resorcinol. A moderately severe delamination test indicated varying levels of water resistance among the polyurethanes, but the resorcinol was completely resistant to delamination. A very severe cyclic delamination test caused severe delamination of polyurethane bonds. However, a recently discovered hydroxy(methylated) resorcinol (HMR) coupling agent dramatically increased delamination resistance of polyurethane adhesives. In a test of resistance to deformation under static loads, polyurethane bonds withstood extreme exposures of temperature and relative humidity for 60 days without deformation.

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One-part polyurethane wood adhesives are a recent addition to the general purpose consumer market. They join a broad array of polyurethane adhesive products that have wide commercial application in textile, foundry, packaging, forest products, automotive, footwear, furniture, and recreational vehicle markets. Polyurethanes are well known for their excellent adhesion, flexibility, high cohesive strength, low-temperature performance, and amenable curing speeds. Because they effectively wet and readily form hydrogen bonds with most substrates, they develop excellent bonds to textile fibers, metals, plastics, wood, glass, sand, ceramics, rubber, and leather.

One-part polyurethanes are based on urethane prepolymers made by reacting an excess of methylene diphenyl diisocyanate (MDI) with a polyol such that a small amount of isocyanate monomere remains. Free isocyanate groups in the adhesive then react with moisture on substrate surfaces to complete the cure. The reaction of isocyanate with water proceeds through intermediate steps to form urea linkages with evolution of carbon dioxide. Carbon dioxide causes the adhesive to foam and expand and, if not properly controlled, entrapped gas bubbles will weaken the cohesive strength of adhesive film. Products within this new class of wood adhesives are marketed as single-component, ready-to-use liquids. They contain essentially 100 percent solids that react and cure on contact with moisture in the air and on the wood substrates.

Since these new wood adhesives were introduced, the Forest Products Laboratory (FPL) has received many inquiries concerning the strength and water resistance of polyurethane bonds to wood. These inquiries usually come from users constructing aircraft boats, outdoor furniture, and other laminated wood assemblies for outdoor exposure. The FPL has technical information on several types of polyurethane and isocyanate-based adhesives for wood. These include MDI binders for composites, emulsion-polymer/isocyanate laminating adhesives, moisture-curing polyurethane construction adhesives, experimental MDI laminating adhesives, and moisture-curing hot-melt adhesives. However, none of these data could be used to credibly predict performance of this new class of polyurethane adhesives on wood, particularly in laminates exposed to wet-use conditions.

The purpose of this study was to determine the strength and durability of representative commercial polyurethane wood adhesives in bonds to hardwood and softwood, according to a battery of standardized testing procedures. These procedures were selected to assess varying levels of bond durability. A resorcinol-formaldehyde adhesive, which is one of the most durable of structural adhesives for wood, was included in these tests to provide a standard of performance for comparison with the polyurethane adhesives.
hesives. Although not included in the original plans of this study, preliminary tests indicated that a recently discovered hydroxy-methylated resorcinol (HMR) coupling agent (9) effectively increases resistance to delamination of polyurethane adhesives in hardwood and softwood lumber laminates. The adhesion-enhancing capabilities of the HMR coupling agent for polyurethanes will be evaluated in a subsequent study and report.

EXPERIMENTAL MATERIALS

Adhesives

Four commercial one-part polyurethane adhesives were obtained directly from U.S. suppliers. Product names have not been disclosed here, nor to any inquirers, as was agreed to by suppliers and authors. All four adhesives were applied and cured in general accordance with the manufacturer’s instructions, unless noted otherwise in the procedures of this report.

A resorcinol-formaldehyde adhesive, supplied by Indspec Chemical Corporation, Pittsburgh, PA and identified as Penacolite G-1131-A resin and G-1131-B hardener, was used as a standard of performance for comparison with polyurethane adhesives.

Woods

Yellow birch and Douglas-fir are representative of moderately high density hardwood and softwood species. The wood from both species was heartwood, straight grained, and free of defects. Orientation of annual rings with respect to the adhesive interface varied between flatsawn and quartersawn. The average and range of annual rings per inch were 24 (14 to 50) for yellow birch and 19 (8 to 42) for Douglas-fir, as determined by sampling 30 pieces of wood.

The wood was conditioned at 22.8°C (73°F) and 50 percent relative humidity (RH) to an equilibrium moisture content (EMC) of approximately 9.5 percent for both species. The bonding surfaces were knife-planed 24 hours before bonding. Laminae for preparation of shear specimens were planed to 8 mm (5/16 in.) thick, and boards for delamination specimens were planed to 19 mm (3/4 in./thick, from a small number of specimens could not provide numerical descriptions of properties that were as meaningful as simple means, ranges and totals, against requirements.

Specimen

Shear strength and wood failure were measured from small compression-loaded block-shear specimens with 6.45-cm² (1.0 in.²) shear areas (8) (Fig. 1). Specimens were cut from two-ply laminates bonded with a test adhesive. Grain direction was parallel in both laminae and was parallel to the shearing stress in the specimens. Individual laminae were 8 mm (5/16 in.) thick, 127 mm (5 in.) wide, and 305 mm (12 in.) long. Twenty-five specimens were tested for each treatment combination.

Compression-loaded block-shear specimens were substituted for tension-loaded lap-shear specimens. The latter are specified in the two plywood standards (2,6). Stress analysis has shown that tension perpendicular to the bondline, not shear, is the primary stress mode causing failure in lap-shear specimens (8). This tensile stress is caused by eccentricities in the direction of pull as the specimen, particularly a wet one, rotates according to the notching pattern and stiffness of the wood adherends. The presence of the tensile stress produces failure loads that are lower in the lap-shear specimens than in the block-shear specimens. Also, block-shear specimens of two-ply and parallel-grain constructions produce much higher failure loads than those of the prescribed three-ply and cross-grain constructions of lap-shear specimens. Strength and wood failure values tend to be more variable in lap-shear specimens than in the comparably sized block-shear specimens.

A delamination specimen with 76 mm (3 in.) sides was cut from the same two-ply laminate that was used for the block-shear specimens. Delamination was measured along the bondlines on all four sides after treatment. Five specimens were tested for each treatment combination.

Deformation under static loading was measured from a block-shear specimen of the same size and configuration as shown in Figure 1. These specimens were cut from two-ply laminae of yellow birch and bonded with only one of the polyurethane adhe-
sives. Individual laminae were 8 mm (5/16 in.) thick, 64 mm (2-1/2 in.) wide, and 305 mm (12 in.) long. Two specimens were tested for each treatment combination.

Delamination of lumber laminates was measured from bondlines on both end-grain surfaces of 76 mm (3 in.) long cross sections cut from six-ply lumber laminates. Laminates were prepared by bonding six pieces of either yellow birch or Douglas-fir with a polyurethane adhesive. Individual laminae were 19 mm (3/4 in.) thick, 76 mm (3 in.) wide and 305 mm (12 in.) long. Twelve specimens, or 914 cm (360 in.) of bondline length, were measured for delamination.

**Specimen Preparation**

All two-ply and six-ply laminations bonded with polyurethane adhesives were prepared by the same procedures. Planed and cut-to-size laminae were sprayed with a fine mist of water under air pressure. Approximately one minute after misting, a weighed amount of adhesive was spread on each surface with a roller at a spread rate of 0.098 kg/m² (0.020 pcf). Both adhesive-spread surfaces were immediately assembled and rubbed together to ensure contact between surfaces. Pressure was applied after approximately 15 minutes closed assembly time at 69 N/cm² (100 pcf) to Douglas-fir laminates and 103 N/cm² (150 pcf) to yellow birch. Constant pressure was maintained for 24 hours at room temperature. After pressing, laminates were reconditioned at 22.8°C (73°F) and 50 percent RH for 7 days before cutting into specimens.

The two-ply laminates bonded with resorcinol adhesives were roller-spread on each surface at a spread rate of 0.146 kg/m² (0.030 pcf). After approximately 5 minutes open assembly time and 20 minutes closed assembly time, pressure was applied and adhesive was cured as previously described.

**Specimen Testing**

Twenty-five randomly selected block-shear specimens representing each adhesive and wood species combination (250 specimens total) were tested for shear strength and wood failure in the dry condition (9.5% EMC), as described in ASTM D 905-94 (3). The rate of loading was 5 mm/minute (0.2 in./min). Load at failure was measured in newtons per square centimeter (pounds per square inch) of shear area, and wood failure was estimated to the nearest 5 percent of shear area.

A second group of 25 specimens representative of each treatment combination was subjected to a vacuum-atmospheric pressure water soak (VAS) (2). The specimens were submerged in water at 49°C (120°F) while a vacuum of 5.06 N/cm² (15 in. Hg) was maintained for 30 minutes. Water soaking continued for 15 hours at atmospheric pressure. While wet, the specimens were loaded to failure as previously described.

A third representative set of 25 block-shear specimens from each treatment combination were subjected to a vacuum-pressure water soak (VPS) (2). The specimens were submerged in cold tap water while a vacuum of 8.4 N/cm² (25 in. Hg) was maintained for 30 minutes, followed immediately by pressure at 41.4 N/cm² (60 psi) for 30 minutes. While wet, the specimens were loaded to failure as previously described.

A fourth group of representative block-shear specimens were subjected to a boil-dry-boil test (BDB) (2). The specimens
were boiled in water for 4 hours, then dried for 20 hours at 63°C (145°F). Then they were boiled for another 4 hours, cooled in tap water, and tested to failure while wet.

Five two-ply delamination specimens representing each adhesive-wood species combination were subjected to a two-cycle boil test (6). Specimens were submerged in boiling water for 4 hours, then dried for 20 hours at 63°C (145°F). They were boiled again for 4 hours, dried for 3 hours, and measured for delamination. Any delamination greater than 25.4 mm (1 in.) in continuous length was considered failure of the specimen. Total delamination was also measured.

Twelve six-ply delamination specimens representing one of two polyurethane adhesives on each of the two wood species were subjected to a severe, three-cycle delamination test (4). In the first cycle, specimens were vacuum-soaked in water at 8.4 N/cm² (25 in. Hg) for 5 minutes, then pressure-soaked at 41.4 N/cm² (60 psi) for one hour. These events were repeated. Drying at 65.5°C (150°F) for 21 hours completed the first cycle. In the second cycle, specimens were steamed for 1.5 hours, followed by pressure-soaking in water at 82.7 N/cm² (120 psi) and drying at 65.5°C (150°F) for 21 hours. The third cycle was a repeat of the first cycle. Immediately after the final cycle, delamination was measured along the bondlines on all end-grain surfaces. Delamination was expressed as a percentage of total bondline length on the end-grain surfaces.

Ten randomly selected block-shear specimens (Fig. 1) made from two-ply laminates of yellow birch and bonded with Polyurethane Adhesive D were statically loaded and exposed to two separate extremes of prolonged service environments. Two specimens were loaded at each of the following static loads: 41.4 N/cm² (60 psi), 82.7 N/cm² (120 psi), 124.1 N/cm² (180 psi), 165.5 N/cm² (240 psi), and 206.9 N/cm² (300 psi), and then exposed to 71°C (160°F) for 60 days. The 165.5 N/cm² (240 psi) static load level and the constant high temperature and high humidity environments were the same as specified in ASTM D 2559-92 (4), however, the single block-shear specimens and static loading apparatus of ASTM D 4680-92 (5) were substituted for the multiple-joint shear specimens and loading apparatus of the ASTM D 2559-92 (4) test. Furthermore, the static loading period was extended from the required 7 days to 60 days in this test.

RESULTS AND DISCUSSION

Shear Strength and Wood Failure in the Dry Test

The four polyurethane adhesives developed dry shear strengths on yellow birch and Douglas-fir woods that were significantly stronger (statistical data not shown) than the resorcinol adhesive (Fig. 2). No significant differences in shear strength were detected between the four polyurethanes. Strengths of all adhesive bonds to yellow birch were almost double those to Douglas-fir, primarily because the wood of yellow birch is of higher density and it is stronger in shear parallel to the grain. All adhesives had dry shear strengths that exceeded by far the requirements for structural bonds on both species of wood, as specified in ASTM D 2559-92 (4). Again, the

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shear strength and wood failure values are not necessarily equivalent.

Typically, wood failure of a high-density wood such as yellow birch is lower than that of a low-density wood such as Douglas-fir, even though the full parallel-to-grain shear strength of both woods has been reached. These data reflect this effect by showing that wood failure was significantly lower for yellow birch than Douglas-fir. However, within either species group, there were no significant differences in wood failure between any of the adhesives. With the exception of borderline wood failures of Adhesives B and C for yellow birch, all adhesives met the minimum 75 percent wood failure requirement of ASTM D 2559-92 (4).

Mean dry shear strength and wood failure values of all adhesives for both wood species are shown in Table 1. Since strength and wood failure values among the polyurethanes were not significantly different, all polyurethane values were averaged and compared with those of resorcinol for yellow birch and Douglas-fir, as shown in Figures 2 and 3, respectively.

**Shear Strength and Wood Failure in the Wet Tests**

In VAS, VPS, and BDB tests of wet shear strength the polyurethane bonds were essentially as strong as the resorcinol bonds on both wood species (Table 1, Figure 2). Statistical comparisons showed no significant differences between strengths of any adhesives in the wet tests, although the resorcinol bonds retained 10 to 15 percent more of dry shear strength than did the polyurethane bonds. In VAS and VPS tests, individual polyurethanes on yellow birch retained 36 to 38 percent of their dry shear strengths, and in the BDB test, they retained between 30 and 34 percent of dry strength. In these same tests resorcinol bonds retained 47, 47 and 42 percent of dry strength, respectively. Greater percentages of dry strength were retained by polyurethane bonds on Douglas-fir: 51 to 53 percent in VAS tests; 53 to 56 percent in VPS tests; and 31 to 41 percent in BDB tests. Resorcinol bonds retained about 15 percent more of their dry shear strength on Douglas-fir than did the polyurethanes.

Wood failure percentages in the wet tests were uniformly alike — all polyurethane adhesives produced very low levels of wood failure while the resorcinol adhesive produced very high levels (Table 1, Figs 2 and 3). Wood failures of resorcinol bonds were significantly higher than those of polyurethane bonds (generally more than 91%) except for the 76 percent wood failure requirement in the BDB test. Essentially, the resorcinol bonds were as durable as the wood itself, as indicated by the high level of wood failure. The polyurethane bonds were much less durable than the resorcinol bonds on both wood species.

AU polyurethane adhesives developed wet wood failure percentages on Douglas-fir that were above the minimum 15 percent required for Technical and Type 1 plywood, as specified in ANSI/HPVA HP-1-1994 (6). On yellow birch, however, only Polyurethane D exceeded the requirement while the other polyurethanes were marginally lower. None of the polyurethanes on either wood species could meet the minimum 80 and 85 percent wet wood failure requirements required of softwood plywood grades in the VPS and BDB tests of U.S. product Standard PS 1-83 (2). Neither could they consistently meet the 45 percent wood failure requirement in the VAS tests.

**Delamination in the Two-Cycle boil Test**

Delamination values of individual polyurethane- and resorcinol-bonded specimens during the two-cycle boil test are shown in Table 1. Adhesives are compared with each other and with the requirements of ANSI/HPVA HP-1-1994 (6). The criteria of failure within a specimen is that delamination must not exceed 25.4 mm (1 in.) of continuous length and that 90 percent of the specimens tested must meet the continuous-length requirement.

No delamination occurred within any of the resorcinol-bonded specimens. How-

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**Table 1 — Delamination after a two-cycle boil test of yellow birch and Douglas-fir laminates bonded with polyurethane (A, B, C, and D) and resorcinol (RF) adhesives.**

<table>
<thead>
<tr>
<th>Delamination</th>
<th>Specimen 1</th>
<th>Specimen 2</th>
<th>Specimen 3</th>
<th>Specimen 4</th>
<th>Specimen 5</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(mm) (in.)</td>
<td>(mm) (in.)</td>
<td>(mm) (in.)</td>
<td>(mm) (in.)</td>
<td>(mm) (in.)</td>
<td></td>
</tr>
<tr>
<td><strong>Yellow birch</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>0.0 (0.00)</td>
<td>22.4 (0.88)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>22.4 (0.88)</td>
</tr>
<tr>
<td>B</td>
<td>47.8 (1.88)</td>
<td>2.8 (0.11)</td>
<td>20.8 (0.82)</td>
<td>54.1 (2.13)</td>
<td>28.2 (1.11)</td>
<td>153.7 (6.05)</td>
</tr>
<tr>
<td>C</td>
<td>31.8 (1.25)</td>
<td>11.7 (0.46)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>19.3 (0.76)</td>
<td>62.8 (2.47)</td>
</tr>
<tr>
<td>D</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>11.9 (0.47)</td>
<td>5.3 (0.21)</td>
<td>0.0 (0.00)</td>
<td>17.2 (0.68)</td>
</tr>
<tr>
<td>RF</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
</tr>
<tr>
<td><strong>Douglas-fir</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>14.0 (0.55)</td>
<td>10.9 (0.43)</td>
<td>0.0 (0.00)</td>
<td>5.1 (0.20)</td>
<td>0.0 (0.00)</td>
<td>30.0 (1.18)</td>
</tr>
<tr>
<td>B</td>
<td>124.5 (4.90)*</td>
<td>66.0 (2.60)*</td>
<td>58.9 (2.32)</td>
<td>138.9 (5.47)*</td>
<td>110.2 (4.34)*</td>
<td>498.5 (19.63)</td>
</tr>
<tr>
<td>C</td>
<td>6.4 (0.25)</td>
<td>11.4 (0.45)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>29.2 (1.15)*</td>
<td>47.0 (1.85)</td>
</tr>
<tr>
<td>D</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>15.2 (0.60)</td>
<td>0.0 (0.00)</td>
<td>0.0 (0.00)</td>
<td>15.2 (0.60)</td>
</tr>
<tr>
<td>RF</td>
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</table>

*Specimens with more than 25.4 mm (1 in.) of continuous delamination failed the test. Ninety percent of the specimens must pass.
ever, delamination within the polyurethane-bonded specimens varied considerably, from complete failure of the test by two polyurethanes to minor delamination by others. Polyurethane Adhesives A and D met delamination requirements on both yellow birch and Douglas-fir. Adhesive C passed on yellow birch but failed on Douglas-fir. Adhesive B did not meet requirements on either wood, but its performance was much better on yellow birch where only one in five specimens delaminated more than 25.4 mm (1 in.) continuously. Four of five specimens failed the delamination test on Douglas-fir.

Delamination in the Cyclic Test

Selected polyurethane adhesives in six-ply lumber laminates of yellow birch and Douglas-fir exhibited low resistances to delamination during the severe cyclic delamination test of ASTM D 2559-92 (4). This test is specified for qualifying adhesives in structural laminated wood products for use under exterior exposure conditions (1). To qualify, adhesives such as resorcinol must not delamate more than 8 percent on hardwoods and 5 percent on softwoods. Polyurethane Adhesive D delaminated 53.5 percent on yellow birch, and Adhesive C delaminated 71.1 percent on Douglas-fir.

Although not included in this report, a recently developed HMR coupling agent (9) produced dramatic increases in delamination resistance during this test by both polyurethane adhesives on both species of wood. After wood surfaces were primed with the coupling agent, Polyurethane Adhesive D delaminated only 5.8 percent on yellow birch, and Adhesive C delaminated 2.1 percent on Douglas-fir. Both polyurethanes on HMR-primed wood met the delamination requirements of ASTM D 2559-92 (4).

Resistance to Deformation Under Static Load

Block-shear specimens of yellow birch laminated with Polyurethane Adhesive D were statically loaded at 41.4 N/cm² (60 psi) intervals up to 206.9 N/cm² (300 psi).

They were exposed to two extreme environments of 71°C (160°F) with no RH control and to 90 percent RH at 26.7°C (80°F). After 60 days of loading, none of the polyurethane bonds deformed, even at the highest load. These polyurethane bonds clearly exceeded the deformation requirements of ASTM D 2559-92 (4), which requires static loading at 165.5 N/cm² (240 psi) at these conditions for only 7 days.

CONCLUSIONS

The dry shear strength and wood failure values indicated that the one-part polyurethane adhesive bonds were at least as strong as bonds of a resorcinol-formaldehyde structural adhesive on yellow birch and Douglas-fir. Wet shear strengths measured after three different water-saturating test procedures indicated that polyurethane bonds were as strong as those of resorcinol. However, measurements of wood failure indicated that polyurethane bonds were not equivalent, and they developed very low levels of wood failure relative to those of the highly durable resorcinol adhesive. A moderately severe two-cycle boil test indicated polyurethane bonds varied from high to low resistance to delamination, while the resorcinol bonds were completely resistant to delamination. A very severe cyclic delamination test that qualifies adhesives for structural laminated wood products for exterior exposure caused severe delamination of polyurethane bonds in lumber laminates of yellow birch and Douglas-fir. In a test of resistance to deformation under static loads, polyurethane bonds to yellow birch withstood extremes of temperature and relative humidity for 60 days without deformation, thereby exceeding structural requirements of ASTM D 2559-92 (4). Limited testing indicated that a recently developed HMR coupling agent dramatically increases delamination resistance of polyurethane adhesives when the wood surfaces are primed with the coupling agent before bonding.