DURABILITY OF ONE-PART POLYURETHANE BONDS TO WOOD IMPROVED BY HMR COUPLING AGENT

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ABSTRACT

In a previous study on the strength and durability of a new class of wood adhesives called one-part polyurethanes, four commercial one-part polyurethanes, along with a resorcinol-formaldehyde adhesive representing a standard of performance, were compared in bonds to yellow birch and Douglas-fir in a series of industry-accepted tests (7). The polyurethanes all performed essentially the same: high in dry and wet strength but low in wet wood failure and resistance to delamination. In the study described in this paper, we look at improving the durability of polyurethane bonds to wood by priming before bonding with a hydroxymethylated resorcinol (HMR) coupling agent that is known to enhance adhesion of all thermosetting wood adhesives. The HMR dramatically increased the wet wood failure and resistance to delamination of polyurethanes that were found deficient in the previous study, to levels comparable with the highly durable resorcinol adhesive. One-part polyurethane adhesives met the strength and durability requirements of the most rigorous of tests, specifically American Society for Testing and Materials (ASTM) standard D 2559 (3), when wood surfaces were primed with the HMR coupling agent.

One-part polyurethane adhesives are a new class of general purpose wood adhesives that are gaining popularity in the consumer market. They are promoted as waterproof, suitable for exterior and interior applications, and capable of bonding wood to metal, plastic, ceramic, and masonry materials. These products are marketed as single-component, ready-to-use, solventless, liquid adhesives. They are essentially 100 percent solids that cure on reaction of isocyanate terminal groups of polyurethane with moisture in the air and on surfaces. The reaction produces carbon dioxide that causes the adhesive to foam and expand. To develop high-strength structural bonds to wood, high pressure must be used to force entrapped air bubbles from the bondline; otherwise, the adhesive film will not develop its inherent cohesive strength.

In response to many inquiries from users constructing airplanes, boats, outdoor furniture, and various exterior laminates, the USDA Forest Service, Forest Products Laboratory (FPL) provided information on the strength and durability of one-part polyurethane bonds to wood (7). Four commercial polyurethane adhesives, along with a resorcinol-formaldehyde (RF) adhesive to represent a standard of performance, were subjected to a series of industry-accepted tests that were intended to assess varying levels of bond strength and durability. The tests indicated that the dry shear strength, dry wood failure, and wet shear strength of polyurethanes were at least comparable with the RF adhesive. In the ASTM D 2559 (3) test of resistance to deformation under static load, polyurethane bonds to yellow birch were loaded to 165.5 N/cm^2 (240 psi) in separate exposures of 71°C (160°F) dry heat and 90 percent relative humidity (RH). No deformation occurred during 60 days of static loading. Wood failure in wet shear tests, however, was far below that of RF. And, in both moderate and severe cyclic delamination tests, the polyurethane bonds had poor resistance to delamination.

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TABLE 1. – Ingredients in HMR coupling agent.

Ingredients	Parts by weight
Water, deionized	90.43
Resorcinol, crystalline	3.34
Formaldehyde, 37% formalin	3.79
Sodium hydroxide, 3 M	2.44

In this study, our objective was to determine if a coupling agent, hydroxymethylated resorcinol (HMR), could improve adhesion of one-part polyurethanes well enough to overcome the deficient wood failure in the wet shear tests and the poor resistance to delamination in the moderate and severe cyclic delamination tests, especially the ASTM D 2559 (3) delamination test. This HMR coupling agent has been shown to enhance adhesion of all thermosetting wood adhesives to wood, including isocyanate-based wood adhesives (6,9). Preliminary experiments to improve adhesion of these one-part polyurethanes demonstrated dramatic increases in resistance to delamination when wood surfaces were primed with HMR prior to adhesive application (7).

EXPERIMENTAL MATERIALS

ADHESIVES

Two commercial one-part polyurethane adhesives were selected. Each one had demonstrated better durability than two others in bonds to either yellow birch or Douglas-fir in the preceding FPL study (7). A commercial two-part RF adhesive, which is generally accepted as among the most durable of structural wood adhesives, was used as a standard of performance for the polyurethanes. Both types of adhesives normally cure at room temperature. They were applied and cured in general accordance with the supplier's instructions, unless noted otherwise in this report.

WOODS

Yellow birch and Douglas-fir have adhesion characteristics that are typical of moderately high density hardwood and softwood species. The wood of both species was straight-grained heartwood, free of defects, with a relatively slow growth rate. Grain orientation with respect to bondlines within laminates varied from angles of 0 to 45 degrees.

The wood was conditioned at 23°C (73°F) and 50 percent RH to an equilibrium moisture content (EMC) of approximately 9.5 percent. The bonding surfaces were knife-planed 24 hours before laminating.

HMR COUPLING AGENT

A 5 percent solids aqueous solution of HMR was used to prime the knife-planed wood surfaces before bonding with the two polyurethanes. The wood was not primed with HMR before bonding with the RF. The ingredients listed in **Table 1** were reacted at room temperature for 4 hours before HMR application. Dodecyl sulfate sodium salt (0.5% by weight) was added to the HMR solution at the end of the 4-hour reaction time to aid wetting of the wood surfaces. This is particularly useful on resinous softwood species. The length of the reaction time is critical to the durability of the resultant adhesive bond (8). Depending on wood species, adhesive type, and level of delamination resistance to be achieved, reaction time could vary between 4 and 6 hours or range more widely between 3 and 8 hours.

EXPERIMENTAL METHODS

EXPERIMENTAL DESIGN AND ANALYSES

Two statistical experiments were conducted to analyze sources of variation (ANOVA) in shear strength and wood failure as affected by three adhesives (two polyurethanes (C and D) and RF); two wood species (yellow birch and Douglas-fir); two surface treatments (unprimed and HMR-primed); and four test methods (dry condition (DRY), vacuum and atmospheric pressure water soak (VAS), vacuum and high-pressure water soak (VPS), and boil-dry-boil (BDB)). Five specimen panels were replicated for each adhesive, species, and surface treatment combination. Since each panel was cut into 20 block-shear specimens, with each specimen randomly assigned to each of four test methods, variation was analyzed as a split plot design and statistical differences were detected by multiple-range testing.

Statistical analyses were not conducted for the two delamination tests. Percentages of delamination were simply reported as means that were compared with specification requirements.

TEST METHODS

Tests of shear strength and wood failure were conducted according to industry-accepted test methods that were designed to assess varying levels of durability of bonds in both hardwood and softwood plywood products. The four levels of test severity were DRY, VAS, VPS, and BDB. These procedures are specified in American National Standard ANSI/HPVA HP-1-1994 (4) and U.S. Product Standard PS 1-83 (1).

Resistance to delamination was measured in a moderately severe two-cycle boil test, specified in ANSI/HPVA HP-1-1994 (4). A more severe test of resistance to delamination was conducted according to ASTM D 2559-92 (3), which is the specification used to qualify adhesives in structural laminated wood products intended for exterior exposure.

SPECIMENS

Shear strength and wood failure were measured from small compression-loaded block-shear specimens that had shear areas of 6.45 cm^2 (1.0 $in.^2$). Test specimens were cut from 2-ply laminated panels with grain direction both parallel to the laminates and in the same direction as the shearing stress.

Small block-shear specimens were substituted for tension-loaded lap-shear specimens of the same shear area. This substitution was made because stress analysis has shown that tensile stress perpendicular to the bondline, not shear, is the primary stressing mode in lap-shear specimens (5).

A 2-ply delamination specimen with 76-mm (3-in.) sides was cut from each test panel. Delamination was measured along the bondlines on all four sides.

The 6-ply lumber laminates were cut into 76-mm (3-in.) sections, which were used to measure delamination of bondlines on both end-grain surfaces of these specimens. Individual laminates were 19 mm (3/4 in.) thick, 76 mm (3 in.) wide, and 305 mm (12 in.) long.

SPECIMEN PREPARATION

All 2- and 6-ply laminates were prepared in the same manner. If the surfaces were to be primed before bonding, the 5 percent HMR solution was spread on

TABLE 2. – Dry and wet shear strengths of polyurethane (C and D) and RF adhesives on unprimed and HMR-primed yellow birch and Douglas-fir:

Adhesive	Dry strength		VAS[a] (wet strength)		VPS[b] (wet strength)		BDB[c] (wet strength)	
	Unprimed	HMR primed	Unprimed	HMR primed	Unprimed	HMR primed	Unprimed	HMR primed
	----------(N/cm^2 (psi))----------							
Yellow birch								
C	2,202 (3,193)	2,253 (3,268)	830 (1,206)	818 (1,187)	838 (1,216)	828 (1,201)	711 (1,032)	738 (1,067)
D	2,200 (3,189)	2,367 (3,433)	840 (1,218)	843 (1,222)	800 (1,159)	812 (1,178)	744 (1,079)	726 (1,053)
RF	1,910 (2,769)	--	901 (1,308)	--	889 (1,269)	--	809 (1,173)	--
Douglas-fir								
C	1,138 (1,650)	1,333 (1,934)	590 (856)	612 (887)	625 (906)	610 (884)	513 (744)	485 (704)
D	1,158 (1,679)	1,258 (1,825)	600 (870)	620 (899)	637 (924)	644 (934)	477 (691)	515 (747)
RF	927 (1,344)	--	652 (946)	--	663 (961)	--	--	509 (738)

[a] VAS = vacuum atmospheric pressure soak.
[b] VPS = vacuum pressure soak.
[c] BDB = boil-dry-boil.

both bonding surfaces with a brush at approximately 0.15 kg/m^2 (0.03 psf). The primed surfaces were dried for 24 hours at 23°C (73°F) and 50 percent RH before bonding.

Primed and unprimed laminates were sprayed with a fine mist of water before adhesive application, as recommended by the adhesive suppliers. Approximately 1 minute after misting, a weighed amount of adhesive was spread on each surface with a roller at a rate of 0.098 kg/m^2 (0.02 psf). Both adhesive-spread surfaces were immediately assembled and rubbed together to assure contact between surfaces. After approximately 15 minutes closed assembly time, pressure was applied at 69 N/cm^2 (100 psf) on Douglas-fir and 103 N/cm^2 (150 psf) on yellow birch. Constant pressure was maintained for 24 hours at room temperature. After being pressed, the laminates were conditioned at 23°C (73°F) and 50 percent RH for 7 days to complete the cure of the adhesives.

The 2-ply laminates that were to be bonded with RF adhesive were roller-spread with adhesive on each surface at a rate of 0.146 kg/m^2 (0.03 psf). After approximately 5 minutes open assembly time and 20 minutes closed assembly time, pressure was applied and the adhesive cured as previously described for the polyurethane adhesives.

SPECIMEN TESTING

Twenty-five randomly selected block-shear specimens representing each treatment combination were tested for shear strength and wood failure in a dry condition (DRY) at 9.5 percent EMC, as described in ASTM D 905-94 (2). The rate of loading was 5 mm/min. (0.2 in./min.). Load at failure is shown in both Newtons per square centimeter and 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 randomly selected block-shear specimens, representing each treatment combination, was subjected to a vacuum, atmospheric-pressure water soak (VAS) (1). The specimens were submerged in water at 49°C (120°F) while a vacuum of 51 N/cm^2 (15 in. Hg) was maintained for 30 minutes. Water soaking was continued for 15 hours at atmospheric pressure. While wet, the specimens were loaded to failure as previously described.

A third group of 25 block-shear specimens, representing each treatment combination, were subjected to a vacuum, high-pressure water soak (VPS) (1). The specimens were submerged in cold tap water while a vacuum of 8.4 N/cm^2 (25 in. Hg) was maintained for 30 minutes, followed immediately by pressure at 41.4 N/cm^2 (60 psf) for 30 minutes. While wet, the specimens were loaded to failure as previously described.

A fourth group of 25 block-shear specimens, representative of each treatment combination, were subjected to a boil-dry-boil (BDB) test (1). The specimens were boiled in water for 4 hours, then dried for 20 hours at 63°C (145°F). They were boiled again for another 4 hours, cooled in tap water, and tested to failure while wet.

Five 2-ply delamination specimens representing each treatment combination were subjected to a two-cycle boil test (4). 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, then measured immediately for delamination along all edges. Any continuous delamination more than 25.4 mm (1.0 in.) was recorded since this criterion is considered a failure in delamination.

Twelve 6-ply delamination specimens representing each treatment combination were subjected to the three-cycle ASTM D 2559 (3) delamination test. In the first cycle, specimens were vacuum-soaked in water at 8.4 N/cm^2 (25 in. Hg) for 5 minutes, then pressure-soaked in water at 41.4 N/cm^2 (60 psf) for 1 hour. These events were repeated. The specimens were dried at 66°C (150°F) for 21 hours to complete the first cycle. In the second cycle, specimens were steamed for 1.5 hours, followed by pressure soaking in water at 41.4 N/cm^2 (60 psf), and drying at 66°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 the end-grain surfaces. Delamination was expressed as a percentage of total bondline length on the end-grain surfaces.

RESULTS AND DISCUSSION

SHEAR STRENGTH AND WOOD FAILURE IN THE DRY TEST

The two polyurethane adhesives essentially performed the same in the dry test. They developed high shear strengths on yellow birch and Douglas-fir that were significantly higher than that of the RF adhesive **(Table 2).** The polyurethanes are more viscoelastic and tougher than the brittle RF adhesive, so they reach a high ultimate strength before they fail. High percentages of wood

failure in the shear joints were further indications that the full parallel-to-grain strengths of both species were nearly reached **(Figs. 1 and 2).** The strengths of all adhesive bonds to yellow birch were about double those to Douglas-fir, primarily because the wood of yellow birch is of higher density and stronger in shear parallel-to-grain. All adhesives easily exceeded the minimum requirements for dry shear strength and wood failure for exterior-use structural adhesives, as specified in ASTM D 2559 (3).

Further significant increases in dry shear strength were achieved by priming the wood surfaces with the HMR coupling agent **(Table 2).** As shown in **Figures 1** and **2,** very high levels of wood failure near the full strength of the wood were achieved, even without priming with HMR. Thus, the primer did not produce significantly higher wood failure in Douglas-fir joints, although it did significantly increase wood failure of adhesive C on yellow birch.

SHEAR STRENGTH AND WOOD FAILURE IN THE WET TESTS

As in the dry tests, the two polyurethanes performed essentially the same in the wet tests **(Table** 2). Their wet strengths did not differ significantly in either the VAS, VPS, or BDB tests. However, the polyurethane bonds were significantly lower in strength than the RF bonds in all three tests. Wet strengths on the higher density and stronger yellow birch were much higher than on Douglas-fir. The HMR primer produced no significant increases in wet strength in any of the three wet tests.

On the unprimed wood surfaces, wet wood failures of the polyurethanes were quite low **(Figs. 1** and **2),** particularly in bonds to the higher density yellow birch. By contrast, the RF adhesive produced bonds that were dramatically higher in wood failure on both species of wood. Essentially, the RF bonds were as strong as the wood itself, as indicated by this high level of wood failure.

Of the polyurethanes, only polyurethane D displayed wet wood failure on unprimed yellow birch **(Fig. 1)** that exceeded the 15 percent required for technical and Type 1 grades of plywood, as specified in ANSI/HPVA HP- 1 - 1994 (4). Both polyurethanes developed much higher levels of wood failure on Douglas-fir **(Fig. 2),** but that standard applies only to hardwoods. Neither could the two polyurethanes meet the 80 and 85 percent wet wood failure of the VPS and BDB tests, respectively, as specified for softwood construction plywood in PS 1-83 (1). The 45 percent requirement specified for the VAS could not be met either.

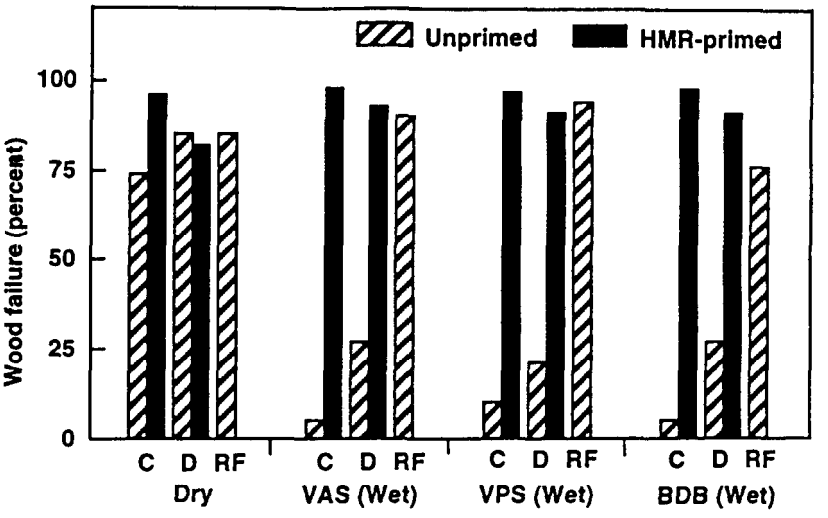

Figure 1 .—Dry and wet wood failures of polyurethane (C and D) and RF adhesives on unprimed and HMR-primed yellow birch; VAS = vacuum and atmospheric pressure water soak; VPS = vacuum and high-pressure water soak: BDB = boil-dry-boil.

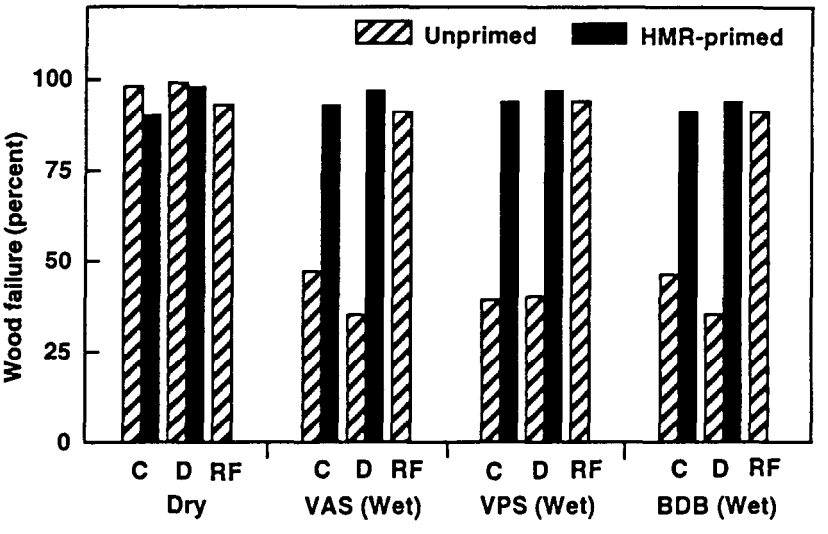

Figure 2.—Dry and wet wood failures of polyurethane (C and D) and RF adhesives on unprimed and HMR-primed Douglas-fir; VAS = vacuum and atmospheric pressure water soak; VPS = vacuum and high-pressure water soak; BDB = boil-dry-boil.

The most important finding is that the HMR primer greatly increased the durability of the polyurethane bonds, as can be seen in all three tests of wet wood failure **(Figs. 1** and **2).** Because of priming with HMR, wet wood failure of polyurethanes increased dramatically compared with that achieved on the unprimed wood surfaces. Not only did high levels of wet wood failure develop on the low-strength Douglas-fir but also on the high-strength yellow birch. Both polyurethanes on primed surfaces performed comparably to the RF, but in the BDB test, they were significantly higher in wet wood failure than the RF on yellow birch **(Fig. 1).** For unknown reasons, 3 of 25 RF-bonded specimens developed atypically low wet wood failure even though wet shear strength of those specimens was very high.

TABLE 3 —Delamination after a two-cycle boil-dry test of unprimed and HMR-primed yellow birch and Douglas-fir bonded with polyurethane (C and D) and RF adhesives.

Specimen	Yellow birch		Douglas-fir	
	Unprimed	HMR-primed	Unprimed	HMR-primed
	(mm (in.))			
Adhesive C				
1	31.8 (1.25)	0.0 (0.00)	6.4 (0.25)	0.0 (0.00)
2	11.7 (0.46)	0.0 (0.00)	11.4 (0.45)	0.0 (0.00)
3	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)
4	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)
5	19.3 (0.76)	0.0 (0.00)	29.2 (1.15)[a]	0.0 (0.00)
Total	62.8 (2.47)	0.0 (0.00)	47.0 (1.85)	0.0 (0.00)
Adhesive D				
1	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)
2	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)
3	11.9 (0.47)	0.0 (0.00)	15.2 (0.60)	0.0 (0.00)
4	5.3 (0.21)	2.5 (0.10)	0.0 (0.00)	2.5 (0.10)
5	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)	0.0 (0.00)
Total	17.2 (0.68)	2.5 (0.10)	15.2 (0.60)	2.5 (0.10)
RF adhesive				
1	0.0 (0.00)	--	0.0 (0.00)	--
2	0.0 (0.00)	--	0.0 (0.00)	--
3	0.0 (0.00)	--	0.0 (0.00)	--
4	0.0 (0.00)	--	0.0 (0.00)	--
5	0.0 (0.00)	--	0.0 (0.00)	--
Total	0.0 (0.00)	--	0.0 (0.00)	--

[a] Specimens with more than 25.4 mm (1 in.) of continuous delamination failed the test. Ninety percent of the specimens must pass.

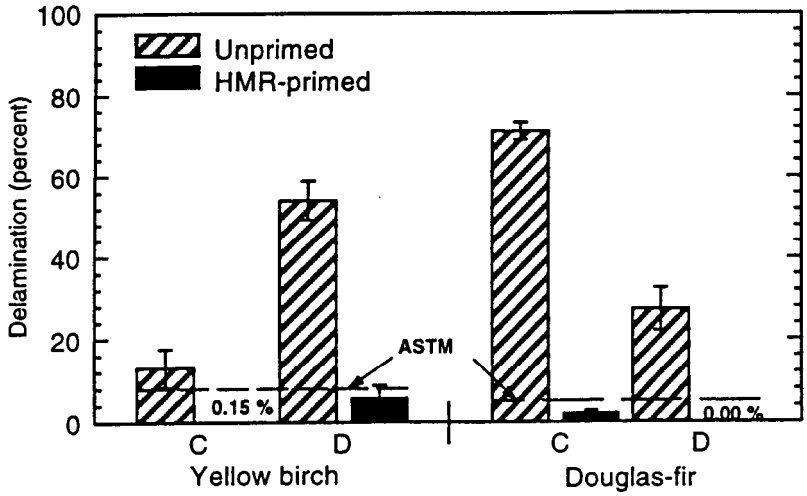

Figure 3. — Delamination of polyurethane bonds in unprimed and HMR-primed yellow birch and Douglas-fir lumber joints after the ASTM D 2559 cyclic delamination test (maximum delamination allowable in ASTM D 2559: 5% softwoods, 8% hardwoods).

DELAMINATION IN THE TWO-CYCLE BOIL TEST

No delamination occurred in any of the RF-bonded yellow birch and Douglas-fir specimens **(Table 3).** Likewise, no significant delamination occurred among the polyurethanes in the HMR-primed specimens. When the surfaces were not primed, however, polyurethane C failed to meet the requirements of ANSI/HPVA HP-1- 1994 (4) on both species of wood. Polyurethane D did meet requirements despite delamination within a few specimens. Clearly, the HMR coupling agent enhanced the resistance to delamination of the polyurethane adhesives on hardwood and softwood species, so they met requirements of the hardwood plywood standard. They also performed as well as the durable RF adhesive.

DELAMINATION IN THE ASTM D 2559 TEST

Both polyurethane adhesives had poor resistance to delamination on the unprimed wood surfaces during the very severe cyclic delamination test of ASTM D 2559 (3) as shown in **Figure 3. Figure 4a** shows delamination of polyurethane D adhesive in unprimed yellow birch lumber joints (53.5%). **Figure 5a** shows delamination of polyurethane C adhesive in unprimed Douglas-fir joints (71.1%). Neither adhesive could meet the maximum allowable delamination requirements on either species, which are specified as 8.0 percent or less for hardwoods and 5.0 percent or less for softwoods. When the wood surfaces were primed with HMR, however, the polyurethanes easily exceeded these requirements. Typical low levels of

delamination on HMR-primed surfaces are shown in **Figures 4b** and **5b,** where polyurethane D delaminated 5.8 percent on yellow birch and polyurethane C delaminated 2.1 percent on Douglas-fir. On closer comparisons within **Figure 3,** it is apparent that polyurethane C resisted delamination on yellow-birch much better than polyurethane D (13.0% compared with 53.5%, respectively). By contrast, polyurethane D evidently performed much better than polyurethane C on unprimed Douglas-fir (27.2% compared with 71.1%, respectively). Once the surfaces were primed with HMR, however, differences between the very low levels of delamination by either polyurethane on either species were minor and inconsequential **(Fig. 3).**

MECHANISMS OF ADHESION ENHANCEMENT

It is quite clear from this study that HMR greatly enhanced the durability of bonding of isocyanate-terminated, one-part polyurethane adhesives to wood, as had been demonstrated in previous experiments with an emulsion-polymer/ isocyanate adhesive (6,9) and a polymeric diphenylmethane-type diisocyanate adhesive (6). These reactive isocyanate groups, which are common to all three types of adhesives, readily react with the hydroxyl groups of water and other molecules such as HMR. Since HMR strongly adsorbs onto the highly polar lignocellulosics of wood, the wood surface becomes enriched with functional hydroxymethyl groups. Thus, at proper conditions, it is conceivable that covalent bonding may occur between functional, hydroxymethyl groups of resorcinol and terminal isocyanate groups to form durable urethane linkages. Even if covalent bonding does not occur, high-density hydrogen bonding may be strong enough to cause HMR-improved adhesion of one-part polyurethanes to the level of the structurally durable RF adhesive.

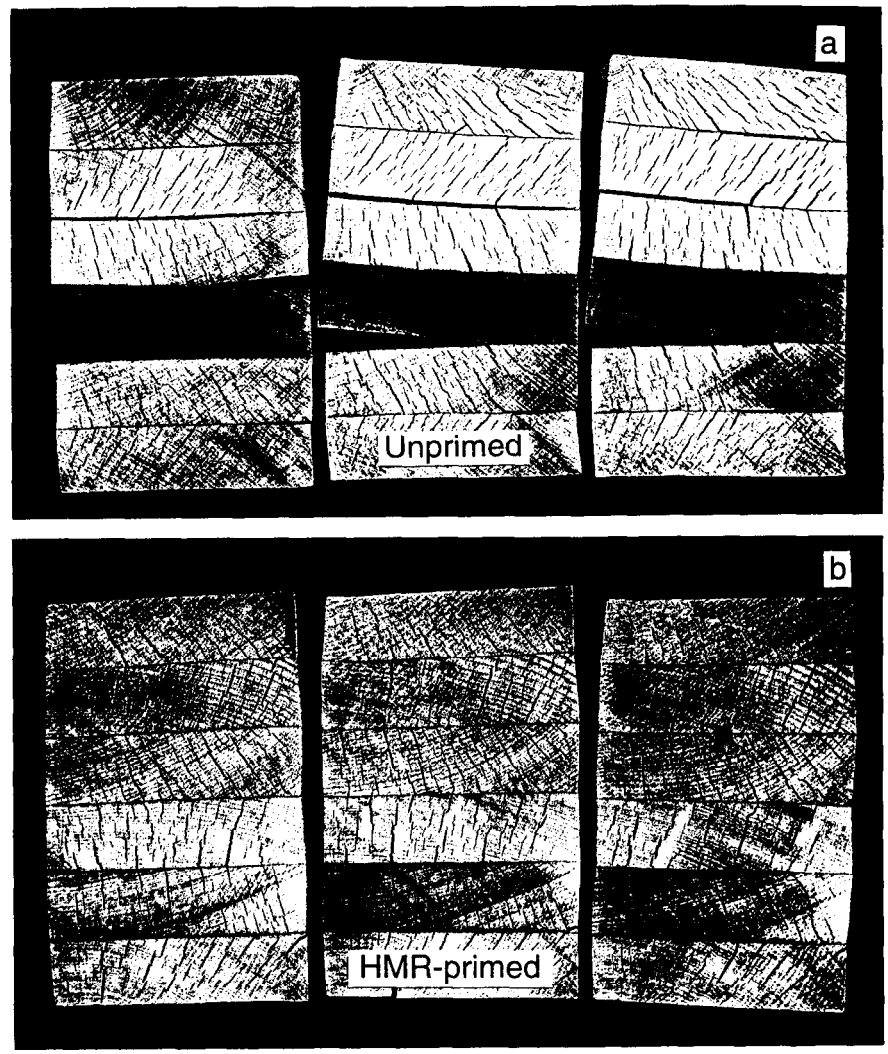

Figure 4. — Cross sections of yellow birch lumber laminates showing (a) 53.5 percent delamination of polyurethane adhesive D in unprimed joints; and (b) 5.8 percent delamination of the same adhesive in HMR-primed joints.

CONCLUSIONS

The HMR coupling agent dramatically improved adhesion of the one-part polyurethane adhesives on yellow birch and Douglas-fir. In tests of wet wood failure and resistance to delamination in both moderate and severe cyclic delamination tests, the polyurethanes performed poorly when the wood surfaces were not primed with HMR. After priming with HMR, though, performance of the polyurethanes was equivalent to the highly durable RF structural adhesive in tests of dry and wet shear strength and wood failure and resistance to delamination. Furthermore, polyurethanes with HMR priming could meet the strength and durability requirements of the most rigorous of tests, specifically ASTM D 2559 (3).

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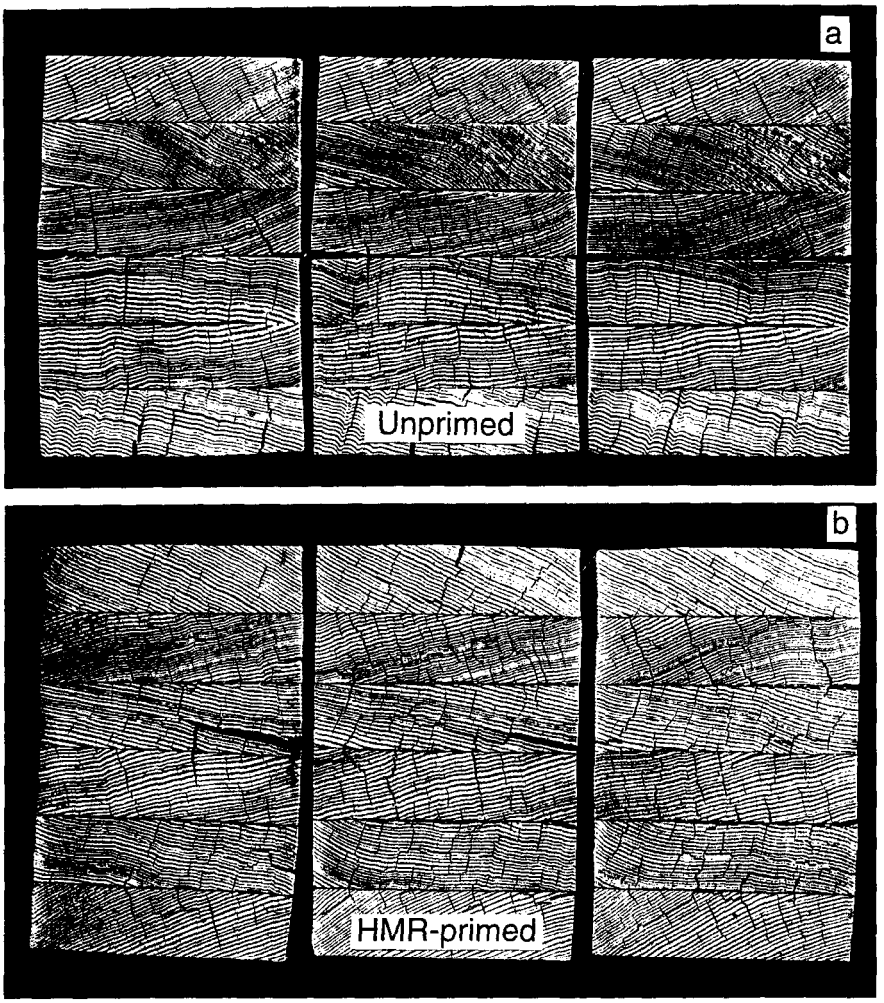

Figure 5. — Cross sections of Douglas-fir lumber laminates showing (a) 71.1 percent delamination of polyurethane adhesive C in unprimed joints, and (b) 2.1 percent delamination of the same adhesive in HMR-primed joints.

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