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What Does Moisture-Related Durability of Wood Bonds Mean?

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ABSTRACT

The accelerated test methods that distinguish between acceptable and unacceptable wood adhesives generally involve subjecting the bonded assembly to abnormally rapid and extreme moisture exposure or cycling. In the United States and Canada, these tests for moisture durability have been established, but selection of the appropriate test methods for the different service classes (extent of water exposure) is still being discussed in some cases. For establishing these standards, a better understanding of the information provided by these tests about the bondline durability is important. Most studies involve different adhesives with the same wood species, and evaluate bond efficacy using strength and percent wood failure. We studied different wood species, especially in durability testing, to provide crucial insight into the factors that contribute to a durable bond and demonstrate the utility of the percent wood failure. The bonding and bond durability of white oak, sugar maple, aspen, Sitka spruce, southern yellow pine, and ipê are covered. The data indicate that the wood properties greatly influence the internal or interfacial stress and thus the durability of the bonded assembly.

KEYWORDS

Wood swelling, bond durability, epoxy, ipê

We selected five species as wood substrates for our durability study: white oak (ring-porous hardwood), sugar maple (diffuse-porous hardwood), aspen (lower density, diffuse-porous hardwood), Sitka spruce (lower density, gradual-transition softwood), and southern yellow pine (higher density, abrupt-transition softwood). We evaluated these species using a single adhesive; an epoxy was used to minimize surface wetting problems, overpenetration, and large adhesive void issues, while still showing bondline failures under durability testing. For durability testing both D 2559 wet/dry cycles and D 905 (wet and dry conditions) were used (ASTM 2007). We also studied the bonding of a tropical timber known internationally as ipê (Tabebuia spp.). Ipê is of interest because it is a high density tropical hardwood able to withstand harsh service environments of exterior exposures. The combination of high density (and thus high intrinsic strength) and high concentrations of certain extractives makes ipê highly resistant to biological attack. However, these properties also are also a severe challenge to bondline durability, and thus, we examined the performance of five different ambient curing exterior adhesives using accelerated durability tests.

METHODS

For the epoxy bonding studies, we used FPL 1A, which involves first mixing 100 parts D.E.R. 33 1 epoxy resin (Dow Chemical Company, Midland, MI) with 12.5 parts of benzyl alcohol-., (Aldrich Chemical Company, Milwaukee, WI) and 2.50 parts of CAB-O-SIL® N70-TS hydrophobic fumed silica (Cabot Corporation, Boston, MA). After thoroughly mixing these materials, 11.1 0 parts of D.E.H. 24 hardener (Dow Chemical, Midland, MI) was well mixed in with epoxy. The five wood species used in these bonding studies were white oak (*Quercus* sp., white group), sugar maple (*Acer saccharum*), southern yellow pine (*Pinus* sp.), Sitka spruce (*Picea stichensis*), and aspen (*Populus tremuloides*) with the lumber purchased from several local distributors, sapwood was used for all species, except the white oak.

The method for wood selection has been discussed in detail as has the bonding and testing procedure for the ASTM D 2559 type tests (Christiansen 2005). For the ASTM D 905 test, all wood strips were conditioned at 27°C and 65% relative humidity until bonded. Specimens were prepared by laminating two strips of wood, 6.4 mm thick, 3 1.8 mm wide and 22.9 cm long. FPL 1A epoxy was spread at an approximate rate of 320 - 340 g/m^2 on both surfaces with a rubber-roll hand spreader. Adhesive spread rate was accurately controlled by automatic tare weighing the adhesive on the laminates as they were spread. Pressure for the epoxy was measured by squeeze out, generally about 10psi. After removing material about 3 mm from all sides and ends, four block-shear specimens with a shear area of 25.4 mm by 25.4 mm were cut from each joint assembly to form shear blocks similar except for size differences as described in ASTM D-905-03 (ASTM 2007) and randomly assigned to either the ambient (−10% EMC, equilibrium moisture content), wet, or wet/ambient (re-equilibration to −10% EMC) shear tests. Eight specimens for each wood species were subjected to a single vacuum pressure soak (VPS) of 85 kPa for 5 minutes and 5 17.6 kPa for one hour, and then tested for shear strength and wood failure while in the water saturated condition. The saturation process procedure consists of submerging specimens in tap water at room temperature in a pressure vessel, maintaining a vacuum of 84.66 kPA for 30 minutes maintaining a pressure of 448 ± 35 kPa for 30 minutes, and submerging in water until tested.

uniform structure. On the other hand, bonds with southern yellow pine can lack durability due its relatively high specific gravity, dense latewood bands, higher swelling for a softwood, and its higher strength.

Although epoxies are too expensive for most wood bonding applications, they are used in repair of wood-to-wood bonds (e.g., wooden bridges, bolted timbers, airplanes, and boats) (Selbo and Bohannan, 1968; Avent et al. 1976). Epoxies are used for these applications because they have good gap filling properties and need low clamping pressures. We chose epoxies because they form bonds easily due to their low surface tension, have limited pH sensitivity, use the same clamping pressure for the different species, are unlikely to form starved bonds due to over penetration, and often produce moisture sensitive wood bonds made it the choice for our study. We used FPL 1-A because it was a known reliable adhesive for critical wood bonding applications, i.e., for aircraft bonding (Myal 1967).

One way to test durability is to examine the shear bond strength of both ambiently stored and re-equilibrated (after water soaking) specimens. We tested the shear strength of the bonded wood specimens not only using standard ambient and wet (water-soaked) conditions of the standard D 905 test but also after allowing the wet specimens to come back to the normal ambient condition. In Figure 1, it is clear that the bond strength varies by wood species and test conditions. The stronger wood species (oak, maple, and pine), see shear parallel to grain in Table A, give higher bond strengths for the ambient specimens (shear strength in figure 1, but the bond strengths for all species decline under the wet conditions, as expected based on the properties of wood itself (Green et al. 1999). However, all the strengths return within 90 % of the original upon allowing the specimens to re-equilibrate back to 10% moisture content. A similar phenomenon was observed previously by Gillespie (1976) in which sugar maple and eastern white pine ASTM D-905 epoxy bonded assemblies were subjected to substantial moisture extremes (i.e., 2% to $\geq$ FSP). He studied what effect shrink-swell and swell-shrink cycles had on the shear strength and wood failure. He found that for epoxy-bonded assemblies, the maple retains 87% of original strength upon going from 12% to 2% and back to 12% EMC, but only retains 50% of original strength in going from 12% to 2 FSP and back to 12% EMC. More dramatically, he found that the epoxy-bonded pine retains full strength upon going from 12% to 2% EMC or 2 FSP and re-equilibration back to 12% EMC.

Because wood strength decreases as its moisture content increases, the data in Figure 1 alone does not indicate whether the" bond or the wood is responsible for these changes in shear strength. The shear strength data needs to be combined with the percent wood failure to understand what is happening in these tests. The data in Figure 2 generally show a lower percent wood failure and therefore greater adhesive failure as the shear strength of the assembly increases. Thus, the epoxy forms reasonably strong bondlines, but when the wood is strong enough to exert high force upon the bond, the epoxy may fracture first. The percent wood failure decreases for all but the aspen and Sitka spruce in the wet specimens compared to the ambient specimens. Thus, even though the wood is weakening and putting less force on the bondline during the shear test, the bondline is failing first more often. The percent wood failure values for the wet/ambient recover to near those of the ambient specimens. Again, the previous study by Gillespie (1976) displayed a similar trend in wood failure results for maple and eastern white pine. Thus, the extra strain due to the differential swelling between the wood and adhesive causes an internal stress on the bond and greater failure within the bond. Those wood species with the greatest swelling have the largest decline in wood failure supporting the model of wood strain (Frihart 2007, Gillespie

In this study, we tested the bond strength of block-shear specimens of ipê under ambient (1 0% EMC) and wet (2 FSP) conditions. Figure 3 shows the results of the compressive shear test. In the ambient (1 0% EMC) condition, it is apparent that most adhesives performed well. The EPI and PRF gave the highest average shear stresses of 21.9 and 20.6 MPa. The EPO, PUR1, and PUR2 gave very similar average shear stresses of 17.1, 17.2, and 18.4 MPa. After the VPS cycle, the compressive shear stress dropped considerably with all adhesives (averaging –5 MPa), except the PRF adhesive, which gave an average shear stress of 14.2 MPa. This dramatic difference in wet-bond strength between the PRF and the other four adhesives displays why phenolic and resorcinolic adhesives still exist in today's market as the true standard for exterior bondline exposure. Under ambient conditions, almost all adhesives showed exceedingly poor wood failure results after the shear test, except for EPI and PRF with averages of 72 and 84%, respectively (Figure 4). After VPS exposure, the wood failure results are again quite dramatic, showing that most adhesives gave average values of only 0-5% while the PRF gave an average of 43%. The dimensional changes measured in the bondline area after the VPS cycle for all adhesives displayed an average change of only 2%. These collective results suggest that most adhesives used to bond ipê cannot withstand the swelling stresses induced at the bondline during the VPS cycle, even with very small final dimensional changes. However, PRF seems to have a way of stabilizing the bondline and the interphase region, thus maintaining a sufficient amount of bondline integrity to allow failure in the wood.

Using fluorescence and transmitted light microscopy, we analyzed cross-sections of several bondlines with all five adhesives. For simplicity, we selected two magnification scales (6.3X and 40X) from all adhesives analyzed and the 6.3X magnification images are shown in Figure 5. From the microscopic images, it is seen that all adhesives have difficulty in penetrating the ipê anatomical structure. Most are able to fill the vessel lumina and voids between fibers fairly well. Vessel elements are not thought to contibute significantly to the overall strength of hardwood xylem. It is the fibers, with their thick secondary cell wall layer and more substantial length, which are thought to contibute most to the bondline integrity. In the wood failure analysis of ipê, we noted that when the wet PRF-bonded specimens failed, the failed surface typically displayed wood only a few cells deep (viewed in Environmental Scanning Electron Microscopic images). We hypothesize that PRF, being known as an adhesive with capabilities of permeating the cell wall, may diffuse into the first few fiber cells and act as stress-deminisher for fractured surfaces.

Testing Moisture-Related Durability

The purpose of these accelerated tests is to predict real performance and bond failure in exterior exposure. The D 2559 is considered to be a minimum requirement for most structural bonding processes. It does allow for 60% greater failure with hardwoods, most likely due to the greater swelling and shrinking of the more dense species. Does a single or a few cycles of high amplitude strain caused by rapid wetting and drying of the wood in the accelerated tests represent the slower wetting and drying in actual end use? The slow natural penetration of moisture deep into the wood and the stress relaxation modes of the wood could indicate that the rapid cycles may not be a good representation of actual end use conditions. However, in general the literature shows a good relationship between those adhesives that hold up in exterior exposure and those that pass the accelerated durability tests (Caster 1980, Frihart 2008, Iwata and N Inagaki 2006, Raknes 1997, Sugimoto et al. 2007). In addition, the tests recommend the use of the same wood species that is used in the end application, thus eliminating the effect of the structural, chemical, and swelling differences from giving an incorrect assessment.

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Table A. Properties of Wood Species used in these Studies

Species	R shrink	T shrink	V shrink	SG	Shear ||	MOE	Growth Rings
white Oak	4.4	8.8	12.7	0.68	13800	12300	Ring Porous
sugar maple	4.8	9.9	14.7	0.63	16100	12600	Diffuse Porous
aspen	3.3	7.9	11.8	0.39	7400	9900	Diffuse Porous
Sitka spruce	4.3	7.5	11.5	0.36	6700	9900	Gradual Transition
SYP	4.8	7.4	12.3	0.51	9600	12300	Abrupt Transition
ipê	6.6	8	13.2	0.92[a]	14600	21600	Diffuse Porous

Notations: R, T, and V shrink are green to oven-dry % shrinkage in the radial, tangential and volumetric modes, respectively; SG is specific gravity (oven-dry weight / volume at 12% moisture content), Shear ll is shear strength parallel to grain in KPa; MOE is modulus of elasticity in MPa,. [a]Ipe specific gravity is oven-dry weight / green volume.

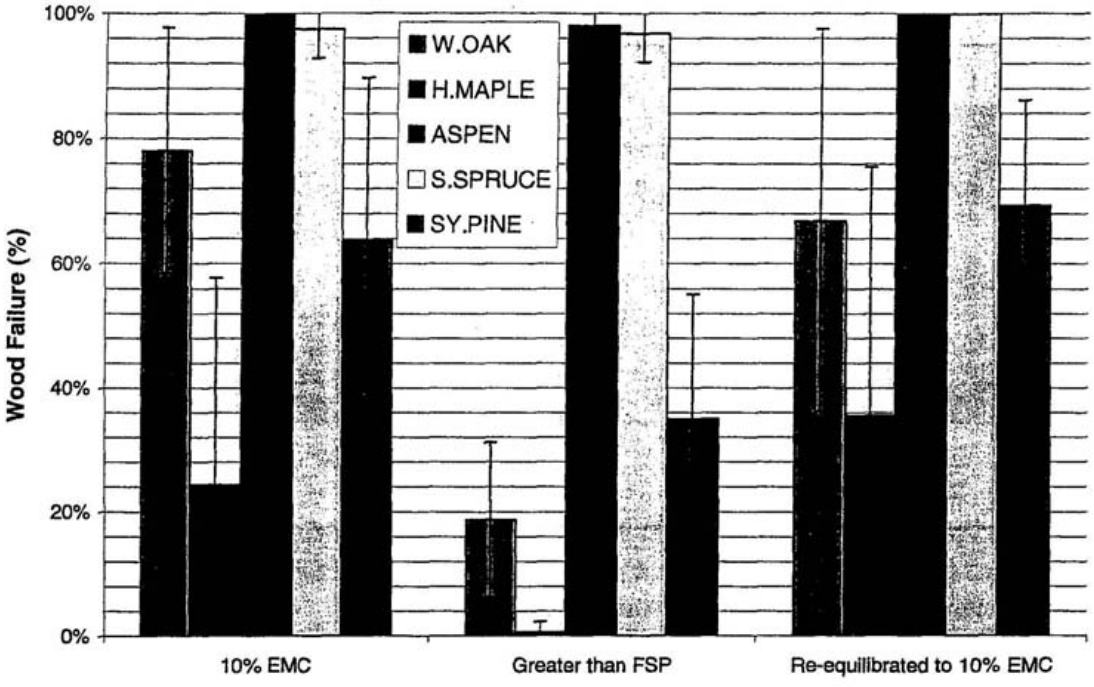
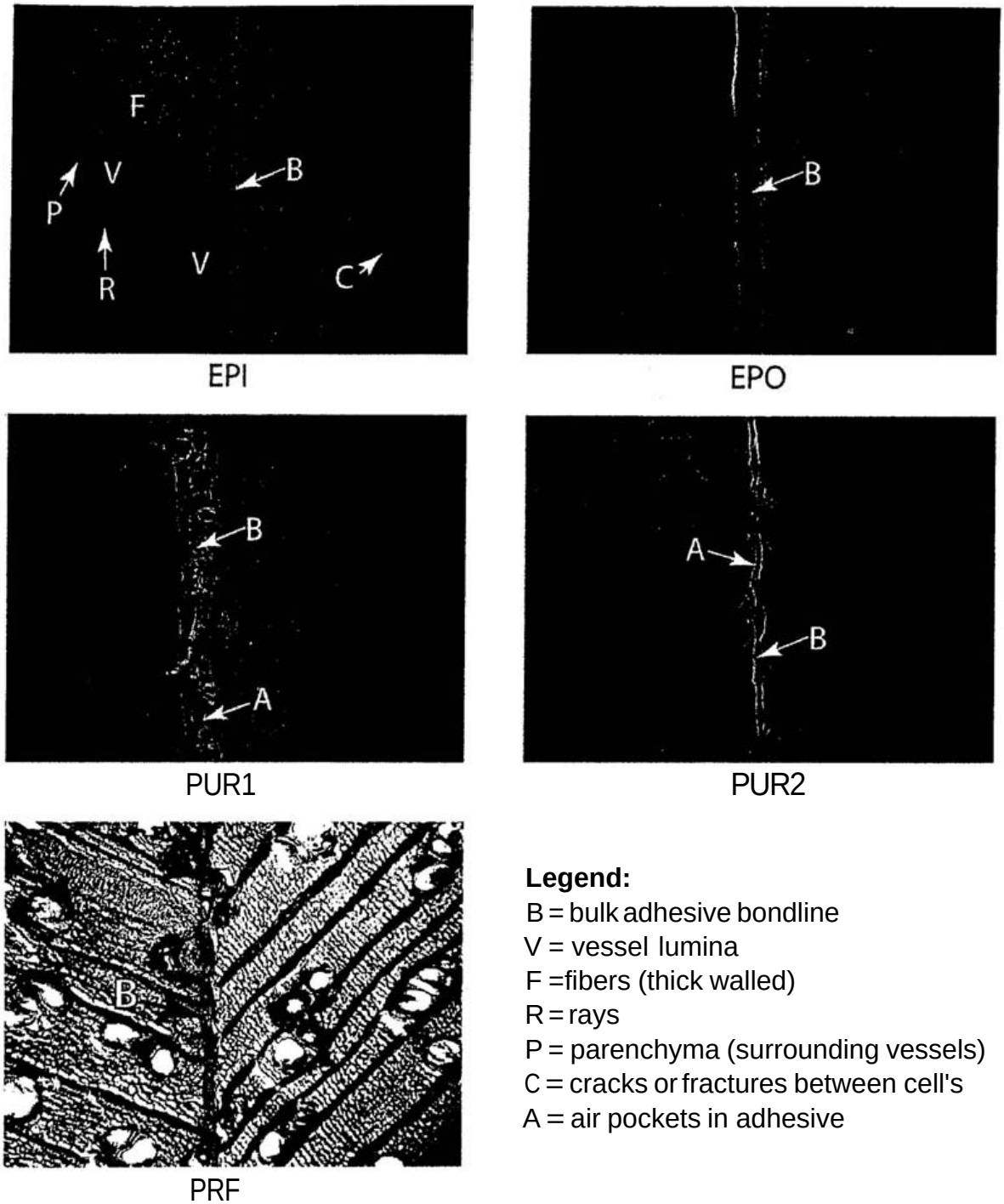


Figure 2. Percentage wood failure for FPL 1A epoxy as determined using ASTM D 905 and ASTM D 5266 testing for different wood species and tested for ambient (1 0% EMC), vacuum pressure soak (>FSP), and vacuum pressure soak followed by re-equilibration to 10% EMC (wet/ambient) specimens. The error bars are ± two standard deviations around the average value.

Table B. % Delamination determined using ASTM D 2559 testing for FPL 1A epoxy with different wood species. Only the Sitka spruce had sufficient durability to meet the standard requirements.

Wood species	**% Delamination**	
	Assembly 1	Assembly 2
White Oak	83.1	83.9
Sugar Maple	79.1	84.2
Aspen	10.5	13.9
Sitka Spruce	0.0	0.0
Southern yellow pine	67.3	51.7



Legend:
B = bulk adhesive bondline
V = vessel lumina
F =fibers (thick walled)
R = rays
P = parenchyma (surrounding vessels)
C = cracks or fractures between cell's
A = air pockets in adhesive

Figure 5. Fluorescence microscopy of the cross-section of ipê bonded with five exterior adhesives. Images are the following: EPI (emulsion polymer isocyanate), EPO (epoxy), PUR1 (polyurethane, fast curing), PUR2 (polyurethane, slow curing), PRF (phenyl-resorcinol formaldehyde). Images are shown at 6.3X. The PRF images are transmitted light only because of non-fluorescence of this adhesive.

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