

Assessment of Increased Wet Wood Bonding for Epoxy-Bonded Samples Using a Melamine-Urea-Formaldehyde Priming Agent

Jermal G. Chandler
Chemist

Charles R. Frihart
Project Leader, Wood Adhesives Science & Technology, USDA Forest Service, Forest Products Laboratory, Madison, Wisconsin, USA

Abstract

Is the hydroxymethylated resorcinol (HMR) primer unique or can a melamine-based primer also increase the wet wood strength of epoxy bonds? Although the exact reason for poor durability with some wood adhesives is not known, the HMR priming agent was found to facilitate durable bonds in most cases tested. A model of cell wall stabilization that is believed to be the mechanism of HMR has led to the consideration of melamine-based primers. Previous studies have shown that melamine resins can increase the hardness of wood cell walls and lower the amount of swelling from water impregnation. Primer formulations of melamine-urea-formaldehyde (MUF) with various solid contents and bonded using a typical epoxy adhesive (FPL-1A) yielded high wood failure of block shear samples under wet conditions. Untreated samples of the same wood species and conditions had a low percent wood failure and shear strength. The primer was most effective at high dilutions, and performed poorly at concentrations greater than 5 %w/w of the solid polymer. Thus, we concluded that MUF, like HMR, can help create more durable bonds and may be related to modification of the wood cell walls.

Introduction

The limited structural durability of epoxy bonds to wood has always been a problem for fabricators of adhesive-bonded wood products intended for service in exterior environments. Epoxy adhesives do not possess the

same durable nature as phenol or resorcinol adhesives in wood assemblies. Although epoxy adhesives develop dry shear strengths that exceed the strength of the wood itself, the epoxy bonds fail in delamination once exposed to the severe stresses associated with water soaking and drying. In the 1960s, Olson and Blomquist (16) worked toward developing more durable epoxy formulations. One such formulation, FPL 16, was capable of exposure to a 120-hour boil and dry test. This formulation was modified, marketed privately as FPL 16A, and was a popular adhesive for aircraft applications. The consensus was that FPL 16A was not as durable as a typical phenol-formaldehyde (PF) resin, however. Caster of the Weyerhaeuser Company, in cooperation with Dow Chemical Company, made further progress toward more durable epoxy bonds by priming wood surfaces with a 2 percent aqueous solution of polyethylenimine (4). This surface treatment allowed two adhesively bonded assemblies to perform comparably to small, solid wood specimens in accelerated aging and exterior exposure tests. Unfortunately, none of these modifications led to sufficient durability to pass ASTM D 2559 (21). Thus, the need for structural epoxy bonds with greater resistance to stresses from repeated water soaking and drying led to further exploration of chemical surface treatments at the USDA Forest Products Laboratory (FPL), Madison, Wisconsin.

Wood Bonding Issues

The mechanism of adhesion between the resin and wood components, which is still currently under debate, is generally thought to involve mechanical interlocking, covalent bonding, and secondary interactions, such as Van der Waals forces and hydrogen bonding (14). Creation of a bond between an adhesive and the wood substrate requires sufficient penetration of the resin into the wood components (13). Good penetration of the adhesive is promoted by excellent adhesive-to-wood surface interactions and excellent adhesive mobility. Experimentally, wood is often resurfaced prior to bonding in efforts to improve the wood–adhesive interaction and provide a smooth surface possessing minimal extractives and atmospheric debris. Other variables, such as processing factors and methods of heating the adhesive bond, will also influence adhesive penetration (18). Grain direction, permeability, porosity, roughness, surface energy, temperature, pressure, and time are among the other wood and processing factors that could influence the adhesive penetration (14,19). Variation in any of these factors can alter the quality and durability of the bond. For wood bonds, when failure is identified, it is typically either wood or bondline failure. Bondline failure is often simplistically thought of as a lack of adhesion because of the difficulty in determining the main failure location, which mostly is due to the complexity of wood surfaces. As seen in **Figure 1**, Marra has identified five locations for failure when moving from bulk wood to bulk adhesive (14).

Epoxy Adhesive Failure Mechanisms

So where and why do epoxies fail? For epoxies, failure often occurs in the bulk wood (links 8 and 9) under dry conditions and in the adhesive interphase layer (links 2 and 3) under wet conditions (7). A valid explanation for why epoxies fail under wet conditions is their inability to withstand strain during expansion of the wood as it absorbs water under soaking conditions. Previous work showed that fractures tend to occur longitudinally (as expected) from swelling forces (8).

In addition to epoxy fracture studies, Frihart et al. (9) showed that epoxy wood samples gave unexpectedly strong bonds to wood (modified by acetylation) under wet conditions. Acetylation with acetic anhydride is a typical chemical modification of the wood cell wall in which the resulting wood has a lower affinity for water. The chemical reaction is detailed in another study (8). The resulting process creates a bulky wood product that is less permeable to water and less subject to dimensional change (17). This study reasoned that acetylation stabilized the wood and created a surface that would help prevent the epoxy bond from failing when the wood was swollen with water.

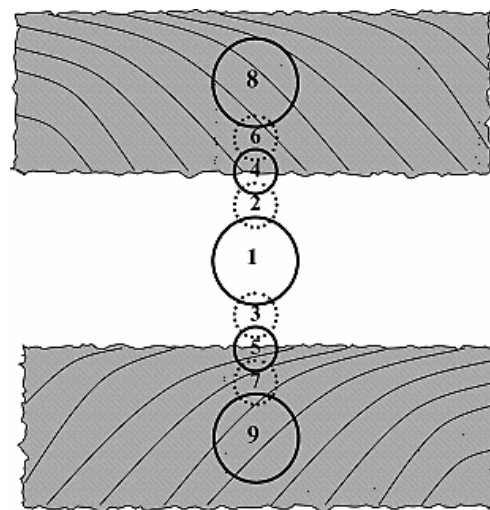


Figure 1. ~ Links in an adhesive bond. Dashed links are the most vulnerable to malformation.

Additional experiments at FPL by Yelle and Frihart (22) showed the recoverable nature of the wood bondline and cell structure after stress and water soaking. Several different wood species bonded with FPL 1A epoxy were subjected to ASTM D 905 testing (2). Dry samples were tested as-is; wet samples were tested after vacuum pressure soaking; and wet-dry samples were allowed to dry for 1 week in an 80°C, 65 percent humidity room. Per usual, wet shear samples gave much lower wood failure than dry shear samples. An unexpected result was that the wet-dry samples nearly equaled the stress value of the dry samples. This was evidence that the wood and its cell walls had recovered from strain and expansion during soaking conditions. The cell wall, in the wet-dry state, was in the process of returning to a “less strained” state. This cell wall stabilization theory has become the new model for explaining why hydroxymethylated resorcinol (HMR) produces bonds of extraordinary durability (5).

HMR

Research at FPL demonstrated that when HMR was used as a priming agent on a wood surface bonded with epoxy, it would produce bonds of extraordinary structural durability (20, 21). Additionally, researchers have never quite understood why HMR worked so well and the conceptual models created to explain the chemistry taking place in HMR-primed wood did not seem valid. The initial model of how HMR worked proposed that a coupling agent adhered to the cellulose and hemicellulose compartments of wood, thus physio-chemically bonding the epoxy adhesive and the wood lignocellulosics (20). This, however, did not explain why the early HMR formulation (non-novolak based) needed to be used 4 to 8 hours after mixing to perform well. Apparently the length of reaction time (defined by the time after mixing) determined the

molecular weight distribution and reactivity of HMR; thus, the reaction time had a strong effect on adhesion. This did not appear to be the act of a coupling agent, whose distinct purpose is to promote a stronger bond at the interface. Current literature has begun to indicate a mechanism involving cell wall stabilization.

HMR has been challenging to study because it has chemical functional groups that are similar to cellulose, hemicellulose, and lignin components in wood (Fig. 2).

It is not known whether or not the similarity in structure promotes good priming ability, but it does make HMR hard to analytically differentiate from wood.

Melamine Priming Agents

Why use melamine-urea-formaldehyde (MUF) as a primer for wood surfaces? Melamine-based compounds have not been previously used as priming agents for wood. These resins are known to have increased stability against hydrolysis (6), which helps make MUF-based wood bonds water resistant. This is due to the stabilization of the C-N bond by the quasi-aromatic ring structure (Fig. 3).

A water resistant compound such as MUF could decrease the amount of swelling seen in epoxy bonds during soaking conditions. Additionally, Gindl et al. (10,11) showed that MUF could be detected in the cell wall by ultraviolet (UV) microspectroscopy, and melamine sucrose formaldehyde (MF) treated cell walls showed an increase in Young's modulus of 33 percent (vs. untreated). Further, Miroy et al. (15) used hexamethoxymethyl melamine (which is also known as hexamethylol-melamine-methyl-ether or MME) to increase the Brinell hardness of wood and lower its volumetric variation in water. Hexamethoxymethyl melamine, a melamine derivative, prefers to react with polyols and phenols. So, it is capable of reacting with the hydroxyl groups of wood (cellulose or lignin). Clearly, melamine resins have the potential to affect the mechanical properties of wood on a micro level.

An important reason for the use of MUF as a primer is for characterization purposes. The MUF synthesis is a straightforward reaction (Fig. 4) and the C-N and the N-H bonds, which are not common in wood, allow melamine components to be easily identified in $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ (1).

Additionally, melamine can be identified in the UV region with a broad absorbance around 240 nm and a small absorbance at 280 nm with little interference from wood (lignin also has a characteristic absorbance at 280 nm) (12). Melamine also has a characteristic spectrum in the mid-IR region. A vibration for the triazine ring sextant occurs at 813 cm^{-1} (12).

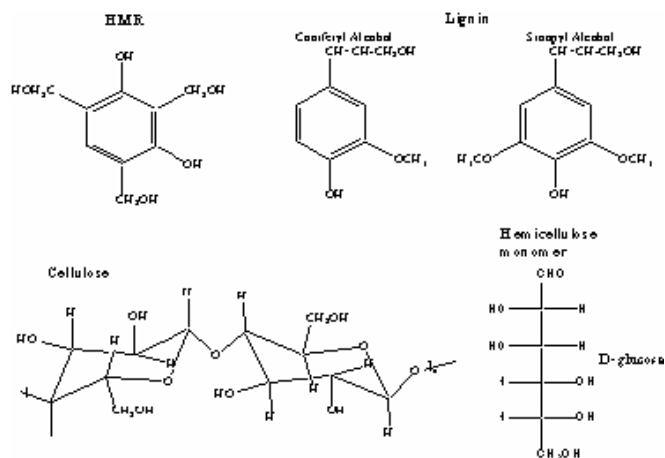


Figure 2. ~ Chemical structures of HMR, cellulose, hemicellulose, and lignin.

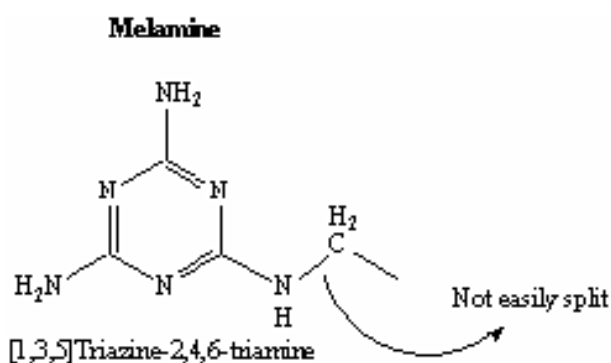


Figure 3. ~ Melamine triazine ring stabilizes against breakdown caused by hydrolysis.

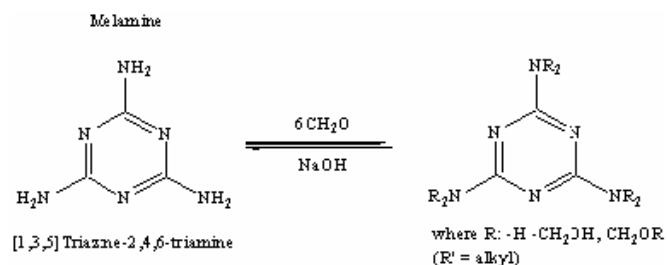


Figure 4. ~ Chemical structure of melamine and product from formaldehyde synthesis.

Experimental

Melamine Primers

Melamine Sucrose Formaldehyde

Liquid melamine sucrose formaldehyde resin was secured from Dynea (Moncure, NC). The resin was 55 percent solid and contained a 3 percent trade secret plasticizer. In this study, the resin is referred to as MF.

MUF

A 50 %w/w solution of formaldehyde was created using paraformaldehyde $[(CH_2O)_m]$ and water to produce 2.96 moles of solution. This solution was heated to $\sim 60^\circ\text{C}$ and stirred for 30 minutes. An additional 4 moles of water were added to the formaldehyde solution and the temperature was raised to 85° to 90°C , which was optimum for melamine dissolution. The pH of the mixture was adjusted to 8.9 to 9.1 using a 25 percent NaOH solution. Immediately upon reaching the desired pH, 1.3 moles of melamine were added to the reactor over a 10-minute period or longer to ensure all melamine was dissolved. After a charge of 4 moles of water to wash the melamine down the reactor, the solution was refluxed for 15 minutes, and then the reactor was removed from heating. At this stage, the haze point and water dilutability of the reaction mixture were checked. When the batch was at a temperature below 60°C , the pH was adjusted to 9.5 to 9.7 using the NaOH solution. Finally, at 35°C , 0.18 moles of urea was added and stirred until completely dissolved. Final product formulation is given below (% mass):

50% w/w formaldehyde	49.6%
Water	8.9%
Melamine	30.5%
Water	8.9%
Urea	2.0%

Overall, this procedure yielded the following ratios:

$$F/M = 2.3 \text{ and } F/(M + U) = 2.0.$$

The final resin specifications were:

Solid content	= 45% to 49%
Brookfield viscosity	= $16.6 \pm 0.2 \text{ mPa}\cdot\text{s}$ (@ 60 rpm and 20°C)
pH	= 9.5 to 9.7
Gel time	= 55.5 min. (Sunshine Gel Meter, 100°C water bath)

Haze point and water dilutability tests often varied from batch to batch depending on small changes in reflux time. This resin was synthesized at the FPL and distinguished by being labeled MUF. Although with the small amount of post-added urea, this is most likely not a MUF resin, but the term is used here to distinguish it from the MF adhesive.

Hexamethylol-Melamine-Methyl-Ether

Hexamethylol-melamine-methyl-ether (MME) was 30 percent solid and was obtained from Structure Probe, Inc. (SPI). The resin was made in Ulm, West Germany and distributed in West Chester, Pennsylvania.

Wood Selection and Priming

For the initial stages of the experiment, yellow-poplar sapwood was chosen for block shear testing. This wood is considered to be even grained, thus eliminating the problems seen with other wood species. The vessel elements

are of consistent structure throughout in this species. In addition, inorganic extractives rising to the surface and creating interference with adhesive surface wetting are not a significant issue because of their low concentration. For purposes of cleanliness and warp minimization, the wood was planed within 24 hours of bonding to ensure a glueable surface.

Experiment 1

The MF resin was used as-is (55 %w/w) and diluted into five solutions (with distilled water): 25, 10, 7, 5, and 3 %w/w. The diluted solutions were mechanically stirred until sufficient mixing was evident. An acid catalyst was used to promote curing that consisted of 10 %w/w p-phenol sulfonic acid in water and a 1:1 molar ratio of acid to morpholine. Two acid levels, 10 %w/w and 1 %w/w, were added to 50 grams of the primer solution just before it was applied to the wood surface. The primer solution-catalyst matrix was applied using the typical spread rate of 0.15 kg/m^2 per bondline. After priming, the samples were stored in a humidity controlled room for 1 week to assure adequate penetration.

Experiments 2 and 3

The MUF resin was cooled to room temperature; five solutions differing in contents of solids were created by dilution with distilled water. This consisted of an as-is solution (45%), 25, 10, 5, 3, and 2 %w/w MUF resin. The diluted solutions were mechanically stirred until sufficient mixing was evident. The same acid catalyst used for the MF (10% w/w p-phenol sulfonic acid in water and a 1:1 molar ratio of acid to morpholine) was used here. One acid level of 10 %w/w was added to 50 grams of the primer solution just before it was applied to the wood surface.

The MME resin (30% solids as-is) was diluted into three solutions: 5, 3, and 2 %w/w. This primer used a different catalyst, which was a crystallized solid of p-toluene sulfonic acid (as provided by SPI). One acid load of 10 %w/w of the solid polymer primer was used. It was then heated in an oven at 40°C for 48 hours and then heated again at 60°C for another 48 hours to promote curing (as instructed by SPI).

The primer solution-catalyst matrix was applied using the typical spread rate of 0.15 kg/m^2 per bondline. After priming, the samples were stored in a humidity-controlled room for 1 week to assure adequate penetration.

Bonding of Surfaces and Shear Test Procedure

Adhesive bonding was done using FPL 1A epoxy adhesive based on diglycidylether of bisphenol-A resin and a triethylenetetramine curing agent, identified by their manufacturer, Dow Chemical Company, as D.E.R. 331 epoxy with D.E.H. 24, respectively. They were mixed 100 parts (weight basis) resin to 11.5 parts curing agent. For diluted mixtures 12.5 parts of non-reactive benzyl alcohol

is added and 2.5 parts of hydrophobic fumed silica (NTS grade Cab-O-Sil by Cabot Corporation) is added to facilitate spreading on the wood surface. Once the hardener is added, there was minimal open assembly time given for each surface (less than 1 minute). A total of 15 to 20 minutes was available before the batch of epoxy fully cured. The spread rate for the epoxy is 0.34 kg/m² per bondline.

Once the epoxy was spread evenly on a surface, it was bonded to its corresponding adherend to form an assembly, and each sample was placed into the cold press to allow even distribution of the load onto the bondlines. Wax paper was placed in between each assembly to prevent sticking, and they remained without pressure for 1 hour. Then, a wood ram was centered on the top and pressure was applied by the cold press until the point of squeeze out or until small amounts of epoxy oozed from the bondline. Experimentally, this pressure was determined to be about 0.7 MPa (10 psi). The cure time under pressure was about 15 hours.

Block shear specimens (3.2 by 2.5 by 0.6 cm) were cut from the joint assemblies and randomly assigned for wet or dry shear test. There was no additional treatment needed for dry specimens; however, wet specimens were subjected to a vacuum pressure soak (VPS) cycle that placed the wood specimens under high stress as well as large quantities of water. The method consisted of 30 minutes under vacuum at 660 mm (26 in.) of mercury (Hg) followed by 30 minutes of pressure at 0.45 MPa (65 psi). The wet and dry specimens were then tested in shear according to ASTM D 905. All specimens were held at a constant load with a test speed of 2.5 by 10⁻³ m (0.10 in.)/min applied until failure. The wood failure was estimated to the nearest 5 percent on the sheared area, according to ASTM D 5266 (3). The compression shear strength data were given by the machine in psi and recorded in MPa. All testing was done using the TestStar 4.0D software and the MTS II material test system both furnished by MTS Testing Corporation. For the presented data, the error bars represent one sigma standard deviation.

Results and Discussion

Experiment 1

This experiment showed that a primer high in solids content (greater than 10%) did not help promote good bonding. These primers produced a glossy film on the surface, and gave minimal, wet and dry, wood failure as can be seen in **Figure 5**.

The importance of catalyst was tested by using the low solids content primers. **Figure 6** is a comparison between MF primers using a 10 percent catalyst and a 1 percent catalyst. The overall result was inconclusive since it was difficult to assess which percentage worked best.

Given the short pot life of the resin and the unknown effect of various additives, we decided that an MUF resin would be created at FPL as opposed to acquiring MF from outside sources. Also, future experiments would only use primer formulations of 5 percent MUF and lower, thus making the formulation closer in solids content to the HMR primer and within range of good performance of the MF resin tested in this experiment. The lower solids content was accomplished by introducing more water during the synthesis of the resin. This concept is reflected in the product formulation given in the experimental section.

Experiment 2

The synthesized MUF solution had an initial solid content of 45 percent, which was lower than the 55 percent solids previously used by the MF. **Figure 7** further demonstrates that primer formulations lower than 5 per-

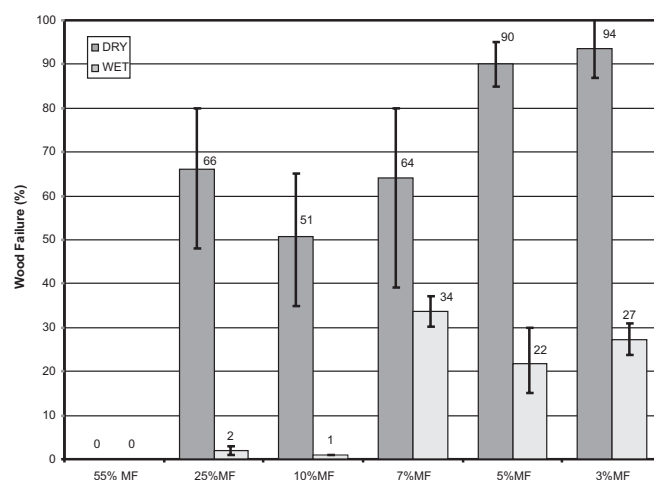


Figure 5. ~ Wood failure of melamine sucrose formaldehyde primers using a 1% catalyst.

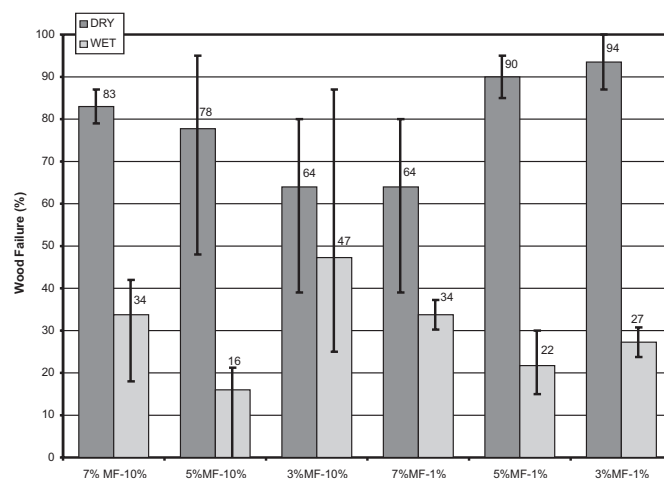


Figure 6. ~ Wood failure of low solids content melamine sucrose formaldehyde primers comparing 10% catalyst vs. 1% catalyst.

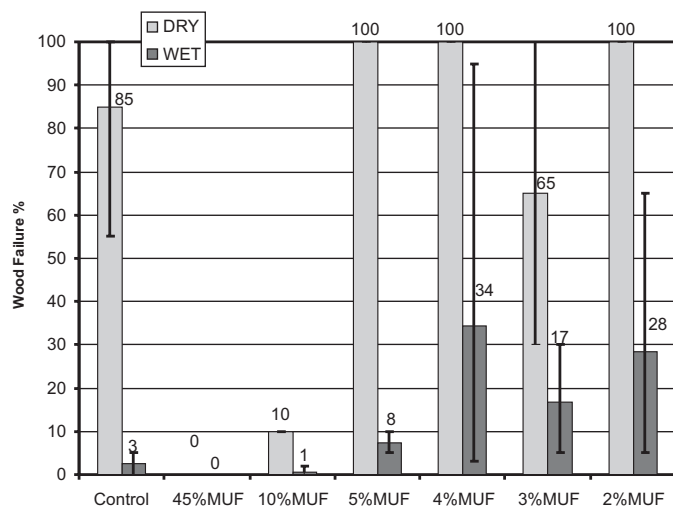


Figure 7. ~ Wood failure of control vs. uncatalyzed MUF primer.

cent, even with no added catalyst, perform better than a controlled sample (epoxy bond with no primer), especially within the wet specimens. Primers equal or *greater* than 10 percent did not achieve noteworthy wet wood failure at all. **Figure 8** shows that an acid catalyst clearly gives better wet wood failure compared to the uncatalyzed MUF samples in **Figure 5**. The 3 percent MUF primer had a wet wood failure of 92 ± 5 percent, and the 2 percent MUF primer had 100 percent failure. These are clearly impressive results.

It seems that primer formulations greater than 3 percent do not soak into the wood completely, thereby making it difficult for the epoxy adhesive to penetrate the wood or bond to the MUF primer. Viscosity, however, may not be the only issue. Perhaps, since the higher solid content primers do not contain as much water (as low solids do) they do not help foster the hydrogen bonding that is important in interfacial attraction between polar adhesives and the hemicellulose and cellulose of wood. Because of these results, 3 percent and 2 percent were identified as optimum solids contents for MUF primers.

Experiment 3

In experiment 3, MUF primers were replicated in an effort to obtain reproducible results. Additionally, MME primers were made and tested for bond durability. All primer solutions were catalyzed with an acid load of 10 percent w/w of the solid polymer (as detailed earlier).

Figure 9 shows that 3 percent and 2 percent MUF primers again gave high wet wood failure and outperformed the control sample in the process. Thus, it is evident that MUF can be used effectively as a priming agent for wood to increase wet wood strength of epoxy bonds. **Figure 10** shows the same control in comparison with the MME priming agent. All samples, both dry and wet, of

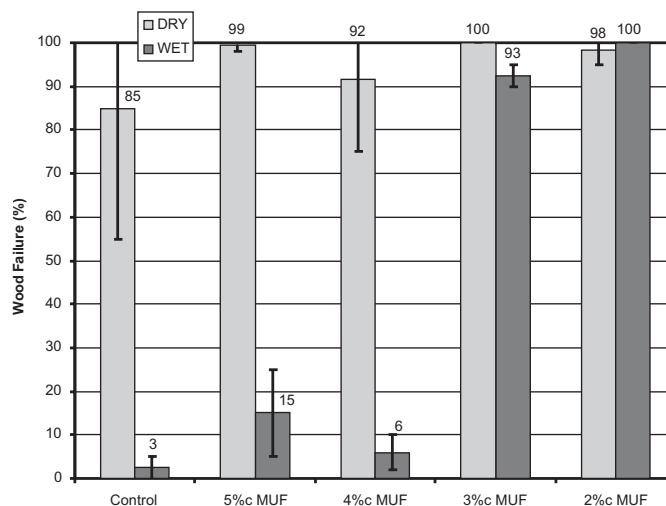


Figure 8. ~ Wood failure of control vs. catalyzed MUF primer.

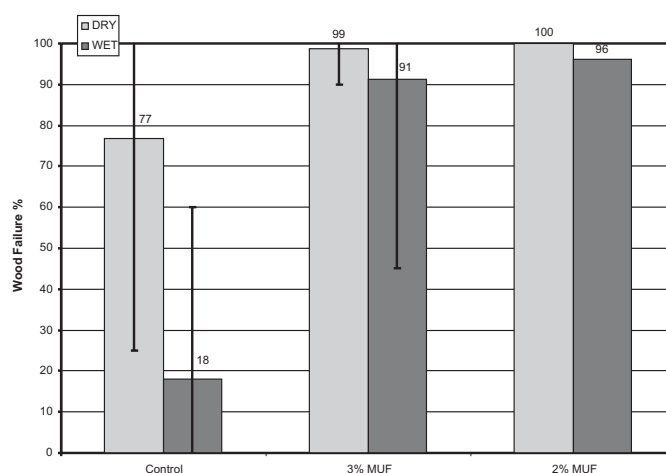


Figure 9. ~ Wood failure of MUF.

these primers tested showed more or less full wood failure. It, too, is an extremely effective priming agent.

The MME primers appear to perform better than MUF primers, however, the similarity or difference of the chemical structures of MME and MUF is unclear. One reason might be that MME rather does not contain any condensed structures whereas the MUF batches have been synthesized to a certain haze point and water dilubility; hence the MUF contain condensed structures which might be hindered or even excluded from penetrating into the cell wall. In addition, the efficacy of the different catalysts used for these primers is uncertain. These and other questions will hopefully be answered with future work.

Conclusion

MUF is a good priming agent for wood. It increased the wet-wood strength of epoxy bonds, which have been char-

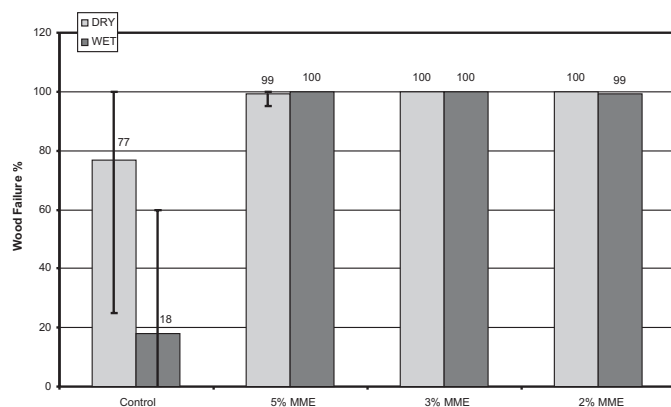


Figure 10. ~ Wood failure of MME.

acteristically weak. This treatment did not chemically deteriorate the wood as can be evidenced by the compression shear strength values in **Table 1** from Experiment 2. Although all MUF primers (except 4% dry) show a higher strength values, they do not differ too much from the control sample. Note that at 100 percent wood failure you are not testing the shear strength of the bondline but only the strength of the solid wood.

The wet wood failure increased by an order of magnitude when changing from wet epoxy adhesive bonds (control) to primed epoxy bonded surfaces. However, research indicates that primers with solid contents greater than 5 percent do not give good bonds, probably due to too much coating of the wood. They are coating the surface of the wood cell wall creating a thick layer of primer on the secondary and primary walls. Additionally, the epoxy adhesive cannot bond well with the MUF primer and gives way because of a weak adhesive bond. Another plausible explanation is that since the higher solid content primers do not contain as much water (as low solids do) they do not help foster the hydrogen bonding that is important in interfacial attraction between polar adhesives and the hemicellulose and cellulose of wood. Since MUF primers at 3 percent and 2 percent solids performed well, it is desirable to look deeper into the chemical make-up of the primer and further substantiate the cell wall stabilization theory of increasing wet wood strength. By utilizing the functional groups of melamine for characterization tests, we hope to obtain suitable information on this topic.

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Table 1. ~ Dry and wet compression shear strength values of MUF priming agents vs. control.

Sample	Dry	Wet
----- (MPa) -----		
Control	14.21	5.07
5%	14.68	5.54
4%	13.33	5.48
3%	15.38	5.78
2%	14.99	5.37

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Charles R. Frihart

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Forest Products Society
2801 Marshall Court
Madison, WI 53705-2295
phone: 608-231-1361
fax: 608-231-2152
www.forestprod.org

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