High heat resistance of adhesive bonds to wood and aluminum

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Abstract: Wood is preferred as a building material due to its high strength, low weight, and ability to retain many of its properties even at moderately high temperatures. However, numerous wood adhesives lose strength as the temperature increases, especially around 200°C. This study examined bond strength at temperatures above that required for initiation of wood degradation for bonds of wood-to-wood and wood-to-aluminum using two oil-based adhesives (resole phenolics and epoxies) and two bio-based adhesives (soy protein isolate and ovalbumin from egg whites). The phenolics not only gave the best strength in the wood-to-wood bonding, but also in the wood-to-aluminum bonding. The second best were the protein adhesives, and the epoxies were the weakest. This research reinforces the use of phenolic adhesives for wood exposed to high temperatures.

Keywords: Wood bonding; High temperature; Phenolic; Protein; Epoxy; Aluminum.

Introduction

For efficient use of wood resources, many wood manufacturers bond smaller wood elements (veneer, strands, particles, and fibers) to make useful larger wood assemblies (composites, various types of beams, and cross-laminated timber) (Frihart and Hunt 2010). Composite wood products need to resist bond failures to maintain structural integrity during extreme events for a sufficient time to allow people to escape injury or death. Heat resistance of wood bonds has been an ongoing issue (Yeh and Brooks 2006), whether it is the creep of structural bonds in hot and humid locations (ASTM 2011) or fire resistance (ANSI/APA PRG 320). For nearly half a century, the adhesives used in laminated wood products such as structural glulam were primarily phenol resorcinol adhesives that maintained bond integrity even when subjected to moisture and fire. The proliferation of mass timber products such as cross-laminated timber (CLT) over the last two decades, however, has resulted in the development of new adhesive formulations that facilitate the production of large-scale structural wood panels. The introduction of these new adhesive formulations has led to the development of new timber product standards and fire testing procedures. Recent research has focused on large-scale tests on thicker wood members (Zelinka et al. 2018), and the ANSI/APA PRG 320: Standard for Performance-Rated Cross-Laminated Timber standard requires full-scale fire qualification testing (ANSI/APA 2018). Because qualification testing at full scale is costly, Miyamoto et al. (2021) and Zelinka et al. (2020a) have demonstrated the validity of small-scale testing for precursory evaluation of adhesive performance at elevated temperatures.

One area not investigated is the resistance to elevated temperature of bonded thin veneers. Thin veneers are bonded into

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products such as laminated veneer lumber (LVL) and plywood, both of which are produced at many scales, including mass timber panels that are comparable to the sizes of CLT. We used an economic small-scale apparatus (ASTM 2020) to address this gap in the literature by testing the strength of both bio-based and synthetic adhesives at temperatures above where wood starts to degrade. The adhesives were evaluated to determine if the thermal degradation of the wood caused the adhesive to de-bond or if the adhesives prevented the veneers peeling off prior to complete degradation of the veneer layer (Poletto et al. 2012). We also tested the wood bonding to aluminum under the same conditions, because aluminum is used in furniture, sporting equipment, and vehicular components. Especially in sporting and transportation applications, laminates need to be lightweight, which adds additional performance criteria for the adhesive. Bonding lightweight aluminum alloys to wood veneers with high strength-to-weight ratios is desirable, yet challenging because of material dissimilarities in porosity, thermal expansion, stiffness, moisture content, and chemical composition, among many other factors that potentially affect bond quality.

Aluminum-to-wood bonding is an area of particular concern with phenolics, which do not normally adhere to the metal platens in the pressing of composites. Historically, aluminum-to-wood bonding was investigated by the USDA Forest Service Forest Products Laboratory (1964) at a time when aircraft structures were made primarily from timber materials. Eickner et al. (1955) directly bonded 5-ply, birch-veneer plywood to aluminum alloy using several different adhesives and processes and tested the lap joints via 10 million cycles of prying to assess fatigue resistance. Black and Blomquist (1959) investigated the thermal degradation of various adhesives bonded to aluminum and stainless steel. Since these studies, new formulations and testing methods have been developed, which led to this current comparison of phenolic, epoxy, and bio-sourced adhesives.

The need to withstand high temperatures (such as 250°C) drastically limits the types of adhesives that can be used and eliminates adhesive types known for their mechanical or chemical thermal instability at higher temperatures: most polyurethanes, melamine-urea-formaldehyde, polyvinyls, and hydrocarbons (Liu et al. 2020; Zelinka et al. 2020b; and Miyamoto et al. 2021). This narrowed down the selection to three types that are known to have good heat resistance: phenolics, proteins, and epoxies (Frihart 2013). Phenolics are widely used in structural wood bonding because of their durability and heat tolerance. Proteins are not widely used in wood bonding mainly because of their limited moisture resistance and cost; however, they have long been used in wood fire door applications. Epoxies are not normally used in wood bonding, except in repairing delaminated wood beams and repairing spots of wood decay. Their advantage is that they are gap filling, and some formulations can bond at room temperature, but outside of wood applications, they are widely used for their durability and heat resistance.

Therefore, the objective of this study was to test the strength of both bio-based and synthetic adhesives at temperatures above where wood starts to degrade with wood-to-wood and wood-to-aluminum bonding.

Materials and methods

Specific adhesives were selected for minimal wood void filling to keep the bonded product weight low (Table 1). Glass Bubbles K15, which are hollow spheres of glass of 60 µ, were obtained from 3M^{TM} (Minneapolis, MN) and added to the adhesives to reduce weight and density at 5%, 10%, or 20% of total solids.

The resoles and epoxies were used as received. The soy protein isolate (SPI, PRO-FAM^{TM} 974) was from ADM (Decatur, IL), and the egg white (dried egg whites, H-40) was from Ballas Egg Products Corp. (Zanesville, OH). The protein adhesives were prepared by dissolving them in water at the concentrations specified and stirring until they were well mixed. The resoles were proprietary commercial aqueous PF adhesives with minimal wood penetration obtained from a major wood adhesive producer, stored at 5°C, and used within a month, according to the manufacturer's instructions. The epoxies were all selected because they were recommended as heat-resistant adhesives or had been previously tested for good wood bonds, and they were mixed with curing agents according to the manufacturers' instructions. Because the different types of adhesives interact differently with the wood surface and cure differently, the bonding conditions are discussed in the results and discussion section.

Veneers from five wood species, bigtooth aspen (*Populus grandidentata)*, yellow birch (*Betula alleghaniensis*), cherry (*Prunus* spp.), red maple (*Acer rubrum*), and yellow poplar (*Liriodendron tulipifera*) were obtained from Great Lakes Veneer (Marion, WI). The veneers were cut into 117 mm × 20 mm × 0.8 mm thick pieces. Aluminum pieces of 117 × 20 × 0.8 mm were cut from sheet stock. Half of the aluminum specimens were lightly sanded with medium grit sandpaper. To make a specimen, 5–10 mg of an adhesive was applied to the end of a piece of veneer and a second piece of veneer was

Table 1. List of adhesives evaluated for bonding wood and aluminum ABES samples.

Adhesive	Adhesive type and label	Details
PF 1	Resole phenol-formaldehyde	Concentration of 43.1% solids obtained from a commercial phenolic producer (Aqueous)
PF 2	Resole phenol-formaldehyde	Concentration of 42.3% solids obtained from a commercial phenolic producer (Aqueous)
	Epoxy 805	Aremco Products Inc (Valley Cottage, NY)
	Epoxy 526N	Aremco Products Inc (Valley Cottage, NY)
	Epoxy 2335	Aremco Products Inc (Valley Cottage, NY)
	EPON 162	Hexion (Houston, TX)
	EPON 862	Hexion (Houston, TX)
	D.E.R. 331	OLIN (Clayton, MO)
SPI 974	PRO-FAM™ 974	Soy protein isolate (SPI) from Archer Daniels Midland Company (Decatur, IL)
H-40	Egg white H-40	Ballas Egg Products (Columbus, OH)

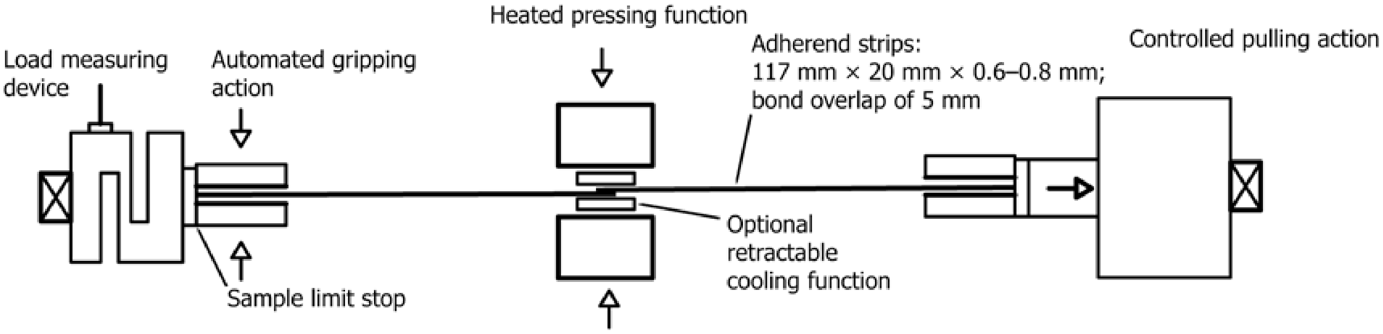

Figure 1. Schematic of the bonding and ABES testing system.

placed over the adhesive, resulting in a 5-mm overlap (Figure 1). The specimens were bonded at 120°C or 150°C for 120 s at 0.2 MPa using the ABES method (ASTM 2020) and tested the next day to ensure that the adhesives were fully cured. The ABES (Automatic Bond Evaluation System) from Adhesive Evaluation Systems, Inc. (Corvallis, OR) is an apparatus that can bond small samples and test them in a relatively short time.

The ABES test method was used for several reasons. The thin veneer ensured rapid heat transfer to the bondline both in the bonding step and 250°C heat treatment for 100 s. Good temperature and time control ensured repeatability in the bonding and heat treatment steps. The platens attain the higher temperatures for testing the heat resistance of the bond and the wood is degraded only near the bonded area. As soon as the platens are retracted, the tensile force is applied, with the bond still at the elevated temperature, and the force required to break the bond is measured in Newtons at the factory setting of 15 on the pull rate control (no unit specified). Using a smooth veneer ensures consistent testing of the bondline strength. The ABES instrument as described in ASTM D-7998-19R24 (ASTM 2024) is illustrated in Figure 1, and the procedure is described in detail (Frihart and Lorenz 2020). The small mass of wood and large mass of the platens assured that the bondline reached the set temperature quickly, as 0.2 MPa pressure was applied to the bondline. Five replicates were run for each test condition, and the mean and 95% confidence interval were calculated for each condition. The ABES is only a screening tool for eliminating the poor performers and not for setting the final adhesive formulations for production of full-scale laminates.

Results and discussion

Four important properties of the adhesives considered are that they need to bond wood laminates with minimum penetration, produce heat resistance bonds to 250°C, adequately bond with added glass bubbles to reduce the density and amount of adhesive used, and bond to aluminum. Wood is a porous material; thus, penetration of the adhesive is required to create strong bonds through mechanical interlock. In making lightweight products, a strong surface bond with minimum penetration is required, since the adhesive is much denser than the displaced air in the wood voids. Wood penetration can be judged by visual and microscopic examination of crosscut samples (Hare and Kutscha 1974). In this study, whether the bond failed because of wood or adhesive failure was not determined, because the surface area of the bond in ABES testing is too small to identify and measure a definitive mode of failure.

Because the ABES data below are for screening purposes for the best wood species and adhesive formulation, the statistical differences were not examined by analysis of variance (ANOVA) and post-hoc tests. Instead, adhesive and veneer selections were made only when there were large and consistent differences apparent in graphs comparing the mean and error bar plots of bond strength at elevated temperatures. Although bond strength is critical, other factors like low adhesive application rates, resistance to peeling of thin veneers on heating, and availability of a consistent wood source were considered. Generally, bonds that exhibited the least change in strength at elevated temperatures, relative to room temperature, were considered top performers.

Phenol-formaldehyde adhesives

Phenol-formaldehyde (PF) adhesives are among the strongest and most durable wood adhesives that are often formulated as low molecular weight oligomers to improve penetration of the cell lumens and the cell walls for more water durable bonds (Frihart 2009). The two proprietary commercial aqueous resole PF adhesives (labelled PF 1 and PF 2) were bonded at 120°C using all five wood species and, in some cases, with glass bubbles added to reduce the adhesive density. The strengths at 21°C and after heating for 100 s at 250°C, without and with 5% glass bubbles, are provided in Figure 2. The strengths were similar for all the veneers, except for the cherry at 250°C. Even though the high temperature tests were above the temperature for the initiation of wood decomposition, there was no delamination in any of the samples at 250°C during the short hot-testing conditions. The 250°C test results were generally significantly lower than the 21°C test results, without and with the addition of glass bubbles, but these differences did not exist in every case. Significant differences were considered for conditions showing non-overlapping 95% confidence interval bars. The greater differences in the temperature effect on the cherry compared to the other species could indicate a greater heat sensitivity of this species.

Wood is often used because it is strong for its weight, but the adhesive has a higher density than the wood. Thus, low density glass bubbles were added to the PF 1 adhesive at 10% and 20%, in contrast to the 5% added previously. The bond shear strength decreased somewhat, and with 20% glass bubbles, this PF was very thick and difficult to spread on the wood. There was no delamination at 250°C, but the glass bubbles did not show any reinforcing effect leading to higher strength (Figure 3). At this point, the yellow poplar was selected based on its relative low density, greater uniformity, and high strength as the wood species to use beyond the preliminary test with each adhesive.

PF 1 (43.1% solids) was used to bond yellow poplar at 150°C, which is a more optimum temperature for bonding the PF adhesives (according to the manufacturer's guidelines), to compare with the samples bonded at 120°C. The 120°C was considered less promising for some applications due to it being difficult to attain in some pressing processes, especially for thicker plywood, which needs higher platen temperatures for all the bonds to reach this temperature. In Figure 4, the bond strengths tested at 21°C were 40% higher when bonded at 150°C, although the strengths tested at 250°C did not increase.

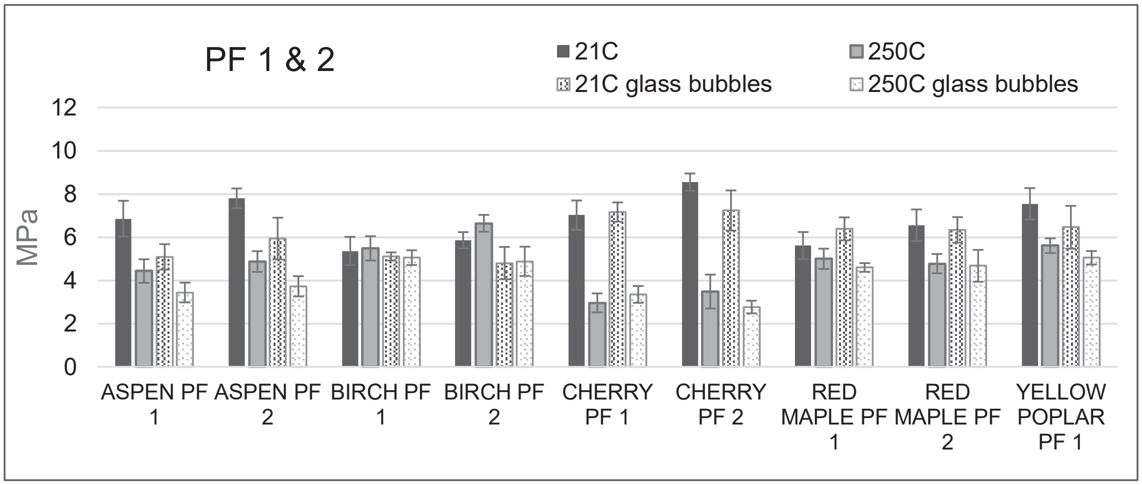

Figure 2. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of phenolics on five different wood species, without or with 5% glass bubbles, bonded at 120°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

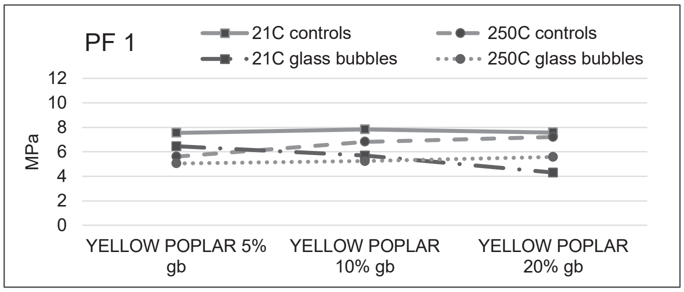

Figure 3. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of PF 1 with 5%, 10%, or 20% glass bubbles (gb) added, on yellow poplar bonded at 120°C for 120 s using ABES.

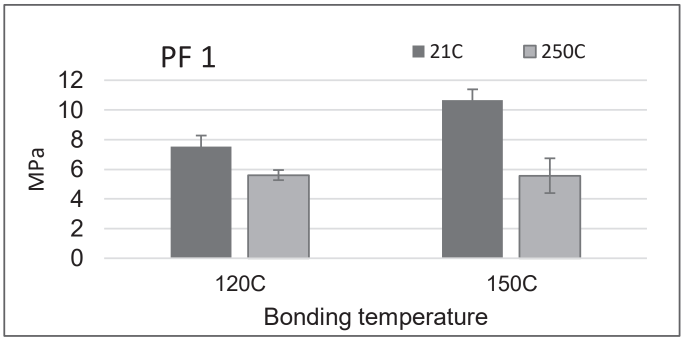

Figure 4. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of a phenolic on yellow poplar bonded at 120°C and 150°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

Based on the results, we supposed that the adhesive is partially cured at 120°C and fully cured at 150°C, before reaching the 250°C test condition.

Proteins

In addition to the commercial phenolics that did very well as high temperature adhesives in the ABES tests, protein and epoxy adhesives were also studied. Protein adhesives were widely used for wood product assembly, including in fire door applications that continue today, until the introduction of synthetic adhesives that have better water resistance gained broader industry acceptance. Soybean proteins were the main commercial adhesives, but the ovalbumin from egg whites is also a good wood adhesive (Frihart and Lorenz 2018; Lorenz and Frihart 2023). Of the three adhesive types considered, the only fully bio-based option is protein adhesives. These adhesives come as powders and are dispersed in water at room temperature just prior to use.

A commercial soy protein isolate (SPI) was tested at 20% solids, which was a thick paste. As shown in Figure 5, the bond shear strengths at both 21°C and 250°C were similar for all the veneers, without and with 5% glass bubbles (based on the weight of soy solids). There was no delamination at 250°C of the SPI bonds, but their higher temperature bond strengths were considerably lower than those for the PF adhesives. The lower strength at higher temperatures may be due to less adhesive in the glueline (adhesive layer between the wood surfaces), since the solids content of the SPI adhesive was dramatically lower than the PF adhesive, at 20% versus 43%, respectively.

An egg white adhesive was tested at 50% solids, which was a thick liquid, but still pourable. In Figure 6, the bond strengths at both 21°C and 250°C were similar for all the veneers (except for the cherry), without and with 5% glass bubbles (based on the weight of egg white solids). There was no delamination at 250°C of the egg white bonds, and their 250°C bond strengths were above those with SPI and close to those with the PF adhesives, with cherry again providing lower strength.

Because of the low viscosity of the egg white dispersion, compared to the soy protein, glass bubbles were added to the egg

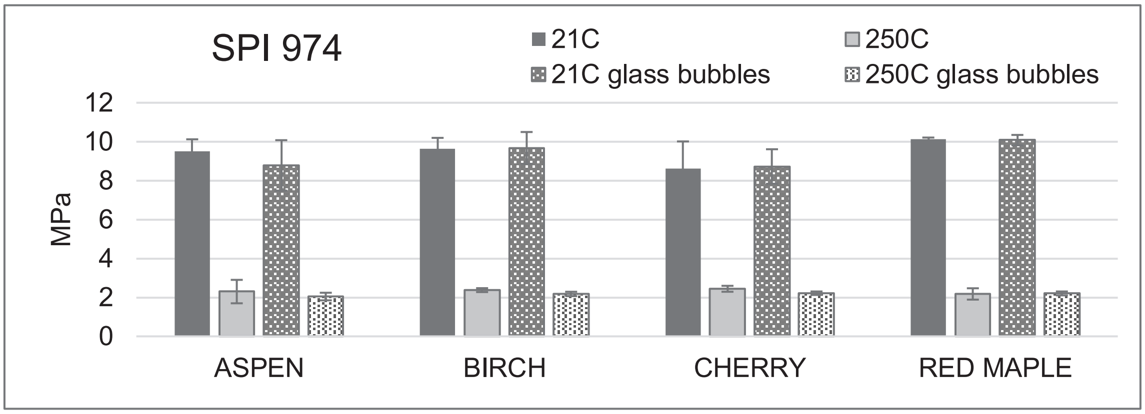

Figure 5. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of soy protein isolate on four different wood species, without or with 5% glass bubbles, bonded at 120°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

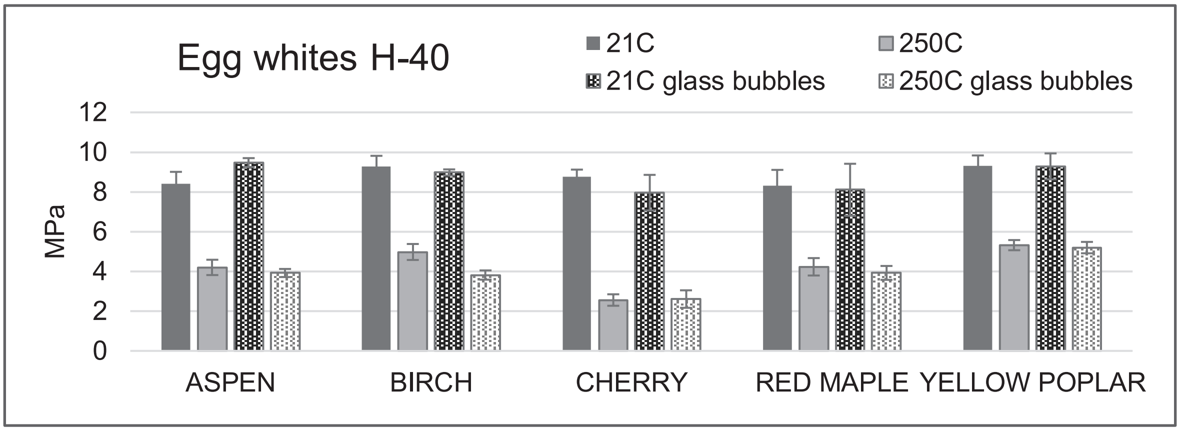

Figure 6. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of egg whites on five different wood species, without or with 5% glass bubbles, bonded at 120°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

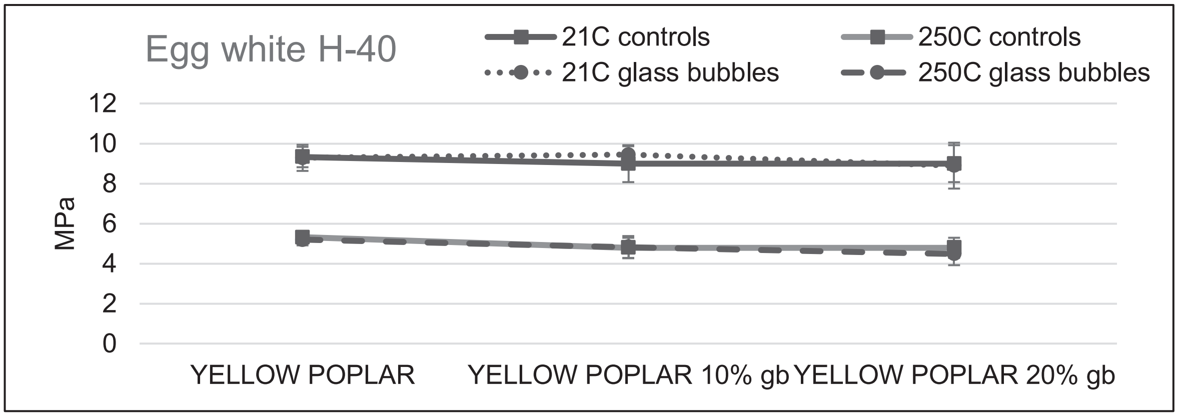

Figure 7. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of egg white adhesive with high amounts of glass bubbles (gb) on yellow poplar bonded at 120°C for 120 s using ABES.

white adhesive at 10% and 20% to compare with 5% added previously. The bond strength was about the same (Figure 7). With 20% glass bubbles the egg white was very thick and difficult to spread on the wood. There was no delamination at 250°C.

Although the protein adhesives performed sufficiently well in this application, the bonds at 250°C were lower than those of the phenolics. However, they could be suitable, with egg whites as the best option, if a bio-based adhesive was a necessary requirement.

Epoxy adhesives

Epoxies are well known as heat resistant adhesives and are widely used for bonds involving metals, composites, and other substrates involving high temperature applications. However, their use in wood bonding has been limited to specialty applications, such as bonding of wooden boats and aircraft, as well as for the repair of degraded, bonded wood products, because of their room temperature-curing and gap filling properties. An advantage of the epoxies is that they are usually not solvent- or water-borne; thus, there is no need to remove these liquids during the bonding process.

Most of the heat-resistant epoxies require bonding conditions at higher temperatures than are useful for wood and produce heavier weight gluelines than preferred. Of potentially useful epoxy adhesives, the most suitable were 805, 526N, and 2335 from AREMCO; these were tested as received and mixed according to the manufacturer's recommended ratios. The recommended cure schedules involving several hours at two different temperatures were not practical for the required veneer assembly and testing with ABES in 120 s, so bonding was tested at the recommended maximum temperature and adjusted depending on whether there was adequate bonding. Because of the porosity of wood, the 24 h at room temperature step was not used to avoid selective migration of adhesive components into the wood of the more polar curing agent, which would have resulted in the wrong ratio for curing. The bond strengths at both 21°C and 250°C were similar for all the veneers, without and with 5% glass bubbles (based on the weight of the epoxy), as shown in Figure 8. There was no delamination at 250°C with any of the epoxies. At this point, the cherry had been dropped due to its poor performance with the other adhesives.

The recommended epoxy 805 cure schedule is room temperature for 24 h and then 93°C for 2 h. The bonding temperature had to be increased to 120°C for adequate bonding in 120 s. With 5% glass bubbles added, the adhesive became difficult to stir in about 20 minutes, and almost impossible to spread on the wood, which is why this data is not available for the last samples bonded, the yellow poplar and red maple.

The recommended epoxy 526N cure schedule is room temperature for 2 h and then 93°C for 2 h, cool to room temperature, then 163°C for 2 h. The bonding temperature had to be increased to 178°C for adequate bonding in 120 s. With 5% glass bubbles added, the adhesive became difficult to stir and spread on the wood after about 2 h. This adhesive provided adequate bond strength at 250°C, without and with glass bubbles (Figure 9).

The recommended epoxy 2335 cure schedule is 93°C for 2 h plus 177°C for 2 h. Bonding at 177°C resulted in good bonding in 120 s (Figure 10). With 5% glass bubbles added, the adhesive became difficult to stir and almost impossible to spread on the wood after about 2 h, which is why the data is incomplete for the aspen, the last samples to be bonded.

The epoxies above were selected because of their recommended heat resistance, but are not usually used for wood adhesion. Three additional epoxies, typically used for wood adhesion in marine and aircraft applications, were tested. The three epoxy adhesives (EPON 162 and 862 from Hexion, and

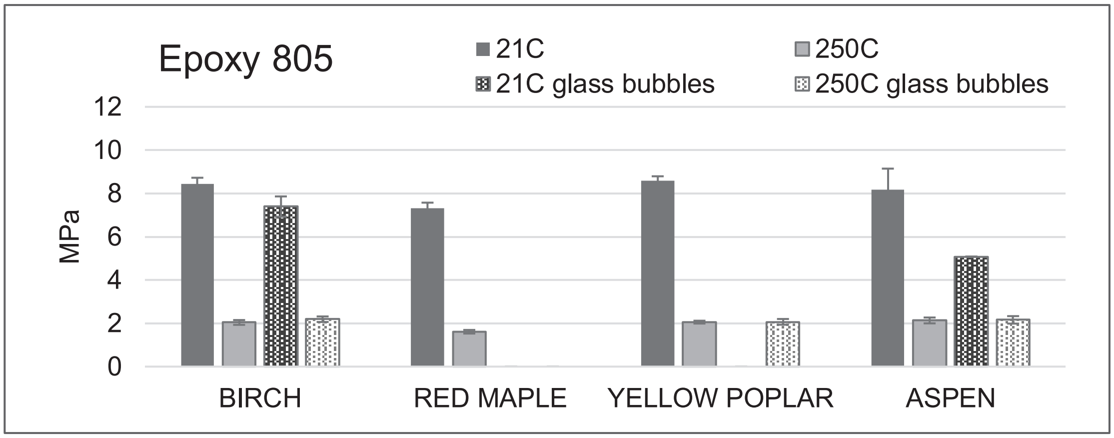

Figure 8. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of epoxy 805, without or with 5% glass bubbles, on four different wood species bonded at 120°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

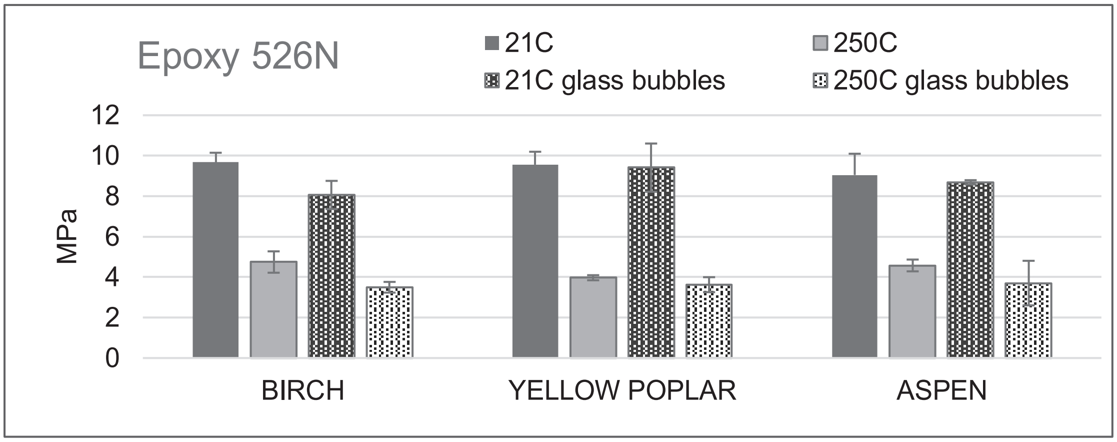

Figure 9. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of epoxy 526N, without or with 5% glass bubbles, on three different wood species bonded at 178°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

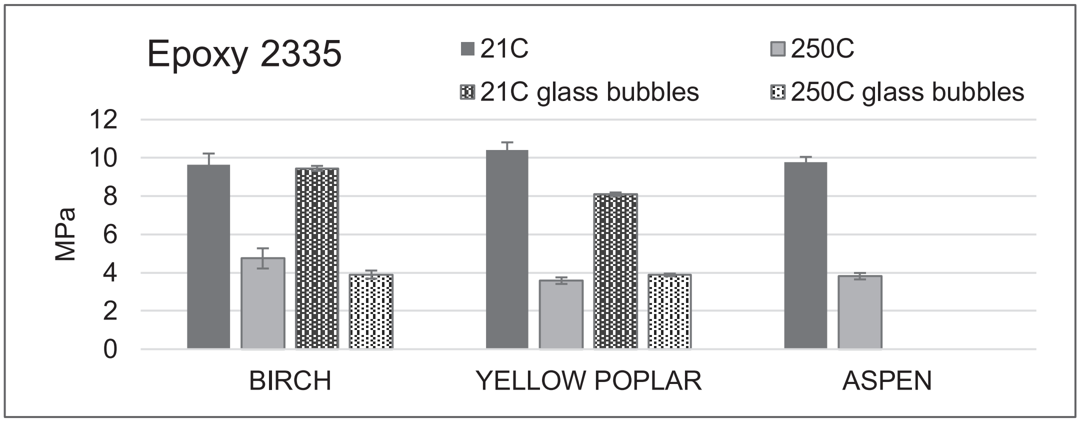

Figure 10. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of epoxy 2335, without or with 5% glass bubbles, on three different wood species bonded at 177°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

D.E.R. 331 from Olin) were tested as received and mixed according to the recommended ratios with two different curing agents (Epikure W from Hexion and D.E.H. 24 from Olin). The Epikure W did not result in adequate bonding of yellow poplar until the temperature was increased to 200°C. All three epoxies had good strengths with D.E.H. 24 when bonded at 100°C for 120 s on yellow poplar. The strengths at both 21°C and 250°C were similar for all three epoxies, and better than or similar to, the epoxies tested previously (Figure 11). There was no delamination at 250°C with any of the epoxies.

Bonding to aluminum

Although the main part of the study was to bond wood-to-wood, there may be some applications that require wood-to-aluminum bonding. The aluminum was bonded to yellow poplar, since it gave good bonding results and was the least dense wood species, which helps to provide low weight composites. Yellow poplar was bonded to untreated and treated (lightly sanded) aluminum at 160°C for 120 s with one of the phenolics. Sanding the aluminum did not increase the bond strength over the untreated aluminum (Figure 12). The aluminum was bonded to the wood at 160°C instead of 150°C, because the bonds were stronger than at 150°C. The epoxies Olin 331 and EPON 162 did not bond to the aluminum at 100°C, the temperature used in bonding the wood.

The wood-to-aluminum bonds with the phenolic were surprisingly strong and as good as the wood-to-wood bonds, even at high temperatures. Thus, the same phenolic could be used for bonding all the substrates.

Conclusions

The results of this evaluation of adhesive wood bonds at temperatures above where wood generally starts to lose strength

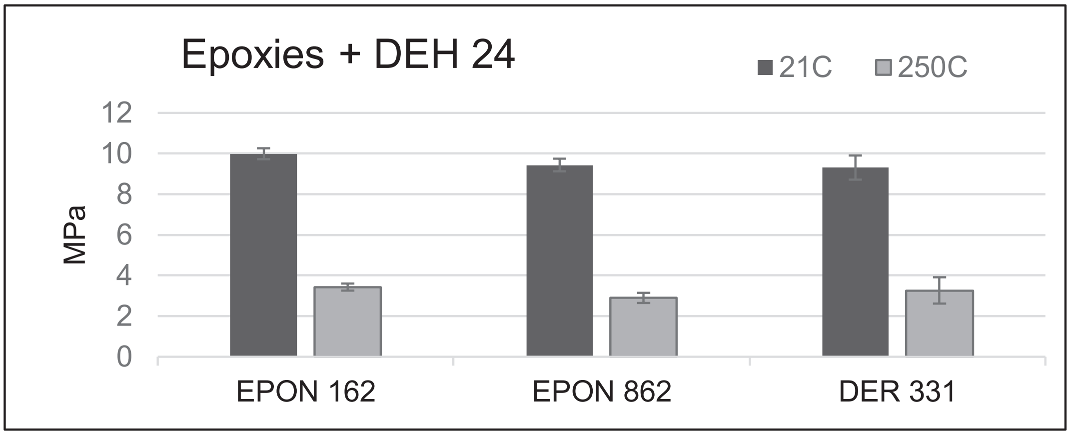

Figure 11. Room temperature (21°C) and high temperature (after 250°C heating for 100 s) bond shear strength of epoxies on yellow poplar bonded at 100°C for 120 s using ABES. The values are the average of five replicates, and the error bars represent one standard deviation.

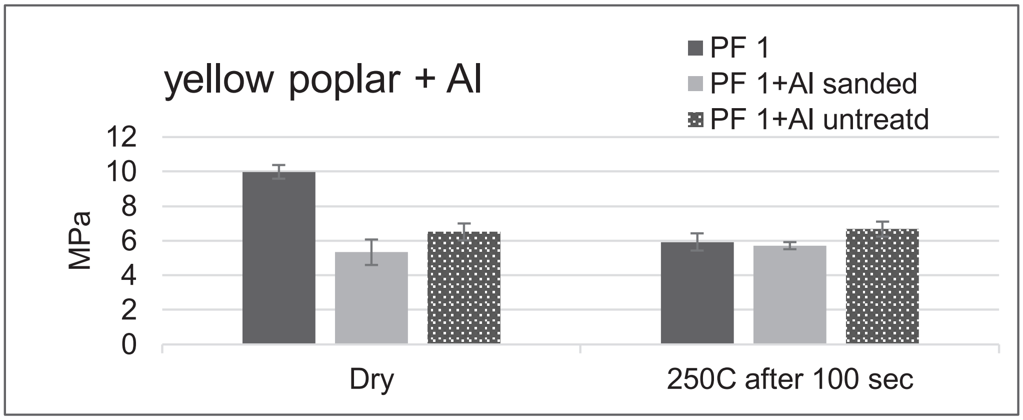

Figure 12. Yellow poplar was bonded to yellow poplar and aluminum (sanded and untreated) at 160°C for 120 s and tested at room temperature (21°C) and high temperature (after 250°C heating for 100 s). The values are the average of five replicates, and the error bars represent one standard deviation.

show that it is possible to make wood products with thin veneers and maintain bond integrity even at elevated temperatures. The lack of delamination indicated that the degradation of the wood would be the controlling factor for good product performance and that the thin laminates can replace solid wood whose properties are better understood. In addition, except for thickening of the adhesives, the glass bubbles generally decreased the 21°C bond shear strength, but had less effect on the 250°C bond test. The PF and egg white adhesives had the best shear strengths, without or with 5% glass bubbles, on the yellow poplar veneers. See Table 2 for the yellow poplar results, which was the preferred wood species due to its lower density and more uniform structure.

With the superior shear strength at 250°C and ease of use by many bonding methods, the PF1 adhesive provided the best results for producing and testing laminates on a larger scale. The proteins had good strengths, but they would require the bonding facility to prepare the adhesives, in contrast to the ready to use PFs. The epoxies were considered the least favorable of the adhesives, based on their lower strengths, thicker gluelines, and the difficulty in spreading them on the wood.

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Table 2. Summary of adhesive shear strengths on yellow poplar.

	ABES shear strength in Pa without glass bubbles		ABES shear strength in Pa with 5% glass bubbles			
Adhesive	21°C	250°C	21°C	250°C	Comments	
PF 1 43.1% 120°C	754.3	560.6	645.7	504.5	Easy to use	
PF 2 42.3% 150°C	1066.6	556.5	NT	NT	Easy to use	
SPI 974 20%	1027.9	241.7	896.7	269.7	Easy to use	Thick paste
Egg whites H-40 50%	932.3	531.9	928.2	519.5	Easy to use	Pourable
Epoxy 805	858.6	205.5	NT	206.8	Very thick	With glass bubbles difficult
Epoxy 2335	1039.6	358.2	810.1	388.2	Very thick	With glass bubbles difficult
EPON 162	999.2	343.1	NT	NT	Very thick	With glass bubbles impossible
EPON 862	943.9	289.8	NT	NT	Very thick	With glass bubbles impossible
D.E.R. 331	931.3	326.6	NT	NT	Very thick	With glass bubbles impossible

NT = not tested

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