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# Durability of the adhesive bond in cross-laminated northern hardwoods and softwoods

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ABSTRACT

In this study, we investigated the durability of adhesive bonds in the cross-laminated lumber of seven hardwood and two softwood species from the Great Lakes region. The 2-layered cross-laminations were glued using phenol resorcinol- and melamine-based structural adhesives. A total of 720 cross-laminated wood blocks were tested for delamination by exposing the samples to cyclic (wet-dry) conditions. Distribution of the adhesive on the bondlines was also studied to understand the effect of adhesive penetration on bond durability. The results indicated that mixed hardwood cross-laminations generally produced better bonds than single hardwood species cross-laminations. Hardwood and softwood hybrid cross-laminations were found to have better bond durability in dry-wet cycles. A high failure rate ($\geq$ 50%) was found in the following single species cross-laminations: aspen, white ash, white pine, and yellow birch. Similarly, several mixed species cross-laminations resulted in a delamination rate of 50% and higher, which raises caution in their use in CLT manufacturing. In addition, the viscosity of the adhesive influences the maximum depth of penetration, which tends to affect the durability of adhesive bonds.

## 1. Introduction

Interest in using hardwood species for advanced structural materials has grwon in the U.S. and globally because of an anticipated increase in hardwood timber resources in the near future. Hardwoods are known for superior strength properties associated with their relatively higher mean density values compared to most of the softwoods. Therefore, incorporation of hardwood species with higher mechanical properties for structure reinforcement, where locally or globally high strength performance is required, has been considered for solid wood-based structural products, such as glulam and cross-laminated timber (CLT). However, the adhesive bond durability of harwood-based structural materials has not been studied as extensively as their softwood counterparts. To ensure the material integrity of mixed-species structural products, the bonding properties, including strength and durability, require further study.

However, due to the complexity of hardwood anatomical features and surface chemistry, bonding properties of hardwoods are more complicated than those of softwoods [1,2]. For example, a greater presence of extractives in hardwoods can interfere with available bonding sites on the wood surface, resulting in poor bond in a bonding assembly. Adhesive penetration is highly influenced by vessel/pore diameter and length that are more variable in hardwoods [2]. Higher density of hardwoods influence bond quality as the dimensional changes resulting from moisture absorption and loss can create stress on the bond line. At similar specific gravities, hardwoods shrink 20% more than softwoods since the lower lignin (hydrophobic) content of hardwoods corresponds to higher contents of the carbohydrates (i.e. cellulose and hemicellulose, both hydrophilic), resulting in increased dimensional changes [3].

Despite the bond challenges, most hardwoods with higher density could lead to the developemnt of next-generation high-performance hybrid CLT. Yelle and Stirgus [4] investigated the influence of anatomical, physical, and mechanical properties of diffuse-porous hardwoods (sugar maple, soft maple, and basswood) on moisture durability of bonded assemblies. They found that density and pore

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distribution played a significant role in how wood adhesive bonds perform under wet–dry cycle durability tests. They reported that an increase in specific gravity (SG) resulted in an increase in shear strength (sugar maple > soft maple > basswood), indicating a positive influence of wood density on hardwood bond performance. In addition, the tension strength perpendicular to the grain for hardwood were shown to reach up to 260% of the softwood strength values with hardwood admitting larger spans and smaller cross sections [5]. Hardwoods, such as aspen and birches, were found to exhibit a higher resistance to planar shear stresses than softwoods [6].

The durability and general adhesive bond performance between wood elements are influenced by the degree of penetration of the adhesive into the porous network of interconnected cells [12]. Therefore, understanding the fundamentals of adhesive penetration is very important in ensuring effective bonding [13–15]. To understand the bonding complexities and effectiveness of hardwood bonds, we need to understand how differences in wood microstructures may lead to different penetration behavior of specific adhesives [7–9]. Adhesive factors such as viscosity, solid content, pressure and temperature, coupled with the structural makeup of the interphase, its volume and shape dictate the magnitude of stress concentrations and bond performance [13–15]. The wood-adhesive bond can be considered as a symmetrical chain across the bond-line [2] where the bond is inferred as the weakest link in the chain [14]. The cross-link chain analogy can be separated into nine levels [8]. The first link is the pure adhesive phase excluding the wood substrate [2]. The second and third levels include the wood substrates and the adhesive boundary layer. The fourth and fifth links represent inter-phase regions of wood and adhesive where the union of the bonds (adhesion) takes place through covalent bonding mechanical interlocking or electrostatic force. All areas of modification to prepare for gluing are considered the sixth and seventh link, while the ninth region representing the unadulterated wood.

To understand the influence of adhesive penetration on bond quality, several studies have been conducted to develop procedures for measuring and monitoring adhesive penetration depth using various methods, including light microscopy [10], scanning thermal microscopy [22,23], florescence microscopy [24 17,25], and scanning electron microscopy (SEM) [26]. Youngquist and Murmanis [27] employed fluorescence microscopy (FM) and SEM to evaluate the surface of bonded wood materials before and after soak-dry treatments. Singh et al. [28] evaluated the wood-adhesive interface in a commercial plywood manufactured by gluing *Pinus radiata* wood plies with a phenol-formaldehyde adhesive using different microscopy technique including scanning electron microscope. The SEM helped in enhancement of differentiation between the adhesive and wood cell walls based on a special technique. There is a need to better understand the durability of adhesive bonds and the resulting performance of bonding mixed hardwood species in CLT applications. In this study, we investigated the adhesive bonding properties of several common hardwood and softwood species from the forest lands in the Great Lakes states, where many hardwood species have been underutilized.

The goal of this study was to assess the feasibility of using hardwood and mixed species from the Great Lakes region to manufacture structural grade CLT panels. The specific objectives were to 1) investigate the adhesive bond penetration of cross laminated mix hardwood species using the scanning electron microscope (SEM), 2) analyze the bond durability based on interactions of adhesives, SG and anatomical features of cross laminated mix species when subjected to cyclic delamination (wet-dry conditions), and 3) investigate the influence of anatomical features such as pore distribution and SG on adhesive penetration of cross-laminated bonds made of mixed species. This current study hypothesized that; the rate of adhesive penetration would affect the bond durability in a wet-dry condition.

## 2. Materials and methods

The species investigated in this study included seven hardwoods and two softwoods from the Great Lake regions. The hardwoods included sugar maple (*Acer saccharum* Marshall), red maple (*Acer rubrum* L.), northern red oak (*Quercus rubra* L.), white ash (*Fraxinus americana* L.), yellow birch (*Betula alleghaniensis* Britton), American basswood (*Tilia americana* L.), and quaking aspen (*Populus tremuloides* Michx); The softwoods were eastern white pine (*Pinus strobus* L.) and red pine (*Pinus resinosa* Aiton). Table 1 shows the species code, SG, and anatomical features of these species. The selected species have a range of anatomic features: diffuse porous (sugar maple, red maple, yellow birch, basswood, and aspen), ring porous (northern red oak and white ash), abrupt early/latewood transition (red pine) and gradual early/latewood transition (white pine). The SG of the seven hardwood species ranges from 0.38 to 0.62 [29]; the SG of red pine and eastern white pine are 0.46 and 0.35 respectively. White pine has the lowest SG (0.35) that has met the minimum SG requirement of ANSI/APA PRG 320 [39]. The minimum SG of 0.35 was set as the lower bound for the CLT connection design since it is the close to the lowest SG of commercially available wood species in North America including the western wood species in the United States and the northern species in Canada. The study selected several species that are abundant in the state of Michigan and while some are considered low-grade. There is strong interest to create new avenues for the use of these species especially in mass timber applications.

### 2.1. Material preparation

A total of 245 kiln-dried hardwood boards were procured from a local hardwood mill in South Range, MI, 35 boards for each of the seven species. The hardwood boards were graded as “Select and Better” based on the National Hardwood Lumber Association (NHLA) grading rules, with the dimensions of 38 mm (1.5 in.) in thickness, 152 to 305 mm (6 to 12 in.) in width, and 2.44 to 3.66 m (8 to 12 ft) in length. In addition, 25 kiln dried red pine and 25 kiln dried white pine boards were procured from a local wood dealer in Houghton, MI. The softwood boards were visually graded No. 2 common with the dimensions of 51 mm × 152 mm × 3.66 m (2×6 nominal × 12 ft long). The boards were first pre-surfaced to 32-mm thick using a planer, edge-trimmed using a jointer, and then

**Table 1**
The wood species used in this study.

| Species Category | Species | Species code | SG [a] | Anatomical feature |
|---|---|---|---|---|
| Hardwood | Sugar maple (*Acer saccharum* Marshall) | SM | 0.63 | Diffuse porous |
| | Red maple (*Acer rubrum* L.) | RM | 0.54 | Diffuse porous |
| | Northern red oak (*Quercus rubra* L.) | RO | 0.63 | Ring porous |
| | White ash (*Fraxinus americana* Buckley) | WA | 0.60 | Ring porous |
| | Yellow birch (*Betula alleghaniensis* L.) | YB | 0.62 | Diffuse porous |
| | Basswood (*Tilia americana* L.) | BW | 0.37 | Diffuse porous |
| | Quaking aspen (*Populus tremuloides* Michx.) | ASP | 0.38 | Diffuse porous |
| Softwood | Red pine (*Pinus resinosa* Aiton) | RP | 0.46 | Abrupt early/latewood transition |
| | Eastern white pine (*Pinus strobus* L.) | WP | 0.35 | Gradual early/latewood transition |

[a] Specific gravity is the average value of the wood species at 12% MC from Table 1A of Miles and Smith[29].

conditioned in an environmental chamber at 20°C and 65% relative humidity (RH) for at least 4 weeks to achieve a 12% equilibrium moisture content (EMC). The conditioned boards were then run through the planer again to reduce the thickness to 25 mm (1 in.) with the allowable variations of ± 0.2032 mm across the width and ± 0.3048 mm along the length. The planed boards were subsequently cut into short boards of 462 mm long.

### 2.2. Species combinations of two-layer cross-laminated billets

The focus of this study was to examine the species effect on bonding hardwood and mixed species for CLT manufacturing. Out of seven hardwood species and two softwood species, we designed four types of two-layer cross-laminated billets: 1) single species; 2) mixed hardwoods; 3) hybrid of hardwood and softwood ; and 4) mixed softwoods. The billet assembly contained 45 combinations as shown in Fig. 1. The single-species billets included 9 configurations, each for an individual species cross-laminations (RM-RM, ASP-ASP, RO-RO, YB-YB, BW-BW, SM-SM, WA-WA, WP-WP, RP-RP). The mixed hardwood billets included three groups: Group 1 had 10 configurations for two diffuse-porous species mixed (SM-RM, SM-BW, SM-YB, SM-ASP, RM-BW, RM-YB, RM-ASP, BW-YB, BW-ASP, and ASP-YB); Group 2 had 10 configurations for diffuse-porous and ring-porous mixed species (RO-YB, RO-SM, RO-RM, RO-BW, RO-ASP, WA-YB, WA-SM, WA-RM, WA-BW, WA-ASP); and Group 3 just had one configuration for two ring-porous species mixed: WA-RO. Three groups together resulted in a total of 21 different mixed hardwood cross-laminated billets. The hardwood-softwood hybrid billets included two groups: Group 1 for the combinations of red pine (abrupt early/latewood transition) with individual hardwood species (diffuse/ring porous), resulting 7 configurations: RP-RO, RP-WA, RP-YB, RP-SM, RP-RM, RP-BW, and RP-ASP; Group 2 for the combinations of white pine (gradual early/latewood transition) with individual hardwood species (diffuse/ring porous), resulting in 7 configurations: WP-RO, WP-WA, WP-YB, WP-SM, WP-RM, WP-BW, and WP-ASP. A total of 14 species combinations were designed for the hybrid cross-laminated billets. The softwood billets just had one configuration: red pine with white pine (gradual and abrupt early/latewood transition).

### 2.3. Fabrication of two-layer cross-laminated billets

Two commercially available adhesive systems were used to bond the two-layer cross-laminated billets. The phenol resorcinol based adhesive system included CASCOPHEN® G-1131A, the resin and G113B, the hardener. The resin-hardener mixture ratio was 5:1 (by weight). The melamine base adhesive system included Cascomel™ 4720 resin and Wonderbond™ 5025A hardener. The resin-hardener mixture ratio was 4: 1 (by weight). Both adhesive systems have been used in manufacturing of CLT and glulam products. The high-density wood spread rate was 50 lbs per 1000 square feet and the less dense species were 60 lbs per 1000 square feet as indicated by the adhesive manufacturer.

A total of 270 two-layer cross-laminated billets were fabricated, 135 billets using phenol resorcinol adhesive and 135 billets using melamine base adhesive. We followed the manufacturer's instructions in the glue application and pressing the two-layer cross-laminated billets (Table 2). Both the shear blocks and delamination blocks were randomly cut from the 135 billets produced in each adhesive category consisting of 3 billets for each 45 combinations. The adhesive was applied using contact adhesive rollers per manufacturer's specified spread rate. The short boards were first edge-glued to form 462×234 mm plates within 24 h after the final planning to 25 mm. Each 462×234 mm plate was then cut into two 229×229 mm laminates. All the laminations were reconditioned before making the two-layer cross-laminated billets. The 229×229 mm billet was pressed under a pressure of 0.86 MPa for 8 h to cure the adhesive at

**Table 2**
CLT manufacturing parameters according to the manufacturer's recommendations.

| Description | Adhesives | |
|---|---|---|
| | Melamine formaldehyde | Phenol- resorcinol formaldehyde |
| Assembly time | 50 min | 45 min |
| Pot life | 3 h | 1 h |
| Curing temperature | 21°C | 21°C |
| Adhesive to hardener ratio | 5:1 | 4:1 |
| Amount of glue for one billet | 56.80 g | 56.80 g |
| Spread | both gluing surfaces | both gluing surface |
| Pressure | 0.862 MPa | 0.862 MPa |
| Total cure time | 24 h | 24 h |
| Clamp time | 8 h | 8 h |
| Maximum speed of mixer | 328 rpm | 328 rpm |

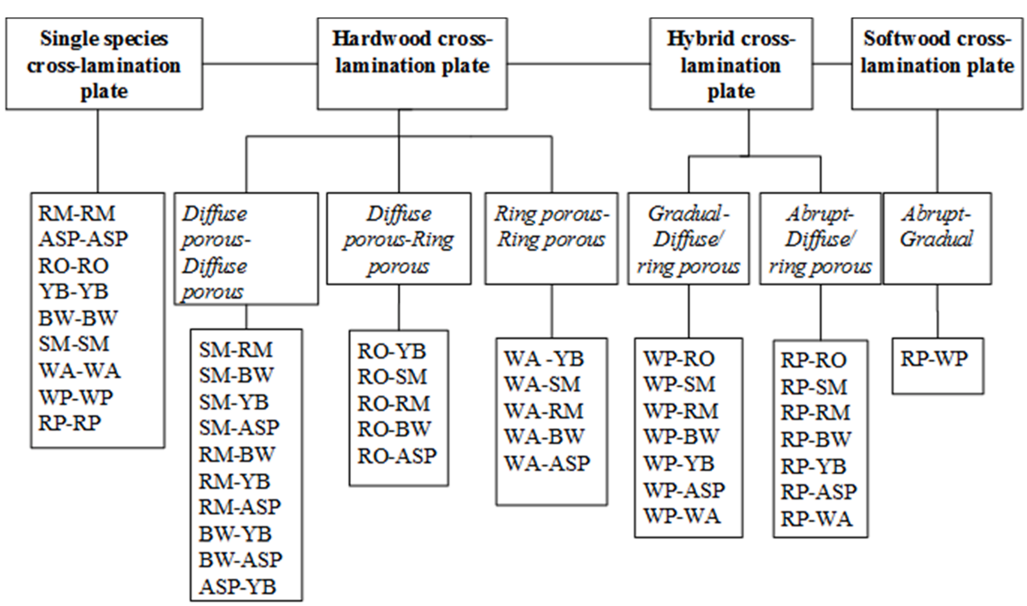


**Fig. 1.** Species combinations of the two-layer cross-laminated billets. Detailed description of the species is in Table 1.

21°C per the recommendations of the manufacturer for industrial applications.

### 2.4. Delamination test blocks

Based on the billet designs, the two-layer cross-laminated billets have 45 species configurations. For each configuration, eight delamination test blocks (51 mm × 76 mm) were cut from the billets, resulting in a total of 360 melamine test blocks and 360 test blocks for each type of adhesive. The delamination test blocks were reconditioned at 20°C temperature and relative humidity of 65% before testing.

### 2.5. Cyclic delamination test

The cyclic delamination test was conducted according to AITC Test T110-2007 (supplementary sheet 1) [30]. ASTM D2559 [31] was referenced for guideline and rules of acceptance of bond delamination. The delamination test blocks were exposed to several conditioning steps. The block specimens were weighed immediately to the nearest 1 g after removing from the conditioning room. They were then placed into a pressure vessel with the end grain surfaces freely exposed to water at a temperature of 18 to 27°C and at 51 mm apart. The specimens remained submerged throughout the cycling. Wire screens were used to separate test specimens. A vacuum of 84.81 kPa was drawn on the specimens for thirty minutes followed by a pressure of 517.11 ± 5 kPa for two hours. The specimens were then placed into the convection oven set at 71 ± 3°C for 10–15 h or until dried to 12–15% of the original weight (initial weight before wet-dry condition) according to the AITC standard. The specimens were arranged in the oven 51 mm apart from one another and oriented so that their end surfaces and glue lines were running parallel with the direction of the airflow. After 10 h of drying, a specimen from each species configuration was weighed hourly to determine whether the specimens had reached their target weights. Once the specimens reached target weight, they were removed from the ovens for bond line evaluation.

The bond lines of the test blocks were visually inspected for delamination, and the cycle test repeated for the specimens with delamination rate above 5%. Delamination is defined as the separation of layers in a laminate due to failure of adhesive, either in the adhesive itself or at the interface between the adhesive and the wood (PRG 320 Standard). The total length of open joints (delamination) on each end-grain surface of each specimen was recorded excluding specimen with failure in the wood joint due to checking or small isolated knots and any delamination that was <2.54 mm in length and >5 mm away from any recordable delamination. All measurements were done immediately after the specimens were taken from the oven to the nearest 1.27 mm. In addition, delamination that resulted from any failure where shallow wood failure was noted and no other factors related to the wood, such as grain angle and growth-ring structure, were influencing the delamination was also recorded. An example of the delamination in white ash-white ash cross lamination before and after it was subjected to cyclic delamination (wet-dry cycle conditions), shows the changes that had occurred (Fig. 2). The delamination was measured along the bond lines and reported as the total of delamination lengths on the sawn faces of the specimen divided by the total length of perimeters of the bond line (76.2 mm × 2 sides + 50.8 mm × 2sides = 254 mm per block), expressed as a percentage,

$$D = \frac{l_{td}}{l_{tg}} \times 100$$

Where, D is the delamination rate; $l_{td}$is the total delamination length (mm) for all 4 surfaces; $l_{tg}$is the perimeter of all bond lines in the test block (mm).

The delamination observed and recorded at the end of the second cycle must not exceed 10% as indicated by AITC Test T110-2007 standard [30] requirement and for adhesive qualification and lot tests, the delamination shall not exceed 5% for softwoods and 8% for hardwoods after a single cycle. A second cycle for qualification is not permitted according to the AITC test standard.

### 2.6. Determination of adhesive viscosity

The dynamic viscosities (η) of the two adhesives (150 mL for each resin and hardener mix) used in this study were measured using a spindle based Fungilab Viscolead One Rotational Viscometer (Fungilab Inc., Hauppauge, NY). Due to the high viscosity of the glue, only the spindle (SPL 4; Fungilab) were able to give an accurate assessment of the adhesives. The resin to hardener mixing ratios as specified by the adhesive manufacturer were used. All testing was conducted under the same conditions [environmental temperature of 21°C (69.8°F) and time intervals (seconds)] after 10 min of initial mixture. A simple linear regression was used to shows the viscosity of the two adhesives used in the study that describes their resistance to flow through time. This corresponds to the informal concept of "thickness" calculated based on the ratio of the shearing stress (F/A) to the velocity gradient ($\Delta v_x/\Delta z$ or $dv_x/dz$) in the adhesive fluid as shown:

$$\left(\eta = \frac{F/A}{\Delta v_x/\Delta z}\right)$$

### 2.7. Adhesive bond penetration and SEM image processing

To investigate the distribution and penetration of adhesive in the bond line region of the mixed species cross-lamination, the electron

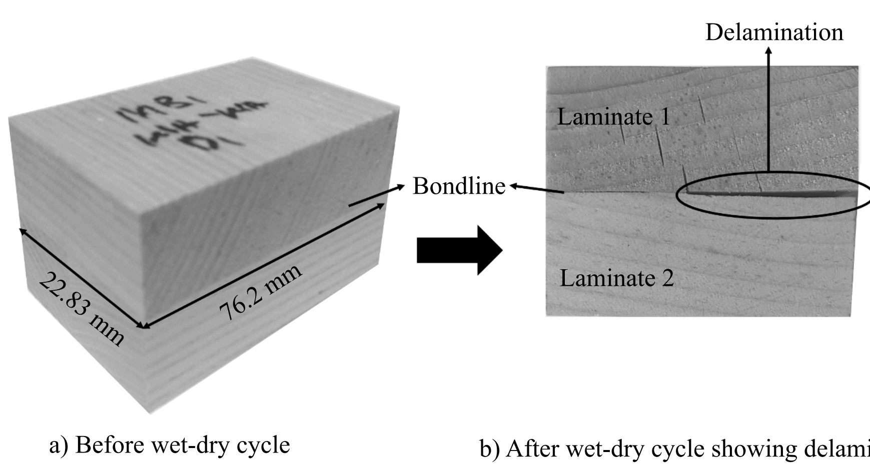


**Fig. 2.** White ash cross-laminated specimen for delamination test. a) before wet-dry cycle; b) after wet-dry cycles.

microscope images were taken from the 10×10 mm square cut from the center of each cross lamination with the bond line placed at the center at 39 mm working distance. The accelerating voltage used was 15 kV under three different magnifications (×300, ×200 and × 100) using a JEOL JSM-6400 scanning electron microscope. These samples were first cut near the bondline of the delamination blocks using band saw, and then soaked in water for a minimum of 3 h before microtoming with LEICA® SM2000 R sliding microtome and then carbon coated with Denton Vacuum DV-502A. Soaking the samples made the wood softened for smooth microtoming. The microtome samples were dried in an oven to remove all moisture before carbon coating. A total of 16 cross-laminated test blocks were chosen including 8 blocks of the melamine adhesive and 8 of the phenol resorcinol adhesive for SEM imaging. The cross-laminated samples were selected based on the pore distribution and or transitioning of the wood blocks. These included; diffuse porous/diffuse porous cross-lamination (SM-YB), ring porous/diffuse porous (RM-SM) cross-lamination, ring porous/abrupt transition (WA-RP) cross-lamination, ring porous/gradual transition (RO-WP) cross-lamination, diffuse porous/gradual transition (BW-WP) cross-lamination, ring porous/ring porous (RO-WA) cross-lamination, diffuse porous/abrupt transition (YB-RP) cross-lamination and abrupt/gradual transition (WP-RP) cross-lamination.

SEM images were taken in both the longitudinal-transverse (LT) and longitudinal-radial (LR) planes. The ImageJ bundled with 64-bit Java 1.8.0_112 (National Health Institute Open Software) was used for the SEM image analysis and calibrated to a micrometer (μm) scale. The calibration was done by using the line tool to draw the same length of the scale bar and change the measurement to μm. The "straight" segmented hand tool was used to measure the adhesive penetration depth from one end of the interphase in one adherend to the other end of the interphase layer in the other adherend that it is cross laminated with. Each cross-laminated block was then measured at 10 different points to determine the averaged depth of penetration. The free hand selection tool was used to trace adhesive area for each block for the two different adhesives used in the study to determine depth area (μm$^2$). The maximum penetration depth lacks important information about the bond line morphology, in regard to the penetration itself, as well as to the pure bond line between the two adherents as identified by [9]. Hence, we employed image analysis tools (ImageJ) to help in analysis of the image taken from the SEM.

### 2.8. Statistic analysis

The normal quantile plot of the delamination percentage *(x)* obtained from the wet dry cycle for both single species and mixed species indicated asymmetrical distributions, suggesting that delamination percentage was not normally distributed. A logistic regression (also known as Squish and Logist) function ($logist_x = \frac{1}{(1+e^{-x})}$)in JMP® PRO 14.0 (SAS Institute Inc., Cary, NC) [32] was used to model the probability by transforming the delamination percentage scores (x) of the cross-laminated samples as a function of the species, SG and anatomical features [33 34]. A delamination percentage score of zero was transformed to 0.5. Normal probability plots and standardized residuals were tested for the assumptions of normality and constant variance of errors using the Shapiro-Wilk test. The single species cross-lamination and mixed species cross-lamination goodness of fit for the transformed data was $p < 0.001$ ($p <$ W) indicating a normal distribution.

An ANOVA and descriptive statistics were performed using the JMP® PRO 14.0 software at an alpha level of 0.05 to test for the significant differences in total delamination percentage based on the logistic transformation. To model the pass/fail of the delamination percentage based on the AITC Test 110 2007, of adhesive qualification of 8% and below, the delamination percentage was assigned a probability of 1 and 0, where 1 (>8%) indicated fail and 0 (≤ 8%) indicated pass for the adhesive qualification test. A mosaic plot was used to illustrate the adhesive qualification based on the failure rate or pass of each sample in a given species. The Alpha level associated with a 95% confidence at 0.05 of the chi-squares $\left(\chi = \sum \frac{(f_o - f_e)}{f_e}\right)$ statistics ($f_o$ and $f_e$represent the observed and expected frequency respectively) were used to test the relationship that exists in the cross-laminated species of the mosaic plot.

The adhesive bond penetration of the mixed species determined through microscope also used ANOVA and descriptive statistics. the species type, anatomical features, and SG was considered a fixed variable ($Y_{ij}$) and tested for the relationship to the delamination percentage. The mean values and standard errors for the delamination was considered independent values ($B_j$), which were calculated using pivot tables with a model of $Y_{ij} = \mu + \alpha_i + B_j + \varepsilon_{ij}$, where μ is the intercept and $\alpha_i$ is the assigned treatment such as the adhesive and $\varepsilon_{ij}$ is the is the error assumed independent and normally distributed with mean 0 and variance $\sigma^2$. Pearson correlation analyses were conducted for the averages of the cyclic delamination test mimicking the bonding performance in an external condition. A post hoc analysis using Tukey's HSD at 95% confidence level was used to identify sample means that were revealed by ANOVA to be significantly different from each other. Linear regression was used to determine the relationship between the SG and delamination percentage of the single species cross-lamination. All statistical analyses were carried out using the JMP® PRO 14.0 software.

## 3. Results

### 3.1. Single species cross-laminations

Fig. 3 shows the mean delamination rate of eight replicates of test blocks for seven single-species cross-laminations. Both red pine and basswood test blocks (RP-RP and BW-BW) showed no delamination, in both the melamine adhesive and the phenol resorcinol adhesive blocks. Aspen and white pine recorded no delamination in resorcinol blocks, and red oak recorded no delamination in melamine blocks. The highest delamination rate was found in white ash with the melamine adhesive (35.6%) and in yellow birch with the phenol resorcinol adhesive (30.9%), respectively. An ANOVA of the transformed delamination rate indicates a significant difference between species ($p$ = 0.0008). The Tukey HSD indicated that there was a significant difference in yellow birch to basswood and red pine ($p$ = 0.0013), and all others were not significant.

A comparison of the mean delamination rate across the range of SG indicated that the phenol resorcinol adhesive recorded little delamination in the species with low SG (0.35–0.46) (Fig. 4). The delamination percentage of species with SG of 0.54 and 0.64 increased but remained below 7%. There was an increase in delamination percentage at SG of 0.62, with the highest delamination percentage at 30.9% and a decrease in delamination percentage at a SG of 0.63 to 10.72%. In the melamine

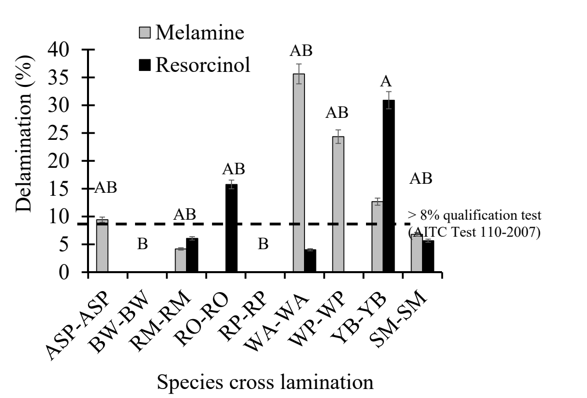


**Fig. 3.** Mean delamination rate (%) of the single species cross-laminations.

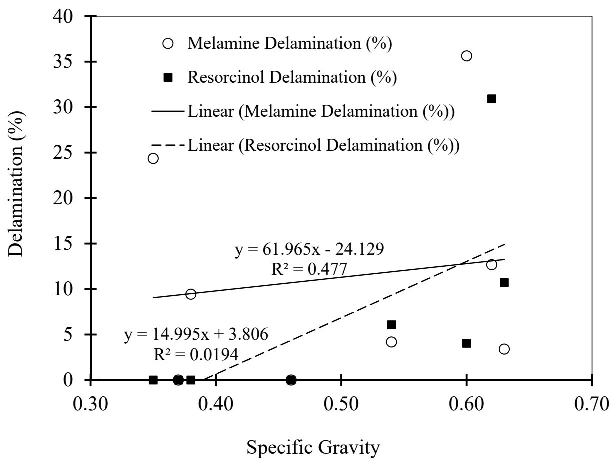


**Fig. 4.** Delamination rate in relation to SG for single species cross-laminations.

adhesive, the species with SG of 0.37 and 0.46 produced no delamination, but the delamination was high at a SG of 0.35 (24.35%). The SG of 0.54 and 0.63 was below 5% total delamination percentage. A linear regression of the delamination percentage by the SG indicates a weak correlation for both adhesives (Fig. 4). However, the phenol resorcinol adhesive produced lower Root Mean Square Error (RMSE = 0.18) and a weak but relatively higher $R^2$ (0.24) compared to melamine with RMSE of 0.23 and $R^2$ of 0.01. The low $R^2$ values for both adhesives implies that the SG and the percentage of delamination are not directly dependent on each other. The differences between the species bonded with the phenol resorcinol adhesive was significant ($P < 0.0001$) but those bonded with melamine was not ($p = 0.23$).

Delamination rate varied with anatomical features (Fig. 5). No delamination was found in red pine cross-laminations, a case of abrupt earlywood-to-latewood transition. In white pine cross-laminations, which have gradual earlywood-to-latewood transition, no delamination was recorded for the phenol resorcinol adhesive, but the highest delamination rate (24.4%) was observed for melamine adhesive. The cross-laminations with ring porous species yielded a delamination rate exceeding 8% for both the phenol resorcinol and the melamine adhesives, 9.9% for the phenol resorcinol and 17.8% for the melamine. The delamination rate fell below 7% in the diffuse-porous cross-laminations for both adhesives.

The pass/fail rate of the bondline delamination determined based on the AITC Test 110 2007 standard provides a clear picture of the delamination tests (Fig. 6). In melamine-bonded specimens, the failure rate of the delamination for aspen, white ash and white pine was over 50%; in the phenol resorcinol-bonded specimens, only yellow birch recorded a failure rate above 50%. However, the delamination failure

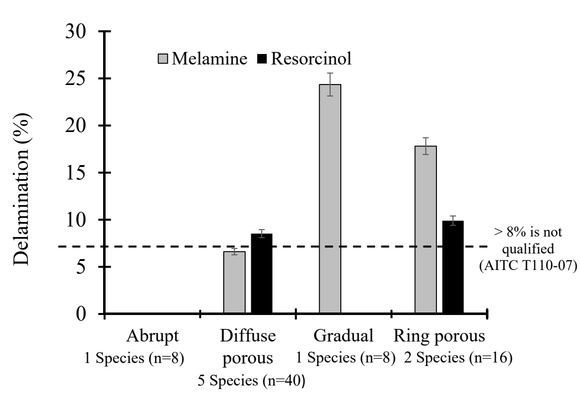


**Fig. 5.** The mean delamination rate for single-species cross-laminations with different anatomical features. (Error bars indicate standard errors).

rate in red oak for the phenol resorcinol was relatively high (37.5%).

### 3.2. Mixed species cross-laminations

The seven hardwoods and two softwoods were mixed and cross-laminated to yield three main categories – mixed hardwood cross-laminations, mixed softwood cross-laminations, and hybrid hardwood-softwood cross-laminations (Fig. 7). The softwood cross-laminations showed no delamination in both adhesives while hardwood cross-laminations indicated the highest amount of delamination rate 7.92 % for the phenol resorcinol and 6.98% for the melamine adhesive. The hybrid cross-laminations exhibited a delamination rate of <3% in both adhesives. The difference between the three categories (hardwood cross-lamination, softwood cross-lamination and hybrid cross-lamination) was significant ($p < 0.0001$). The Tukey-Kramer HSD test at 95% confidence level indicated a significant difference between the hardwood cross-lamination and the hybrid ($p < 0.0002$) and softwood cross-laminations ($p = 0.0239$). No significant difference was found between the softwood cross-lamination and the hybrid cross-lamination ($p = 0.4295$).

The groupings of the mixed species cross-laminations showed a varied delamination percentage in relation to the anatomical features (Fig. 8). Also, the relationship between the melamine adhesive and the phenol resorcinol adhesive in the mixed species cross lamination was weak based on the summary fit ($R^2 = 0.043$). However, there were significant differences among the sample means ($p = 0.0007$), with differences only found between abrupt/diffuse porous and diffuse porous/diffuse porous cross lamination ($p = 0.0101$). The diffuse porous/ring porous cross-lamination recorded the highest mean delamination percentage (8.34%) with melamine in comparison with other pore distributions. However, the lowest performance (highest delamination) based on the individual species cross-lamination, was for RM-ASP (29.41%) in the diffuse-porous/diffuse-porous cross-lamination (Fig. 8).

Considering the phenol resorcinol adhesive diffuse-porous/diffuse-porous combination had the two lowest overall delamination percentage (22.10%) based on their anatomical features and the worst species delamination seen in RM-SM cross-lamination (46.66%). RP-ASP cross lamination in the abrupt/diffuse porous cross-lamination (A) produced no delamination in either adhesive while red pine cross-laminated with sugar maple and basswood produced no delamination only for melamine adhesive and red pine cross-laminated with red maple only for the phenol resorcinol adhesive. In the abrupt/ring-porous cross-lamination (B) there was no delamination for the phenol resorcinol adhesive in the red pine cross-laminated with red oak and white ash. The abrupt/gradual cross-lamination (C) consisting of RP-WP cross lamination also recorded no delamination in both adhesives. Other bond properties that showed no delamination in both adhesives included YB-BW cross lamination in the diffuse-porous/diffuse-porous cross lamination (D) and in the WP-YB cross-lamination in the gradual/diffuse porous (F) anatomical properties. Analysis of the mix species cross-lamination based on their anatomic features showed significant differences ($p < 0.001$*) in relation to the delamination percentages (Supplementary sheet 1) and individual species that did not meet the AITC 110 2007.

The logistic regression of the mixed-species cross-laminations indicates high pass rate in most of the mixed-species except in RM-ASP, RM-WA, and RO-SM cross laminations with the melamine adhesive that had a failure rate of 50% and above (Fig. 9). With the phenol resorcinol adhesive, high failure rate was seen in ASP-SM, ASP-YB, RM-SM, and RO-WA cross-laminations. The differences in the mixed-species cross-laminations were significant for both adhesives ($p < 0.0001$).

### 3.3. Viscosity and adhesive penetration depth in the cross-laminated layers

The results from the viscosity of the adhesives showed a strong

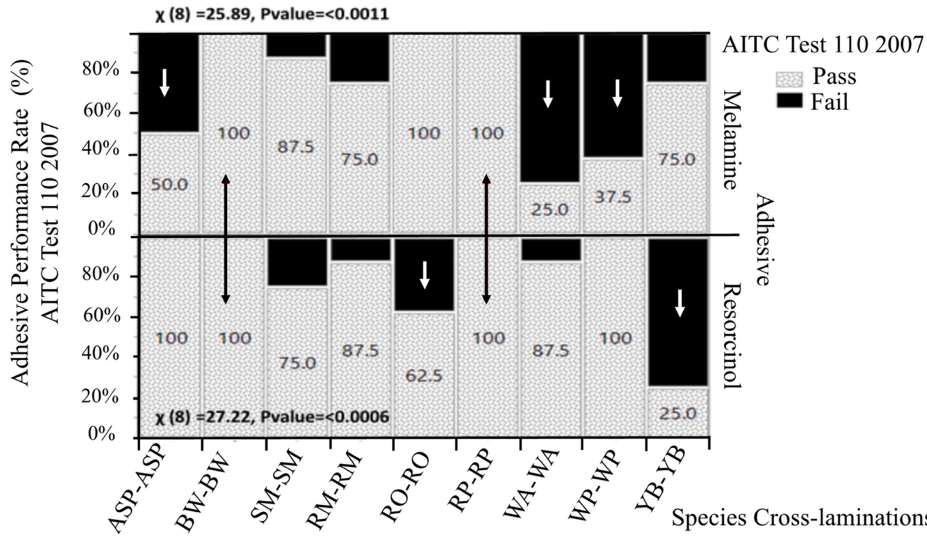


**Fig. 6.** The Mosaic plot of the failure rate of single species cross-laminations based on the AITC standard.

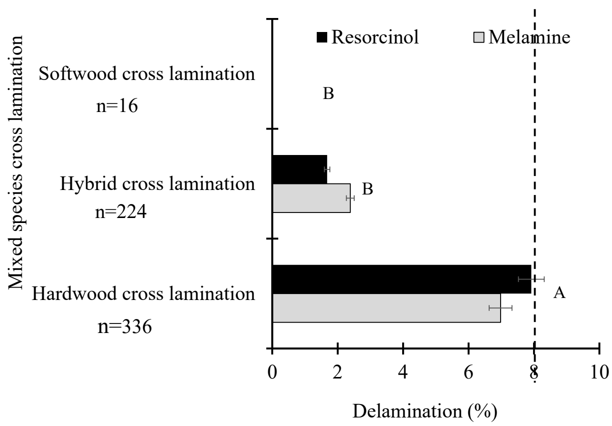


**Fig. 7.** Delamination rate of mixed species cross-laminations (Letters indicates significant differences).

positive r-squared ($R^2 > 0.75$) for both adhesive with the melamine-based adhesive (>1900 cP) being more viscous compared to the phenol resorcinol adhesive (<1400 cP) which may have effect on its flow and penetration at the bond interphase (1 cP = 1 mPa.s) (Supplementary sheet 1).

An analysis of variance (ANOVA) of the adhesive maximum penetration depth (μm) indicates a significant difference in the anatomical features ($p < 0.0126$; Fig. 10). Pairwise comparison using Tukey-Kramer HSD at 0.05 α-level indicates a variable significance in the mixed species cross laminations with RP-YB (abrupt diffuse/porous being) being significantly different from SM-YB (diffuse porous/diffuse porous) ($p = 0.0122$), SM-RO (diffuse porous/ring porous) ($p = 0.0210$), RP-WA (ring-porous/abrupt) ($p = 0.0334$) and RO-WA (ring porous/ring porous) ($p = 0.0456$). The percentage delamination recorded based on the selected species for this section was not significantly different ($p = 0.3444$; Fig. 10).

The Linear regression of the delamination percentage and the maximum penetration depth (μm) for the selected mix species cross lamination based on their anatomical features indicates a significant difference in melamine adhesive ($p = 0.0159$) with an $R^2$ value of 0.65. The phenol resorcinol adhesive was shown not to be significant ($p = 0.4669$) with an $R^2$ value of 0.09 (Fig. 11).

## 4. Discussion

### 4.1. Delamination of single species cross-laminations

Considering the delamination results of the single species cross-laminations, some of the hardwood species did not meet the requirements stated in the AITC Test T110-2007 [30] and ASTM D2559 [31]. This is similar to the results reported by Franke et al. [35] where the outcome of delamination tests for more than half of the specimen did not meet the requirement. Sikora [36] also conveyed similar results. However, there was no delamination for red pine (RP) and basswood (BW) with the two commercial adhesives (melamine and resorcinol) used in this current study. This is likely due to the low SGs of these two species. Basswood is known to show high volumetric swelling and shrinkage which may be as a result of the considerably more tension wood than most hardwood species [4]. Several studies have shown that tension wood consisting of gelatinous (G) layer formed towards the lumen with nearly pure cellulose and can form in place of the S2 or S3 layers making the G-layer much more vulnerable to moisture absorption with more dramatic dimensional changes [37,38].

In addition, RO recorded no delamination with the melamine whereas ASP and WP recorded no delamination with the phenol resorcinol adhesive. Although SM and RM recorded some level of delamination with both adhesives, their delamination rates were below the 8% requirement of the AITC Test T110-2007 standard. However, when the ASTM D2559 is applied as their bases for qualification then they do not meet the requirement. The ASTM D2559 indicates that total delamination percentage shall not exceed 1 % for any bond line in the laminated test member for softwoods or 1.6 % for hardwoods. This current study used the AITC Test T110-2007 [30] as the basis for adhesive qualification. YB did not meet any of the delamination standard requirements for either adhesives which could be attributed to its high SG (0.62). A general comparison of the results indicated that, the phenol resorcinol adhesive had a better performance than the melamine-based adhesive when subjected to a wet-dry cycle conditions (cyclic delamination) than melamine. Although previous studies by the authors (unpublished) suggest that melamine adhesive bonds were stronger in shear under compression loading, where the mean shear strength was 2% higher in hardwood single combinations compared with resorcinol hardwood single combinations and 18% higher than resorcinol softwood respectively [39]. Other studies presented similar results, where the compressive shear strength of wood bonded with MUF adhesive in 2% (w/w) formic acid solution at 12 and 18% MC stabilized at 10.6 and 10.0 MPa, respectively, which are 17% and 16% higher than that with

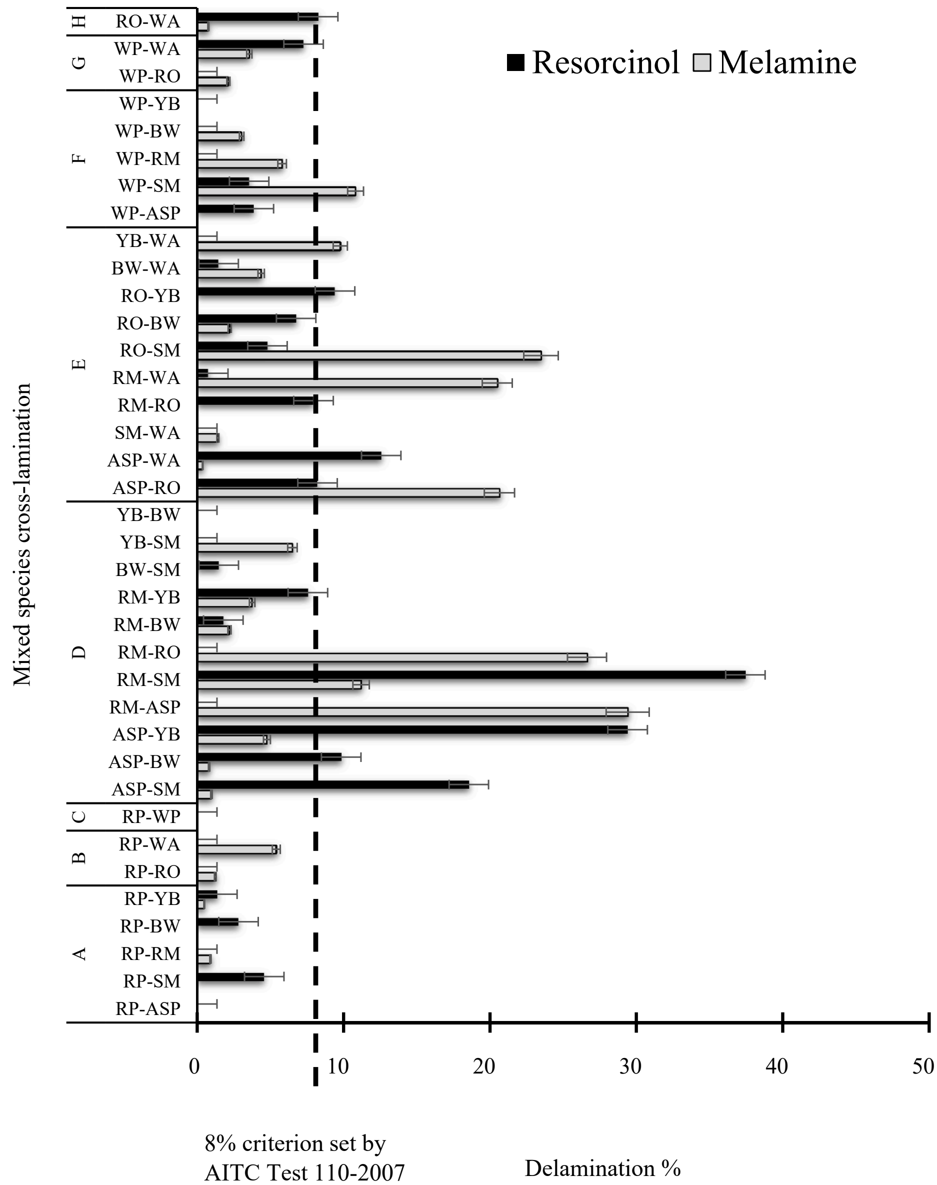


**Fig. 8.** Delamination rate of the mixed species cross-laminations in accordance with the anatomical features. A: abrupt/diffuse porous (n = 40); B: abrupt/ring porous (n = 32); C: abrupt/gradual (n = 16); D: diffuse porous/diffuse porous (n = 161); E: diffuse porous/ring porous (n = 159); F: gradual/diffuse porous (n = 80); G: gradual/ring porous (n = 32); and H: ring porous/ring porous cross laminations (n = 16).

PRF adhesive at the same condition [40].

### 4.2. *Effects of SG and anatomical features on delamination*

For the samples bonded with the phenol resorcinol adhesive, most of the species with lower SGs (≤ 0.5) had no delamination but the delamination percentage increased in the species of higher SGs (Fig. 4). A study by Koch [43] showed similar results in a plywood made from eight loblolly pine trees selected to exhibit a range of SG and growth rate. In the study, high SG wood delaminated more rapidly than low SG wood, particularly when the glue application rate was low or the assembly time was long. The melamine adhesive performed poorly at SG of 0.35 and SG of 0.60 which could be attributed to the rate of penetration. The phenol resorcinol adhesive was more effective when subjected to wet-dry conditions in most species except for the SG of 0.62 (yellow birch cross lamination). The better performance of the phenol resorcinol adhesive can be atrributed to the similarity between the adhesive and the lignin in wood surfaces as both have aromatic ring structures. This similarity contributes to the formation of durable glue bonds [16,18]. As softwoods usually have a higher lignin content than hardwoods, the phenol resorcinol adhesive was seen performed better in this study with the two pine species than in the hardwoods.

The primary structural difference between hardwood and softwood (cellulose and lignin) could have played a role in the better performance of softwood with cross laminated timber. The benzene ring in the resorcinol adhesive and the softwood (EtOH/Benzene in softwood > EtOH/Benzene hardwood) could have increased the bondabilty in softwood as there may be strong affinity between them that could results in good chain link analogy for adhesion and cohesion which has a strong influence on optimum conditions for good bonding [41].

The $R^2$ of the linear regression between the delamination percentage and SG for both adhesives for the single species cross lamination of delamination percentage and specific gravity was low (<0.25), suggesting a weak association between the SGand the delamination percentage (Fig. 4). However, the predictor, SG, in the phenol resorcinol adhesive were statistically significant indicating that the changes in the

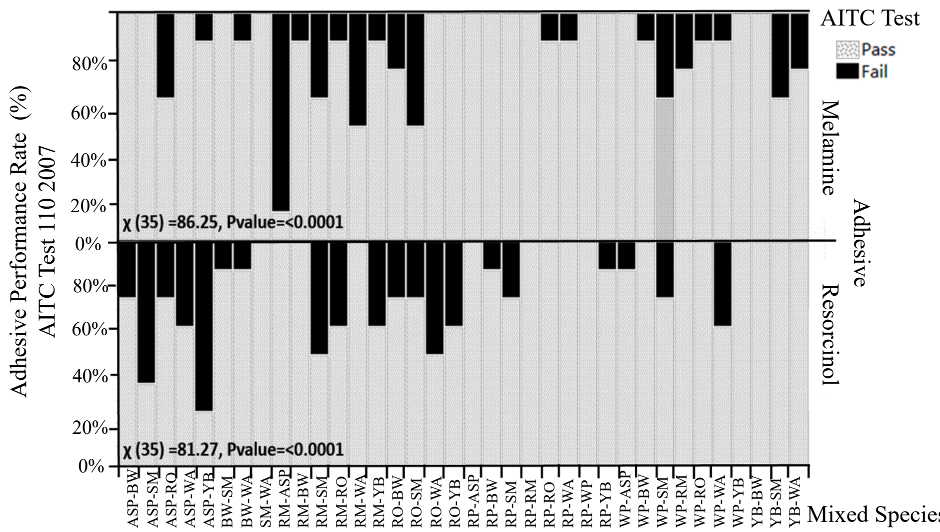


**Fig. 9.** The failure rate of mix species cross lamination based on the AITC standard.

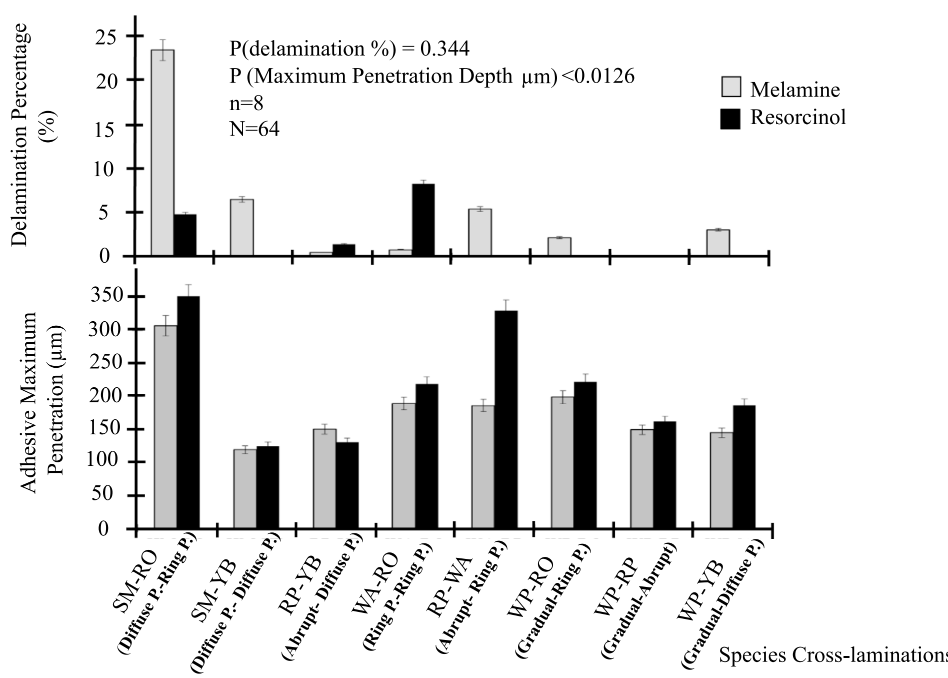


**Fig. 10.** Anatomical features effects on adhesive penetration depth and delamination percentage.

predictor values (specific gravity) were associated with changes in the responding value (delamination percentage).

Wood anatomical parameters had an influence on the delamination percentage of cross-laminated species (Fig. 5). The 5 diffuse-porous species worked better with both adhesives compared to the 2 ring porous species in terms of bond durability. However only the combinations of the diffuse porous species and the melamine adhesive in this study met the 8% delamination requirement of the AITC standard. The highest delamination percentage was seen in the combination of ring porous species and the melamine adhesive. When the phenol resorcinol adhesive was used, the average delamination percentages were below 10% for both the diffuse porous and the ring porous species. But neither of them met the delamination requirement. Previous studies [14] and [11] suggested several factors, including anatomical features such as pore distribution affect the adhesive penetration and bondability of the wood. In the softwood, the adhesive filled the tracheids which are effectively interconnected, compared to the complicated hardwood structure where adhesive filled vessels which appear to be isolated from the bond line [22]. Insufficient penetration of adhesive limits surface contact, leaving a thick film of adhesive on the surface, whereas over-penetration creates starved bond lines, thus leading to weak bonding in both cases [25].

The logistic regression plotted as mosaic provides guidelines for selecting hardwood species for cross lamination applications, such as CLT. It provides the rate of failure of the single species cross laminations based on the AITC Test 110 2007 (Fig. 6). A species with $\geq$ 50% rate of failure may require special attention on the durability of the bondlines in cross-laminations. However, the bondline perfomance can be optimized for combinations of specific species and adhesive system.

### 4.3. Mixed species delamination

Our results indicated a better bonding performance for the mixed species cross-laminations compared to the single species cross-

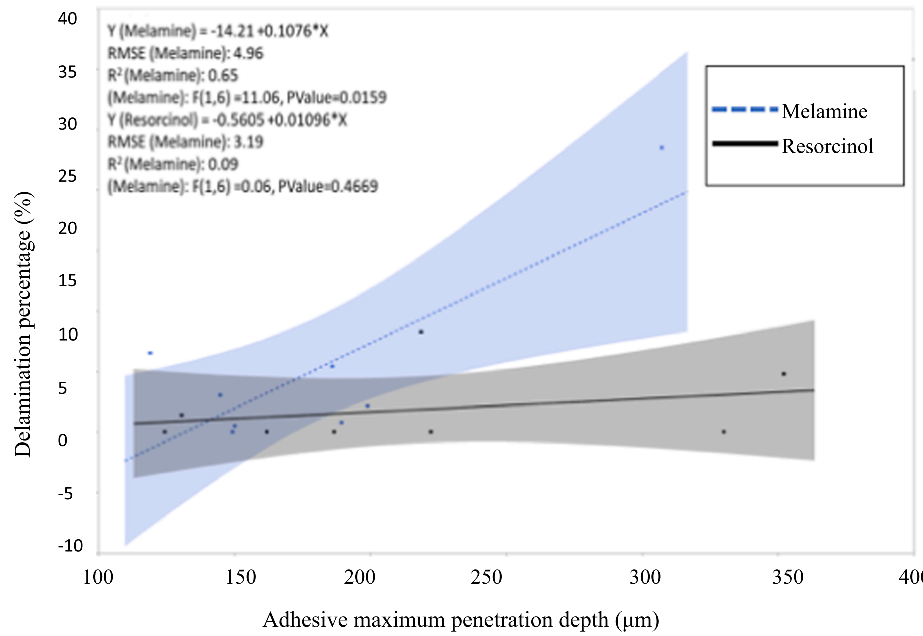


**Fig. 11.** Relationship between delamination rate and maximum adhesive penetration depth.

laminations, with a delamination rate of <8% (Fig. 7). The differences in wood anatomical features, such as pore distribution, earlywood/latewood transitioning, and types of wood cells, influence the bond quality. Mixing species of different anatomical features may have reduced the negative effects of some features that would be exacerbated in single species cross-laminations. Other factors, such as wood density and extractives may affect the bond quality as well. The hybrid (hardwood and softwood mixed) cross-laminations also had a better bonding performance compared to the mixed hardwood cross-laminations. These findings expand the discussion on the use of hardwood lumber in CLT productions towards the consideration of using mixed species and hybrid species.

Considering the individual combinations of different species in Fig. 8, the ASP-RO, RM-ASP, RM-SM, RM-WA, RO-SM, YB-WA, and SM-WP combinations had delamination rates higher than the standard requirement when the melamine adhesive was used. For the phenol resorcinol adhesive, the mix species that did not qualify included ASP-SM, ASP-RO, ASP-BW, ASP-WA, ASP-YB, RM-SM, RO-WA, and RO-YB. The average coefficients of variation in mixed hardwood cross-laminations and hybrid cross laminations for the two commercial adhesives were below 40% .

The rate of failure in the mix species indicates that caution should be taken when bonding some species with the melamine adhesive, such as RM-ASP, RM-WA, and RO-SM combinations. For the phenol resorcinol adhesive, the combinations included ASP-YB, RM-SM, and RO-WA. All those combinations had failure rates of 50% and above (Fig. 9).

### 4.4. Adhesive penetration

The melamine adhesive was seen to be more viscous ($1972 \leq X \leq 2095$ cP) compared to the phenol resorcinol adhesive ($1366 \leq X \leq 1396$ cP) under the same testing condition. Several studies have indicated that viscosity affects bond penetration that intends to affect bond durability [14,17]. The penol resorcinol adhesive produced deeper penetration in most species compared to the melamine base adhesive(Fig. 10). The higher penetration of the phenol resorcinol adhesive did not necessarily indicate less delamination percentage or better bond performance in the wet-dry cycle as too much penetration results in 'dry-out' at the interface. However, less penetration of the melamine adhesive did not necessarily mean better bonds either as shallow penetration could also limit the formation of the three-dimensional zone at the interphase. Optimal adhesive penetration will be required for proper bond formation [16,21] that may vary from each combination of mixed species in cross-laminations. The study by Hass et al. [11] on two different types of glue (viscosity for glue I was 5.32 ± 0.01 Pa·s and for glue II was 12.05 ± 0.03 Pa·s) indicated that the penetration of the adhesive in beech wood is affected by the vessel network characteristics and viscosity. Melamine adhesive in previous studies was seen to have higher shear strength and wood failure percentage, but when subjected to the wet-dry conditions, resorcinol was seen to perform better.

The analysis of the SEM images showed high maximum penetration of resorcinol in all the species except for the SM-RO (diffuse-porous/ring-porous) cross lamination (Fig. 10). The low penetration of the melamine adhesive could be attributed to its high viscosity.

The surface morphology from the SEM images explains how the adhesive and wood surfaces interplays to result in bond formation. This study only looked at the rate of adhesive penetration however, several other factors relating to surface energy also play major roles influencing face bond durability. Factors such as adhesives spreading rate, bondline thickness, curing pressure are necessary to achieve good mechanically stable bond [42]. Nonetheless, as suggested by Ren and Frazier [43], it is necessary for adhesive to penetrate deep enough to bond with undamaged cell walls for a durable bond. The difference in adhesive maximum penetration depth (μm) was significant ($P < 0.0126$) with the selected species based on their anatomical features indicating that the anatomical features has influence over the degree of penetration. The linear regression indicates a relatively higher association of the selected cross lamination species with melamine adhesive ($R^2 = 0.65$) compared to resorcinol ($R^2 = 0.09$).

## 5. Conclusions

We investigated the durability of the adhesive bonds in the cross-laminated panels fabricated with seven hardwood and two softwood species from the Great Lakes region. The main findings are summarized as follows:

(1) The mixed hardwood cross-laminations and hybrid cross-laminations all met the requirements per the AITC 110 2007 standard at wet and dry conditions (cyclic delamination

performance). Both the melamine- and the phenol resorcinol-based adhesives performed adequately in softwood and hardwood cross-laminations. The phenol resorcinol adhesive was more effective in bonding mixed hardwood cross-laminations.

(2) Anatomical features and SG were found to link to the delamination in both single and mixed hardwood species. The cross-laminations of the diffuse porous species showed adequate performance compared to those of the ring porous species in the single species cross-laminations. SM-RM, ASP-RM, ASP-RO, ASP-BW, ASP-WA, RO-WA, RO-YB, and ASP-YB cross-laminations with the phenol resorcinol adhesive did not pass the AITC T110 bondline durability test. Based on the frequency of failure rate, this study indicated that caution should be exercised when face bonding ASP, WA, and WP with the melamine adhesive and and YB with the phenol resorcinol adhesive.

(3) Mixed hardwood species generally performed better in the delamination test than the single species. The results demonstrated the feasibility of using mixed hardwood species in cross-laminations. The hardwood-softwood hybrid was found to be the best species combinations for the cross-laminations fabricated in this study.

(4) The viscosity of the adhesive influenced the maximum depth of glue penetration into wood, which may have affected the delamination rate. However, such effects were further complicated by wood properties, such as species, anatomical features, and SG.

The durability of adhesive bonds is critical to the cross-laminated wood products that have a hardwood component, and it is affected by both the wood anatomical features and the adhesive system used. Although the results of this study demonstrated the feasibility of incorporating hardwoods in cross-laminations using more than one species, specific combinations will need detailed studies to investigate gluing parameters for optimal bond performance.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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