Effect of Relative Humidity Level on Bonding Properties of Black Spruce Glulam

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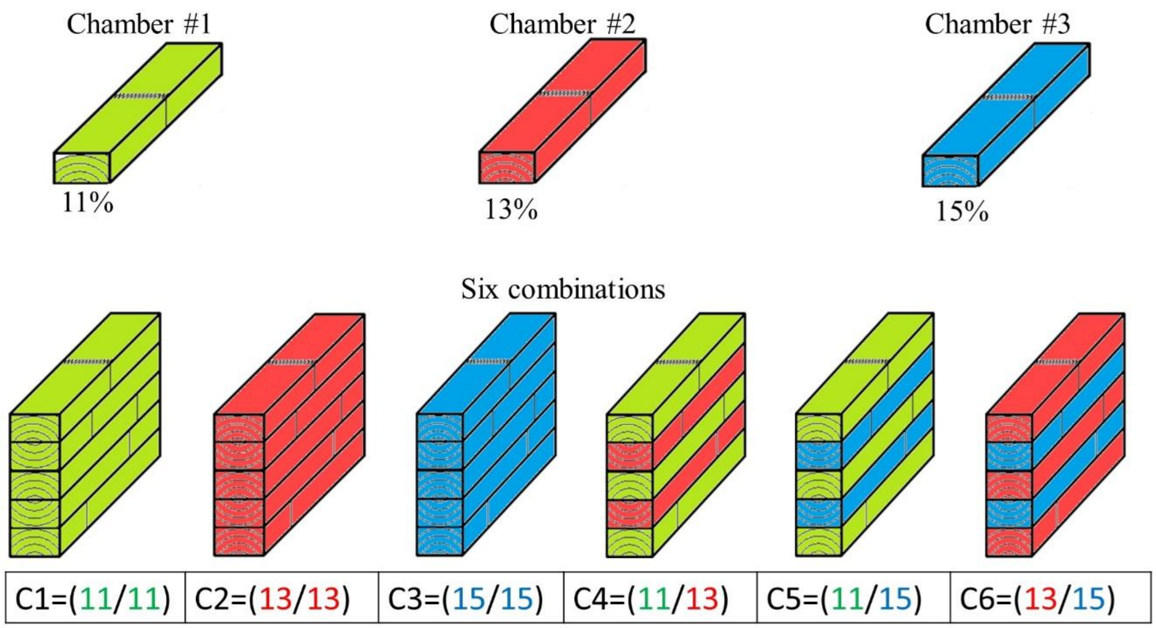
GRAPHICAL ABSTRACT



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Glued-laminated timber (glulam) is a structural wood-based composite widely used in construction. One of the constraints to its production is the requirement that lamellae must have the same moisture content before bonding. This study analyzed the effect of joining lamellae having different moisture content levels on the bonding performance of glulam elements. Black spruce [*Picea mariana* (Mill.) B.S.P.] wood with different levels of moisture content (11%, 13%, and 15%) were bonded with two component polyurethane and placed in three different environments with different relative humidities. Block shear and delamination tests were performed according to ASTM D905-08 (2021) and EN 302-2 (2013), respectively, and the glue line profile was assessed with a micro-CT scanner. The results showed that the relative humidity had more influence on the results than the initial moisture of the wood. The results obtained for block shear and delamination tests respected the limits of the standard, but the adhesive profile of mixed moisture glulam presented some undesirable characteristics (thinner and irregular adhesive distribution). Micro-CT scan reconstructed images were found to be an interesting tool for this type of evaluation.

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Keywords: Block-shear; Delamination; CT-Scan; Polyurethane; Softwood

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INTRODUCTION

Even though wood is one of the oldest building materials, in the last two decades it has received increased interest due to questions involving climate change and the sustainable management of renewable resources (Essoua and Blanchet 2017). Proof of that is the update of the National Building Code of Canada in 2020, now allowing constructions up to 12 stories with wood and wood-based structural composites (National Research Council Canada 2020).

Wood engineered composites such as plywood, glued laminated timber (glulam), and cross-laminated timber (CLT) provide products with higher dimensional stability, mechanical performance, and manufacturing flexibility in comparison to raw timber equivalents, as they are produced by gluing wood items with synthetic adhesives (Vella *et al.* 2019; Li *et al.* 2022). Glulam beams are manufactured from lamellas bonded horizontally or vertically, and their final quality is dependent on many factors, such as species, moisture content (MC), production parameters, and adhesive characteristics (Mirski *et al.* 2021; Reinprecht *et al.* 2022).

One of the limitations of the glulam industry on this subject is the requirement of lamellae classification according to its MC, *i.e.*, the demand for adjacent pieces to have the same MC. If this limitation is overcome, there will be economic and environmental benefits, as the kiln drying will require less time and, therefore, less energy, and the efforts to pre-classify wood according to moisture content will be reduced. This study evaluated the effect of humidity on glulam bonding performance in three different targeted MCs, *i.e.* 11%, 13%, and 15%. The difference among the chosen MCs, even though small, falls inside the spectrum of the glue manufacturer's practices for mass timber manufacturing and, although widely accepted, has still not been deeply investigated (Purba *et al.* 2022; Tran *et al.* 2024).

It is known that the moisture content from lamellas and variation between them influence bonding properties, which is dependent on the chemical and mechanical properties of the adhesive and the conditions the product is exposed to during its application (Kläusler *et al.* 2013; Hänsel *et al.* 2021; Li *et al.* 2022). The change in humidity conditions influences the properties of the wood-based composites because it affects the wood properties, the adhesive layer, and their interplay (Kläusler *et al.* 2013). However, quantifying this effect in the bonding properties of glulam is not easy, as several factors, as mentioned, play a role in the final performance of the product. Therefore, this study employed lamellae conditioned in rigorously regulated climatic chambers that allowed the assessment with precision of the MC effect on the bonding performance and adhesive distribution profile of glulam.

In this study the species chosen was Black spruce (*Picea mariana* (Mill.) B.S.P.). It is one of the most important industrial species of Eastern Canadian boreal forest, which is widely used in construction applications such as lumber and glued structural members (Lei *et al.* 2005; Kuljich *et al.* 2012; Francezon and Stevanovic 2017).

Considering the importance of this species to the Canadian building sector and the lack of knowledge of how its moisture content affect the final performance of glulam, this paper aimed to evaluate the influence of initial MC and relative humidities on the bonding properties of black spruce.

EXPERIMENTAL

Materials

Black spruce kiln-dried planks were acquired from PFResolute sawmills in Québec, Canada, with nominal dimensions of 3.5 m x 15 cm x 5 cm. These planks were placed in three different climatized chambers to alter their moisture content, Chamber #1 had 40% relative humidity (RH) at 20 °C and equilibrium moisture content (EMQ) of 11%, Chamber #2 had 60% RH at 20 °C and EMQ of 13%, and Chamber #3 had 75% RH at 20 °C and EMQ of 15%.

Five moisture content (MC) specimens, produced according to ASTM D4442 (2020) method with dimensions of 60 mm x 50 mm x 19 mm, were tested for each chamber after the wood was climatized for 21 days. These specimens had their initial mass measured before being placed in a forced convection oven with temperature of 103 ± 2 °C. Then they had their mass measured until there was less than 0.5% difference between consecutive measurements.

Wood presented average MC values of 10.7% (±0.3%), 13.3% (±0.1%), and 14.8% (±0.3%) and average apparent densities of 601 kg/m³ (580 to 656), 610 kg/m³ (562 to 649), and 650 kg/m³ (571 to 733), respectively. The experimental design consisted of bonding black spruce wood climatized in different environments, so there were six different combinations, as shown in Table 1. Samples produced for each combination were climatized after bonding and before testing in different chambers depending on the initial moisture content of the blocks (Table 1). Samples of combinations where both blocks had the same initial moisture content (C1, C2, and C3) were climatized in the same chamber of the referred blocks, for mixed combinations (C4, C5, and C6) the chamber chosen was the same that climatized the block with the lowest moisture content of such combination.

The planks were then cut to the dimensions required to produce samples for block shear and delamination tests and had their surfaces planed the same day of production. The primer Loctite PR 3105 Purbond (Bridgewater, NJ, USA) was diluted in water (1:20) and applied to the surfaces 60 min before placing the glue (Pradhan *et al.* 2024). A bi-component polyurethane Purbond GT20 and hardener Purbond GT205 (Bridgewater, NJ, USA) was prepared in a proportion of 100:15 and spread across the bonding surfaces with a disposable painting roll with a spread rate of 192 g/m². Blocks were weighted during glue applications to make sure that the target amount of adhesive was correctly placed.

Table 1. Experimental Design for Bonding Evaluation

Combination code	Block 1 MC	Block 2 MC	Climatizing Chamber for specimens
C1 (reference)	11	11	Chamber #1
C2	13	13	Chamber #2
C3	15	15	Chamber #3
C4	11	13	Chamber #1
C5	11	15	Chamber #1
C6	13	15	Chamber #2

Samples were pressed in a Dieffenbacher vapor heated press (Eppingen, Germany) at room temperature for 138 min with 1.0 MPa before being placed in the climatizing chamber according to the combination code. Samples were cut into test specimens after seven days to allow full adhesive curing.

Block Shear Tests

Block shear tests were based on the ASTM D905-08 standard (2021), where 10 specimens from each combination were cut from the produce samples. Each specimen had dimensions of 50.8 mm x 50.8 mm x 38.1 mm with one 6.35 mm notch on each side. This bonding evaluation methodology and delamination tests were chosen as they are considered important for ensuring the proper performance of glulam during their service life (Li *et al.* 2015; Hänsel *et al.* 2021).

The contact area between each lamella was measured with a caliper and the shear test occurred in an MTS QTest load frame (Eden Prairie, USA) fitted with a 50 kN load cell and a compression shearing tool at 5.0 mm/min testing speed. Shearing strength was determined by Eq. 1,

$$f_v = F_{max}/S \tag{1}$$

where f_v is the shear strength of the glue line (MPa), F_{max} is the maximum load supported by the specimen before failure (N), and S is the contact surface area (mm²).

Two other properties were assessed for the block shear tests: the percentage of wood in the contact surface after the specimens' failure (by visual analysis) and the maximum displacement of the specimen before its failure.

Delamination Tests

Delamination tests were performed according to the European standard EN 302-2 (2013) for samples with six layers of 25 mm and total dimensions of 100 mm x 80 mm x 150 mm. Three specimens were tested for each combination in three vacuum-pressure cycles. Each cycle comprised two 15-min vacuums at 25 ± 5 kPa followed by 60 min of 600 ± 25 kPa, totaling 150 min.

After each cycle, the samples were dried in a forced convection oven with a temperature of 65 ± 3 °C until their masses were between 102% and 108% of the initial mass. The samples had their delamination measured for each glue line to determinate their maximum and total delamination (Vella *et al.* 2019).

The placement of lamella of different moisture content was alternated, as shown in Fig. 1.

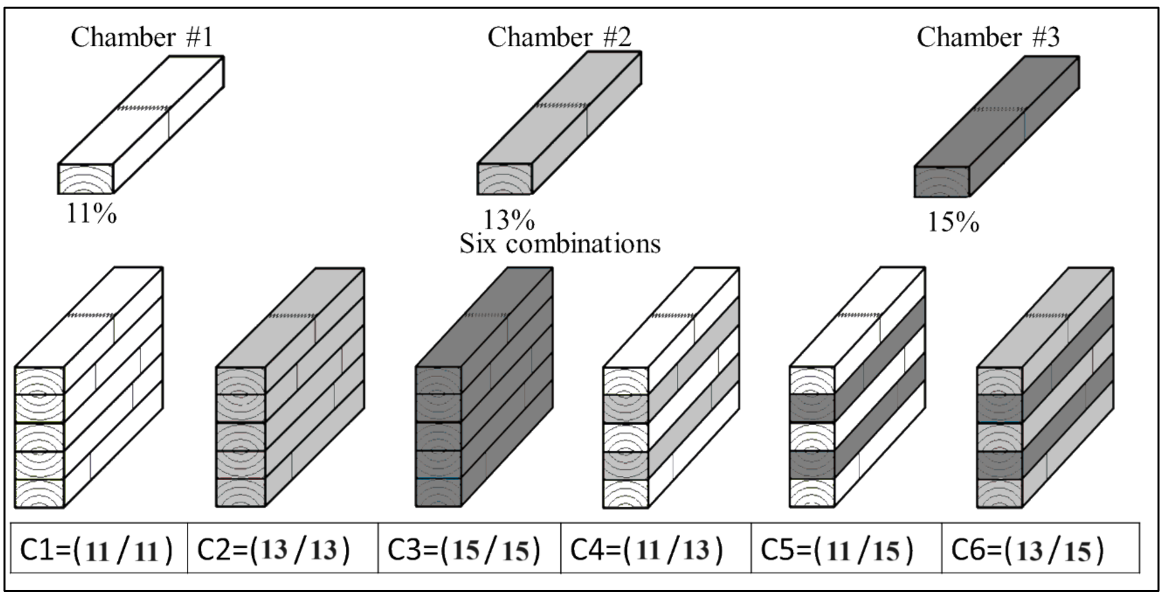

Fig. 1. Lamellae placement according to moisture content and combination

X-Ray Micro Computer Tomography

One small sample of 5 mm x 5 mm x 5 mm x 5 mm was extracted close to the bond line of the block shear billets for analysis with a micro-CT scanner Bruker SkyScan 1272 (Billerica, MA, USA). Specimens' fiber direction was positioned vertically, allowing the 2D image reconstruction of the transversal plane to observe pores and adhesive profile. The parameters used for image acquisition were: 4032 x 2688 resolution, 360 ms exposure time, angular increments of 0.167°, 3 µm image pixel size, and x-ray with 50 kV voltage and 200 µA current.

Digital images were reconstructed using the Nrecon software layered reconstruction software version 1.7.4.2 (Bruke Corp, Kontich, Belgium), where automatic corrections (beam hardening and ring artifact removal) were performed to reduce image artifacts generated during the image acquisition procedure. The software ImageJ was used to analyze the images and to add the scale in the lower right corner of it. Due to the high resolution used for image acquisition, a total of 8680 reconstructed pictures were produced and analyzed and those representatives of each combination were shown in the results.

Statistical analyses were performed to compare the averages obtained for block shear strength and displacement during the tests. Data normality was evaluated using the Shapiro-Wilk test, and the variance analysis was performed using the Tukey test, which showed 5% significance with aid from R software version 4.3.2.

RESULTS AND DISCUSSION

The block shear strength and the wood failure percentage (average and range of measured values) are shown in Fig. 2, along with the statistical analysis. A decrease in bonding strength was observed for combinations bonded with the same moisture content (C1, C2, and C3), which can be attributed to the fact that these combinations were placed in different climatizing chambers after production with increasing RH levels, as described in Table 1, providing a combining effect of adhesion and viscoelastic behavior of wood. The moisture content increase lowers the wood's mechanical strength if it is below the fiber saturation point (Li *et al.* 2022).

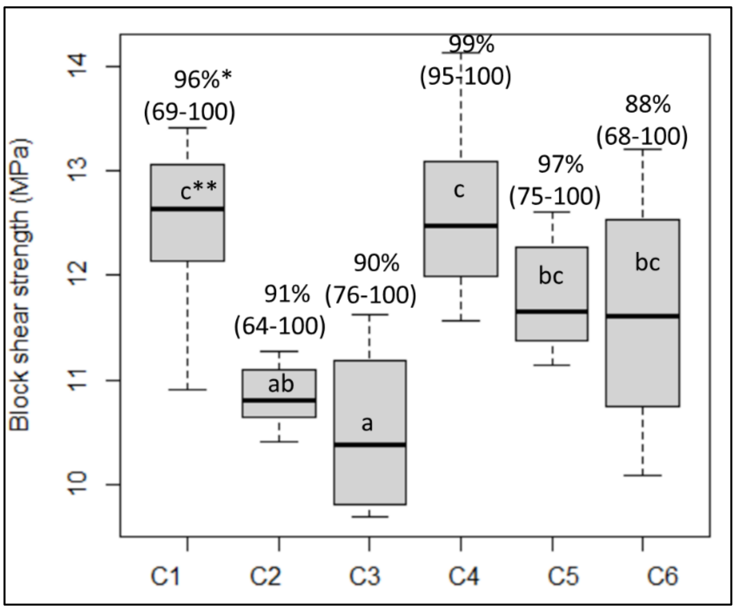

Fig. 2. Block shear strength of the six combinations tested (*percentage of wood failure; **same letter means no statistical difference with 5% significance)

The influence of the moisture content level was also observed in the stiffness of the block shear samples, as seen in Fig. 3, where maximal displacement was also increased. Li *et al.* (2022) noted an increased deformation for Douglas fir plywood with PF adhesive conditioned for higher RH (60, 80, and 95%) and Kläusler *et al.* (2013) and Zhan *et al.* (2019) describe that a higher RH lower young modulus for polyurethane adhesive, making it more ductile, and decreases the stiffness of wood, respectively.

This trend was visualized in the wood failure percentage in the broken shear surface, as combinations placed in higher moisture content conditions (C2, C3, and C6) showed lower failure rates in wood than other combinations, as the wood had higher flexibility than the adhesive in these cases. No correlation was found between the sample strength and its maximum displacement (p-value 0.33 and R^2 0.02).

The moisture content effect was also witnessed in the reconstructed micro tomography images (Fig. 4), as C1 showed a denser and regular bond line, C2 showed a bond line thicker but less densified and C3 presented an even less dense and irregular profile with lacking glue in some parts. The change in adhesive distribution can be associated with the curing of polyurethane based adhesives, which is driven by the reactivity of the MDI isomers with moisture from the wood and ambient humidity (Kläusler *et al.* 2013), but to a lesser extent from wood surface roughness, as it did not seem to have a large effect on the gluing process according to Hänsel *et al.* (2021). At any rate, the analysis of the wood surface was out of the scope of this study.

The image analysis in combination with the micro-CT scan together were able to explain the results of the other bonding evaluation tests (Li *et al.* 2018); however it is important to mention that each reconstructed image shown in Fig. 4 presents concentric rings in their center, which are artifacts created by the method of data obtention (the sample turned around its own axis) and does not reflect the anatomy of wood or the structure of the adhesive.

There was no significant difference between the Combination 1 (reference) and the combinations with mixed moisture contents (C4, C5, and C6), for the block shear strength and maximum displacement. However, the adhesive profile for these mixed combinations showed a highly irregular profile (*i.e.*, variable adhesive thickness and its absence in some points), with low densified glue region and even some areas where there is no glue at all, especially for the combination C5, where lamellas had the highest difference in moisture content (Table 1).

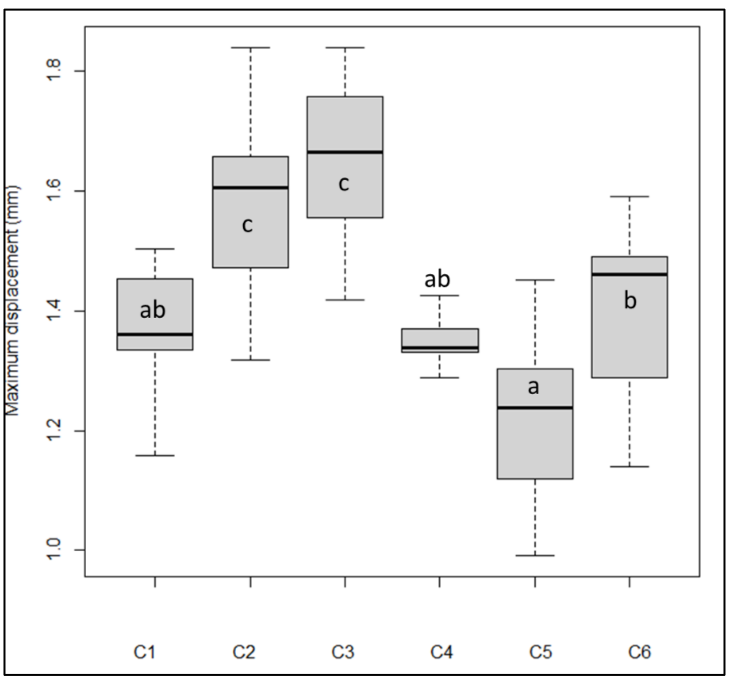

Fig. 3. Maximum displacement during the block shear tests for each combination (same letter means no statistical difference with 5% significance)

Even with the difference in initial moisture content and the humidity levels in the climatizing chambers, all combinations performed adequately for the block shear tests.

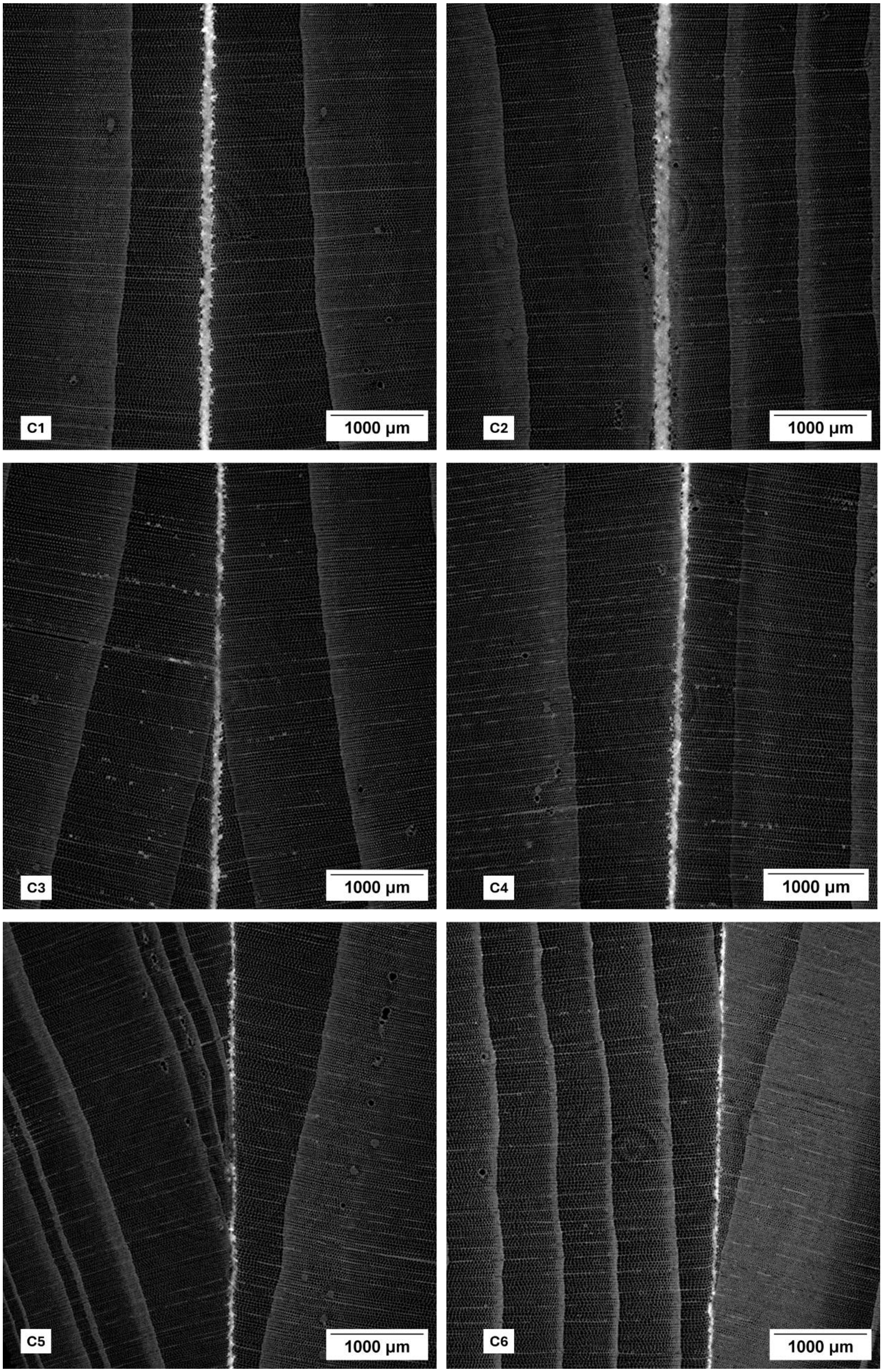

Fig. 4. Two-dimension reconstruction of a transversal layer for each combination tested

Wood percentage obtained for the block shear tests respected the requirement of ASTM D2559-12a (2016) of over 75% and strength ranged from 9 to 14 MPa. Results were coherent with previous results reported by Zhang *et al.* (2011) for Black Spruce evaluated different types of adhesives and Okkonen and River (1989) for softwoods (Douglas-fir and southern pine).

Maximum and total delamination obtained after each cycle are shown in Figs. 5 and 6, respectively. As expected, delamination was increased after each cycle, and the results obtained were adequate according to EN 14080 (2013), which requires less than 30% for maximum and 4% for total delamination. The combination C3 had a more pronounced delamination (23% for maximum delamination and over 2.5% for total delamination) than other combinations tested, which relates to its significantly poorer performance in the block shear tests, as explained earlier, thus showing lower quality of the gluing process (Hanegraaf 2018).

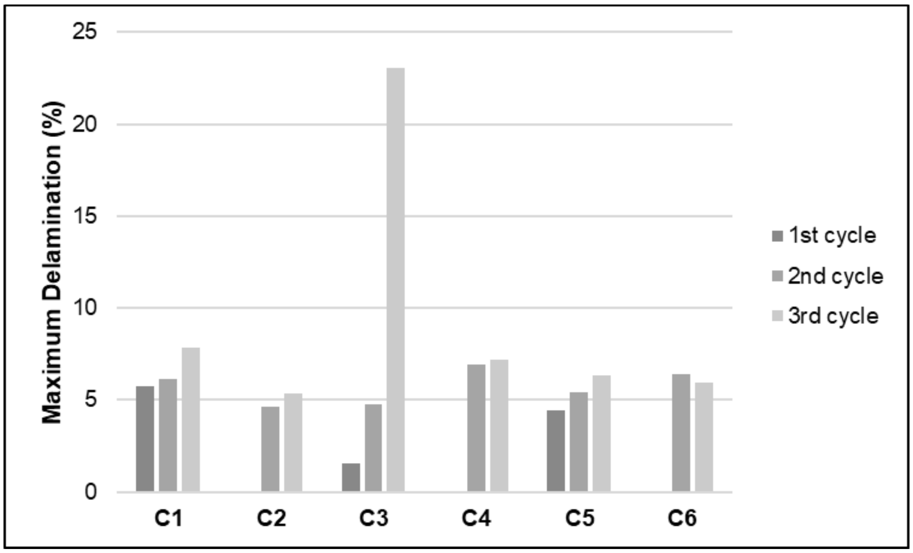

Fig. 5. Maximum delamination obtained after each vacuum-pressure cycle

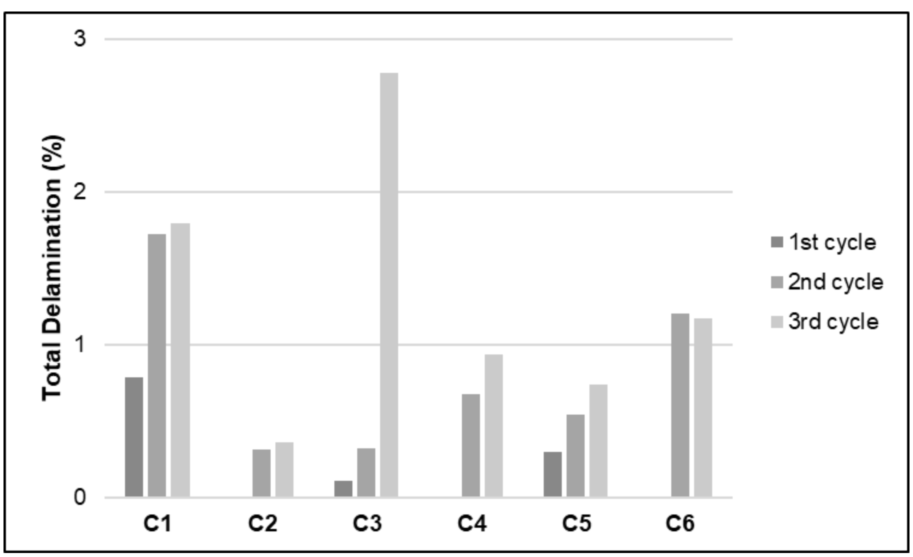

Fig. 6. Total delamination obtained after each vacuum-pressure cycle

Therefore, these results can be attributed to the negative effect of samples with high moisture contents, which affected the setting of the polyurethane and provided a weaker bond strength that increased delamination (Vella *et al.* 2019; Hänsel *et al.* 2021). The characteristics of the adhesive profile for the C3 combination was like those of the mixed combinations (C4, C5, and C6), as discussed previously, while the performances of the latter were comparable to the reference for maximum delamination and even slightly better for total delamination.

Although the good performance for the mixed combinations was achieved, their adhesive profile may be a concern for long-term applications due to their thinner glue profile distribution. Hence, the evaluation of accelerated aging and weathering for this type of element should be considered for future research projects.

CONCLUSIONS

1. The samples' moisture content before testing had more influence in the shear strength and delamination than the initial moisture content of the lamellae before bonding. The higher the moisture content was, the lower the bonding performance of the glulam.
2. Mixing lamellae with different initial moisture contents provided glulam with the same bonding performance of the reference, but with undesirable glue line profiles for the combinations tested.
3. The block shear strength and the delamination obtained, for all combinations evaluated of lamellae initial moisture content and RH climatizing conditions, respected the requirements imposed by the respective standards.
4. Analyzing the adhesive distribution in the interface region was possible with images obtained by the micro-CT scan method, which proved to be an interesting tool to assess the glue line profile, adhesive penetration, and wood interface densification.
5. Even if proper moisture content conditions before lamellas lamination is good practice, this study indicates that 2% moisture content variations have a limited effect on the glue line performance, especially for variation in a single glulam.

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