



Article **Feasibility of Bonding High-Moisture-Content Wood Using** *Nothofagus chilean* Species

Víctor Rosales ^{1,2}, Gonzalo Rodríguez-Grau ^{2,3,*}, Carlos Galarce ³, Claudio Montero ^{4,5}, Claudia Alvarado ^{3,6}, Lisa Muñoz ⁷, and Régis Pommier ⁸

- ¹ Department of Construction, Universidad del Bío-Bío, Concepción 4051381, Chile; vrosaleg@ubiobio.cl
- ² National Excellence Center for the Timber Industry (CENAMAD), Pontificia Universidad Católica de Chile, Santiago 7820436, Chile; Universidad del Bío-Bío, Concepción 4051381, Chile
- ³ School of Civil Construction, Faculty of Engineering, Pontificia Universidad Católica de Chile, Santiago 7820436, Chile; cegalarc@uc.cl (C.G.); claudia.alvarado.g@gmail.com (C.A.)
- ⁴ Adhesives & Composites Material Laboratory, Universidad del Bío-Bío, Concepción 4051381, Chile; cmontero@ubiobio.cl
- ⁵ Wood Design & Technology Laboratory, Universidad del Bío-Bío, Concepción 4051381, Chile
- ⁶ Centro de Investigación y Desarrollo en Ciencias Aeroespaciales, Fuerza Aérea de Chile, Av. José Miguel Carrera 11087, Santiago 8020744, Chile
- ⁷ Instituto de Química, Facultad de Ciencias, Pontificia Universidad Católica de Valparaíso, Avda. Universidad 330, Placilla (Curauma), Valparaíso 2373223, Chile; lisa.munoz@pucv.cl
- ⁸ Institut de Mécanique et D'Ingienierie (I2M), Arts et Métiers, University Bordeaux, UMR 5295, 351 Cours de la Libération, 33400 Talence, France; regis.pommier@u-bordeaux.fr
- * Correspondence: grodriguezg@uc.cl

Abstract: Appraising and protecting forests requires a management plan and the creation of innovative products for the market. The development of the green gluing technique could add value to native timber. However, there is a lack of knowledge concerning the response and the productive process of Nothofagus species using this technique. This work investigated the viability of implementing the green gluing method using three types of Nothofagus. Wood pieces were made using a one-component polyurethane adhesive. Delamination, shear tests, morphological characterization, and bond line thickness analysis tested their capacity. The results showed a variable response depending on the Nothofagus type, where the surface treatment could improve the green gluing performance. The findings highlight the relevance of increasing knowledge about the essayed species and their preparation to maintain their natural moisture condition.

Keywords: green gluing; Nothofagus; adhesion; polyurethane; wettability; wood surface

1. Introduction

Forests are critical habitats for biodiversity. They are also essential for providing various ecosystem services vital for human well-being. At the same time, forests provide a wide range of critically important ecosystem services such as climate regulation, biomass production, water supply and purification, pollination, and providing habitats for forest species [1]. One of the ways to protect the forest is by increasing species value through management plans and creating innovative products for the market and consumers.

The productive use of Chile's native forests is currently marginal in terms of exports but of great importance in timber production [2]. The industrial consumption of native wood has decreased since 1994 to the current day, from 4 MM of m³ to close to 0.5 MM of m³. The representative native species correspond to *Nothofagus alpina*, *Nothofagus obliqua*, and *Nothofagus dombeyi*, with more than 6 MM of ha together and a potential volume of 25 MM of m³ [2]. In particular, *Nothofagus alpina*, *Nothofagus obliqua*, and *Nothofagus dombeyi* are very interesting owing to their location and availability. Recently, Riquelme-Buitano et al. [3] have reported good experiences with plantations with native species, which have



Citation: Rosales, V.; Rodríguez-Grau, G.; Galarce, C.; Montero, C.; Alvarado, C.; Muñoz, L.; Pommier, R. Feasibility of Bonding High-Moisture-Content Wood Using *Nothofagus chilean* Species. *Forests* 2023, 14, 2386. https://doi.org/10.3390/f14122386

1111ps.//doi.org/10.5550/114122500

Academic Editors: Radosław Mirski, Jakub Kawalerczyk and Joanna Walkiewicz

Received: 30 October 2023 Revised: 10 November 2023 Accepted: 13 November 2023 Published: 7 December 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). shown a high productive potential for *Nothofagus alpina*, *Nothofagus dombeyi*, and *Nothofagus obliqua* [3].

The species *Nothofagus alpina* is an endemic tree of the sub-Antarctic forests of Chile and in fragmented form in Argentina. It occupies a total area of 1.6 MM of ha [4]. The wood of *Nothofagus alpina* is considered one of the most valuable native woods, so the species has been widely exploited. The sapwood is pinkish-yellow, and the heartwood is pinkish-brown with well-defined rings. The wood is semi-heavy and semi-hard, and resistant to bending and rotting (heartwood), with a nominal density of 531 kg/m³ [4].

Similarly, *Nothofagus obliqua* is a tree endemic to the sub-Antarctic forests of Chile and Argentina. It occupies an area of 1.8 MM, of which 1.3 MM of ha is in southern Chile. This species has a thick bark with dark-brown cracks in mature specimens and which is smooth and gray-white in young trees, with simple deciduous leaves 2–5 cm long and 1–2 cm wide with short petiole and lamina aovado-lanceolate. Its wood has high mechanical strength and durability [5] with a nominal density of 607 kg/m³ [4].

Finally, *Nothofagus dombeyi* is a monoecious heliophyte tree considered a colonizer. It is an evergreen species that covers an area of about 6.2 MM of ha. It is an easily workable wood with grayish-white sapwood and pale pinkish-white heartwood, and a fine and homogeneous texture with visible and well-differentiated annual rings, with a nominal density of 594 kg/m³.

Despite excellent mechanical properties, the trees' use is limited by their medium growth [6] and limited development of management plans. Besides this, due to the high proportion of Chile's native wood species sold as lumber, its favorable mechanical properties and durability are wasted, and only its calorific potential as fuel is used [2].

Green gluing is a method used with unseasoned, freshly sawn wood that has never been dried [7]. This means that the construction of glued laminated timber uses pieces of wood with a high moisture content (or green wood) and a polyurethane (PU) adhesive that works with this level of moisture [8]. This technique could help stabilize the wood dimensionally for raw materials considered lower quality to produce structural elements [8].

One of the factors driving the development of the technique is the possibility of reducing energy consumption for products associated with construction, as hardwood lumber requires a lot of energy in the drying process, which contributes to the environmental load [9], a topic of particular relevance today. Additionally, the green gluing technique can help to optimize raw materials by minimizing defects in wet wood while also promoting native wood species and opening new markets for manufacturing products [2,8].

Due to this, we propose to develop a green gluing technique that promotes using native trees to establish construction elements and reverse their inadequate use. In this way, the applicability of green gluing in native Chilean wood was evaluated. Delamination, shear tests, morphological characterization, and glue line thickness analysis were performed to assess the green gluing technique. The results are a first in using green gluing techniques in Nothofagus species.

2. Materials and Methods

The experimental procedure was divided into two steps. The first step was preparing the sample, and the second step was the application of diverse tests with the primary goal of probing the feasibility of the proposal study. Figure 1 summarizes the experimental design.

2.1. Origin of Raw Material

The wood sample laminate pieces used in the study were built with *Nothofagus dombeyi*, *Nothofagus alpina*, and *Nothofagus obliqua* from the Araucanía and Los Ríos Regions of Chile, purchased through Maderas Nalcahue and Maderas Martini. The humidity ranges studied were between 21.0% and 28.9%. Anhydrous woods were used as a control and the gold standard. The cut of wood used corresponds mainly to the tangential type.



Figure 1. Summary of the experimental design.

2.2. Characterization of Physical and Morphological Properties of Wood

2.2.1. Physical Parameters

The moisture content, reference, and oven-dry density were determined according to the procedures stipulated by the Chilean standard NCh176/1: 2003 [10] and the NCh176/2 standard 1986 Modified 1988 [11]. The samples were sized with an SCM brand square saw. To determine the dimensions and mass of the test pieces, a 0.01 mm precision Mitutoyo digital caliper and a Drawell 0.01 g precision balance were used. A Drawell brand drying oven was used, in which the samples were brought to an anhydrous state at 103 °C \pm 2 °C for 24 h.

The integrity or degradation of the wood pieces, their density, and some mechanical characteristics, such as the dynamic modulus, were measured by determination of the velocity of ultrasonic waves using a FAKOPP Microsecond Timer[©] (Agfalva, Hungary), designed explicitly for wood measurements by the Hungarian company Fakopp Enterprise (Agfalva, Hungary). The device measures the time (μ s) between the emission of an ultrasonic wave (23 kHz) generated by the emitting probe by a non-instrumented hammer blow (200 gr) and its reception by the receiving searcher. The time is measured with an accuracy of $\pm 1 \mu$ s [12] and displayed on a digital display. The basis of the ultrasonic test is the passage of an ultrasonic wave through the wood. The ultrasonic wave will take the most direct path between emitting and receiving points. Its propagation speed will initially depend on the elastic properties of the material through which it passes [12,13].

2.2.2. Micro-Computed Tomography (Micro-CT)

A high-resolution Bruker SkyScan 1272 microtomograph (Kontich, Belgium) was used with a rotation of 0.4 at 80 kV, 125 mA, utilizing an aluminum filter of 0.25 mm and a voxel size of 12 μ m resolution. Three-dimensional scanned images were obtained using NRecon reconstruction software (Version 3.1.1). The images were reoriented in space using DataViewer software v1.5.1.9 to standardize sample positioning. Quantitative assessments were performed using micro-CT and software analysis in a volume of interest (VOI) of approximately 2 cm³ in the transversal plane. Finally, images of each sample were obtained using visualization software. The pixel size was 24 μ m. Voxels in the 3D image with grayscale values between 25 and 255 were considered solid material, while voxels with values between 24 and 0 were considered void space [14].

Image processing and analysis were performed with Image J software version 154e (The National Institutes of Health, Bethesda, MD, USA). Images obtained from micro-CT were corrected by removing the background. The corrected image was then converted to a binary black-and-white format for postprocessing and parameter estimation. The thresholding procedure used was the iterative selection method. Finally, the percent porosity was the black pixels divided by the total pixels times one hundred [15].

2.2.3. Contact Angle Testing

The physicochemical properties of the surface were determined through contact angle measurements of the samples at 25 °C, using the sessile drop method, or static drop, using a contact angle device (Drop Shape Analyzer DSA25S; KRUSS, Hamburg, Germany) controlled by AVANCE software version 1.5.1 (KRUSS, Germany). The procedure consists of depositing on the surface perpendicular to the grain 8 μ L of water drops and 8 μ L of diiodomethane drops in triplicate, thus establishing the components, polar and dispersed, obtaining the surface free energy and contact angle [16]. The dry and wet woods were used to measure the contact angle.

2.3. Manufacture of Green Glued Laminate

Wood samples from three different species were collected from southern Chile. Dry and wet heartwood without defects or knots was used. Lamellae were cut to have clean and flat surfaces, with an average size of 20 mm thickness, 60 mm width, and 60 mm length. Gluing operations were conducted in laboratories at the Pontificia Universidad Católica and Universidad del Bío-Bío, where the moisture content of the wood during gluing varied from $23 \pm 2\%$ (around the FSP for each species [17]). Through experience, part of the timber was conditioned to preserve the moisture, while the other part was dried naturally.

Ten laminated blocks of wood (bilaminated) were manufactured for each species, each shaft consisting of two lamellae by the conditioned samples. At the same time, six laminated blocks of dry and six laminated blocks of moisture-native species were used. A spatula applied a 150 g/m² adhesive rate to each contact surface. The correct amount of adhesive was determined based on the dimensions and weight of the wood piece. The glue was applied to the wood piece, which was then weighed. The open and closed times were 1 min and less than 7 min, respectively. A clamping pressure of approximately 0.6 MPa was maintained for two hours, and gluing and pressing operations were carried out in a controlled environment of 20 ± 3 °C and relative moisture of less than $60 \pm 5\%$. After the binding phase, the laminate was stabilized in an environmentally controlled environment at $20\% \pm 3\%$ °C. The maximum time between planning and gluing operations was approximately 15 min.

2.4. Mechanical Tests

2.4.1. Shear Test

Dry and wet samples were tested in a compression-loading shearing tool described in the ASTM Method D905 using a universal uniaxial reaction press with a controlled displacement control system and a maximum tensile and compression capacity of 10,000 Kgf. The model of the press was TC10001, and it was made by a local manufacturer. The cutting device was manufactured to meet the requirements of ASTM D 905 [18]. The device's geometry is provided with a transverse ball-joint system to guarantee a shear stress in the gross cross-section. The machine maintains a uniform loading rate. The load may be applied with a continuous motion of the movable head at a rate of 0.6 mm/min with a permissible variation of less than 25%. The test specimens were obtained after cutting an extensive laminate. The size of each sample was 60 mm in length by 60 mm in width by 40 mm in height. The maximum load at failure was recorded, and then shear strength was calculated for each specimen based on the shear area. For the evaluation of delamination at the glue line in the bilaminated Nothofagus beams, 16 samples were used. These samples, $40 \times 40 \times 75 \text{ mm}^3$, were prepared by the Laboratory of Wood Construction and Recycled Materials of the Pontifical Catholic University of Chile. The Adhesives and Composite Materials Laboratory of the Universidad del Bío-Bío tested the samples. The reference standard used to evaluate the delamination at the glue line was NCh 2148:2013 [19], extracted from ISO 12580/2007 [20]. The glued lines of each specimen were measured with a 0.01 mm precision ruler. The samples were then weighed on a balance with an accuracy of 0.01 g. The specimens were subjected to a vacuum of 70 kPa for 0.5 h and a water pressure of 600 kPa for 2 h. Finally, they were dried in an oven at 70 °C for 15 h or until the specimens reached 100% to 115% of the initial mass. The samples were then placed in a desiccator, and the delamination generated by the applied cycle was measured. This procedure allowed the total delamination to be determined as a percentage (%) of the complete delamination divided by the length of the glued line measured before the cycle was applied. The following formula was used to calculate this percentage:

Total delamination (%) =
$$\frac{l_{tot, delam}}{l_{tot,glue \ line}} \times 100$$

where:

 $l_{tot, delam}$: Delamination length of glue lines on the two end grain surfaces in the test piece (mm).

 $l_{tot, glue line}$: Length of the glued lines before the delamination cycle (mm).

2.5. Statistical Analysis

A one-way ANOVA was conducted using Excel (Microsoft, 2023) to compare species under each condition. Significant differences (p < 0.05) were noted on graphs.

3. Results

3.1. Characterization of Physical Properties

High quality and stiffness were evident from the physical properties of each species. Table 1 summarizes the average values for each wood. The modulus of elasticity (MOE) is one of solid wood's most critical mechanical properties determining a cut piece's structural performance and market value [21,22]. Elastic properties relate the material's resistance to deformation under applied stress to the ability of the material to return to its original dimensions when the pressure is removed. In this sense, the Nothofagus specimens showed a high MOE value, confirming its good mechanical properties. At the same time, the density value allowed the categorization of the timber with a medium level. No significant differences in the propagation velocity were observed, which could provide clues about the similar internal structure.

Table 1. Summary of the physical properties of the Nothofagus species used.

Specie	Properties at 6% MC	Density (kg/m ³)	MOE (GPa)
Nothofagus alpina	Mean	571.2	8.80
	SD	47.3	2.4
	Cov (%)	8.3	26.9
	Mean	568.5	8.84
Nothofagus obliqua	SD	34.7	3.4
	Cov (%)	6.1	39.02
Nothofagus dombeyi	Mean	598	10.2
	SD	48.2	1.5
	Cov (%)	8.1	14.4

SD: Standard deviation. Cov: Variation coefficient.

3.2. Mechanical Tests

3.2.1. Shear Test

Mechanical tests were performed on the different laminated samples of naturally dried and conditioned wood. Figure 2 shows the shear test results for the naturally dried samples. They all exceed the standards established for use as construction material, presenting average values of higher than 9 N/mm².



Figure 2. Shear test values of different native species naturally dried. The dashed line indicates the standard value required.

The laminated *Nothofagus alpina* samples constructed with conditioned woods presented the best results among the wood tested. The average of the shear tests yielded a value of 6.82 N/mm² with a standard deviation of 1.6, with a 58.3% success rate for moisture range (21%–28.9%). However, this value increased to 7.42 N/mm² \pm 0.55 with a 77.8% success rate for samples with less than 24% moisture content. The rest of the species showed lower values than the norm demands (Figure 3).



Figure 3. Shear test values of conditioned native species. The dashed line indicates the standard value required.

Finally, the results were not good when using wood with high moisture content directly from the forest, where the shear tests of the three species presented still lower values (Figure 4).



Figure 4. Shear test values using moisture timber of native species. The dashed line indicates the standard value required.

3.2.2. Delamination Test

Samples of all types of wood naturally dried were used for delamination tests. The results indicate that *Nothofagus obliqua* did not pass the delamination test using naturally dried wood, showing an average delamination of 41.54% for a diverse range of 21% to 29% for moisture. On the other hand, *Nothofagus alpina* showed a delamination average value of 15.17% and the value for *Nothofagus dombeyi* was 11.22%, reaching the delamination probes (Figure 5A).



Figure 5. Summary of delamination test value. (A) Results using dry and (B) native timber conditioned. The asterisk indicates a significative difference (p < 0.05). The dashed line indicates the standard value required.

Because the *Nothofagus alpina* samples constructed with conditioned woods were the only samples of this condition that approved the mechanical test, these were considered in the delamination tests. However, the results were unfavorable and showed a high delamination value, with an average of around 80% (Figure 5B).

3.3. Morphological Characterization

3.3.1. Characterization by Microcomputed Tomography (Micro-CT)

Micro-CT was carried out for the internal morphology of the laminates of native wood. The image shows two tangential cut pieces joined together and one corresponding to the glued line. The laminates made with naturally dried wood showed a similar and uniform pore distribution in the analyzed piece's transverse layers (Figure 6). However, the porosity values varied among the other native woods, as seen in Table 2.



Figure 6. Distribution of porosity into laminates of naturally dried *Nothofagus alpina* (**A**) and *Nothofagus alpina* (**B**) by micro-CT.

Specie	Average Porosity (%)	Average Porosity in Glue Line (%)	
Nothofagus alpina	59.1	38.6	
Nothofagus obliqua	43.1	27.7	
Nothofagus dombeyi	57.7	37.9	

Table 2. Average porosity of the wood species and adhesive lines using dry native timbers.

Despite the differences in porosity of each species, it can be seen that the penetration of the adhesive was similar, covering approximately 20% of the pores in each laminate. With these results, a similar performance in the mechanical tests regarding adhesive bond strength is expected. On the other hand, the glue line analysis shows that the average thickness of all the laminates made with dry native wood was about 232 μ m.

A similar analysis was conducted for the samples that passed the expected values in the shear tests with conditioned *Nothofagus alpina*. The results show a lower percentage of porosity associated with a larger pore size, giving an average porosity value of 34.64%. Another change observed was the thickness of the glued line, which was approximately 400 μ m (Figure 7).



Figure 7. Distribution of porosity into the glue line of dry *Nothofagus alpina* (**A**) and conditioned *Nothofagus alpina* (**B**) using the micro-CT technique.

3.3.2. Measurement of the Surface Wettability of Wood by the Contact Angle Test

The contact angle and its influence on adhesion were evaluated using the sessile drop technique [23] (Figure 8). The surface energies of the dispersed and polar components are presented in Table 3. The contact angle and its influence on adhesion were evaluated using the sessile drop technique, according to Young's equation (Equation (1)) [24]

$$\cos\theta = \frac{\gamma SG - \gamma SL}{\gamma LG} \tag{1}$$

where θ represents the contact angle; and γLG , γSG , and γSL symbolize the liquid–vapor, solid–vapor, and solid–liquid interfacial tensions, respectively.



Figure 8. Image of a drop of water in contact with the surface of the wood before gluing with naturally dried timber at a temperature of 20 °C, with a detection time of 3 s for (**A**) *Nothofagus alpina*, (**B**) *Nothofagus obliqua*, and (**C**) *Nothofagus dombeyi*.

Table 3. Summary of superficial characteristics of samples made with dry wood. Features of the types of pieces in terms of physical properties: free energy (γ) and polar (γ^{p}_{S}) and dispersive (γ^{d}_{S}) components obtained by static contact angle measurements (θ) in water and diiodomethane.

Specie	θ (°)	γ (mN/m)	$\gamma^{d}{}_{S}$ (mN/m)	$\gamma^{P}{}_{S}(mN/m)$
Nothofagus alpina Nothofagus obliqua	54.00 40.55	$\begin{array}{c} 50.29 \pm 0.44 \\ 57.97 \pm 6.00 \end{array}$	$\begin{array}{c} 29.02 \pm 0.23 \\ 28.02 \pm 0.77 \end{array}$	$\begin{array}{c} 21.27 \pm 0.21 \\ 29.96 \pm 5.00 \end{array}$
Nothofagus dombeyi	65.60	53.76 ± 14.00	40.87 ± 8.00	12.89 ± 6.00

The surface energies of the dispersed and polar components are presented in Table 3. The surfaces were superficially evaluated in their condition before gluing.

For the samples that naturally dried, the highest contact angle was obtained on the *Nothofagus dombeyi* surface, with a value of 65.50°, followed in decreasing order by *Nothofagus* alpina with a value of 52.40° and *Nothofagus obliqua* with 40.86°. The surface free energy was higher on the *Nothofagus alpina* surfaces. In decreasing order, it was followed by *Nothofagus dombeyi* and, finally, *Nothofagus obliqua*, as presented in Table 3.

Very similar contact angles were obtained for the surfaces analyzed with high moisture. For *Nothofagus alpina*, a contact angle of 54.92° was obtained, and for *Nothofagus obliqua*, it was 59.30°. No data were recorded for *Nothofagus dombeyi* due to its high wettability (Figure 9). A summary is presented in Table 4.



Figure 9. Image of a drop of water in contact with the wood surface before gluing with naturally moist timber at a temperature of 20 °C, with a detection time of 3 s for (**A**) *Nothofagus alpina* and (**B**) *Nothofagus obliqua*.

Table 4. Summary of superficial characteristics of samples made with high moisture content. Features of the types of pieces in terms of physical properties: free energy (γ) and polar (γ^{p}_{S}) and dispersive (γ^{d}_{S}) components obtained by static contact angle measurements (θ) in water and diiodomethane.

Specie	θ (°)	γ (mN/m)	$\gamma^{d}{}_{S}$ (mN/m)	$\gamma^{P}{}_{S}$ (mN/m)
Nothofagus alpina	60.30	54.92 ± 2.20	41.085 ± 0.31	13.08 ± 1.89
Nothofagus obliqua	59.30	39.16 ± 4.07	30.15 ± 3.98	0.01 ± 0.09
Nothofagus dombeyi	n.d.	n.d.	n.d.	n.d.

n.d.: Not determined.

When we analyze the surface of the naturally dried glued species, we observe that *Nothofagus obliqua* has the highest surface energy but is very similar to *Nothofagus dombeyi*. The result would be related to a better condition for accession. In the case of wet glued wood, the surface free energy naturally changes, with *Nothofagus alpina* being the species with the highest energy.

Jankowska and colleagues found that a lower contact angle value indicates a higher roughness of wood, leading to a higher wettability [25]. It is important to note that using a sanded surface can increase wettability due to the activated hydrophilic group. This situation can produce voids filled with powder, altering the roughness and changing the contact angle.

4. Discussion

The results of this study are the first steps in establishing the technical feasibility of green-glued laminated hardwoods of the Nothofagus species harvested in Chile. The results highlight the behavior of the bond lines when the gluing parameters are constant, while their moisture content varies at 24%.

The shear tests showed a good performance of the pieces made with dry wood, with values exceeding the established Chilean standard. However, some failures were observed in the delamination test by the *Nothofagus obliqua* specimens, which did not reach the minimum required. On the other hand, the shear test results did not show the desired performance using conditioned timbers to 24% MC. The *Nothofagus alpina* species achieved the best value, averaging 6.82 N/mm² (Figure 3). At the same time, a low average weight and a high data dispersion in *Nothofagus obliqua* and *Nothofagus dombeyi* were observed compared with the results using dry timber. Previously, Setter et al. [26] indicated that the forest species' density and porosity influence the mechanical resistance to shear. In the same way, Alia-Syahirah et al. [27] reported that a higher density can negatively affect

adhesion in wood products. Despite these preceding studies, wood density was discarded as a negative factor because all the species showed a similar density value. The porosity, wettability, and roughness were probably more responsible for the results seen.

Porosity and density are essential parameters that significantly influence wood's material properties, such as flow, adsorption, impregnability, conductivity, and tensile and bending strength [28]. Consequently, the porosity and internal wood structure of the species used were examined using micro-CT. The analysis revealed no form or internal order distinctive among the species tested. Nevertheless, a modification of porosity during wood conditioning cannot be discarded. Wood is a material that can swell or shrink due to changes in its moisture content because it is non-homogeneous and hygroscopic [29]. During the drying process, the fibers are connected in a water-covered cluster. The free water menisci will retreat during the drying process but will halt at the small openings of bordered pits between the fibers. As the drying process continues, the meniscus in the widest gap cannot withstand the capillary suction and retreats into the corresponding fiber, which gradually empties and repeats the process [30]. That could mean that the moisture content in the conditioned wood pieces could have affected adhesive wettability produced by modifying pore size [31].

The adhesion process involves various steps. The first is wettability, the adhesive's capacity to establish contact with the wood surface. This property plays a crucial role in the ability of liquid sealants to spread and infiltrate wood cells, ultimately impacting the bonding strength between the two surfaces [27]. In this step, the adhesive molecules interact with the wood surface through molecular forces such as Van der Waals, hydrogen bonding, and dipole–dipole interactions [32]. After that, the adhesive penetrates the wood surface's pores, filling up the voids and crevices. Finally, the adhesive solidifies, forming a bond between the two surfaces. In the case of PUR adhesive, solidification occurs when the glue cools down and creates an initial bond with the wood surface. After that, it starts reacting with moisture in the air, which causes it to expand slightly and form a strong bond between the covers. However, this process could be modified in the sample using conditioned lumber.

Fibers' uneven distribution and orientation can create internal tension, making adhesion difficult and weakening the bond strength. A product's strength and durability depend heavily on its bonding properties, particularly the shear bond strength and bond stiffness. These properties can be influenced by several factors, including the direction of the bond, the way lamellae are combined, and the type of failure mode [33]. According to a study conducted by Li et al. [33], bonded wood samples with uniform fiber orientation and wood species in all plies exhibited higher bond strength and stiffness compared to samples where the wood species used in the outer plies differed from the wood used in the inner plies, leading to a mixed failure mode. In the same way, Jakob et al. [34] reported that even slight deviations in fiber orientation during the assembly of intermediates can cause a loss in strength or stiffness due to re-assembly by adhesive bonding. This highlights the significant impact of fiber orientation on the mechanical properties of wood products. Additionally, the article emphasizes the importance of unidirectional fiber orientation in achieving high composite strength and stiffness, indicating that fiber orientation plays a crucial role in the mechanical performance of composite materials. The different porosity of wood species and adhesion in different directions probably influenced the mechanical performance of our study.

Based on the micro-CT results, the glue line's thickness in conditioned lumber was twice that of dry timber. In the research of Lissouck et al. [35], similar values of bond line thickness using a block of wood with similar density in a dry state were reported. The adhesion mechanism was probably significantly affected by moisture distribution and the drying process. The findings suggest that in conditioned pieces with an inverted moisture gradient (i.e., the surface moisture content level is higher than that in the center), the glue interacts with the superficial water, reducing the adhesive–wood interaction. Although adhesive penetration occurs, the reaction mainly occurs with water instead of wood, resulting in a weak union.

Understanding surface roughness is essential for achieving successful adhesion when gluing wood products. Studies such as Alia-Syahirah et al. [27] have shown that surface roughness can significantly affect adhesive penetration and contact angle. Additionally, surface roughness has been identified as a critical factor in wood's surface wettability [36]. It is crucial to consider the relationship between surface treatment, roughness, and their effects on contact angle to achieve optimal adhesive bonds [36,37]. In this sense, the contact angle results showed that the behavior of the specimens varied depending on the condition of the wood used. Contact angle values were smaller for dry wood. A high surface tension on dry wood probably modified the contact angle value.

This behavior was reported previously in another type of wood. Benkreif et al. [38] reported that changes in moisture content significantly affect the angle and surface tension values of sanded birch wood surfaces. Specifically, as moisture content increases, the contact angle of sanded birch samples increases logarithmically while the surface tension decreases logarithmically. These results have significant implications for the wettability and adhesion of adhesives and wood coatings on birch wood surfaces. A similar phenomenon occurred in the samples using wood conditioning, where the moisture content on the surface could be higher owing to the conditioning process itself, which might explain the unexpected results of this research.

The contact angle could change depending on diverse scenarios. When surfaces are rough, two states can occur: the Wenzel and Cassie–Baxter states. In the Wenzel state, the surface under the liquid is wet, which increases the contact area between solid and liquid [39]. This results in an increase in the apparent contact angle for hydrophobic surfaces and a decrease for hydrophilic surfaces. In the Cassie–Baxter state, air bubbles get trapped between the liquid droplets and the solid surface [39]. According to the characteristics of the Cassie–Baxter state, more porous and heterogeneous surfaces have a greater contact angle due to air bubbles [39,40]. Therefore, the trapped air bubbles at the surface–liquid interface could explain the greater contact angle observed in some samples.

Moisture content plays a crucial role in the curing process of adhesives, and it is imperative to analyze its effect. When water is present during adhesive curing, it initiates chemical reactions that promote the formation of strong bonds. Adhesives such as moisture-curing adhesives require moisture to start the curing process. The moisture in the environment or on the substrate surface reacts with specific chemical groups in the glue, such as isocyanate or silane groups, to form cross-links and create a strong bond [41]. The strength of adhesion was likely affected by the moisture profile in the conditioned and natural wet samples, which decreased its performance. Additionally, the cellular structure of softwoods and hardwoods influences the penetration of 1C PUR adhesive, as shown by Shirmohammadli et al. [42]. Therefore, it is impossible to discern the influence of porosity on the results. Finally, a multifactorial study must be carried out, considering the effect of the anatomical structure of wood, moisture, surface treatment, and gluing procedure to understand the process better.

5. Conclusions

Green gluing is a promising technique for increasing the value of various wood species. Despite their desirable mechanical properties, Nothofagus species are not available in the required size, so developing this technique could increase its use. This study highlights the importance of wood surfaces in achieving optimal performance with green gluing supported by the contact angle results. The moisture profile on the wood should be considered since this could change the penetration grade of some adhesives. At the same time, our experience indicates that proper management and maintenance of timber moisture impacts the adhesion process. Finally, more studies about the anatomical structure of Nothofagus will help to comprehend the disparate behavior observed in this study's mechanical tests. Future research using recently harvested wood and varying amounts of adhesive may provide improved outcomes.

Author Contributions: Conceptualization, G.R.-G. and V.R.; methodology, R.P., G.R.-G., V.R., C.G. and C.M.; formal analysis, C.M. and C.G.; investigation, C.G., C.A., L.M. and C.M.; resources, G.R.-G., V.R. and L.M.; writing—original draft preparation, C.G. All authors contributed to the review and editing of the manuscript. All authors have read and agreed to the published version of the manuscript.

Funding: The authors would like to acknowledge funding from Centro Nacional de Excelencia para la Industria de la Madera (CENAMAD, FB210015). In particular, Víctor Rosales, Gonzalo Rodríguez-Grau, Claudio Montero, Claudia Alvarado, Carlos Galarce, and Régis Pommier thank the financial support of ANID + FONDEF/Concurso IDeA I+D, FONDEF/ANID 2021 ID 21 | 10241.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Acknowledgments: The authors would like to acknowledge the technical support of Maria Jose Salas (FONDEQUIP project EQM 130028) in the micro-CT measurements. Moreover, the authors thank Camilo Guzmán and Sergio Valenzuela for their collaboration and support in the mechanical tests and preparation of samples.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Brockerhoff, E.G.; Barbaro, L.; Castagneyrol, B.; Forrester, D.I.; Gardiner, B.; González-Olabarria, J.R.; Lyver, P.O.; Meurisse, N.; Oxbrough, A.; Taki, H.; et al. Forest Biodiversity, Ecosystem Functioning and the Provision of Ecosystem Services. *Biodivers. Conserv.* 2017, 26, 3005–3035. [CrossRef]
- 2. Rodríguez-Grau, G.; Pinto-González, F.; Cortés-Rodríguez, P.; Marín-Uribe, C. Native Wood Revaluation through Green Gluing: A Systematic Review. In *World Conference on Timber Engineering*; WCTE: Chile, Santiago, 2021.
- Riquelme-Buitano, T.; González, P.F.O.; Donoso, P.J. Comparing Growth of Nothofagus Alpina and Nothofagus Obliqua in Pure and Mixed Plantations in the Intermediate Depression of the Los Ríos Region, Chile. *Bosque* 2023, 44, 263–272. [CrossRef]
- 4. Instituto Forestal. Memoria INFOR 2019; INFOR: Sino Plaza, Hong Kong, 2020.
- Juacida, P.R.; Siebert, W.H.; Torres, U.M.; Edad, Y. Durabilidad Natural de Renovales de Nothofagus Obliqua. *Bosque* 2000, 21, 73–77. [CrossRef]
- 6. Ugarte Hidalgo, M. Caracterización Del Crecimiento En Renovales de Roble (Nothofagus Obliqua (Mirb). Oerst) En La Comuna de Curacautín, Región de La Araucanía; Universidad de Chile: Santiago, Chile, 2014.
- 7. Sterley, M. Green Gluing of Wood; KTH-Royal Institute of Technology: Stockholm, Sweden, 2004.
- Rodríguez-Grau, G.; Marín-Uribe, C.; Cortés-Rodríguez, P.; Montero, C.; Rosales, V.; Galarce, C. Bibliometric Analysis of the Green Gluing Technique (2000–2020): Trends and Perspectives. *Forests* 2022, *13*, 1714. [CrossRef]
- 9. Sultana, R.; Rashedi, A.; Khanam, T.; Jeong, B.; Hosseinzadeh-Bandbafha, H.; Hussain, M. Life Cycle Environmental Sustainability and Energy Assessment of Timber Wall Construction: A Comprehensive Overview. *Sustainability* **2022**, *14*, 4161. [CrossRef]
- 10. *NCh176/1;* Madera-Parte 1: Determinación Del Contenido de Humedad. Instituto Nacional de Normalización: Santiago, Chile, 2019.
- 11. NCh176/2; Madera-Parte 2: Determinación de Densidad. Instituto Nacional de Normalización: Santiago, Chile, 1988.
- Balmori Roiz, J.A.; Acuña Rello, L.B.; Otero, L.A. Estudio de La Influencia de La Dirección de La Fibra En La Velocidad de Propagación de Ultrasonidos (FAKOPP) En Madera Estructural de "Pinus Sylvestris L. y "Pinus Radiata D. Don.". In Proceedings of the 6th Rehabend Congress, Burgos, Spain, 24–27 May 2016.
- 13. Grabianowski, M.; Manley, B.; Walker, J.C.F. Acoustic Measurements on Standing Trees, Logs and Green Lumber. *Wood Sci. Technol.* **2006**, *40*, 205–216. [CrossRef]
- 14. Romano-Matos, V.; Tundidor-Camba, A.; Vera, S.; Videla, A.R. Improvement of Plasterboard Properties by the Control of Polymethylhydrosiloxane Dosage, Stirring Time, and Drying Temperature Applied to the Calcium Sulfate Hemihydrate and Water Mixture. *Materials* **2023**, *16*, 5084. [CrossRef]
- Kazup, Á.; Fegyverneki, G.; Gácsi, Z. Evaluation of the Applicability of Computer-Aided Porosity Testing Methods for Different Pore Structures. *Metallogr. Microstruct. Anal.* 2022, 11, 774–789. [CrossRef]
- Qin, Z.; Gao, Q.; Zhang, S.; Li, J. Surface Free Energy and Dynamic Wettability of Differently Machined Poplar Woods. *Bioresources* 2014, 9, 3088–3103. [CrossRef]
- 17. Popper, R.; Niemz, P.; Eberle, G. ETH Library Influence of Extractives on Water Vapour Sorption by the Example of Wood Species from Chile Influence of Extractives on Water Vapour Sorption by the Example of Wood Species from Chile. *Wood Res.* **2006**, *51*, 95–96.

- 18. *ASTM* (*D905*); Standard Test Method for Strength Properties of Adhesive Bonds in Shear by Compression Loading. ASTM International: West Conshohocken, PA, USA, 2021.
- 19. NCh 2148; Madera Laminada Encolada Estructural—Requisitos, Métodos de Muestreo e Inspección. Instituto Nacional de Normalización: Santiago, Chile, 2013.
- ISO 12580:2007; Timber Structures—Glued Laminated Timber—Methods of Test for Glue-Line Delamination. ISO: Geneva, Switzerland, 2007.
- Yang, J.L.; Evans, R. Prediction of MOE of Eucalypt Wood from Microfibril Angle and Density. *Holz als Roh—Und Werkst.* 2003, 61, 449–452. [CrossRef]
- 22. Kumar, C.; Psaltis, S.; Turner, I.; Brancheriau, L.; Hopewell, G.; Lee, D.; Bailleres, H. *Predicting Performance of Sawn Boards by Using Transverse Cores of Standing Trees*; University of Queensland: Brisbane, Australia, 2019.
- 23. Cordeiro, L.A.; Miranda, B.D.; Carneiro, M.E.; Missio, A.L.; Klock, U.; Cademartori, P.H.G.D. The Effect of Sanding on the Wettability and Surface Quality of Imbuia, Red Oak and Pine Wood Veneers. *Maderas. Cienc. y Tecnol.* 2023, 25. [CrossRef]
- Hebbar, R.S.; Isloor, A.M.; Ismail, A.F. Contact Angle Measurements. In *Membrane Characterization*; Elsevier Inc.: Amsterdam, The Netherlands, 2017; pp. 219–255. ISBN 9780444637918.
- Jankowska, A.; Zbieć, M.; Kozakiewicz, P.; Koczan, G.; Oleńska, S.; Beer, P. The Wettability and Surface Free Energy of Sawn, Sliced and Sanded European Oak Wood. *Cienc. y Tecnol.* 2018, 20, 443–454. [CrossRef]
- Setter, C.; Lira Zidanes, U.; Hélio de Novais Miranda, E.; Maria Silva Brito, F.; Marin Mendes, L.; Benedito Guimarães Junior, J. Influence of Wood Species and Adhesive Type on the Performance of Multilaminated Plywood. *Environ. Sci. Pollut. Res.* 2021, 28, 50835–50846. [CrossRef]
- Alia-Syahirah, Y.; Paridah, M.T.; Hamdan, H.; Anwar, U.M.K.; Nordahlia, A.S.; Lee, S.H. Effects of Anatomical Characteristics and Wood Density on Surface Roughness and Their Relation to Surface Wettability of Hardwood. *J. Trop. For. Sci.* 2019, *31*, 269–277. [CrossRef]
- 28. Plötze, M.; Niemz, P. Porosity and Pore Size Distribution of Different Wood Types as Determined by Mercury Intrusion Porosimetry. *Eur. J. Wood Wood Prod.* 2011, 69, 649–657. [CrossRef]
- Jang, E.-S.; Kang, C.-W. Changes in Gas Permeability and Pore Structure of Wood under Heat Treating Temperature Conditions. J. Wood Sci. 2019, 65, 37. [CrossRef]
- 30. Salin, J.G. Drying of Liquid Water in Wood as Influenced by the Capillary Fiber Network. *Dry. Technol.* **2008**, *26*, 560–567. [CrossRef]
- Arai Mizutani, K.M.; Oshima, K.; Iida, I. Physical and Mechanical Properties of Wood after Moisture Conditioning. J. Wood Sci. 2001, 47, 185–191.
- 32. Bockel, S.; Harling, S.; Konnerth, J.; Niemz, P.; Weiland, G.; Hogger, E.; Pichelin, F. Modifying Elastic Modulus of Two-Component Polyurethane Adhesive for Structural Hardwood Bonding. *J. Wood Sci.* **2020**, *66*, 69. [CrossRef]
- 33. Li, X.; Ashraf, M.; Kafle, B.; Subhani, M. Effect of Fibre Orientation on the Bond Properties of Softwood and Hardwood Interfaces. *Buildings* **2023**, *13*, 1011. [CrossRef]
- Jakob, M.; Mahendran, A.R.; Gindl-Altmutter, W.; Bliem, P.; Konnerth, J.; Müller, U.; Veigel, S. The Strength and Stiffness of Oriented Wood and Cellulose-Fibre Materials: A Review. *Prog. Mater. Sci.* 2022, 125, 100916. [CrossRef]
- 35. Lissouck, R.O.; Ngono Mvondo, R.R.; Ateba, F.R.; Pommier, R. Investigation of Green-Glued Laminated Timber from the Congo Basin: Durability, Mechanical Strength and Variability Trends of the Bondlines. *South. For.* **2022**, *84*, 225–241. [CrossRef]
- Wang, J.; Wu, Y.; Cao, Y.; Li, G.; Liao, Y. Influence of Surface Roughness on Contact Angle Hysteresis and Spreading Work. *Colloid.* Polym. Sci. 2020, 298, 1107–1112. [CrossRef]
- 37. Sinderski, L.G.Z. Contact Angle and Roughness of Wood, a Short Review. Ciência da Madeira 2020, 11, 1–11. [CrossRef]
- Benkreif, R.; Brahmia, F.Z.; Csiha, C. Influence of Moisture Content on the Contact Angle and Surface Tension Measured on Birch Wood Surfaces. *Eur. J. Wood Wood Prod.* 2021, 79, 907–913. [CrossRef]
- Milne, A.J.B.; Amirfazli, A. The Cassie Equation: How It Is Meant to Be Used. Adv. Colloid. Interface Sci. 2012, 170, 48–55. [CrossRef]
- Bobji, M.S.; Kumar, S.V.; Asthana, A.; Govardhan, R.N. Underwater Sustainability of the "Cassie" State of Wetting. *Langmuir* 2009, 25, 12120–12126. [CrossRef]
- Xiang, H.; Xia, H.; Chen, H.; Mi, H. Influence of Moisture-Curing Reaction on the Mechanical Properties of One-Component Polyurethane Mixtures. J. Mater. Sci. 2021, 56, 15069–15086. [CrossRef]
- 42. Shirmohammadli, Y.; Pizzi, A.; Raftery, G.M.; Hashemi, A. One-Component Polyurethane Adhesives in Timber Engineering Applications: A Review. *Int. J. Adhes.* **2023**, *123*, 103358. [CrossRef]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.