WOOD RESEARCH doi.org/10.37763/wr.1336-4561/67.4.598611
e-ISSN 2729-8906
67(4): 2022 598-611 pp.

STUDY OF WOOD ADHESIVES ON THE BONDING PROPERTIES IN SOLID AND HOLLOW GLULAM BEAMS OF *PINUS RADIATA*

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(RECEIVED JANUARY 2022)

ABSTRACT

The aim of this research was study of polyurethane (PUR), isocyanate polymer emulsions (EPI) and melamine-urea-formaldehyde (MUF) adhesives, on the bonding properties of solid and hollow glulam beams of Pinus radiata. The thermomechanical analysis (DMA) of the adhesives was carried out to evaluate their stiffness and reactivity. Glulam beams were evaluated by a bending test. The quality of the bonding was evaluated by resistance to shear and delamination. The morphology of the bonding was studied by microscopy. The DMA study showed that the MUF adhesive had the highest level of stiffness and reactivity. The results of the bending test showed that the highest modulus of rupture results were obtained in solid and hollow laminated beams with MUF adhesive, achieving increases of 30% over the PUR adhesive. The lowest delamination results were obtained in solid glulam beams with MUF and EPI adhesives, while the highest results were 32% and 47% for the PUR adhesive. Finally, glulam beams manufactured with MUF adhesive presented the best performance and results.

KEYWORDS: Wood adhesive, bonding properties, glulam, *Pinus radiata*.

INTRODUCTION

In Chile, forest plantations cover approximately 2.3 million hectares, equivalent to 13.6% of the country's total forests, according to figures from the 2020 Forestry Yearbook. Of this area, approximately 56% corresponds to radiata pine and 37% to eucalyptus species, located mainly between the O'Higgins and Los Ríos regions (INFOR).

According to FAO data, in 2019 Chile was responsible for 2.24% of the total value of exports of wood products traded worldwide, highlighting construction products. The study carried out by the World Bank entitled "The construction of wooden houses in Chile - A pillar for sustainable development and the reactivation agenda", indicates that in Chile 20.8% of homes with 1 and 2 stories high they are built of wood, a figure that is still low compared to the average of 70% that is built in developed countries such as the United Kingdom (70%) and the United States (85%) (2020).

The forestry and timber industry in Chile currently produces engineering products based on wood and adhesive for the construction sector. These products include medium density particleboard, medium density fiberboard, plywood, oriented strand board, composite beams, glued laminated timber (glulam) and structural wood. Within solid wood structural products, one of the most important is glulam, whose properties depend on the wood quality, adhesives properties, and the process conditions (Pirayesh et al. 2013). The adhesives used in the manufacture of glulam and other solid wood-based structural products are melamine-urea-formaldehyde, phenol-resorcinol-formaldehyde, polyurethane (PUR), and isocyanate polymer emulsions (Almeida et al. 2014). Currently the most used adhesives in solid wood products are MUF and PUR, a thermosetting adhesive and a thermoplastic adhesive respectively. Both adhesives are characterized by being highly reactive, which favors production levels. Once set, these adhesives leave a transparent joint line, which benefits the aesthetic part of wood-based products (Ramage et al. 2017, Sikora et al. 2016).

The aim of this research was to manufacture and evaluate the properties of solid and hollow GLULAM beams, with polyurethane adhesives, isocyanate polymer emulsions and melamine-urea-formaldehyde, by evaluating their physical and mechanical properties, as well as the study of morphological and bond adhesive properties.

MATERIALS AND METHODS

Materials

One hundred and twenty pieces of dried and planed *Pinus radiata* D. DON wood with an average moisture content of 12% were used. The pieces thickness was 32 mm and 47 mm, with a width of 95 mm and a length of 3200 mm. Three commercial structural wood adhesives were used, a one-component polyurethane adhesive (PUR) Itapur200, a two-component isocyanate polymer emulsion adhesive (EPI) Ita300-100/15 and a two-component melamine urea-formaldehyde adhesive (MUF) 1247/2526-100/20. The wood and the adhesives were provided by the company Anwood SpA, located in Los Angeles city, Chile.

Preparation and characterization of adhesives

The PUR, EPI and MUF adhesives were prepared in a plastic container by adding the catalytic agents by adhesive weight (w/w), and mixed at 20°C by using a mechanical stirrer IKA, model RW 20 D, of 4 stainless steel blades, at a speed of 300 rpm; during 10 min. At the end, samples were taken to study the adhesives. The adhesives study was carried out by determining the pH and viscosity at 25°C, and the solids content, in triplicate measurements. The pH was measured using a Hanna pH meter, provided with a combined electrode (temperature, pH), according to the standard ASTM E70-07 (2015). The viscosity was determined by means of a Brookfield RV model DV2T viscosity meter, using a spindle No.6 at 100 rpm and 25°C, according to standard ASTM 1084-97 method B (1998). The solids content, expressed as a percentage value, was determined by removing the water present in one gram of adhesive, by drying in oven at 103 ± 2°C during 24 hours, based on the standard ASTM D1490-01 (2013).

Dynamic thermomechanical analysis (DMA) of wood-adhesive composites

The stiffness of the wood-adhesive composites was measured using a dynamic thermo-mechanical analyzer, Perkin Elmer DMA 7e, in a three-point bending test mode with a distance between supports of 15 mm. Earlywood slices of *Pinus radiata* D. DON were used as wood species (Gao et al. 2015, Núñez-Decap et al. 2018a, 2019b, Labat et al. 2008, Park et al. 2008, Zhen 2002). Two veneers 0.5 mm thick, 4 mm wide and 20 mm long were bonded together with 200 $g\cdot m^{-2}$ on a dry basis (Fig. 1). The sets were subjected to a non-isothermal test between 30 and 200°C, with a heating rate of 10°C min^{-1} and a frequency of 1 Hz. Other sets were subjected to isothermal test at 25°C for 120 min and frequency of 1 Hz. The storage modulus was monitored during the whole test. Three replicates were evaluated for each sample.

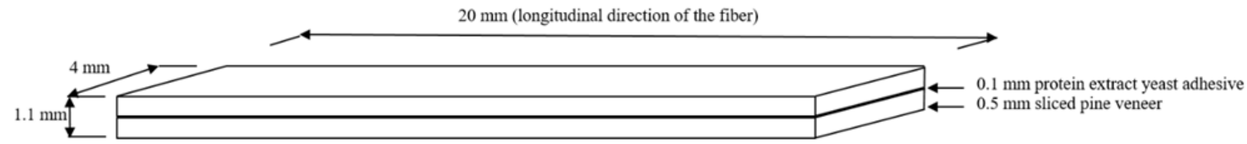

Fig. 1: Dynamic thermomechanical analysis by means of 3 points bending test of two wood sheets with an adhesive solution in the middle.

Visual classification of radiata pine structural wood

The 120 pieces of dry wood samples were sanded on both sides until obtaining the final thicknesses, 31 and 36 mm. Pieces width was 95 mm and length 3200 mm. The structural classification of the wood was carried out according to the Chilean standard NCh 1207 (2005), which classifies the wood visually, identifying knots, dead edge, fiber inclination and pith presence. The wood pieces were classified into three grades: select grade (GS), grade 1 (G1) and grade 2 (G2). The classified pieces of wood were marked at their ends according to the structural grade. Moisture content was measured using a Wagner L622 model moisture meter. Dimension and weight of pieces were also measured to calculate their density. Within the total of classified pieces, 11 were rejected and the remaining 109 were grouped according to thickness and structural grade (Tab. 1).

Tab. 1: Visual classification of pieces by structural grade (units).

		Visual structural grade Chilean standard NCh 1207		
Group	**Thickness**	**GS**	**G1**	**G2**
1	31 mm	13	11	9
2	36 mm	37	27	12

Manufacture of glued laminated timber beams

Glulam beams were manufactured with two different profiles, one solid and the other hollowed out. Fig. 2 shows the cross section of each kind of glulam.

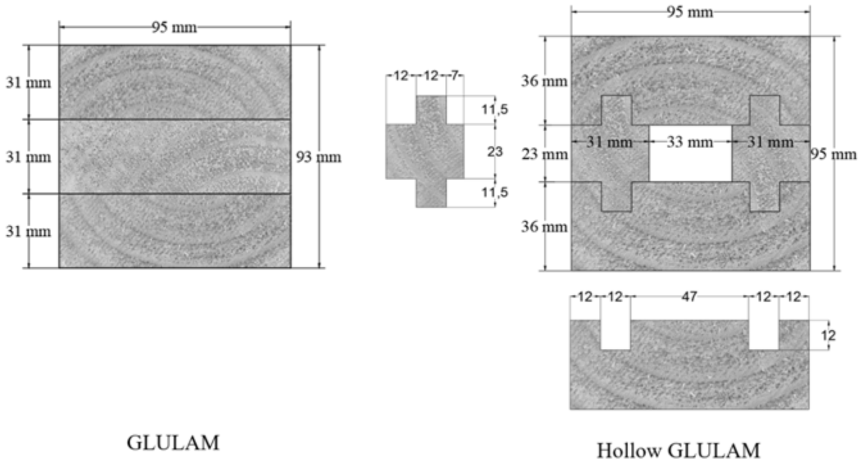

Fig. 2: Cross section of solid wood laminate beams and hollow wood laminate beams.

According to the availability of radiata pine wood and visual structural classification results, 30 laminated wooden beams of 3200 mm long were manufactured, according to the detail presented in Fig. 3.

GLULAM beams	Adhesive	Configuration of wood sheets in beams						
Solid	PUR	G1 / G2 / GS	G1 / G2 / GS	G1 / G2 / GS	G1 / G1 / GS	GS / G2 / GS		
	EPI	G1 / G2 / GS	G1 / G2 / GS	G1 / G1 / GS				
	MUF	G1 / G2 / GS	G1 / G2 / GS	GS / G2 / GS				
Hollow	PUR	G1 / G2 G2 / GS	G1 / G2 G2 / GS	G1 / G1 G1 / GS	G1 / G1 G1 / GS	G1 / G1 G1 / GS	GS / GS GS / GS	GS / GS GS / GS
	EPI	G1 / G2 G2 / GS	G1 / G2 G2 / GS	G1 / G1 G1 / GS	G1 / G1 G1 / GS	GS / GS GS / GS	GS / GS GS / GS	
	MUF	G1 / G2 G2 / GS	G1 / G2 G2 / GS	G1 / G1 G1 / GS	G1 / G1 G1 / GS	GS / GS GS / GS	GS / GS GS / GS	

Glulam: glued laminated timber; PUR: polyurethane; EPI: isocyanate polymer emulsions; MUF: melamine-urea-formaldehyde.

Fig. 3: Configuration of manufactured glulam beams.

Sheets gluing was applied manually on one side, through rubber rollers that allowed to achieve a uniform spread. The amount of adhesive used was 300 $g \cdot m^{-2}$ for each adhesive. Glulam beams pressing, was carried out by means of a hydraulic press for 180 min, with specific pressure of 8 kg·m^{-2}. Subsequently, the beams were stored for seven days, under controlled conditions of temperature and relative humidity (25°C +/-3°C, 60% +/-5%). Finally, glulam beams were sanded on their four faces and dimensioned in their length, obtaining final dimensions of 90 mm width, 90 mm thickness and 2500 mm long.

Evaluation of glued laminated timber beams

The specimens extraction scheme for mechanical tests and physical properties evaluation of Glulam beams is presented in Fig. 4. Specimens were extracted to run bending test, delamination adhesive bond test and bond shear tests. Also, specimens to measure density and moisture content, and samples to study of morphological and adhesive bond properties were extracted.

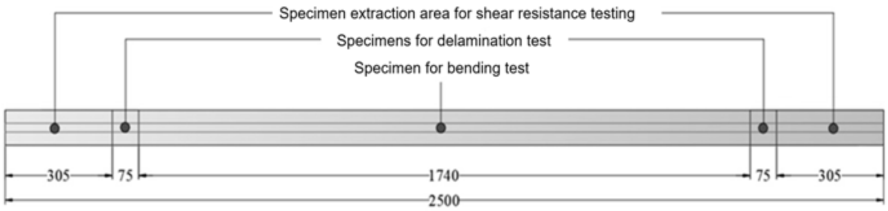

Fig. 4: Extraction scheme of the specimens for mechanical tests and evaluation of physical properties of glued laminated timber beams, dimensions in mm.

Bending test

Bending test of four-point was carried out according to ISO 8375 (2017) and ASTM D198 (2015). In Fig. 5, the diagram of bending test is presented.

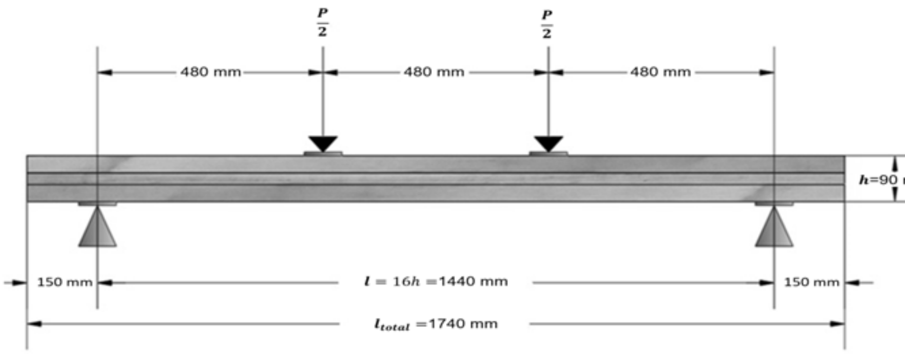

Fig. 5: Bending test specimen according to ISO 8375 (2017).

A test span was used between the supports of 1440 mm. The load was applied with a hydraulic cylinder and an Enerpac brand manual pump, using a 100 kN load cell at constant speed, with load intervals of 50 kg, until failure. Duration of applied load did not exceed 5 min, as ISO 8375 standard indications. Specimens displacement produced during the test were measured with a precision deformometer 0.01 mm and the loads were recorded through a connected interface to a Data Logger equipment, Handheld brand. The type of failure of the beams was observed and recorded according to ASTM D143 (2014) standard. The flexion rupture modulus (MR_f) and the elasticity modulus (MoE) were calculated. The MR_f was obtained by Eq. 1 and the MoE by Eq. 2:

$$MR_f = \frac{P_u * l}{b * h^2} \; [MPa] \tag{1}$$

where: P_u - maximum load (N), l - span test (mm), b - width sample (mm), h - thickness sample (mm).

$$MoE = \frac{l^2 * (P_2 - P_1)}{b * h^2 * (\delta_2 - \delta_1)} * \left[\left(\frac{3 * a}{4 * l}\right) - \left(\frac{1}{l}\right)^3\right] (MPa) \tag{2}$$

where: $(P_2 - P_1)$ - load increase in the linear area of the load-displacement curve (N), $(\delta_2 - \delta_1)$ – increase in deformation corresponding to $(P_2 - P_1)$, (mm), a - distance between a load application point and the nearest support (mm).

Adhesive bonding delamination test

Glulam beams adhesives bond delamination were evaluated based on the Chilean standard NCh 2148 (2013). Two specimens of 75 mm long, 90 mm width and 90 mm thickness, were obtained and evaluated from each beam. The specimens were subjected to water immersion process under vacuum (500 mm Hg for 30 min) and pressure (0.50 ± 0.04 MPa for 120 min) in an autoclave, to be dried later in an oven at 70°C for 15 hours. Delamination was measured along the gluing lines at the ends of the specimens as a percentage of these.

Shear strength test of the adhesive bonding

The shear strength of the glulam beams adhesive bond was evaluated based on the Chilean standard NCh 2148 (2013). From each beam, eight specimens were extracted and evaluated. The test was carried out by a universal testing machine (Instron 23-100), with a shear device, at a speed of 0.6 mm·min^{-1}. After the test, the maximum breaking load and percentage of wood failure were recorded.

Determination of density and moisture content

The moisture content and density of glued laminated wood beams were determined according to the Chilean standards NCh 176/1 and NCh 176/2, respectively. Five specimens from each beam were extracted and evaluated.

Study of the morphology of the adhesive bonding

The morphology of glulam beams adhesive bonding lines was studied microscopically, by a Leica EZ4 HD stereo microscope and a JEOL JSM-6610LV scanning electron microscope. A morphological analysis of the adhesive bond was performed and the penetration of the adhesive into the wood-adhesive-wood interface was quantified. Three 10 mm wooden cubes with the adhesive bond in the center were removed from each beam sample. Then, the samples were prepared to be studied in the stereomicroscope and scanning electron microscope (Moon et al. 2009).

RESULTS AND DISCUSSION

Adhesive properties

In Tab. 2 shows the results of adhesives properties. PUR adhesive presented the highest solids content, followed by EPI adhesive and MUF respectively, which was expected according to its composition and curing method, with a difference of 40% between the highest and lowest results. EPI adhesive viscosity, presented the highest value, followed by PUR and MUF adhesives respectively, according to their technical specifications. Increasing the viscosity makes it more difficult for the adhesive to spread on the wood surface. The pH of the three adhesives was acidic, as was the pH of radiata pine wood.

Tab. 2: Adhesive properties.

Adhesive	Ratio base/catalyst mix (% p/p)	Solid content (%)	Viscosity (cP)	pH
PUR*	100 : 0	98.28	7760	4.67
EPI**	100 : 15	65.50	12197	3.80
MUF***	100 : 20	59.51	6695	6.79

PUR- polyurethane; EPI- isocyanate polymer emulsions; MUF- melamine urea formaldehyde.

Dynamic thermomechanical analysis of wood-adhesive composites

Results of non-isothermal temperature analysis of adhesive-wood compounds, showed that the wood-MUF adhesive system (Fig. 6, Tab. 3), presented the highest value of stiffness and reactivity, reaching 4.3 GPa stiffness at 128°C, while EPI and PUR wood-adhesive systems reached 2.5 GPa and 2.8 GPa at 221°C and 268°C, resp. Similar behavior as results obtained from other study, that tested different adhesive systems (Park and Kim 2008, Núnez et al. 2018).

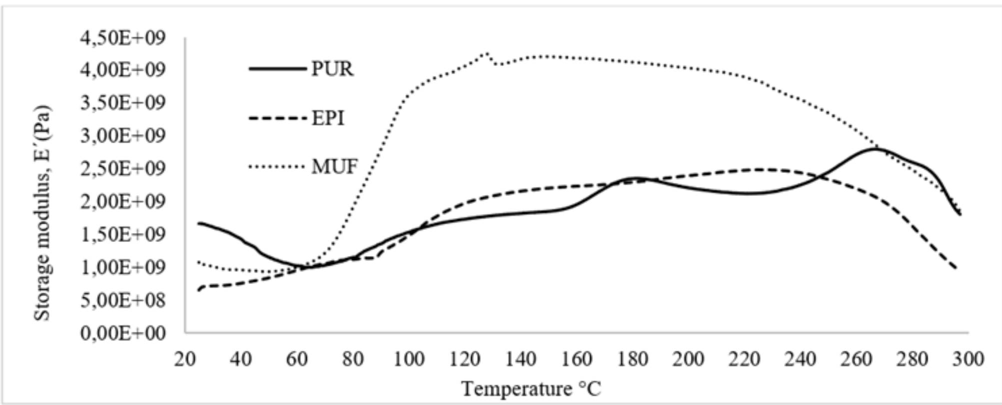

Fig. 6: Thermomechanical dynamic analysis curves of the wood-adhesive system.

Isothermal analysis results at 20°C of adhesive-wood compounds showed that wood-MUF system adhesive (Fig. 7), presented the highest stiffness value after 120 min of the analysis (4.3 GPa), while EPI and PUR wood-adhesive systems reached 2.2 GPa and 2.9 GPa, respectively. Similar as levels of stiffness achieved for others adhesives to manufacture wood-based composites (Gao et al. 2012, Gao et al. 2015, He and Riedl 2003, Núnez et al. 2018).

Tab. 3: Thermomechanical dynamic results of the wood-adhesive system.

Adhesive	Minimun temperature (°C)	E´min (Pa)	Maximum temperatura (°C)	E´max (Pa)
PUR	67.0	1.01E+09	268.0	2.80E+09
EPI	25.0	6.51E+08	221.0	2.48E+09
MUF	49.0	9.40E+08	127.5	4.26E+09

PUR- polyurethane; EPI- isocyanate polymer emulsions; MUF- melamine urea formaldehyde; E´min- minimum initial storage modulus; E´máx- maximum storage modulus.

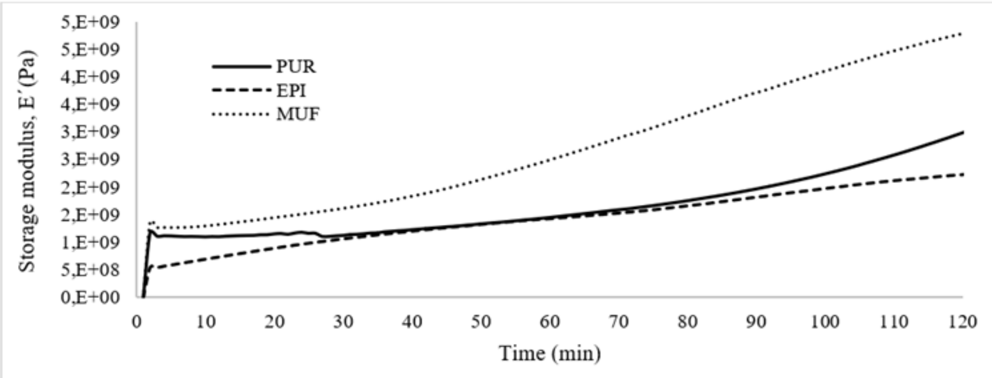

Fig. 7: Dynamic thermomechanical analysis curves of the adhesive at a temperature of 20°C.

Glued laminated timber beams properties

In Tab. 4 shows the average results of mechanical and physical properties and the quality of the adhesive bond of glulam beams, solid and hollow, with PUR, EPI and MUF adhesives.

Tab. 4: Average results of the mechanical, physical and quality properties of the adhesive bonding of glulam beams.

Glulam beams	Adhesive	Bending test			Shear test			Delamination test (%)	MC (%)	Density ($kg \cdot m^{-3}$)
		MR_f (MPa)	MoE (MPa)	P_u (KN)	Strength (MPa)	Max load (kN)	Wood fault (%)			
Solid	PUR	50^3	7670^1	25.16^3	10.0^1	22.3^1	74^2	32^2	8.9^1	446^1
	EPI	54^2	7901^1	27.56^2	10.6^1	23.7^1	80^2	0^1	9.4^1	449^1
	MUF	64^1	7360^1	32.14^1	10.5^1	23.3^1	90^1	0^1	9.3^1	456^1
Hollow	PUR	45^3	7714^1	22.52^3	6.1^2	2.9^2	33^3	47^2	9.0^1	484^1
	EPI	51^3	7901^1	25.39^3	7.7^2	3.8^2	47^3	29^2	9.2^1	479^1
	MUF	56^2	7991^1	27.86^2	10.0^1	4.9^2	70^2	30^2	10.2^1	499^1

Glulam: glued laminated timber; PUR: polyurethane; EPI: isocyanate polymer emulsions; MUF: melamine-urea-formaldehyde. Superscript numbers show the significant differences according to LSD test, with confidence interval of 95%. Statistical analysis ANOVA. Software Statgraphics Centurion XVII.

Flexural test results showed that there are statistically significant differences among rupture modulus (MR_f) and the maximum load (P_u) values, but not among values of elasticity modulus (MoE) which depended on type of adhesive and the glulam beams kind. Values of MR_f and P_u

were higher in solid glulam beams than the hollow ones, presenting the highest values in melamine-urea-formaldehyde (MUF) adhesive. The highest MR_f and P_u were 64 MPa and 32.14 kN, respectively, obtained by solid glulam beams glued with MUF adhesive, while the lowest results, 45 MPa and 22.52 kN, were presented by the hollow glulam beams glued with PUR adhesive. The resistance difference between the highest and lowest values of MR_f and P_u was 30%. EPI adhesive presented intermediate values of MR_f and P_u for MUF and PUR adhesives in solid glulam beams and showed similar results to those obtained by PUR adhesive, applied to hollow glulam beams.

The results of the *MoE* did not present statistically significant differences, which means that the rigidity of the glulam beams is not affected by type of adhesive neither by kind of profile. Similar results were obtained in a study that evaluated MUF, EPI, PUR and phenol-resorcinol-formaldehyde (PRF) adhesives in glulam beams manufactured by five species of Colombian wood, where the maximum resistance results were obtained through MUF and PRF adhesives, followed by EPI and PUR adhesive. In the elasticity modulus case, this did showed differences, being higher for glulam beams glued with MUF and PRF adhesive over EPI and PUR adhesives (López et al. 2013).

Adhesive bond shear strength test results showed, that there are statistically significant differences respect to the strength, maximum load and failure of wood. The highest shear resistance values were obtained for solid glulam beams with similar results among the three adhesives (10.0 MPa to 10.6 MPa), while in the hollow ones, the highest values were obtained with MUF adhesive (10.0 MPa) compared to EPI and PUR adhesives (7.7 MPa and 6.1 MPa, respectively). The highest maximum shear load results were obtained for solid glulam beams with similar results among the three adhesives (23.3 kN to 23.7 kN), while in hollow ones the results were significantly lower, but similar among the three adhesives (2.9 kN to 4.9 kN). This, because of the smaller area that was subjected to apply the shear test over specimens extracted from hollow glulam beams. Wood failure was bigger in solid glulam beams glued with MUF adhesive (90%) showing the highest values, followed by EPI (80%) and PUR (74%) adhesives, respectively. In hollow glulam beams case, the highest value was obtained for MUF adhesive (70%), followed by EPI and PUR adhesive. Finally, it should be noted that Chilean standard NCh2148 establishes as requirements for this test, a shear level resistance of 8 MPa and a wood failure of 70%, which implies that all glulam beams gotten the requirements of the standard, except for the hollow ones samples glued with EPI and PUR adhesive.

Adhesive bond delamination test results, showed that there are statistically significant differences in percentage. The lowest percentage values of delamination were obtained in solid glulam beams with 0% for MUF and EPI adhesives, while PUR adhesive presented 32%. In hollow glulam beams, the percentage of delamination was higher for the three types of adhesives (29% to 47%), which could be expected considering the fact of bonding line is shorter than the solid glulam beams, at the ends of its one, which disfavors the resistance to delamination as a result of conditioning. On the other hand, it should be noted that Chilean standard NCh2148 establishes a maximum delamination level of 5% as requirement for this test, which implies that only solid glulam beams glued with MUF and EPI adhesive gotten the requirement.

In studies of adhesive as MUF and phenol-resorcinol-formaldehyde on Bamboo and Portuguese maritime pine wood presented higher values of shear resistance and better delamination behavior than EPI and PUR adhesives, as similar result obtained in the present study (Martins et al. 2019, Weiqi et al. 2019).

Finally, it can be noted that the moisture content and the density of glulam beams were homogeneous in all the samples, with an average moisture content of 9.3% and the average density of 469 $kg \cdot m^{-3}$, expected values for radiata pine wood, used to manufacture glulam beams in Chile.

Morphology of the adhesive bonding

Wood-adhesive-wood interface images of glue line in solid and hollow glulam beams bonded by PUR, EPI and MUF adhesives, were obtained using stereomicroscope and scanning electron microscopy (SEM) are presented in Figs. 8 and 9, resp.

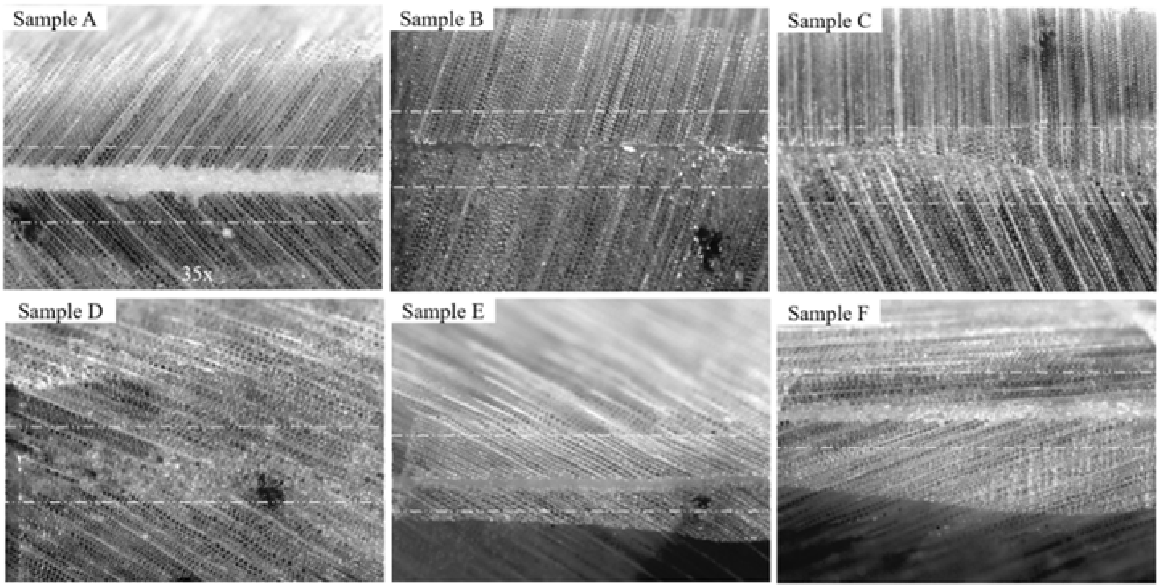

Fig. 8: Stereo-microscope images of the adhesive bonding in glulam *beams at 35x. Sample A, solid beam with PUR adhesive. Sample B, solid beam with EPI adhesive. Sample C, solid beam with MUF adhesive. Sample D, hollow beam with PUR adhesive. Sample E, hollow beam with EPI adhesive. Sample F, hollow beam with MUF adhesive.*

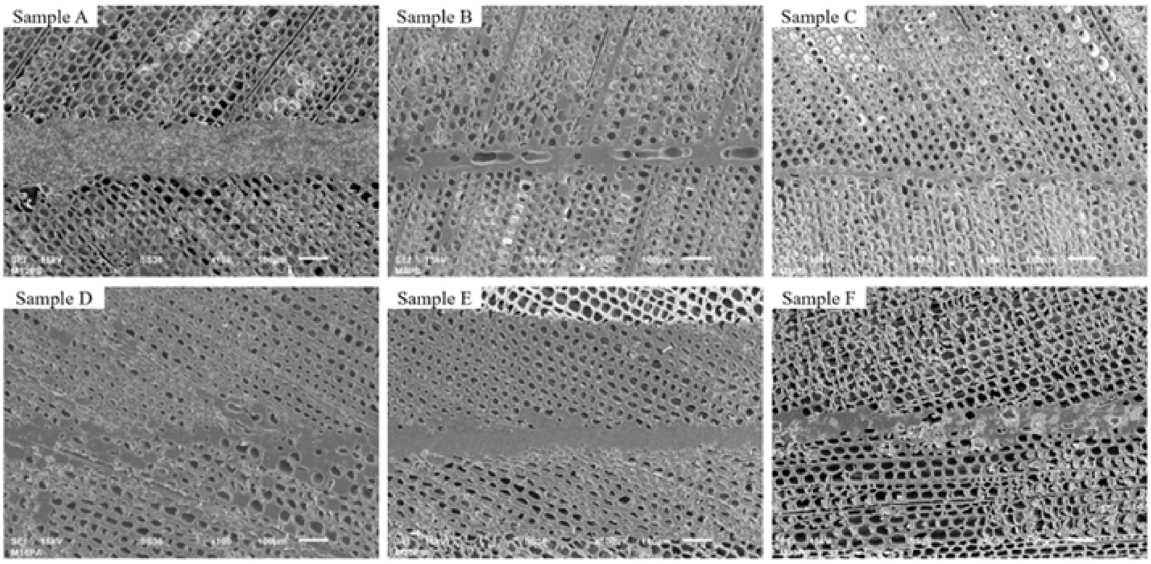

Fig. 9: Scanning electron microscopy images of the adhesive bonding in glulam *beams 100x. Sample A, solid beam with PUR adhesive. Sample B, solid beam with EPI adhesive. Sample C, solid beam with MUF adhesive. Sample D, hollow beam with PUR adhesive. Sample E, hollow beam with EPI adhesive. Sample F, hollow beam with MUF adhesive.*

The images obtained by stereomicroscope and SEM showed that interface area of adhesive bond with MUF adhesive, for solid and hollow glulam beams, is more homogeneous presenting less penetration of the adhesive into the wood than EPI and PUR adhesives. PUR adhesive exhibited a higher penetration into the wood, presenting a heterogeneous form. In Tab. 5 shows the result of the total interface thickness of the adhesive bond line measured by SEM.

Tab. 5: Average thickness of the wood-adhesive-wood interface in solid and hollow GLULAM beams.

GLULAM beams	Adhesive	Average adhesive interface thickness (μm)
SOLID	PUR	227.91^{3}
	EPI	131.75^{2}
	MUF	56.40^{1}
HOLLOW	PUR	224.09^{3}
	EPI	100.58^{2}
	MUF	97.88^{2}

Glulam: glued laminated timber; PUR: polyurethane; EPI: isocyanate polymer emulsions; MUF: melamine-urea-formaldehyde. Superscript numbers show the significant differences according to LSD test, with confidence interval of 95%. Statistical analysis ANOVA. Software Statgraphics Centurion XVII.

Total interface thickness results of adhesive bond line showed that range of mean values varied between 56.40 μm and 227.91 μm. MUF adhesives presented low thickness values and more homogeneous penetration distribution, 56.40 μm and 97.88 μm, for solid and hollow glulam beams. EPI adhesives presented intermediate thickness values between 131.75 μm and 100.58 μm and PUR adhesives presented the highest values of thickness of bond line with values between 224.09 μm and 227.91 μm.

Other investigations have found similar ranks (from 20 μm to 250 μm), for bond line thicknesses in adhesives applied to wood as well as MUF, EPI, PUR and phenol-resorcinol-formaldehyde. Wood anatomy is a condition that can affect preparation of samples surfaces, also conditions of gluing and pressing process, strongly can affect the thickness of the bond line of these adhesives, the properties of strength to shear and delamination (Knorz et al. 2015a,b).

CONCLUSIONS

(1) MUF adhesives showed the highest level of reactivity and stiffness according to the results of dynamic thermomechanical analysis (DMA). *(2)* The evaluation of the flexural properties showed that the solid and hollow glulam beams made with MUF adhesives presented the highest values of modulus of rupture, not appreciating significant differences in the modulus of elasticity. *(3)* The highest results of strength shear and failure of wood of the bonding line were obtained in the solid glulam beams for the three adhesives, and in the hollow glulam beams with MUF adhesive. *(4)* The best delamination results of the bonding line were obtained in solid glulam beams with MUF and EPI adhesive. *(5)* The bonding lines of the glulam beams with MUF adhesive showed a more homogeneous penetration of the adhesive into the wood, with a lesser thickness than the bonding line. *(6)* Finally, the MUF adhesive presented the best comparative performance in the manufacture of solid and hollow glulam beams of Pinus radiata wood.

ACKNOWLEDGEMENTS

We would like to thank the collaboration, equipment and infrastructure provided by the Wood and Adhesive Engineering Products Laboratory (PRODIMA-LAB), of the Department of Civil and Environmental Engineering of the Universidad del Bío-Bío. We also want to thank the ANID BASAL FB210015.

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