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Comparison of different bonding parameters in the production of beech and combined beechspruce CLT by standard and optimized tests methods

Michele Brunetti¹, Michela Nocetti^{1,2*}, Benedetto Pizzo¹, Francesco Negro³, Giovanni Aminti¹, Paolo Burato¹, Corrado Cremonini³, Roberto Zanuttini³

¹CNR-IBE, Sesto Fiorentino (FI), 50019, Italy

²Department of Forest and Wood Science, Stellenbosch University, Stellenbosch, 7600, South Africa ³Department of Agricultural, Forest and Food Sciences, University of Torino, Grugliasco (TO), 10095, Italy

*Corresponding author Michela Nocetti CNR – IBE, Institute for Bioeconomy Via Madonna del Piano 10, I - 50019 Sesto Fiorentino (FI), Italy e-mail: michela.nocetti@cnr.it Tel: 0039-055-5225654 Fax: 0039-055-5225643

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Abstract

The interest in the use of beech wood in construction is growing steadily. Considering its high mechanical performance, it has a large potential for the production of glued timber structural products. However, the issue of structural bonding remains the one to be solved for an effective production and a safe use. Three adhesives (one-component polyurethane – PUR, PUR + primer and melamine-urea-formaldehyde – MUF) and two press systems (hydraulic and vacuum press) were investigated in the production of pure beech and combined beech-spruce-beech cross laminated timber (CLT). The evaluation of the bonding quality was performed with both standardized and optimized (the specimen layers were oriented with the wood grain forming an angle of 45° in respect to the load application) delamination and shear test methods. None of the adhesive tested met the requirements for delamination tests provided by the current standardization for softwood. As for the adhesive, PUR was that with the poorest performance in the production of the CLT panels entirely made of beech; the addition of a primer improved the bonding, permitting to achieve results comparable to those observed for MUF. On the contrary, in the production of beech-spruce panels, the three adhesive gave similar outcomes. The press system was not a relevant factor in terms of bonding quality.

As for the testing methods, a size effect was noticed in the delamination test: the larger the specimen and the greater the delaminations observed. The shear tests on dry specimens were little sensitive, even if a 45° grain orientation seemed to reduce the rolling shear during shear test and to better highlight the effect of the bonding parameters. Combining delamination pretreatment and shear made the test more sensitive and subjective.

Keywords: delamination test; shear test; glued timber; wood adhesive; hydraulic press; vacuum press

1. Introduction

The development of Cross Laminated Timber (CLT) has greatly changed the use of structural timber in construction. CLT, in fact, enables a new structural typology, based on load-bearing walls and ceilings, that came up beside the traditional wooden carpentry. CLT is widely appreciated by the market and its annual global production is expected to reach 3,000,000 m³ by 2025 [1,2]. The success of CLT can be attributed to several aspects: lightness and high mechanical performance, possibility to realize prefabricated structures, short building sites required, thermal insulation properties, limited cost. Furthermore, CLT meets the increasing demand for wood-based materials in construction that is linked with their sustainability, low environmental impacts and carbon storage.

In Europe, CLT can only be made of softwood or poplar (*Populus* spp.) wood according to EN 16351 [3]. The most used species are Norway spruce (*Picea abies*), white fir (*Abies alba*), Scots pine (*Pinus sylvestris*) and less frequently Doulglas fir (*Pseudotsuga menziesii*). Anyway, the interest in the use of hardwood in construction is growing steadily [4,5]. Beech, in particular, is an abundant forest resource in Europe, which is fundamental for assuring a regular supply to industrial productions. Considering also its high mechanical performance [6–8], beech wood has a large potential for the production of Glue Laminated Timber (GLT) [9–12] and CLT, both in pure [11,13] and combined layup with softwood [14,15].

However, determining the bonding quality between layers is a fundamental step to the efficient and safe use of any glued wood-based structural product. The characteristics of a structural bonding has to be implemented and verified for each species, since the performance can considerably vary depending on the combination between species and adhesives. This is especially valid for hardwoods which are characterized by a high variability in terms of anatomical structure and physico-mechanical properties. The technical process and the bonding properties cannot be simply transferred from a well know species to another, especially if the wood characteristics are different between the two [16].

As a rule, the most critical aspect of bonding lays in the performance after variations in humidity, durability or ageing. Over the past years, several investigations were performed on the bonding quality of beech and various aspects were studied: gluability of beech wood containing red heartwood [17–21]; performance of different adhesives [16,18,19,21,22]; influence of closed time during bonding process [20,22]; thickness of lamellae in the final glued product [20]; use of several primers before the application of one-component polyurethane adhesive [20,23–25]; influence of the angle of the growth rings of lamellae [20]; surface preparation before gluing [24,26].

Commonly, the evaluation of the structural bonding is performed with two kinds of tests: one aimed to measure the shear strength of the glueline (test that can be executed both on specimens in dry condition or after a specific pretreatment); the other specifically intended to evaluate the durability of the bonding. This last, named delamination test, is designed to induce high stresses on the glueline by wet and re-drying cycles and to observe the splits eventually occurred between two adjacent wooden layers. In the existing literature on hardwoods, it was frequently observed that the shear tests performed on dry specimens easily met the requirements of the technical standardization on gluing of structural products. Instead, meeting the standard requirements after humid pretreatments was often difficult [16,18–20,27–29].

In the same way, the delamination test designed for softwood glulam is generally recognized as severe when applied to hardwood laminated timber and some authors proposed to lower the acceptance limits respect to those envisaged for softwood [16,27].

It is also appropriate to consider that the researches cited above were performed on specimens with the lamellae oriented in parallel each other (GLT). In CLT, instead, the perpendicular orientation of boards constitutes a relevant variable, both on the results of shear tests and, mainly, on delamination. In this regard, Knorz et al. [30] investigated the delamination test of CLT made of spruce, concluding that the method, taken from GLT, is not suited to CLT and requires a specific adaptation. The delamination test envisaged by EN 16351 can be considered severe for coniferous CLT as well. For this reason, Sikora et al. [31] proposed to replace the delamination with the shear test, as an easier and more reliable method.

The advantage of shear tests is that they quickly provide an objective and easy-to-measure value. The block shear test could be consider an effective method for quality control of bond lines because the preparation of

the specimens is simple and the procedure easy to carry out [32,33]. At the same time, weaknesses are reported in the literature, mainly when the test is applied to cross laminations and rolling shear failure may occur. Therefore, some authors proposed and verified a new approach to prevent rolling shear, i.e. to cut the specimen with the testing surfaces inclined at 45° with respect to the load axis, so that both surfaces show the same wood grain inclination with respect to the load, avoiding the presence of one layer weaker than the other due to their orientation [34].

Another step forward was the combination of delamination and shear test on the same specimen [34,35]. The concept arose from the subjectivity of the evaluation of splitting in delamination test, improvable by the easiness of measure of shear strength. On the other hand, the shear test performed on dry specimen often resulted to be little sensitive, as stated few lines above, and a suitable pretreatment could increase the effectiveness of the test. Previous works on this subject experienced promising results [35]. As for the several bonding parameters, the adhesive is surely one of the firsts to be verified. Today softwood CLT is mainly bonded using formaldehyde-free polyurethane (PUR), but urea-melamine-formaldehyde (MUF) and phenol-resorcinol-formaldehyde (PRF) adhesive systems are used as well. PUR resins are less suited to hardwood because, even if they enable passing the shear tests, their use does not meet the delamination requirements. Anyway, some advantages of PUR resins (fast curing at room temperature, transparent glue lines, absence of formaldehyde, easy to handle one-component formulation) make worth trying to improve their performance by using primers [24].

Pressing is another crucial phase of CLT manufacturing. Today, two types of press are used by the industries. The hydraulic press with rigid plates can generate high vertical pressures. To minimize the potential gaps between the boards, application of side clamping is sometimes applied. Instead, the vacuum press with flexible membrane generates lower pressures and exploits vacuum to favor the penetration of the adhesives inside wood.

In this context, the objectives of this study were: (1) to compare the influence of different pressing methods, adhesives and compositions on the production of beech and beech-spruce CLT panels; (2) to analyse different testing methods for evaluating the bonding quality of beech CLT. The outcomes represent relevant advances toward the production of beech wood CLT.

2. Materials and Methods

2.1 Materials

Boards of beech (*Fagus sylvatica* L.) and spruce (*Picea abies* L.) originating from North-eastern Italy and Austria respectively, were supplied as raw material for this study. The boards were kiln-dried until the achievement of the equilibrium moisture content of about 12%, then cut into lamellae of 25x100x475 mm³ (BxHxL). During the process, lamellae with major defects such as large knots or cracks were discarded. Three commercial adhesives were used for manufacturing the experimental CLT panels: 1-component polyurethane adhesive (PUR), the same polyurethane plus primer (PUR+P), and melamine-urea-formaldehyde (MUF).

2.2 CLT manufacture

3-layered CLT panels were assembled with nominal dimensions of $450x450x60 \text{ mm}^3$ and two different compositions, namely *beech-beech-beech* ('beech' in the following) and *beech-spruce-beech* ('combined'). The boards were sanded just before bonding in order to obtain plane surfaces suited to the process. The spread rate of PUR was about 150 g/m² and, when used, primer amounted to 20 g/m²; MUF adhesive spread was about 300 g/m². The primer was applied on each beech adherend and an open time of 30 minutes was waited before the application of the PUR. The closed assembly time for PUR was not more than 25 minutes, while with MUF it was extended to 60 minutes, since previous works stressed on the benefits of a longer closed assembly time that allows a better penetration of the MUF into the wood, with better results of delamination tests [20,22,27,29].

The manufacture of the panels was carried out in two industrial plans: one using a hydraulic press (H) and the other a membrane-vacuum press (V). By hydraulic press, the pressure of 1.2 MPa was applied and held for 2 h with PUR, 3 h with PUR+P and 24 h with MUF; by membrane-vacuum press, the pressure of 0.09 MPa was applied for 2 h with PUR, 3 h with PUR + P and 4.5 h with MUF. After manufacturing, CLT panels were stored for 30 days at 20 °C and 65% of relative humidity.

2.3 Testing procedures

The CLT testing material, consisting of 3 panels for each of the 12 experimental thesis (2 press typologies x 2 species combinations x 3 adhesives), were cut into samples for delamination, shear and merged delamination + shear tests (the test methods are described in the following paragraphs). From each panel, specimens for all the testing procedures were cut and the positions of the specimens within the panel were changed randomly so as to ensure that the specimens for a certain test did not all originate from the same position in their respective panels. Approximately, 4 specimens per panel for delamination tests and 3 specimens per panel for shear and delamination + shear tests were obtained.

The specimens were cut from CLT panels with the grain direction of each lamella oriented with an angle of 90° (and 0°) and 45° with respect to the sides of the panel. Thickness was always of 60 mm, corresponding to the thickness of the CLT panels, whereas the cross section was always squared but of different sizes (Table 1 and Fig. 1).

Table 1

Outline of the test methods: name, test type, specimen size, grain angle with respect to the side of the panel and number of specimens.

Test	Test methodology	Specimen size	Grain angle	Ν
		(mm^2)	(°)	
D_100	Delamination (standard)	100x100	90	136
S 50 90	Shear on dry specimen (standard)	50x50	90	100
DS 50 90	Delamination pre-treatment + Shear	50x50	90	97
s <u>50</u> 45	Shear on dry specimen	50x50	45	80
DS 50 45	Delamination pre-treatment + Shear	50x50	45	98
s 70 45	Shear on dry specimen	70x70	45	102
DS_70_45	Delamination pre-treatment + Shear	70x70	45	94



Fig. 1. Example of the different specimens cut from a portion of the panel (acronym explained in Table 1).

2.3.1 Delamination tests (D)

The delamination test was performed according to Annex C of EN 16351 [3]. The cross section of the specimens was of 100x100 mm. Grain formed an angle of 90° with the sides of the specimens. These were immersed in water at 10-20 °C applying for 30 min a vacuum between 15 and 30 kPa. Following, a pressure of 600-700 kPa was applied for 2 h. The specimens where then dried at 75 °C for 10-15 h until their mass reached the 100-110% of the initial value. After the above pre-treatment, the length of delaminations was measured. The total delamination D_{tot} and the maximum delamination D_{max} were calculated as:

$$D_{tot} = 100 \times \frac{l_{tot,delam}}{l_{tot,glueline}}$$

$$D_{max} = 100 \times \frac{l_{max,delam}}{l_{glueline}}$$

where $l_{tot,delam}$ is the total delamination length in the specimen, in mm; $l_{tot,glueline}$ is the sum of the perimeters of all glue lines in the specimen, in mm; $l_{max,delam}$ is the maximum delamination length, in mm; $l_{glueline}$ is the perimeter, in mm, of the glue line in which the maximum delamination occurred. Openings between adjacent layers were considered as delamination only when presenting the criteria described in Annex C of EN 16351 [3].

After delamination readings the specimens were completely split with hammer and chisel and the wood failure percentage (WFP) observable on the breaking surfaces was visually assessed by an experienced operator and rounded to the nearest 5%.

2.3.2 Shear tests (S)

Specimens were cut from CLT in order to cover the whole thickness of the panels, then 2 gluelines for each specimen were tested. Their cross sections were of 50x50 mm, with grain oriented at 90° (method S_50_90) and 45° (S_50_45), and of 70x70 mm with grain oriented at 45° (S_70_45).

The specimens S_50_90 were placed in the shearing tool so that the vertical load was applied in the direction of the wood grain on one side of the glueline and perpendicular to the grain on the other side of the glueline. As for the specimens S_50_45 and S_70_45, the load was applied with and angle of 45° in respect to the wood grain for both the two sides of the glueline (Fig. 2). The shear strength f_v was calculated as

$$f_{v} = \frac{F_{u}}{A}$$

where F_u is the failure load, in N, and A is the shared area, in mm².

2.3.3 Merged delamination + shear tests (DS)

In the merged tests which combined delamination + shear test, the specimens first underwent the wet and redrying cycles as for delamination test (see above) and then were subjected to shear test after the delamination readings. Overall, this resulted in test methods DS_50_90 , DS_50_45 and DS_70_45 . Both delamination and shear strength were determined as already described above.

After test, the wood failure percentage (WFP) was visually assessed by an experienced operator and rounded to the nearest 5%.



Fig. 2. Load configuration for specimens with 90° (left) and 45° (right) grain orientation.

2.4 Data analysis

A pass/fail evaluation was conducted for the delamination test results according to the requirements settled in EN 16351 [3]: the D_{tot} should not exceed 10% of the sum of all glue lines and D_{max} should not exceed 40 % of the total length of a single glue line (named D pass). When the requirements for delamination were not met, the glue lines were split and the wood failure percentage assessed: the minimum WFP for the single glue area should be not less than 50% and the minimum WFP of the sum of all split glued areas of the specimen should be not less than 70% (WFP pass). The same requirements were kept for the pass/fail analysis of the smaller specimens (DS_50_90, DS_50_45 and DS_70_45).

In order to explore the influence of the gluing parameters on the bonding quality, mean comparisons were performed after check of data for normality (Shapiro-Wilk normality test) and homogeneity of variances across groups (Levene test). Variables expressed in terms of percentage were arcsin transformed in order to obtain normal distributions. An analysis of variance was implemented including shear strength or delamination as dependent variables and species combination, adhesive, press and their interactions as independent categorical variables. For significant differences as resulted from the ANOVA, a post hoc analysis (Tukey's HSD test) was computed.

Finally, Pearson correlation coefficients were calculated to investigate the agreements between the response of the several test methods.

3. Results and discussions

3.1 Gluing parameters

3.1.1 Delamination

The results of the pass and fail analysis for the several thesis and test methods are reported in figure 3. Overall, the specimens that met the requirement of the standard EN 16351 [3] for delamination length were very few: only 2 specimens (1.5 % of the total) tested according to the standard (method D_100). Considering the second step of the evaluation, the number of specimens that reached the minimum WFP provided by the standard rose to 14 (10.3 %). Changing the dimension of the specimens (DS_50 or DS_70) the percentage of *pass* rose a bit, but this result will be discussed in the next paragraph concerning the comparisons between methods.

The highest percentages of *pass* were reached by the thesis with MUF in the combined beech-spruce panels (both for H and V press). PUR was the adhesive with the highest percentages of *fail*, reaching the 100% with the panel entirely made of beech; while it performed better with the combined panels. Adding the primer before gluing with PUR adhesive, the percentages of *pass* rose.

More details about the effects of the several factors investigated were detected by the analysis of the average delamination values. Here, only the results of the D_{tot} are shown, since the D_{max} followed the same trend. The average values and the variation are represented in Figure 4, while the results of the analysis of variance are summarized in Tables 2 and 3. As for the factors examined, the species and the adhesive were highly significant factors influencing delamination, while the press used was not so important (Table 2). Generally, beech panels delaminated more than the combined ones, while the use of hydraulic or vacuum press led to very similar results in terms of delamination. The same conclusions about the influence of the press used were reported by Knorz et al [30] in a study on the bonding evaluation of spruce CLT. Even if the pressure is very different between the two systems, the vacuum likely plays a role in improving the penetration of the adhesive into the wood layers.

The analysis went in depth investigating the interaction between species and adhesive, which was also a highly significant factor as resulted from the ANOVA. Looking at the post hoc tests (Table 3), it was evident that the group which clearly differentiated from the others was the beech panels glued with PUR, with the far higher D_{tot} values. On the opposite side, lowest D_{tot} were observed for the combined panels glued with MUF. Considering the results separately for species (or panel's layup), PUR displayed the worst results in terms of delaminations when used on beech, while MUF and PUR+P did not differentiate. Otherwise, the combined panels showed on average the same values of D_{tot} when glued with PUR or PUR+P, but the use of MUF resulted in a significantly lower D_{tot} .

The same findings were confirmed by all the test methods, with the exception of DS_50_90 which did not consent to distinguish the MUF adhesive for the combined panels.

The delamination test confirmed to be a very severe evaluation method for CLT, particularly when applied to hardwoods.



Fig. 3. Pass/fail results for the several test methods compared. D pass: percentage of specimens which met the requirements of the standard EN 16351 for total and maximum delamination; WFP pass: percentage that exceeded the limits for delamination but met the requirements for wood failure.



Fig. 4. Mean values of total delamination for the several thesis and the different test methods (bars indicate the standard deviation).

Table 2

F values and significance as results of the three-way analysis of variance for the D_{tot} observed in the several test methods.

	D_100	DS_50_90	DS_50_45	DS_70_45
species	103.5 ***	23.8 ***	25.9 ***	17.9 ***
adhesive	79.7 ***	27.9 ***	40.5 ***	53.0 ***
press	0.1	4.2 *	4.8 *	3.6
species × adhesive	22.3 ***	16.8 ***	21.2 ***	18.5 ***
species × press	5.4 *	3.7	4.9 *	1.9
adhesive × press	0.6	0.8	3.1	5.0 **
species × adhesive × press	2.4	0.7	0.4	0.3

*significant at 5% level; **significant at 1% level; ***significant at 0.1% level; ' ' not significant

Table 3

Species and adhesive interaction: mean values of D_{tot} and comparison among groups calculated for the different test methods. Same letters indicate that the means do not differ at 5% significant level.

	D_100	DS_50_90	DS_50_45	DS_70_45
beech_PUR	98.5 a	89.0 a	86.5 a	91.0 a
beech_MUF	55.7 b	19.6 b	22.6 bc	49.5 bc
beech_PUR+P	47.3 b	32.7 b	29.7 bc	29.8 cd
comb_PUR	55.2 b	26.0 b	30.4 b	64.8 b
comb_PUR+P	44.3 b	26.3 b	37.3 b	47.8 bc
comb_MUF	29.2 с	20.5 b	10.4 c	17.5 d

3.1.2 Shear strength

The median and the variability of the shear strength measured on the specimens dry and after the delamination cycles are graphed for all the thesis in figure 5. In table 4, the results of the analysis of variance are reported, the shear strength being the dependent variable and the species, adhesive, press and their interaction the factors included in the model.

Looking at the tests performed on the dry specimens, it was evident that the species was the most important factor determining the shear strength of bonding, having the beech panels a strength significantly higher than the combined ones. This difference was more evident in the specimens tested with the load forming an angle of 45° in respect to the wood grain (S_50_45 and S_70_45 in figure 5).

The Tukey test was performed for the significant factors in the ANOVA and the outcomes (mean values and significance of difference between means) for the *species x adhesive* interaction are summarized in table 5. The test S_{50}_{90} did not highlight any difference among adhesives, while PUR performed worse in beech panels, as evidenced both by S_{50}_{45} and S_{70}_{45} . For combined panels no differences were detected by S_{50}_{45} test method and only a slightly higher values for PUR was noted in the S_{70}_{45} results. As concerns the press used, a low-level significance was observed only in the S_{45}_{90} test. So, the interaction *species x pressure* was investigated and the hydraulic press seemed to result in a higher shear strength for combined panels, while no significant difference was recorded for beech panels (data not shown).

When the shear test was performed after the delamination cycles, the effect of the species decreased, while the importance of the adhesive increased (Table 4). The not significance of the adhesive factor in the DS_70_45 specimens is due to the fact that the PUR specimens were not included in the analysis (the shear test was not possible due to the complete split of the layers). Again, the interaction *species x adhesive* (Table 5) displayed very low shear values for beech glued with PUR and similar higher values when MUF and PUR+P were used (only DS_50_45 evidenced significant higher shear values for MUF in respect to PUR+P). No difference regarding the adhesive was found out for combined panels (Table 5).

Small differences were observed between the two press methods: the hydraulic press resulted in little higher shear strength for combined panels and the vacuum press for the beech specimens (data not shown). Any comment on the shear strength values in terms of acceptability of the performance of the glued product is not possible due to the lack of technical standardization, as well as of reference values in the scientific literature. The only earlier study on the bonding of CLT made of hardwood was the one from Eucalyptus [35], for which mean values of shear strength were lower than what detected here (4.12 MPa for the 90° specimens and 5.62 MPa for the 45° orientation). This underlines the high variability of hardwood properties, already highlighted also for other species like beech, oak and ash [24], and makes even more difficult to set reference values for an ad hoc minimum requirement of bonding quality. Instead, what can be observed is that usually in the literature the shear test on the glue lines are found to be not able to distinguish the several factors that can affect the bonding quality, especially when tests are carried out on the dry specimens [18–20,27–29]; here the main outcomes deduced from the delamination test can be detected also by the shear tests (with the exception of the S_50_90 method).



Fig. 5. Boxplot of the shear strength for the several thesis and test methods (median; 5, 25, 75, 95 percentile; min/max).

Table 4

F values and significance as results of the three-way analysis of variance for the shear strength observed in the several test methods, dry and after delamination.

	DRY		AFTE	AFTER DELAMINATION		
	S_50_90	S_50_45	S_70_45	DS_50_90	DS_50_45	DS_70_45 ⁽¹
species	105.4 ***	262.9 ***	415.6 ***	40.7 ***	16.3 ***	59.8 ***
adhesive	3.8 *	4.8 **	2.7	52.5 ***	50.6 ***	0.3
press	5.5 *	0.3	2.9	0.4	0.0	4.0 *
species × adhesive	0.1	6.6 **	15.3 ***	30.1 ***	27.4 ***	8.2 **
species × press	17.3 ***	0.4	7.7 **	6.5 *	6.2 *	5.6 *
adhesive × press	2.5	0.9	0.7	0.9	0.4	1.6
species × adhesive	0.0	0.8	2.1	0.5	1.0	0.2

*significant at 5% level; **significant at 1% level; ***significant at 0.1% level; ' ' not significant

(1) specimens glued with PUR adhesive were not included

Table 5

Species and adhesive interaction: mean values of shear strength (dry and after delamination) and comparison among groups calculated for the different test methods. Same letters indicate that the means do not differ at 5% significant level.

	DRY strength (N/mm ²)		Strength A	FTER DELAM (N/mm ²)	INATION	
	S_50_90	S_50_45	S_70_45	DS_50_90	DS_50_45	DS_70_45
beech MUF	6.7 a	9.6 a	7.2 ab	5.1 a	7.3 a	4.6 a
beech_PUR+P	6.8 a	9.1 a	7.5 a	4.8 a	5.4 b	5.4 a
beech_PUR	6.1 a	7.6 b	6.3 b	1.3 c	0.8 d	-
comb_MUF	4.3 b	4.6 c	3.7 d	2.6 b	3.7 c	3.4 b
comb_PUR+P	4.7 b	4.6 c	3.9 cd	2.7 b	2.4 c	2.3 b
comb_PUR	4.0 b	4.8 c	4.7 c	2.3 bc	2.7 c	-

3.2 Comparing methods

The different test methods can be compared on the basis of delamination and shear strength values.

3.2.1 Delamination

The wet and drying cycles applied in the several tests were all the same, but the sizes of the specimens were different and a dimensional trend was observed: the larger the specimen, the higher the occurred delamination. Comparing the average values of D_{tot} for the four methods, D_100 was the one with the higher delaminations (on average 55.0%), followed by DS_70_45 (49.5%), DS_50_45 and DS_50_90 (39.3% and 35.9% respectively). Thus, by the comparison of means it appeared that the results of DS_50_45 and DS_50_90 did not differ each other, while they both distinguished from D_100 and D_70_45. The last two also were different, although only at 5% level of significance (data not shown).

Despite the "size effect", according to the outcomes of the delamination readings (see paragraph above), the four test methods agreed very well in the evaluation of the several bonding parameters. The agreement was "measured" by the calculation of the correlation coefficients between the D_{tot} averaged by panels of the several methods (Table 6); the species were kept separate. The correlations were high and highly significant for the beech panels, while for the combined the values of the coefficients were lower and sometimes not significant. In particular, DS_50_90 for combined species did not correlate with any of the other methods. This outcome could be explained by the high variability of delaminations observed in the beech panels, for which the PUR adhesive let to broad splits among the layers. The less variability in the combined specimens

could be insufficient to detect significant correlations, but it helped to highlight another clue: even though the size of the specimens was the same, the 90° orientation of the layers most likely behaved differently to the 45° in respect to the sorption of water and the re-drying during the test, thus inducing different stresses on the glue-line. The average values of delamination were very similar between the two series, but the DS_50_90 seemed less sensitive to small bonding quality variations, without a clear explanation for that. However, due to the findings described in the paragraph 3.1 and the size effect above mentioned, it can be concluded that the 50 mm size could be large enough to get a proper evaluation of the delamination behaviour for beech and combined beech – spruce CLT panels.

Table 6

Pearson correlation coefficients (r) between the several methods for the mean values for panel of D_{tot}

	DS_50_90	DS_50_45	DS_70_45
Beech			
D 100	0.86***	0.91***	0.87***
$\overline{\text{DS}}_{50}90$		0.87***	0.75***
DS_50_45			0.81***
Combined			
D_100	-	0.58*	0.59*
DS_50_90		-	-
DS_50_45			0.65**
*	1 *** * * * * * * * * *	10/1 1 1 1 1 1	· C · · · O 10/

*significant at 5% level; **significant at 1% level; ***significant at 0.1% level; (-) not significant

3.2.2 Shear strength

From table 5, where the mean values of shear strength were displayed for the several test methods, it can be noticed that the load of dry specimens resulted in the highest strength values for the DS_50_45 test, the lowest for the DS_50_90 and intermediate values for the DS_70_45. The difference between the two 50 mm sized specimens was the grain orientation of the layers in respect to the load direction. The 90° (and 0°) orientation let to the presence of rolling shear during the test, therefore, to lower maximum loads; the rolling shear effect could be reduced with the 45° orientation and in that case the ultimate load were higher. The same was observe in a similar work on eucalyptus [35].

On the other hand, the lower stress values obtained with the 70 mm sized specimens compared to the 50 mm ones could be explained by a size effect on the shear test method. Okkonen and River [36], studying the factors that can affect the block shear test on several species, found out the specimen size as an important aspect, being the shear strength of the smaller specimens higher than what was measured on the bigger ones. Comparing the "S" and the "DS" tests results, the shear strength values dropped after the wet and re-drying pretreatment. The shear test methods after delamination were able to discriminate the specimens which delaminated more from the others that did not or did to a lower extent. This statement was confirmed by the very high correlations values obtained between the shear strength and D_{tot} for all the merged DS test methods (Table 7 and Figure 5). Consequently, it can be hypothesised the effectively use of the shear strength values after delamination cycles as a "measure" of the delamination extent and, therefore, of the durability of the bonding. This could be a more objective assessment than the subjective evaluation of the splits between layers made by an operator and may help to combine the information of the strength of the bonding with its durability. Similar conclusions were reported by Dugmore et al. [35].

Finally, the methods were compared by means of correlations determined for the shear strength values averaged by panels (table 8). Table 8 shows the results of beech panels, since for the combined beech-spruce specimens no significant correlation was found. As for the delamination results, the low variability among the several thesis in combined panels could be the explanation of a scarce significance of the correlation coefficients.

Anyhow, from table 8, the results obtained from the specimens tested in dry condition did not correlate each other; the S_50_90 strength values did not correlate either with the results after delamination, while the other

"dry" tests did; most important, the tests after pretreatment displayed high and significant correlation each other. This difference of behaviours in dry (no correlation) and wet (very good correlation) conditions was possibly due to the occurrence of microcracks due to delamination in pretreated samples, which enhances the sensitiveness to test the bonding quality in assemblies, thus allowing their differentiation irrespective on the adopted geometry. It is worth evidencing that this occurrence was not obtained in CLT made with spruce, for which the specimens were 40 mm sized and did not delaminate [34].

On the other side, the "dry" tests were less effective, even though the ones with the 45° orientation were able to better characterize the bonding quality (the correlation with the results after delamination were moderate but significant).

Table 7

Pearson correlation coefficients (r) between the shear strength measured after delamination and the D_{tot} values measured on the same glue line for the several methods

	beech	combined	All
DS 50 90	-0.83***	-0.44***	-0.56***
DS_50_45	-0.91***	-0.68***	-0.72***
DS_70_45	-0.92***	-0.82***	-0.80***

***significant at 0.1% level



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Fig. 5. Scatterplot of D_{tot} vs shear strength measured after delamination on the same glue line. Example for the method DS_50_45.

Table 8

Pearson correlation coefficients (r) between the several methods for the mean values for *beech* panel of shear strength

	S_50_45	S_70_45	DS_50_90	DS_50_45	DS_70_45
S 50 90	-	-	-	-	-
s ⁵⁰ 45		-	0.59*	0.53*	0.51*
S_70_45			0.61**	0.52*	0.76**
DS_50_90				0.91***	0.82***
DS_50_45					0.71***

*significant at 5% level; **significant at 1% level; ***significant at 0.1% level; (-) not significant

4. Conclusions

Engineered beech products, such as CLT can provide opportunities for greater use in larger and more sustainable timber constructions. However, the issue of structural bonding remains the one to be solved for the production of glued structural beam or panels made of beech. Here, three adhesives and two press systems were investigated in the production of beech and combined beech-spruce-beech CLT panels; while the evaluation of the bonding parameters was carried out with both standardized and optimized shear and delamination test methods.

None of the adhesive tested met the requirements for delamination tests provided by the current standardization for softwood, while no assessment is possible for shear strength due to the lack of reference values. Anyhow, useful conclusions about the parameters investigated can be drawn.

As for the adhesive, PUR was the one with the poorest performance in the production of beech CLT panels, with very high delaminations and low values of shear strength, both in dry specimens and after pretreatment. The addition of the primer before the spreading of the adhesive improved the bonding quality, so to achieve results comparable to those observed with MUF.

On the contrary, in the production of beech-spruce panels, the three adhesive gave similar outcomes: the shear strength did not distinguished them. The use of MUF resulted in slightly less delaminations, while the delaminations registered for PUR and PUR+P were similar to what observed for PUR+P and MUF in beech panels.

The press system was not a relevant factor. Even if the pressures reached in the two systems were very different, the results in terms of bonding quality were mostly the same.

As concerns the several testing methods used in the work for evaluating the bonding quality, again the delamination test proved to be very severe for CLT and even more for hardwood CLT. A size effect was noticed: the larger the specimen and the greater the delaminations. Despite this, the several methods agreed very well in the evaluation of bonding parameters, so to lead to the conclusion that a specimen size of 50 mm could be enough for the assessment of bonding durability.

The shear tests on dry specimens are decidedly less sensitive; of all, the less effective is the one with the 90° grain orientation in respect to the load, while the 45° orientation seems to better highlight the effects of the gluing parameters. After delamination, the tests become more sensitive and correlate with each other. High correlation was also observed between the total delamination and the shear strength measured after pretreatment, so that this could be used as a measure of the effects of the delamination cycles and also as a appropriate way for evaluating glueline strength and durability.

The test method which combines the delamination pretreatment and the shear strength determination on block specimens of 50 mm size and the grain orientation at 45° in respect to the side of the panel could be suitable for the quality control of the bond lines in CLT panels.

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5. References

[1] R. Brandner, G. Flatscher, A. Ringhofer, G. Schickhofer, A. Thiel, Cross laminated timber (CLT):

overview and development, Eur. J. Wood Prod. 74 (2016) 331-351. doi:10.1007/s00107-015-0999-5.

[2] L. Muszynski, E. Hansen, S. Fernando, G. Schwarzmann, J. Rainer, Insights into the Global Cross-Laminated Timber Industry, 2 (2017) 77–92. doi:biobus.swst.org.

[3] EN 16351, Timber structures. Cross laminated timber. Requirements, CEN - European Committee for Standardization, Brussels, 2015.

[4] S. Aicher, Z. Christian, G. Dill-Langer, Hardwood glulams. Emerging timber products of superior mechanical properties, in: Proceedings of the World Conference on Timber Engineering, WCTE, Québec City, Canada, 2014.

[5] S. Torno, M. Knorz, J.-W. van de Kuilen, Supply of beech lamellas for the production of glued laminated timber, in: Proceedings of the International Scientific Conference on Hardwood Processing, ISCHP, Firenze, Italy, 2013: pp. 210–217.

[6] P. Glos, J.K. Denzler, P.W. Linsenmann, Strength and stiffness behaviour of beech laminations for high strength glulam, in: Proceedings of the CIB W18, Meeting 37, Edinburgh, Scotland, paper 37-6-3, 2004.

[7] T. Ehrhart, G. Fink, R. Steiger, A. Frangi, Experimental investigation of tensile strength and stiffness indicators regarding European beech timber, in: Proceedings of the World Conference on Timber Engineering, WCTE, Vienna, Austria, 2016: pp. 600–607.

[8] T. Ehrhart, G. Fink, R. Steiger, Strength grading of European beech lamellas for the production of GLT & CLT, in: Proceedings of the International Network on Timber Engineering Research, INTER, Meeting 49, Graz, Austria, paper 49-05-1, 2016.

[9] M. Frese, H.J. Blaß, Characteristic bending strength of beech glulam, Mater Struct. 40 (2007) 3–13. doi:10.1617/s11527-006-9117-9.

[10] S. Aicher, D. Ohnesorge, Shear strength of glued laminated timber made from European beech timber, Eur. J. Wood Prod. 69 (2011) 143–154. doi:10.1007/s00107-009-0399-9.

[11] S. Aicher, Z. Christian, M. Hirsch, Rolling shear modulus and strength of beech wood laminations, Holzforschung. 70 (2016). doi:10.1515/hf-2015-0229.

[12] T. Ehrhart, R. Steiger, P. Palma, A. Frangi, Mechanical properties of European beech glued laminated timber, in: Proceedings of the International Network on Timber Engineering Research, INTER, Meeting 51, Tallin, Estonia, paper 51-12-4, 2018.

[13] S. Franke, Mechanical properties of beech CLT, in: Proceedings of the World Conference on Timber Engineering, WCTE, Vienna, Austria, 2016.

[14] S. Aicher, M. Hirsch, Z. Christian, Hybrid cross-laminated timber plates with beech wood crosslayers, Construction and Building Materials. 124 (2016) 1007–1018.

doi:10.1016/j.conbuildmat.2016.08.051.

[15] S. Aicher, M. Hirsch, Z. Christian, Hybrid beech and spruce cross-laminated timber, in: Proceedings of the World Conference on Timber Engineering, WCTE, Vienna, Austria, 2016.

[16] J. Konnerth, M. Kluge, G. Schweizer, M. Miljković, W. Gindl-Altmutter, Survey of selected adhesive bonding properties of nine European softwood and hardwood species, Eur. J. Wood Prod. 74 (2016) 809–819. doi:10.1007/s00107-016-1087-1.

[17] E. Pöhler, R. Klingner, T. Künniger, Beech (*Fagus sylvatica* L.) – Technological properties, adhesion behaviour and colour stability with and without coatings of the red heartwood, Ann. For. Sci. 63 (2006) 129–137. doi:10.1051/forest:2005105.

[18] D. Ohnesorge, K. Richter, U. Seeling, Project "Innovation for Beech" Gluability of Beech Wood Containing Red Heartwood, in: Wood Structure and Properties' 06, Edited by S. Kurjatko, J. Kúdela & R. Lagaňa, Arbora Publishers, Zvolen, Slovakia, 2006: pp. 471–474.

[19] S. Aicher, H.-W. Reinhardt, Delaminierungseigenschaften und Scherfestigkeiten von verklebten rotkernigen Buchenholzlamellen, Holz Roh Werkst. 65 (2007) 125–136. doi:10.1007/s00107-006-0135-7.

[20] D. Ohnesorge, K. Richter, G. Becker, Influence of wood properties and bonding parameters on bond durability of European Beech (Fagus sylvatica L.) glulams, Ann. For. Sci. 67 (2010) 601–601. doi:10.1051/forest/2010002.

[21] A. Brandmair, S. Clauß, P. Haß, P. Niemz, Bonding of hardwoods with 1C PUR adhesives for

timber construction, Bauphysik. 34 (2012) 210-216. doi:10.1002/bapi.201200025.

[22] M. Schmidt, P. Glos, G. Wegener, Gluing of European beech wood for load bearing timber structures, Eur. J. Wood Prod. 68 (2010) 43–57. doi:10.1007/s00107-009-0382-5.

[23] O. Kläusler, P. Hass, C. Amen, S. Schlegel, P. Niemz, Improvement of tensile shear strength and wood failure percentage of 1C PUR bonded wooden joints at wet stage by means of DMF priming, Eur. J. Wood Prod. 72 (2014) 343–354. doi:10.1007/s00107-014-0786-8.

[24] J. Luedtke, C. Amen, A. van Ofen, C. Lehringer, 1C-PUR-bonded hardwoods for engineered wood products: influence of selected processing parameters, Eur. J. Wood Prod. 73 (2015) 167–178. doi:10.1007/s00107-014-0875-8.

[25] K. Casdorff, O. Kläusler, J. Gabriel, C. Amen, C. Lehringer, I. Burgert, T. Keplinger, About the influence of a water-based priming system on the interactions between wood and one-component polyurethane adhesive studied by atomic force microscopy and confocal Raman spectroscopy imaging, International Journal of Adhesion and Adhesives. 80 (2018) 52–59. doi:10.1016/j.ijadhadh.2017.10.001.

[26] O. Kläusler, K. Rehm, F. Elstermann, P. Niemz, Influence of wood machining on tensile shear strength and wood failure percentage of one-component polyurethane bonded wooden joints after wetting, International Wood Products Journal. 5 (2014) 18–26. doi:10.1179/2042645313Y.0000000039.

[27] M. Knorz, M. Schmidt, S. Torno, J.-W. van de Kuilen, Structural bonding of ash (Fraxinus excelsior L.): resistance to delamination and performance in shearing tests, Eur. J. Wood Prod. 72 (2014) 297–309. doi:10.1007/s00107-014-0778-8.

[28] M. Knorz, E. Neuhaeuser, S. Torno, J.-W. van de Kuilen, Influence of surface preparation methods on moisture-related performance of structural hardwood–adhesive bonds, International Journal of Adhesion and Adhesives. 57 (2015) 40–48. doi:10.1016/j.ijadhadh.2014.10.003.

[29] S. Ammann, S. Schlegel, M. Beyer, K. Aehlig, M. Lehmann, H. Jung, P. Niemz, Quality assessment of glued ash wood for construction engineering, Eur. J. Wood Prod. 74 (2016) 67–74. doi:10.1007/s00107-015-0981-2.

[30] M. Knorz, S. Torno, J.-W. van de Kuilen, Bonding quality of industrially produced cross-laminated timber (CLT) as determined in delamination tests, Construction and Building Materials. 133 (2017) 219–225. doi:10.1016/j.conbuildmat.2016.12.057.

[31] K.S. Sikora, D.O. McPolin, A.M. Harte, Shear Strength and Durability Testing of Adhesive Bonds in Cross-laminated Timber, The Journal of Adhesion. 92 (2016) 758–777.

doi:10.1080/00218464.2015.1094391.

[32] R. Steiger, E. Gehri, K. Richter, Quality control of glulam: shear testing of bondlines, Eur. J. Wood Prod. 68 (2010) 243–256. doi:10.1007/s00107-010-0456-4.

[33] P. Lavisci, S. Berti, B. Pizzo, P. Triboulot, R. Zanuttini, A shear test for structural adhesives used in the consolidation of old timber, Holz Als Roh- Und Werkstoff. 59 (2001) 145–152. doi:10.1007/s001070050486.

[34] M. Betti, M. Brunetti, M.P. Lauriola, M. Nocetti, F. Ravalli, B. Pizzo, Comparison of newly proposed test methods to evaluate the bonding quality of Cross-Laminated Timber (CLT) panels by means of experimental data and finite element (FE) analysis, Construction and Building Materials. 125 (2016) 952–963. doi:10.1016/j.conbuildmat.2016.08.113.

[35] M. Dugmore, M. Nocetti, M. Brunetti, Z. Naghizadeh, C.B. Wessels, Bonding quality of crosslaminated timber: Evaluation of test methods on Eucalyptus grandis panels, Construction and Building Materials. 211 (2019) 217–227. doi:10.1016/j.conbuildmat.2019.03.240.

[36] E.A. Okkonen, B.H. River, Factors affecting the strength of block-shear specimens, Forest Product Journal. (1989) 8.