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Assessment of bonding durability of CLT and glulam made from oak and mixed poplar-oak according to bonding pressure and glue type

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- 3 4

5 Abstract

6 The objective of this study is to evaluate the influence of manufacturing parameters on the bonding quality of CLT 7 and glulam made from oak and mixing poplar and oak species. Studied bonding parameters were bonding pressure 8 and glue type. To test the bonding durability, the specimens were subjected to a vacuum pressure cycle followed 9 by drying to the initial weight. The bonding quality was then measured from delamination, residual shear strength, 10 and wood failure percentage (WFP). Bonding pressure and type of glue appeared to have a significant influence 11 on the bonding quality of CLT or glulam, with also significant interactions with the kind of species bonded. If a 12 0.8 N/mm² bonding pressure seemed to provide better results, vacuum-pressed (0.085 N/mm² bonding pressure) 13 mixed poplar-oak CLT or glulam glued with PUR were very close to reaching the requirements of the standards. 14 Generally, CLT or glulam entirely made from oak delaminated more than the mixed specimens. However, their 15 residual shear strength can be comparable or even superior to what was obtained with mixed poplar-oak specimens. As a result, residual shear strength after delamination test may be interesting to consider as an additional criterion 16 17 to assess glue line integrity of hardwood CLT or glulam products. 18 Keywords: Engineered wood products, hardwood, gluing, delamination, shear 19 20 Citra Yanto Ciki Purba 21 Arts et Métiers Institute of Technology, LaBoMaP 22 Rue Porte de Paris, 71250 Cluny, France 23 e-mail: citra.purba@ensam.eu 24 25 Guillaume Pot 26 Arts et Métiers Institute of Technology, LaBoMaP 27 Rue Porte de Paris, 71250 Cluny, France 28 29 Robert Collet 30

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40 1 Introduction

- 41 A shift toward forests mixing more softwood and hardwood species in the future has been discussed in many
- 42 studies [1–3]. In France, oak is already a widely distributed hardwood species which represents 27% of the total
- volume of standing trees [4]. In addition, as it has a higher tolerance to warmer and drier climates it is considered
- 44 more adapted to the future climates in comparison to beech, for example [5]. An increasing interest is shown in
- the manufacturing of engineered wood products (EWP) made from hardwoods, such as cross-laminated timber (CLT) or glued laminated timber (glulam). Both of these EWPs show a strong potential to enable the use of the
- 47 lowest qualities of hardwoods in the construction industry [6,7].

48 Currently, both CLT and glulam are almost exclusively manufactured from softwood lumber, mainly spruce and 49 fir. EWP made from softwood generally has lower wood density than hardwood ones, thus has also lower stiffness and strength, and especially low rolling shear strength [8,9]. High density is usually associated with enhanced 50 51 strength and stiffness characteristics as well as increased shrinkage and swelling under moisture changes. 52 Therefore, the stresses in bonds induced by moisture changes may be significantly higher [10]. Higher wood density also means lower porosity and higher cell wall thickness and as a result, less penetration of adhesive which 53 54 may cause weaker bonds. For this reason, it is generally assumed that high-density woods tend to have a lower 55 bonding performance [11–13]. In addition, more complex hardwood anatomic features and chemical compounds 56 make hardwood gluing more sophisticated. A study by Sikora et al. [11] demonstrated the positive correlation 57 between the wood density with rolling shear strength and the negative correlation with bonding durability.

58 The wood bonding durability is generally assessed through so-called delamination tests consisting in wetting and

59 drying cycles which induce high stresses in the glue lines. Adhesives generally exhibit low swelling capacity,

60 whereas wood swells with moisture in an anisotropic manner. This constraint caused by the adhesive leads to very

61 high stresses in the glue line [14]. The percentage of delamination length and wood failure percentage (WFP) are 62 the criteria to quantify the delamination. The delamination length is the extent of splitting that occurs between two

63 neighboring layers of wood. It is reported as a percentage of the total length of the joint. WFP is a different criterion

- 64 measured after the complete splitting of the glue line and related to the percentage of the glued surface in which
- 65 the rupture occurred in or between wood fibers.

A common way to measure the bonding strength is through a dry shear test. This method, however, is considered
 ineffective in determining the influence of various bonding parameters on the bonding quality as it lacks
 consistency due to the presence of rolling shear failure especially in CLT [15]. However, performing a shear test

69 after wetting and drying cycle which led to delamination make the test more sensitive and objective to the influence

- 70 of the manufacturing process [13,15,16].
- Several studies have been conducted to characterize the bonding ability of oak, showing the good shear strength of glue lines of oak glulam glued with polyurethane (PUR) or melamine-urea-formaldehyde (MUF) at dry
- conditions [17–19]. However, in terms of resistance to stresses caused by moisture changes as in a delamination
- test, the results in the literature are more contrasted. A study by Konnerth et al. [18] on six plies glulam made of
- 75 oak or beech showed that samples using MUF, PUR, and phenol-resorcinol-formaldehyde (PRF) adhesives pass
- the delamination requirements, but glulam samples made of poplar glued with MUF did not pass the requirement.
- In contrast, Luedtke et al. [17] showed that six plies glulam made from oak glued with PUR did not meet the delamination standard. These authors added that within the tested hardwood species, the diffuse-porous species
- outperform the ring-porous species *i.e.* oak. The problem with gluing oak is that the wood pattern is ring porous which means that the variations in density and permeability are very distinct between earlywood and latewood
- 81 [20].

The behavior of CLT is not the same as glulam in delamination tests since the orientations of the lamellas are different. Beech glulam may pass the delamination requirements [18,21] but not beech CLT. Brunetti et al. [15]

- 84 showed that neither the MUF nor the PUR being used to bond beech CLT met the delamination test requirements
- standard originally made for softwood [22]. Based on previous studies, it is clear that EWPs made of hardwood
- species, especially CLT, are unlikely to pass the European standard requirement for delamination but can easily exceed the dry shear strength requirements. To date, there has been no report on the bonding durability of oak
- 87 exceed the88 CLT.
- Fabrication of EWP mixing wood species is a growing research topic [15,23–26]. Special attention needs to be given since strong wood density differences may affect negatively the bonding properties especially in delamination tests [10]. The advantage of using mixed species has been shown by Castro and Paganini [23] in
- 92 which mixed glulam made from poplar and eucalyptus showed higher structural efficiency in bending than those

- 93 entirely constructed from poplar or eucalyptus. Poplar has also shown interest in the fabrication of mixed-species
- 94 CLT with Douglas-fir and Monterey pine (Pinus radiata), for which the mechanical properties were comparable to
- 95 those made of non-poplar wood [27]. The utilization of poplar as a single CLT material has been also studied
- 96 before [27–29].
- 97 Several studies showed that MUF bonded hardwood produced more than 2 times lower delamination compared to
- 98 PUR bonding [30–32]. A recent study on white oak, white ash, yellow birch, and birch glulam specimens bonded
- 99 with melamine-formaldehyde (MF) and two-component PUR (2C-PUR) showed that the loss of strength induced
- by the moisture and differential wood swelling caused by the vacuum-pressure cycle was considerable (50%), especially for high-density specimens bonded with 2C-PUR adhesive (around 60% reduction) [32]. Knorz et al.
- [30] compared the bonding quality of glulam made of ash with various adhesives *i.e.* PRF, MUF, PUR, emulsion
- polymer isocyanate (EPI), and varying closed assembly times as a bonding parameter. For all tested adhesives and
- 104 closed assembly times, the shear tests showed high WFPs and strength values that are comparable to solid ash. In
- 105 contrast, for delamination tests, significant differences were found between the adhesives as well as between closed
- assembly times, with improving resistance to delamination for increased closed assembly times. However, none
- 107 of the five adhesives satisfied the requirement of the current standard. The application of a primer product in
- 108 combination with a 1C-PUR adhesive appears to be a promising approach for hardwood, and especially oak [17].
- 109 The two main surface bonding machines used by CLT producers are hydraulic press and vacuum press, which
- 110 differ considerably with regard to bonding pressures. The pressure obtained in a vacuum press lies below 0.1
- 111 N/mm² while the pressure obtained with a hydraulic press is generally above 0.4 N/mm². Several studies have
- reported the influence of the bonding pressure on the bonding quality. Knorz et al. [33] studied the influence of
- bonding pressure (0.085 vs 0.8 N/mm²) and the timber thickness (20 mm vs 40 mm) on the delamination of spruce
- 114 CLT glued with 1C-PUR. Their results show that both parameters had no impact on the results of delamination 115 tests. A more recent study on eucalyptus CLT glued with 1C-PUR showed that lower pressure (0.1 N/mm²) results
- 115 tests. A more recent study on eucalyptus CLT glued with 1C-PUR showed that lower pressure (0.1 N/mm²) results 116 in higher delamination failure [13]. Therefore, a higher pressure seems preferable for hardwood CLT. More
- pressure results in deeper adhesive penetration and, therefore, better bond durability [11]. For these authors, CLT
- bonded under high pressure (1 N/mm²) showed up to 100% WFP in the bonding line while CLT bonded under low
- 119 pressure $(0.4-0.6 \text{ N/mm}^2)$ can reach as low as 40% WFP.
- 120 There is still no reported results in the available literature investigating whether oak CLT resist to delamination 121 tests or not, nor exploring what is the bonding performance when oak is mixed with another species. Therefore,
- tests or not, nor exploring what is the bonding performance when oak is mixed with another species. Therefore, the objectives of this study are to provide the first results of oak CLT delamination and residual shear strength
- 123 obtained after the treatment intended for delamination test. The influence of glue type and bonding pressure on the
- delamination and residual shear strength will be studied for CLT and glulam made from oak and mixed CLT and
- 125 glulam made from poplar and oak.

126 2 Material and methods

127 2.1 Wood material

- 128 Oak (*Quercus petraea* (Matt.) Liebl) boards with cross-sections of 88 mm x 25 mm as well as poplar (*populous alba*) boards with dimensions of 88 mm x 23 mm coming from a local forest in Burgundy, France were used for
- 130 the production of 3-plies CLT and glulam panels. All lamellas showed annual growth rings with tangential
- 131 orientation (flat-sawn boards). Flat sawing was chosen to ensure high rolling shear properties. A previous study
- by Aicher et al. [34] showed that quarter sawn boards resulted in the lowest rolling shear thus were less suitable
- 133 for CLT fabrication.
- 134 Less than 24 hours before fabrication, all sides of the oak boards were planed to the desired thicknesses of 19.5
- 135 mm for the outer layers and 23 mm for the inner layer, with a width of 86 mm. The poplar inner layer thickness
- 136 was reduced to 21 mm. The mean density for oak wood was 0.707 kg/m^3 with a standard deviation of 0.107 kg/m^3
- 137 while poplar mean density was 0.341 kg/m³ with a standard deviation of 0.014 kg/m³. The mean value and standard
- 138 deviation of oak boards' moisture content (MC) were 9.2 ± 0.5 % while poplar MC was 9.3 ± 0.3 % which satisfies
- the glue manufacturer's requirement which is a MC between 6 % to 15 %.

140 2.2 Manufacturing of CLT and glulam

- 141 To study the influence of the adhesives on bonding quality, PUR and MUF were employed in this study. PUR is
- broadly used in the industry since it is more eco-friendly. MUF is cheaper compared to PUR but less eco-friendly
- 143 as it contains formaldehyde. However, MUF had been proved to provide higher wood bonding quality than PUR.

144 Both PUR and MUF are cold-setting chemical adhesive systems that meet the requirements of adhesive type I 145 according to EN 15425 [35] for structural bonding of wood.

146 3-layered CLT and glulam panels were assembled in two different compositions. First composition was CLT or

147 glulam entirely made from (named oak) with nominal dimensions of 400 mm x 400 mm x 62 mm. The second

148 composition was CLT or glulam made from oak as outer layers and poplar as inner layer (oak-poplar-oak, named

149 mixed poplar-oak) with nominal dimensions of 400 mm x 400 mm x 60 mm. CLT lamellas orientation was cross-

150 layered while glulam lamellas were parallel. The boards were sanded just before bonding in order to obtain smooth

- surfaces suited to the process and to assemble oak panels 62 mm thick and mixed poplar-oak panels 60 mm thick. 151
- 152 The time between surface preparation and bonding was at most 6 h. Surfaces were cleaned using filtered
- 153 compressed air before gluing, in order to remove dust.

The adhesive was applied to the faces of the lamellas using a spatula. The amount of adhesive spread, as well as 154

155 assembly time and pressing time, were in accordance with adhesive manufacturers' recommendations. The spread

- rate of PUR was about 150 g/m² and, when used, the primer amount was of 20 g/m²; MUF adhesive spread was 156
- about 300 g/m². The primer was applied to each adherend and was followed by an open time of 30 min before the 157
- 158 application of PUR. The closed assembly time for PUR was not more than 60 min, while for MUF it was 30 min. Table 1. Outline of the treatment, number of specimens used and code used to shorten the names

No	Code	Type of assembly	Species	Pressure	Glue type	Nb layers	Nb specimens
1	CMHM	CLT	Mix	Hydraulic	MUF	3	9
2	CMHP	CLT	Mix	Hydraulic	PUR	3	9
3	CMVM	CLT	Mix	Vacuum	MUF	3	9
4	CMVP	CLT	Mix	Vacuum	PUR	3	9
5	COHM	CLT	Oak	Hydraulic	MUF	3	9
6	COHP	CLT	Oak	Hydraulic	PUR	3	9
7	COVM	CLT	Oak	Vacuum	MUF	3	9
8	COVP	CLT	Oak	Vacuum	PUR	3	9
9	GMHM	Glulam	Mix	Hydraulic	MUF	3	9
10	GMHP	Glulam	Mix	Hydraulic	PUR	3	9
11	GMVM	Glulam	Mix	Vacuum	MUF	3	9
12	GMVP	Glulam	Mix	Vacuum	PUR	3	9
13	GOHM	Glulam	Oak	Hydraulic	MUF	3	9
14	GOHP	Glulam	Oak	Hydraulic	PUR	3	9
15	GOVM	Glulam	Oak	Vacuum	MUF	3	9
16	GOVP	Glulam	Oak	Vacuum	PUR	3	9

160

159

161 To study the influence of bonding pressure on the bonding quality, a vacuum press (pressure: 0.085 ± 0.05 N/mm²)

162 and a hydraulic press (pressure: 0.8 N/mm²) were used. After manufacturing, the panels were stored for 30 days 163 at 20.2 C and 37.5 % of relative humidity.

164 2.3 Measurement of bonding quality

The testing materials, consisting of one 400 x 400 mm² panel for each of 16 experimental modalities (2 lamella 165 166 orientation x 2 press typologies x 2 species combinations x 2 adhesives), were cut into 9 specimens each with dimensions of 100 mm x 100 mm x 60 (or 62) mm. The 100 mm x 100 mm dimension corresponds to the 167 168 dimensions required for the CLT delamination test of EN 16351:2015 [22]. It has been chosen to keep the same 169 dimensions for the glulam specimens, although EN 14080:2013 [36] specify a specimen length in grain direction 170 of 75 ± 5 mm. The position of the specimens inside the panels is presented in Fig. 1. Wood bonding quality was 171 then assessed through the delamination test, measurement of wood failure percentage, and shear test after the

172 delamination cycle.



174 175

Fig. 1. Sampling position and configuration of oak and mix poplar oak specimen.

176 **Delamination test**

177 In this research study, the delamination test as defined in EN 16351:2015 [22] for CLT was applied for both CLT and glulam (it corresponds to method B of EN 14080:2013 [36] for glulam). The test was performed in one cycle. 178 179 The specimens were placed in a vacuum pressure vessel with end grain surfaces exposed to water. A vacuum of 180 75 kPa was first drawn for 30 min. The vacuum was then released and pressure was applied around 550 kPa for 2 181 h. The test pieces were then dried at 70°C until the mass of the test pieces has returned to 110 % of the original 182 mass. The specimen weight before and after the wetting and drying cycle was measured using an analytical scale Sauter RC-8021 with 0.1 g resolution. After reaching their target weight, the specimens were immediately removed 183 184 from the oven for visual inspection.

185

188

As defined in EN 16351:2015 [22] for CLT the total delamination length (%) of a test piece has been calculated
 from Eq. (1):

$$D_{tot \ CLT} = 100 \frac{l_{tot,delam}}{l_{tot,glue \ line}} \tag{1}$$

189 Where $l_{tot, delam}$ is the total delamination length, in mm; $l_{tot, glue line}$ is the sum of the perimeters of all glue lines in a

delamination specimen, in mm. The EN 16351:2015 [22] standard requires that the total delamination length

- 191 should not exceed 10% of the sum of all glue lines. The delamination length was measured using a ruler with a ± 1 mm precision.
- 193 The maximum delamination (%) for CLT was calculated using Eq. (2):

$$D_{\max CLT} = 100 \frac{l_{\max,delam}}{l_{glue \ line}}$$
(2)

Where $l_{max, delam}$ is the maximum delamination length, in mm; $l_{glue \ line}$ is the perimeter of one glue line in a delamination specimen, in mm. The EN 16351:2015 [22] requires that the maximum delamination length of each specimen should not exceed 40% of the total length of a single glue line.

As defined in EN 14080:2013 [36] for glulam the total delamination length (%) of a test piece has been calculated
from Eq. (3):

200
$$D_{tot glulam} = 100 \frac{l_{end,tot,delam}}{l_{end,tot,dlue line}}$$
(3)

Where $l_{end,tot,delam}$ is the total delamination length on both end-grain surfaces, in mm; $l_{end,tot,glue \ line}$ is the entire length of the glue lines on both end-grain surfaces, in mm. The EN 14080:2013 [36] standard requires that the total delamination length on both end-grain surfaces should not exceed 4% of the entire length of the glue lines on
 both end-grain surfaces.

- 205 The maximum delamination for glulam (%) was calculated based on Eq. (4):
- 206 207

$$D_{\max glulam} = 100 \frac{l_{end.max,delam}}{2.l_{end} glue line}$$
 (4)

208 Where $l_{end.max,delam}$ is the maximum delamination length, in mm; $l_{end glue line}$ is the length of one glue line on 209 the end-grain surface, in mm. The EN 14080:2013 [36] standard requires that the maximum delamination length 210 of each specimen should not exceed 30 %.

211 Shear test

The shear strenght behavior was determined in a block shear test according to EN 16351:2015 [22] and EN 14080:2013 [36] as presented in Fig. 2. After the delamination procedure, the specimens were tested parallel to the grain with the shear plane corresponding to the adhesive layer. The shear test was performed with a universal testing machine (ZWICK) using a displacement rate of 2 mm/min. The shear strength f_v was calculated by means of the load at failure F_u and the cross-section A of the respective specimen (Eq. 5):

$$f_{\nu} = \frac{F_{u}}{4} \tag{5}$$

218 F_u is the ultimate load, in N; A = 100 x 100 mm² is the sheared area.



219

220

Fig. 2. Loading position in the shear test for glulam and CLT.

221 Measurement of WFP

The measurement of WFP was performed right after the shear test. For most specimens, the glued surfaces were fully split. For the rest, which was not completely split, each glue line has been cut open with a chisel. For panels made of MUF, the areas where the glue failed were white, smooth, and shiny while the areas of wood failure were rough due to rip-off wood grain or extra fiber added to the surface. In contrast, for PU, the glue failure areas were rough with a color often similar to the wood. These factors make it difficult to automate the measurement of WFP, as it relies heavily on color contrast.

228 A contrast between the area with solid wood failures and non-solid wood failures was enhanced by applying black 229 ink on the plain area or area of non-solid wood failures (Fig. 3a). An image of each glued surface was then taken. 230 An image analysis system (ImageJ) was then used to calculate the wood failure percentage. The WFP of a split 231 glued area was calculated as the ratio of the area with solid wood failures and the glued area before splitting. WFP 232 total was defined as the ratio of the total area with solid wood failures to the total area of glue lines whereas WFP 233 minimum was determined as the ratio of the area where the solid wood failures are minimum to the glued area 234 before splitting. The colored image was first converted into 3 channels of grey level image (red, green, blue) 235 followed by the application of a common color threshold on the red channel image in order to select the designated 236 wood failure area. The particle analysis of image-J was then used to calculate the wood failure percentage as 237 presented in Fig. 3. The same procedure was performed to calculate WFP on the glulam samples.

238 For CLT, the EN 16351:2015 [22] standard allows to measure the WFP after the delamination test and splitting

the specimens, so that if the delamination lengths do not fulfill the requirements, the test can be passed if the

minimum WFP of a single bonding area is higher than 50% and the minimum WFP of the sum of all split bondingareas in the sample is higher than 70%.





242

Fig. 3. Measurement of WFP using image analysis: a) red channel image, b) thresholded image (0,85), c) particle
 analysis to calculate the area of non-joint failure.

247 Data analysis

An analysis of variance (ANOVA) was performed to evaluate the influence of each fabrication parameter and their interaction. Therefore, delamination, WFP, and residual shear strength were used as the dependent variables while pressure, adhesive, assembly type, species, and their interactions were used as the independent variables. A post hoc analysis (Tukey's HSD test) was calculated for those that showed significant differences as a result of the ANOVA. The Pearson correlation coefficients were also calculated to investigate the correlations between residual shear strength, delamination, and WFP.

254 3 Results and discussion

255 Delamination and WFP

256 The average values and standard deviations for the different combinations of parameters and testing methods are 257 presented in Fig. 4 while the results of the analysis of variance are summarized in Table 2. The mean total 258 delamination length for all specimens was 23.18 % while the mean WFP was 69.89 %. The mean total delamination 259 length for oak CLT (41.02 %) was nearly two times the one found for mixed poplar-oak CLT (20.85 %). 260 Meanwhile, the total delamination length for oak glulam was five times higher than mixed poplar-oak glulam 261 (27.43% VS 4.41%). Consistent with the delamination, the mean WFP of mixed poplar-oak CLT (81.36%) was two times higher than oak CLT (39.07%). The mean WFP of mixed poplar-oak glulam (92.93%) was also higher 262 263 than oak glulam (65.02%).



267 Fig. 4. Barplot of the percentage of total delamination, maximum delamination of the single glue line, WFP total, and minimum WFP in a single glue line along with the limit value required by EN 16351:2015. The error bar 268 269 represents the standard deviation. The dashed line shows the threshold for CLT according to EN 16351:2015. The 270 dotted line shows the threshold for glulam according to EN 14080:2013 [36].

271 Concerning the influence of the factors examined, the assembly type, wood species, and pressure were highly 272 predictive and statistically significant factors influencing both total delamination length and WFP, while the glue 273 used was much less predictive and statistically significant for WFP only (Table 2). Generally, specimens entirely made of oak exhibited more delamination than the mixed ones. Glulam and hydraulic press configurations also 274 275 seem to produce lower delamination and higher WFP. Concerning the significant influence of interaction between 276 glue and assembly type, it appears that glulam bonded with PUR generates the lowest delamination and highest 277 WFP while CLT bonded with PUR produces the highest delamination. There was also a significant and high 278 influence of the interaction between pressure and glue type: delamination results were better for MUF when the 279 0.8 N/mm² pressure in a hydraulic press was applied instead of the 0.085 N/mm² pressure in a vacuum press, 280 whereas no clear conclusion can be drawn when PUR was used. For similar reasons, the interaction between 281 species, glue, and assembly type appears to be highly significant.

- 282
- 283
- 284
- 285

Table 2. F values and significance as results of the four-way analysis of variance for the total delamination and
 WFP observed in the CLT and Glulam

	D _{tot}	WFP	
pressure	21.10***	29.88***	
species	71.15***	189.78***	
glue	1.44	5.39*	
type	29.25***	51.55***	
pressure * species	3.90	4.06*	
pressure *glue	30.03***	40.84***	
species *glue	9.74**	0.13	
pressure *type	0.67	1.16	
species *type	0.01	8.65**	
glue*type	15.69***	18.64***	
pressure * species *glue	0.00	0.00	
pressure * species *type	0.93	2.21	
pressure *glue*type	2.56	0.10	
species *glue*type	26.96***	11.79***	
pressure * species *glue*type	1.48	1.64	

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

289 Table 3 presents the results of the Tukey test, which was performed to evaluate the influence of species, glue, and

assembly type on the difference in mean total delamination and WFP. The glulam made from mixed poplar-oak

bonded with PUR showed the lowest delamination and the highest WFP. For CLT configuration, mixed poplar-

292 oak bonded with PUR seemed to produce the lowest delamination and highest WFP.

Table 3. Tukey test for all significant combinations. C, CLT; G, glulam; O, oak; M, mixed poplar-oak; P, PUR;
 M, MUF. Means that do not share a letter are significantly different at 5 % level based on the Tukey Post Hoc test

turno* annoina *alua	Number	Meen D	Meen WED
type species give	Nulliber	Ivieali D _{tot}	
СОР	17	55.60 ^A	31.60 ^A
GOM	18	37.00 ^B	52.50 ^B
СОМ	18	27 .00 ^{BC}	46.40 ^{AB}
СММ	19	27 .00 ^{BC}	79.60 [°]
GOM	18	18.30 ^{CD}	77.50 [°]
СМР	16	12.70^{CDE}	84.30 ^{CD}
GMM	18	9.60 ^{DE}	88.30 ^{CD}
GMP	19	1.67^{E}	97.40 ^D

295 The results of the pass and fail analysis for all treatment and test methods are reported in Table 4. Overall, only 296 27.14 % of CLT specimens met the requirement of the standard EN 16351:2015 [22] for both total delamination 297 and maximum delamination. Considering the second step of the evaluation, the percentage of specimens that reached the minimum WFP provided by the standard rose to 60.1 %. For glulam, 60.64 % of specimens pass the 298 299 30 % threshold of maximum delamination and 49.31 % pass the total delamination required by standard EN 14080:2013 [36]. Only two glulam configurations (GMHM and GMHP) had a 100% passing rate based on 300 301 delamination while there was only one CLT configuration (CMHM) that passed the minimum requirement based 302 on WFP. All of those three panels were constituted of mixed poplar-oak layers and were assembled using the hydraulic press. However, both of the vacuum-pressed mixed poplar-oak glulam and CLT bonded with PU (CMVP 303 304 and GMVP) were close to reaching the requirements of the standard with more than 85% of the specimen that 305 fulfilled the criteria.

None of the specimens entirely made of oak had a 100 % passing rate for both delamination and WFP. In addition, there were two configurations (COVP and GOVM) made from oak that had a 0 % passing rate for both delamination and WFP. Among all 4 criteria presented in Table 4, total delamination was found to be the most downgrading criteria for CLT (27.14 % of all CLT specimens) while WFP total is the one that gave the highest rate of success (49.13 % of all CLT specimens).

A previous study by Konnerth et al. [18] has shown that glulam made from oak glued with MUF passed the delamination minimum according to EN 301-2 unlike what we have found in the present work. Added to this, the

313 pressure used by them was 40% higher than the pressure used in our study ($1.4 \text{ N/mm}^2 \text{ vs } 1 \text{ N/mm}^2$). Concerning

the performance of oak CLT in the delamination test, the delamination resistance was still better compared to the

315 other major European hardwood species *i.e.* beech [15]. These results highlight how the minimum delamination

rates required by EN 16351:2015 [22] are more difficult to achieve for CLT entirely made of hardwood such as oak or beech than for CLT made of softwood. A too harsh delamination test (which has been initially developed for softwood species and glulam type with the grain oriented in the same direction) or an inappropriate delamination limit for hardwood has been discussed in many reports [15,16,18,30].

Table 4. Percentage of the specimen that passes tests related to total delamination, maximum delamination of the
 single glue line, total solid wood failure, and minimum WFP in single glue line required by EN 16351:2015 [22].
 C, CLT; G, glulam; O, oak; M, mixed poplar-oak; H, Hydraulic; V, Vacuum; P, PUR; M, MUF. NA: not applicable

323

NT.	0.1	Percentage of specimen passing the test by criteria						
NO	Code	$D_{tot}(\%)$	D _{max} (%)	WFP (%)	WFP min (%)	All criteria		
1	CMHM	44,44	66,67	100,00	100,00	100,00		
2	CMHP	25,00	75,00	75,00	75,00	75,00		
3	CMVM	20,00	20,00	50,00	50,00	50,00		
4	CMVP	75,00	87,50	100,00	87,50	87,50		
5	COHM	55,56	100,00	55,56	55,56	55,56		
6	COHP	0,00	12,50	12,50	25,00	12,50		
7	COVM	0,00	33,33	0,00	0,00	0,00		
8	COVP	0,00	0,00	0,00	0,00	0,00		
9	GMHM	100,00	100,00	NA	NA	100,00		
10	GMHP	100,00	100,00	NA	NA	100,00		
11	GMVM	22,22	55,56	NA	NA	22,22		
12	GMVP	88,89	88,89	NA	NA	88,89		
13	GOHM	11,11	33,33	NA	NA	11,11		
14	GOHP	22,22	55,56	NA	NA	22,22		
15	GOVM	0,00	0,00	NA	NA	0,00		
16	GOVP	44.44	55.56	NA	NA	44.44		

324

A higher passing rate with WFP criteria than with the delamination length criteria is in accordance with a previous study by Brunetti et al. [15]. However, measuring WFP is rather more subjective than measuring delamination length because it requires an operator to open the glue line in which each operator proceeds in his own way, and the WFP is measured visually by an operator and not by the image analysis used in the present work. Measurement

329 of WFP by image analysis after a shear test seems to be more objective and repetitive.

330 Lower delamination for mixed poplar-oak CLT specimens in this study is also in accordance with the study of 331 Brunetti et al. [15] on CLT constituted of beech and spruce. However, these authors did not explain the reason for this behavior. The proposed explanation is as follows. For these 3 plies specimens, the middle layer is the one 332 333 subjected to the higher stresses because most of the outer layers are freer to swell, not having any boundary 334 condition on their free faces. The inner layer of a CLT specimen is subjected to high compressive stresses because 335 its swelling is blocked by the longitudinal outer layers which cannot stretch due to high wood anisotropy. The 336 interface between inner and outer layers is thus subjected to shear stresses. Delamination should occur when 337 specimens are swelling when the shear stresses in the glue line become greater than the shear strength of the glue 338 line. If the middle lamella is made from poplar, the swelling is less important than for oak, thus the shear stresses 339 in the glue line are less important. In addition, poplar having a low compressive strength, a plastification/damaging 340 perpendicular to the grain phenomenon may occur in the poplar middle lamella, releasing some energy and thus 341 limiting the shear stresses in the glue lines. Oak having a much greater perpendicular to the grain compressive 342 strength in addition to a higher swelling, when the middle lamella is made from oak, the shear stresses induced are 343 greater than when poplar is used, and they can be greater than glue line shear strength. Moreover, it should be 344 noticed that the shear strength of the glue line may be different between an oak/poplar interface and an oak/oak 345 interface.

346 Shear strength after delamination

347 The residual shear strength of specimens after the delamination cycle is presented in Fig. 5. In Table 5, the results

of the analysis of variance are reported, with shear strength as the dependent variable and assembly type, species, pressure, glue, and their interaction as the independent factors included in the model. The type of assembly was

the most important factor determining the residual shear strength, with glulam specimens demonstrating higher

351 residual shear strength. There is only the wood species parameter that did not show a significant influence on the

- 352 residual strength. However, for three-way interaction, it can be seen that the interaction of species, glue, and type
- 353 of assembly have a significant influence on the shear strength.

Table 5. F-values and significance as results of the four-way analysis of variance for residual shear strength observed in the CLT and Glulam

	F	P-value	significance
pressure	6.67	0.01	*
species	0.32	0.57	
glue	4.50	0.04	*
type	195.67	0.00	***
pressure * species	0.27	0.60	
pressure *glue	19.81	0.00	***
species *glue	0.15	0.70	
pressure *type	0.18	0.67	
species *type	0.90	0.34	
glue*type	21.76	0.00	***
pressure * species *glue	0.51	0.48	
pressure * species *type	0.40	0.53	
pressure *glue *type	3.19	0.08	
species *glue *type	9.14	0.00	**
pressure * species * glue *type	0.01	0.94	

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

The Tukey test was performed for the significant factors in the ANOVA and the outcomes (mean values and significance of the difference between means) for the glue, type of assembly, and species interaction are

summarized in Table 6. It can be seen that glulam made from oak or mixed poplar-oak bonded with PUR provided

360 the highest residual shear strength.

Table 6. Tukey test type*species* glue for the mean of residual shear strength. C, CLT; G, glulam; O, oak; M as
 a second character, mixed poplar-oak; P, PUR; M as a third character, MUF

type* species* glue	Number	Mean	Grouping				
GOP	18	6,50	А				
G M P	19	5,93	А	В			
GMM	18	4,88		В	С		
GOM	18	4,11			С	D	
СОМ	18	2,76				D	E
СМР	16	1,73					Е
СММ	19	1,51					Е
СОР	17	1,24					E

363 Means that do not share a letter are significantly different at 5 % level based on the Tukey Post Hoc test.

The mean residual strength of all glulam specimens was higher (5.36 N/mm²) than CLT specimens (1.81 N/mm²). 364 365 Performing block shear test after the delamination procedure is out of the standards: both the EN 16351:2015 [22] and EN 14080:2013 [36] propose to perform these tests on CLT of glulam without any pre-treatment. For 366 367 reference, the standard for CLT production with softwood, EN 16351:2015 [22] sets as sufficient the characteristic value of 1.25 N/mm² for the bonding strength of glue lines between crosswise bonded layers, with no single value 368 under 1 N/mm² (tests performed on dry specimens). Among all the CLT specimens, 71.42 % have shear strength 369 higher than this reference value (80 % of mixed poplar-oak and 62.85 % of oak CLT). Considering all the CLT 370 371 configurations, only one (CMVP) had 100 % specimens passing 1.25 N/mm². However, CMHM, CMHP, and 372 COHM configurations had all but one specimen above the limit of 1.25 N/mm², which is very encouraging considering that these are residual shear strengths. The EN 14080:2013 [36] requires for glulam a shear strength 373 374 of at least 6 N/mm², or between 4 N/mm² and 6 N/mm² if wood failure percentage is 100 %. Among all glulam 375 specimens, 43.83 % had residual shear strength above the 6 N/mm² limit required by EN 14080:2013 [36] for the 376 dry shear test. Among all glulam configurations, GOVP has the highest percentage of specimens with residual

377 shear strength higher than 6 N/mm^2 (77.78 %).

378





380

Fig. 5. Boxplot residual shear strength after delamination cycle

The mean shear strength for CLT made from oak specimens was 3.69 N/mm² and for mixed poplar-oak CLT it 381 382 was 3.56 N/mm². Thus, the residual shear strength of oak and mixed poplar-oak CLT was about equal. Indeed, the 383 trade-off between delamination and residual shear strength is visible here. The higher delamination of the oak 384 specimens results in low mean residual shear strength. However, since oak has superior mechanical properties to poplar, the average residual shear strength of these heavily delaminated oak specimens was fairly high. It should 385 386 be remembered that some oak specimens were 100% delaminated, so the corresponding mean residual shear 387 strength can be computed with zero values. As a result, while a CLT specimen entirely made from oak is not 100% 388 split, it may still resist to the same shear stresses as a mixed poplar-oak CLT specimen exhibiting much lower 389 delamination.

390 The block shear test has been developed to test the laminations in shear parallel to the grain. Consequently, when 391 applied to CLT, it very often results in rolling shear failure which depends more on the wood mechanical properties 392 rather than the bonding strength [16]. Regarding the residual shear strength tested in our study, rolling shear failure rarely happened. The oak specimens have generally shown shear failure in the glue line. For mixed poplar-oak 393 394 CLT, the specimens typically showed evidence of compression failure in the poplar lamella (before rolling shear 395 final failure), especially for specimens with practically no delamination. Fig. 6 shows the larger displacements obtained for mixed poplar-oak CLT compared to oak CLT which is due to this compression failure. For specimens 396 397 with severe delamination, shear failure in the glue line was the primary type of failure. For CLT, the best residual 398 strength was achieved for specimens entirely made from oak bonded using MUF at 0.8 N/mm² pressude. This is 399 in accordance with the rather low delamination obtained for this configuration. The higher residual shear strength 400 than mixed poplar-oak CLT despite the lower delamination of these latter samples may be explained by the very 401 high rolling shear strength of oak and the observed compression failure in poplar lamella.



403 Fig. 6. A typical plot of force and displacements in the shear test for oak and mixed poplar-oak CLT specimens.

404

405 Correlations

The correlations among the percentage of total delamination, WFP, and residual shear strength of glulam are presented in Table 7 while for CLT, it is presented in table 8. For both oak and mixed poplar-oak glulam configurations, it can be seen that the WFP and total delamination were correlated with the residual shear strength. Indeed, the residual shear strength increased with the decrease of delamination and increase of WFP. For both oak and mixed poplar-oak configurations, residual shear strength seems to correlate better with WFP than with

411 delamination length.

Table 7. Coefficient of correlation between WFP, delamination, and shear strength for glulam specimens made
 from oak (upper triangle) and mixed poplar-oak (lower triangle)

	WFP	D _{tot}	Residual shear strength
WFP	1	0.74***	0.82***
D _{tot}	-0.86***	1	-0.64***
Residual shear strength	0.6***	-0.59***	1

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

415 A positive correlation between WFP and shear strength and a negative correlation between delamination and shear 416 strength was also found for CLT configurations. It may be noticed that the coefficients of correlation between 417 delamination length or WFP and residual shear strength were generally higher than for glulam.

417 detainmation length of w17 and lesidual shear strength were generally ingher than for grutam.

418 Since all these possible criteria are rather well correlated, they may overlap and thus may seem unnecessary.
419 However, the correlation is not perfect, and thus using both delamination length and WFP as criteria as in the EN

419 However, the contention is not perfect, and thus using both defamination length and wFF as criteria as in the EN 420 16351:2015 [22] standard allows avoiding to fail too easily to the delamination test. Post delamination shear test

421 may be added to these criteria for the same reason.

Table 8. Coefficient of correlation between WFP, delamination, and shear strength for CLT specimens made from
 oak (upper triangle) and mixed oak-poplar (lower triangle)

Pearson	WFP	D _{tot}	Residual shear strength
WFP	1	-0.75***	0.51***
D _{tot}	-0.82***	1	-0.81***
Residual shear strength	0.78***	-0.83***	1

424 *Significant at 5% level; **significant at 1% level; ***significant at 0.1% level; '' not significant.

425 4 Conclusion

- 426 Bonding pressure and type of glue parameters appeared to have a significant influence on the bonding quality of
- 427 CLT or glulam, with also significant interactions with the kind of species bonded. Using a poplar middle lamella
- 428 in a 3-plies poplar-oak CLT or glulam induced less delamination than with specimens entirely made from oak
- samples. Only mixed poplar-oak CLT and glulam bonded with a hydraulic press with 0.8 N/mm² bonding pressure
- showed 100% passing rates for all the samples according to the EN 16351:2015 and EN 14080:2013 requirements.
 If a high bonding pressure seemed to provide better results, vacuum-pressed (0.085 N/mm² bonding pressure)
- 432 mixed poplar-oak CLT or glulam bonded with PUR were very close to reaching the requirements of the standards,
- 433 only a few samples exhibiting too much delamination. As a result, it seems possible to use a vacuum press for
- 434 glulam or CLT bonding when a poplar middle layer is used with oak outer layers. It would be interesting to confirm
- 435 all these observations with 5-plies CLT or glulam with more plies. Moreover, other tests with more specimens and
- 436 variability would be needed to quantify more precisely the observed difference between configurations.
- 437 For CLT or glulam entirely made from oak, using MUF with 0.8 N/mm² bonding pressure provided the lowest 438 delamination and the highest residual shear strength. The shear strength was superior to what is obtained with 439 mixed poplar-oak specimens manufactured in the same conditions. Mixed poplar-oak CLT exhibited compression 440 failure in the poplar middle lamella, whereas CLT entirely made from oak exhibited shear failure in the glue line. 441 This highlights the very high transverse strength of oak, hence its interest as a middle layer when gluing is 442 performed efficiently. In particular, the CLT entirely made from oak glued with MUF at 0.8 N/mm² bonding 443 pressure exhibited significantly higher residual shear strength than other CLT configurations, but it did not pass 444 the delamination and WFP criteria of EN 16351:2015 on the contrary to mixed poplar-oak CLT. As a result, it 445 may be considered that either these criteria may be changed or the post-delamination shear test may be considered 446 as an additional test to qualify CLT resistance to delamination, especially for hardwood species that are very
- 447 resistant to rolling shear.
- Delamination length, WFP, and residual shear strength were rather well correlated for both CLT and glulam.
 However, allowing to use the WFP criterion if delamination length criterion is not fulfilled, then using residual
 shear strength criterion if WFP criterion is not fulfilled looks like an interesting solution to avoid being too
- 451 restrictive in the assessment of bonding strength.
- As a result of this research, mixing poplar and oak species in CLT or glulam manufacturing appears to provide good bonding durability, with potentially other benefits like lighter structure and better thermal insulation. Further research is needed to understand the mechanical properties of these hybrid products. In addition, this study only presents the experimental results of bond durability with phenomenological explanations. Future studies would require a strain field analysis and/or finite element modeling to understand what happens in the joint, and especially what makes hybrid CLT or glulam perform better than CLT or glulam entirely made from oak.

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