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BONDING OF FIRE-RETARDANT TREATED SPRUCE LAMELLAE FOR USE IN CROSS LAMINATED TIMBER (CLT)

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ABSTRACT: This paper presents results from bond line testing to investigate the influence of fire-retardant (FR) treatment on the bondability of spruce, tested in glued laminated timber (GLT) specimens as requested by standards for testing of the actual adhesive systems, and specimens of cross-laminated timber (CLT) produced under laboratory conditions to investigate a product bonded with the adhesives. One MUF- and two PUR adhesives were chosen for the investigations, selected on the basis of their use in CLT-production today as this product was the main focus. They were tested in five different material combinations: lamellae with high uptake of fire-retardant against lamellae with high uptake of fire-retardant, high uptake against untreated lamellae, low uptake against low uptake, low uptake against untreated lamellae and untreated against untreated lamellae.
All 22 GLT specimens fulfilled the requirements for resistance to delamination according to EN 302-2:2017/EN 301:2017. The requirements for compressive shear strength according to EN 14080:2013 were fulfilled by all 24 specimens cut from the GLT samples.
73 of 75 CLT specimens fulfilled the requirements for resistance to delamination according to EN 16351:2021.
The results do not show any influence of the FR treatment on the bondability of the FR treated spruce lamellae. Potential explanations for this might be that a) the adhesives are fully compatible with the FR, or b) the FR-treated wood was removed by the slight planing of the lamellae prior to bonding, leaving untreated but insufficiently planed surfaces for bonding.

KEYWORDS: Fire-retardant, fire-retardant impregnated spruce, B-s1,d0, CLT, shear strength, delamination

1 INTRODUCTION

The objective of this work is to look into the possibility of using commercially available fire retardant (FR) treated wood in the outer layers of Cross Laminated Timber (CLT) panels for interior use in order to achieve reaction-to-fire class B-s1,d0.

CLT is often used in walls, often covered in gypsum boards to achieve the necessary reaction-to-fire classification. Alternatively, the wooden surface can be treated with a FR after installing the element in the building to maintain the wooden surface. But this is found to be time consuming, and the possibility of receiving the element with the right properties from the factory would be more efficient.
CLT produced in Norway consists exclusively of spruce wood, the outer lamellae usually fulfil the requirements of strength class C24 [1]. Therefore, this raw material has been used in this study.

1.1 APPROACH

Several types of fire retardants and their application processes were screened to select the most suitable one for application in CLT-production. The aim is to make the treated layer a part of the panel and avoid adding extra thickness, maintaining the same properties of the panel except for the improved fire properties.
The different types of fire retardants – paint/varnish and impregnation – and some of the challenges by using each type in different processes, were identified and discussed, focusing on those adding the treatment before leaving the production facility.
The different processes were mainly divided into "before-" and "after pressing" of the panel. Discussions about the various challenges led to one process chosen for further investigations – to add FR impregnated spruce lamellae to the production line of CLT before pressing of the panels. A such panel would consist of untreated wood in the middle layers and only FR impregnation in the surface layer(s), as reaction-to-fire is mainly affected by the surface properties of the panel.

A potential reduction of mechanical properties of the FR treated lamellae and issues linked to the hygroscopicity of the FR were ignored in this initial study, as well as the challenge about maintaining the fire properties after transportation and construction, where possible damage to the surface can be expected to have an impact on the reaction-to-fire properties.

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Two questions were raised to the chosen solution: 1) Is it possible to FR impregnate C24 spruce and obtain a B-s1,d0 classification? 2) Is it possible to bond FR impregnated spruce and ensure adequate attachment of treated lamellae? These two questions were investigated, and the results are presented in this paper.

1.2 STATE OF THE ART

1.2.1 Fire-retardant impregnation of spruce

Spruce is difficult to impregnate due to the blockage of the fluid pathways in the wood due to aspiration (irreversible closure) of up to 75 % of the bordered latewood pits during drying of the wood [3], relatively small ray size and high percentage of heartwood which is impermeable to impregnation [4]. Biotechnological attempts to increase the permeability of spruce have been developed on laboratory scale [5, 6], but are not yet commercialised.

As a consequence, a pressure impregnation will only penetrate the outer millimetres of spruce, an area prone to be removed during machining. Therefore, the surface of the treated lamellae cannot be machined [7], if necessary, only lightly sanded to maintain the improved reaction-to-fire classification.

Screening the market for FR treated wood showed that spruce lamellae treated with 5.7% (arto/arto, weight percent gain calculated based on dry matter) FirePRO® and coated fulfilled the required classification [2]. The purpose for coating of the wood is to protect the surface of the wood from high relative humidity which is known to cause migration of the hygroscopic components of the FR which often results in discoloration and loss of FR over time. This aspect was neglected in this study because the interior surface of the CLT-panels is usually not exposed to relative humidity exceeding 60% (Service class 1). Following recommendations by the treater, desired uptake in this case is set to 6.5%.
With this, the first of the two questions was answered.

1.2.2 Bonding of fire-retardant treated wood

Bonding of fire-retardant treated wood, mostly veneers for plywood, has been done on at commercial scale since 1954 [8]. [9] investigated the influence of monoammonium phosphate, diammonium phosphate, ammonium sulphate, ammonium sulfamate, borax and boric acid on adhesives urea-formaldehyde (UF) and phenol-formaldehyde (PF) adhesives used in plywood production. Impregnation of veneers with borax and boric acid gave severe embrittlement rendering the use of a mechanic glue spreader not feasible. Black reports that it was these two treatments which caused most of the problems in achieving good bonds. He ascribes the difficulties to the low pH of borax and the high pH of boric acid. However, all fire-retardant treatments reduced the water resistance of the adhesives. [9] states that it is difficult to qualify the influence of the fire-retardant treatments on the performance of PF due to the variability of their chemical composition and complexity of their curing reaction.
[8] summarizes results from trials including veneers impregnated with monoammonium phosphate, diammonium phosphate, ammonium sulphate, borax, boric acid, zinc chloride and chromated zinc chlorate bonded with various glues and adhesives. The author recommends phenol, resorcinol and combinations of these two resin types for all treatments. Melamine, fortified urea and urea adhesives are recommended for a treatment combining boric acid and borax. [10] conducted bonding trials with solid wood from Douglas fir and Western Hemlock impregnated with a mixture of ammonium sulfate, zinc chloride, boric acid and sodium dichromate. The surfaces were slightly planed before bonding of the members with a resorcinol resin (PRF) and a paraformaldehyde hardener (type A) and a resorcinol adhesive and a hardener with 54% formaldehyde content (type B). The shear strength of the bond lines of all samples made from treated wood were lower than the shear strength of the samples made from untreated wood. Schaeffer identified chemical interaction between the fire-retardant salts and the adhesives as reasons for the reduced bond strength: Ammonium sulphate, zinc chloride and sodium dichromate showed increased rate of gelation, boric acid was found to slightly retard the rate of gelation of a resorcinol-resin adhesive. However, also changes in the wood surface affect the adhesion properties of wood. [11] investigated the influence of natural weathering on the tensile shear strength of Desmodur-vinyl trie ketonol acetate bond lines between pieces of Scots pine and Oriental beech that had been dip-treated with borax and boric acid. Without natural weathering, the reduction of tensile shear strength was higher for boric acid compared to borax for both wood species. After four years of outdoor exposure, the Oriental beech samples treated with fire retardant showed higher tensile shear strength than the untreated controls, the values for the borax-treated samples were higher than those for the samples treated with boric acid. In case of the Scots pine samples, the borax treated samples showed lower tensile shear strength compared to the controls and the samples treated with boric acid.
[12] investigated the influence of diammonium phosphate and sodium silicate on the adhesion properties of impregnated birch veneer. The former increased the glue bond strength measure by ABES, the latter showed unchanged or slightly reduced glue bond strength for 15% and 30% concentration in the impregnation solution. [13] consider using lamellae impregnated with a fire-retardant for CLT-production problematic because of the reduced bondability of fire-retardant treated wood and issues linked to the disposal of planer shavings from fire-retardant treated wood. Investigations of potential alternatives to planing, hot pressure treatment, chemical treatment with a confidential substance and plasma treatment showed that planing provided bond lines with highest shear strength and wood failure percentage. The adhesive systems used were PRF and polyurethane (PUR).

2 MATERIAL AND METHODS

2.1 PRODUCTION OF TEST SAMPLES

Lamellae and fire-retardant treatment

Lamellae of spruce (*Picea abies* L.) graded C24 were fire-retardant treated with FirePRO®, a fire-retardant containing Borax and boric acid, in a commercial treatment process.

The uptake of FR for each lamella was compared to the desired uptake to ensure reaction to fire-class B-s1,d0 according to product certificate of constancy of performance [2] (Figure 1, Figure 2). The lamellae with a deviation of -10% to +35% from the desired uptake were sorted out for other tests. The rest of the lamellae were divided into two groups; High uptake (H) (40–62.5 kg dry fire retardant/m^3 wood) and low uptake (L) (17.5–26.8 kg dry fire retardant/m^3 wood) and were used for preparation of the samples for bondability testing. Machining of the impregnated surfaces was kept to a minimum to remove as little fire retardant as possible from the face of the lamellae intended for bonding, but still provide favourable surfaces for bonding.

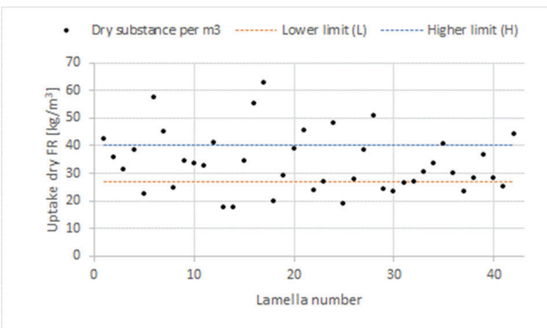

Figure 1: Distribution of impregnation liquid uptake in the lamellae (lamella no. 1-43).

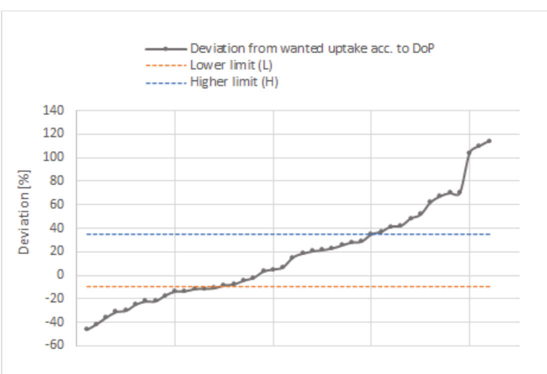

***Figure 2:** Distribution of dry substance uptake in the impregnated lamellae in relation to the desired uptake. 0% deviation marks the desired uptake, corresponding to 6,5% dry substance of the total weight after impregnation.*

Samples for testing of bondability

Glued laminated timber (GLT) samples were produced as described in EN 14080 [15] with the following combinations of treatments: L-U, H-U, L-L (L) and H-H (H) (illustration in Figure 3). With regard to CLT-testing, samples with the following combinations of treatments were assembled: L-U and H-U (illustration in Figure 4). For both GLT and CLT, reference samples of untreated lamellae (U) were assembled. Three different state-of-the-art adhesive systems for use in CLT were used: a 2-component MUF-system and two different 1-component PUR-systems.

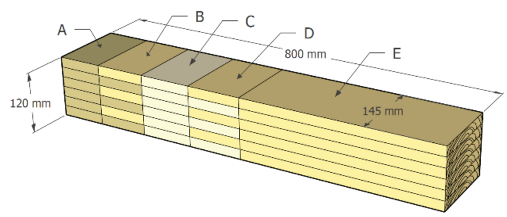

***Figure 3.** Illustration of the beams produced for adhesive testing. Each combination is partly shown, A (H) – B (HU) – C (L) – D (LU) – E (U).*

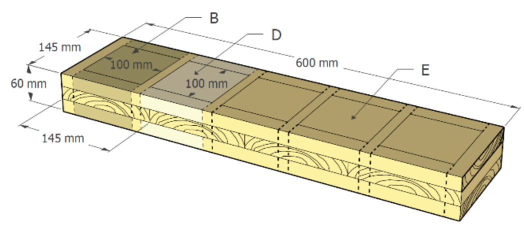

***Figure 4.** Illustration of the beams produced for product testing of small CLT specimens. Two beams were produced per combination B (HU), D (LU) and E (U) with the different adhesive systems. The marked areas illustrate the five specimens that are cut from each beam for testing of resistance to delamination, measuring 100 x 100 x 60 mm^3.*

2.2 TESTING BONDABILITY

The investigations of the material's bondability was divided into adhesive- and product testing. Adhesive testing covers testing of resistance to delamination of glulam samples according to EN 302-2 [14] and testing of compressive shear strength according to EN 14080 Annex D [15] on GLT as requested for testing of the actual adhesive systems.
Delamination was tested on two specimens per combination of treatment and adhesive with five bond lines per specimen (Figure 3). Compressive shear strength (CSS) was tested on two specimens per combination with five bond lines per specimen (Figure 3).

As the focus of the study is on CLT, product testing on CLT samples was conducted according to EN 16351 Annex A [16]. The standard requires maximum 40%

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opening per individual bond line and an average of 10% opening of the sum of all bond lines. If a specimen exceeds these requirements, the bond areas are to be opened to quantify the wood failure percentage which may not be less than 50% for the individual bond area and not less than 70% for the total bond area per specimen.
In this study we evaluate the conformity of the bondings with the requirements defined in EN 16351. Additionally, we use the average opening of the bond lines in each specimen for further analyses.
The test was performed on five specimens per CLT-element, each specimen contained two bond lines (Figure 4).

3 RESULTS AND DISCUSSION

3.1 RESISTANCE TO DELAMINATION

The requirements for resistance to delamination of GLT according to EN 301 [17] were fulfilled by 22 of 24 specimens (Table 1). The evenly one-sided distribution of the delaminated areas on the samples produced from GLT.9 (6.2 and 6.4% delamination) (Figure 5) indicates deviation in thickness as reason for the failure of the specimens rather than the fire-retardant treatment. The results are therefore rejected.

***Table 1**: Delamination in GLT-specimens according to EN 302-2 in % of total bond line length, requirement: ≤ 5 % (Spec.= specimen).*

Sample	Adhesive and treatment	Delamination [%]	
		Spec. 1	Spec.2
GLT.1	1/MUF H	0.0	1.1
GLT.2	1/MUF HU	0.8	1.8
GLT.3	1/MUF L	0.6	1.4
GLT.4	1/MUF LU	0.0	0.4
GLT.R1	1/MUF U	3.8	0.7
GLT.5	2/PUR HU	1.7	3.3
GLT.R2	2/PUR U	0.0	0.4
GLT.6	3/PUR H	2.0	1.4
GLT.7	3/PUR HU	2.6	1.0
GLT.8	3/PUR L	4.8	2.2
GLT.9	3/PUR LU	N*	N*
GLT.R3	3/PUR U	0.7	1.9

N* Results rejected due to planing error

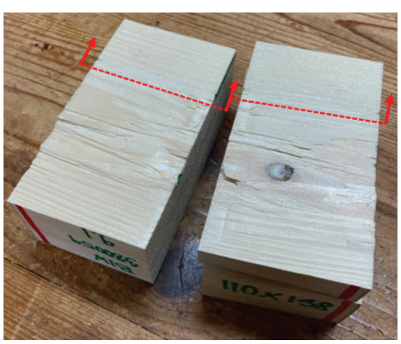

***Figure 5:** Evenly one-sided delamination on specimen GLT.9.1*

The average delamination of the visible bond line length per CLT element is compiled in Table 2. The specimens that did not fulfil the requirement were opened for further investigation of wood failure percentage in the glue line surface (results are found in Table 3).
Five specimens (Table 3) were rejected due to knots or resin pockets in the bond area and are therefore not included in all further analyses. Besides from these, two specimens did not fulfil the requirements given in EN 16351 (CLT3.1 and CLT10.1). The delamination of all remaining specimens was below the requirement of max. 30% of the bond area.

The delamination of the CLT-specimen was significantly higher than the delamination of the GLT-specimen (Figure 6). This is due to the higher stress on the bond line between cross-laminated lamellae in CLT compared to parallel laminated lamellae in GLT.

***Table 2.** Average ($\overline{x}$) and standard deviation (σ_x) of delamination according to EN 16351 Annex A in % of total bond line length, requirement: ≤ 10 %.*

Sample	Adhesive and treatment	Delamination [%]		Opened*
		$\overline{x}$	σ_x	
CLT1	1/MUF HU	7.0	3.4	1/5
CLT2		4.7	4.2	0/5
CLT3	1/MUF LU	21.8	6.1	5/5
CLT4		26.3	19.7	4/5
CLT.R1	1/MUF U	10.0	6.6	3/5
CLT.R2		9.6	6.6	4/5
CLT5	2/PUR HU	10.9	9.2	2/5
CLT6		5.2	2.4	0/5
CLT.R3	2/PUR U	9.9	8.2	3/5
CLT.R4		6.4	3.1	3/5
CLT7	3/PUR HU	12.0	7.7	3/5
CLT8		9.1	6.6	2/5
CLT9	3/PUR LU	13.0	3.4	4/5
CLT10		9.8	6.0	4/5
CLT.R5	3/PUR U	5.7	4.7	1/5
CLT.R.6		1.3	2.0	0/5

*Number of specimens that did not fulfil the requirement of total bond line length opening and therefore were opened for further investigation of wood failure percentage in bond area (Table 3).

***Table 3.** Average ($\overline{x}$) wood failure in the bond area of opened specimens in %, according to EN 16351 Annex A. Requirement: ≥ 70%.*

Sample	Adhesive and treatment	$\overline{x}$ wood failure of opened specimens [%]
		Specimen 1 – 2 – 3 – 4 – 5
CLT1	1/MUF HU	X – X – 98 – X – X
CLT2		X – X – X – X – X
CLT3	1/MUF LU	65^{N} – 70 – 83 – 98 – 78
CLT4		50^{N*} – 83 – 80 – X – 80
CLT.R1	1/MUF U	X – X – 88 – 85 – 88

CLT.R2		83 – 88 – 78 – X – 90
CLT5	2/PUR HU	80 – X – 55^{N*} – X – X
CLT6		X – X – X – X – X
CLT.R3	2/PUR U	88 – 88 – 70 – X – X
CLT.R4		X – 60^{N*} – 80 – X – 83
CLT7	3/PUR HU	X – 83 – 70 – X – 80
CLT8		X – X – 88 – 80* – X
CLT9	3/PUR LU	70* – 85* – X – 97 – 68^{N*}
CLT10		40^{N} – 80 – 90 – X – 75^{N*}
CLT.R5	3/PUR U	X – X – X – 100 – X
CLT.R6		X – X – X – X – X

*One opened bond line only.
^{N} Delamination does not fulfil the requirement.
^{N*} The specimens had knots or resin pockets in the bond line area and are therefore excluded from further analyses.

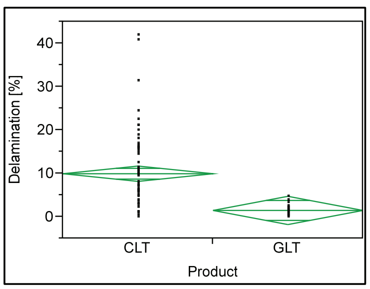

Figure 6: *Delamination in bond line of CLT and GLT specimens investigated according to EN 16351 and EN 302-2, respectively.*

3.2 COMPRESSIVE SHEAR STRENGTH

The compressive shear strength (CSS) of all 24 specimens fulfilled the requirements for shear strength according to EN 14080:2013 Annex D, the average results are shown in Table 4. The specimens showed wood failure only. Results from specimens with knots in the shear area are excluded from further analyses.

Table 4. *Average ($\bar{x}$) and standard deviation (σ_x) of compressive shear strength (CSS) according to EN 14080 Annex D. The requirement was ≥ 4.0 % for individual and ≥ 4.9 % for average values.*

Sample	Adhesive and treatment	CSS [N/mm^2] $\bar{x}$	σ_x
GLT.1	1/MUF H	8.3	0.9
GLT.2	1/MUF HU	8.1	1.4
GLT.3	1/MUF L	9.8	0.9
GLT.4	1/MUF LU	9.3	1.0
GLT.R1	1/MUF U	9.5	0.9
GLT.5	2/PUR HU	7.5	0.7
GLT.R2	2/PUR U	9.0	0.9
GLT.6	3/PUR H	9.2	1.0
GLT.7	3/PUR HU	8.9	0.9
GLT.8	3/PUR L	8.7	1.2
GLT.9	3/PUR LU	8.3	0.8
GLT.R3	3/PUR U	8.3	0.7

3.3 INFLUENCE OF FIRE-RETARDANT TREATMENT ON OVERALL BONDABILITY

The delamination results of both GLT and CLT samples do not indicate any influence of the treatment intensity on the delamination of the respective specimen (Figure 7), only the delamination of treatment LU was significantly different from the remaining treatments. This difference can be ascribed to the CLT specimens 4.2 (40.9% delamination of bond line) and 4.3 (42.0 % delamination of bond line). Both specimens, however, fulfil the requirement of ≥70% wood failure in bond area according to EN 16351 (Table 3).
The latter are decisive for the significant difference between the delamination results for CLT-specimen from treatment LU and UU (Figure 8). The delamination results for the GLT-specimen from the different treatments do not differ significantly (Figure 9).

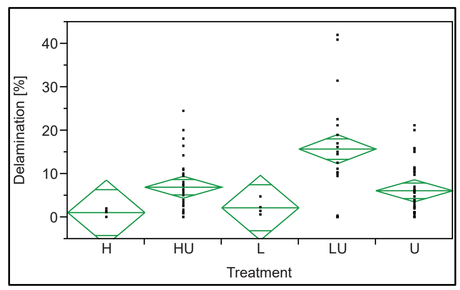

Figure 7: *Influence of treatment on the bond line delamination [%] of GLT and CLT samples.*

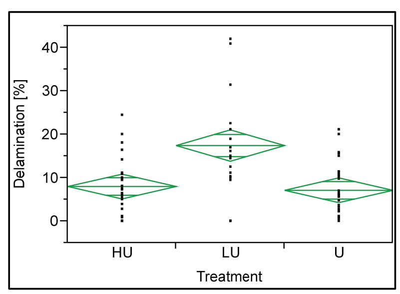

Figure 8: *Delamination of bond line of CLT samples based on the treatment.*

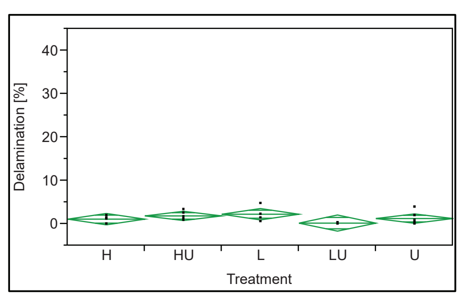

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Figure 9: Delamination of GLT samples based on the treatments.

The compressive shear strength (CSS) of the GLT samples (Figure 10) did not differ significantly between the treatments. Thus, an influence of the treatment on the bondability was not found here either.

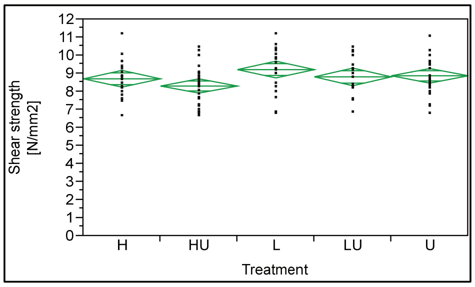

Figure 10: *Compressive shear strength of the bond lines in the GLT samples based on their treatments.*

3.4 INFLUENCE OF ADHESIVE

No significant differences between adhesives were found for the

- overall delamination (Figure 11),
- overall delamination as function of the treatment,
- delamination of CLT and GLT and
- delamination as function of the treatment within the specimens from CLT and GLT.

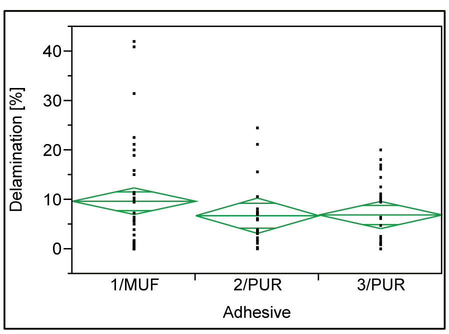

Figure 11*: Delamination in bond lines found in GLT and CLT samples as function of the adhesive.*

The compressive shear strength (CSS) of the bond lines of 1/MUF was significantly higher than the CCS of bond lines with 2/PUR (Figure 12), the same was observed for all treatments.
This is surprising as the delamination in the bond lines bonded with the different adhesives did not differ significantly. Additionally, bonding with 3/PUR yielded higher CSS than bonding with 2/PUR. Thus, the authors assume a combination of locally uneven uptake of FR or uneven plaining with resulting local deviations in bond quality as explanation because incompatibility between PUR-adhesives and the FR can be excluded.

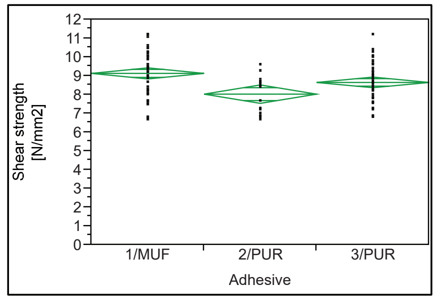

Figure 12*: Compressive shear strength as function of the adhesive*

4 CONCLUSIONS

The study did not show any influence of the fire-retardant treatment on the bondability of the spruce lamellae. This might be due to two reasons: Either the adhesives applied are fully compatible with the fire-retardant, or the fire-retardant has been removed from surfaces to be bonded during sample preparation.
The manufacturer of the impregnated material used in this study, as well as literature, advises against machining of fire-retardant treated wood as this will remove some of the impregnated surface [18, 19]. This is valid for all fire-retardant treated wood, but especially for refractory wood species such as spruce. Before this background, planing was limited to an absolute minimum, leading to wood surfaces of lower quality than usually accepted for bonding activities.
The combination of these two factors leads to the conclusion that the results from the current study do not represent the full bonding potential of the investigated adhesives and the fire-retardant treated wood.
Further investigations should be conducted where the planed surfaces are carefully re-treated with FR prior to bonding.

5 ACKNOWLEDGEMENTS

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