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GLUED-IN HARDWOOD RODS USING BIO-SOURCED ADHESIVES — PART I: INVESTIGATIONS UNDER LABORATORY CONDITIONS

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ABSTRACT: Glued-in Rods (GiR) have proven to be a particularly good method for connecting timber structures, in which steel or FRP steel rods are yet commonly bonded with two-component epoxy resin or polyurethane adhesive systems. However, from a sustainability point of view, this poses a problem. This paper summarises preliminary results of a research project investigating to which extent the currently used glued-in steel rods in softwood can be substituted by rods made of hardwood for load-bearing functions. The project also reports on the performance of bio-sources adhesives to substitute the chemical adhesives currently used. The results presented herein focus on the performance of corresponding small scale GiR under laboratory conditions; the companion paper, also presented at the WCTE2023, will extend these findings towards more critical environmental conditions.

KEYWORDS: glued-in-rods, structural, joints, adhesion, bio-sourced

1 INTRODUCTION

1.1 SETTING THE FRAMEWORK

Glued-in-rods (abbreviated as GiR in the following) form a subset of bonded connections, in which load is transferred from one timber block to another via one (or several) bonded rods [1–3]. Design parameters involved for GiR are the choice of materials (timber, adhesive, and rods) and their sizing (cross-section of the timber members, adhesive layer thickness, and embedment length). Because of the complexity of this joint type, failure can take multiple forms. Figure 1 summarises them graphically, for more details refer to [1–3]. Each of these is associated with specific material and geometry parameters of the GiR, which cannot be discussed at length in this paper [4–6].

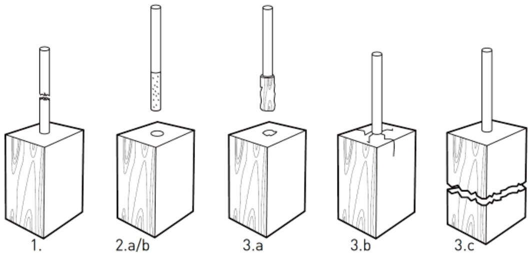

Figure 1: *Failure modes of glued-in-rod connections*

For most current applications, the rods are either threaded rods [7] or rebars [8], seldom fibre reinforced polymers [9]. Regarding the adhesive, almost all experimental investigations consider two-component epoxies (2K-EPX) or 2K-polyurethanes (2K-PUR) [2], both being of petrochemical origin. While timber engineering is widely acclaimed for its sustainability [10], the use of metallic and polymeric material for the joints may be questioned.

The research summarised in this paper addresses the question to which extent the concept of glued-in rods can be pushed towards sustainability by substituting the metallic rods by hardwood ones, and the "chemical" adhesives by bio-sources alternatives.

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Surprisingly little research has been documented on gluing-in hardwood rods to connect structures in timber engineering. Among those, Koizumi and Jensen [11–13] presented experimental results on dowels (in fact rods) made of Maple (*Acer saccharum*) glued into Cedar (*Cupressus japonica*). The results showed that average shear strengths of beyond 8 MPa were achieved with a 2K-PUR, and beyond 10 MPa using a 2K-EPX.

Adhesives based on renewable resources have been used since the dawn of humanity in conjunction with timber. Both their availability, and performance, proved sufficient to fulfil all demands up to World War II, where they were still used to manufacture aeroplanes [14]. Nowadays, adhesives based on natural raw materials are classified into glutine (in the following referred to as animal glue), casein, lignin, tannin, and dextrin [15,16]. They achieve, on spruce shear strengths up to 9.6 MPa, and for beech strength up to 11 MPa

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1.2 OBJECTIVES

The research summarised in this publication aimed to three different objectives: (1) Investigate the potential hardwood dowels (or bars) offer as an ecologically sustainable alternative to steel, and FRP, rods; and (2) extend the sustainability of GiR through the substitution of "chemical adhesives" by "natural" ones.

2 MATERIALS AND METHODS

2.1 Adhesives

The adhesives used in this study were all derivatives of "Technical Gelatine 400/115" from the Company Fritz Häcker GmbH in Germany. The basic adhesive, is a gelatine currently commercially available. A feature relevant for this study is the relatively high water-content during processing of the adhesives.

The technical gelatine is delivered in a dried state in form of granulate. As a first step it is swollen in water. Therefore 25 parts of technical gelatine is mixed with 75 parts of water. The gelatine must be soaked in water for at least two hours before it can be further processed. To improve laboratory workflow, the mixtures are usually soaked overnight. In the next step the adhesive was molten in a water bath at 50 °C. In this state, the adhesive is now ready for processing.

Modifications of the basic formulation involve aspects such as reduction of the water-content, the addition of solid particles (as chalk, saw dust), and further reactants (as tannin etc.) A summary of the modifications, all labelled M*XX*, is shown in Table 1and Table 2

Table 1: Summary of adhesive modifications to improve the formation of larger adhesive layer thickness (% are related to the mass)

REF	Technical Gelatine 400/115: water-content 25:75
M29	REF with water-content reduced to 25:60
M30	REF with water-content reduced to 25:45
M31	REF + 30% chalk
M32	REF + 50% chalk
M33	REF + 30% sawdust

The modifications of the adhesive were performed following two objectives:

Firstly, the one discussed in this part, aimed at enabling the adhesive to form the relatively large layers (0.2 to 0.5mm) required for the glued-in rods, as opposed to the almost nil thickness needed in lap shear samples. The issue herein is related to the large water content of the gelatine-based adhesives.

Secondly, gluing with technical gelatine means that the processed adhesive contains 75 % of water. For the usual applications for this adhesive, like gluing books, folding boxes or cardboard this is not an issue. Animal glues are adhesives which have been used for centuries to bond wood. However, because of their high water-content, they have problems to fill large adhesive layer gaps above 200 µm. To overcome this limitation the idea was to increase the solid content. One way was to work with less water. The other way was to incorporate fillers. Therefor we chose two different fillers: Chalk and sawdust. Chalk is used very often in adhesives. We worked with the product Omyacarb HSB from Omya.GMBH / Germany. And another interesting filler for us was sawdust, due to the application of gluing wood. Working with a filler, is changing a lot of the adhesive characteristics like viscosity, stiffness or the thermal expansion coefficient. By choosing sawdust, we saw a high chance to improve several characteristics of the adhesive and additionally get a bio-based filler so to fulfil the requirement of sustainability. We used the product HAHO 120/F from JELU-WERK J. Ehrler GmbH & Co. KG / Germany for the sawdust. Secondly, improving—if necessary—the mechanical performance of the adhesives under elevated moisture contents, an aspect further developed in the second part of this publication.

Table 2: Summary of adhesive modifications to improve the performance under elevated moisture environment

M7	1 % tannic acid
M10	3 % kalinite
M14	0,3 % gallic acid
M26	1 % tannic acid + 9 % linseed oil
M10C	M10 + 30% chalk
M14C	M14 + 30% chalk

2.2 Investigations on lap shear samples

The preliminary investigations focused on a comparison of the mechanical performance of the adhesive used in lap shear samples (acc. to DIN EN 1465).

The substrates consisted in beech wood (*Fagus sylvatica*) in the dimensions 19.5 × 5.2 × 80.5 mm³, which were conditioned to 23 °C and 50% relative humidity (rH) for at least 7 days prior to bonding.

During the bonding, a pressure of 0.7±0.1 MPa was exerted on the overlap, the resulting adhesive layer thickness was almost nil.

For the results presented in this part, bonded lap shear samples were conditioned for 7d @ 23 °C and 50% rH prior to testing.

Testing occurred in an UTM under displacement control, with 5mm/min, until failure occurred. Ten samples were tested in each series.

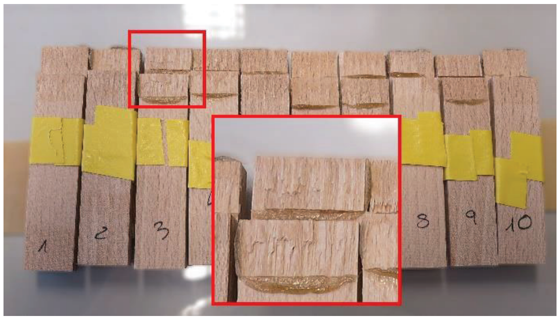

Figure 2: A set of ten lap shear samples after testing (the enlargement shows a typical substrate failure)

The results subsequently presented herein are given as average lap shear strength, which corresponds to the failure load divided by the bonded area surface, which was measure using a calibrated calliper for each individual sample.

Additionally, the fracture surface has been characterised so to distinguish between substrate failure (SF) and adhesive failure (AF).

2.3 Investigations on glued-in rods

The mechanical performance of bio-sourced adhesives was compared to that of widely used "chemical" ones. Tests were performed on the following wood species. Spruce, which constitutes the base material of all GiR, and beech for the rods. Two increasing levels of complexity were considered. Firstly, lap shear samples acc. to DIN EN 302; secondly, small scale GiR consisting of 45x45x120 mm³ spruce blocks in which ø6mm hardwood rods were bonded

The spruce blocks are drilled to precisely 6.4 mm with an automatic drilling machine, to achieve an accurate adhesive layer thickness between the two substrates. This results in a radial distance of 0.2 mm.

To ensure axiality, the glued in rods are centred during the gluing process with thin spacers around the hole top. The wooden rods are deburred and thoroughly cleaned with isopropyl alcohol.

Preparing the lap shear samples with the ecological adhesive is identical to the process of the lap shear samples, however the application process is different. After mixing of the adhesive, the viscous fluid is transferred to a syringe and subsequently used to fill the lateral hole in the spruce block with adhesive. The outside of the hardwood dowel is coated in a film of the same adhesive. The rod is then pressed into the hole with a spring-loaded guide mechanism until it bottoms out, for further details regarding the bonding process cf. [17].

These bonded samples are then cured and conditioned under the same environmental conditions as the lap shear samples (7d @ 23 °C and 50% rH).

Figure 3: Process of injecting the adhesive into the hole, coating the rod and combining them

2.3.1 Test setup

Testing the adhesive performance is conducted by rigidly clamping each side of the sample in the UTM, under displacement control and 2mm/min, until failure.

Clamping the hardwood rod directly did not produce adequate findings, due to deformation of the wood fibres and therefore induced stress fractures and reduced tensile strength of the dowel. This resulted in the premature failure and breakage of the rod during testing.

To limit the compression of the wood across the grain, multiple options were followed.

Bonding an aluminium sleeve with a chemical adhesive to protect the wood from crushing, did not achieve satisfactory results due to poor adhesion between the metal and wooden substrate.

Gluing a block of beech wood to the exposed part of the dowel still resulted in both brittle fractures as well as the wood splitting along the grain.

Figure 4: (Top) Aluminium sleeve bonding failure, (bottom) rod failure with beech block

The hardwood rods therefore not only break due to excessive compression and reduced surface area, but also due to pulling forces not being parallel to its grain structure.
To finally solve the issue, a secondary spruce block with identical dimensions was affixed to the opposite end of the rod using double the adhesive layer length. Either block is also drilled through on the side. A specific testing setup was devised to ensure perfect coaxiality.

During testing the samples are fixed in the UTM by inserting metal rods into these holes. This allows in conjunction with a bearing fixture for up to five degrees of freedom during the test. This setup achieved adhesive or substrate failure every time, and no hardwood rod failed.

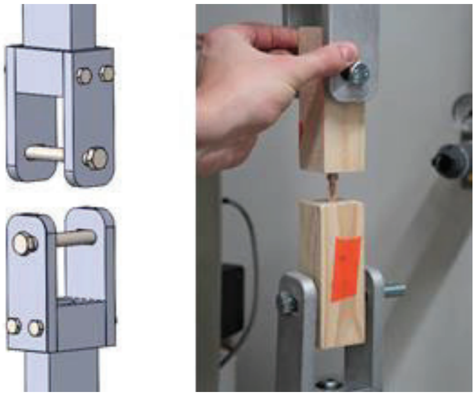

Figure 5: Test setup for the GiR

3 RESULTS AND DISCUSSION

3.1 Investigations on lap shear samples

The results of the lap shear tests with the different adhesive modifications described in

Table 1 are summarised below in Figure 6.
The original formulation of the adhesive, labelled herein REF, resulted in a lap shear strength of 12.7 MPa, with a variance (defined as standard deviation divided by the average) of 10%. Reducing the water-content, i.e., considering the modification M30, resulted in a reduction of the average lap shear strength by roughly one third, while increasing the variance to a relatively value of 25%. Regarding chalk, it is to be noted that the addition of 30% (M31) did neither affect strength nor variance; for 50% (M32), however, the average lap shear strength increased marginally by 8%, with an almost unchanged variance of 12%. Lastly, adding 30% sawdust to the basic adhesive formulation (M33) reduced the average lap shear strength by 20%, without affecting the variance.

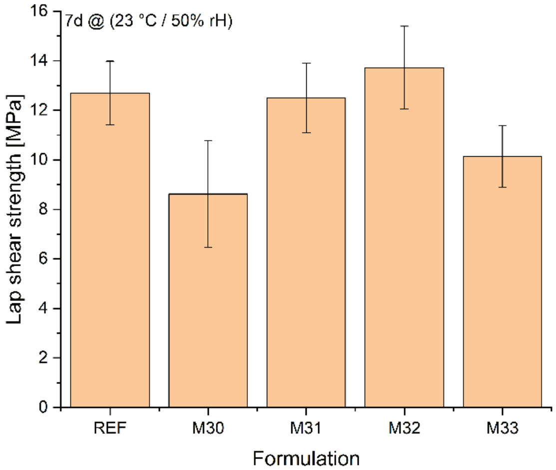

Figure 6: Experimentally determined lap shear strengths with the formulations allowing for larger adhesive layer thickness

Animal glue is made from collagen. While the polymer molecules in collagen are present as a triple helix, in animal glues they are converted into random coils. During swelling, melting (processing of the adhesive) and drying (within the adhesive layer), the polymer molecules partially convert back to triple helix. These triple-helix structures act like nodes in a physical network. Reducing the water content during processing of the gelatine-based adhesive affects this conversion from random coils to triple helix. The decreased adhesive bonding strength of the samples with reduced water content may be an indication of a weaker physical network due to the lower content of these triple-helix structures.

The aforesaid short summary of the results may lead to the conclusion that the different adhesive formulations may be easily ranked in terms of mechanical performance. However, because of the statistical nature of all experimental work, a closer statistical look through an analysis of variance (ANOVA) is required. Accordingly, the data was viewed through Tukey's test at a significance level of 0.05, and allowed for conclusions with regard to the statistical significance of differences. The ANOVA showed that reducing the water content resulted in a significant decrease of strength compared to the reference, both chalk series, but not if compared to the addition of sawdust. On the other hand, adding the chalk (in both proportions, 30 and 50%) did neither significantly increase nor decrease strength. Lastly, the addition of sawdust, although resulting in lower average strength, did not significantly impact the mechanical performance.

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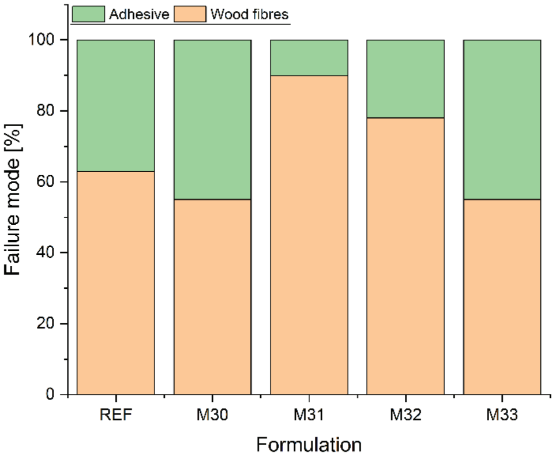

Figure 7: Failure modes observed in the lap shear tests

Focusing on the failure patterns, summarised in Figure 7, it appears that there are significant differences between the different adhesive formulations. While for the reference adhesive, slightly over 60% of all failure surface was withing the wood, this proportion slightly decreases when the water content is reduced. Chalk on the other hand seems to result in much more wood being torn off, although this results also depends on the chalk content, as higher values tend to increase the proportion of adhesive failure. Lastly, the addition of sawdust leads to the lowest observed proportion of wood being ripped-off—barely more than 50%.

Regarding the second aspect, which was to prepare adhesive formulations prone to better perform under moist conditions, and which resulted in the modifications described in Table 2 only preliminary results obtained under "normal" conditions, i.e. +23 °C and 50% rH, are presented herein in Figure 8.

The reference gelatine adhesive mixtures were modified using different techniques. For M7, Technical gelatine was first swollen overnight in demineralized water, and then tannic acid was added. The mixture was heated to 50°C, and a 10% solution of tannic acid (by weight) was introduced, resulting in gelation of the adhesive. For M10, Technical gelatine was first swollen overnight in a solution of gallic acid, and the mixture was then melted and stirred at 50°C. During this process, compressed air was introduced for 1.5 hours to modify the adhesive. In the case of M14, Technical gelatine was first swollen overnight in a solution of kalinite. The mixture was then heated to 50°C and stirred, resulting in adhesive modification. Finally, for M26, Technical gelatine was first swollen overnight in demineralized water and mixed with linseed oil before the addition of tannic acid. The mixture was heated to 50°C and stirred, resulting in the modification of the adhesive. This method is similar to M7, with the addition of linseed oil as a modifying agent.

To improve moisture resistance, we tried two different ideas: using Potassium alum, and crosslinking. Previous studies [18] suggest that Potassium improves the moisture resistance of glutine adhesives. Kalinite is also used as tannin agent and surely as in impact on collagene.

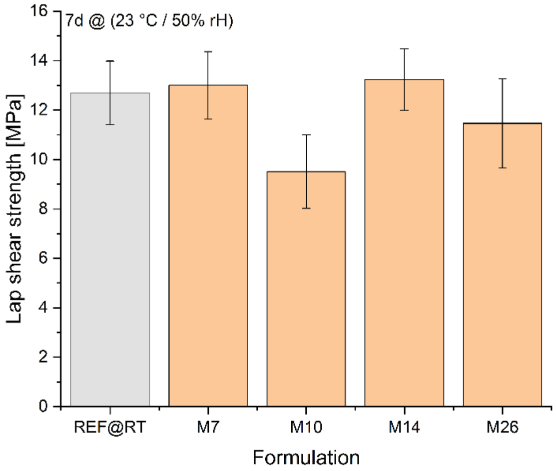

Figure 8: Experimentally determined lap shear strengths with the formulations allowing for higher moisture contents (tested under RT conditions)

It appears that, if related to the original formulation (REF), all but one (M10) modification result in very similar lap shear strengths under normal conditions that cannot be distinguished statistically (using an ANOVA, Tukey's test, *p*=0.05) from the original one. Similar is the situation regarding the scatter, expressed as variance. The mechanical behaviour under moist conditions will be the subject of the second part of this study.

3.2 Investigations on glued-in rods

An initial round of testing to determine an optimal adhesive layer thickness between the rod and the drilled hole resulted in the results presented in Figure 9. The minimum hole diameter is limited in part due to the natural deviations in roundness of the rod and the pressure required for inserting it into pre-filled holes. With an initial layer thickness of 0.2 mm and a gradual increase in thickness up to 0.5 mm an obvious trend of reduced adhesion strength between the substrates is identifiable.

Figure 10 presents a summary of the results from the GiR tests conducted using the various adhesive modifications. The different formulations include but are not limited to those shown in

Table 1. An epoxy adhesive series was also added as a comparison.

Some increases in average shear strength were observed when comparing the original REF formulation to the subsequent M29 and M30 modifications, which have the same components but differ only in water content. The M29 showed little to no effect on strength, though the variance increased from 14% to 18%. Further reduction of the water content to 60% of the REF formulation with the M30 however, increased shear strength from 4.9 MPa to 5.4 MPa, while also slightly increasing the variance. The addition of chalk to the original formulations as a filler, resulted in increased adhesive strength in both cases. With M31 outperforming the reference by 27% on average shear strength and decreasing the relative variance by a third.

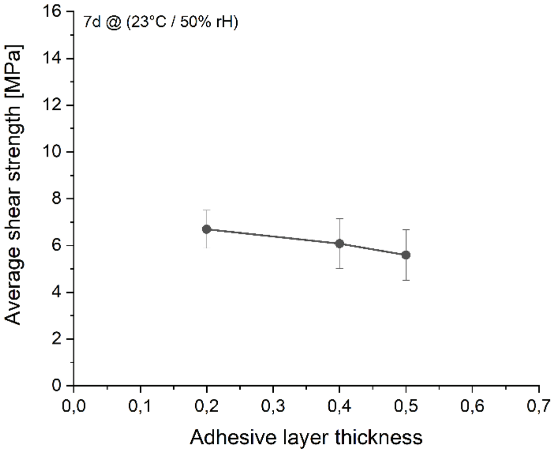

Figure 9: Test results of the GiR with selected ecological adhesives *(t_a indicates the adhesive layer thickness)*

Although still surpassing the previously observed strength values at 5.8 MPa, the M32 modification exhibits a slightly lower mean value compared to M31, with a corresponding increase in variance to 16%. Finally, adding fine sawdust to the adhesive resulted in an improved strength performance compared to the original formulation with 5.2 MPa, though with a comparatively high variance of 32%.

The 2K-epoxy which was chosen as a comparison for an adhesive able to bridge very high layer gaps, was very comparable to the M31, with an average shear strength of 6.7 MPa, which is only around 7% higher than the best ecological adhesive from this series.

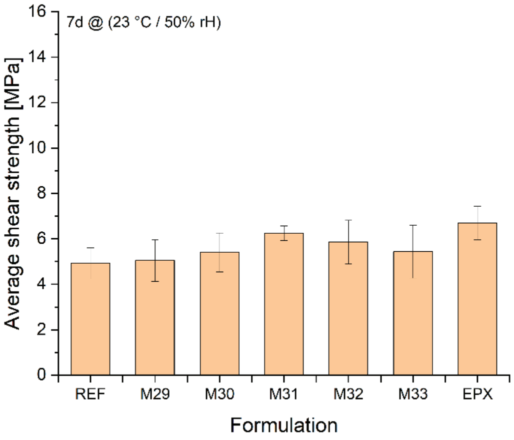

Figure 10: Test results of the GiR with selected adhesive formulations *(0.2 mm adhesive layer thickness, all embedment lengths are 30 mm)*

The testing revealed that the most common failure modes encountered were related to wood tear-out of the spruce block, which is a typical behaviour of this relatively soft material. This kind of failure occurs when the strength of the adhesive exceeds that of the wood, which can cause the wood fibres to tear apart.

Figure 11: *Typical failure of GiR with hardwood rods and eco-sourced adhesives*

Figure 11 depicts a common scenario of wood failure over adhesive failure. However, accurately quantifying the amount of wood tear-out versus adhesive failure is challenging due to the similarities in colour between the adhesive and the substrate. As the embedment length increases, the failure mode shifts from wood tear-out to splitting or fracturing of the hardwood rod either along or across the grain.

Shear strength tests on small, and large, scale glued-in rods (GiR) composed of hardwood rods glued into softwood (spruce) blocks confirmed the results obtained on the lap shear tests; mechanical performance of the "natural" adhesives ranged just slightly below that of the "chemical" ones.

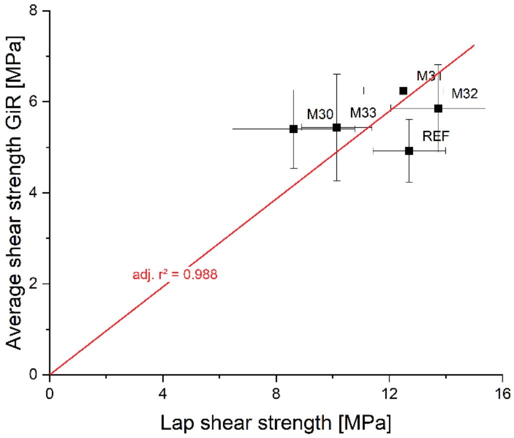

Figure 12: Comparison between lap shear strength (obtained in 1/lap shear samples) and average shear strength (obtained on GiR) for selected adhesives

When comparing the lap shear strengths to the average shear strength of the GiR, it may be difficult to compare the adhesive formulations individually from each testing method. Figure 12 aims to compare each mixture directly between lap shear and average shear strength.

While the absolute strength values differ, a direct correlation between the result can more easily identified. It is shown that some formulations form a close correlation between the two testing methods, like the M31, M32 and M33, showing that the adhesive mixtures are cross-comparable between the glued-in rods and the lap shear samples. However, outliers such as the M30

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which performed better in GiR samples, or the reference mixture which performed very well in lap shear samples, show trials of both testing variants has to be done, especially for elevated moisture conditions like those done in the second part of this study [19], to ensure accurate findings.

In order to ensure comparability between the lap shear test samples and the GiR samples for the adhesive formulations designed to perform under moist conditions, their average shear strength was also tested under RT conditions.

To also adapt these formulations to increased adhesive layer thicknesses, a preselection was made for the M10 and M14 and the mixtures were expanded to include a further series with chalk as a filler. For both formulations 30% filler was added, additionally to their original formulation. The results of this series of tests can be seen in Figure 12. For simplified comparability of the results, both the reference formulation and M31, which have the same chalk content as the M10C and the M14C, were included. The M10 adhesive was 25% weaker than the reference mixture at only 3.93 MPa and a variance of around 11%. However, the basic M14 formulation outperformed not only the reference mixture by over 10% at RT, but also some of the formulations directly aimed at improving layer adhesive gaps. The M10C, modified with 30% chalk improved on its adhesive strength, though still underperforming compared to the reference adhesive. The M14C even outperformed the M31 mixture and had very similar strengths to that of the 2K epoxy adhesive at 6.60 MPa and a variance of 8%.

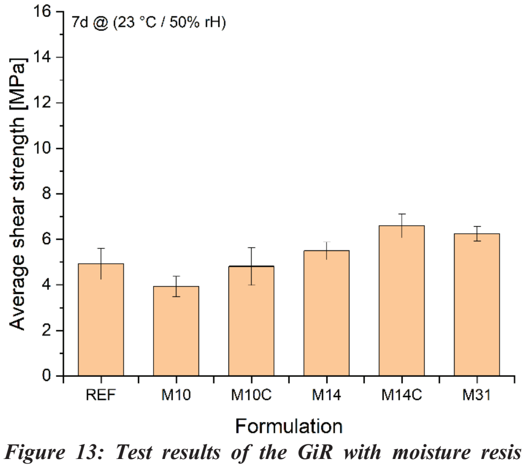

Figure 13: Test results of the GiR with moisture resistant adhesive formulations *(0.2 mm adhesive layer thickness, all embedment lengths are 30 mm)*

4 CONCLUSION

As a first conclusion, it appears that the considered ecological, or naturally sourced, adhesive, has the potential to achieve relatively high levels of lap shear strength. The measures taken to mitigate the effect of the high water-content, most prominently the addition of chalk and sawdust, improved the mechanical performance, especially for increased adhesive layer thicknesses.

Similarly, a series of modifications to ensure that strength is not degraded under moist conditions have been developed. Most of them resulted in lap shear strength in essence statistically not distinguishable from that of the reference formulation. The glued-in rods had very similar results and showed promising improvements for increased strength when additional filler was added to the mixture. However, due to the different mechanical properties of the hardwood rods, if compared to steel, it proved necessary to adapt geometrical parameters (as embedment length, slenderness ratio l/d). Numerical modelling proved adequate to predict the load bearing capacity of all experimental series with sufficient accuracy.

The follow-up study, also submitted to the WCTE [19], will investigate if these promising results are also obtained under adverse environmental conditions (in particular high moisture content).

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