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GLUED-IN HARDWOOD RODS USING BIO-SOURCED ADHESIVES — PART II: IFNFLUENCE OF ENVIRONMENTAL CONDITIONS

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ABSTRACT: The first of this two-part series presented an ecological alternative to the commonly considered metallic rods glued into wood using conventional, petrochemical based, adhesives. A series of tests spanning from lap shear samples, small-scale glued-in rods (GiR), to large scale GiR featuring hardwood rods in spruce blocks and eco-sourced adhesives, proved the feasibility of a substitution concept under laboratory conditions. This second part investigates the influence of environmental conditions on the load-capacity of aforedescribed GiR.

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]. While most investigations focus on so-called laboratory conditions (e.g. 23 °C and 50-65% rel. humidity), it is well known that conditions deviating therefrom are more critical. On one hand, temperature directly affects the performance of the adhesive [4], on the other hand moisture strongly influences the behaviour of wood [5]; additional effects results from the temperature and moisture induced deformations [6]. All these effects potentially lead to a reduction of the load-bearing capacity of bonded joints involving wood. Aforesaid is also true for glued-in rods (GiR) [7–9].

Gelatine adhesive is a type of adhesive that has been used for a long time, but its durability and resistance to environmental conditions have been questioned. Recent studies have shown that gelatine adhesive has good potential for use in various applications, such as wood and paper conservation. However, its performance may depend on factors such as the preparation method and the environmental conditions it is exposed to. Gelatine adhesive may be vulnerable to degradation by factors such as humidity, temperature, and microorganisms, which can affect its long-term stability. Some researchers have suggested that gelatine adhesive can be improved by modifying its properties or using it in combination with other materials. Overall, more research is needed to fully understand the durability of gelatine adhesive and how it can be optimized for different applications. To improve the durability of gelatine-based adhesive to environmental conditions, several additives can be used. One effective approach is to modify the gelatine itself to enhance its properties. For instance, adding crosslinking agents or modifying the gelatine with other polymers or resins can improve its mechanical and water resistance properties. Other additives that may improve the durability of gelatine-based adhesive include plasticizers, which can improve flexibility and reduce brittleness, and preservatives, which can protect against microbial degradation. Additionally, fillers and reinforcement materials, such as cellulose or silica, can be added to improve mechanical properties and reduce shrinkage. The specific choice and amount of additives will depend on the specific application and the desired properties of the adhesive.

1.2 OBJECTIVES

The research summarised in this publication continue those presented in the companion paper [10], in which the potential substitution of metal by hardwood for rods and petrochemical adhesives by eco-sourced ones has been demonstrated under laboratory conditions. The focus of this second part is set on environmental conditions considered critical for most eco-sourced adhesives, and timber engineering in general.

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2 MATERIALS AND METHODS

2.1 Adhesives

The fundamental information related to the adhesive used, Technische Gelantine 400/115, a gelatine marketed by the German company Fritz Häcker GmbH, was reformulated in several modifications so to result in two types of improvements. Firstly, 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, improving—if necessary—the mechanical performance of the adhesives under elevated moisture contents. Both aims were achieved to a series of modifications summarised in Table 1.

Table 1: Summary of the adhesive formulations

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

Modification with tannic acid: One part of Technical Gelatine was mixed with three parts of demineralised waters and left to swell overnight. This prepared adhesive was melted in combination with stirring in a water bath at 50 °C. A solution of tannic acid (10 mass %) was adjusted to pH 8 with the aid of NaOH and added to the adhesive. The reaction started immediately and gelation of the adhesive begun.
Modification with gallic acid: A solution of gallic acid (0.1 mass %) was adjusted to pH 8 with the aid of NaOH. One part of Technical gelatine was mixed with three parts of gallic acid solution and left to swell overnight. The adhesive was melted with stirring in a water bath at 50 °C and clean compressed air was introduced into the reaction mixture for 1.5 hours.
Modification with kalinite: One part of Technical gelatine was mixed with three parts of kalinite solution (1 masse %) and left to swell overnight. The adhesive was melted with stirring in a water bath at 50 °C
Modification with tannic acid and linseed oil: same procedure like modification with tannic acid alone. Linseed oil is added to the molten adhesive (before tannic acid is added).

This second part of the study focuses on the mechanical performance of the original adhesive's modification described above. For more details refer to the first part of this study [10]. For that, several series of ten lap shear samples (DIN EN 1465) were manufactured from beech (*Fagus sylvatica*) substrates (19.5 × 5.2 × 80.5 mm³) conditioned at 23 °C and 50% relative humidity (rH), bonded, and subjected to different environmental conditions. The first thereof was a co-called climatic Box briefly described as follows: samples were placed in a lockable plastic box over a saturated salt solution. A sensor recorded temperature and moisture. We worked with saturated solution of sodium chloride. The box was placed in a circulating air-drying cabinet at 40 °C for 7 days. Inside the box 75 % relative humidity was measured; that was exactly what was to be expected. Lap shear samples were removed immediately bevor testing without drying time at room temperature.

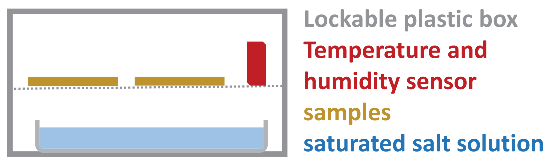

Figure 1: Principle of the climatic box

The second being a classical climate chamber set up for 45 °C and 85% rH. Samples were removed from the chamber and stored at room temperature until tested.

All tests were performed in a UTM under displacement control, with 5mm/min, until failure occurred. 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.

2.2 Glued-in rods (GiR)

As part of the second round of testing with changed environmental conditions, the GiR are also subjected to different moisture and temperature levels.

Hence, the adhesives listed in Table 1 were evaluated against commonly used "chemical" adhesives by subjecting them to elevated levels of humidity and thermal conditions to determine their mechanical performance. The tests were conducted on various wood species including spruce, which served as the base material for all GiR, as well as beech, ash, and oak, which were used for the rods. The GiR samples comprised spruce blocks measuring 45 x 45 x 120 mm³, to which ø6 mm hardwood rods were affixed using the adhesives under evaluation.

Overcoming the challenges of manufacturing and testing of the GiR samples was thoroughly discussed in the first part of this study [10]. Therefore, the manufacturing process is herein summarized. The adhesive layer thickness was carefully controlled to achieve a radial adhesive layer thickness of 0.2 mm, and spacers were used during the gluing process to ensure concentricity of the wooden rods. The rods were also cleaned with isopropyl alcohol to remove any debris and left to fully evaporate.

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In the initial study described in reference [10], the adhesive application process involved using a syringe to inject the adhesive into the lateral hole in the spruce block, while simultaneously coating the hardwood rod with the adhesive. The rods were then cantered and pressed into the hole using spacers to ensure precise concentricity.
However, during the process of liquefying the M10 adhesives at 50° C, the resulting mixture remained very viscous and would gelatinize immediately. This meant that the adhesive had to be applied immediately, within seconds, after melting so to avoid it solidifying in the syringe.
To speed up the process of pressing the gelatinous adhesive mixture into the gap, a different bonding method was used. Specifically, in large-scale GiR test trials involving chemical adhesives, the adhesive is injected laterally into a hole that is perpendicular to the hole for the hardwood rod. The rods are inserted into the hole beforehand, and the adhesive is then pressed in until it visibly emerges from the sides of the rod at the top opening of the hole. Adopting this process however was not as straight forward.
Because the adhesive layer thickness for the ecological glues is smaller by a factor of 5 than that of chemical adhesives, increased hydraulic pressure was generated when the adhesive was pressed in, which caused the wooden rods to be forced out like a piston. To prevent this from happening, a specially designed bonding device, which is shown in Figure 2, was used to pretension and centre the hardwood rods using tension springs and guide rods.

Figure 2: Bonding device designed for the rapid application of adhesives

After applying the adhesive, all samples were cured and conditioned for 7 days at 23°C and 50% rH. In the same fashion as the second variant of environmental conditions of the lap shear samples, all GiR were placed in a climate chamber at 45°C and 85% rH for 14 days after curing.

Testing the adhesive performance involved rigidly clamping each side of the sample in the UTM under displacement control and 2 mm/min until failure. However, clamping the hardwood rod directly resulted in inadequate findings due to deformation of the wood fibres, inducing stress fractures and reducing tensile strength of the dowel. To limit compression of the wood across the grain, to stop the rod from failing before the adhesive, several options were tried.
Increasing the hardwood rod diameter for instance still did not achieve satisfactory results, due to the dowel still breaking during testing. Other different attempts are thoroughly discussed in part one of the study [10].
The hardwood rods broke due to excessive compression, as well as pulling forces not being parallel to its grain structure. To reduce the pulling forces and combat the other issues a combination of different elements solved this problem.

One of these elements involved reducing the bonding depth of the glued-in rod to decrease the tensile load on the rod during testing. However, the most significant improvement was achieved by affixing a secondary spruce block with identical dimensions to the opposite end of the rod using double the adhesive layer length. Either block was also drilled through on the side, and a specific testing setup was devised to ensure perfect coaxiality. During testing, the samples were held in the UTM by inserting metal rods into the side holes, allowing for up to five degrees of freedom during the test. This setup achieved adhesive or substrate failure every time, and no hardwood rod failed.

3 RESULTS AND DISCUSSION

3.1 Lap shear samples

In a first round of experiments, samples were first conditioned for 7days at 23 °C and 50% rH, and then placed in the aforedescribed climatic box (40 °C and 75% rH) for another 7 days.

The resulting lap shear strengths, shown in Figure 3, ranged from extremely low 0.95 MPa (for the basic formulation, labelled REF) to 12.87 MPa (for M26, with 1 % tannic acid + 9 % linseed oil). The scatter, if expressed as standard deviation divided by the average, ranged from 9 to 15%, which remains acceptable for wood-based samples. The poor performance of the REF-samples was attributed to the favourable conditions for fungi growth in the climatic box, which contrasted with the results observed in the classical climatic chamber. After conducting an analysis of variance using Tukey's test with a significance level of 0.05, it was concluded that there was no statistically significant difference in measured strength among all series except for the REF adhesive, which was excluded from the analysis.

Comparing the performance of the aforesaid adhesive modifications after conditioning in the climatic box with that under RT (i.e., 23 °C and 50% rH) resulted in the

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following: save for the REF-series, which exhibited the already presented fungi issue, lap shear strength was on average the same, with M7 presenting a 9% lower and M10 a 20% higher value. The differences series-wise being not statistically significant—again taking out the REF-series.

Turning the focus to the failure modes (again discarding the REF-series for the reasons already stated), almost all series exhibited a clear substrate failure that manifested in ripping of a thin layer of wood, as shown in Figure 4.

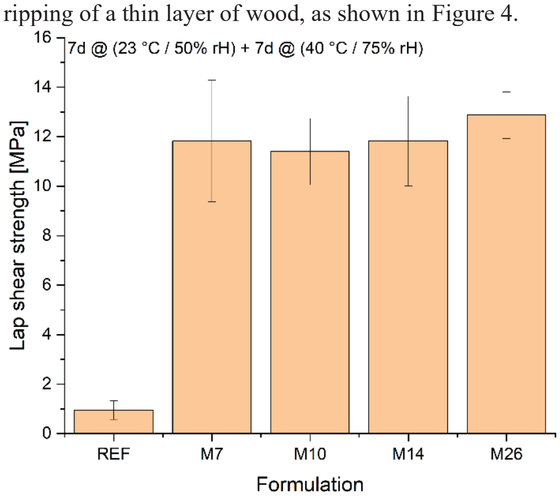

Figure 3: Experimental results obtained in the climatic box (without reconditioning to RT)

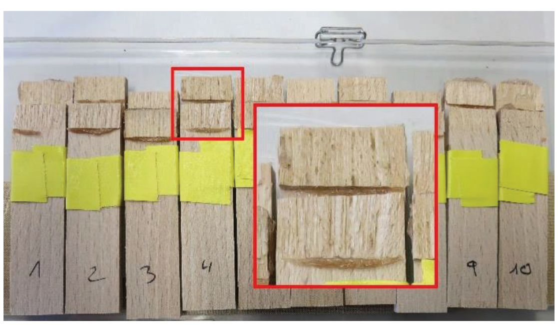

Figure 4: Typical failure mode observed

In the second round of tests, which involved a classical climatic chamber, samples were first conditioned for 7 days at 23 °C and 50% rH, then 14 days at 45 °C and 85% rH before being tested. The experimental results are presented in

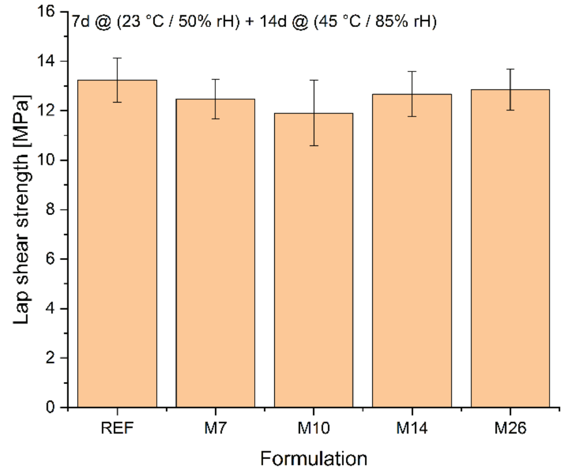

Figure 5: Experimental results obtained in the climatic chamber (with reconditioning to RT)

According to the results, the average lap shear strength for each series ranged from 11.9 MPa (for modification M10) to 13.2 MPa (which was surprisingly observed for the unmodified REF-series that no longer had fungi-related problems). On average, the variation, indicated by the standard deviation relative to the average, was 8% (with M7 having 6% and M10 having 11%).

In comparison to the lap shear strength tests carried out under room temperature (RT) conditions, the lap shear strength was observed to be higher by an average of 7%. However, statistical analysis through ANOVA using Tukey's test with a significance level of 0.05 revealed that these differences are not statistically significant. Similarly, when compared to the lap shear strength tests conducted in the climatic box, the average lap shear strength was higher by 4%, but not statistically significant, except for the REF-series, which exhibited fungal issues.

The issue with the moisture resistance of technical gelatine is due to the fact that technical gelatine can absorb a lot of water and swells. This reduces the mechanical properties, including the cohesive strength.
The first step was storing samples in a climate chamber. After removing the specimens after 7 in the climate chamber, these samples lay for an indefinite time at RT and had the opportunity to dry back. This means that at the time of testing, the technical gelatine was no longer in a swollen state. The test result shows two things:

- The technical gelatine is able to re-dry very well.
- Secondly, no ageing in the form of a loss of strength due to hydrolytic degradation could be detected.

In order to measure the strength of the bonds in the swollen state of the technical gelatine, we carried out the humidification in the box over a saturated NaCl solution. The box was taken to the testing machine after being exposed to the increased temperatures and moisture in the closed state. The samples were removed from the box just

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before they were clamped in the testing machine and then tested immediately. The adhesive had no time to dry back. These tests show:
The unmodified technical gelatine has a low bond strength of less than 1 MPa in the swollen state (as was to be expected). The modifications, however, significantly improved the moisture resistance with an average lap shear strength of 12 MPa.

3.2 Glued-in Rods

The measurement of moisture content in the wood is a critical aspect of evaluating and comparing the adhesive strength to those not exposed to higher levels of humidity. To analyse the moisture content of the test samples after conditioning in the climate chamber (14d @ 45°C/85% rH), the glued in rods were measured in regular intervals during their period in the climate-controlled environment.
For this study, we employed two methods to measure the moisture content of wooden blocks. The first method was a pin-type moisture meter, which is a widely used tool for determining the moisture content of wood. This type of moisture meter can only measure the moisture content up to a depth of a few millimetres. This limited depth measurement can be a challenge when trying to assess the overall moisture level of a wooden block, as the surface of the wood can become saturated with ambient humidity very quickly, while the interior may remain dry.

To address this issue, we also measured the weight of each wooden block at regular intervals. This method provides a more accurate and comprehensive assessment of the moisture content of the wood, as it captures the changes in weight over time, reflecting the moisture uptake or loss. Figure 6 shows the results of both the moisture meter and weight measurements.

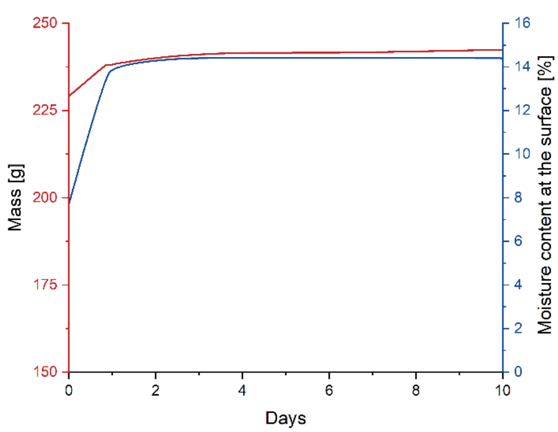

Figure 6: Measured wood moisture in comparison to weight increase over time

Our findings revealed that although the humidity values measured with the moisture meter reached a stable plateau within the first 48 hours, the moisture level in the wooden blocks continued to increase significantly until day four of their exposure in the climate chamber. After that, the moisture content plateaued and did not increase any further.

The first part of this study [10] conducted a series of GiR tests comparing the different variations and mixtures of the adhesive formulations, also listed in Table 1 (Ref, M29-M33). Following the findings of that study, a preselection of those mixtures, based on the adhesive strength for higher layer thickness was made, as seen in Table 2. The highest performing as well as formulations without additional fillers were chosen.

Table 2: GiR adhesive selection

		Shear strength dry [MPa]
M10	3 % kalinite	3.93 ± 11%
M10C	M10 + 30% chalk	4.81 ± 17%
M14	75% gallic acid + 25% gelatine	5.49 ± 7%
M14C	M10 + 30% chalk	6.59 ± 8%
M30	REF with water-content reduced to 60%	5.40 ± 16%
M31	REF + 30% chalk	6.25 ± 5%
M33	REF + 30% sawdust	5.44 ± 22%
EPX	Chemical Epoxy	6.70 ± 11%

The effect of warm temperatures and high moisture levels (45 °C and 85% rH) for 14 days on the adhesive properties was investigated with the aforementioned adhesives. The results as presented in Figure 7 showed that all ecological, gelatine-based adhesives performed poorly.

The ecological formulations exhibited a reduction in tensile strength on average by approximately 80% after exposure in the climate chamber. Differentiating between the different mixtures, the M33 showed the highest average tensile strength of 2.50 MPa with a variance of 5%. On the other hand, the M30 exhibited an average tensile strength of 2.00 MPa similar to the M31, with a variance of 6%.

To compare the GiR samples against an adhesive that is moisture resistant, the same 2 component chemical epoxy from the first study [10] was used. It performed only 20% worse compared to its dry tensile strength counterpart at 5.92 MPa. It is noteworthy that the failure mode exhibited mostly wood tear-out and not adhesive failure. The decreased tensile strength performance can be explained by the fact that when wood is exposed to warm temperatures and high moisture levels, the lignin, which is the natural glue holding the wood fibres together, can degrade and lose its adhesive properties. This can lead to a weakening of the intermolecular bonds between wood fibres and result in decreased strength of the wood. In addition, moisture can cause wood to swell and deform, further reducing its strength and durability.

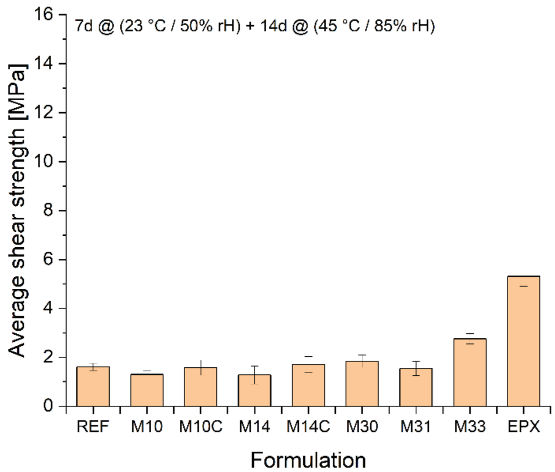

Figure 7: Average shear strength of the GiR (without reconditioning to RT)

In conclusion, the results of the tests of the mixtures tested at RT part one of this study [10] indicate that the ecological adhesive formulations can perform well under room temperature and humidity conditions. However, they are not be suitable for applications where the wood will be exposed to elevated temperatures and high moisture levels for extended periods.

To improve the performance of the ecological, gelatine-based adhesives, the specially formulated mixtures for humid environments (M10, M14 and M10C, M14C) were also tested as GiR samples. A comparison of the M10 and M14 and their chalk modified counterpart, against the other eco-adhesive variations from Table 2: GiR adhesive selectionTable 2 and the chemical epoxy, is depicted in the same Figure 7.

The reference adhesive, consisting of 3-parts water and 1-part technical gelatine, was used as the basis for the new formulations. The M10 mixture, which substituted the water for a kalinite solution, exhibited an average strength of only 1.30 MPa during the climate chamber experiment. Further regression was observed with the M14 formulation, which substituted the water with gallic acid, resulting in a further 2% decrease in adhesive performance. Both eco-adhesives displayed a high variance with the M14 mixture reaching 30%.

When compared to the gelatine-based adhesives not specially formulated to withstand higher levels of moisture, it was evident that the M10 and M14 eco-adhesives were not able to effectively adhere to wood. The series of tests with additional 30% chalk content, while marginally better at 1.57 MPa and 1.69 MPa for the M10C and M14C respectively, could not realistically improve on the adhesive performance.

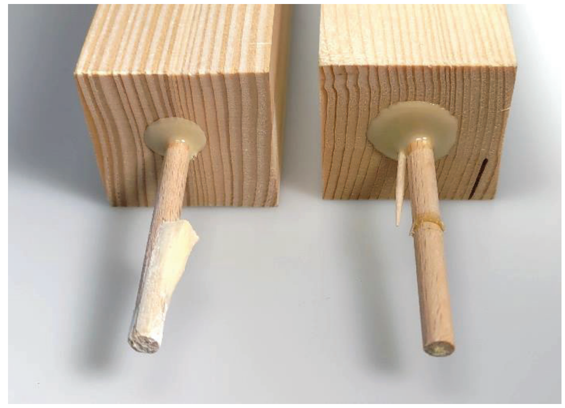

Figure 8: Wood tear out (left) and adhesion failure (right)

Quantifying the failure modes of the adhesives, all ecological adhesives showed considerable adhesion failure, with little to no wood failure. Figure 8 depicts a comparison of the typical wood tear out experienced during the tests performed at RT versus the same formulation under the increased moisture and temperature levels. The hardwood rod is clear of any adhesive and shows virtually no sign of any adhesion.

4 CONCLUSION

In this second part of a study devoted to ecological, or sustainable adhesives, and their use in a structural context, the investigations presented in [10], which were carried out at RT, were extended towards more severe environmental conditions (+45 °C and 85% rH).

Firstly, an existing gelatine-based adhesive was modified so to improve its performance under the previously mentioned severe environmental conditions. This resulted in four additional mixtures.

Secondly, the lap shear strength of the aforesaid formulations, additionally to that of the reference gelatine, were determined under two different environmental scenarios: in a climatic box, with tests performed without reconditioning at RT, then in a climatic chamber with reconditioning to RT. The results indicated that it is possible to achieve similar lap shear strength under these climatic conditions, if compared to RT. It is to be noted that in the lap shear samples, the adhesive layer thickness was almost zero.

Lastly, tests on glued-in rods (GiR) were performed. A first round of tests was performed with the original formulation, to which different fillers were added (for more details refer to [10])—which were not optimised for humid environments and tested without reconditioning. These were, in a second run, compared to some of the formulations that were optimised, also without reconditioning to RT. Unlike the lap shear samples, the adhesive layer thickness in the GiR tests was set to 0.2 mm due to technological reasons. If compared to the similar tests described in [10], which were performed

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under RT, joint capacities significantly decreased by around 80% (averaged over all series). If comparing the performance of a 2K-epoxid taken as reference, exposure to elevated temperature and moisture only resulted in a decrease of roughly 20% in strength.

Regarding the resistance to temperature and moisture of ecological gelatine-based adhesives, the study showed that the influence of the adhesive layer is paramount. It is therefore not sufficient to assess the performance of such adhesives only at the level of lap shear samples (with very thin layer thicknesses).

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