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Enhancing the performance of beech-timber concrete hybrids by a wood surface pre-treatment using sol-gel chemistry

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Abstract

Timber-concrete composites require reliable connections between both components, which are usually obtained by metal fasteners or slots in the wood. In this study, an alternative approach is presented based on a fully glued connection in combination with a primer treated wood surface, to enhance the compatibility and the adhesion properties at the interface between beech wood and concrete. Prior to the gluing and the concrete application in a wet-on-wet process, the wood surface was functionalised with a xerogel obtained by means of a sol-gel process, consisting of two layers of silane nanofilms, with different functional groups, which are capable of undergoing further chemical crosslinking reactions with the adhesive. The coating with its functionalities allows for reducing the penetration of the epoxy adhesives into the wood structure and an additional chemical connection to the adhesive can be established. The main objective of this study was to analyse the effect of the surface treatment on the mechanical properties of such composites in 3-point and 4-point bending tests as well as push-out-tests. The results showed that the pre-treatment can improve the load bearing capacity of the timber-

concrete composites, but that a ductile behaviour cannot be achieved with the tested adhesives.

Keyword: Materials science

1. Introduction

Several connection systems have been developed to connect timber and concrete. Many of them are based on steel fasteners such as screws or bolts, which are indented into the timber or notched connections [1, 2, 3, 4]. Though the mechanical fastenings provide an adequate solution, certain drawbacks – in particular corrosion of the steel fasteners – can reduce the service time of the composite and have to be considered. While several studies report on the use of adhesives as an additional connection together with the steel fasteners [5, 6, 7], not much is reported on using fully glued connections. However, there might be several benefits of using an entire adhesive approach, not only with regard to the corrosion issue, but also in terms of addressing the issue of grey energy, i.e. reduction of the use of steel fasteners in wood-concrete composite structures and the possibility to establish a more time efficient and cost saving application.

Brunner et al. [8] were the first to report on an application method, in which wet concrete was directly poured on an adhesive-treated timber in a so-called “wet-on-wet” process. Among the challenges encountered, it was not possible to obtain an equal adhesive distribution at the interface as the wet concrete displaced the thin adhesive layer on the wood surface, resulting in a non-optimal overall performance of the composite. In addition, the partial penetration of the adhesive into the porous wood structure led to an adhesive depletion at the interface, causing further compatibilization issues. To address the drawbacks identified by Brunner et al. [8], a sol-gel based modification of the wood surface has been reported [9]. This study uses a chemical treatment of wood by sol-gel chemistry not only to achieve a tighter bonding of the adhesive to the wood surface via a priming system, but also to achieve an enhanced surface performance towards wettability [10, 11, 12, 13].

The wood surface pre-treatment has to fulfil certain requirements, with the aim to enhance the performance of the composite. Hence, a double layer pre-treatment was applied by spraying, in order to generate a primer [9]. This primer is composed of two alkoxysilanes with different functional groups. Fig. 1 displays the different layer of the primer and it also shows that the first layer is mainly consisting of tetraethyl orthosilicate (TEOS). The first layer is providing a hydrophobisation of the surface as it is decreasing the water transport phenomena at the wood-concrete interface, and is therefore also helping to avoid issues with concrete dehydration

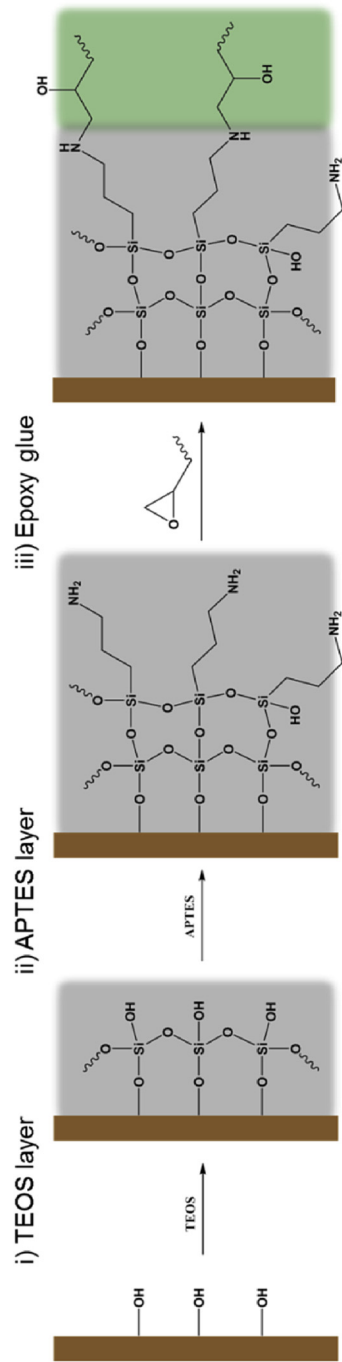


Fig. 1. Wood surface treatment with polysilanes layers. The treatment consists of two different layers, which is TEOS as a first, so called hydrophobizing layer and APTES as a second layer, the so-called functional layer.

while it protects the wood against wetting with alkali water from the concrete [14]. The second layer, which consists of (Aminopropyl) triethoxysilane (APTES), is also beneficial in providing a hydrophobic surface. In addition, through the attachment of functional group from APTES, namely primary amines, a more reactive group is present at the wood surface to establish covalent bonds, since the amine groups are capable of reacting with the epoxy functionalities from the adhesive [15, 16]. Contact angle measurements confirmed the hydrophobic behaviour of the wood treatment and it could also be shown by penetration studies that the silanization treatment influences the extent of adhesive penetration in the wood scaffold. Moreover, the study also indicated that a minimal amount of glue penetration of the adhesive is needed to establish mechanical interlocking. However, an excessive penetration is detrimental since it results in the depletion of the glue line. The developed TEOS/APTES primer treatment decreased the adhesive penetration while maintaining mechanical interlocking and chemical interlocking of the wood scaffold with the adhesive, thereby facilitating the compatibilization with concrete.

The main objective of this study was to test the impact of the developed primer system on the mechanical properties of wood/concrete composites with two different epoxy-based adhesive systems. For this purpose, comparative mechanical tests were conducted, which could be based on some preliminary mechanical tests on the impact of different surface pre-treatments on the mechanical behaviour of the composites [9]. In a first investigation it was important to test how different amounts of adhesive and the different adhesive systems affect the mechanical behaviour, which was studied in 3-point bending tests on small wood-cement paste specimens. For a more application-relevant testing setup, 4-point bending tests were conducted on larger wood-concrete specimens based on the obtained data. The test series was complemented by push-out-tests to analyse, whether the shear resistance of the interface was affected by the primer system and to reach a conclusive assessment of the impact of the pre-treatment on the ductility of the composites.

2. Materials and methods

Tetraethyl orthosilicate (TEOS), (3-Aminopropyl) triethoxysilane (APTES), Ammonium hydroxide solution, and ethanol were obtained from Sigma Aldrich. The Sikafloor®-156 and Sikadur®-31 EF were provided by SIKA AG. Beech wood (*Fagus sylvatica*) with different dimensions were prepared and set to fiber saturation point (FSP) for the 3-point bending specimens and to a standard wood water content of ~12% (storage at 20 °C and 65% relative humidity) for the 4-point bending test and the Push-Out Tests.

2.1. Preparation of TEOS sol

The sol was prepared based on an alkaline hydrolysis of TEOS in a solution of NH_4OH in ethanol [17, 18]. 20 ml of NH_4OH were dissolved in 300 ml of EtOH. The solution was kept at 30 °C for 15 min followed by the dropwise addition of 20 ml (89.5 mmol) of TEOS while stirring. The stirring was continued for 120 min to form a milky blueish sol-gel.

2.2. Preparation of APTES sol

20 ml of APTES were dissolved in a 300 ml 50:50 mixture of ethanol and water. The solution was kept at RT. the stirring was continued for 120 min in order to produce the sol-gel.

2.3. Treatment of the wood specimens

The generated sol-gels were transferred to a spraying device and applied to the wood surface. The treatment was first conducted with TEOS, of which 5 layers were sprayed, followed by curing of the sol-gel over night at 80 °C. The pre-treated specimens were allowed to cool down at room temperature and further modified by five layers of APTES. Finally modified wood specimens were cured at 105 °C for 5 h, followed by storing the specimens at a humidity of 65%, RT, until constant mass was obtained, and weight percent gain (%) was determined.

2.4. Preparation of the cement paste

The cement used for the preparation of the 3-point bending tests was a standard Portland cement with the trademark name Normo 5R (CEM I 52.5R) by Holcim AG. The water/cement ratio (w/c) was set to 0.4. The cement paste was cured for 28 days in order to reach full compressive strength, which is also referred as the standard strength.

2.5. Preparation of the concrete

The basis of the cement, which was used exclusively for the 4-point bending test and push-out-test, was the same one as for the cement paste, Normo 5R. The density of the cement was 339.9 kg/m³. The amount of the aggregates was set to be 1991 kg/m³, which is composed of 35% of the total amount out of aggregates (sand and gravel) size 0–4 mm, 30% of the total amount with an aggregate size of 4–8 mm and the residual 35% were aggregate size of 8–16 mm.

2.6. Preparation of the adhesive

Two 2-component epoxy-based adhesive systems provided by SIKA AG were used. For the first system Sikafloor®-156, the ratio between component A and B was set to

3:1 and 0.0127 g of thickening media per gram of glue was added to adjust the viscosity. The pot life of the glue was set to 60 minutes. The second epoxy adhesive was Sikadur®-31 EF, which was prepared as recommended by the manufacturer. In brief, the components A and B were mixed in ratio of 2:1 and the pot life is 75 minutes.

2.7. Preparation of the composites for mechanical testing

The specimens for 3-point and 4-point bending as well as the specimens for the push-out tests were prepared by a wet-on-wet process [8], in which the wet concrete is casted on the uncured glue. The specimen for the 3-point bending test were prepared in a steel mold. Prior to the application, the wood was set close to the fiber saturation point to avoid any future cracks in the cement paste due to wood shrinkage. Once the wood specimens reached constant weight, they were put into steel moulds, in which they were kept for the entire time of cement paste curing (see Fig. 2). The specimens were cured for 28 days under the same climatic conditions. After reaching day 28, they were immediately subjected to bending tests. The reason for the different treatment was due to the fact that concrete instead of cement paste was used for the 4-point bending test, which is supposed to be more resistant towards the internal stresses caused by the swelling and shrinkage of wood. The wood was then placed into the mold and the glue was poured directly on top of the wood where a certain setting time was allowed, which was 20 minutes for the Sikafloor®-156 and 30 minutes for the Sikadur®-31 EF. The wet cement paste or concrete, respectively, were then poured directly onto the glue and the specimens were stored. The wet-on-wet procedure was also applied for the push-out tests. Since the specimen has two sides with concrete, it was decided that one side was prepared first and followed by the next side after allowing the concrete to set for two days. The molds for the three different testing setups are shown in Fig. 2.

2.8. 3-Point bending tests

The three-point bending tests were performed on a Zwick and Roel Type BZ1-MMZ100.ZW03v (Fig. 3). The maximal force, which can be applied on that device is 100 kN. The size of the specimens arise from $160 \times 40 \times 10 \text{ mm}^3$ wood layer and $160 \times 40 \times 30 \text{ mm}^3$ cement paste layer and the distance between the two bearing loads was 14 cm. The test was performed according to Swiss SIA Norm 265, which prescribes a test setup as follows: duration of the test 300 seconds at 65% relative humidity and 20 °C.

2.9. 4-Point bending test

The four-point bending tests were performed on a Walter und Bai Type 502/4000/100v (Fig. 3). The maximal force, which can be applied on that device is 100 kN.

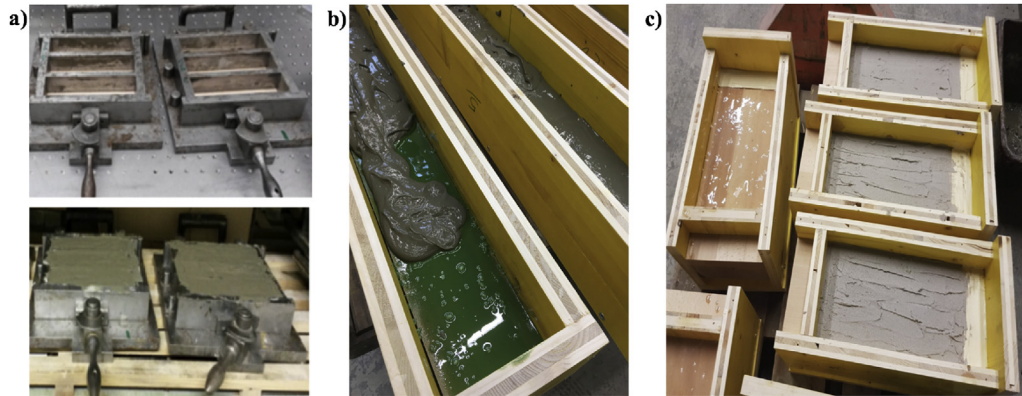


Fig. 2. Pictures of the three different composite preparation setups. a) upper photograph: Steel molds for the preparation of the specimens for the 3-point bending tests. Each apparatus allowed for a preparation of three specimens simultaneously. Bottom: Steel molds with wood and fresh cement paste on top. The thickness of the wood was 10 mm and 30 mm of cement paste were added. b) Preparation of specimens for the 4-point bending tests, which were prepared in a wooden mold. The glue was stained green to investigate whether the glue stayed in place during casting. c) The molds for the preparation of the push-out tests show the two different adhesive systems, right the Sikadur®-31 EF and left the Sikaflow®-156.



Fig. 3. Setups of all three loading conditions after the failure of the composites. The image on the upper left side shows the 3-point-bending test; the image on the upper right side shows the push-out test, with the described ‘sandwich’ geometry of the specimen and at the bottom is the 4-point-bending test. All pictures show the cracked specimen in order to illustrate the failure behaviour.

The size of the specimens arise from $100 \times 12 \times 3 \text{ cm}^3$ wood layer and $100 \times 12 \times 12 \text{ cm}^3$ concrete layer. The distance between the two testing heads was 28 cm and the distance between the two bearing loads was 84 cm. For testing, a velocity of $v = 0.05 \text{ mm/s}$ was chosen and the end of the test was set to 100 seconds after the first noticeable crack. Tests were performed at $20 \text{ }^\circ\text{C}$. The bending test was analyzed by the provided software testXpert II.

2.10. Push-out test

The push-out tests were performed on a Schenck Hydropuls. The geometry and size of the specimens was changed to a 'sandwich' geometry, where a $10 \times 30 \times 30 \text{ cm}^3$ wood specimen was glued to two concrete layers ($60 \times 25 \times 30 \text{ cm}^3$) with an offset of 5 cm. Fig. 3 is illustrating the specimen and it shows that the adhesive was applied on both sides of the specimen. The load was applied on the wood, which is in the middle of the 'sandwich' in order to test the ductility of the system. The test was conducted in two phases. In an initial phase, a constant force of 100 kN with a velocity of $v = 0.01 \text{ mm/s}$ was applied. The duration of the initial test was 30 seconds and then the force was reset to 0 kN. The actual test phase was conducted subsequently to the initial phase, where the specimen was fully loaded with the same velocity. The end of the test was set to be after the first noticeable crack. All tests were performed at the standard condition of $20 \text{ }^\circ\text{C}$. The results were analyzed by the provided software of the device. Fig. 3 shows the setups of all three loading conditions after the failure of the composites.

2.11. Statistics

The differences between the arithmetic means of maximum load values was tested on significance by a t-test at the 0.05 level.

3. Results and discussion

3.1. 3-Point bending tests

In a first study, the influence of the amount of the Sikafloor®-156 adhesive was tested for unmodified and pre-treated surfaces of wood-cement paste composites. Fig. 4 shows the force-deflection curves obtained from the 3-point bending tests. The plain lines are used for the untreated specimens with two different amounts of adhesive. Although both curves show a similar slope at the beginning of the test, the unmodified specimens glued with a higher adhesive amount show a larger deflection before rupture. However, since the differences between the slopes of the corresponding curves were rather minor, one may conclude that the stiffness of the unmodified composite is not much affected by the glue amount. In addition, the maximum force values for the untreated specimen were almost equal despite a variation of the glue

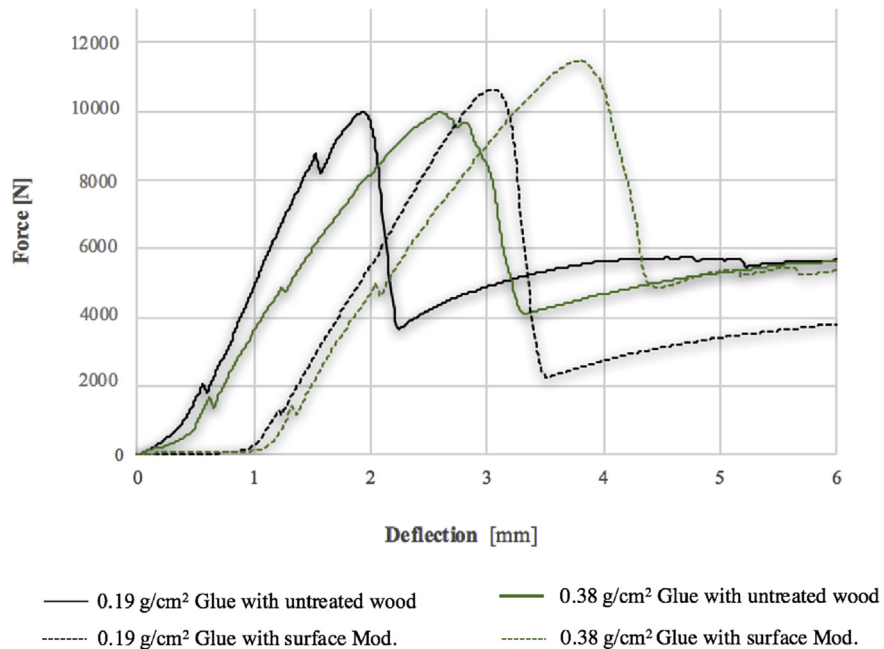


Fig. 4. 3-point bending tests on wood-cement paste composites glued with Sikafloor®-156 and Sikadur®-31 to compare treated and untreated wood surfaces with different adhesive amounts. The force-deflection curves of the specimens with surface treatment is shifted along the x-axis, indicating that the loading of the specimens started not directly, but after 1 mm transverse movement.

amount. For the treated specimens (dotted lines) again no effect on the slope in the initial phase of the tests was observed, but a larger deflection of those samples with a higher adhesive amount could be observed at higher load levels. In terms of the maximum force, almost the same average values were measured for untreated specimens glued with the different amounts of adhesive. For the treated specimens, higher values were reached for the higher adhesive amount, but without a significant difference to the specimens glued with the lower adhesive amount.

When comparing the unmodified and modified specimens with the same adhesive amounts, the experimental force-deflection curves indicate that there is no effect of the surface pretreatment on their slope, but that the treatment allows for reaching higher load bearing capacity. However, the difference was only significant for the samples treated with the higher amount of adhesive, which showed also the higher loading-carrying capacity. For the 0.38 g/cm^2 glue amount, composites with an unmodified surface could withstand an average maximal load bearing force (F_{\max}) of 10.28 kN, whereas the composite with a treated surface failed at an average F_{\max} of 11.78 kN.

The first 3-point bending tests revealed that the effect of the amount of adhesive is rather minor. The Sika Floor adhesive is a product usually used for flooring applications, which means that this product can be used in combination with concrete to provide a good adherence. Brunner et al. [8] showed in their publication that this

epoxy based system can be used as an adhesive, since it has also good adherence to timber.

Further, it could be extracted from force-deflection curves that the surface treatment may have an impact on the load bearing capacities of the composites. Since the differences were only minor, further 3-point bending tests were conducted with the higher adhesive amount, this time for a comparison of the Sikafloor®-156 and the Sikadur®-31 EF. The latter adhesive is also known for its high affinity towards concrete, but has a much higher viscosity in comparison to the Sikafloor®-156. Although it is more recommended for the gluing of dry concrete, also the Sikadur®-31 glue was subjected to a wet-on-wet application and the glue amount was set to 0.38 g/cm^2 for both glue systems based on the first investigation.

The results of the second 3-point bending test series are summarised in Fig. 5 and Table 1. For a direct comparison of the influence of the two adhesives on the mechanical performance of the untreated and treated composites, new specimens were prepared for the Sikafloor®-156 glue composites. Likewise for the first test series for both adhesive types, only a minor difference between the slopes of the untreated and the surface treated specimens was observed. The slopes of both samples glued with Sikafloor®-156 were slightly steeper than those glued with Sikadur®-31, which had also a larger total deflection at maximum load. In terms of the maximum load values no significant differences were found between samples glued with Sikafloor®-156 and samples glued with Sikadur®-31 (comparing unmodified with unmodified and modified with modified samples). However, when

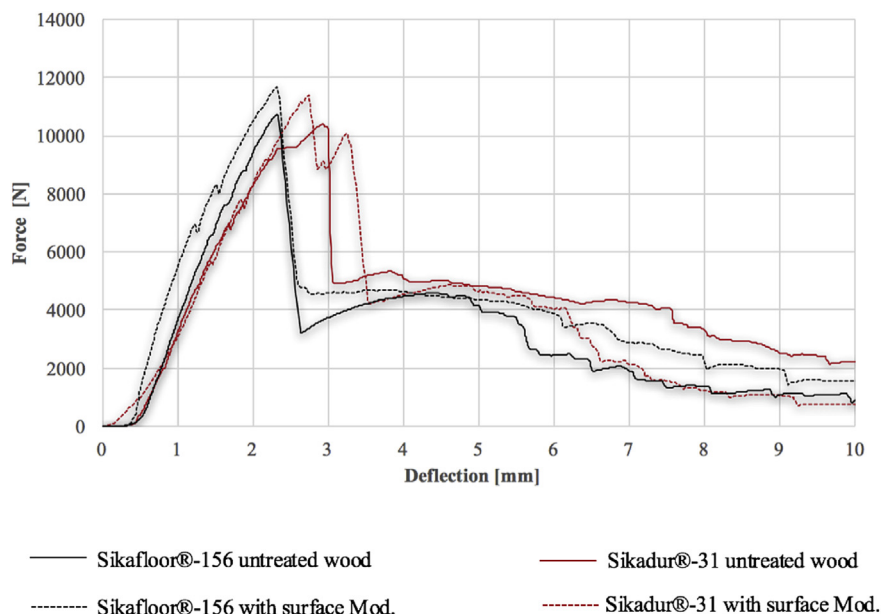


Fig. 5. 3-point bending tests for the comparison of Sikafloor®-156 and Sikadur®-31 glued composites with treated and untreated wood surfaces. Each graph shows a reference curve of 10 measurements.

Table 1. Arithmetic means and standard deviations for Fmax as an indicator of the load bearing capacities of the timber-cement paste composites in 3-point bending tests.

Glue Type	Specimen	Glue [g/cm ²]	Mean value Fmax [kN]	SD [kN]
Sikadur®-31 EF	10	0.38	9.87	2.56
	10 (Modified)	0.38	11.34	1.89
Sikafloor®-156	10	0.38	9.32	1.56
	10 (Modified)	0.38	11.12	1.02

comparing untreated and surface-treated composites, again a higher load bearing capacity was found for the surface-treated samples. This difference was significant for Sikafloor®-156, but not for Sikadur®-31, due to the large standard deviation of the unmodified samples.

The obtained load bearing capacities show that epoxy adhesives can be utilized for the wet-on-wet process without losing adherence properties at high pH. In addition, there was a slight positive effect observed of the wood surface pre-treatment with the sol-gel chemistry on the load bearing capacity of the small beams, which indicates that the wood modification may enhance the compatibility of the composite components. The similar behaviour for both epoxy adhesive types is an indication that the priming system is well adapted to the chemistry of epoxy-based adhesives. The curves show no plastic deformation behaviour, hence a beneficial ductility of the composites was not observed.

3.2. 4-Point bending tests

The three-point bending tests helped to adjust the glue amount and also showed that there is a slight impact of the surface treatment. For more application relevant measurements, 4-point bending tests on larger specimens and concrete instead of cement paste were performed. In terms of wood preparation, in this case the modified and unmodified beech wood were stored at 65% humidity and 20 °C till mass equilibrium was reached to adjust a moisture content of approximately 12%. During the wet-on-wet process, it could be observed that the pouring of the wet concrete did not push the adhesives away and therefore the viscosity of the adhesives was appropriate, which in terms of Sikafloor®-156 was mainly obtained by the thickening media. However, the application of Sikadur®-31 on the one-meter scale was more difficult than the application of Sikafloor®-156, taking more effort and time and one can state that the high viscosity of the Sikadur®-31 glue is not very suitable for the wet-on-wet procedure.

The mechanical behaviour of the composite is shown in Fig. 6 in terms of force deflection curves and Table 2 summarises the measured Fmax values of the 4-

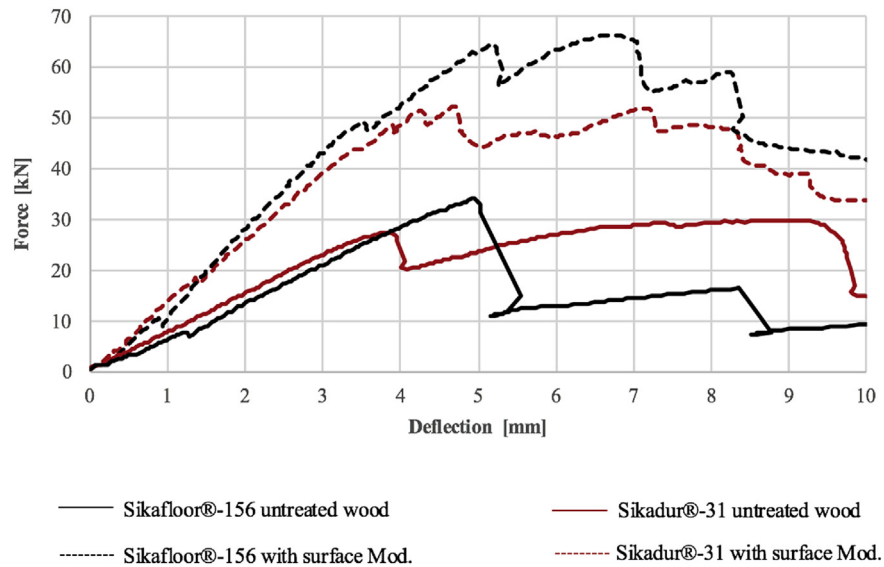


Fig. 6. 4-point bending tests for the comparison of Sikafloor®-156 and Sikadur®-31 glued timber-concrete composites with treated and untreated wood surfaces. Each graph shows a reference curve of 10 measurements.

point bending tests. Again, both epoxy adhesives showed very similar results in load bearing capacity and stiffness for both untreated and surface treated composites. However, in contrast to the 3-point bending tests, large differences between modified and unmodified specimens could be observed in 4-point bending tests for both adhesive systems. In terms of Sikafloor®-156 the maximal force (F_{max}) values for untreated specimens were only about half the ones of the modified specimen and a similar relationship was found for Sikadur®-31 EF. Moreover, the 4-point bending tests indicate an impact of the surface treatment on the stiffness of the beams. For both adhesive systems a much higher slope can be observed for the surface treated samples in comparison to the untreated samples.

The failure of the composite appeared to be less brittle than in the 3-point bending tests with large plastic deformation, in particular for both surface treated specimens. However, the failure of the glue line and the separation of the wood and concrete

Table 2. Arithmetic means and standard deviations for F_{max} as an indicator of the load bearing capacities of the timber-concrete composites in 4-point bending tests.

Glue Type	Specimen	Glue [g/cm^2]	Mean value F_{max} [kN]	SD [kN]
Sikadur®-31 EF	10	0.38	30.34	6.03
	10 (Modified)	0.38	59.79	6.42
Sikafloor®-156	10	0.38	36.84	5.45
	10 (Modified)	0.38	61.77	7.29

layers could not be assigned to a specific point on the force-deflection curves. Therefore, regions of load bearing of the entire, intact composite and of the mechanical behaviour of the separated composite layers could be distinguished. As a consequence, one cannot state on the basis of these measurements, whether the adhesives and the wood surface treatments have the potential to contribute to a ductile behaviour of the composites or not. In order to gain further insight on this particular aspect, push-out-tests were performed with the objective to investigate a potential plastic deformation behaviour of the entire composites.

3.3. Push-out tests

The push-out tests were conducted with a specific focus on the glue line performance. In order to perform the tests, the shape and geometry of the specimen had to be changed to a so called ‘sandwich’ shape where the wood was used with a moisture content of 12% and had two concrete slabs glued on each side. As the applied adhesives were rather viscous, the preparation of the specimen was a difficult task as both sides of the test specimen had to be prepared by the wet-on-wet process. Therefore, only five specimens each were prepared and tested after a concrete curing time of 28 days at a humidity of 95% and 20 °C.

The load-time curves for the push-out tests are shown in Fig. 7 and the obtained values are summarized in Table 3. The load-time curves for the composites during a duration of up to 400 seconds, display the effect of the surface treatment and the

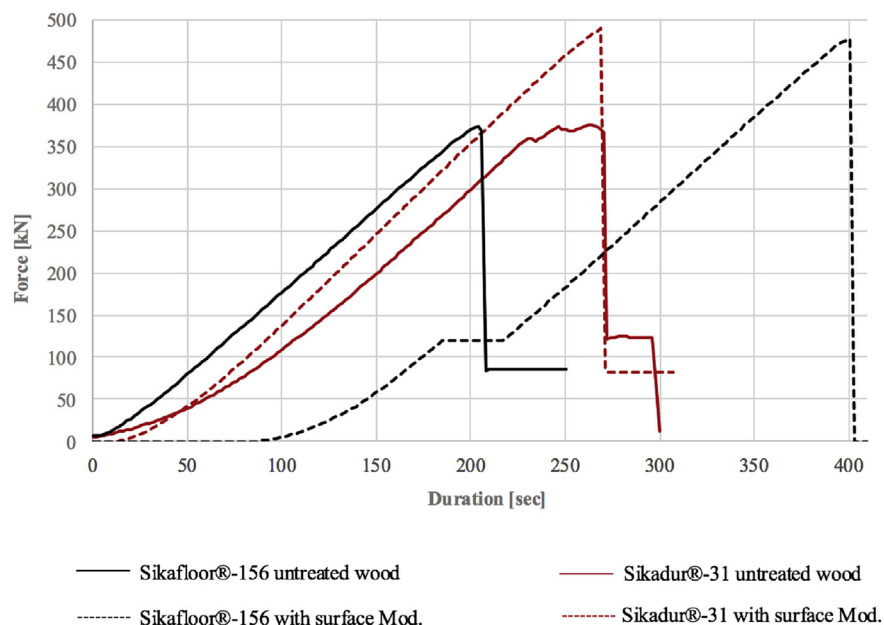


Fig. 7. Push-out tests for the comparison of Sikafloor®-156 and Sikadur®-31 glued timber-concrete composites with treated and untreated wood surfaces. Each graph is the reference curve of 5 measurements.

Table 3. Arithmetic means and standard deviations for Fmax as an indicator of the load bearing capacities of the timber-concrete composites in push-out tests. The glue amount is the amount, which was applied on each timber surface.

Glue Type	Specimen	Glue [g/cm ²]	Mean value Fmax [kN]	SD [kN]
Sikadur®-31 EF	5	0.38	374.93	69.25
	5 (Modified)	0.38	488.78	18.20
Sikafloor®-156	5	0.38	373.56	62.50
	5 (Modified)	0.38	477.45	17.84

comparison between the two adhesive systems. Again, for both adhesive systems higher Fmax values could be observed for the surface treated composites in comparison to the non-treated ones. The Fmax values for the Sikafloor®-156 adhesive were 373.56 kN for the non-treated and 477.45 kN for the treated specimens, whereas for the Sikadur®-31 EF adhesive 374.93 kN for the non-treated and 488.78 kN for the treated specimen were obtained. In contrast, the slopes of the curves are very similar.

The specimens showed very brittle fracture behaviour, with the exception of the untreated specimens glued with Sikadur®-31, which showed a short phase of plastic deformation. Hence, the results of the push-out tests clearly show that the epoxy adhesives cannot contribute to a ductile behaviour of the composites, although the force-deflection curves of the 4-point bending were in part pointing into this direction. However, it was found that the glued connections can withstand high loads and that a fully glued connection in a wet-on-wet process is a promising alternative to the use of steel fasteners. Moreover, it could be shown by the push-out test that the glue might be the key factor for a composite with ductile behavior and therefore, adjusting the system with adhesives providing a higher ductility compared to the rather brittle epoxy system could be a promising research direction. However, further studies have to be conducted on the adhesive in order to go towards more ductile behaviour without losing the required load bearing capacity.

4. Conclusion

Sol-gel chemistry can be used to generate a versatile primer to not only establish a hydrophobic surface but also to enhance the compatibility of two rather dissimilar materials in timber-concrete composites. In all mechanical testing setups evaluated, the wood surface treated composites reached higher load bearing capacities compared to the untreated reference composites. A versatility of the priming system for epoxy-based systems can be assumed, since Sikafloor®-156 and Sikadur®-31 EF showed very similar behaviour not only for untreated but also for surface treated composites. Hence, the results indicate that the surface treatments contribute to a

tighter mechanical and chemical interlocking. However, it needs to be stated that all performed tests do not allow for drawing conclusions on the long-term behaviour of the composites. This has to be studied in further investigations to reach a more comprehensive assessment of the feasibility of the primer system under real application conditions. Furthermore, specimens tend to fracture in a brittle manner, and hence the fully glued connections with epoxy resins do not contribute to a ductile behaviour of the composites. An enhanced plastic deformation of the composites would be highly beneficial in construction applications and hence, further studies have to address alternative adhesive systems, which can add ductility to the system.

Declarations

Author contribution statement

Sanja Kostić: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Sandro Meier: Performed the experiments.

Etienne Cabane: Analyzed and interpreted the data.

Ingo Burgert: Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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