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Small-Scale Test of Transparent Adhesives in Glass Structures under Shear Stress and Their Ageing Resistance

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Abstract: Glass load-bearing structures, which have been popular in recent years, require careful attention to detail, because with improper design there is a concentration of stress leading to failure and subsequent collapse of the structure. Mechanical connections are mainly used but there are disadvantages such as stress peaks, weakening of the cross-section, or aesthetic reasons. Adhesive connections eliminate these disadvantages. The lack of information and standards about adhesives (except for silicones) obstruct their wide-ranging use. Especially problematic is an ageing of adhesive and its influence on the mechanical properties. This study is focused on experimental testing of two transparent epoxy adhesives that were used for small-scale specimens with float glass. Two sets of specimens were prepared for each adhesive. The first set was an unaged reference set, the second set was exposed to an artificial ageing method based on EN ISO 9142. The specimens were subjected to shear stress in a displacement controlled test. One adhesive showed good mechanical properties in the reference set, but resistance to artificial ageing was poor. The second adhesive does not achieve good mechanical properties in the reference set in comparison to the first adhesive. The artificial ageing method was devastating for the second adhesive. Poor mechanical properties of the second adhesive could be caused by the age of the uncured adhesive. The first adhesive can only be recommended for the interior where the adhesive is not exposed to aggressive environmental effects.

Keywords: Transparent Adhesive, Glass, Shear Strength, Artificial Ageing, Durability, Epoxy

Introduction

When one says glass, most people think of a window in connection with civil engineering. But glass is also used for other parts of buildings, especially in the last decades. Its strength and other material properties allows use glass for load-bearing structures like canopies, balustrades, facades but also glass beams or columns (Glassonweb, 2016). Nowadays, research on glass is relatively far and there are a few standards for design of glass structures.

Joints of glass structures, both glass-to-glass, glass-to-metal, or other materials, are problematic and represent one of the most critical aspects of glass design. Haldimann *et al.* (2008) define two main types of connections, mechanical and glued. They introduce common types of mechanical supports, their advantages and disadvantages. Bedon and Santarsiero (2018) define four types of glass connections: Clamped, friction-grip, bolted and adhesive connections which include laminated adhesive connections and explain differences, load transfer, etc. Centelles *et al.* (2019) compare the experimental results of the different connections (mechanical, adhesive, laminated) with the influence of different materials, type of test, load duration, pre- and post-breakage performance, failure mode and resistance to artificial ageing. Kassnel-Henneberg (2016) presents important connecting parts of their recent all-glass structures.

The effort to design adhesive connections is evident from the above-cited papers. Not only architects and investors prefer adhesive joints because of their invisibility and keeping the flat surface of the glass, but engineers consider them suitable from a mechanical point of view. Adhesive connections provide uniform stress distribution (according to the stiffness of the adhesive) and hence eliminate stress peaks in glass which are unsuitable and critical for glass. Adhesive connections

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can eliminate vibrations, interrupt thermal bridges, etc.

Adhesives are viscoelastic materials, which means stress-strain relationship is time-dependent. The short- and long-term load-bearing capacity is different. It depends on the chemical composition, molecular structure and molecular free volume. Temperature is another important parameter in relation to mechanical properties of adhesives. The adhesives are stiff and brittle in low temperature and on the other hand, they are more flexible in high temperature (Petrie, 2007).

Regarding the environmental effects, ageing is another complication with adhesives. Many studies in different branches, including civil engineering, investigate the durability of adhesives (Unuk *et al*., 2019; Fevery *et al*., 2021; Nicklisch and Weller, 2016; Aßmus *et al*., 2018; Machalická *et al*., 2019). There is no standard or regulation in civil engineering specifying an exact method of artificial ageing that represents the real years of exposure of the adhesive to the external environment. The ETAG 002 (2012) guideline has a procedure and specifies artificial ageing, the geometry of the specimen, but the standard aimed at structural sealant glazing kits. It cannot be used generally for all kinds of adhesives. The EN ISO 9142 (2003) standard offers more methods and procedures, but it is general and every applicant of the standard can do whatever he wants. The standard does not state the conversion of artificial ageing to real-time exposure. There are other standards or regulations, but there is no general procedure for testing the durability of the adhesive for civil engineering.

Each part of the structure determines the load-bearing capacity of the entire construction. The lack of information and standards prevents the wider use of adhesives for load-bearing structures in civil engineering.

Experimental Details

This study is focused on small-scale experimental testing of adhesively connected glass joints with transparent epoxy adhesive. Small-scale tests were performed to select the appropriate adhesive for the bonding of glass connections. Two epoxy adhesives were tested unaged as a reference set and after one artificial ageing method.

In total, 4 sets of specimens were prepared (2 unaged, 2 aged), each set contained 5 specimens.

Materials

The specimens consist of three float glass panes with dimensions 50 x 50 mm and thickness 19 mm. Float glass with a flat surface was used. The specimens were prepared as double-lap shear joints, see the geometry in Fig. 1.

Two epoxy adhesives were selected, Polycol 117+593 and SPT Epoxy. Polycol is a two-component stiff epoxy adhesive. SPT Epoxy is a two-component impact resistant adhesive. Both adhesives are assumed as resistant to ageing. Glass panes were bonded with 1 mm adhesives.

Artificial Ageing

The EN ISO 9142 standard consists of 12 artificial ageing methods, 4 special methods and one method of artificial ageing with exposure to chemicals. A method of artificial ageing called D2 was chosen. This method has been modified and supplemented for this experiment in order to ensure the effect of weather conditions to which the resulting joint may be exposed.

The D2 method includes heating, cooling (temperature shock) and humidity. The basic cycle consists of 16h ±1h heating at 40°C with 90% RH, shock cooling at - 20°C for 3h ±1h and then heating at 70°C with (50±5) % RH for 5h ±1h; Fig. 2.

This method was supplemented with method E3, which contains exposure to laboratory light source, UV - radiation. An extended cycle included a five-time repeated basic cycle D2 followed by exposition to UV - radiation for two days. The complete artificial ageing method consisted of three time-repeated extended cycle; Fig. 3.

Test Procedure

The test was performed using a Shimadzu test machine. Specimens were placed on the elastic pad, which prevents direct contact between glass and steel and eliminates stress peaks in the glass. The same elastic pad was located on the loaded surface of the middle glass; Fig. 4.

Specimens were exposed to the shear test, which was induced by the compression load on the middle glass. This compression caused shear stress in the adhesive connection. The test was controlled by displacement which was applied continuously until the destruction of the specimen. The speed of the displacement was 0.05 mm/min.

Four linear potentiometers were used to measure the displacement. Two potentiometers measured the displacement of the middle glass and two potentiometers measured the compression of the glass into the pad.

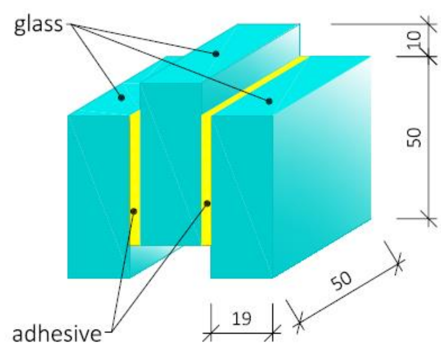

Fig. 1: Specimen geometry in mm

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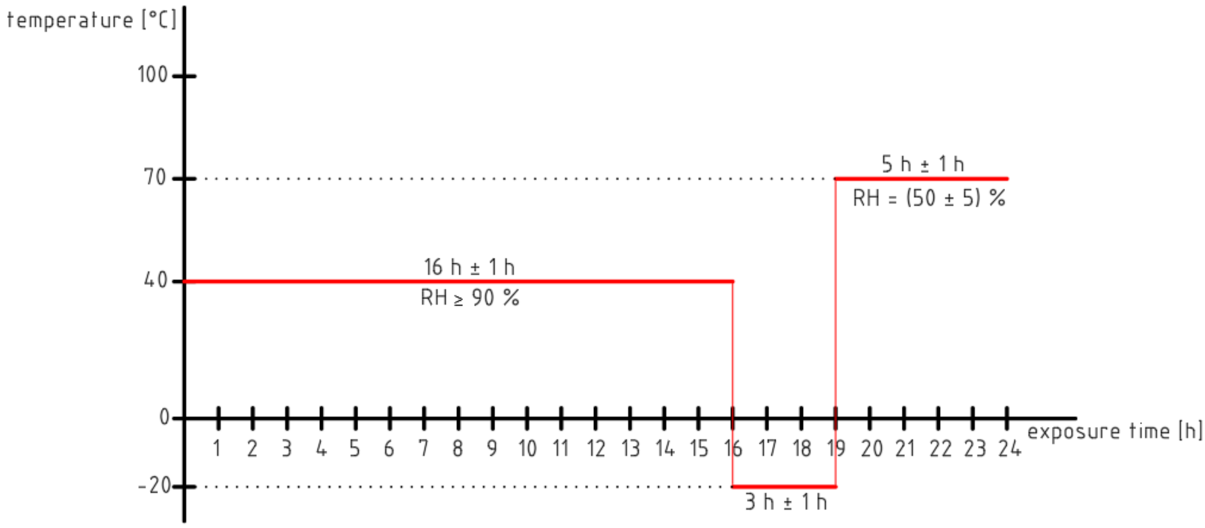

Fig. 2: Basic cycle D2 according to EN ISO 9142

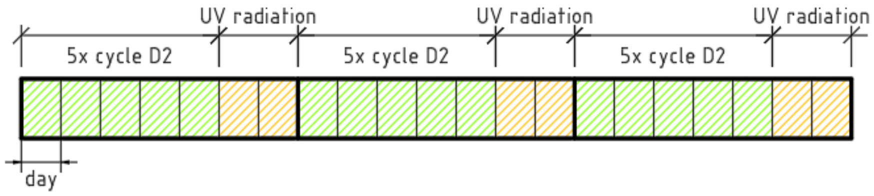

Fig. 3: The ageing method based on EN ISO 9142

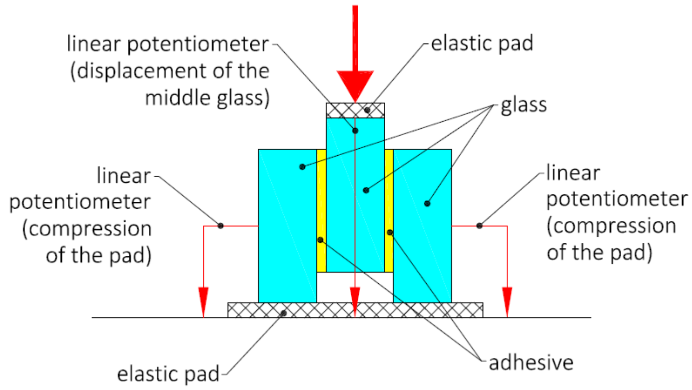

Fig. 4: Schema of the mechanical test

Results and Discussion

Reference Set

SPT Epoxy tested at room temperature showed three times higher shear strength compared to Polycol; Table 1. Specimens with SPT Epoxy adhesive showed relatively low standard deviation, only 0.928 MPa; Fig. 5. Relatively good adhesion to glass was observed. Failure occurred mostly in A-SS mode or S mode; Fig. 6 and 7.

The failure mode of the Polycol adhesive was different for almost every specimen. Generally, it could be said that the predominant failure mode was a combination mode with dominant adhesion failure; Fig. 6 and 8. One specimen with the lowest adhesion failure and dominant substrate failure, Fig. 9, showed much higher (more than twice) shear strength compared to other specimens in the set; Fig. 5. The adhesion of the adhesive to the glass was much higher, which caused higher shear strength of the specimen. The lower adhesion of the Polycol adhesive, for almost all specimens, could be caused by the age of the uncured adhesive. The adhesive was not delivered in the original packaging and the expiration of the adhesive was not specified even when the glue was poured from the original packaging. This process can significantly affect the mechanical properties of the adhesive.

Ageing Method Based on EN ISO 9142

Specimens with Polycol adhesive failed during artificial ageing. All specimens unglued themselves during the first five days of the method (Procedure D2). The specimen after the test is shown in Fig. 10, where the yellowish of the adhesive is obvious.

The set of specimens with SPT Epoxy adhesive had a similar problem, but one specimen got through the artificial ageing successfully, without ungluing. Two of the specimens failed during the D2 procedure, the last two specimens unglued during UV-radiation. Two specimens failed in the first cycle, one failed in the second cycle and one failed in the third cycle of the test. Specimen unglued during the procedure is shown in Fig. 11. Different degree of yellowing was observed.

One specimen, which passed the test, was tested and reached shear strength 0.332 MPa, Table 2, which is only 3.6% of shear strength of the specimens in the reference set. The loss of adhesion was the only mode of failure and the color of the adhesive was dark yellow, Fig. 12.

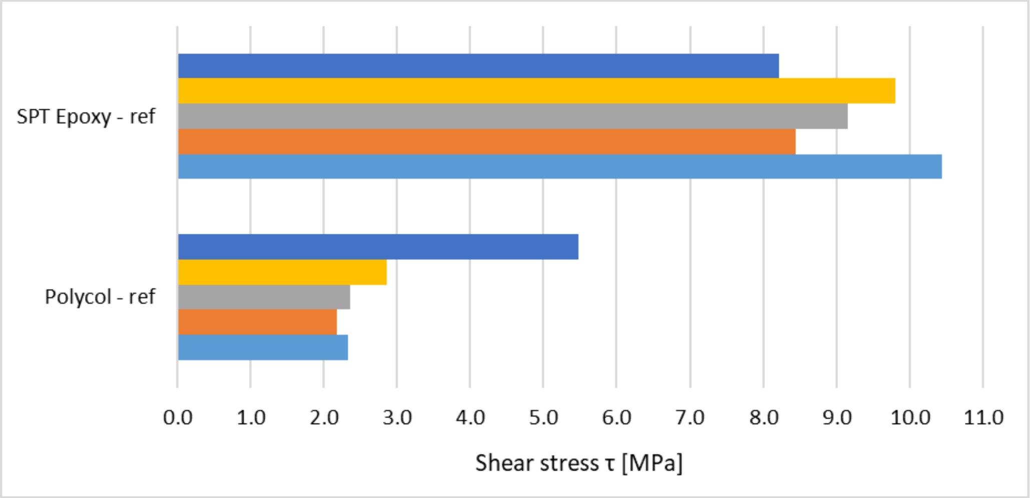

Fig. 5: Shear strength of reference sets of specimens

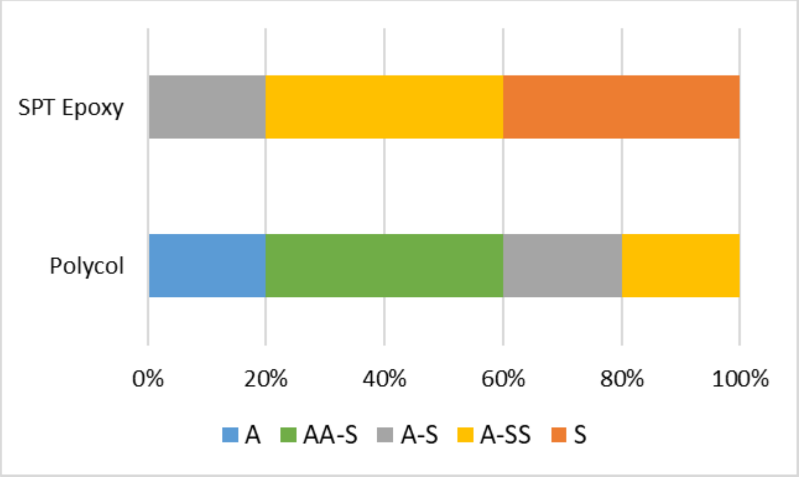

Fig. 6: Failure mode of the specimens in reference set, A-adhesion failure, AA-S – combination mode of failure with dominant adhesion failure, A-S – combination failure mode adhesion-substrate failure, A-SS – combination failure mode with dominant substrate failure, S – substrate failure

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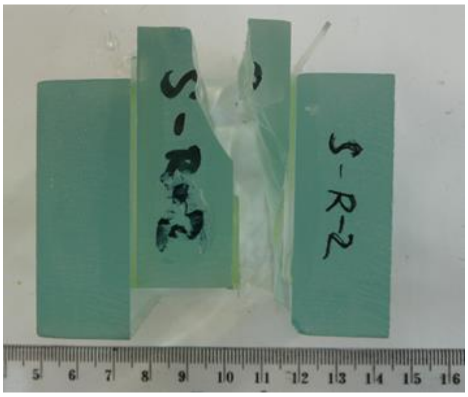

Fig. 7: Specimen with SPT Epoxy adhesive after the test (reference set) - combination mode of specimen's failure with dominant substrate failure

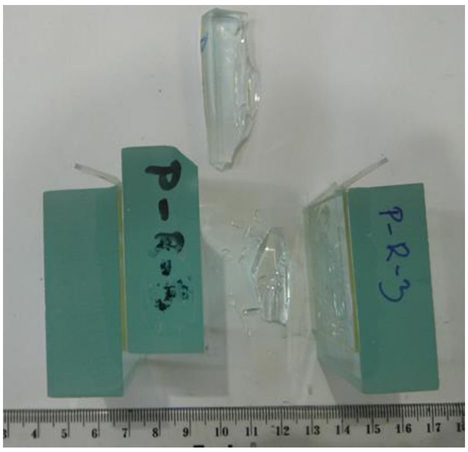

Fig. 8: Specimen with Polycol adhesive after the test (reference set) – combination mode of specimen's failure with dominant adhesion failure

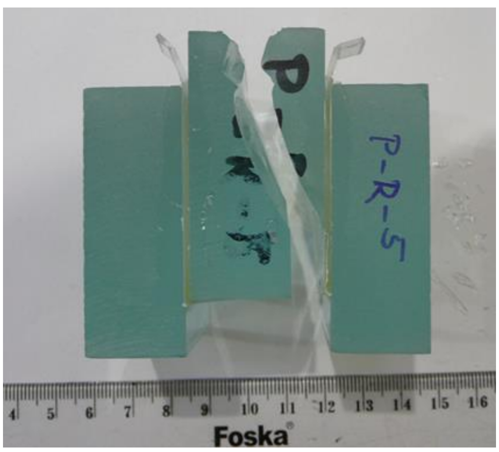

Fig. 9: Specimen with Polycol adhesive after the test (reference set) - combination mode of specimen's failure with dominant substrate failure

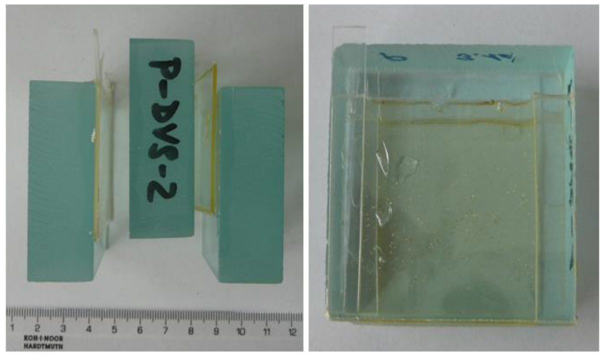

Fig. 10: Specimen with Polycol adhesive after artificial ageing method based on EN ISO 9142

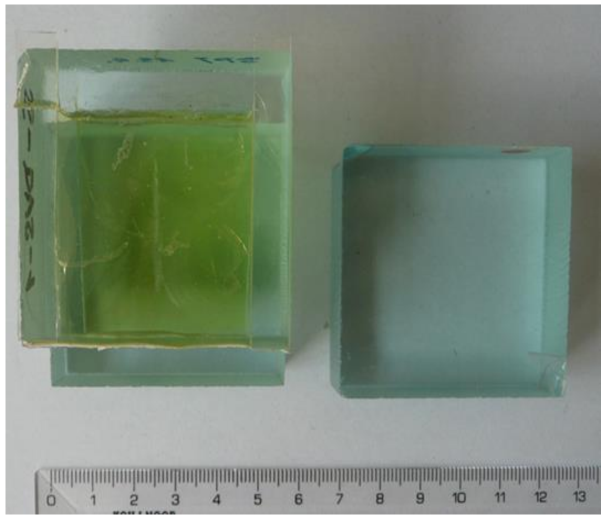

Fig. 11: Specimen with SPT Epoxy adhesive after artificial ageing method based on EN ISO 9142

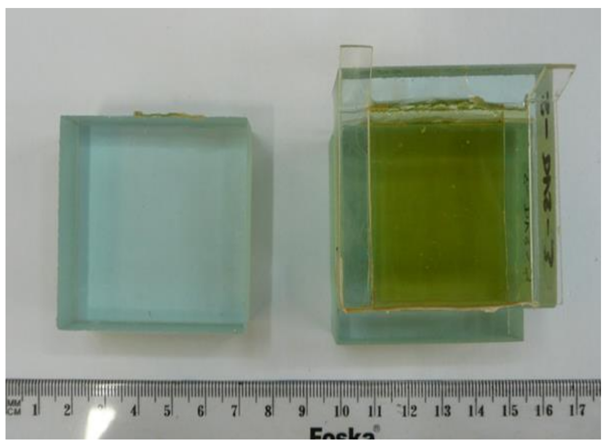

Fig. 12: Aged specimen with SPT epoxy after shear test

Table 1: Reference sets of specimens: average shear strength, standard deviation and mode of failure

Adhesive	Average shear strength	Standard deviation	Mode of failure
	MPa	MPa	-
SPT Epoxy	9.212	0.928	A-**S**[a]
Polycol	3.043	1.387	**A**-S[b]

a. A-**S** – combination failure mode with dominant substrate failure (fracture of glass)
b. **A**-S – combination failure mode with dominant adhesion failure

Table 2: Set of specimens exposed to the artificial ageing method based on EN ISO 9142 – average shear strength, standard deviation and mode of failure

Adhesive	Average shear strength	Standard deviation	Mode of failure
	MPa	MPa	-
SPT Epoxy	0.332	-[b]	A[a]
Polycol	-[b]	-[b]	-[b]

a. A - adhesion failure
b. The results were not obtained during the shear test due to failure of specimens in artificial ageing

Conclusion

SPT Epoxy adhesive showed good mechanical properties in the reference set. Although the adhesion of the adhesive was not perfect, no considerable differences in shear strength were observed between the specimens with lower adhesion and the specimens that failed due to glass fracture.

However, poor mechanical properties were obtained after artificial ageing. Only one specimen managed the artificial ageing method based on EN ISO 9142. The shear strength of the specimen was 0.332 MPa, which is only 3.6% of the reference specimen strength and adhesion failure was observed. Other specimens failed at various stages of artificial ageing. Two specimens failed in the first cycle, one in the second cycle and one in the third cycle. Ungluing was for two specimens during UV- radiation and for two specimens in the climate chamber.

The specimens with Polycol adhesive showed confusing and unsatisfactory results in the reference set. A small adhesion strength to glass was observed. Three specimens failed with adhesion failure or in combination mode with dominant adhesion failure and low shear strength (average strength of these three specimens is 2.293 MPa). One specimen failed in combination mode with adhesion-substrate failure and with a little higher shear strength (2.856 MPa). One specimen failed in combination adhesion-substrate mode with dominant substrate failure and reached the highest shear strength (5.482 MPa). This strength was almost twice higher compared to specimens with dominant adhesion failure.

All specimens with Polycol adhesive failed during the first cycle in the climate chamber using the artificial ageing method based on EN ISO 9142. No differences were observed between the specimens in the set. Yellowing of the adhesive was observed.

The poor mechanical properties of the Polycol adhesive could be caused by the age of the uncured adhesive at the application time.

When we compared both adhesives, SPT Epoxy achieved better mechanical properties. The reference set showed good shear strength and mostly good adhesion to glass. Also, this adhesive achieved better results in the artificial ageing method based on EN ISO 9142 in comparison to the Polycol adhesive. One specimen with SPT Epoxy passed the test and other specimens failed at various stages of the artificial ageing method. Any specimen with Polycol adhesive did not pass the artificial ageing.

Any of the adhesives could not be recommended as a suitable adhesive for bonding in an external environment. SPT Epoxy adhesive showed good mechanical properties for internal application. The Polycol adhesive should be tested again with the fresh adhesive. The influence of different surface treatments (not only degreasing but use primer or plasma treatment) of the glass, especially for Polycol adhesive, could be good to investigate in other research.

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Author’s Contributions

Markéta Zikmundová: Prepared the specimens, performed the experimental test, evaluated the test results, wrote the article.

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Martina Eliášová: Supervision of the research and the ongoing work.

Ethics

This article is original and contains unpublished material. The corresponding author confirms that all authors have read approved this manuscript and there are no conflicts of interest.

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