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ADHESIVE-STRENGTH ASSESSMENT OF LIME INJECTION GROUT USING STANDARDISED AND MODIFIED TEST METHODS

OCENA SPRIJEMNE TRDNOSTI APNENE INJEKCIJSKE MASE S STANDARDNO IN MODIFICIRANO PRESKUSNO METODO

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The adhesive strength of non-structural lime-based grouts used to stabilise sensitive, detached, decorative plasters is an important mechanical property. However, it is difficult to determine it due to the lack of suitable standard test methods. The existing standard procedures are mainly aimed at testing the properties of hydraulic binders and are not suitable for injection grouts or mortar specimens based on a lime binder. In the present study, the focus is on a comparison of the pull-off results between the standardised method (EN 1015-12) performed on pre-drilled specimens on sandwich panels (PMS) and the modified method using sandwich discs (DSS). It was found that the modified method with sandwich discs (DSS) achieved an up to 58 % higher adhesive strength than the standard method with sandwich panels. In the sandwich-panel specimens, fracture occurred in the grout since pre-drilling reduced the cohesive strength of the grout. For the sandwich discs (DSS), fractures occurred predominantly at the interface between the mortar and the grout.

Keywords: lime-based grout, pull-off strength, modified test method, sandwich panel, sandwich disc

Sprijemna trdnost nekonstrukcijskih injekcijskih mešanic, ki so namenjene stabilizaciji občutljivih dekorativnih ometov, je ključna lastnost za učinkovitost utrditvenega ukrepa. Realno ovrednotenje sprijemne trdnosti je še vedno težavna naloga, saj za tovrstne aplikacije ne obstajajo standardne metode. Večinoma so obstoječi standardi namenjeni hidravličnim vezivom in zato niso primerni za injekcijske mase ali malte z apnenim vezivom. V tej študiji je poudarek na primerjavi rezultatov sprijemne trdnosti med standardizirano metodo, ki vključuje vrtanje vzorcev na sendvič ploščah (PMS), in modificirano metodo s sendvič diski (DSS). Izkazalo se je, da z modificirano metodo s sendvič diski (DSS) dosežemo do 58 % višje sprijemne trdnosti kot pri standardni metodi na sendvič ploščah. V primeru sendvič plošč se je zaradi vibracij med vrtanjem vzorcev porušitev pojavila v sami injekcijski masi. Ocenjujemo, da so vibracije poslabšale kohezivne trdnosti injekcijske mase. Porušitev na sendvič diskih (DSS) je bila večinoma na stiku med malto in injekcijsko maso.

Ključne besede: apnena injekcijska masa, odtržna sprijemna trdost, modificirana preskusna metoda, panelni sendvič, sendvič disk

1 INTRODUCTION

Stabilization intervention by grouting is a common method to restore the adhesion between delaminated plaster layers. When the grout is injected behind the plaster to fill voids and cracks, it becomes an irremovable part of the wall and results in an irreversible intervention. Therefore, the physical-mechanical compatibility between the grout and the original plaster is crucial. The requirements for the hardened state of the grout are very often specific in relation to the properties of delaminated plasters: porosity, water vapour permeability and capillary water absorption similar to the original materials; mechanical strength similar to or lower than that of the original plasters; and good adhesion properties.[1]

However, there are still some issues related to a realistic evaluation of the mechanical properties of the lime-based grouts. This applies in particular to the adhesive strength of non-structural pure lime grouts, as there are still no specific international laboratory standard test methods in this area. In a limited number of previous studies, the adhesive strength of grouts was measured predominantly by a pull-off test described in the standard EN 1015-12.[2] However, the specimen preparation differs considerably among different institutions.[1,3] Azeiteiro et al.[3] and Padovnik et al.[1] studied the pull-off strength using a specimen that simulated the detachment between plaster layers that were later injected with grout. This approach, referred to as the "panel sandwich test," was used to simulate detachments of 2 mm to 5 mm between fine plaster and rough plaster. The specimen preparation consists of first applying rough plaster and then fine plaster with purposely fabricated detachments, to a substrate, and cutting/drilling a circular specimen of a specific diameter in the detachment area. After that, gluing a circular metal pull-head plate with the same diameter to the specimen surface is carried out to transfer the increasing tensile force to the specimen until its rupture.

In addition, it was found that there are problems in evaluating the actual adhesion strength using the stan-

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dard test method EN 1015-12, especially when pre-cutting a circular area after the plastered panel has been cured.[5] During cutting, the vibrations of the drilling machine might cause the weak plaster or grout layer to detach or significantly reduce the cohesion of the grout and/or plaster. Another limitation of the pull-off method, when using standard circular pull-head plates with a diameter of 50 mm, is the low adhesion strength of the lime-based grout. As a result, the standard EN 1015-12 procedure shows a low repeatability and accuracy of the test results because the tensile load is often below 10 % of the capacity of the pull-off testing machine.

In the latest study, Pasian et al.[4] developed a sandwich system in which the grout is injected between two plaster layers without support. The advantages of the new proposal for sample preparation are that such a system does not require drilling a circular area and that the larger load-bearing surface allows for a more realistic adhesion-strength evaluation as well as greater accuracy of the test results. On the other hand, the proposed sample-preparation procedure also has a weakness. The injected sandwich samples are sealed in a non-porous plastic container for 28 d. Thus, CO_2 access required for the carbonation of the lime binder is restricted. In addition, the authors highlighted that the proposed sandwich sample allows evaluating the behaviour of the whole plaster-grout-plaster system in terms of water-vapour permeability and capillary-water absorption.

This study presents a new approach to evaluating the lime-grout adhesive strength based on the preparation and testing of a 100-mm disc-sandwich model. The grout is injected into a deliberately prepared air pocket between the fine and rough plaster layers, and subsequent core drilling is omitted. The obtained test results are compared to those of the panel sandwich test, performed by the standard pull-off method. It is important to note that this study does not provide information about fresh grout properties, which is generally very important in the assessment of the test results. Since the same grout compositions as in earlier studies were used to prepare the samples, fresh grout and mortar properties reported there represent relevant data, if needed.

2 EXPERIMENTAL PART

2.1 Composition and testing of grouts and plasters

Two lime grouts were used to compare the efficiency of the standard EN 1015-12 and a modified test method to evaluate their adhesive strength. The grout compositions are shown in **Table 1**. Commercially available dry hydrated lime of class CL 90-S (EN 459-1)[6] was used as a binder. A finely ground limestone from Slovenia (CALCIT, Stahovica, Slovenia) and thin-walled soda-lime-borosilicate glass microspheres (3M Glass Bubbles K1) were used as fillers. To achieve an adequate viscosity and injectability of the grout in a fresh state, polycarboxylate ether-based superplasticiser (PCE-SP) was used to reduce the water content of the grout (tap water at a temperature of (20 ± 1) °C). The grout mixtures were prepared using a KitchenAid mixer with a power of 300 W and a stainless-steel gate anchor blade. First, the lime and filler were mixed. Then 70 % of the water was added and mixed for 2 min at low revolutions (540 min^{-1}). In the last 15 s of the low-speed mixing, the PCE-SP and 30 % of the water were added. Finally, each grout was mixed at high revolutions (1200 min^{-1}) for 3 min.

For the sandwich samples, rough and fine lime plasters were used (**Table 1**). The rough plaster was prepared from (1 : 3) lime putty : coarse sand (0/4 mm) lime mortar, and the fine plaster from (1 : 2) lime putty : fine sand (0/1 mm) lime mortar, and they were mixed with a RILEM-CEN mortar mixer for 180 s.

The hardened properties of the lime grouts, and the rough and fine lime mortar plasters, were evaluated after 90 d. The fresh mixtures were cast in cylindrical moulds with a diameter and height of 50 mm and demoulded after 48 h. Curing was carried out under controlled ambient conditions (RH (60 ± 10) % and (19 ± 1) °C) until the test day.

The compressive test was performed according to the standard EN 1015–11.[7] The splitting tensile test followed the ASTM C496/C496 M-1 standard.[8] The compressive and splitting tensile strengths were determined on four specimens per composition. The tests were performed with a Roel Amsler HA 100 servo-hydraulic testing machine (Zwick GmbH & Co. KG, Ulm, Germany), complemented by a load cell with the capacity adjusted to the compressive (25 kN) and splitting tensile (5 kN) strengths of the tested specimens.

Table 1: Composition of injection grout mixtures and lime plasters

	Grout A	Grout B	Fine plaster	Rough plaster
Binder – hydrated lime CL70 volume ratio	1	1	–	–
Binder – slaked lime putty[1] volume ratio			1	1
Limestone aggregate 0/4 mm volume ratio	–	–	–	3
Limestone aggregate 0/1 mm volume ratio	–	–	2	
Limestone filler: glass microspheres volume ratio	3 : 0	2 : 1	-	-
Water/binder mass ratio	1.86	1.76	–[2]	–[2]
Water/(binder, limestone filler and glass microspheres) mass ratio	0.41	0.50	–	–
PCE-SP (%)	0.5	0.5	–	–

[1] Slaked lime putty is 2 years old and it contains about 51 % water and 49 % $Ca(OH)_2$

[2] the only water used for the preparation of rough and fine plaster was the water already contained in the slaked lime putty

2.2 Panel sandwich test

The panel-sandwich models were prepared based on the instructions in Ref. 1 and Ref. 3 to simulate a smaller (2 mm) and a larger (5 mm) detachment of the fine plaster from the rough plaster due to air pockets. At the age of 1 year, the air pockets were first prewetted and then grouted using a syringe.

2.3 Modification of the sandwich model

In this study, the preparation of the sandwich system from Ref. 4 was modified. The sandwich-system modification focuses on simplification of the model preparation procedure. The newly proposed sandwich model consists of two discs made of lime mortar, representing two plaster layers.

The discs are cast using cylindrical moulds with an inner diameter of 100 mm and a height of 20 mm. The rough plaster discs are prepared as 20-mm-thick slabs. In the fine plaster discs, however, a cavity of 90 mm in diameter and 2 mm or 5 mm in height is made. Before casting the fine plaster, a plastic disc with a diameter of 90 mm and a height of 2 mm or 5 mm is inserted and attached to the mould base centre. In this way, a fine plaster model with a cavity height of 2 mm or 5 mm and an edge-rim thickness of about 5 mm is prepared. In addition, two small holes are cut in the rim at opposite positions to ensure efficient cavity grouting and air release during grouting (**Figure 1a**). By joining the two 1-year-old discs, detachment of the fine plaster from the rough plaster is simulated (**Figure 1b**).

After 1 year, the rough and the fine discs with the cavity are assembled into a model using 40-mm-wide adhesive tape (**Figure 1c**). Surfaces of the cavity are pre-wetted with a syringe through one of the holes to reduce the absorption of the grout water and grout shrinkage.[9] After pre-wetting, the sandwich model is turned into position with one hole facing the laboratory room ceiling. Through this hole, the lime grout is slowly injected using a syringe. The air pocket is considered filled when the grout flows from the opposite hole, and the grouting is stopped (**Figure 1c**). The adhesive tape is re-

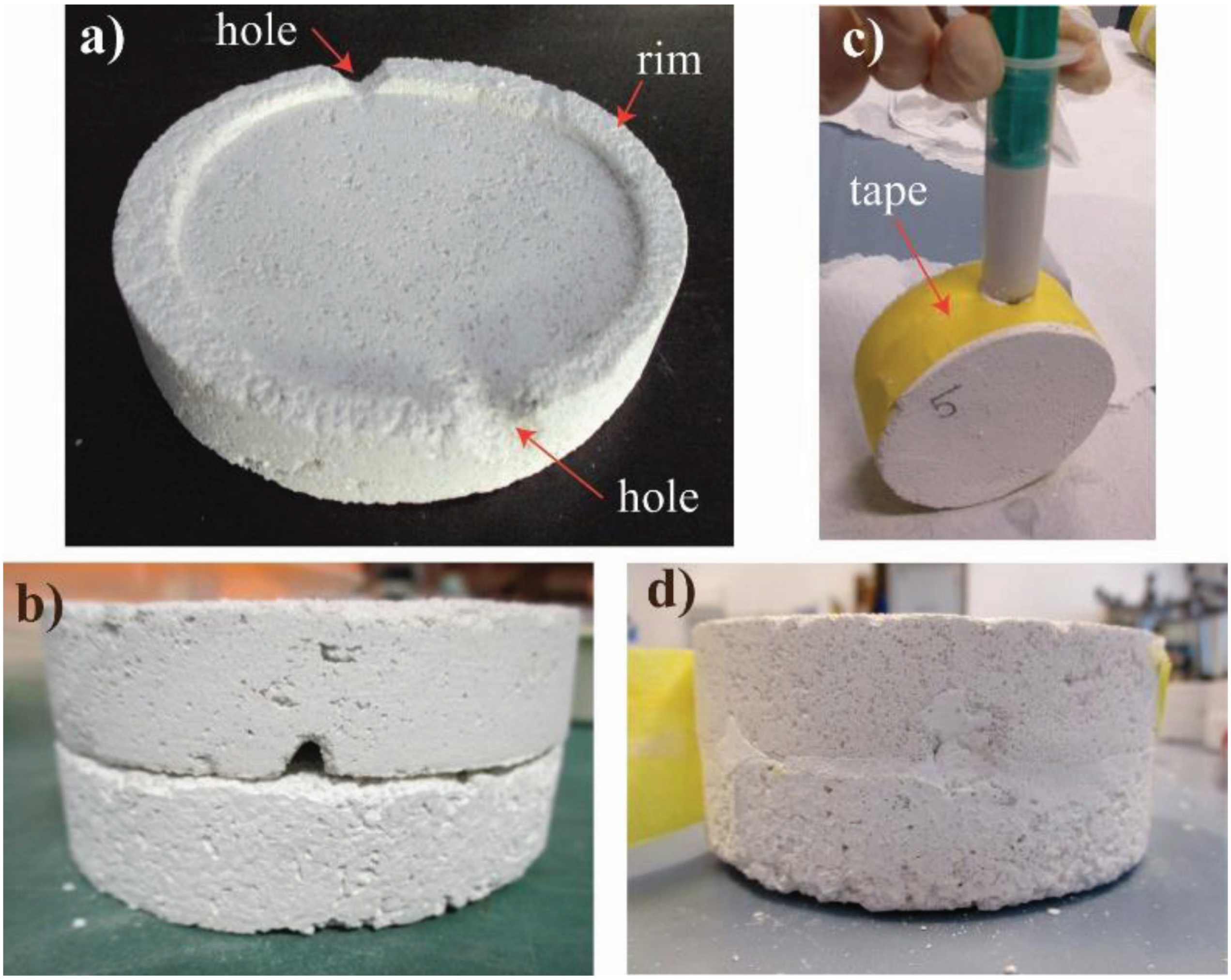

Figure 1: Test specimens: a) a cylindrical cavity disc with a flat bottom and small holes at opposite sides of the rim, b) two discs are joined to the sandwich model, c) grout injection through the hole, d) sandwich system after grout injection

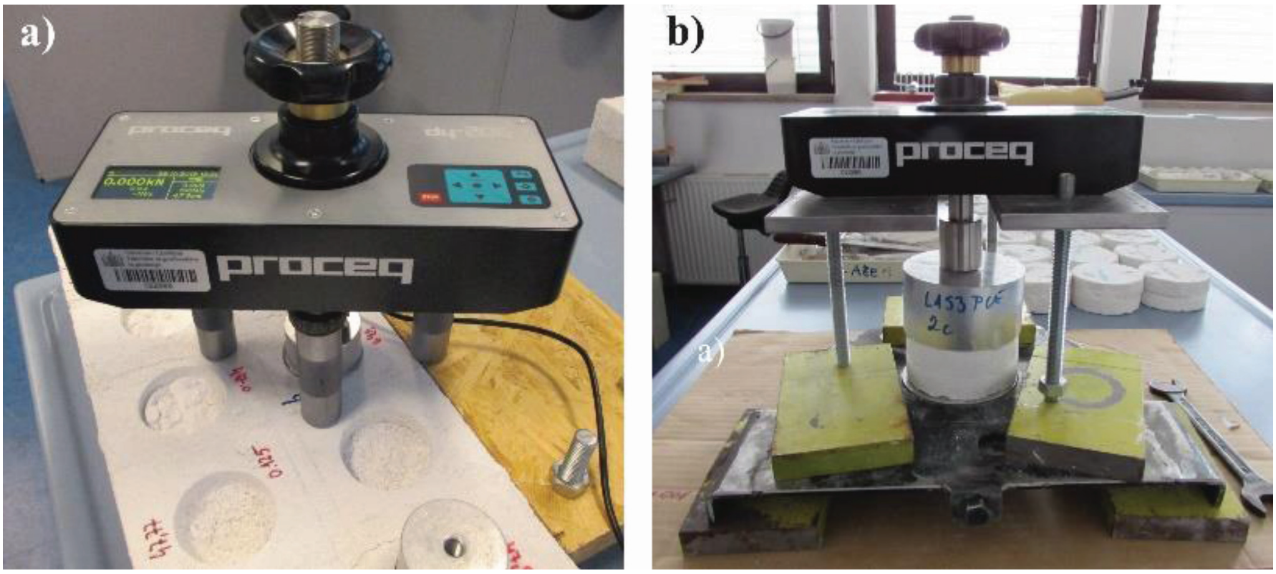

Figure 2: Pull-off test: a) according to the EN 1015-12 for panel-sandwich models (PSM), b) according to the modified standard for disc-sandwich systems (DSS)

moved from the injected sandwich after 24 h (**Figure 1d**).

The modified sandwich model was cured under controlled ambient conditions (RH (60 ± 10) % and (19 ± 1) °C) for 90 d. The same curing regime was adopted for the panel sandwich model.

2.4 Adhesive strength

The adhesive strength of the grout was evaluated on the panel-sandwich models by following the standard EN 1015-12 procedures (**Figure 2a**). Circular test areas of 50 mm in diameter were cut through the grouted plaster layers and 2 mm into the substrate using a core-drilling machine. The metal pull-heads with a diameter of 50 mm were glued centrally on the test areas. The Proceq pull-off tester DY-206 with an operating range of 0.3–3.1 MPa (0.6–6 kN for the 50 mm test disc/pull-head) was used to apply the tensile load to the test surface.

On the modified disc-sandwich model with a diameter of 100 mm, the adhesive strength of the grout was evaluated using the same testing machine (Proceq DY-206 pull-off tester). In Pasian et al. a diameter of 95 mm was proposed for the sandwich specimens. The operating range of the tester was thus shifted from 0.08 MPa to 0.8 MPa, i.e., to an interval where the adhesive strength of the lime grout and mortar values fall. On the test day, two circular aluminium pull-head discs (diameter 100 mm and height 50 mm) are glued to the rough and fine plaster surfaces of the sandwich model using epoxy resin or thermoplastic hot glue. Special care should be taken to ensure that the discs are horizontal and parallel. Prefabricated discs for the sandwich specimen with the same diameter as the pull-head discs eliminate the cutting of the test areas and thus possible damage to the grout-plaster bond or reduced cohesion of the grout and/or plasters. The specimen with the glued pull-heads is placed in a specially designed metal frame, which allows efficient clamping of the lower pull-head and consequent execution of the pull-off test (**Figure 2b**). Due to the larger specimen diameter, the proposed modified test method also provides higher accuracy of the test results compared to the pull-off test procedure in the EN 1015-12.

3 RESULTS AND DISCUSSION

The comparison of adhesive strengths measured using the panel-sandwich test (PSM) and the disc-sandwich system (DSS) with a 2-mm or 5-mm air pocket is shown in **Figure 3**. The type of rupture is presented in **Table 2**.

It can be seen that the adhesive strengths evaluated by DSS are higher than those measured in the PSM. The

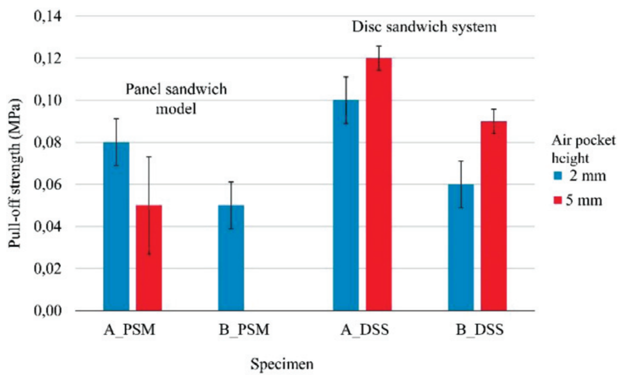

Figure 3: Adhesive strengths of grouts A and B injected into 2-mm or 5-mm air pocket, for the panel-sandwich models (PMS) and disc-sandwich systems (DSS)

Table 2: Location of failure after the pull-off test

Air pocket height/ specimen	2 mm	5 mm
A_PSM	100 % within the grout	90 % within the grout 10 % along the grout–fine plaster interface
B_PSM	70 % within the grout 30 % along the grout–rough plaster interface	90 % within the grout 10 % along the grout–fine plaster interface
A_DSS	90 % along the grout–rough plaster interface 10 % within the rough plaster	40 % within the grout 50 % along the grout–rough plaster interface 10 % within the rough plaster
B_DSS	100 % along the grout–rough plaster interface	20 % along the grout–fine plaster interface 60 % within the grout 20 % along the grout–rough plaster interface

values for the A_PSM and B_PSM grouts injected into the 2-mm or 5-mm air pockets ranged from 0.05–0.08 MPa, i.e., 10–58 % lower than for DSS.

When comparing the results for the 2-mm and 5-mm air pockets, two opposite trends can be observed, depending on the type of sandwich model used. For the PSM, the adhesive strength decreases with increasing thickness of the air pocket, while for the DSS, the strength is higher for the thicker 5-mm air pocket. A plausible explanation for the observed trends is the damage to the grout-render layer interface and/or reduced cohesion of the lime grout and/or mortar related to the core-drilling procedure in the PSM.

According to the average pull-off strengths (**Figure 3**), the difference between the PSM and DSS sandwich models with the 2-mm air pocket is less significant, i.e., 20 % for grout A and 17 % for grout B. The reference strength is that of the DSS specimen. The adhesive strength of grout A in the 2-mm air pocket reached the value of 0.08 MPa and 0.10 MPa for the PSM and the DSS model, respectively. The failure occurred within the grout (100 %) in the PSM. In the DSS, it occurred predominantly along the grout to the rough-plaster interface (90 %). Therefore, the average tensile strength of grout A seems to be higher than 0.10 MPa, since, in the DSS model, the adhesive strength between the grout and the rough plaster was the weakest sandwich layer. Thus, the lower tensile strength of grout A measured for the PSM model can be related to the reduced grout cohesion due to core-drilling damage. However, its value is at the same time well below the operating range of the pull-off tester. Grout B injected into the 2-mm air pocket of the PSM and DSS models showed lower average pull-off strengths of 0.05 MPa and 0.06 MPa, respectively. For the PSM model, the rupture occurred predominantly in the grout (70 %) and along the interface between the grout and the rough plaster (30 %), while for the DSS model, the failure was in the interface (100 %). Thus, the cohesion of grout B can be estimated as higher than 0.06 MPa, and reduced cohesion or interface damage is responsible for the lower pull-off strength of the PSM. These results show that the interface between the lime plaster and grouts A or B is the weakest layer in the disc-sandwich specimens (DSS) with a 2-mm air pocket. The tensile rupture of the grouted layer in the panel-sandwich specimens (PSM) is caused by a reduced cohesion of the grout due to core drilling. The same trend was observed in Faria et al., where the authors showed that cutting specimens by core drilling harms the adhesive strength of the grout.

For the 5-mm air pocket, the differences between the average pull-off strengths of the panel and the disc-sandwich models are higher than for the 2-mm air pocket. A difference of 58 % was obtained for grout A. On the other hand, the pull-off test was not performed for grout B and the PSM model, because the specimens failed during the drilling process due to a failure within the grout and the grout to fine plaster interface. The rupture of the A_PSM specimens occurred predominantly in the grout (90 %) and partly through the interface between the grout and the fine plaster layer (10 %), resulting in a pull-off strength of 0.05 MPa. In the case of the A_DSS specimens, a considerably higher average pull-off strength was measured (0.12 MPa), and the mixed-fracture mode was partly along the grout (40 %), partly at the interface between the grout and the rough plaster (50 %) and in the rough plaster itself (10 %). These results indicate approximately the same values of cohesive strengths in the grout and rough plaster and the adhesive strength between the grout and the rough plaster. For the B_DSS specimens with the 5-mm air pocket, the average pull-off strength reached 0.09 MPa, with the fracture in the fine plaster layer (20 %), through the grout (60 %), and through the interface between the grout and the rough plaster (20 %). The results indicate that grout B's cohesive strength of 0.09 MPa is probably the weakest link in the B_DSS specimens.

A relatively large, injected area in the disc-sandwich system also allows the effectiveness of the grouting process to be evaluated by the presence of voids not occupied by the grout and by the presence of drying cracks formed in the grout. These features were visible on the specimens after the pull-off test. It was observed that the grout did not completely fill the air pocket due to the formation of air bubbles during the injection process. Despite their good working properties, partial filling of the air pockets was observed for both grouts A and B.

Comparing the results in **Figure 3** with the pull-of strengths reported in comparable studies[3,4] shows that the values obtained in this study are higher than those in the referred studies. In Ref. 4 the 150-d pull-of strengths of the lime-grout-injected disc sandwiches were 0.032–0.041 MPa when injected into an 8-mm air pocket. These results are similar to the pull-off strengths of specimens A_PSM and B_PSM in the present study. On the other hand, specimens A_DSS and B_DSS that are more comparable to those in Ref. 4. provided much higher adhesive strengths for comparable grout compositions. Moreover, the maximum pull-off strength in Ref. 3 after 28 d and 60 d was 0.015 MPa. The sandwich model used is similar to the PSM system in our study, but the lime-based grouts contained 10–30 % of metakaolin.

The values of different studies cannot be directly compared with each other, as parameters, such as grout and plaster composition, air-pocket shape and thickness, and curing time and conditions, can influence the test results.[4]

Table 3: Average compressive strength and splitting tensile strength at 90 d

Specimen	Average compressive strength (MPa)	Average splitting tensile strength (MPa)
A	3.5 ± 0.3	0.16 ± 0.04
B	2.6 ± 0.2	0.18 ± 0.04
Fine plaster	2.2 ± 0.1	0.18 ± 0.01
Rough plaster	1.3 ± 0.1	0.13 ± 0.01

The average mechanical strengths of grouts A and B after 90 d are shown in **Table 3**, along with the corresponding standard deviation. The compressive strengths of grouts A and B were 3.5 MPa, and 2.6 MPa, respectively. The splitting tensile strengths were 0.16 MPa and 0.18 MPa for grouts A and B, respectively, which is close to the same strength of the lime mortars for the fine and rough plasters (**Table 3**). The reported results differ from those in Ref. 1 where the same compositions were used; the measured values are, as a rule, higher. The mixing procedure was modified for this study and may be responsible for the increased strength properties.

A comparison of the splitting tensile-strength values in **Table 3** with the DSS pull-off strengths in **Figure 3** shows that the weakest link in the A_DSS samples is most likely the interface between the grout and the rough plaster layer, and the rough mortar itself, since the A_DSS average pull-off strength is the same as the rough plaster average splitting tensile strength for the 5-mm air pocket. The grout governs the failure mode and the pull-off strengths for the B_DSS samples with a 5-mm air pocket. Due to its much lower pull-off strength compared to the splitting tensile strength of the grout, the presence of entrapped air pores in the lower-density grout B is a plausible explanation for the measured values. In the 2-mm air-pocket samples, the adhesive strength between the grout and the rough plaster seems to control the pull-off strength, which is considerably lower than the grout-splitting tensile strength of the individual grout. It seems that efficient grouting is more complex for the 2-mm air pocket than for the 5-mm one, and the reduced pull-off strength may be due to the incomplete filling of the detachment model. Lower-density grout B is less efficient in air-pocket filling than grout A.

4 CONCLUSIONS

The study presents a new approach to the evaluation of lime grout's adhesive strength, based on the 100-mm disc-sandwich system (DSS) and pull-off tester, and compares the results of this modified adhesive-strength test with those of the standard EN 1015-12 adhesive-strength test (PSM). The PSM test involves cutting the samples by core drilling. In both methods, the adhesive strength of the lime grout is evaluated by injecting the grout into the air pocket between two plaster layers.

The DSS sample simulates two plaster layers with detachment in terms of the composition of each layer and the detachment size between two layers, which can be easily adjusted to the actual situation on site. Moreover, the DSS model with a diameter of 100 mm makes it possible to evaluate the filling of the space between two plaster layers and the formation of drying cracks after the pull-off test.

In addition, the shape of the upper disc with a 5-mm-thick wall of mortar around the cavity makes it more difficult for CO_2 to access the injected grout in the disc-sandwich model, which reflects the situation on site more closely than the solution in [4].

Increased test area (100 mm in diameter) compared to the standard method (EN 1015-12, 50 mm in diameter) allows for higher measurement accuracy and shifts the working operating range of the pull-off tester to adhesive strengths typical for the grouts and mortars with a predominantly hydrated lime binder.

The core-drilling elimination in the modified DSS resulted in increased pull-off strength values for all the tested combinations compared to the PSM test. The difference was 20 % (grout A) and 17 % (grout B) for the 2-mm air pocket and 58 % for the 5-mm air pocket. These results confirm that the core-drilling procedure is a detrimental influence when testing low-strength lime-based materials.

The tests of the DSS samples also revealed that the 2-mm air pocket represents a more challenging environment for the grout injection than the 5-mm one. The failure mode was predominantly through the interface between the grout and the rough plaster, which indicates poorer adhesive strength of the grout, most probably due to the presence of entrapped air.

Finally, it can be concluded that the modified sandwich model proposed in this study is a better solution than the EN 1015-12 test method when the lime-based grout's adhesive strength is evaluated. However, a more extensive testing campaign with different grout and mortar compositions is needed for the future.

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