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A 'Sandwich' Specimen Preparation and Testing Procedure for the Evaluation of Non-Structural Injection Grouts for the Re-Adhesion of Historic Plasters

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ABSTRACT
Non-structural injection grouting aims to stabilise delaminated plaster by introducing in the void, typically between delaminated plaster layers, a compatible adhesive material with bulking properties (the injection grout). Injection grout formulations are firstly tested in the laboratory to determine their performance characteristics including their physical-mechanical compatibility with the original materials (plaster or other building materials). However, laboratory tests on grouts are often not sufficiently representative of actual cases in which the grout sets between two plaster layers. This problem is particularly significant when water vapour permeability, capillary water absorption and adhesion need to be assessed. A study was conducted on the development of a 'sandwich' system (plaster-grout-plaster) to better simulate the real situation. This paper describes a new methodology for such specimen preparation and the adapted procedures for laboratory testing. The results are discussed comparing them with those obtained from standard specimen preparation and testing procedure.

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'Sandwich' system; injection grout; adhesion; capillary water absorption; water vapour permeability; plaster

1. Introduction

Wall paintings and historic plasters are complex, multi-layered porous systems which often suffer from failure of adhesion between the different layers. Injection grouting aims to stabilise the delamination introducing a compatible adhesive material with bulking properties (Griffin 2004; Rickerby et al. 2010). Although among the general performance criteria for an intervention retreatability is ideally required (Cather 2006), grouting is essentially not re-treatable and is therefore irreversible, since when set injection grouts become a non-extractable part of the wall (Rickerby et al. 2010). For this reason, physical-mechanical compatibility of the set grout with the original plaster(s) is crucial. Non-structural injection grouts should meet the following criteria: minimal grout volume change on setting; porosity and water vapour permeability similar to or higher than those of the original materials; mechanical strength similar to or lower than that of the original materials; good adhesive properties (Griffin 2004).

There are no specific international laboratory standards for non-structural injection grouts testing, although research on this has been carried out in this field by different institutes (among them Azeiteiro et al. 2014; Biçer-Şimşir and Rainer 2013; Padovnik et al. 2016; Papayianni and Pachta 2015; Pasian et al. 2016a; Pasian, Piqué, and Jornet 2017). Properties of hardened grouts and plasters are typically tested separately following the same standard procedures; in both cases, the specimens set in laboratory condition that are very different from the condition of use on site.

The present research considers and develops a multi-layer system which simulates the actual case in which the grout sets within porous layers. In this kind of specimen, the grout (G) sets between two plaster layers (plaster P1 and plaster P2) (see Figure 1). Testing of this kind of multi-layer specimen is desirable to evaluate the properties of the grout within the plaster layers, giving the opportunity of assessing how the grout behaves in a system in which two interfaces are present (in a plaster1-grout-plaster2 system–P1-G-P2–, interfaces P1-G and G-P2; see Figure 1).[1] When a case study is considered, the plaster of the multi-layer specimen should simulate as far as possible the original plaster for which the grout is designed (even if its ageing obviously cannot be similar to that of the historic plaster). This can help to predict how

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[1]In an actual case, the situation is obviously more complex: more interfaces may be involved, the geometry may be not as regular, and so forth. Moreover, the layers of material to stabilise (in the scheme, generically 'plaster') are not necessarily the same.

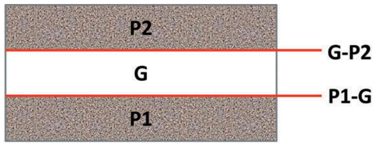

Figure 1. Stratigraphy (plaster P1-grout G-plaster P2) of 'sandwich' specimen in which the grout (G) sets between two plaster layers (plaster P1 and plaster P2). Two interfaces are involved (red lines in the graph): P1-G and G-P2.

the material may behave on site. In this paper, the 'sandwich' system includes a set grout between two slabs of the same plaster; the system, however, is versatile and can potentially include a grout between two layers of different plasters, or between a support (stone, brick, and so forth) and a plaster layer, according to the specific case study under consideration.

In previous studies, a multi-layer specimen prepared in the laboratory was considered to simulate the stabilisation of a delamination with injection grouting and was employed for adhesion tests (Azeiteiro et al. 2014; Padovnik et al. 2016; Pasian, Piqué, and Jornet 2017). Such multi-layer specimens included in stratigraphy support-rough plaster-grout in Pasian, Piqué, and Jornet (2017), while in other studies (Azeiteiro et al. 2014; Padovnik et al. 2016) support-rough plaster-grout-fine plaster. While in Pasian, Piqué, and Jornet (2017) the grout does not set between two layers of plaster, and the specimen is therefore not fully representative, in Azeiteiro et al. (2014) and Padovnik et al. (2016), the grout sets between two plaster layers, but multiple interfaces and heterogeneous materials are involved, potentially giving ambiguous qualitative results while testing adhesion (where the rupture occurs in the specimen).

The aim of this research is to propose a rigorous methodology for testing injection grouts that set between two plaster layers (with the stratigraphic sequence plaster-grout-plaster, which will be called in this paper 'sandwich'). Properties of the grouts are assessed by testing the hardened material separately and in the multi-layer 'sandwich' system. The methodology includes:

- preparation of 'sandwich' specimens to simulate the setting of the grout in a situation similar to the real case of a wall painting or historic plaster (set grout between two plaster layers; such plaster layers are here of the same type to reduce variables);
- adaptation of standard testing procedure for this kind of testing specimen (water vapour permeability, capillary water absorption and adhesion); and
- interpretation of results taking into consideration data collected from the 'sandwich' system and from the single materials composing such multi-layer specimen (isolated grout specimen and isolated plaster specimen).

Compared to previous studies in which a multi-layer system was considered to assess just adhesion (Azeiteiro et al. 2014; Padovnik et al. 2016; Pasian, Piqué, and Jornet 2017), in the present research, the multi-layer sample is developed in order to assess a range of properties of the grout while set within porous plaster. The same 'sandwich' specimen typology can be tested for water vapour permeability, capillary water absorption and adhesion.

It is important to underline that the aims of this research do not include the evaluation of the performance of the different grouts considered. For this reason, although fresh properties of grouts are in general very important to evaluate such materials (Baltazar et al. 2013; Biçer-Şimşir and Rainer 2013; Papayianni and Pachta 2015), working properties of the fluid grouts are not discussed here, because not relevant for the objective of the paper (working properties of such grouts are discussed in Pasian 2017 regarding grouts A and B, and in Pasian et al. 2016a regarding grout C). Properties of the hardened grouts as single materials were tested with traditional specimens in Pasian et al. (2016a) regarding grout C (compressive and tensile strength, porosity, water vapour permeability and capillary water absorption). In Pasian et al. (2018), compressive and tensile strength are reported for grouts A and B, while for such grouts porosity, water vapour permeability and capillary water absorption are reported in Pasian et al. (2016b).

It is also important to highlight that when testing a grout, in addition to the testing of sandwich specimens, it is still necessary to test the grout as a single material with 'traditional' specimens (Padovnik et al. 2016; Papayianni and Pachta 2015; Pasian, Piqué, and Jornet 2017) to determine properties such as compressive, flexural strength and so forth.

2. Materials and methods

In this research, the proposed 'sandwich' system represents the simplest case: set grout between two slabs of the same material, with two interfaces (Figure 1). As the research does not reflect a specific case study, a reference plaster prepared in the laboratory was used as the material between which the grout sets.

Three injection grouts and the reference plaster prepared in the laboratory were considered in the preparation and testing of the 'sandwich' specimen. Water vapour permeability and capillary water absorption were tested separately for both grout and plaster, as well as for the whole corresponding 'sandwich' system; adhesion was evaluated only on the 'sandwich' systems.

2.1. Plaster and injection grout mixtures

The plaster and two of the grouts considered (grout A and grout B) are lime-based, while the third grout (grout C) is hydraulic lime-based. The reference plaster is prepared with slaked lime putty and standard sand (CEN-Standard sand EN 196–1 2016). In Table 1 formulations of injection grout mixtures and of the reference plaster are reported. The grouts were prepared using an electric whisk. Grout A and B were prepared as follows: firstly slaked lime was mixed with 1 pt/V suspension medium for 60 s; then aggregates were added and mixed for other 60 s; finally 0.2 pt/V and 0.7 pt/V suspension medium were added, respectively, to grout A and to grout B, and the grout was mixed for other 60 s. For grout C, binder and aggregate were firstly mixed together (dry) and then the suspension medium was added, mixing with the electric whisk for 180 s. The plaster was prepared using a Hobart mortar mixer N-50 and mixing the paste for 180 s. Grout and plaster specimens were prepared as cylinders with 95 mm diameter and 20 mm height; they were stored at 65–70% RH and 20–23°C.

2.2. 'Sandwich' system preparation

A 'sandwich' system is a multi-material specimen, and because the materials need to adhere to the interfaces, its preparation is more complex compared to the preparation of a grout or a plaster specimen. The set-up proposed simulates the injection of a grout into a delamination between two plaster layers, and it is the following: a cylindrical plastic container (95 mm diameter and 48 mm height) holds the specimen during its preparation (Figure 2(a)); three nails, hammered to a wooden surface, are used to mark the bottom of the plastic container where holes are drilled (Figure 2(b)) to facilitate later the extraction of the specimen from the plastic container. Two pre-prepared plaster slabs (95 mm diameter and 20 mm height) are pre-wetted with 30 mL 25% water: 75% ethyl alcohol solution, spread with a syringe on the plaster surface to reduce the absorption of the liquid contained in the grout mix and reduce grout shrinkage (B. Biçer-Şimşir and Rainer 2013); they are then introduced at the two far ends of the plastic container–ensuring that they are parallel one to the other–so that a void (empty space) of 8 mm remains between the two slabs (Figure 2(d)). On the external wall of the container, four holes are drilled along the perimeter of the empty space at intervals of 75 mm circa (Figure 2(d)). The plastic container with the plaster slabs is placed between two bricks, so that the two plaster slabs are in a vertical position (simulating a wall) and the set-up is stable; the grout is injected with a syringe via a catheter, progressively injected from the four holes to ensure homogeneity of the specimen (Figure 3). The entire plaster-grout-plaster system is removed from the plastic container placing the system on the three nails (Figure 2(b)) and gently pushing down the container to extract the 'sandwich' (Figure 4). The plastic container holding the system is non-porous, and CO_2 access is therefore restricted. This is not a problem for grout C, as its set is hydraulic (being NHL-based); grouts A and B, instead, are lime-pozzolan-based and their set requires CO_2 (in their set both carbonation and hydraulic lime-pozzolan reactions are involved [Cizer, Van Balen, and Van Gemert 2010]); therefore, it is important to extract the sample from the container. It is advised an overall curing time of at least 150 days at 65–70% RH. Sandwich specimens with lime-based grouts should be removed from the plastic container after 28 days, so that enough time is provided to avoid damage and deformation of the specimen during the removal, but enough time is given also for carbonation to occur outside of the container (with free access of CO_2). Sandwich specimens with hydraulic lime-based grouts can be removed from the plastic container after 28 days. The rest of the curing occurs at the RH conditions indicated, which may promote hydraulic reactions. The resulting 'sandwich' specimen is a cylinder with 95 mm diameter and 48 mm height. The specimens were stored at 65–70% RH and 20–23°C.

The nomenclature used for identifying the grouts, the plaster and the 'sandwich' specimens are shown in Table 2.

Table 1. Formulations of injection grout mixtures and of the reference plaster.

	Reference plaster	Grout A	Grout B	Grout C
Binder:				
Slaked lime putty[a]	1 pt/V*	1 pt/V	1 pt/V	–
Natural hydraulic lime (NHL 2)	–	–	–	1 pt/V
Aggregates:				
Scotchlite K1®	–	3 pt/V	3 pt/V	–
Pumice <90 µm	–	1 pt/V	1 pt/V	–
Quartz sand < 125 µm	–	–	1 pt/V	–
Marble dust < 90 µm	–	–	–	3 pt/V
CEN-standard sand EN 196–1	3 pt/V	–	–	–
Suspension medium:				
Deionised water	—[b]	1.2 pt/V	–	1.5 pt/V
Deionised water 15%: ethanol 85%[c]	–	–	1.7 pt/V	–

* where pt/V = parts by volume
[a] Aged 48 months; it contains ca. 50% water and 50% $Ca(OH)_2$
[b] The only water used for the reference plaster was the water already contained in the slaked lime putty; as the slaked lime putty is 50% water and 50% $Ca(OH)_2$, the water/binder ratio of the plaster is 1.
[c] Such suspension medium has been used on the basis of results obtained in the previous research on water-reduced injection grouts, prepared partially substituting water with ethyl alcohol (Pasian et al. 2018).

2.3. Testing procedures

Three properties–water vapour permeability; capillary water absorption; and adhesion–were tested in this

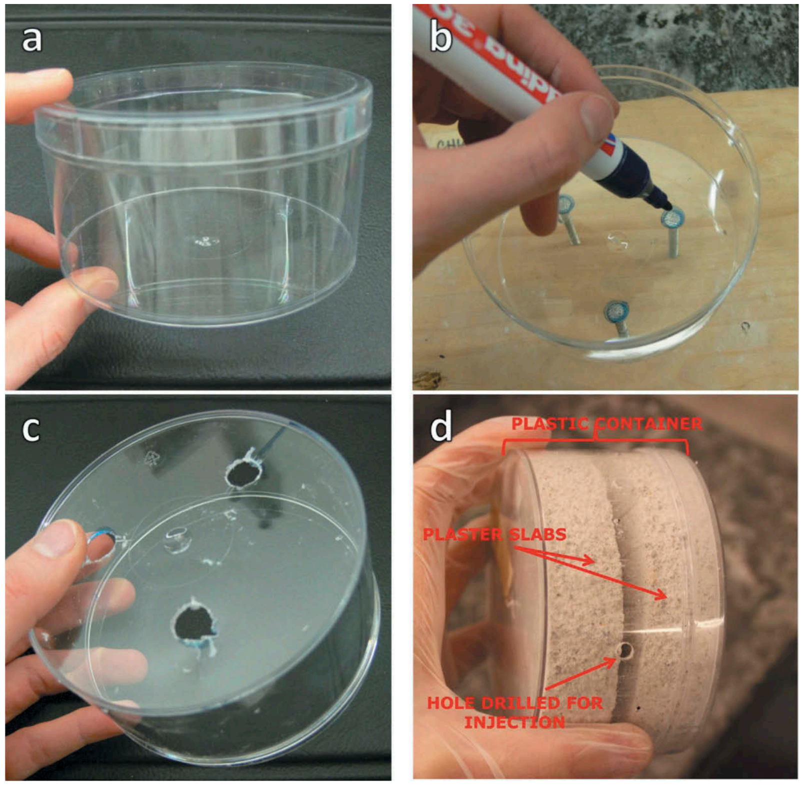

Figure 2. (a) Plastic container for the preparation of a 'sandwich' system specimen. (b) To facilitate extraction of the specimen from the plastic container, three nails allow it to be pushed from the bottom. Marks are drawn on the plastic in proximity of the nails to drill holes. (c) Plastic container with drilled holes for the extraction of the specimen. (d) Set up for the system preparation: two plaster slabs are placed at the two ends of a plaster container; in the middle, holes are drilled to allow injection of the grout between the two plaster slabs.

Figure 3. Injection of the grout between the two plaster slabs through the holes drilled in the plastic container.

research using the 'sandwich' testing specimen described above. These properties are essential to understand the physical-mechanical behaviour of the grout set between plaster layers. Water can move throughout the multi-layer system in both liquid and vapour form; its continuity of movement may be disturbed at interfaces between internal structures. Where liquid flow is disrupted, contaminants such as soluble salts are deposited (Pender 1999). Therefore, ideally, the liquid water absorption–and also the water vapour diffusion–through the set grout and the plaster layers should be continuous without interruptions

Figure 4. 'Sandwich' system composed of plaster 1-grout–plaster 2.

Table 2. Specimen nomenclature.

Nomenclature	Material
P	Reference plaster
A	Grout A
B	Grout B
C	Grout C
S_A	'sandwich' system: plaster-grout A-plaster
S_B	'sandwich' system: plaster-grout B-plaster
S_C	'sandwich' system plaster-grout C-plaster

or delays. Finally, adhesion of the grout to the plaster layers is paramount for a successful grouting intervention.

For each test, plaster (P) and grouts specimens (A, B and C) were firstly tested separately (the plaster after 365 days and the grouts after 150 days from preparation), and then the corresponding 'sandwich' systems (S_A, S_B and S_C, after 150 days from preparation). For each test, at least five specimens of each type were tested; the average values and standard deviations are reported here.

2.3.1. Water vapour permeability

The water vapour permeability test determines the permeability factor (δ) of a material. Permeability is the quantity of water vapour transmitted per time unit through a unit area of the material per unit of vapour pressure difference between its faces for a unit thickness (as per standard EN 12086 2013). The higher the permeability value the easier the water vapour can pass through the specimen.

The standard EN 12086 was adopted for the test. Given that the plaster is the same for all the 'sandwich' system types, its contribution to permeability δ can be expected to be the same for all the 'sandwich' systems. Instead, the contribution of the set grout to permeability δ in the 'sandwich' system changes according to the type of grout present in the specimen.

Permeability is the product of the permeance and the thickness of the test specimen (EN 12086 2013), expressed in mg/[m·h·Pa], where h is the thickness of the specimen. Having tested separately first the permeability δ of the grout and of the plaster, considering the permeability δ of the whole 'sandwich' system, each material (grout and plaster) should contribute half in the 'sandwich' system permeability; it is not necessary to calculate the contribution of the two different materials according to their thickness in the sample. As a result, the permeability of the system could be estimated as the average of the permeability of plaster and grout (for instance in a system S_C: $\delta_{S\_C} = [\delta_P + \delta_C]/2$, where P is plaster and C is grout C). However, this mathematical approach does not fully reflect the complexity of a specimen composed of two materials and two interfaces; variables may be involved in how water vapour passes through a multi-layered material, including the porosity of the set grout (which may differ from the same grout prepared as a separate specimen), the degree of adhesion of the materials at the interfaces (possible empty spaces may lead to a more rapid water vapour passage), and how porosity is interconnected at the interfaces.

2.3.2. Capillary water absorption

The capillary absorption test used (EN 1015-18 2004) determines the amount of water absorbed by the specimen (capillary absorption W, weight to surface ratio), while the coefficient of absorption is calculated as a function of the square root of time (w). The time interval before weighing the specimens was shorter compared to the procedure described in EN 1015–18:2004 in order to follow in detail the initial absorption trend. The perimeter surface of the specimens was not sealed (as EN 1015-18 2004 indicates) in order to follow the capillary front on the side of the specimen, which, as it gets wet, becomes darker in colour. In particular, for the 'sandwich' systems, the capillary front was followed during the test to assess when water was reaching the interfaces. The main aim in testing the 'sandwich' systems was to determine if the water absorption was linear or it would slow down at the interfaces.

2.3.3. Adhesion

Adhesion is the maximum tensile strength with direct load (pull-off) perpendicular to the surface of the tested material applied on support (as described in EN 1015-12 2016); this definition implies just one interface between the material to test and the support. This is the case for instance of repair mortars and plasters; in previous studies their adhesion has been tested with EN 1015–12 (pull-off test), as reported in Isebaert, Van Parys, and Cnudde (2014), Pachta, Marinou, and Stefanidou (2018), Veiga, Velosa, and Magalhães (2009). Adhesion of injection grouts has also been tested in samples with just one interface (system support-grout; Pasian, Piqué, and Jornet 2017); however, this is not representative of a real situation. In other studies (Azeiteiro et al. 2014; Padovnik et al. 2016), grouts adhesion was tested on a multi-layer specimen. In Azeiteiro et al. (2014) the system consists of a brick support, a spatter dash layer and two layers of plaster with delamination in between, where the grout is injected (five interfaces involved). In Padovnik et al. (2016) a 'panel sandwich test' was developed: an aerated concrete panel is used as a support and detachments with a different thickness (to fill with the grout) are created between the rough and the fine plaster (four interfaces involved). In both Azeiteiro et al. (2014) and Padovnik et al. (2016) the load is applied just in one direction according to standard EN 1015–12. Such settings may have few disadvantages: they involve multiple interfaces and heterogeneous materials, and testing requires drilling (as per standard EN 1015–12) which provides vibrations and can, therefore, influence the adhesion at the various interfaces.

In the present research, standard DIN 1048–2 (1991) was applied instead. Such test is typically performed to assess the cohesion of a specimen composed of just one material; here it was performed on the 'sandwich' specimen to assess the adhesion of the grout to the plaster. The standard takes into consideration specimens having not less than 50 mm diameter, to which steel platens are glued. In this paper, as seen, specimens of 95 mm diameter are considered; platens in aluminium (95 mm diameter and 20 mm height, having the same diameter of the 'sandwich' systems) were used, being lighter than steel platens, and therefore allowing to handle the system specimen-glued platens more easily. The platens have a central part–perpendicular to the main, circular body of the platen–on which a borehole is present (see Figure 5(a)); the borehole is used to fix the platen with a tie rod to the tensile testing machine, as shown in Figure 5(b). The two platens are uniformly glued with epoxy resin on the two plaster slabs of the system, so that the two perpendicular central parts of the platens are rotated by 90° with respect to each other (see Figure 5(b)); this allows a good tension distribution which minimises potential errors through sheer force during the test. In addition to this, particular care needs to be taken during the sandwich sample preparation so that the two plaster slabs are parallel one to the other.

After the described set up was prepared, the specimen underwent an uni-axial tensile force in the two opposite directions until failure (see Figure 6).

As seen, just two interfaces are present in the sample; two additional interfaces are present in the test set-up, i.e. the two interfaces plaster-platen (see Figure 6). Compared to standard EN 1015–12, the proposed method does not require drilling. In addition to this, the testing area in the proposed method–and therefore the load-bearing surface of the specimen–is bigger (95 mm diameter) compared to the testing area in EN 1015–12 (50 mm diameter), and thus determines a potentially higher accuracy of the result compared to the pull-off test.

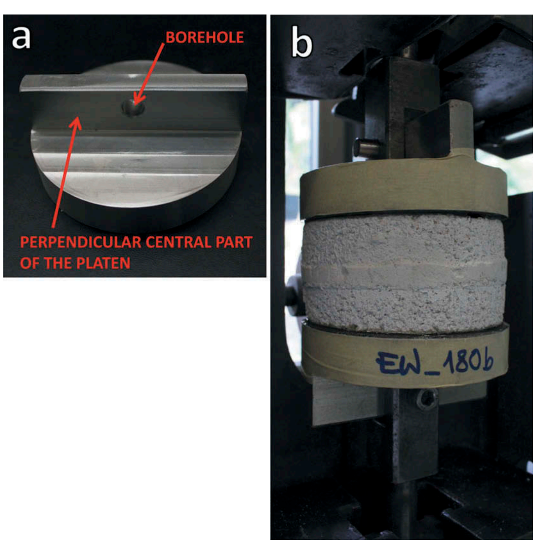

Figure 5. (a) Aluminium platen, 95 mm diameter. A borehole is present on the perpendicular central part of the platen; the borehole is used to fix the platen with a tie rod to the tensile testing machine. (b) Sandwich specimen fixed to the tensile testing machine and ready to be tested; the two platens are glued, respectively, on the two ends of the specimen so that the two perpendicular central parts of the platens are rotated by 90° with respect to each other. The platen is fixed with tie rods to the tensile testing machine.

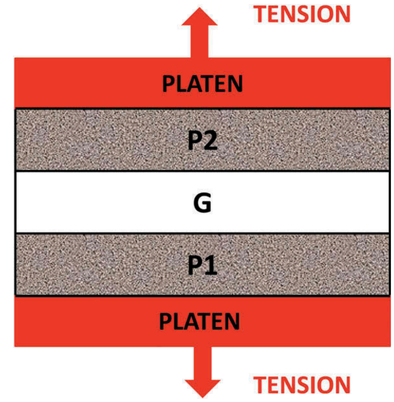

Figure 6. Diagram of a 'sandwich' system, where P1 and P2 are plaster slabs, and G is the set grout; uni-axial load perpendicular to the system is applied in the two opposite directions. Adhesion is measured as the maximum load before failure.

3. Results and discussion

In the following sections, results are presented and discussed separately for each test. Porosity of the different materials has an influence on properties such as water vapour permeability, capillary water absorption and mechanical strength. The porosity of the plaster P, grout A and grout B is reported in Pasian et al. (2016b): the plaster has a total porosity of 14.92%, of which capillary porosity 12.28% and air pores amount 2.64%; grout A has a total porosity of 43.07%, of which capillary porosity 38.61% and air pores amount 4.46% (of which air pores >120 μm 0.39%); grout B has a total porosity of 43.68%, of which capillary porosity 37.32% and air pores amount 6.36% (of which air pores >120 μm 3.99%). The porosity of grout C is reported in Pasian et al. (2016a) and is total porosity 44.24%, of which capillary porosity 41.50% and air pores amount of 2.74%.

3.1. Water vapour permeability

Results of water vapour permeability are given in Table 3; permeability δ of plaster P and of the separate grouts A, B and C are reported, as well as the permeability δ of the 'sandwich' systems. In Table 3, in addition to δ measured for the 'sandwich' systems, also the expected δ is reported (average of the plaster and grout values).

The fact grout A, B and C have a different water vapour permeability is related to their different porosities (reported in Pasian et al. 2016a, 2016b); grout B has a higher water vapour permeability compared to the other two grouts because it has a higher percentage of air pores, in particular of air pores >120 μm. Similar materials such as lime-based

Table 3. Water vapour permeability results.

Specimen	Measured δ mg/(m·h·Pa)	*Standard deviation*	Expected δ mg/(m·h·Pa)	Difference between measured and expected δ
Plaster P	0.0374	0.0033	–	–
Grout A	0.0566	0.0035	–	–
Grout B	0.0756	0.0034	–	–
Grout C	0.0487	0.0045	–	–
S_A	0.0476	0.0032	0.0470	1.2%
S_B	0.0552	0.0031	0.0565	2.3%
S_C	0.0450	0.0058	0.0430	4.4%

plasters and lime- and hydraulic lime-based grouts tested in other studies have values in the range of those found in this research (for plasters: Jornet and Romer 2008; Jornet et al. 2012; for grouts: Pasian et al. 2016a; Padovnik et al. 2016; Pasian, Piqué, and Jornet 2017).[2]

Table 3 indicates that for all the sandwich systems the value of permeability is higher than that of the plaster and lower than that of the corresponding grout. All the systems have high permeability; none of the materials seems to impede or visibly slow the water vapour passage down. The difference in permeability among the 'sandwich' systems is linked to the different grouts in the specimen. The system S_B has a higher permeability because grout B has a higher permeability. Specimens S_A and S_C have a comparable permeability (just slightly lower–0.0026 mg/(m·h·Pa)–the one of S_C), although grout C has a lower permeability compared to grout A; this is probably due to the fact that grout C only partially filled the gap in the S_C system (see below in this section and Section 3.3).

The permeability value measured for the 'sandwich' system S_A (plaster–grout A–plaster) differs 1.2% from the estimated permeability value; it differs 2.3% for the 'sandwich' system S_B (plaster–grout B–plaster) and 4.4% for the 'sandwich' system S_C (plaster–grout C–plaster). The measured permeability value of system S_C has a higher standard deviation compared to the values of systems S_A and S_B; also the result of grout C has a higher standard deviation compared to the other two grouts. The high standard deviation probably has an influence on the difference between the measured value of permeability δ for system S_C and the expected one (see Table 3). In addition to this, the value of the measured δ is probably affected by how the grout fills the space between the two plaster layers. If the 8 mm layer between the two plaster slabs is not completely filled by the grout but includes empty spaces, water vapour passage is easier, and δ of the 'sandwich' specimen is higher. This hypothesis was verified for S_C specimens during the adhesion test (see Section 3.3). It was observed that the space between the two plaster slabs was not completely filled by grout C, and empty spaces in the centre of the specimen were left. This was due to the poor injectability and flow of grout C (Pasian et al. 2016a). On the other hand, grouts A and B completely filled the layer in between the plaster layers, having good injectability and flow (Pasian 2017).

Overall, there is a difference between the measured and the expected δ of the 'sandwich' systems; however, this difference is within 2.3% for grouts completely filling the gap between the two plaster layers in the specimen. Furthermore, one needs to consider that specimens with the same composition can have slightly different δ. For plaster and grouts it may depend on a number of factors, for instance slightly different porosities; porosity is never exactly the same even if the specimens are composed of the same material and are from the same batch. Water vapour permeability is not influenced only by the percentage of porosity, but also by pore connectivity and tortuosity (Pender 1999), which can be different in each specimen. In general, if the permeability of the grout is much lower compared to that of the historic plaster, the material is not suitable (Griffin 2004). If the permeability of the grout is similar to or higher than the permeability of the plaster (Griffin 2004) and the 'sandwich' system is tested, the permeability value δ of the 'sandwich' is supposed to be close to the average of δ of the separate materials (plaster and grout). If the value of the 'sandwich' system is substantially different from that of the estimated value, this may be an indication that the materials have poor adhesion at the interfaces or that the grout does not completely fill the void in between the plaster layers. Testing a 'sandwich' specimen allows to verify that the system plaster-grout-plaster as a whole retains high permeability (typical of historic plasters), highly desirable for the stabilisation of plasters through injection grouting.

3.2. Capillary water absorption

Results of the capillary water absorption test for plaster and grouts at 60 s and 24 h from the beginning of the test are reported in Table 4. For the 'sandwich' systems, values of absorption W and coefficient of absorption w are also

[2]In such references cited, the results are expressed in water vapour resistance (μ) instead of water vapour permeability (δ). For easier reference, the water vapour resistance values of the materials tested in the present research are also reported here: plaster P has $\mu = 17$; grout A has $\mu = 10.33$, grout B has $\mu = 9$, grout C has $\mu = 15$.

Table 4. Capillary water absorption results.

Specimen	W_{60s}[Kg/m^2]	Standard deviation	w_{60s}[Kg/(m^2·√h)]	Standard deviation	W_{24h}[Kg/m^2]	Standard deviation	w_{24h}[Kg/(m^2·√h)]	Standard deviation
Plaster P	1.83	0.10	13.96	0.10	8.02	0.13	1.64	0.13
Grout A	3.54	0.17	27.40	0.17	14.40	0.10	2.94	0.10
Grout B	3.21	0.12	24.90	0.12	12.94	0.18	2.69	0.18
Grout C	4.25	0.11	28.41	0.11	18.48	0.11	3.77	0.11
S_A	1.79	0.15	12.85	0.15	8.72	0.17	1.78	0.17
S_B	1.85	0.18	13.90	0.18	8.57	0.19	1.75	0.19
S_C	1.91	0.14	14.79	0.14	9.30	0.16	1.88	0.16

reported; interesting for such multi-layer specimens are the graphs showing the absorption trend (Figure 7).

Plaster P has a lower absorption *W* and coefficient of absorption *w* compared to the grouts, both at 60 s and 24 h. This is related to the fact that plaster *P* is less porous compared to the grouts. Grouts A and B have a comparable absorption *W* at 60 s–having a comparable capillary porosity–, while grout A has a higher absorption *W* at 24 h. Grout A also absorbs faster than grout B (higher coefficient of absorption *w* at 60 s; comparable *w* at 24 h), because it has a lower amount of air pores, in particular of air pores >120 μm, compared to grout B. Grout C absorbs more and faster than grouts A and B (higher values for both *W* and *w*), having a high capillary porosity, and a lower amount of air pores compared to grouts A and B. Similar materials such as lime-based plasters and lime- and hydraulic lime-based grouts tested in other studies have values in the range of those found in this research (for plasters: Jornet and Romer 2008; for grouts: Pasian et al. 2016a; Padovnik et al. 2016; Pasian, Piqué, and Jornet 2017).

The 'sandwich' systems have values of absorption *W* and coefficient of absorption *w* at 60 s comparable to those of the plaster; at 24 h their *W* and *w* are slightly higher than those of the plaster and lower than those of the corresponding grouts.

For the 'sandwich' systems, graphs of capillary absorption (Figure 7) illustrate the absorption trend of the specimen; they show whether the absorption is linear, if it speeds up or slows down at the interfaces (when water encounters a different material). In order to better visualise it, in the graphs (Figure 7) the absorption line is marked differently according to the material the water passes through (blue for the plaster, red for the grout).

The 'sandwich' system with grout A (S_A) has a capillary absorption which is quite linear. The capillary absorption graph (Figure 7(a)) shows a slight acceleration in absorption at interface 1 (there is a change in the slope of the second segment line–red–referring to grout A). Overall, though, the absorption appears linear and is not visibly altered when the water passes through the different layers. The same can be said for the 'sandwich' system with grout B (S_B) where the capillary absorption curve is quite linear. Observing the capillary absorption graph (Figure 7(b)) and the slopes of the segment lines, a slight acceleration in absorption can be seen at both interfaces. Overall, however, the absorption again appears linear and is not visibly modified when the water passes through different materials. A different case is the 'sandwich' system with grout C (S_C); the capillary absorption graph (Figure 7(c)) shows how the slope of the second segment line–red–referring to grout C is much flatter compared to the slopes of the segment line referring to the plaster slabs–blue. This indicates that at the interface P1-C there is a strong deceleration in absorption, which then speeds up again at the second interface C-P2, when the water encounters the plaster. In this case, the water absorption is modified at both interfaces of plaster-grout. In the adhesion test (Section 3.3) it was verified that 'sandwich' systems S_C was not completely filled with grout C; empty spaces were left in the middle. This is likely the cause of the strong reduction in the speed of absorption at the first interface.

Testing the capillary absorption of a 'sandwich' specimen allows to verify that in the system plaster-grout-plaster the water absorption is linear, and it is not slowed down at the interfaces; a potential accumulation of water at the interfaces (potentially occurring if the grout is poorly permeable to liquid water, but also if the absorption slows down) may be potentially detrimental for the original materials.

3.3. Adhesion

After performing an adhesion test, it is crucial to assess the qualitative nature of the rupture in the specimen, i.e. where the rupture occurs in the system (Padovnik et al. 2016; Pasian, Piqué, and Jornet 2017; Szemerey-Kiss and Török 2017). If the rupture occurs at either of the two interfaces, the numerical datum actually relates to the adhesion of the grout to the reference plaster. If the rupture occurs within the plaster or in the grout, it relates to the internal cohesion of the plaster or of the grout. In addition, if the failure occurs in the grout, it means that the grout cohesion is lower than the bond strength between grout and plaster (adhesion), which is

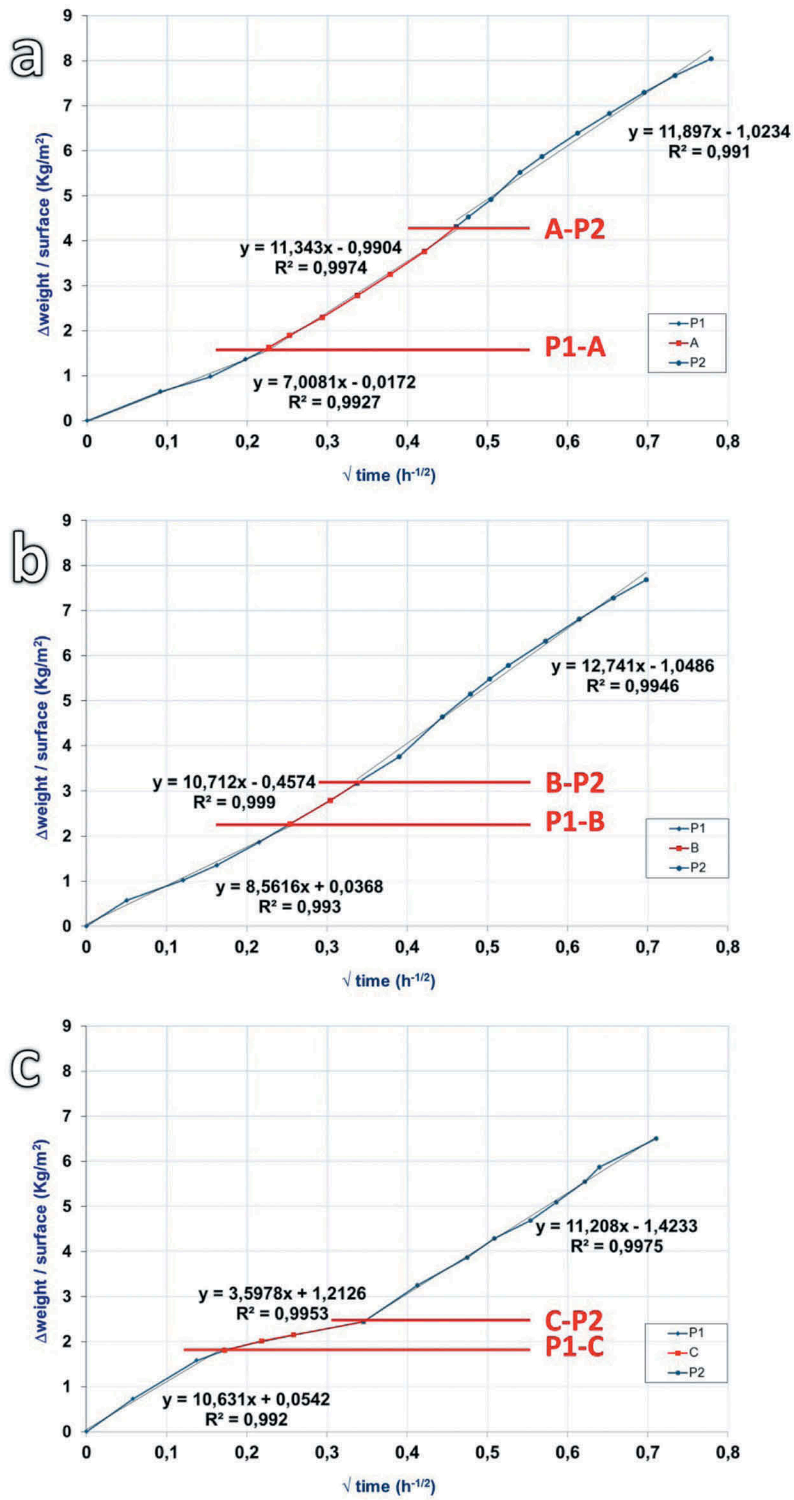

Figure 7. Capillary absorption graph of (a) S_A, where P1-A is the interface plaster1-groutA and A-P2 is the interface groutA-plaster2; (b) S_B, where P1-B is the interface plaster1-groutB and B-P2 is the interface groutB-plaster2; (c) S_C, where P1-C is the interface plaster1-groutC and C-P2 is the interface groutC-plaster2. From the equations in the graphs, the rate of water absorption through the particular layer can be determined.

what may be desirable in the case of non-structural grouts for historic plasters.

Results of the adhesion test and observations are reported in Table 5. Representative specimens are shown in Figure 8 after the adhesion test. Direct tensile strength and compressive strength values of grouts A, B and C as separate samples are also reported in Table 6.

Table 5. Adhesion test results.

Specimen	Direct tensile strength σ_t *[N/mm²]*	*Standard deviation*	*Rupture: observations*
S_A	0.041	0.010	The rupture occurs at the plaster-grout interface in all the specimens tested
S_B	0.032	0.009	The rupture occurs in the middle of the grout in all the specimens tested
S_C	–	–	It was not possible to perform the test; the adhesion was so poor that just the specimen handling was sufficient to break it at the plaster-grout interface

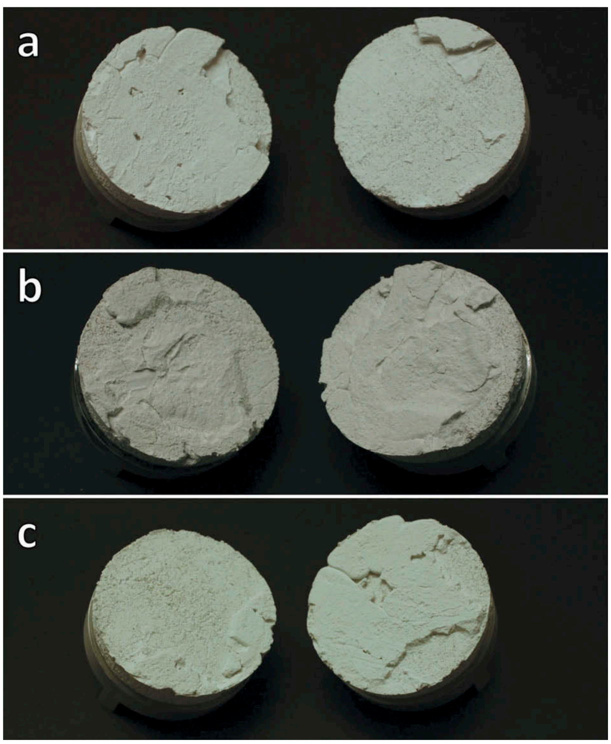

Figure 8. 'Sandwich' system after the adhesion test; (a) specimen S_A: the failure is at the interface between the grout and the plaster; (b) specimen S_B: the failure is in the middle of the grout; (c) specimen S_C: the failure is at the interface between the grout and the plaster; in specimen S_C the grout did not completely fill the gap between the two slabs of plaster.

Table 6. Direct tensile strength and compressive strength of grouts.

Specimen	Direct tensile strength σ_t*[N/mm²]*	*Standard deviation*	Compressive strength σ_c[N/mm²]	*Standard deviation*
A	0.35	0.16	3.07	0.24
B	0.29	0.14	3.08	0.22
C	0.21	0.12	2.72	0.20

The three systems (S_A, S_B, S_C) considered in this study behaved differently in the adhesion test, depending on the grout present (see Table 5). In S_A specimens (plaster–grout A–plaster), the rupture occurred at the plaster–grout interface, and it was flat and relatively homogenous (Figure 8(a)). The plaster-grout bond strength (adhesion) is lower than the cohesive strength of the grout. The value of direct tensile strength for S_A corresponds therefore to adhesion and it is σ_t = 0.041 N/mm². In S_B specimens the rupture occurred in the middle of the grout (Figure 8(b)); this means that the plaster-grout bond strength is higher than the cohesion of the grout, which may be desirable in the stabilisation of wall paintings or historic plasters. The σ_t value is 0.032 N/mm² and refers therefore to grout B direct tensile strength in the sandwich system. It is interesting to notice that the direct tensile strength of grout B tested as a single material is 0.29 N/mm², i.e. ca. 9 times more than the value obtained for the sandwich system. Such results show that injection grouts tested in a multi-layer system potentially show a different mechanical strength (in this case much lower) compared to when tested as single materials. This may be due to a number of factors, including sample preparation (single material vs. 'sandwich'), which in turn may determine grout parameters affecting mechanical strength, including porosity.

It was not possible to perform the adhesion test for S_C specimens, as the adhesion itself was so poor that specimen handling was sufficient to break it at the plaster–grout interface. This is, however, an important qualitative datum; grout C probably does not have a good initial tackiness and therefore it developed no adhesion to the plaster after setting. The poor adhesion may be also related to the grout shrinkage. Furthermore, it was observed in all S_C systems that the space between the two plaster slabs was not completely filled by grout C (Figure 8(c)) due to the poor injectability and flow of grout C (Pasian et al. 2016a).

In Azeiteiro et al. (2014), the values obtained in the adhesion test were much lower (in the range of 0.008–0.015 N/mm²) compared to those obtained in the present study (0.032 and 0.041 N/mm²) and in Padovnik et al. (2016) (in the range of 0.040–0.100 N/mm²); such values in Azeiteiro et al. (2014), though, correspond to weaker grouts (ca. 0.5 N/mm² compressive strength, despite the presence of a pozzolanic material–i.e. metakaolin–in the mixture) compared to the ones considered in Padovnik et al. (2016) and in the present study.

In general, the values obtained in the adhesion test depend on a number of factors involved in such complex system, which include (but are not limited to) the composition of the grout and of the other layers of the system, the structure of the layered system (including the thickness of the void filled by the hardened grout), the ageing time and condition. This is why values found in the different studies cited cannot be directly compared. Nonetheless, it is interesting to note that the result obtained for the grout injected in the 5 mm void of the 'panel sandwich' system in Padovnik et al. (2016) is close to that obtained for specimen S_A of the present study, which has been injected in an 8 mm void (0.040 N/mm^2 in Padovnik et al. 2016 vs. 0.041 N/mm^2 in this study). These two grouts also have a similar compressive strength (respectively, 3.13 N/mm^2 [Padovnik et al. 2016] and 3.07 N/mm^2 in this study). However, while the rupture in Padovnik et al. (2016) is mixed (both in the grout and in the plaster layer–it is not specified which plaster), the rupture in the present study in sample S_A is at the interface plaster-grout and regards therefore adhesion. Since the fracture may occur both in the grout and in the plaster layer, the fact that in the system two different types of plaster are present (as in Azeiteiro et al. 2014; Padovnik et al. 2016) may give ambiguous results. In addition to this, as seen the pull-off test (EN 1015–12) requires drilling, which may lead to vibrations and mechanical stress potentially affecting not just the adhesion of the grout, but also the cohesion of the grout and of the plaster; this may determine a mixed rupture.

4. Final discussion and conclusions

The present research proposes a methodology for testing of injection grouts in their context, i.e. between porous building materials layer, in addition to the testing typically performed on separate grout specimens. This methodology includes: preparation and testing (water vapour permeability, capillary water absorption and adhesion) of 'sandwich' systems (plaster-grout-plaster specimens), and interpretation of results taking into account the multi-material nature of the 'sandwich' specimens.

The test of water vapour permeability (EN 12086 2013) and capillary water absorption (EN 1015-18 2004) on a 'sandwich' specimen allows to assess the behaviour of the whole plaster-grout-plaster system towards water in its vapour and liquid form. This is fundamental in highly porous historic building materials (very often contaminated with soluble salts), where physical compatibility involving capillary absorption and drying behaviour should be ensured (TC 203-RHM (Main author: Groot C.) 2012) when a conservation material is added. To the best of the authors' knowledge, no testing was published so far regarding water vapour permeability and capillary water absorption in plaster-grout-plaster 'sandwich' systems.

Adhesion is of paramount importance for injection grouts, and the present research tries to improve its testing, proposing a system which reduces the number of materials and of interfaces in the specimen, therefore decreasing the potential ambiguity of the qualitative result. Such system does not require core drilling, which may have an influence on the grout adhesion, particularly for the relatively weak lime-based grouts. In addition to this, the 'sandwich' specimen in this research has a larger load-bearing surface (95 mm diameter) compared to the one in the pull-off test (EN 1015–12: 50 mm diameter), and thus leads to a potentially higher accuracy of the result compared to EN 1015–12.

The plaster prepared for the 'sandwich' system may simulate the original plaster to stabilise on site. The present research did not consider a specific case study, and therefore a reference plaster was adopted; when considering a case study, the plaster of the 'sandwich' system should simulate as far as possible the original plaster for which the grout is designed. In the process of designing and testing an injection grout for a case study, the methodology proposed in this paper can help to predict and understand how the material may behave on site, and to refine the designing process in order to obtain a suitable grout with physical and mechanical properties compatible with those of the original plaster.

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No potential conflict of interest was reported by the authors.

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