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Which methods to assess the effectiveness of chemical injections in laboratory?

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Abstract

Resin injection is a widespread technique to fight rising damp in historic brick masonry buildings, but the evaluation of its drying effectiveness is often hard, also due to the lack of standard procedures to test the different damp-proofing materials. Some testing procedures have been proposed in national guidelines and scientific literature to assess the effectiveness of chemical injection in laboratory, but they reflect very different approaches and are hard to compare. This paper firstly provides a critical overview of the presently available methods to assess the effectiveness of chemical injections in brick masonry at the laboratory scale, highlighting their main advantages and drawbacks. Then, two different experimental test walls are proposed, having large and small size, respectively. The test walls were manufactured and subjected to a continuous capillary rise of water, monitoring the moisture amount by a micro-destructive method based on “permanent sampling holes”. Both test walls allowed to effectively reproduce rising damp occurring in real masonry walls and to monitor the moisture amount during time by an easy and reliable method. Large walls involve a long curing time (more than 1 year in laboratory conditions) and are quite space consuming, but they are less affected by the microclimatic variations. Conversely, small walls are more easy to handle, but also more affected by RH changes. The proposed test walls aim at contributing to the future development of a new testing procedure to evaluate chemical damp-proof courses in laboratory.

Keywords: Resin injection; chemical damp-proof courses; standard procedure; chemical barrier; fired-clay bricks; lime mortars; moisture measurement; capillary flow.

1. Introduction

The mitigation of rising damp has been recognized as a key issue for the conservation, repair and maintenance of value of historical masonry buildings [1-2], as damp affects their comfort and

thermal performance, and is a primary cause of materials' deterioration [3].

Although an increasing research effort has been addressed in recent years to the development and testing of solutions to this problem [4], there is still a long route to go towards fully satisfactory repair measures [3]. Several materials and techniques are currently employed to mitigate, if not eliminate, the presence of capillary water rise in masonry walls and their operating principles have been collectively described in some review papers [1, 3-7]. However, compared to the dimension of the problem of rising damp all around the world, the amount of quantitative data about the effectiveness of these repair solutions, from both in-the-field monitoring and laboratory testing, is still limited [4]. One of the causes of this limited availability of data is the lack of shared, quantitative and reliable methods to measure and monitor the moisture amount in masonry walls [8-9]. This hinders a systematic investigation of the drying effect of the different repair solutions and feeds a certain confusion in the field [1].

Among the available methods to cope with rising damp in brick masonry, chemical damp proof (CDP) courses are one of the most frequently applied [10-12]. This method, introduced in the Sixties, consists in injecting liquid damp-proofing products in a row of holes previously drilled in the wall just above the ground level, in order to create a horizontal 'chemical barrier' preventing the water from rising above it [12-13]. Several products can be used for this purpose, such as pore-clogging materials (silicates, acrylic amide gels, etc.) or, mostly, hydrophobic resins (silicone resins, silanes, siloxanes, aluminium stearate, etc.), the latter increasing the contact angle between water and pore walls and hence hindering capillary absorption of water across the treated zone [10]. CDP courses are attractive for historic buildings as they can be easily hidden (this is of paramount importance in heritage buildings), they involve a limited impact on the structural behaviour (differently, for example, from wall cut) and they are based on a solid working principle, but they cannot be used in masonry with large void and cracks, as for example in rubble masonry (because the resin would be drained there) and their long-term performance is still scarcely known. All things considered, there are still several aspects to fully elucidate, although chemical injections have been investigated in the laboratory since the Seventies and many papers are available in the literature on this subject [3]. The main goal of those studies was the assessment of the capability of the injected products to spread around the holes and to create a continuous barrier in the masonry, also when the pores are partially or completely full of water, a common occurrence in real walls [14]. The ability of the invading injection fluid to displace water already present in the pores was investigated, for both water-based and solvent-based fluids, in the Seventies and Eighties [12, 15-16]. Afterwards, different aspects of this problem and, more in general, the effectiveness of damp-proofing injection

products were investigated in laboratory by different researchers, who performed tests on single materials (brick, mortar) [12-13, 17-19, 20], small-size masonry blocks [15, 16, 18, 21], or real-scale walls [11, 22-25]. Some papers also reported on the drying effect of chemical injections on-site, in real buildings affected by rising damp [26-30].

However, the results of the above reported studies are sometimes difficult to interpret and compare. Monitoring real buildings treated with resin injection may appear as the most straightforward and unbiased method to assess the effectiveness of chemical barriers, but there may be many uncertainties in the results, due to:

- the peculiarities of historic masonries and the heterogeneity of materials [9, 31], that make it difficult to find two identical walls to test (one with resin injection and one untreated, for comparison);
- the non-controllable environment, both in terms of microclimate (air temperature, relative humidity, ventilation, heating and cooling systems, etc.) and water supply from the ground (depth of the water table, infiltrations of rain in the soil, leakage from pipes, etc.) [32-33];
- the difficulty in performing an accurate and unbiased moisture measurement in heterogeneous and often salt-laden historic bricks [9, 34];
- the limited possibility to destructively collect samples for characterization, especially in heritage buildings, where the authorities in charge of conservation do not allow the collection of large samples. This is a problem for the assessment of the drying effectiveness of the repair measures, but also for research, as it hinders a proper understanding of the reasons for the success or failure of the repair [3];
- the long time necessary for walls drying in humid climates [29], non-ventilated buildings and thick walls [32].

Also laboratory studies are hard to compare, as they follow different procedures and methods: (i) to pre-wet the masonry, (ii) to inject the fluid, (iii) to pre-condition the treated masonry, (iv) to monitor the moisture amount, and (v) to characterise the treated materials. Unfortunately, no international standard or recommendation is presently available in this field, so there is no shared procedure to assess the effectiveness of chemical injections in the laboratory. There are only some national guidelines and literature papers which suggest evaluation protocols, but they are quite different from each other. Notably, a robust and easily applicable laboratory test method is of paramount importance, as it is the first step to assess the performance of the different resins and products in simplified conditions, posing the bases for the improvement of these products and for a more sensible and successful application on-site.

In this paper, an overview of the available protocols is firstly provided, then some new proposals of

test laboratory walls are presented and tested. The aim of these tests was to investigate whether the walls proposed are able to reproduce the rising damp occurrence in laboratory, so no resin injection was carried out in the present study. Considering the state-of-the-art of research in this field, we focussed on brick masonry walls, as they represent a large portion of the historic building stock affected by rising damp; moreover, the research on stone masonry walls is still very limited.

2. Overview on the existing methods for the assessment of CDP courses

Testing procedures may be divided into three groups, according to their approach:

- methods aimed at assessing the spreading ability of the resin in specific materials, which is the key parameter responsible for injections' effectiveness;
- methods aimed at assessing the drying effectiveness of chemical injections in laboratory, using small-scale masonry specimens;
- methods and protocols aimed at assessing the drying effectiveness of chemical injections on-site.

This paper focuses on the first two groups, namely on the methods of assessment in laboratory. These methods are expected to help in better understanding the performance of commercially available materials and in developing innovative ones with improved performances. In fact, laboratory tests allow to test several resins, to investigate in detail their behaviour in different conditions, to perform destructive analyses for materials' characterization and to possibly improve the products' formulation for a better effectiveness. Hence, laboratory testing is fundamental to carry out a preliminary investigation, evaluation and selection of the materials to be used for on-site injection. Conversely, on-site testing is aimed at a final evaluation of the effectiveness of resin injection in real but uncontrolled conditions and with limited characterization possibilities, hence the protocols for the assessment of chemical injections on-site [10, 25, 29, 30], although extremely important, will be not discussed here, as they go beyond the scope of the present paper.

2.1 Evaluation of the spreading ability of the resin

As the spreading ability of the injected fluids is the key for the success of any damp-proof course [15, 20], several methods in the literature aim at assessing this property in different kinds of materials, either single or coupled. Samples of limited size are used in this case, to allow an easier characterization after injection.

Hacquebord et al. [18] proposed the use of specimens made of two bricks (with specified porosity, mean pore radius and water absorption coefficient) and a mortar joint made of slaked lime, cement and quartz sand in a volume ratio 3:1:10 (water is added up to a specified workability). After curing

by artificial carbonation, two distinct procedures are proposed for liquid injection products and creams, respectively. For liquid products, cores are drilled from the specimens and their lateral surface is sealed. Then, they are let to absorb an amount of water corresponding to a 50% saturation degree, kept in wrapped conditions at least one day to allow moisture redistribution, and finally the damp-proofing liquid is poured on a plastic tube glued to the top surface. After one day, the residual liquid is removed from the top and the cores are allowed to dry for at least 3 weeks. The two bricks are then separated and their water absorption by capillarity is measured, to assess the ability of the damp-proofing product to spread along the entire height of the cylindrical sample, which approximately corresponds to the distance between two injection holes. For creams, an injection hole is drilled at the centre of the specimen, perpendicular to the mortar joint (Figure 1a), stopping 1 cm from the opposite face, then the specimens are divided into three groups and brought to different saturation conditions: dry, water saturated with evaporation possible and water saturated with no evaporation possible. Then, the cream is poured into the hole and the hole is plugged and kept for 3 months in different conditions: the dry specimens are kept at 50% RH, the saturated specimens are kept in a water head of 1 cm for 4 weeks (specimens with evaporation allowed) or up to the end of the test (specimens with evaporation inhibited). After these 3 months, the samples are firstly cut into slices (Figure 1a) and then dried out in ventilated oven at 40°C. Cutting the samples before the oven drying is very important to avoid the redistribution of the resin during drying. The capillary absorption of the slices is finally determined as an indication of the spreading and water-proofing ability of the resin at increasing distance from the injection hole.

In the two methods described above, no indication is given about the threshold for a satisfactory spreading, but the resin is clearly expected to cover half the distance between two adjacent holes, according to the manufacturer's recommendation.

A cheap and fast method to evaluate the spreading ability of the resin in the brick alone was also proposed in [21]. The specimen is made of two bricks glued by an epoxy adhesive, and its lateral faces are covered with a 1-cm thick cement-based mortar, while the bottom face is sealed, as in Fig. 1b. An injection hole is drilled along the bricks' joint and then the specimen is impregnated with water or with a 3 wt% saline solution (mixture of sodium sulphate, chloride and carbonate and potassium nitrate), according to the circumstances under testing. After pouring or pressure injecting the resin in the hole until the product starts to emanate at all sides of the test piece, the specimens are cured for one month at RH 65% or in waterproof packing, depending on the nature of the product, and then cut along their diagonal. The effectiveness of the resin is evaluated in terms of spreading (assessed wetting the cut surface and observing the dark areas) and damp-proofing (in terms of capillary absorption).

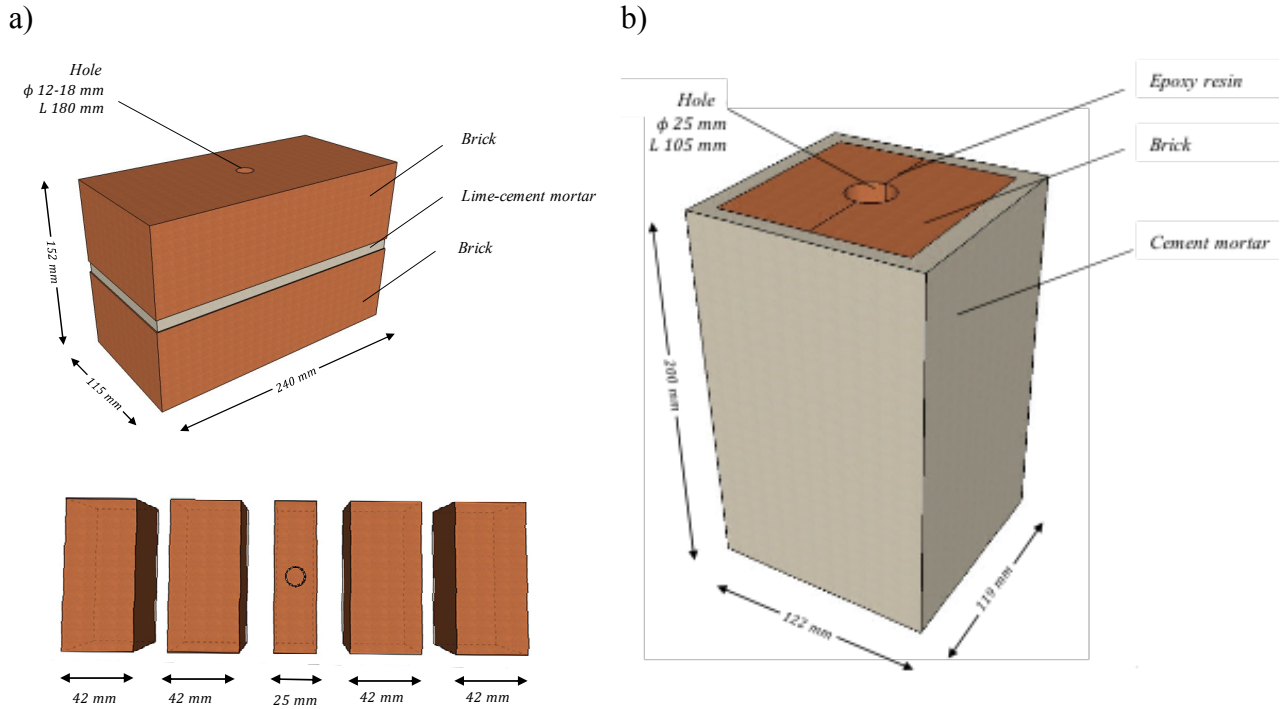


Figure 1: a) Test specimen and cut slices proposed in [18] for cream products; b) test specimen proposed in [21]. Sizes in mm.

A method involving the use of small prisms of calcium silicate with specified porosity and pores mean size was proposed in [19] (Fig. 2). Injection holes are drilled in the prisms and then the specimens are allowed to absorb different amounts of a saline solution and to stabilise for one week, to achieve a saturation degree equal to 40%, 60% and 80%, respectively. The resin is poured into the hole in an amount equal to 25% of the one indicated by the manufacturer, sealing the prism for 28 days. The effectiveness of the resin is evaluated determining the capillary absorption of the prism and the water repellency along a cross section, after cutting the prism in the middle, similarly to the previous method.

All these methods represent a useful tool to evaluate the spreading ability of the injected fluid, however they exhibit two main limitations. The first one is the fact that the spreading of the resin is evaluated when moisture is present inside the pores, but in absence of any water flow, so the possibility that the resin is displaced and redistributed inside the materials is not considered. The second and main limitation is that the spreading of the resin certainly influences its effectiveness in walls, but there is no quantitative correlation available between the cross section reached by the resin and the final outcome in terms of drying. In fact, some authors suggested that, even if injection products provide an only partial pore occlusion, drying is favoured over suction in the wall and some benefits are provided anyway [6, 11], but none of those authors suggested how to estimate the decrease in the moisture amount and/or in the height of the damp zone in presence of a

partial reduction of the suction area.

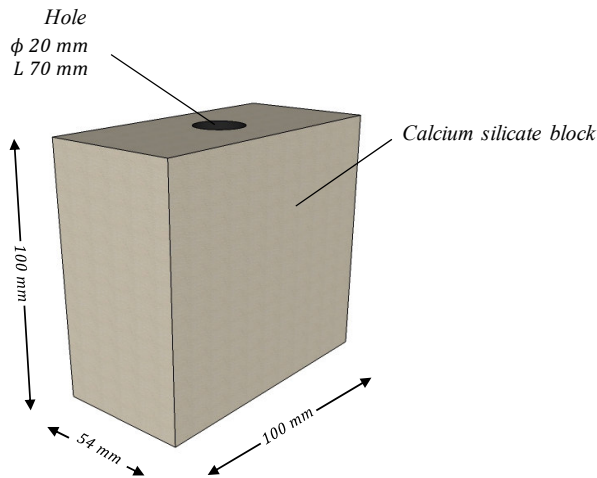


Figure 2: Test specimen proposed in [19] (sizes in mm).

2.2 Evaluation of the drying effectiveness

The German guideline WTA 4-4-04/D [35] proposes two different protocols, for gravity feed and pressure injection, respectively. Two different kinds of test walls are built, as in Fig. 3, using bricks supplied by a specified manufacturer and mortars of different formulation (hydrated lime and sand for gravity feed and hydrated lime, cement and sand for pressure injection). The walls are allowed to cure for 28 days, then holes are drilled as in Fig. 3. Afterwards, the walls are pre-conditioned to a desired moisture content, according to the following procedure. The walls are fully soaked in water and weighed to determine the mass increase at saturation, then they are dried to a mass corresponding to 60%, 80% or 95% saturation degrees, respectively, wrapped and left in this condition until a uniform moisture distribution is achieved (usually 1-3 months). Immediately after the removal of the wrapping, the resin is injected. For each saturation level selected, two walls are treated and one is left untreated, for comparison. After injection, different procedures are followed:

- For 95% saturation degree, the lateral faces of the wall are sealed and the wall is put in a water basin to allow continuous capillary absorption. The evaluation of efficiency is carried out periodically by three methods: measurement of the amount of water evaporating through the top face of the wall, determination of moisture amount in the injection face of the wall by microwave technique, volumetric measurement of the water used to refill the tank. Effectiveness is assessed if the monitored parameter is reduced by at least 50% with respect to the reference wall after a period of 90 days.
- For 60% and 80% saturation degrees, three lateral faces of the wall are sealed immediately

after injection and the evaluation of efficiency is carried out not later than 28 days after, according to the manufacturer's instructions.

The main issue of this test is the long testing time, due to the large size of the specimens, the time needed for internal moisture redistribution and the fact that evaporation is prevented on 3 lateral surfaces of the walls. Moreover, weighing the walls to determine their initial moisture amount and monitoring moisture in the walls is very complex from an operative point of view.

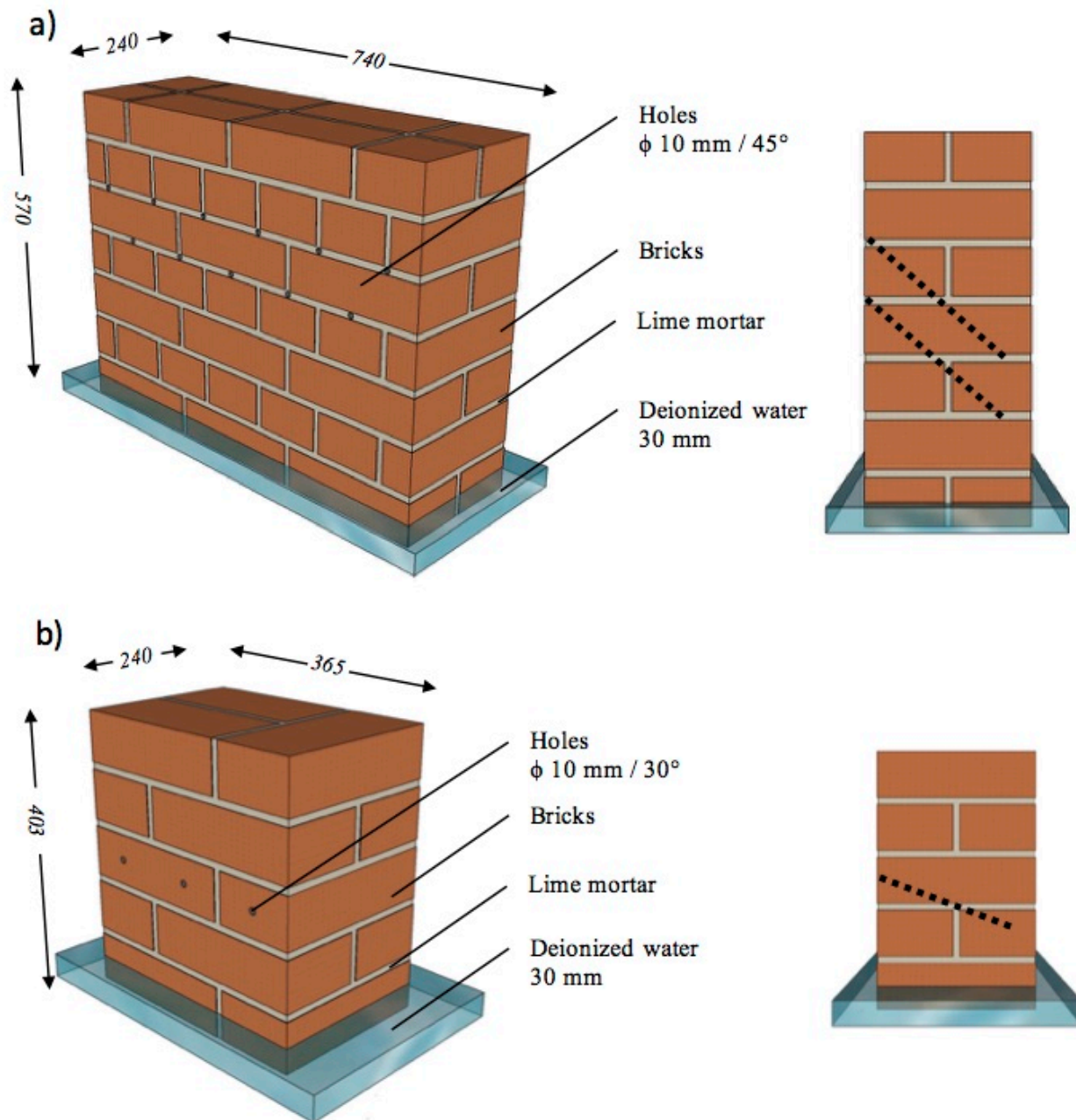


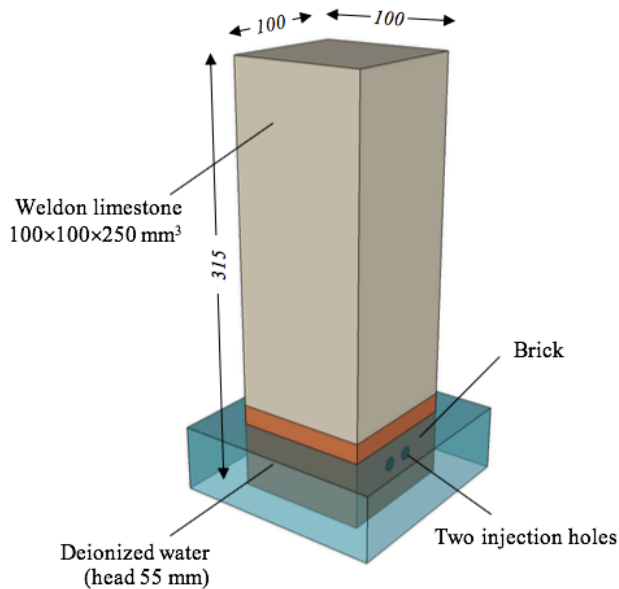
Figure 3: Test walls proposed by WTA 4-4-04/D [35] for: a) pressure injection; b) gravity feed.

In 1988, the British Board of Agreements (BBA) proposed a method [36], to test the effectiveness of 'chemical fluids', by the use of small-scale test pillars (Fig. 4a). A porous limestone prism

(Weldon limestone or similar ones) is placed over a brick piece (supplied by a specific manufacturer), in which two holes have been drilled, after sawing the mating surfaces and possibly interposing cotton gauze to achieve a good contact between the two materials. After pouring water in the bottom tank (head 5 cm), the stone prism is left to equilibrate until constant moisture amount is achieved. The moisture amount is determined by daily weighing the sandstone prism and repositioning it. Then, the resin is injected in the two holes and left to cure for 48 hours, during which the stone prism is placed on a different brick block to keep it moist and avoid any contamination by the resin. After repositioning the stone block, it is weighed daily to assess the drying effectiveness of the injection. An untreated pillar and a pillar in which a physical membrane is inserted between the brick and the stone prism are used as references.

For ‘injection mortars’, a slightly different pillar is used (Fig. 4b), in which the basis is composed of a masonry block (3 brick pieces with two mortar joints). The procedure is the same as above, apart from the fact that, after 2-day curing of the injected product, a dwell period of 3 weeks is introduced, with no water in the tank, and then the water is poured again and the test pillar is left to absorb water by capillarity for 28-100 days, during which the stone pillar is periodically weighed. The dwell period is introduced as the rate of capillary absorption achieved in the test is much greater than that found on site and also to take into account the occurrence of seasonal drying periods in real buildings.

a)



b)

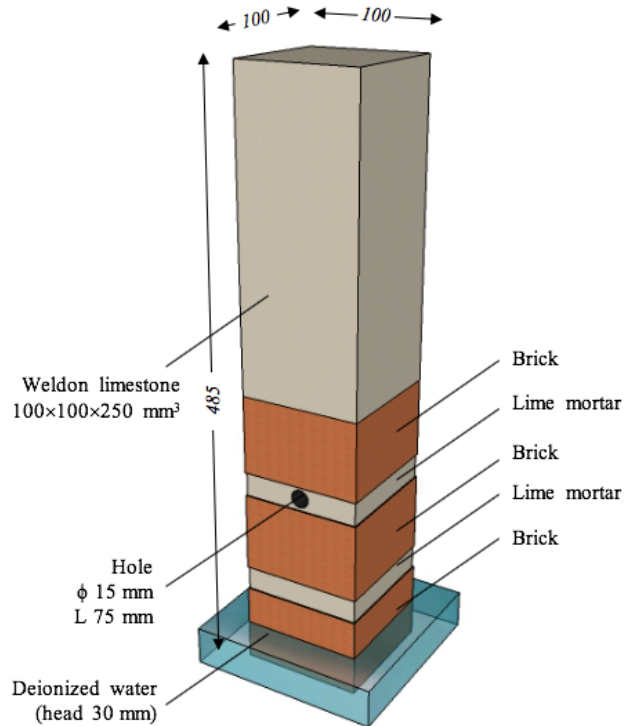


Figure 4: Test walls proposed by BBA MOAT No. 39:1988 [36]: (a) for chemical fluids; (b) for injection mortars. Sizes in mm.

In both the experimental set-ups, the drying effectiveness of the injection product is assessed by comparison with the reference samples.

One possible limitation of this test is that restoring the same capillary flow path between the brick and the stone after removing the latter one for daily measurement may be difficult, or in any case it should be preliminarily ascertained. One source of potential error in the method is the variation in the degree of contact between the limestone measuring block and the damp surface. Moreover, the brick condition at the moment of the injection is very harsh, as the material is basically saturated. On the contrary, the 3-week dwelling period seems a bit too favourable, as it probably allows an almost complete drying of the specimen and hence a satisfactory spreading of the injected fluid across the empty pores.

The use of large-scale masonry walls to test chemical barriers was proposed also in two recent literature papers [24-25]. In [25], 7m×3.5m×2m walls were built, cured for 30 days and stored outdoor (with an upper shelter to avoid the contact with rainfall), with a continuous supply of water (added with NaCl) through basins located under the walls. After the resin injection, the moisture amount at different heights was periodically measured by brick powder extraction by drilling at different heights and gravimetry; hygroscopic moisture determination was carried out as well. In [24], 52 cm×62 cm× 9 cm walls (having a mass of about 50 kg) were built over a basis of reinforced mortar aimed at allowing their movement by two people and subsequent weighing. After curing, the walls were supplied with water at the base and then, after a stable mass was attained, three different products were injected according to the manufacturers' instructions, still keeping the water supply at the base. The monitoring was carried out by periodically weighing the entire walls and, only at the end of the tests, by extracting of samples at different heights and determining the moisture amount by gravimetry. Although these two research papers provide interesting suggestions and results, both kinds of test walls were built with lime-cement mortar joints. This caused a limited absorption of water and hence a quite low water front, so the injection zone was very close to the level of water rise and this may have had an impact on the results, as the water amount in that zone is generally quite variable and unevenly distributed.

2.3 Critical issues in the existing methods and guidelines for a new method

The methods proposed so far to evaluate the effectiveness of DPC are very different in terms of specimens and approach. Based on the literature survey, some critical aspects can be highlighted, as in the following. These aspects should be taken into account also in the development of a new testing protocol in laboratory.

A. Types of masonry materials used.

A.1. Representativity

Although the test walls cannot reproduce the variety and heterogeneity of real masonry walls in terms of internal structure (thickness, bricks disposition, defects, etc.), which can be taken into account only by on-site testing, the bricks and mortar used in the walls should be representative of the materials found in real masonry buildings, in order to reproduce the rising damp occurrence. All the protocols generally suggest the use of solid fired-clay brick exhibiting high porosity and lime-cement based mortar joints. As far as the bricks are concerned, representativity seems not an issue, as porous bricks presently available in the market are quite similar to those used in the past, which were moreover characterised by a large heterogeneity due to the variable firing temperature in the kilns. Conversely, lime-cement based mortars are not so representative of ancient masonry walls, even if the properties of real mortars of damp buildings were found to vary widely, for example in the UK [37]. Lime-cement based mortars were probably proposed in the protocols in order to overcome the problems related to aerial lime-based mortars (too long curing time) and to cement-based mortars (too low capillary absorption) alone, but more representative formulations would guarantee more realistic test walls.

In contrast to the other testing protocols, the Belgian one [19] suggests the use of calcium silicate blocks (rather than brick or brick+mortar), which are porous, but not necessarily representative of historic masonry materials. Additionally, the pH of the pore solution in the mortar might influence the efficiency of the treatment, once resins are injected [23, 37-38].

A.2. Reproducibility

Brick and mortars must have specified characteristics, in order to guarantee a sufficient reproducibility of the testing protocol, as in any standard procedure. Most of the protocols discussed above detail the characteristics of bricks and/or the formulation of the mortar [18-19, 24-25], while others indicate the brand name of the materials and the relevant manufacturers [35, 36]. In both cases, it is impossible to guarantee that the properties of the materials are exactly identical over time, as differences among batches of brick, sand, binder, etc. cannot be avoided. In fact, even a slight difference in the grain size of the binder and/or the aggregate may change the mass proportions, if the recipe is given in volume, or the volume proportions, if the recipe is given in mass. In the case of specified materials' suppliers, the test somehow depends on the suppliers' availability, which may be a further drawback. Thus, the use of reference test walls, which are left untreated for comparison, is fundamental to overcome the problems connected to possible slight variations in the materials' features. In fact, if the drying

effect is evaluated comparing treated and untreated test walls manufactured with exactly the same mortar, any slight differences in the mortar formulation with respect to the ‘standard recipe’ is expected to have a limited impact on the final assessment of resins’ effectiveness. The use of reference specimens is provided for in all the protocols above.

B. Size of the test specimens.

A large size of the specimens [11, 22-25] is positive as it allows to test almost real-scale masonry walls, which are more realistic than single materials or small samples. However, large walls also make the conditioning of the environment (temperature and relative humidity) and the measurement of the total mass very difficult, for practical reasons. Large test walls involve a very long curing time if lime-based mortar joints are used, as in real historic walls, and a long time to attain the equilibrium in each phase the test, from pre-wetting to post-treatment drying. On the other hand, small specimens [15, 16, 18, 21] are more manageable and affordable in terms of testing time, but they exhibit a major drawback: bricks, stone and mortars are highly porous and hence sorptive, so any small laboratory sample which is put in contact with water will quickly get fully saturated, which may be a too severe condition for the test, as discussed in the following.

C. Water saturation degree at the injection time.

This is one of the most critical issues. Real walls usually exhibit a moisture profile decreasing with height [30, 39- 41], hence the moisture amount at the bottom of the wall, meaning in the injection zone, is obviously maximum. However, full saturation can be found only sometimes at the basis of real walls, and “building materials in use are far more commonly unsaturated than saturated” [42]. In fact, it was pointed out that “a realistic performance test for damp-proofing treatments must utilise a wall with a substantial moisture gradient and moisture contents should not be unrealistically high at the treatment level” [38]. Hence, injecting the resins in completely saturated building materials may be too harsh, especially considering that the success of chemical damp-proofing is known to depend on the saturation degree of the existing materials [14, 18, 20]. Aware of this issue, most of the protocols suggest to pre-condition the specimens to specified saturation degrees, by letting them absorb an amount of water corresponding to the expected moisture percent and then keeping them wrapped until water distributes uniformly in the specimens (e.g., [18, 35].

D. Dynamic or static moisture?

The injection and subsequent curing of the resin can be carried out either in specimens in which a specified water saturation degree was induced (see the issue C above) or in specimen which are subjected to a continuous capillary water flow. This latter condition is obviously more

realistic, as this is what happens in real walls, in which the spreading of the resin, its curing and the capillary water rise occur all at the same time. It derives that dynamic conditions should be preferred over static ones, as they are more realistic. However, subjecting the wall specimens to a continuous water supply through their bottom surface usually leads to full saturation, due to the high sorptivity of materials (see the issue B). The protocols described above either involve a continuous water supply [24-25, 36] and thus an almost complete materials' saturation, or static moisture conditions at different saturation degree [18-19, 21, 35].

In order to provide the specimens with a continuous water supply and at the same time, maintain unsaturated conditions (with a saturation gradient from the bottom to the top of the specimens), some authors recently proposed to interpose a layer of material having low porosity between the water reservoir and the brick specimen [43]. This strategy was proposed in a test aimed at assessing the effectiveness of chemical injection on-site, but it could be useful also for laboratory tests.

E. Conditions after the injection.

After the resin injection, different conditions can be selected in the monitoring of the drying effectiveness. A first option is to keep the test walls or specimens under a continuous water supply by capillary absorption (dynamic conditions) [24-25, 35-36]. This is quite realistic, although the flow speed in laboratory test specimens is not necessarily the same experienced by real walls. Moreover, permanently saturated conditions, as explained above, may be too harsh, thus some authors proposed to alternate soaking and drying periods to take account of a seasonal cycle [36]. However, seasonal variations rarely lead to complete drying, hence this condition is not so representative and it may boost the spreading of the resin unrealistically. A second option consists in keeping the moisture level constant after the injection, for example by wrapping or sealing the sample, for a certain time. This approach is usually aimed at evaluating the spreading ability of the resin, rather than its drying effectiveness, although of course these two parameters are related.

F. Evaporation surface.

In some of the protocols described above, the external surface of the sample is partially sealed, in order to make evaporation occur through a desired face. However, the purpose of the sealing and why it is supposed to reproduce on-site conditions is not explained.

G. Methods to measure the moisture amount and to evaluate the drying effectiveness.

When the testing procedure is aimed at evaluating the spreading ability of the resin, the effectiveness is usually evaluated by determining the water repellency and capillary absorption in the cross-section of the material under testing, with and without the treatment. Conversely,

evaluating the drying effectiveness of the treatment in a test wall is much more challenging, related to the difficulty of measuring moisture in building materials [34]. The techniques suggested in the available protocols for the monitoring of moisture after the treatment are very different: microwave method, measurement of the water evaporation, gravimetric measurement of the moisture in a stone probe put on top of the material, weighing of the entire wall, etc.. Several innovative NDT techniques have been recently proposed for moisture measurement [34], but some of them cannot easily be applied to large masonry specimens, or they are very expensive, or few laboratories have this kind of facilities, or simply they have not been tested for this purpose yet.

3. Materials and methods

3.1 Testing walls

Two different kinds of testing wall, having large and small size, were manufactured: Wall Type-A and Wall Type-B, respectively (Fig. 5).

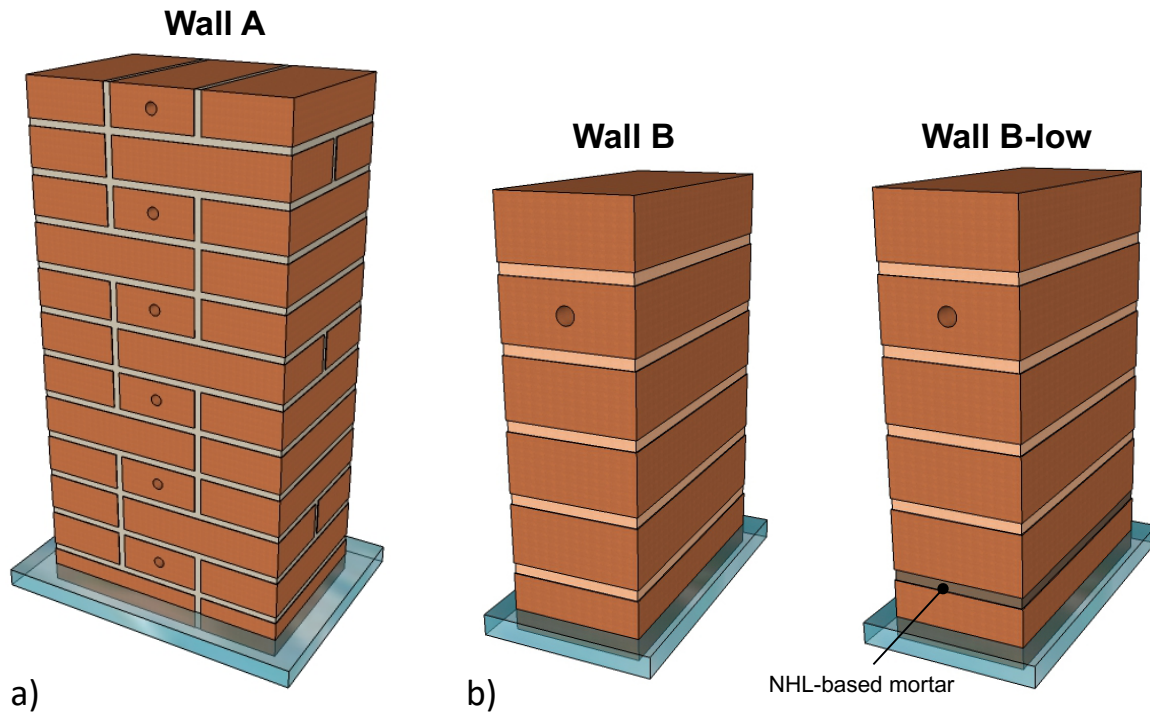


Figure 5: The test walls proposed in this study: a) wall A ($\approx 240 \times 380 \times 765 \text{ mm}^3$); b) walls B and B-low ($\approx 120 \times 250 \times 338 \text{ mm}^3$).

Wall Type-A was built with $240 \times 120 \times 50 \text{ mm}^3$ solid bricks (Stabila, Italy), while wall Type-B was

built with $250 \times 120 \times 55 \text{ mm}^3$ solid bricks (Terreal, Italy). Despite the different manufacturers, the two bricks selected are expected to have similar characteristics. The slight difference in bricks' sizes was not considered critical in these tests, as the two bricks were used in test walls having very different size and geometry.

The mortar for the joints (1.5 cm thick) was manufactured mixing quartz sand, slaked lime, brick powder ($< 0.15 \text{ mm}$) and deionised water, in volume proportions 2:1:1:1 (corresponding to 1450 g river sand, 291 g slaked lime, 408 g brick powder and 382 g water). In a previous study, this mortar was shown to exhibit a high porosity, hence it was expected to favour the capillary absorption by the walls [39]. Moreover, despite being cement free, the presence of the pozzolanic brick dust is expected to reduce the time necessary for hardening with respect to plain lime-based mortars. The use of brick dust (or, in alternative, of natural pozzolana) together with lime was a common practice in historic buildings, given the low mechanical strength and low resistance to water of plain lime. Manufacturing a sufficiently sorptive mortar, providing at the same time an acceptable curing time is not easy [44], but good results were obtained in the past with lime-brick based mortars [38].

Three replicates of each wall type were built. However, as walls B were expected to quickly get saturated due to their high porosity and small size, a further group of three walls was manufactured and labelled B-low. These walls are identical to walls B, except for the fact that a more compact mortar was used for the first joint only, in order to investigate if a limited saturation degree could be introduced in the samples. This second mortar was manufactured mixing quartz sand, slaked lime, hydraulic lime FL C2 (according to EN 459-1 [45]) and water, in volume proportions 3:1:1:1 (corresponding to 1450 g quartz sand, 145 g slaked lime, 291 g hydraulic lime and 291 g water). Both the mortars were prepared in a Hobart mixer (EN 196-1 [46]), following a constant mixing procedure during the entire manufacturing of the walls. During the preparation of the mortars, three $40 \times 40 \times 160 \text{ mm}^3$ mortar prisms were also separately manufactured in a steel mould (EN 196-1) and were allowed to cure for one month at $T=20 \pm 2^\circ\text{C}$ and $\text{RH} > 95\%$ and for one further month in lab conditions, for characterisation purposes.

Prior to building the walls, some of the bricks had been drilled to create holes (diameter 16 mm, length $\approx \frac{3}{4}$ of the brick) for the periodic monitoring of moisture, according to the protocol described in [9]. This protocol suggests that brick fragments taken from exactly the same brick in which the hole is drilled are inserted in the holes and the holes are sealed, allowing the fragments to equilibrate with the surrounding brick over time. The fragments are periodically extracted and used to determine moisture by gravimetry, then they are re-inserted in the holes for further monitoring. The locations of the holes for moisture monitoring in walls A are reported in Fig. 5a: six holes were drilled at different heights. In Wall Type-B and B-low, a single hole was prepared (Figure 5b); in

this case, a row of vertical measurement holes was avoided not to alter the water flow too much. The walls were built directly inside basins, previously soaking the bricks in water for 24 hours to avoid any water depletion of the mortar joints. Walls A were cured in laboratory conditions for 12 months, and walls B for 3 months; in both cases, curing was carried out for the first two weeks under a plastic sheet to prevent water evaporation.

The theoretical position of the holes for resin injection is the following: in Walls A, three holes drilled from the front face in the fourth mortar joint from the bottom (stopping about 3 cm from the opposite face); in Walls B and B-low, three holes drilled from the lateral face in the third mortar joint from the bottom (again stopping about 3 cm from the opposite face). However, no injection was carried out in this study, hence these holes were not drilled.

After curing, deionised water was put in the basins. A constant water head equal to 2 cm was maintained during the test, refilling when necessary.

3.2 Methods

Mercury intrusion porosimetry (Porosimeter 2000 Carlo Erba and Fisons Macropore Unit 120) was carried out on the two bricks used for walls A and B and on the mortar prisms. The mortars cast in the prismatic steel moulds were considered representative of the ones in the joints, because the wall bricks had been saturated in water before the walls manufacturing, hence they were expected not to absorb any mixing water from the mortars, not altering their porosity.

The determination of the capillary water absorption coefficient, CA , of the bricks and the mortar prisms was carried out according to EN 15801 [47]. At the end of the test, the samples were immersed in water up to constant mass and their water absorption at saturation at room pressure (WA_{sat}) was determined too.

The measurement of moisture in the holes was carried out periodically according to the protocol described above. All fragments, having size 1-2 g, were allowed to equilibrate in the holes for at least 3 weeks before any measurement.

The temperature and air relative humidity in the laboratory were measured, in the days of moisture measurement in the test walls, by a thermal-hygrometric probe Testo 635.

The amount of $CaCO_3$ in the efflorescence appeared on the top of walls A (see Paragraph 4.2) was determined gently scratching the efflorescence from the substrate and analysing them by gas volumetric method after HCl attack, in a Dietrich-Frühling calcimeter.

4. Results and discussion

4.1 Materials characterization

The pore size distribution curves of the two bricks and the two mortars are reported in Figure 6. The bricks used in Wall Type-A exhibited a total open porosity equal to 34.0% and a mean pore radius equal to 1.2 μm , while the bricks used in Wall Type-B exhibited an open porosity equal to 34.2% and a mean pores radius equal to 2.5 μm . As expected, the two kinds of brick selected are similar and characterised by high porosity, like historic bricks [42, 48]. The water absorption at saturation resulted equal to $22.7\pm0.7\%$ for the bricks of walls A, and equal to $22.0\pm0.3\%$ for the bricks of Walls Type-B. The mortar with lime and brick powder that was used for all the walls exhibited an open porosity equal to 26.1% and a mean pore radius equal to 0.4 μm , while the mortar used for the first joint of walls B-low exhibited an open porosity equal to 24.8% and a mean pore radius equal to 0.1 μm . Although the total porosity of this latter mortar is only slightly lower with respect to the other one, its smaller pores mean radius is expected to slow down the rate of capillary absorption of water [49-50].

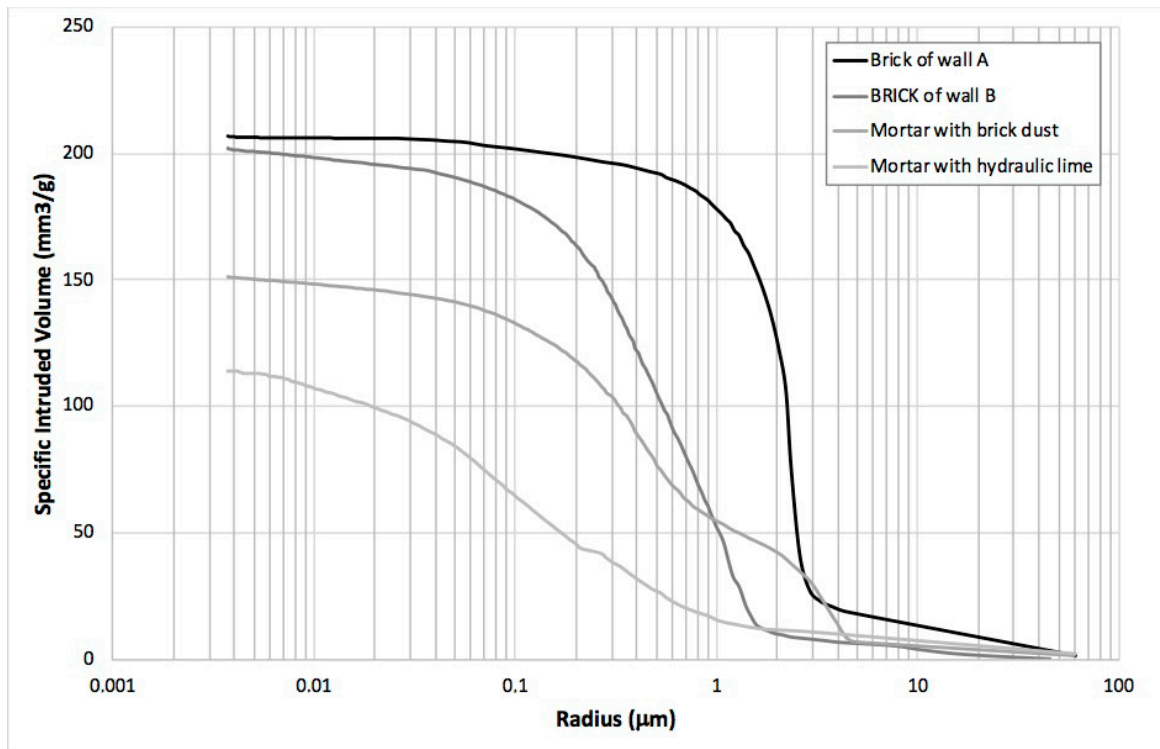


Figure 6: Pore size distribution curves of the materials used in the test walls.

The capillary water absorption coefficients of the materials used are reported in Table 1. The two bricks behave quite similarly. Due to their finer pores, the mortars exhibit a slower absorption rate than the bricks, although not dramatically. The CA values of the mortar with lime and brick dust

and the mortar with hydraulic lime are basically comparable.

Table 1: Capillary water absorption coefficients (CA) of the materials used.

Sample	CA (mg/cm ² s ^{0.5})
Brick walls A	25.8
Brick walls B	25.0
Mortar with hydraulic lime (first joint of walls B-low)	18.5
Mortar with brick dust (all the other joints)	19.3

4.2 Wall Type-A

Moisture was measured 1, 2 and 3 months after the water supply in walls A and the results are shown in Table 2 and Figure 7. Due to the high CA of both bricks and mortar joints, the capillary flow reached the top of the walls after just 1 month, although an additional month in contact with water caused a further slight increase in moisture content (+2.3 wt% on average, passing from 30 to 60 days). Considering that the water absorption of the bricks used is equal to $22.7 \pm 0.7\%$ at saturation, it can be noticed that the bottom bricks were basically saturated at two months, and the moisture amount decreased with height, reaching an average saturation degree at the top measurement hole equal to 79%. Such moisture decrease with height is observed also in real buildings, hence the test walls were successful in reproducing this occurrence.

However, the monitoring of the laboratory environment revealed that the air conditions changed during the testing period. In the first two measurement dates, temperature was $21 \pm 2^\circ\text{C}$ and relative humidity was $55 \pm 5\%$, while in the third one temperature was $22 \pm 2^\circ\text{C}$ and relative humidity was $35 \pm 5\%$. This change was ascribed to the switching on of the heating and ventilation system in the labs, happened 5 days before the second measurement and causing an unexpected drop in the air relative humidity. In fact, the moisture profile at 90 days exhibited a decrease with respect to the previous measurement, but such decrease was small (-1.2 wt% on average, passing from 60 to 90 days), hence the sensitivity of these large test walls to microclimate changes seems quite limited. Some efflorescence was observed at the top of the walls (Fig. 8), probably due to salts impurities already present in the bricks. However, the calcimetry analysis performed on the efflorescence alone revealed that they contained about 20% of calcium carbonate, which clearly indicates that the lime-based mortar joints, notwithstanding the long curing and probably also due to averagely low air relative humidity of the laboratory, still contained some not carbonated calcium hydroxide, that

was leached by the capillary water flow. This aspect may be of particular significance in case of injection of materials that are sensitive to pH [37-38], so artificial carbonation would be necessary in that case. Efflorescence formed at the top of the walls seems not to have affected the results, as the moisture distribution with height and time is fully consistent and no uneven moisture amount was found in the top measurement points.

Table 2: Walls A: moisture amount in the monitoring holes at different times.

Wall	Height of measurement hole (cm)	Moisture (wt.%)		
		30 days	60 days	90 days
A-1	9	20.4	21.8	21.4
	21	18.3	20.1	19.0
	33	17.9	20.2	18.0
	47	17.0	20.0	17.9
	59	17.3	18.4	17.9
	71	16.1	17.0	16.7
A-2	9	20.8	23.4	21.4
	21	18.3	19.7	18.9
	33	16.9	19.4	18.8
	47	16.7	18.2	17.9
	59	15.8	18.0	17.1
	71	16.0	17.3	16.6
A-3	9	18.5	25.9	21.9
	21	18.4	23.7	19.5
	33	17.1	22.0	19.0
	47	17.2	20.2	18.1
	59	16.0	19.7	17.8
	71	16.8	19.6	17.6

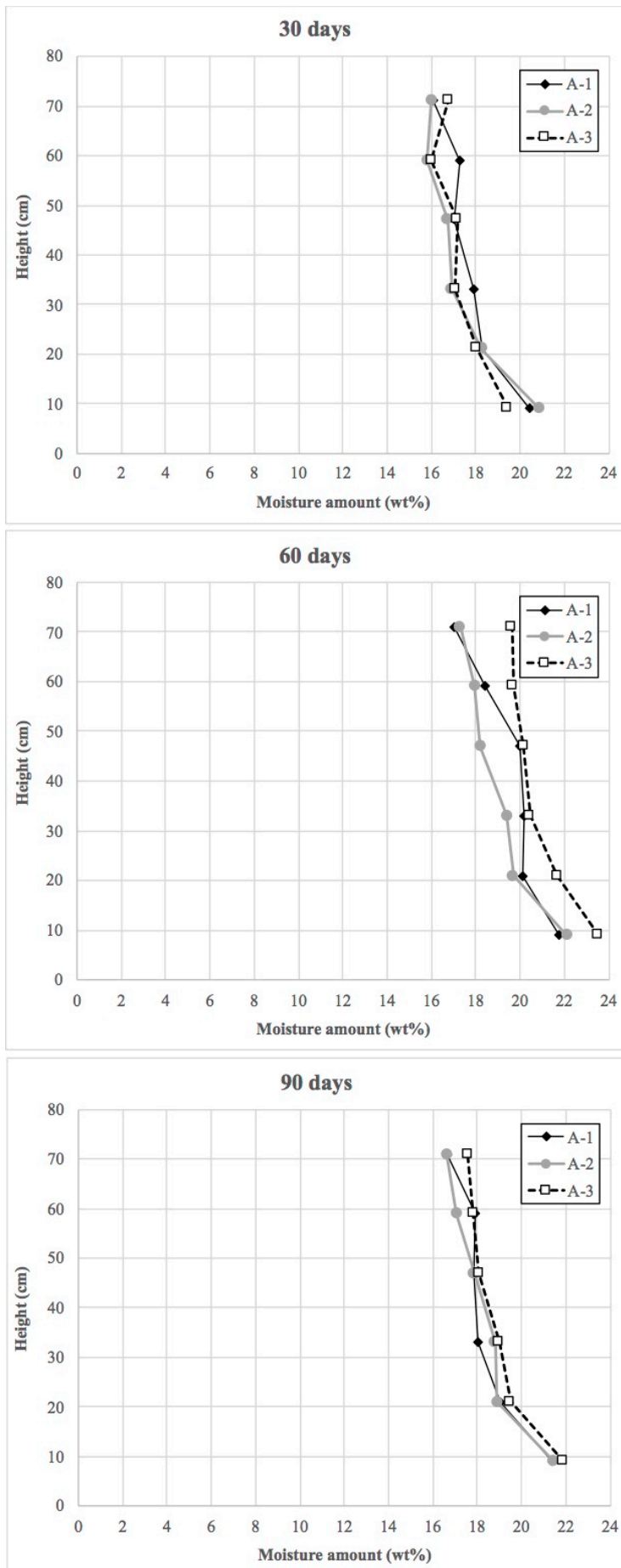


Figure 7: Walls A: moisture distribution with height at the three measurement times.



Figure 8: Efflorescence formation at the top of walls A.

4.3 Wall Type-B

Three days after the water supply in the basins, Wall B appeared already completely wet (dark zones in Fig. 9), as expected due to the high CA of bricks and mortar joint. At the same date, walls B-low appeared only partially wet (Fig. 9), due to the presence of hydraulic lime-based mortar in the first joint. These observations corroborate the idea that a lower amount of water and a lower front level can be induced by inserting a layer with reduced porosity and pore size at the basis of the test wall. However, the switch on of the heating system, occurred in this case at day 15 since the water supply, caused a quick drying of the walls, so that at the first measurement (day 30), the moisture amount in the measurement points appeared limited (Fig. 10) and at day 45 and 60 the measurement points were basically dry. This clearly shows that these small walls are extremely sensitive to relative humidity variations.

In order to restore the initial level of moisture in the walls, at day 61, the walls were covered with nylon boxes, after placing containers with a saturated solution of NaCl inside the boxes, to establish a constant $76 \pm 2\%$ air relative humidity around the walls. The attainment of this relative humidity was checked by probes. Due to this, the evaporation rate was slowed down and walls moisture progressively increased, so that at the final measurement (day 150) wall B was close to saturation, while the water front in walls B-low went up, but it did not reach the measurement point, which remained basically dry (Fig. 10).

A negligible amount of efflorescence appeared on the top of the specimens, due to salts originally

present in the materials, with no evidence of incomplete carbonation of the mortar joints, so in this case two months of curing can be considered enough for the joint mortars.



Figure 9: Walls B (left) and B-low (right) 3 days after the supply of deionised water in the basins.

Wall	Moisture (wt%)				
	30 days	45 days	60 days	105 days	150 days
B-1	7.0	1.7	1.1	11.8	21.0
B-2	1.2	2.4	1.2	5.7	15.7
B-3	6.2	2.2	1.4	7.4	15.2
B-low-1	5.6	1.4	1.2	0.5	0.5
B-low-2	6.3	1.7	0.9	0.6	0.6
B-low-3	6.7	2.1	1.2	0.7	0.7

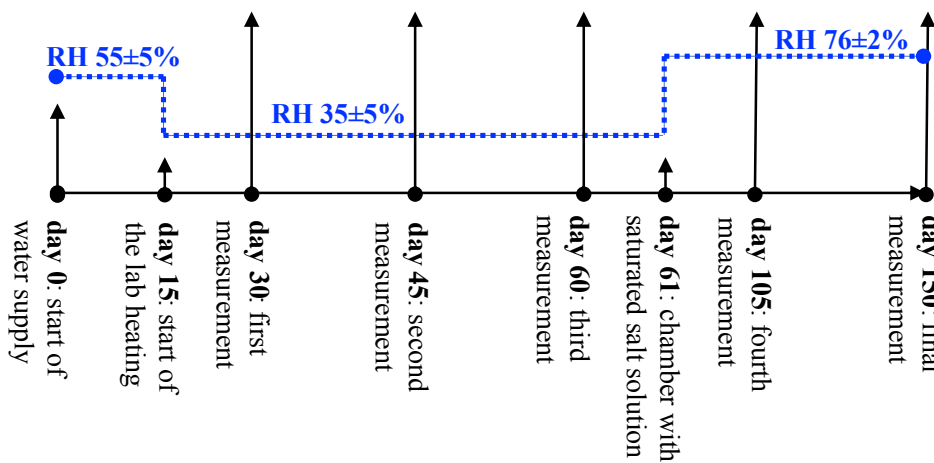


Figure 10: Moisture amount at different times from water supply, in parallel with a scheme of relative humidity in the laboratory.

4.4 Discussion on the advantages and disadvantages of the testing walls

Based on the results above reported, the advantages and disadvantages of the proposed testing walls are discussed in the following, with reference to the issues of Section 2.3.

A. Types of masonry materials used.

A.1. Representativity

The materials used to build the walls, which are commonly available in the market, are similar to those that can be found in historic buildings. Fired-clay bricks exhibited a porosity of about 34% and a mean pore radius close to 1 μm (1.2 and 2.5 μm , respectively), which are common features for both current and historic bricks [5, 42, 48, 51]. Mortar with slaked lime and brick dust was widely used in the past, as well as mortar with natural hydraulic lime. The absence of cement in the joint mortars is important not only because cement was not used in ancient buildings, but also because its use is not recommended in the repair of historic ones [52-53]. Hence, the materials used for building the walls can be considered fairly representative of those used in historic brick masonry walls, although it is obviously impossible to reproduce the variety of historic formulations with a single mortar.

A.2. Reproducibility

Although the main characteristics of the brick and the formulations of the two mortars are reported in this paper, their exact reproduction is made impossible by the unavoidable differences always present in different batches of building materials. For example, the pozzolanic behaviour of the brick powder and the exact composition of the natural hydraulic lime, as well as the pore size distribution of the brick, cannot be perfectly reproduced in future tests, due to a certain degree of variability in the raw materials. However, this problem can be overcome by the use of reference walls that are left untreated in the same laboratory conditions, for comparison, provided that all the materials used for the manufacturing of the test walls come from the same batches and are manufactured by the same procedure. In such conditions, it was shown in the present study that the walls specimens within the same group have comparable behaviour, hence the use of reference untreated walls seems a viable solution for the testing of resin injection's effectiveness.

B. Size of the test specimens.

Large walls (walls A) involve a very long time for testing. In fact, after 1-year curing the carbonation of lime in the mortar joints was still incomplete and some non-carbonated calcium hydroxide was leached by the capillary water flow, as demonstrated by the presence of calcite in the efflorescence at the top of the walls. Artificial carbonation would be necessary for a

complete hardening of the mortar in a reasonable time, but large carbonation chambers are usually not so easily available in laboratories. After the water was supplied to the basins, two months were necessary to reach a steady state of the capillary flow in these walls.

Small walls (B and B-low) apparently reached a satisfactory hardening of the mortar joints in 2 months, due to their limited cross-section, as suggested by the very limited presence of efflorescence on top of the walls. Once supplied to the basins, water reached the top of the walls in few days, although in this study it was not possible to determine the time necessary to reach the steady state, due to an unexpected change in air relative humidity caused by the switching on of the heating and ventilation system in the labs some days before the second measurement of moisture.

However, in terms of moisture amount and distribution, Wall Type-B exhibited an extreme sensitivity to air relative humidity changes, so it was necessary to keep them in a controlled environment, which was done quite easily, by covering the specimens with boxes and putting inside them containers with a saturated NaCl solution. Wall Type-A is much less sensitive to air relative humidity variations.

C. Water saturation degree at the injection time.

In the present study, no injection of resin took place, but a steady state was reached in the capillary flow of water, so it is possible to discuss the saturation degree in the bricks. Walls A exhibited a moisture profile decreasing with height, as in real walls, which was a positive result. The saturation degree at the basis of the wall was basically 100% and the one at the top was about 79%. This means that the resin, if injected at the height originally planned (fourth mortar joint from the bottom), would have to spread in a masonry having a saturation degree approximately equal to 88%, which is representative of a harsh but not unrealistic condition found in the injection zone of damp masonry walls. In the case of walls B, when a steady state was attained, the moisture amount in the single measurement point, namely in the second brick from the top, was 17.2 wt% on average, corresponding to a saturation degree equal to 78%, suggesting also in this case a gradual decrease of moisture content with height, as in real walls. In the case of walls B-low, the hydraulic-lime based mortar in the first joint was successful in slowing down the capillary absorption, but in such a way that the water did not reach the measurement hole, not allowing to quantify the moisture. Thus, the presence of a different, less sorptive mortar in the first joints seems a promising route to establish a lower moisture amount in the walls with respect to saturation, but a higher number of measurement holes is necessary to investigate the moisture distribution with height. However, it is noteworthy that drilling a vertical series of measurement holes in these walls might alter the water flow, so this needs

further investigations.

Moreover, it must be noticed that the CA of the two mortars, determined on mortar prisms, was basically comparable, although their pore size was slightly different, hence this aspect needs further investigations and fine tuning.

D. Dynamic or static moisture?

In both test walls A and B, a dynamic water flow was established. This is positive, as the condition occurring in real walls is reproduced, but it will be necessary to determine the velocity of the capillary flow in saturated conditions, as it might have an impact on the resin displacement after injection. This aspect will be considered in future tests.

E. Conditions after the injection.

The test walls proposed allow a continuous water supply by capillarity also after the resin injection, as in real walls.

F. Evaporation surface.

The evaporation was not prevented in any of the wall surfaces. However, it will be necessary to determine the speed of the water flow, to evaluate if it is realistic and, in case it is too fast, if it may alter the results by causing an excessive resin migration along the capillary path. In such case, a partial sealing of the surfaces could be considered, although it is expected to increase the time necessary to reach the equilibrium and to dry, and to affect the moisture distribution along the walls' height.

G. Methods to measure the moisture amount and to evaluate the drying effectiveness.

The method proposed to measure moisture in the test walls allowed to obtain representative and consistent data. Comparable moisture amounts were found within the same group of masonry walls, confirming the reliability of the measurement method. Moreover, the method is cheap and easy, not requiring any special equipment.

5. Conclusions

After a review of the methods proposed in literature papers and national guidelines to test the effectiveness of resin injection in laboratory, two new types of test walls were proposed in this paper, both of them made of fired-clay bricks and mortars similar to those used in historic buildings. These test walls are aimed at reproducing the occurrence of rising damp, for the testing of chemical injections. The walls were subjected to water supply at the basis and the moisture amount in the bricks was monitored for some months by the use of 'permanent sampling holes'. The results obtained allow to derive the following conclusions:

- The proposed test wall are able to reproduce the rising damp occurring on-site, by exhibiting

a continuous capillary flow and a gradient of moisture with height. By selecting the height of injection, it seems possible to investigate the resin's effectiveness in different saturation degrees

- The use of a less sorptive mortar in the first joint of the test walls seems a promising route to promote unsaturated conditions in the walls and to control the water rise front
- The use of 'permanent sampling holes' provides reliable and consistent results and allows to monitor moisture for any duration that may be necessary, because the brick fragments can be re-introduced in the corresponding holes for an indefinite number of times
- The large test walls are scarcely sensitive to microclimate changes, hence more stable, but they involve a long time for mortar curing (> 1 years) and for the attainment of the steady state in the capillary absorption of water (2 months). Moreover, the construction of big samples is quite space consuming.
- The small test walls involve a shorter curing, but they are very sensitive to microclimate changes, so they must be located in a room or container with constant RH, which is however quite easy due to the small size of the specimens. It seems that a multiple number of permanent sampling holes is necessary also in these small test walls, as in large ones, to take into account the possible variations in the water front and moisture amount
- Given the influence of the environmental conditions on the moisture distribution within the test walls, it is extremely important that the performance of the injected resins will be carried out in comparison with untreated walls located in the same laboratory conditions.

Moisture distribution and water front height in small walls was affected by microclimate (evaporation rate, basically), but it may be also sensitive to other parameters, such as the characteristics of the bricks and the mortars joints (porosity, sorptivity, etc.) and the resistance to water flow at the interface mortar/brick. For this reason, future studies will be address to a modelling-assisted design of test walls, in which the behaviour of the test walls will be predicted starting from all the above mentioned parameters. In fact, modelling may be a useful support tool and may allow to skip some of the issues arising in laboratory experimental campaigns.

The developed test walls represent a promising route for the testing of resin injection and possibly also other repair methods in laboratory. They will be used in future studies for the investigation of different damp-proofing materials for masonry repair.

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