

Injection capacity of hydraulic lime grouts in different porous media

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Abstract Grout injection is a widely used technique for consolidation of multi-leaves masonries, aimed at increasing the compactness and to create links between the internal and external leaves that will improve shear, flexural and compressive resistances. Grouts can be seen as mixtures of binder with water, admixtures and/or additives, which should present low viscosity and high penetrability. The definition of a grout composition should involve the knowledge of the injection capacity within a specific type of masonry and good physical and chemical compatibility with the original materials present in the historic structures.

The flow of the grout through the masonry depends on the fresh grout properties, such as stability, water retention and rheological behaviour. Thus, the evaluation of the performance of the grout as function of a porous medium is firstly started by checking the intrinsic properties of the grout (namely rheological and stability) and then by controlling the injectability of masonry by injection tests on cylinders. Since it is difficult to reproduce a real masonry and to visualize what is happening inside the porous medium being injected, masonry samples were created by filling plexiglass cylinders with a fraction of limestone sands and crushed brick. These materials are sieved to obtain different grain size distributions to enable the simulation of different permeabilities and internal structures for the masonry. The lack of information about the performance of hydraulic lime based grouts as a function of the properties of the porous medium to be injected enhances the need of a detailed research on the subject.

KEYWORDS *Grout injection technique, multi-leaves masonry, hydraulic lime grouts, rheological properties, injectability tests, grout penetrability.*

Abbreviations PM: porous medium

1. Introduction

Grout injection has been regarded as a suitable technique to enhance the homogeneity, uniformity of strength and cohesion of masonry walls. One aspect of extreme importance in the consolidation of monuments is that injections are almost invisible, although not reversible. Several researchers have been studying the effectiveness of the technique in these last two decades [(Binda, et al., 1997), (Binda, et al., 2001), (Binda, et al., 2003), (Bras, et al., 2012) and (Van Rickstal, et al., 2003)].

The grout injection allows the increase of masonry compactness and creates bonds between the internal and external leaves, therefore improving the masonry mechanical strength and monolithic behaviour after hardening of the grout (Ignoul, et al., 2004). Thus, the brittle mechanisms characterized by the out-of-plane detachment of the leaves are minimized (Valluzzi, et al., 2004). Grouts for injection should be adequately designed to achieve the best performance from the injectability point of view. This means that fresh grout properties, such as rheological properties are of prime importance, since adequate rheological properties are an essential criterion to allow the correct flow of the grout inside the masonry to ensure the filling of the voids [(Valluzzi, 2005), (Kalagri, et al., 2010)]. The most important rheological parameters are the fluidity, characterized by the plastic viscosity, the yield stress and the flow time for control of composition of the grout. Other important properties are the granularity of the binder before mixing (Ignoul, et al., 2004), since both rheology and penetrability depend on the particle size of the grout. According to Eriksson (Eriksson, 2002) the most important porous medium (PM) features affecting penetrability are aperture size, variability in aperture and magnitude of contact areas, sometimes referred to as tortuosity. In relation to this issue, the aim of this work is to investigate how the penetration of hydraulic lime grouts stops or gets blocked, and to increase the understanding for the different mechanisms affecting this effect. The stop mechanisms are important to consider during grouting as well as in grouting design in order to understand the processes and to optimise the grouting performance. Research work from different domains of the literature (masonry grouting and soil/rock grouting) has been carried out on penetrability, with some authors defending the importance of the ratio between available opening of the void/channel of the PM to be injected and the maximum particles sizes of the solid phase of the grout [(Eklund and Stille, 2008), (Miltiadou-Fezans and Tassios, 2012) and (Axelsson, et al., 2009)]. In the present article, several criteria already established by different authors were evaluated and related with the grout injection capacity of the present study. Thus, it was possible to identify the appropriate criteria to express the penetrability of the grout used in different PM.

The effectiveness of a grout injection depends not only on the characteristic of the mix, but also on the knowledge of wall type (Valluzzi, 2005). Therefore, it is of utmost importance to know precisely the morphology of the wall section, the composition of the materials constituting the wall, distribution and size of cracks and percentage and distribution of voids [(Binda, et al., 1997), (Binda, et al., 2003)]. It is noted that the permeability and moisture content are also important properties in the assessment of injectability (Van Rickstal, 2000). The relation between the parameters mentioned above (calculated by standard tests) and grout injectability tests is evaluated in the present research.

Injectability tests were used to study the penetrability of grouts. Since it is hard to reproduce a real masonry and because it is difficult to visualize what is happening inside

the PM being injected, reproducible masonry samples of cylindrical shape were created. Two materials with different water absorption coefficients were used in order to study the influence of grout water loss to PM in grout injectability. These materials and the injection setup are similar to that used by other authors in their grout injection tests [(Bras, et al., 2012), (Valluzzi, 2005), (Van Rickstal, et al., 2003)]. However, in this article the outputs achieved are different: the injectability of the grout was analysed based on two equations. One of them was proposed by Bras (Bras, et al., 2012) and takes into account the time and injection height. Another one was a result from this study and expresses the percentage of voids volume that is filled after grout injection. Different grout injectability results are obtained for the various porous media studied. Furthermore, these results are compared with the results obtained by other authors [(Bras, et al., 2012), (Valluzzi, 2005) and (Van Rickstal, et al., 2003)].

Additionally, one of the main criteria for the choice of the binder in the assessment and study on the composition of the grout, is the evaluation of potential incompatibility problems with the materials from the original walls. According to some authors [(Bras, et al., 2012), (Miltiadou-Fezans and Tassios, 2012), (Valluzzi, et al., 2004), (Valluzzi, 2005), (Vintzileou, 2006)] , natural hydraulic-lime binders provide potentially more compatible grouts because of similarity in chemical, physical and mechanical properties to historic materials, when compared with those based on pure cement or organic resins, thereby enhancing durability.

2. Literature survey

2.1. Penetrability of grout

2.1.1. Penetration capability

The penetrability is related to the filling of the existing voids and fissures, directly contributing to the strength, tightness and durability of the masonry. To do so, the grout should be able to pass through the "narrowest" possible width of such discontinuities and overcome flow-resistances, in order to reach the maximum possible internal volume of masonry voids. The limiting factors appear to be the rheology (flow properties) and filtration tendency (plug formation) of the grout (Eklund and Stille, 2008). Both must be optimised to attain adequate penetration of the grout. According to several researchers [(Axelsson, et al., 2009),(Eriksson, 2002)], yield stress is one of the important rheological parameters in predicting penetration capability of fresh grout given that no plug formation has occurred. The filtration tendency is a characteristic (Fig. 1) whereby a plug of particles can be formed at a void opening or in a constriction within the void/channel preventing further penetration (Eklund and Stille, 2008).

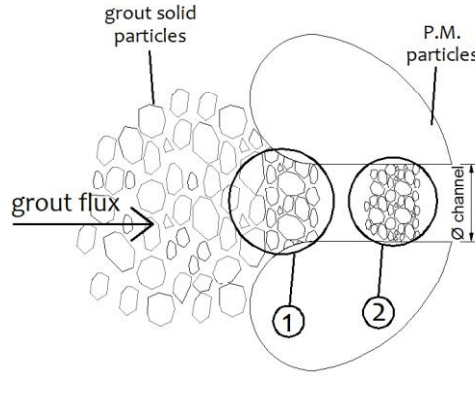


Fig. 1 Plug formation at the entry to a void (1) and obstruction of the void (2). Adapted from (Eklund and Stille, 2008)

The filtration tendency is related to the grain-sizes of the grout. There are various ways of describing directly or indirectly the grain-size distribution. One common method refers to the use of the d_{95} value, which corresponds to the mesh size of a sieve through which 95% of the binder material passes. This value is also used for describing the maximum grain-size, which in reality may not be correct. Since, it should be taking into account the increased tendency for small particles to flocculate into larger agglomerates (Eklund and Stille, 2008), when compared to larger grain-sizes. In fact, small grain-sizes are also important in what refers the analysis of filtration tendency of the grout.

2.1.2. Apertures calculated by different methods

The flow path through the PM can be described by means of porosity, pore size distribution, hydraulic conductivity or theoretical aperture. The advantage of using the latter is that it can be easily compared to the particle size of the grout, thus making it easy to describe the injectability.

Two different methods have been used in order to determine the theoretical aperture with sands b . One method is to use the Kozeny–Carman equation (Carman, 1956), see Eq. (1). The porosity n is determined for each porous medium, as is mentioned in the section 0, the specific surface S (mm^2/mm^3) is determined according to Axelsson (Axelsson, et al., 2007) (Table 8). The shape factor constant (to take into account the shape and tortuosity of channels) C_3 was set at 0.2. This value included simultaneously the notions of equivalent capillary channel cross-section and tortuosity.

$$b_{K-C} = \frac{n}{(1-n) \times S} \times \sqrt{12 \times C_3} \quad (1)$$

Since this equation was developed considering the total available volume between the particles of porous medium, it can be considered as a model for determining the available aperture for a Newtonian fluid. The Kozeny–Carman equation was developed for hydraulic characterisation of the sand (in the present work the PM studied were considered as sand) and implies that a Newtonian fluid (water) is used. However, since grouting is generally performed with a Bingham fluid, there is a rheological difference. According to Axelsson (Axelsson, et al., 2009), b_{eqv} for a Bingham fluid can be expressed using the porosity (n) and specific surface (S):

$$b_{eqv} = \frac{8}{\pi \times S \times (1-n)} \quad (2)$$

This expression was developed for a Bingham fluid and, therefore, it is not describing the same available aperture as the previous equation, as illustrated in Fig. 2. The shaded area in the right of the figure represents the area available for a suspension (Bingham fluid) whereas closer to the contact point between the porous medium particles, only water (Newtonian fluid) will be able to penetrate. Thus, the penetrability of a Newtonian fluid is in the range of 3-5 times bigger than for a Bingham fluid, such as suspensions (Axelsson, et al., 2009). This means that b_{eqv} should be in the range of 3-5 times larger than the apertures developed for Newtonian fluids (b_{K-C}).

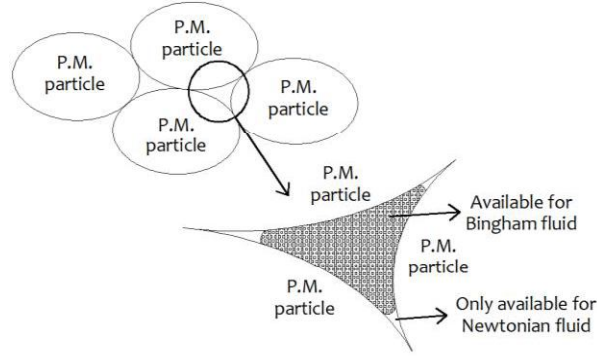


Fig. 2 The available area between PM particles for a Newtonian fluid (e.g. water) and a Bingham fluid (suspension). Adapted from (Axelsson, et al., 2009)

In the field of soil grouting, the penetrability of hydraulic grouts has been studied by a number of authors, e.g. Mitchell (Mitchell, 1982) who established in 1982 several rules of thumb for the injectability of PM. More recently, the same rules were used by Axelsson (Axelsson, et al., 2009). The use of these rules for the present PM leads to the results presented in Table 1.

Table 1 Rules of thumb of Mitchell (Mitchell, 1982) for injectability as a function of the grain distribution for PM

Criteria	Grouting consistently possible	Grouting not possible
$\frac{D_{15}^{soil}}{d_{85}^{grout}}$	> 25	< 11
$\frac{b_{K-C}}{d_{95}^{grout}}$	> 5	< 3

Notation: D_{15} = diameter of the soil grain, corresponding to 15% passing
 d_{85} = diameter of the grout grain, corresponding to 85% passing
 d_{95} = diameter of the grout grain, corresponding to 95% passing

2.1.3. Different criteria to evaluate the penetrability of the grout

As already stated in different literature [(Axelsson, et al., 2009), (Eklund, 2005), (Miltiadou-Fezans and Tassios, 2012), (Paillère, et al., 1984)], for a granular suspension (such as hydraulic lime grout) to be able to penetrate in a certain PM, the grain size distribution of its solid phase should be compatible with the characteristic dimensions of the PM (apertures, voids, interfaces, etc) to be injected. Thereby, penetrability conditions are frequently expressed in terms of the ratio (n) between the size of the larger solid particles of the grout (d) and a “representative” diameter of channels or a width of channels to be injected (W_{nom}). This ratio expresses the practical need of the grout solid particles to be significantly smaller than the characteristic aperture to be penetrated.

Several phenomena are behind this ratio, namely the friction that exists due to the irregular form of the solid particles, the electrostatic connections between particles and the agglomeration due to immediate hydration of the fines (Miltiadou-Fezans and Tassios, 2012). In last decades different authors have established different relationships (criteria) in order to assess the penetrability of hydraulic grouts. More recently, Miltiadou-Fezans and Tassios created one table (Table 2) which gives the different criteria the same format: $d < W_{nom} / n$. According to the same author, when W_{nom} is not known, as in the case of granular media, it is possible to assume the approximation: $W_{nom} \sim 0.15 \times D_{15}$. In accordance with Dantu (Dantu, 1961), this value corresponds to the diameter of the smallest path passing through grains of the same size D_{15} .

Table 2 - Grain penetrability conditions, according to literature. Table adapted from Miltiadou-Fezans and Tassios (Miltiadou-Fezans and Tassios, 2012)

Author	Criterion: $d < W_{nom} / n$	Grouted medium
Johnson (Johnson, 1958)	$d_{85} < W_{nom} / 3,75$	Fine granular soil
Mitchell (Mitchell, 1970)	$d_{100} < W_{nom} / 3$	Fissured medium
Littlejohn (Littlejohn, 1983)	$d_{85} < W_{nom} / 3,75$	Fine granular soil
Littlejohn (Littlejohn, 1983)	$d_{100} < W_{nom} / 5$	Fissured medium
Hutchinson (Hutchinson, 1981)	$d_{max} < W_{nom} / 3$	Fine granular soil
Cambefort (Cambefort, 1977)	$d_{100} < W_{nom} / 1,5 \text{ to } 2$	Fissured medium
Léonard (Léonard, 1961)	$d_{85} < W_{nom} / 0,75 \text{ to } 3$	Fine granular soil
Papadakis (Papadakis, 1959)	$d_{100} < W_{nom} / 1,5 \text{ to } 3$	Fine granular soil
Paillère & Guiney (Paillère, et al., 1984)	$d_{100} < W_{nom} / 1,5 \text{ to } 2,3$	Tests in "sand column"
Miltiadou-Fezans (Miltiadou-Fezans, et al., 2012)	$d_{85} < W_{nom} / 5 \pm 1$	Tests in "sand column"
Miltiadou-Fezans (Miltiadou-Fezans, et al., 2012)	$d_{99} < W_{nom} / 2$	Tests in "sand column"

Notation: d_{85} = diameter of the grout grain, corresponding to 85% passing

$d_{100} = d_{max}$ = "maximum" diameter of the grout grains

From Table 2, it should be emphasized that the criteria from Paillère and Guiney (Paillère, et al., 1984) and Miltiadou-Fezans and Tassios (Miltiadou-Fezans and Tassios, 2012) are resulted of the evaluation of the grout penetrability using sand column tests (used in the standard NF P18-891). These authors studied in a more precise way the relationship that exists between the grading of the solid phase of the grout and the penetrability of hydraulic grouts. In this way, in the experimental results of Miltiadou and Tassios (Miltiadou-Fezans and Tassios, 2012), a great variety of solid phases (of materials commonly used in the composition of hydraulic grout) was taken into account for the formulation of penetrability grading criteria. The criteria are: $d_{85} < W_{nom} / 5 \pm 1$ that relates d_{85} and W_{nom} and studies the phenomenon "wall flocculation blocking effects". The other criterion $d_{99} < W_{nom} / 2$ aims to ensure that the few grains with a size of " d_{99} " do not produce any "friction blocking".

2.1.4. Relation between penetrability and yield stress

According to the conclusions of some authors [(Baltazar, et al., 2013), (Bras, 2011), (Eriksson, 2002)], yield stress can be associated to the ability of the grout to fill the voids and its ability to flow when a given shear stress is applied. The knowledge of the yield stress τ_0 enables to understand if a fluid will flow or not, since it represents that threshold. According to Buckingham Reiner equation (Bras, et al., 2012), the shear stress (τ) at the wall of the cylindrical channel will be:

$$\tau = \frac{\Delta P}{L} \times \frac{D}{4} \quad (3)$$

Where D is the diameter of the void, ΔP is the difference of injection pressure in the channel and L the length of the channel. Since the injection pressure is constant, the shear stress at the wall will decrease when the grout penetrates the channel because L , the length filled by grout, is increasing. When shear stress at the wall is lower than the yield stress of grout the flow will stop. This is expressed by the following equation:

$$\frac{\Delta P}{L} \times \frac{D}{4} \leq \tau_0 \quad (4)$$

Knowing the maximum L of the injection tests, the pressure adopted in the injection tests (see the section 4.2) and the yield stress (see the section 3.1), according to equ. (4) it is possible to obtain the minimum void diameter (D_{\min}) of the porous media to be injected. It is important to emphasize that two of various assumptions followed by Buckingham equation are: the grout flow does not change in time and occurs inside a void with the shape of a cylindrical tube. Given the heterogeneity (variability in size of the apertures) of PM studied in this work, these assumptions are hardly respected.

3. Materials studied

3.1. Grout design

Grout design involves the study of its behaviour in the fresh state, requiring some performance characteristics such as high fluidity, good water retention, stability and limited or no bleeding [(Toumbakari, et al., 1999),(Valluzzi, 2005), (Miltiadou-Fezans and Tassios, 2013)] in order to maximize the injectability of grout in the PM, thus maximizing the penetration and diffusion of grout (Valluzzi, 2005). All these characteristics have their particular role in the success of the grout injection. If they are not satisfied, the grout will hardly be injectable regardless of PM [(Kalagri, et al., 2010), (Miltiadou-Fezans and Tassios, 2012)]. In order to analyse these characteristics several tests based on the following experimental procedures were performed.

Grout fluidity can be evaluated by its rheological behaviour at fresh state, which could be characterised by several rheological parameters including yield stress and plastic viscosity. From a practical point of view, yield stress is associated to the ability of the grout to fill the voids and to flow when a given shear stress is applied. The knowledge of the yield stress enables the understanding if a grout will flow or not, since it represents a threshold value, meaning that as long as the applied stress is below this value the grout does not flow (Miltiadou-Fezans and Tassios, 2012). On the other hand, plastic viscosity represents the flow resistance once flow is initiated. The lower the grout viscosity the easier and faster the grout will flow and therefore the grout will lose less mixing water by absorption (Van Rickstal, 2000). The rheological measurements were performed using a Bohlin Gemin HR^{nano} rotational rheometer equipped with a plate-plate geometry. Knowing the behaviour of hydraulic lime grouts as a shear-thinning fluid [(Baltazar, et al., 2013), (Bras, et al., 2009)] the Bingham model was used to fit the experimental data in order to estimate the plastic viscosity and yield stress. Another parameter to characterize the fluidity of grouts is the flow time, which was measured through the procedure of funnel flow time (Marsh cone test), according to standard ASTM C939-02. Based on this standard the measurement of flow time is connected to the grout fluidity; the longer the flow time, the lower will be the grout fluidity. However, some authors, e.g. Bras (Bras, 2011), concluded that Marsh cone is inadequate for grout design for the injection of a porous media where fine material is present (< 4mm), as is the case of

certain PM studied in this work. Consequently, the measurement of flow time with a Marsh cone with d=10mm seems to be inadequate in case of grouts designed to fill fine voids, as it is not sufficiently sensitive. For this reason, Miltiadou-Fezans and Tassios (Miltiadou-Fezans and Tassios, 2012) used in their tests a Marsh cone with 3mm nozzle-diameter. In order to improve the physical significance of Marsh-cone test, Miltiadou-Fezans and Tassios propose the fluidity factor test (FFT). This test has some changes relative to standard ASTM C939-02, namely the fact the flow time being measured for a flow of only $Q=100\text{cm}^3$ instead of conventional 800/1000 cm^3 used in different works [(Bras, 2011), (Baltazar, et al., 2014)]. The author argues that by this way the fluid pressure acting on the nozzle is practically kept constant during the test. Moreover, the influence of the roughness of cone's walls is minimized. The concept of a fluidity factor F_l is obtained by following the equation:

$$F_l = \frac{Q}{A \times t_f} \quad (5)$$

where A denotes the area of the cross section of the nozzle, t_f is the flow time. Based on this equation, it is possible to state that "more fluid grouts are characterized by higher F_l values, i.e., higher velocities of flow" (Miltiadou-Fezans and Tassios, 2012).

Concerning stability, the segregation of solid particles or excessive bleeding should be avoided since otherwise blockage may soon appear and the quality of the grouting intervention could be severely affected (Valluzzi, 2005). In this research the stability of the grouts was analysed by the bleeding test, based on standard ASTM C 940. According to this standard 800 ml of freshly mixed grout was poured into a 1000 ml graduated glass cylinder and covered. The height of bleed water was noted after complete sedimentation (three hours after the grout mixture). The final bleeding was calculated according to the expression:

$$\text{Final Bleeding}(\%) = \frac{V_w}{V_l} \times 100 \quad (6)$$

where, V_w = volume of decanted bleed water, ml; V_l = volume of sample at beginning of test, ml.

An excess of bleeding in a grout means that there is a substantial amount of free water on the surface of the unset grout. According to Toumbakari (Toumbakari, 2002) a grout has a good behaviour if the bleeding is less than 5%. Thus, in accordance with Table 6, it is possible to conclude that in terms of stability, the grout chosen presents a good behaviour. However, it should be noted that this test only gives discrete results that do not allow to check the evolution of density gradient of the grouts. Consequently, a new test procedure - the stability test - was developed, based on the test proposed by Van Rickstal (Van Rickstal, 2000) aimed at checking the density variation along the test time of a grout in resting conditions. The analysis of the results was done with the coefficient of variation of density throughout the test in relation to the initial density. So, a small coefficient of variation represents a low variation of grout density, consequently meaning reduced segregation and bleeding.

The water retention capability is another important property to be assured in order to maximize the injectability of grout, since it represents the ability of a grout to retain the mixing water during the injection inside dry and high absorptive masonries. The ability to

preserve water within grout suspension for the longest possible time will allow to maintain good rheological behaviour and grout stability in order to ensure a successful injection. The measurement of water retention was performed in accordance with ASTM C941-02.

The grout composition used in the injection tests (shown in Table 3) takes into account the findings reported in the literature [(Valluzzi, 2005), (Miltiadou-Fezans and Tassios, 2012)] and previous research of the team [(Bras, et al., 2012), (Baltazar, et al., 2012) and (Baltazar, et al., 2013)]. In these works, the materials studied were the same (binder and superplasticizer). The main goal was to assess the fluidity, stability and penetrability characteristics in function of the water to solids ratio and/or the percentage of SP.

Table 3 - Grout composition tested

Binder	W/b	SP	% SP
NHL5	0,5	Glenium Sky 617 (BASF)	1.2

The binder used was the NHL5 hydraulic lime (EN459-1) produced in Portugal by Secil-Martingança, which has the characteristics presented in Table 4 according to the information of the quality control system provided by the manufacturer. The grain size distribution is represented in Fig. 3. From this curve, it is possible to obtain d_{85} (98 μ m), d_{95} (129 μ m) and d_{99} (206 μ m) values.

Table 4 - Density and fineness of NHL5 using Blaine permeameter

Sample	Density (g/cm ³)	Fineness (Blaine) (cm ² /g)
NHL5	2,7	9400

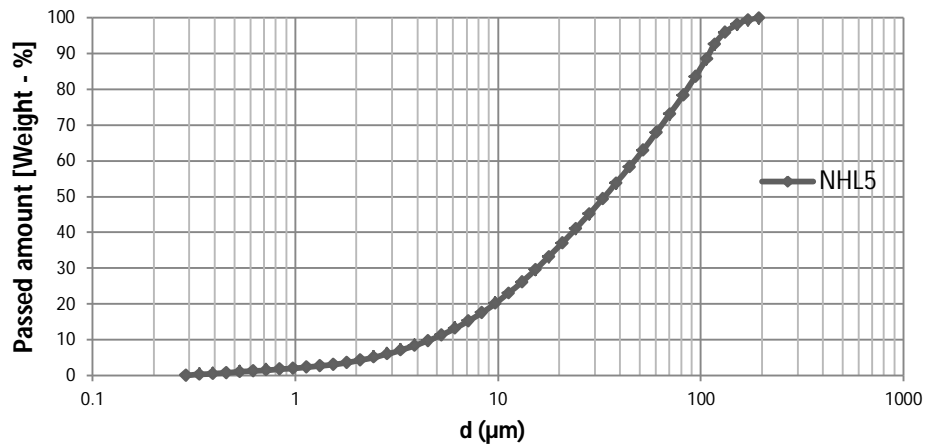


Fig. 3 - Grain size distribution of NHL5 used in the injection tests

Table 5 presents chemical characterizations of NHL5 according to XRF results. The superplasticizer was BASF Glenium Sky 617.

Table 5 - Chemical characterization of NHL5 according to XRF results

Compound Name	Conc. (%)
MgO	0,99
Al ₂ O ₃	2,96
SiO ₂	10,86
SO ₃	1,97
K ₂ O	0,89
CaO	78,97
TiO ₂	0,30
MnO	0,04
Fe ₂ O ₃	2,96
SrO	0,06

The water/binder ratio (w/b) tested was 50% in weight (Table 3). According to the literature and particularly following the recommendations proposed by Valluzzi (Valluzzi, 2005), a minimum value of water/binder of 55% (in weight) should be used, although in this case only 50% was used due to the presence of the superplasticizer.

Concerning the use of the superplasticizer it became evident throughout the test campaign that it was virtually impossible to formulate a grout with adequate injectability without using either a superplasticizer or an excessive amount of water that would lead to a grout with stability problems of bleeding or segregation. Analogous results were obtained by Valluzzi (Valluzzi, 2005). After various preliminary attempts with different brands and dosages of superplasticizer (Baltazar, et al., 2013), a polycarboxylate-based superplasticizer was chosen. This superplasticizer belongs to the third generation whose repulsion is a combination of coupled steric and electrostatic effects, known as electrosteric repulsion, which contributes to an increase of the distance between solid particles. Therefore, the addition of a superplasticizer results in lower particles flocculation and segregation and improves the rheological parameters, by reducing both plastic viscosity and yield stress [(Baltazar, et al., 2012),(Vikan, 2005)].

Comparing the injectability characteristics of the grout chosen with hydraulic lime grouts tested by other authors (Table 6), it can be concluded that this grout has a high performance in terms of fluidity, stability and water retention. **It should be stressed that the materials and tests used in this article are the same as in other works mentioned in Table 6.**

Table 6 Injectability characteristics of the grout selected in comparison with literature

	Parameters / Tests	Grout selected	Literature
Injectability characteristics	Yield stress [Pa], resting time = 0 s	0.63	1.04 (Bras, et al., 2012); 12.74; 0.47 (Baltazar, et al., 2012)
	Plastic Viscosity [Pa.s], resting time = 0 s	0.10	0.15 (Bras, et al., 2012); 0.057 (Baltazar, et al., 2012)
	Flow time (s) (Marsh cone test Diam.=10 mm), resting time = 0 s	9.3	9.1 (Baltazar, et al., 2014); 22 (Bras, 2011)
	FFT (mm/s x 10 ⁻³) (Marsh cone test Diam.=6 mm), resting time = 0 s	1.5	0.1-2.2* (Miltiadou-Fezans, et al., 2012)
	Final Bleeding (Stability Test) [%]	2.1	0.33 - 4.8 (Bras, et al., 2012)
	Coefficient of variation of density (Stability Test) [-]	0.07	0.12 - 0.31 (Baltazar, 2012) (Van Rickstal, 2000)
	Water retention capability (time needed to remove 30 ml of water) (sec)	3582	1626 (Baltazar, et al., 2012); 1650-3574 (Baltazar, 2012)

* Marsh cone diameter = 3mm

3.2. Porous media for injection tests

In order to study the grout injection capacity some injectability tests were made. Given the difficulties to reproduce a historical masonry due to their high heterogeneity (Valluzzi, 2005) and to the difficulty of reproducing the characteristics of ancient mortars [(Binda, et al., 1997), (Almeida, et al., 2012)], masonry samples were simulated by combining three different crushed limestone sands (hereafter mentioned as stone) and three different crushed bricks (hereafter mentioned as brick) (Fig. 4). The same method but with different materials was used in works of other authors [(Bras, et al., 2012),(Valluzzi, 2005),(Van Rickstal, 2000)]. The materials were washed, dried and sieved to obtain diverse grain size distributions to enable the simulation of different

permeability of masonries. Five different grain size media types were adopted to simulate different masonries (Table 7 and ig. 5).



Fig. 4 Three different grain size ranges (fine, medium, coarse). Limestone sand (left picture) and Crushed brick (right picture)

Table 7 Different PM studied

Porous Media	Grain size ranges		
	0.15 - 2mm (<i>fine</i>)	2- 4.75mm (<i>medium</i>)	4.75 - 9.5mm (<i>coarse</i>)
A	1/3	1/3	1/3
B	1/3	-	2/3
C	1/6	-	5/6
D	-	1	-
E	-	1/2	1/2



Fig. 5 - PM A_{stone} (left picture) and E_{stone} (right picture)

In accordance with the survey of the sections of multi-leaves masonry done by certain authors [(Bras, 2011), (Binda, et al., 1997)] some important parameters were adopted (Table 8) **to characterize the dimension and distribution of voids of the different PM.** These parameters are: the voids size average (which correspond to d_{50} - the diameter through which 50% of the total mass passes) (Bras, et al., 2012), as well as the parameter $d(90)$, $d(15)$ and $d(10)$ (respectively the diameter through which 90%, 15% and 10% of the total mass passes) and the % of the total mass that passes through ASTM n°20 sieve (0.85mm). **The parameters above mentioned are obtained from grain size distribution curves shown in Fig. 6. The impact of these parameters on injectability value will be analysed in the section 5.1.1. Furthermore, the grain size distribution of each PM (Fig. 6) allows the determination of nominal lower value of the aperture of voids or interfaces to be injected. Since the calculation of the aperture depends of the specific surface value (shown in Table 8) and the latter is calculated from the grain size distribution of each PM.**

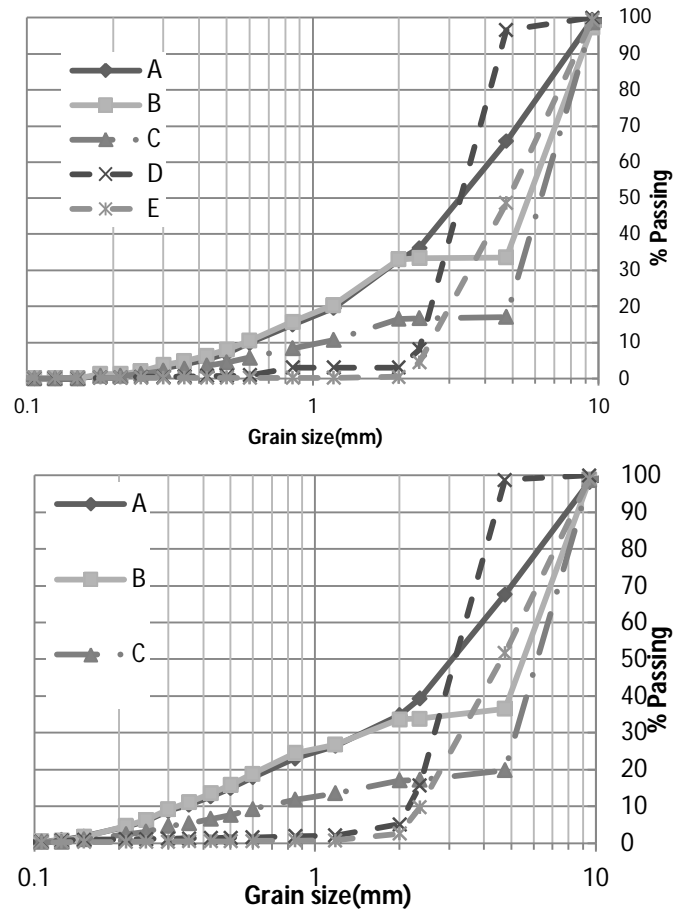


Fig. 6 - Grain size distribution for media types A, B, C, D, E used for cylinders grout injection. Limestone porous media (above picture) and crushed brick porous media (below picture)

According to other authors [(Bras, et al., 2012), (Valluzzi, 2005)], the total porosity of each PM type was evaluated by measuring the volume of water that could be injected inside each cylinder (Table 8). It is important to note that this parameter does not give the value of porosity that the grout can penetrate inside the PM, which in fact is much smaller. In section 5.1.5 is explained why this happens. As shown in Table 8 the percentage of total porosity measured was approximately between 40-55%, which is a typical range of percentages in research of masonry walls, if only the dimension of the inner core are computed (Valluzzi, 2005).

Table 8 PM characteristics

	Porous media type									
	A		B		C		D		E	
	stone	Brick	stone	Brick	stone	Brick	stone	Brick	stone	Brick
Voids size average [mm]	2.67	2.42	5.23	5.02	6.09	5.97	3.22	3.06	4.28	4.09
d(90) [mm]	8.17	8.22	8.97	8.82	9.00	8.97	4.58	4.50	8.62	8.59
d(15) [mm]	0,85	0,50	0,81	0,47	1,78	1,52	2,55	2,34	2,93	2,66
d(10)[mm]	0.60	0.34	0.58	0.32	1.08	0.67	2.41	2.17	2.66	2.38
% of the total mass that passes through n° 20 sieve	15.0	23.0	15.7	24.4	8.4	11.8	3.1	1.8	0.3	0.7
P.M. porosity [%]	41.2	48.1	39.3	48.7	44.6	51.5	50.4	56.6	48.4	55.4
WA (%)	5.6	19.4	3.9	19.1	2.2	16.2	2.2	14.3	1.5	12.5
Specific Surface (mm ² /mm ³)	5,03	6,06	4,90	5,84	3,17	3,38	2,86	2,63	2,02	1,72

To study the water absorption (WA) capacity of each PM the European standard EN 1097-6 was used. From Table 8 is possible to observe the high water absorption capacity of brick PM when compared to the limestone PM, which is in accordance to the literature (Cachim, 2009). The knowledge of water absorption capacity of the particles of PM is of utmost importance during the injection of grouts. This allows a perception of the amount of water absorbed by the particles of PM during the injection process, which influences the grout fluidity and consequently the grout injection capacity. The results show that brick PM create more resistance to the flow of grout.

4. Procedure

4.1. Mixing procedures

The hydraulic lime mixes were prepared at room temperature $22\pm1.5^{\circ}\text{C}$ and 53% of relative humidity. For the preparation of grouts ordinary tap water was used and dry hydraulic lime was hand mixed to ensure a homogeneous distribution before the beginning of the mechanical mixing. The mixing procedure was chosen in accordance with previous research of Baltazar *et al* (Baltazar, 2012). The adopted mixing procedure was the following: the whole binder is added to 70% of total mix water and mixed during 10 min. The remaining water (with diluted SP) is added within 30 s (without stopping the mixer). After all materials had been added, the mixing was maintained for 3 min at 800 rpm. The mixer cup had a capacity of 5 litres, with 177 mm diameter and a height of 244 mm. The blade used had a helicoidally shape and the gap at the bottom between the blade and the cup was $4\text{ mm} \pm 1\text{ mm}$. Each grout sample was passed through a 1.18 mm sieve (n. 16 ASTM) before being injected into the cylinders.

4.2. Injection Tests

Injectability tests were performed to study the penetrability of the grouts. Since it is hard to reproduce a real masonry and it is difficult to visualize what is happening inside the PM being injected, simplified models were created to analyze the penetration of the grout in the masonry. The models involved the use of transparent Plexiglas cylinders with diameter 152 mm and height 300 mm, as in ASTM C943. They were filled with one of the media types trying to reproduce as much as possible real situations. For each PM three samples were used, i.e., three cylinders were filled, for a total of 60 elements. Following the assumptions of Van Rickstal (Van Rickstal, 2000), the cylinders were filled in three fractions (10cm per fraction). At each fraction, they were densified by vibrating the cylinder. At the bottom of the cylinder, a distributing layer of coarse grains ensures a good distribution of the grout. For injection purposes a device based on previous works [(Binda, et al., 2003) ,(Bras, et al., 2012) and (Valluzzi, 2005)] was used (Fig. 7). The filled cylinders were injected with the fresh natural hydraulic lime grouts immediately after grout preparation. Injections tests were performed at constant pressure of 1 bar, with the gout being injected unidirectionally from bottom to top [(Bras, et al., 2012),(Valluzzi, 2005)].

It is worth noting that these injection tests do not simulate the injection within the masonry, but offer the possibility of experimentally:

- evaluate the minimum fineness of the PM that allows the chosen grout to be injected;
- understand the grout flow inside PM for the different grain size distributions used;

- observe the relationships between injection capacity of grout and media grain size distributions.

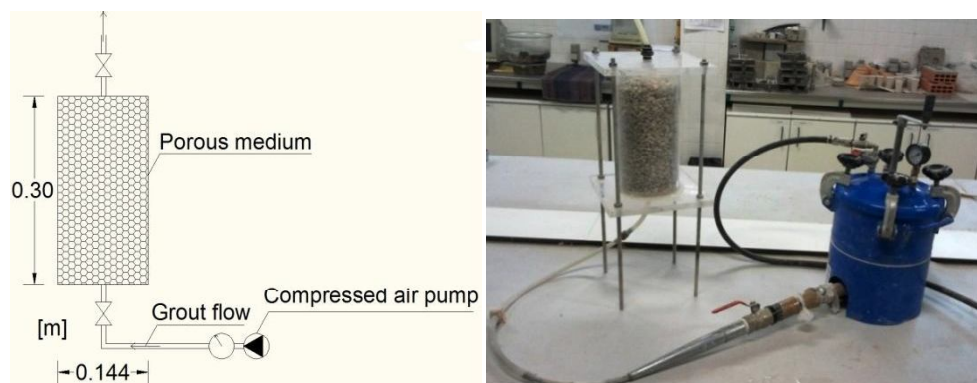


Fig. 7 Setup for injection tests used in lab

In the field of hydraulic grouts for injecting historic masonry structures, systematic research has been undertaken, using the standardized sand column test (NF P18-891, EN 1771), that was conceived by Paillère and Rizoulières in the Laboratoire Central des Ponts et Chaussées (LCPC, France). The test was developed initially for the control of injectability of polymers for the repair of concrete structures (Paillère, et al., 1978). These studies conducted in LCPC, demonstrated that the fulfilment of a "groutability ratio" criterion is not sufficient to ensure penetrability into very fine cracks of masonry. These studies proved that the upper part of the grading curve of the solid phase of the grout has to comply with specific grading criteria, depending on the granularity of the sand to be injected (Miltiadou-Fezans and Tassios, 2012). Thus, using this standard the goals intended in the present study are reached, however this was not used due to three reasons: a) the diameter of the column (22.2mm) is too small for aggregate sizes studied in this work (especially the coarser porous media with size between 4.75-9.5mm); b) due to geometric configuration of the column, the path of the grout during the injection is only one-dimensional, rather than three-dimensional, as in the case of adopted model and reality; c) the material used in the standard is sand which is a material with low water absorption capacity and with low roughness, such characteristics are very different regarding the materials that compose old masonries. Nevertheless, the concepts of the standard regarding the penetrability of the grout are respected along the present article.

4.3. Injection capacity of the grout

According to Kalagri (Kalogri, et al., 2010), the injectability capacity of the grout constitutes a key parameter for a successful intervention. Thus, in present work the goal was to get the injection capacity of the grout as a function of different PM. Each PM is characterized by porosity, grain size distribution and voids size average (Table 8). In the absence of a formal quantitative definition for injectability, Bras created an equation to quantify the injectability of the grout (Bras, et al., 2012), taking into account the quantity of the grout injected (through the height of injection) and the time of injection (rate of injectability of the grout). In the present work, it was decided to express the concept of the injectability as the ratio between the volume of grout injected (m/ρ) and the volume available to grout injection inside the PM (V_v). Defined in this way, injectability can be expressed in l/m^3 (litres of injected grout per cubic metre of voids to be injected), dimensionless (if the volume of the grout is expressed in m^3) or in percentage of the total

volume to be injected. Thus, the following expression for grout injectability (at a given injection pressure) is proposed:

$$I = \frac{m}{\frac{\rho}{V_v}} \quad (7)$$

where I is the grout injectability (-), m the injected mass during the injection process (kg), ρ the density of grout (kg/m³) and V_v is the voids volume of PM (m³). By measuring the weight of the cylinders before and after injection it was possible to determine (by knowing the density of the grout) the quantity (volume) of injected product. Additionally, knowing the volume of voids (measured by the saturation of water before injection), it was possible to calculate the effective performance of injection in each PM.

In the section 5.1.1 and 5.1.2, the equations aforementioned are used and the results obtained are analysed.

4.3.1. Porous media with different moisture content

Since it is not expected that masonries are always dry, the media of some cylinders were pre-wetted by simple injection of water (in accordance with experiments of Valluzzi (Valluzzi, 2005), Van Rickstal (Van Rickstal, 2000) and Anzani (Anzani, et al., 2006), as shown in Fig. 8. After the injection of water the valve at the bottom of the cylinder was opened to allow the water to flow out of the sample. Half an hour later the same sample was injected with the hydraulic lime grout. It was noticed that water injection washed out the finer particles, creating major flow channels. Injection tests for the five media types were done with and without pre-wetting of the PM. Through the comparison of the values of injectability for these two categories it is possible to evaluate the effect of the water content of PM on the injectability of the grout. Some authors, like Bras (Bras, et al., 2012) and Van Rickstal (Van Rickstal, 2000), noted the influence of water content of PM to be injected as a factor able to influence grout injection capacity.



Fig. 8 Cylinders filled with media type E_{stone} (left), A_{stone} (central) and A_{brick} (right) being injected by water. The flow was uniform but with different velocities of injection

5. Results and discussion

5.1. Injection tests

5.1.1. Injection capacity of grout for the different porous media

The main objective of this research was the comparison of the performance of the selected grout in terms of its capacity of penetration and injection in different PM. Injectability (calculated by Eq. 7) was analyzed for two different situations: (i) grout in dry PM and (ii) grout in pre-wetted PM. From Table 9 it is possible to observe that coarser PM (C, D and E) with higher porosity and higher size of finer particles - $d(10)$ (Table 8) - have injectability values roughly 1,5 times higher than those of the finer PM (A and B). This is observed especially in limestone PM. In what concerns brick PM, this difference is slighter and PM C shows an intermediate behaviour between fine and coarse PM. Comparing the injectability of PM between these two materials, in general terms it is observed that there is no great difference. The total mean-square-deviation (MSD) for each material confirms this fact (Table 9). On the one hand, the crushed brick PM have higher porosity (Table 8) which leads to higher mass/volumes of grout to be injected; furthermore, the particle surfaces have lower roughness, which cause a decrease of the resistance to the injection flow (this phenomenon is more pronounced in a fluid more viscous like grout than in the water). But has significantly higher water absorption (Table 8) provoking an increase of the overall resistance to the grout flow. This is due to the increase in viscosity and a decrease of the aperture (Van Rickstal, 2000) resulting from the absorption of water by the PM that renders a good penetration more difficult to achieve. In what refers to finer PM (A and B), for both materials, PM B (with only fine and coarse size particles) present lower injectabilities than PM A (with the three different range particles size) (Table 7). The reason is related with the fact that during injection, when a grout reaches a large void, no pressure can be built up in the neighbourhood of that void. Due to this low pressure, the grout will enter the fine channels only over a short distance, with thixotropy, water absorption and instability of the grout causing the blocking for further injection in these finer channels. When the large void is finally filled, the pressure can increase again, but too much water of the grout is absorbed in the fine channels to restart flowing.

In terms of the injectability of dry or pre-wetted PM, the former present higher values for both of the materials studied. The bigger differences are present in crushed brick PM (see MSD in Table 9), especially for finer media. In this case, the parameter with the highest impact is the water absorption due to the higher specific surface that results from the finer media (Table 8). Since water absorption is higher in the case of crushed brick PM (Table 8), these PM absorb more water in the pre-wetting phase, leaving a lower free volume for the grout to be injected. Thus, their injectability values will decrease.

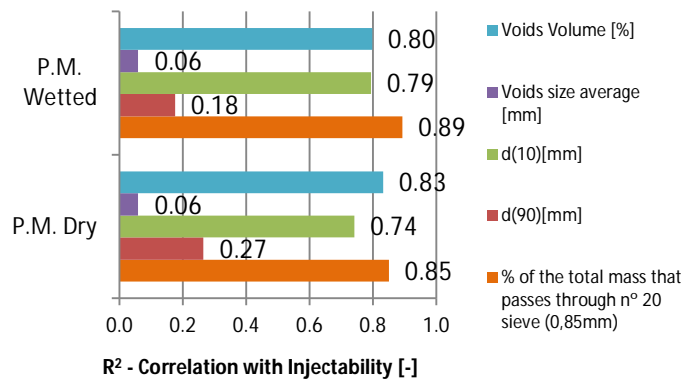
Table 9 Injectability (-) and Volume injection (l) for different PM

	Injectability (-)					volume injection (l)			
P.M.	Lime stone		Crushed Brick		P.M	Lime stone		Crushed Brick	
	Dry	wetted	Dry	wetted		Dry	wetted	Dry	wetted
A	0,57	0,58	0,75	0,61	A	1,27	1,17	1,66	1,27
B	0,54	0,48	0,71	0,46	B	0,85	0,80	1,39	1,00
C	0,96	0,88	0,80	0,74	C	2,32	1,95	2,14	1,92
D	0,96	0,91	0,97	0,82	D	2,67	2,34	2,82	2,25
E	0,97	0,92	0,89	0,84	E	2,61	2,36	2,56	2,30
MSD*	0,08	0,10	0,04	0,11	Average	1,94	1,72	2,11	1,75
MSD*	0,09		0,08		Average	1,83		1,93	

$$* MSD = \frac{(y_1 - y_0)^2 + (y_2 - y_0)^2 + \dots + (y_n - y_0)^2}{n}; y_0 = \text{target value} = 1$$

From the analysis **Table 9** it is possible to observe a close relation with some of the PM characteristics presented in Table 8, namely with parameter d(10), % of the total mass that passes through n°20 sieve, and voids volume. According to **Fig. 9** these parameters are the most important for masonry characterization regarding injectability, independently whether PM is dry or wet at the time of injection. In fact three ranges of values for these parameters can be identified: one for the finer PM A and B, a second for the coarse PM D and E and a third to PM C that lies between the other two ranges (**Fig. 10**). The same happens with the values of injectability. Thus, the above parameters revealed in general to be adequate for the establishment of an injectability characteristic. The other parameters - voids size average and d(90) - did not seem appropriate since the correlation with injectability is very low (**Fig. 9**).

According to Hazen equation (David Carrier, 2003), the permeability is related to the parameter d(10). As injectability is related to the permeability of the porous medium, then it is possible to state that injectability is related to the parameter d(10) and not d(90), which was confirmed in results obtained in this work.

**Fig. 9** Correlation between Injectability and PM characteristics (taking into account the injection results of limestone and crushed brick PM)

The analysis of the relations between injectability and certain PM parameters is presented in Fig. 10, from which it can be seen that higher value of voids volume are associated with higher injectabilities. This association, however, can only be established for the same material. When the analysis is between different materials, there are other parameters, as the water absorption capacity, that may have a relevant preponderance. Thus, in Fig. 10 and Table 8 it is possible to observe that brick PM have a higher porosity. In what concerns the grout volume injected (Table 9) they are similar in both PM, confirming what was previously written about the Newtonian fluids (water, the fluid

that was used to determine the voids volume) able to penetrate into more voids than Bingham fluids (grout suspension). The higher the water absorption of a PM, the higher is the difference between grout injected and the voids volume.

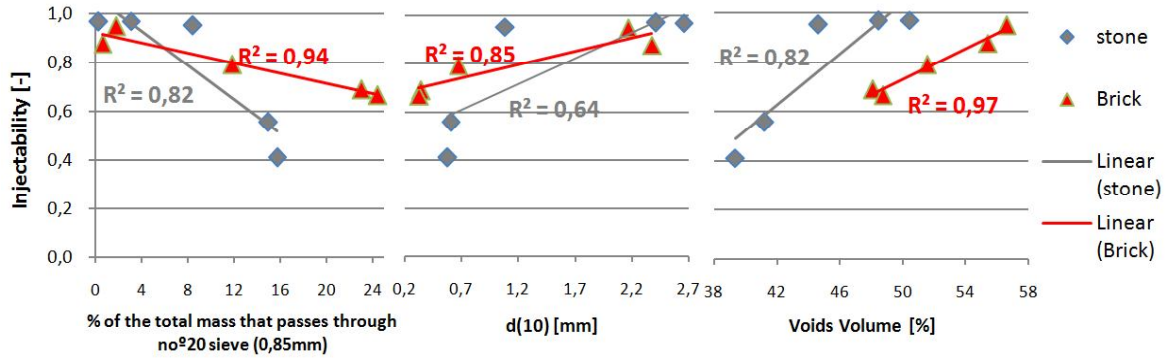


Fig. 10 Relation between Injectability values and certain parameters of the PM

5.1.2. Injection capacity of grout taking account the injection time

As mentioned in section 4.3, authors as Ana Bras followed a different approach to study the injectability of the grout (Bras, et al., 2012). Comparing with the equation proposed in this article, the main difference is the introduction of another parameter: the time of injection. Thus, following the approach of Bras, the rate of grout injectability has a great importance. Fig. 11 is an outcome of using this approach.

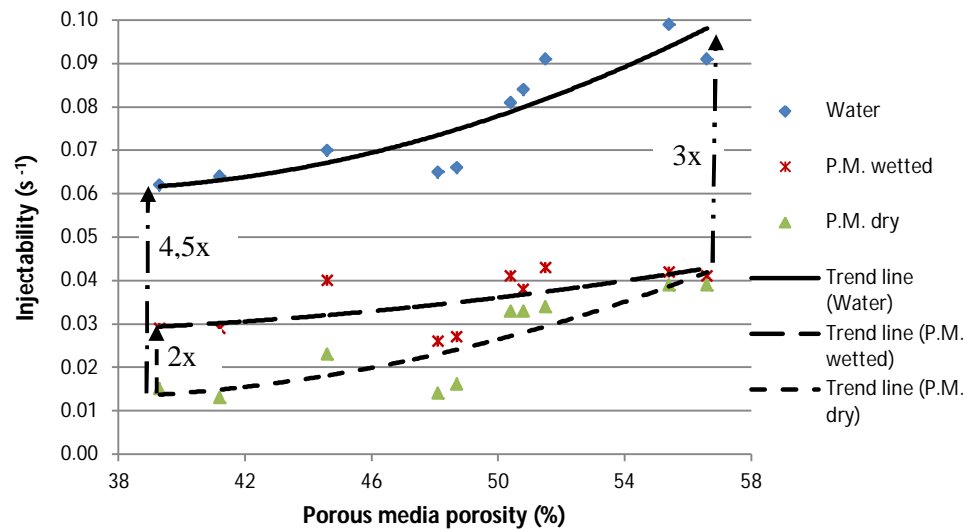


Fig. 11 - Injectability (calculated by the equation of Bras) curves for water, PM wetted, PM dry for the different PM tested, taking account the porous media porosity injected [%]

The injectability of grouts when compared with the results obtained with water presents significantly lower values (Fig. 11), in particular for the low porous media porosity, where the ratio between injection capacities reached 4,5 times. However, for higher porosity the ratio decreases to 3 times. These results fit with the conclusions obtained by Bras (Bras, et al., 2012). In fact, it seems that the yield stress value has more relevance in injectability of a fluid for finer PM, whereas for coarser PM, viscosity forces and inertia effects become more expressive.

From Fig. 11 it is possible to observe that coarser porous media with higher porous media porosity (Table 8) have values of grout injectability approximately 2-3 times higher than those of the finer porous media. The amount of grout injected, as well as the

rate of grout injectability are greater for the coarse porous media because the resistance to the grout flow is lower once the pore system has voids/channels with higher aperture (Table 10).

The grout injectability is higher when porous media is wetted at the time of injection (Fig. 11). This difference (in average about 37%) can be explained by considering that the resistance to flow has been reduced by the water injection, leading to the porous media with a higher conductivity (Van Rickstal, 2000), consequently higher velocity injection (i.e. rate of grout injectability). The higher differences are present in finer porous media due to higher water absorption capacity of finer particles (Table 8). Thus, in what concerns the rate of grout injectability, it is more beneficial to use pre-wetting in finer PM than in coarse PM. In fact, in these cases the grout will only flow through the larger voids, since at the time of grout injection the finer voids are already filled with water (during the pre-wetting these capillaries absorb water due to the high capillary pressure - *Kelvin law*), hence hindering the penetration of the grout.

In general the values of injectability (s^{-1}) obtained are higher than the values obtained by Bras (Bras, et al., 2012). One reason can be the higher porous media porosity and voids size average of the PM studied in this work (Table 8), compared to the PM used by Bras. Thus, the aperture of the voids in the pore system is higher which results in higher injectability of the grout. Other reason can be the better rheological behaviour of the grout used in injection tests compared to the grouts used by Bras (Table 6). Indeed, the lower viscosity and yield stress contribute to obtain a grout with higher fluidity and penetrability, respectively (Miltiadou-Fezans and Tassios, 2012).

5.1.3. Visual inspections during the injection of the cylindrical models

During the injection test a movie was made to allow the visual analysis of grout penetration inside the cylinders. The following remarks can be made: (a) while injecting the dry material a segregation took place between the water (absorbed by the finer material) and the remaining part of the grout (Fig. 12.a); (b) when the finer material formed a complete layer through the section of the cylinder, the flow was interrupted (Fig. 12.a and Fig. 12.c); (c) when an injection blocks, it is not possible to restart the flow by increasing the pressure; d) when the finer material does not exist or is just present in a small quantities the injection was successful (Fig. 13). These results are in accordance with the literature [(Bras, et al., 2012),(Valluzzi, 2005),(Kalagri, et al., 2010)].

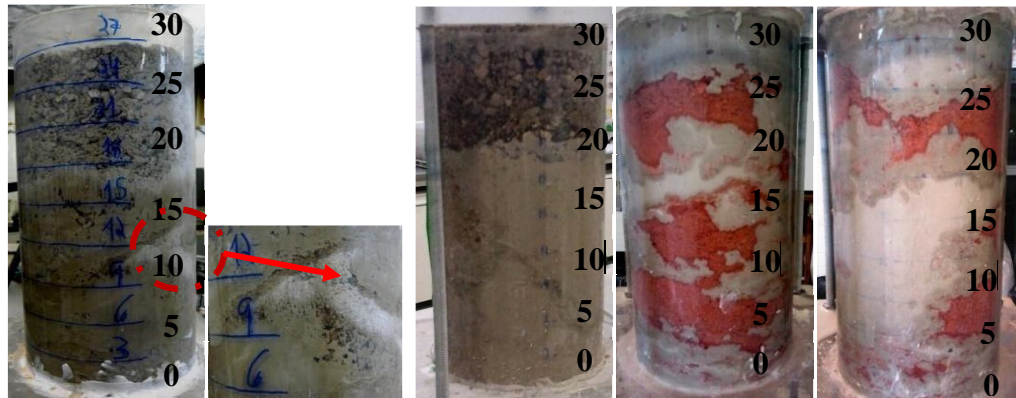


Fig. 12 Cylinders filled with media type a) and b) B_{stone} dry, c) B_{stone} wetted, d) B_{brick} dry and e) B_{brick} wetted after injection

From Fig. 12.a and Fig. 12.c it can be observed that both the height and the area of injection with the same limestone porous medium is higher when the porous medium is

wetted previously to the injection of the grout. One of the reasons is the reduction of flow resistance due to the water injection, leading to a porous medium with a higher conductivity. Van Rickstal (Van Rickstal, 2000) noted that the conductivity to water of a dry porous medium is smaller than the one of a wet sample, concluding that the conductivity of a porous medium is related to water content. Considering the results obtained the same phenomenon happens with the grout. In what concerns the crushed brick PM, it was noticed that the grout was able to advance until reaching the top but leaving part of the voids empty because the resistance to fill these voids is high (Fig. 12.d, Fig. 12.e and Fig. 13.d). Moreover, as explained above, in the crushed brick PM the water absorption is significantly higher, what hinders the grout injection process.



Fig. 13 Cylinders filled with media type a) $D_{\text{stone,dry}}$ b) $E_{\text{stone,dry}}$ c) $D_{\text{brick,dry}}$ and d) $C_{\text{brick,dry}}$ being injected. The flow was uniform but with different velocities of injection

The injection of PM A and B (PM composition with higher amount of fines) was not successful in laboratory, even when PM were wetted. Taking into account that the situation in the cylindrical containers is better than in reality from the point of view of injectability, given the fact that they have a larger number of connected voids when compared with real masonry (Binda, et al., 2003), it can be concluded that this grout will not be injectable inside a masonry with similar characteristics. However, these types of PM (A and B) only represent the cases where the voids cannot be directly reached (when the grout is not allowed to flow through paths with discontinuities or small openings) and/or when the width of the voids is not large enough when compared with the dimension of the grout's grains [(Miltiadou-Fezans and Tassios, 2012), (Valluzzi, et al., 2004)]. In contrast, the grout injection inside PM D and E has good penetration and diffusion of the grout. Regarding PM C, it was observed that the injection inside the cylinders of this PM were not complete, as shown in Fig. 13.d, Fig. 14 and Fig. 15. The reason for this fact is the presence of dry fine particles (with small size and high water absorption) which prevents the penetration of the grout flux. In section 5.1.5 this issue will be addressed in detail.

From these tests it becomes clear that pre-wetting cannot solve the penetrability issues which is in accordance with the injectability tests performed by Valluzzi (Valluzzi, 2005), where no differences were found in cylinders preliminarily wetted, in comparison with the anhydrous ones. Moreover, according to some authors [(Valluzzi, 2005), (Van Rickstal, 2000)] pre-wetting causes a lower mechanical strength of the samples. Therefore, pre-wetting has to be used with much precaution.

5.1.4. Visual inspections after injection of the cylindrical models

The cylinders of Fig. 14 were cut into three slices (bottom, medium and top levels) 45 days after being injected and an inspection of each slice was made (Fig. 15) to assess the

degree of success of the injection in terms of penetration and diffusion of the grout. A remark can be made regarding the presence of high quantity of zones not injected during the injection of PM with finer material (PM A and B), leaving large voids as shown in Fig. 14 and Fig. 15.

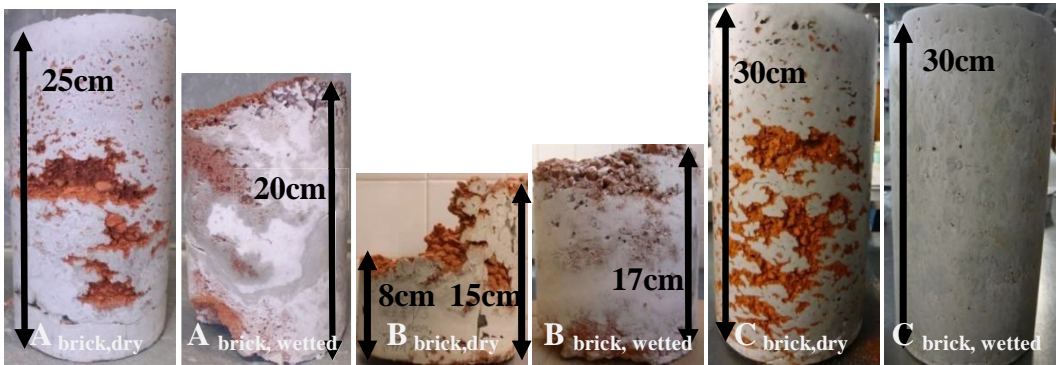


Fig. 14 Cylinders 45 days after being injected

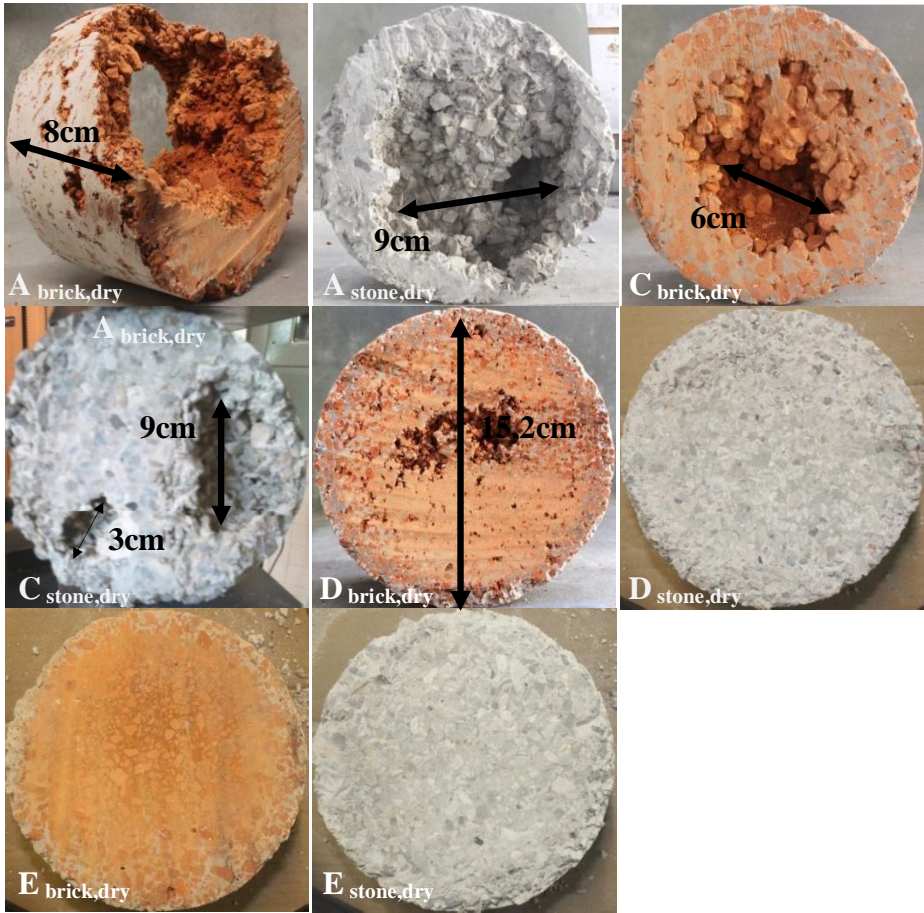


Fig. 15 Slices of cylinders at different levels

5.1.5. Penetrability results

Evaluation of different apertures in grout penetrability

Using the porosity and the specific surface determined from the grain size distribution curves (Fig. 6) of each porous medium presented in Table 8, an evaluation of the aperture (b_{K-C}) in the PM was performed (Table 10). The results obtained imply that the PM A and B are hardly injectable, if the rules aforementioned (see 2.1.2) are considered, which may explain the failure of the injection. In relation to the PM D and E the rules are verified

(exception D_{brick}), thus not causing any fail during the injection process. In particular the PM C are close to what is considered to be injectable (the rules are not totally verified). This may explain why the injection was not totally successful in some parts of the cylinder. These observations are in strict accordance with visual inspections (Fig. 14 and Fig. 15).

Table 10 Determined apertures for the different PM used in the experiments and the ratios between the PM and the grout

Porous Media	b_{K-C} (mm)	b_{K-C} / d_{95}^{Grout}	$D_{15}^{soil} / d_{85}^{Grout}$
A	stone	0,22	1,7*
	Brick	0,24	1,8*
B	stone	0,20	1,6*
	Brick	0,25	2,0*
C	stone	0,39	3,1**
	Brick	0,49	3,8**
D	stone	0,55	5,0
	Brick	0,77	6,0
E	stone	0,72	5,6
	Brick	1,12	8,7

* PM not injectable; ** PM starts to create plug formation (filtration tendency)

According to Eriksson (Eriksson, 2002) and Eklund (Eklund, 2005) the most important PM features affecting penetrability are aperture size, variability in apertures and magnitude of contact areas. As the finer PM present lower aperture sizes and higher variability in apertures and magnitude of contact areas (i.e., higher specific surface), grout penetrability problems may arise.

Newtonian fluid vs Bingham fluid

The aperture determined for the Bingham fluid (called equivalent aperture - b_{eqv}) is typically 3–5 times larger than the Newtonian fluid (water), as shown in Table 11. Thus, as stated earlier, the penetrability of a Newtonian fluid (water) or a Bingham fluid (grout suspension) differs. In fact, the solid particles of the grout suspension cannot enter all of the voids a Newtonian fluid can. It also means that the available space in each PM that can be filled with water is not the same as the space that may be filled with the grout. In fact, the results show that for each PM the volume of injected grout is lower than the volume of injected water. For this reason injectability values are lower than 1 (**Table 9**). Considering the aforementioned, it is possible to state that the measured porosity, which is done with water, is not a representative measurement for the available volume for a grout suspension.

Table 11 The equivalent aperture for a Bingham fluid and the ratio compared to the aperture determinant for Newtonian fluids

Porous Media	b_{eqv} (mm)	b_{eqv} / b_{K-C}
A	stone	0,86
	Brick	0,81
B	stone	0,86
	Brick	0,85
C	stone	1,45
	Brick	1,56
D	stone	1,80
	Brick	2,23
E	stone	2,44
	Brick	3,32

Analysis of the influence of filtration rate on grout penetration blockage

The curves that represent the mass of injected grout over time for each PM were plotted in Fig. 16. During the first part of injection process the injection curves have a linear increasing, while for the last part of the injection the curves show a different behaviour. The coarse PM present a constant flow of the same magnitude in all the tests. In relation to finer PM, from Fig. 16 it can be seen that a limited penetration occurred. After an initial penetration, the inclination of injection curves become null, what means that the penetration in the PM was not total (the top of the cylinder was not reached). The evolution of injection curves are in full agreement with the injectability values (Table 9) and the visual inspections performed the cylinders after 45 days of injection tests (Fig. 14 and Fig. 15).

By analysing the grout injection curves in Fig. 16 and the grout injectability values present in Table 9, certain conclusions regarding the penetrability and stop mechanisms can be made:

1. For PM A and B, the results presented in Table 10 indicate that limited penetration occurs due to the narrow voids between the particles of PM. In the lower part of the cylinder the presence of grout was obvious, but along the height the flow paths were successively blocked, the grout solid particles being unable to enter the available aperture – a stop mechanism called clogging (Axelsson, et al., 2009), leading to an absence of grout at the top as shown in Fig. 12.a. As referred by Eklund and Stille (Eklund and Stille, 2008), the plug formation (Fig. 1) is probably influenced by a stochastic phenomenon. From the experiments it was possible to observe that both the time for the plug to occur and the position where it occurred vary in the different tests.
2. According to Table 10, PM C is close to the threshold for which penetration is possible. The hypothesis is made that in critical parts of PM only single grout particles, but not the entire grout suspension, are able to penetrate the PM, therefore starting the filtration of grout solid particles as shown in Fig. 13.d, Fig. 14 and Fig. 15.
3. The grout will be able to penetrate PM D and E, as confirmed by visual inspection which showed homogenous grout penetration along the cylinder height (Fig. 14 and Fig. 15).

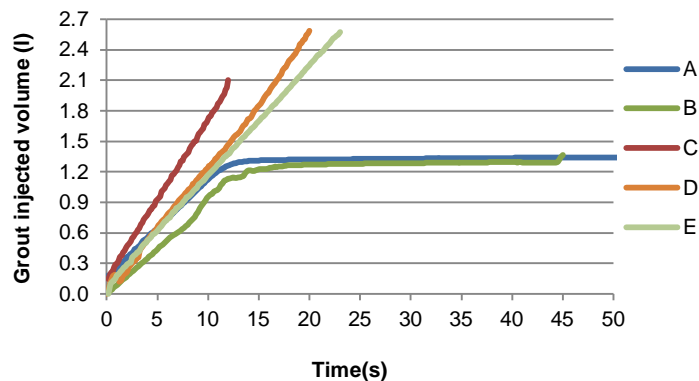


Fig. 16 Grout volume injection vs time to brick PM wetted

Concerning whether pre-wetting of PM before the grout injection may have any benefit concerning the penetration, it was observed that in finer PM the magnitude of filtration process is lower. In fact, as shown in Fig. 12.a and Fig. 12.c a higher height of injection is reached when the PM is pre-wetted. However, the grout volume injected is lower (Table 9), because as already stated during the pre-wetting the capillaries can absorb water due to capillary pressure what subsequently hinders the grout injection process.

Analysis of the grout penetrability using different criteria

Table 12 - Verification of the condition $d < W_{nom}/n$ based on different authors

Author	n value	P.M.									
		A		B		C		D		E	
		stone	Brick	stone	Brick	stone	Brick	stone	Brick	stone	Brick
Johnson	W_{nom}/d_{85} ($n > 3,75$)	1,31	0,77	1,25	0,73	2,73	2,34	<u>4,02</u>	<u>3,88</u>	<u>4,49</u>	<u>4,08</u>
Mitchell	W_{nom}/d_{100} ($n > 3$)	0,66	0,39	0,63	0,36	1,37	1,17	2,08	2,01	2,32	2,11
Littlejohn	W_{nom}/d_{85} ($n > 3,75$)	1,31	0,77	1,25	0,73	2,73	2,34	<u>4,02</u>	<u>3,88</u>	<u>4,49</u>	<u>4,08</u>
Littlejohn	W_{nom}/d_{100} ($n > 5$)	0,66	0,39	0,63	0,36	1,37	1,17	2,08	2,01	2,32	2,11
Hutchinso	W_{nom}/d_{max} ($n > 3$)	0,66	0,39	0,63	0,36	1,37	1,17	2,08	2,01	2,32	2,11
Cambefort	W_{nom}/d_{100} ($n > 1,5$ to 2)	0,66	0,39	0,63	0,36	1,37	1,17	<u>2,08</u>	<u>2,01</u>	<u>2,32</u>	<u>2,11</u>
Léonard	W_{nom}/d_{85} ($n > 0,75$ to 3)	1,31	0,77	1,25	0,73	2,73	2,34	<u>4,02</u>	<u>3,88</u>	<u>4,49</u>	<u>4,08</u>
Papadakis	W_{nom}/d_{100} ($n > 1,5$ to 3)	0,66	0,39	0,63	0,36	1,37	1,17	2,08	2,01	2,32	2,11
Paillère & Guinez	W_{nom}/d_{100} ($n > 1,5$ to 2,3)	0,66	0,39	0,63	0,36	1,37	1,17	2,08	2,01	2,32	2,11
Miltiadou-Fezans and Tassios	W_{nom}/d_{85} ($n > 5 \pm 1$)	1,31	0,77	1,25	0,73	2,73	2,34	<u>4,02</u>	3,88	<u>4,49</u>	<u>4,08</u>
Miltiadou-Fezans and Tassios	W_{nom}/d_{99} ($n > 2,0$)	0,66	0,39	0,63	0,36	1,37	1,17	<u>2,08</u>	<u>2,01</u>	<u>2,32</u>	<u>2,11</u>

Bolded values indicate that the condition $d < W_{nom}/n$ is partially satisfied.

Bolded and underlined values indicate that the condition $d < W_{nom}/n$ is satisfied.

According to the values of Table 12, some criteria created from different fields of the present work showed a slight agreement with the injectability results, namely the criteria proposed by Johnson, Littlejohn, Cambefort, Papadakis and Paillère & Guinez. The criteria created by Miltiadou-Fezans and Tassios showed to be the most suitable. As highlight, for the cylinder D the criterion $d_{85} < W_{nom}/5 \pm 1$ is verified to limestone PM and not for the brick PM (although the criterion has been almost fulfilled). In fact, as can be seen in Fig. 14 the injection was not entirely perfect for D_{brick}. The reason can be lower size of particles to brick PM (Table 8), resulting in lower W_{nom} value, consequently the magnitude of filtration process is higher. As regards the criterion $d_{99} < W_{nom}/2$, for both PM is found to be verified which in the reality it is not correct, as mentioned above. Therefore, the use of a single groutability ratio criterion is not sufficient to ensure penetrability into very fine voids of PM. Indeed, as concluded by Miltiadou-Fezans and Tassios (Miltiadou-Fezans and Tassios, 2012) the upper part of the entire grading curve of the binder must be taken into account. Thus, specific grading criteria should be verified in order to evaluate if the grout can be injected in a certain porous medium.

Buckingham Reiner model (yield stress vs penetrability)

Given the maximum L of the injection tests (0.3m), the pressure adopted in the injection tests ($\Delta P = 1$ bar) and the yield stress ($\tau_0 = 0,63$ Pa), the minimum void diameter (D_{min}) of the porous media to be injected is equal to 0,0075 mm (according to Eq. (4)). As can be seen in Table 10 and Table 11, the apertures calculated for all PM are much bigger than D_{min} . Therefore, according to Buckingham Reiner model an homogeneous filling in all PM should have been achieved. Nevertheless, as can be seen in 5.1.3 and 5.1.4, in PM A and B it is not observed. As aforementioned the variability of shape of channels inside porous media (not only cylindrical channel such as it is considered in the model of Buckingham Reiner) could also be a possible explanation for the difficulty that occurs when the grout is injected in these PM. Another possible explanation for the non

homogeneous filling observed in these PM can be the higher water absorption of these PM (Table 8). The loss of water that occurs in grout leads to an increase of yield stress which means that Eq. (2) may be satisfied - flow will stop in some channels - meaning that grout will try to flow through other available channels, leading to the non homogeneous filling.

6. Conclusions

The present study allows the following conclusions:

(a) Given the large variety of masonry types and materials and in order to better take into account the mentioned difficulties of hydraulic-lime grout to penetrate in extremely fine voids, the importance of evaluating the injectability of a grout for each specific case before intervention (by injecting plexiglas cylinders containing samples of the real materials of the masonry to be repaired) was highlighted in this work. Similar conclusions were also obtained by other authors, such as Bras (Bras, et al., 2012), Kalagri (Kalogri, et al., 2010), Valluzzi (Valluzzi, 2005) and Van Rickstal (Van Rickstal, 2000).

(b) The results of injectability show that the percentage of filling varied between 46% and 97% for the different PM. The reason for the upper value to be lower than 100% is due to the fact that the voids volume was determined with water (Newtonian fluid), which is able to penetrate into more voids than the grout (Bingham fluid). This phenomenon may also explain the similarity of grout volume injected in both PM, in spite of the brick PM having a higher porosity.

The value of injectability of a grout in a given media is mainly affected by the volume of voids, the quantity of fine particles and the water absorption of the media particles. Depending on the grain size distribution and the type of material of PM, the parameters referred have different influence on injectability. Thus, it is necessary to characterize all parameters of the porous medium so that the injection capacity of the grout can be estimated.

Comparing the differences of injectability values between coarse and fine PM, it is observed that these values are higher when using the equation proposed by Bras et al. In addition to the amount of grout injected, the equation by Bras also takes into account the rate of grout injectability, which has a great influence on the results. Furthermore, from these injectability results it seems that the yield stress value has more relevance for finer PM, whereas for coarse PM, viscosity forces and inertia effects become more significant.

(c) According to the visual inspections during the injection of the cylinders, it was concluded that for high amounts (over 33 wt%) of the finer material (0.15-2 mm) the reliability of the injection technique is jeopardized. In these cases the grout flow tends to stop during the injection process, enabling an increase of the particle flocculation phenomena, as observed with PM A and B. Thus, it can be concluded that this grout will not be injectable inside a masonry with similar internal characteristics. The visual inspections also showed that pre-wetting of the PM cannot solve the grout penetrability, since at the time of grout injection the finer capillaries are already filled with water (due to its high capillary pressure - Kelvin law), hence hindering the penetration of the grout. The only advantage of pre-wetting is to increase the grout injection velocity, since the flow resistance is reduced by the water injection, therefore increasing the rate of injectability. However, pre-wetting should be used with much precaution, as also stated by some authors [(Valluzzi, 2005), (Van Rickstal, 2000)].

(d) In the light of the achieved results, some conclusions can be drawn regarding the penetrability of hydraulic lime grout:

1- The penetrability depends on the ratio between the aperture size and the maximum particle size in the grout. When the grout solid particles are not able to enter the available aperture pathways clogging occurs. This happens in PM A and B, for which the rules established by Axelsson and colleagues are not observed. Close to the limit of what is considered penetrable according these rules filtration occurs, i.e. the grout enters the available aperture but some solid particles of the grout stop in constrictions and gradually block the pathways. This stop mechanism happens in some critical zones of PM C. When the rules are respected (cases of PM D and E), homogeneous penetration of grout suspension is achieved.

2- The penetrability of a fluid is dependent on the aperture of the area between the particles of PM and a function of the fluid type. Moreover, the variability in apertures and the magnitude of contact areas are also important PM features that affect the fluid penetrability.

3- The water content of PM before the injection affects the filtration rate; more water means lower filtration tendency. However, as already mentioned, in general this fact does not improve grout penetration.

4 - From the study of hydraulic lime grout penetrability following different approaches, it was possible to conclude: a) the grain size distribution of the grout solid phase should be compatible with the characteristic dimensions of the discontinuities to be injected (voids, fissures, interfaces, etc); b) one "groutability ratio" criterion is not sufficient to ensure penetrability into very fine discontinuities of masonry. In reality, as already established by Miltiadou-Fezans and Tassios (Miltiadou-Fezans and Tassios, 2012) the upper part of the grading curve of the grout solid phase has to comply with specific grading criteria, depending on the granularity of the PM to be injectable; c) knowing grading curve of the binder can predict if a given porous media can be injectable.

5- Regarding the Buckingham Reiner equation, according to the grout penetrability results obtained it is not the best way to check whether a grout is able to penetrate in a specific porous media. The high variability of shape of the channels inside these types of porous media contradicts the assumptions of the equation, which should not be used. However, as already concluded by other authors [(Axelsson, et al., 2009), (Bras, et al., 2012), (Eriksson, 2002)], the injection tests revealed that yield stress may be used as a control index for the application of injection grouts.

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References

- Almeida, C., et al. (2012) *Physical characterization and compression tests of one leaf stone masonry walls*. s.l. : Constr. Build. Mater. 30:188-197.
- Anzani, A., et al. (2006) *Use of Sonic and GPR tests to control the effectiveness of grout injections of stone masonry*. s.l. : 9th European NDT Conference (ECNDT), September, 25-29, 2006 - Berlin, Germany.

Axelsson, M., Gustafson, G. and Fransson, A. (2009) *Stop mechanism for cementitious grouts at different water-to-cement ratios*. s.l. : Tunn and Undergr Space Technology 24, pp. 390-397.

Axelsson, Magnus and Gustafson, Gunnar. (2007) *Grouting with high water/solid-ratios - Literature and laboratory study*. s.l. : Department of Civil and Environmental Engineering - CHALMERS UNIVERSITY OF TECHNOLOGY, GÖTEBORG, SWEDEN.

Baltazar, L. G., Henriques, F. M. A. e Jorne, F. (2012) *Optimisation of flow behaviour and stability of superplasticized fresh hydraulic lime grouts through design of experiments*. s.l. : Constr. Build. Mater. vol. 35, pp. 838–845.

Baltazar, L.G., et al. (2014) *Combined effect of superplasticizer, silica fume and temperature in the performance of natural hydraulic lime grouts*. s.l. : Constr. Build. Mater. 50: 584-597, Elsevier. DOI: 10.1016/j.

Baltazar, Luis G., et al. (2013) *The use of rheology in the study of the composition effects on the fresh behaviour of hydraulic lime grouts for injection of masonry walls*. s.l. : Rheol Acta (2013) 52:127–138.

Baltazar, Luis G., Henriques, Fernando M.A. and Jorne, Fernando. (2012). *Hydraulic lime grouts for masonry injection - effects of admixtures on the fresh grout properties*. s.l. : Structural Analysis of Historical Constructions , Wroclaw, Poland.

Binda, L. and Anzani, A. (1997) *Structural behaviour and durability of stone masonry, saving our architectural heritage: the conservation of historic stone structures*. s.l. : New York: Wiley, 1997:112-48.

Binda, L., et al. (2003) *Experimental research for the choice of adequate materials for the reconstruction of the cathedral of Noto*. s.l. : Constr. Build. Mater. 17:629-639.

Binda, L., et al. (1997) *Repair and investigation techniques for stone masonry walls*. s.l. : Constr. Build. Mater. 11(3): 133-42.

Binda, L., Saisi, A. and Tiraboschi, C. (2001) *Application of sonic tests to the diagnosis of damaged and repaired structures*. s.l. : NDT&E International 34:123-138.

Bras, A. and Henriques, F.M.A. (2009). *The influence of the mixing procedures on the optimization of fresh grout properties*. s.l. : Mater Struct, 42,1423-1432.

Bras, A. (2011) *Grout Optimization for masonry consolidation*. s.l. : Ph.D. thesis, Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa.

Bras, Ana and Henriques, Fernando M.A. (2012) *Natural hydraulic lime based grouts - The selection of grout injection parameters for masonry consolidation*. s.l. : Constr. Build. Mater. 26:135–144.

Bras, Ana, Henriques, Fernando M.A. and Cidade, M.T. (2012) *Rheological behaviour of hydraulic lime-based grouts. Shear-time and temperature dependence*. s.l. : Mech Time-Depend Mater. DOI 10.1007/s11043-012-9202-0.

Cachim, Paulo B. (2009) *Mechanical properties of brick aggregate concrete*. s.l. : Constr. Build. Mater. 23:1292–1297.

Cambefort, H. (1977) *Principes et applications de l' injection*. s.l. : Annales de l'ITBTP, Paris, Supp. no. 353, Série: Sols et fondations, no. 144, 23 pp.

Carman, P.C. (1956) *Flow of Gases Through Porous Media*. s.l. : Butterworths Scientific Publications, London, UK..

Dantu, P. (1961) *Etude mécanique d'un milieu pulvérulent formé de sphères égales de compacité maxima*. s.l. : 5ème Congrès International de Mécanique des sols et des Travaux de Fondations, Paris, Dunod, Publication 61-3, 10 pp.

David Carrier, W. (2003) *TECHNICAL NOTES: Goodbye, Hazen; Hello, Kozeny-Carman*. s.l. : Journal of Geotechnical and Geoenvironmental Engineering, Vol. 129, No. 11, pp. 1054-1056. DOI: 10.1061/(ASCE)1090-0241(2003)129:11(1054).

Eklund, D. and Stille, H. (2008) *Penetrability due to filtration tendency of cement based grouts*. s.l. : Tunn and Undergr Space Technology, pp. 389-398.

Eklund, D. (2005) *Penetrability Due to Filtration Tendency of Cement Based Grouts*. s.l. : Doctoral Thesis, Division of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm, Sweden.

Eriksson, M. (2002) *Prediction of Grout Spread and Sealing Effect, a Probabilistic Approach*. s.l. : Doctoral Thesis, Division of Soil and Rock Mechanics, Royal Institute of Technology, Stockholm, Sweden.

Hutchinson, M.T. (1981) *Principles of grouting II*. s.l. : Summary of a lecture given the 16-9-1981 in the Cement and Concrete Association Conference and Training Centre, UK, TDH 4710, pp 6.

Ignoul, S., Van Rickstal, F. and Van Gemert, D. (2004) *Application of mineral grouts. Case study and impact on structural behaviour: Church of St. Catharina at Duisburg (B)*. s.l. : Proceedings International Seminar IV 'Structural Analysis of Historical Constructions', Padua, 10-12 November 2004, Balkema Publ., pp.719-726.

Johnson, S.J. (1958) *Cement and clay grouting of foundations: grouting with clay-cement grouts*. s.l. : J Soil Mech Found Eng Div ASCE 84(1):1-12.

Kalagri, A., Miltiadou-Fezans, A. and Vintzileou, E. (2010) *Design and evaluation of hydraulic lime grouts for the strengthening of stone masonry historic structures*. s.l. : Mater Struct 43:1135-1146

Léonard, Z.F. (1961) *Grouting: clay based and chemical*. s.l. : The Engineer 26:864-866.

Littlejohn, G.S. (1983) *Chemical grouting*. s.l. : South African Institution of Civil Engineers, University of Wetwatersrand Johannesburg, 4-6 July 1983.

Miltiadou-Fezans, A. and Tassios, T. P. (2012) *Fuidity of hydraulic grouts for masonry strengthening*. s.l. : Mater Struct 45:1817-1828.

Miltiadou-Fezans, A. and Tassios, T. P. (2012) *Penetrability of hydraulic grouts*. s.l. : Mater Struct 46:1653-1671.

Miltiadou-Fezans, A. and Tassios, T. P. (2013). *Stability of hydraulic grouts for masonry strengthening*. s.l. : Mater Struct 46:1631-1652.

Mitchell, J.K. (1982) *Soil improvement – state of the art*. s.l. : In: Proceedings, 10th International Conference on Soil Mechanics and Foundation Engineering, vol. 4. Stockholm, Sweden, pp. 509-566.

Mitchell, K.J. (1970) *In-plane treatment of foundation soils*. s.l. : Journal of the Soil Mechanics and Foundations Division, Proc. ASCE, SM1, pp. 73-110.

Paillère, A-M. and Guinez, R. (1984) *Recherche d'une formulation de coulis à base de liants hydrauliques pour l'injection dans les fines fissures et les cavités*. s.l. : Bulletin de liaison des Laboratoires des Ponts et Chaussées, Paris, no 130, pp 51-57.

Paillère, A-M. and Rizoulières, Y. (1978) *Réparation des structures en béton par injection de polymères*. s.l. : Bulletin de Liaison des Laboratoires des Ponts et Chaussées 96:17–23.

Papadakis, M. (1959) *L'injectabilité des coulis et mortiers de ciments*. s.l. : Revue des matériaux de construction. 531, publication technique no. 11 CERILH, 48 pp.

Toumbakari, E. (2002) *Lime-pozzolan-Cement grouts and their structural effects on composite masonry walls*. s.l. : Ph.D. Thesis, Katholieke Universiteit Leuven.

Toumbakari, E.-E., et al. (1999) *Effect of mixing procedure on injectability of cementitious grouts*. s.l. : Cem Concr Res 29:867–872.

Valluzzi, M. R., Da Porto, F. and Modena, C. (2004) *Behavior and modeling of strengthened three-leaf stone masonry walls*. s.l. : Mater Struct, Vol. 37, April 2004, pp 184-192.

Valluzzi, M.R. (2005) *Requirements for the choice of mortar and grouts for consolidation of three-leaf stone masonry walls*. s.l. : Paper presented at the workshop repair mortars for historic masonry. Delft University of Technology, Faculty of civil Engineering and Geosciences, Delft; 26-28 January 2005.

Van Rickstal, F. (2000) *Grout injection of masonry, scientific approach and modeling*. s.l. : Dissertation. Katholieke Universiteit Leuven.

Van Rickstal, Filip, et al. (2003) *Development of mineral grouts for consolidation injection*. s.l. : In Consolidation of Masonry, Ed. D. Van Gemert, Advances in Materials Science and Restoration, pp. 61-70.

Vikan, H. (2005) *Rheology and reactivity of cementitious binders with plasticizers*. s.l. : Ph.D. thesis at Norwegian University of Science and Technology.

Vintzileou, E. (2006) *Grouting of Three-Leaf Stone Masonry: Types of Grouts, Mechanical properties of Masonry before and after Grouting*. New Delhi : Structural Analysis of Historical Constructions.