Repeatability and reproducibility in measuring injection grouts properties: three grouts, two operators

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Abstract Non–structural injection grouts are typically tested following standards concerning plasters, mortars and concretes, as currently there are not specific tests which are internationally agreed for injection grouts. A lab testing program is important to assess how the material is going to behave, in its fluid state and in its hardened state. The objectives of the research was to assess repeatability and reproducibility of testing procedures. Three grout mixtures (with different binding mechanisms) were tested. To assess the *repeatability* the same operator repeated the measure, while to assess *reproducibility* two different operators conducted the tests. The tests performed are repeatable and in most of cases reproducible. However, some properties showed different results when performed by the two different operators. The differences are probably to be sought in the grouts intrinsic properties.

1 Introduction and objectives

Non-structural injection grouting provides stabilisation of delaminated wall paintings/historic plasters introducing a compatible adhesive material with bulking properties, which –once injected– becomes a non-extractable part of the layered system [1]. A lab testing program is crucial to assess how the material is going to behave, short-term in its fluid state (working properties) and long-term over time in its hardened state (performance characteristics). There are not specific tests for injection grouts which are internationally agreed –even if recent publications and international committees' work are trying to fill this gap. Grouts are thus typically tested following national standards or international norms, concerning plasters, mortars and concretes, which have different characteristics and properties compared to non-structural grouts.

The objectives of the research was to assess repeatability and reproducibility of testing procedures for injection grouts. Three grout mixtures were tested, each having a different binding mechanism: one lime-based, one lime-based with addition of a pozzolan, and one hydraulic lime-based. To assess the *repeatability* the same operator repeated the measure, while to assess *reproducibility* two different operators conducted the tests. Each measure was carried out at least on 3 specimens.

2 Materials and mixtures

In this research three grouts with a different binding mechanism were chosen, in order to have materials with properties expected to be in the same range of values, but anyhow different due to the different binder/combination of components.

Grout A was lime-based with addition of a filler (calcite sand 12-70 µm) and a superplasticiser (PCE) to increase fluidity; grout B was lime-based with addition of a pozzolan (pumice 0-90 µm –hydraulic reaction is involved) and a filler (glass microspheres 40-100 µm); grout C was hydraulic lime-based (NHL 2) with addition of a filler (marble powder 0-36 µm). For all grouts the suspension medium was deionised water, in different proportions. The set criteria was that the grouts needed to be easily injected through a 100 mL syringe with 3 mm exit. Specifications regarding grouts formulation are given in Table 1.

Table 1 Grouts formulations

	Grout A	***pt/V***	**Grout B**	***pt/V***	**Grout C**	***pt/V***
Binder	hydrated lime	1	slaked lime	1	NHL 2	1
Aggregates	calcite sand	3	glass microspheres	3	marble powder	3
			pumice	1		
Additives	PCE	0.5% w/w	-	-	-	-
Susp.medium	deionised water	4	deionised water	1	deionised water	1.5

Considering the total volume (solid components + suspension medium), the grouts contain the following amount of water (in volume): grout A 50%; grout B 25% (this includes the amount of water added as suspension medium plus the amount of water contained in the slaked lime –made of 50% $Ca[OH]_2$ and 50%

water); grout C 33%. The amount of water in the grouts will have an influence on grout properties, both working properties and performance characteristics.

3 Testing program, procedures and results

Grout A was tested by operator 1 (repeatability), grout B was tested by operator 2 (repeatability) and grout C was tested by both operator 1 and 2 (repeatability and reproducibility). In this paper grout C tested by operator 1 will be called C1 and grout C tested by operator 2 will be called C2.

The procedures were chosen from international standards for plasters/cements – adapting them, when required, to the materials under consideration– and from the literature [2].

3.1 Working properties

3.1.1 Injectability with syringe, flow with syringe and on plastered tile

Both injectability and flow were assessed with qualitative field tests suggested in the literature [2]. In the injectability test [2:69-70] the grout is injected in a syringe filled with crushed building material and the syphon of the hosting syringe is pressed down so that the grout flows among the crushed material interstices when pressure is applied. In the flow test with syringe [2:72-73], the grout is poured in a syringe filled with crushed building material and it is let flow without any external pressure. A crushed lime-sand mortar was used for the test (particle size 2–4 mm) instead of the crushed travertine or brick suggested in the literature. Both tests were performed with both dry and pre-wet crushed material. Grouts B, C1 and C2 showed *difficult* injectability. Grout A showed *feasible/easy* injectability (for qualitative classification see reference 2).

All grouts showed *difficult* flow (for qualitative classification see reference 2). Results (showed in Fig.1) slightly differentiate for grout C1 and C2 in terms of distance reached. This can be due to the difference in pressure applied by operator 1 and 2 on the syringe syphon. Overall, though, both tests showed to be repeatable and reproducible.

Flow on plastered tile [2:75-76] was performed with both dry and pre-wetted plaster. A controlled weight pressing on the syphon was applied in order to have a constant and repeatable pressure to inject the grouts into the channels. Grout A resulted to have *medium/high* flow, while the other grouts *low* flow (for qualitative classification see reference 2). The test showed to be repeatable and reproducible.

In these tests the grout prepared with the highest amount of water and the superplasticiser, i.e. grout A, is the one with the highest injectability and flow.

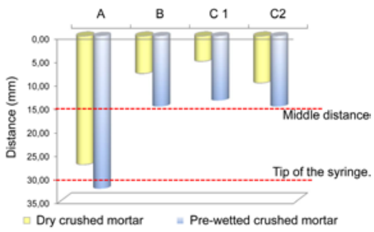

Fig. 1 Injectability with syringe: distance reached by grouts in the syringe

3.1.2 Wet density

To assess wet density both a lab test following the standard ASTM C 185-02 and a field test suggested in the literature [2:81] were performed. Results are listed in Table 2. Although the lab test is more precise than the field one by definition, the two tests for the same grout showed similar results. Grouts C1 and C2 have comparable results. The tests are repeatable and reproducible.

3.1.3 Expansion and bleeding

The test was performed following standard ASTM C940-03, with a reduced volume of grout (80 mL instead of 500 mL). Results are listed in Table 2. Grouts C1 and C2 have the same bleeding. Grout B has a lower bleeding due to its lower water content in preparation. The test showed to be repeatable and reproducible, also with a reduced amount of grout involved.

3.1.4 Water retention and release

Standard DIN 18 555 part 7 was used for the water retention and release test. Results are listed in Table 2. Grout B has a relatively low water content: together with a low bleeding, also a low water release (and therefore a high water retention) was expected. However, grout B water release is higher compared to the one of the other grouts. Probably the grout would show lower water release if no weight were applied on the sample (see standard procedure). Grout A and C1-C2 (C1 and C2 have comparable results) have a high water retention, over 80%. The test showed to be repeatable and reproducible.

Table 2 Wet density, bleeding and water retention and release

Mixture	Wet density cylinder (g/cm^3)	Wet density syringe (g/cm^3)	Bleeding (%)	Water retention (%)	Water release (%)
A	1.78	1.74	2.1	83.1	16.7
B	1.82	1.80	1.4	54.1	45.9
C1	1.82	1.81	2.1	85.1	14.9

C2	1.81	1.82	2.1	86.7	13.3

3.2 Properties during setting and curing

3.2.1 Drying shrinkage

A field test suggested in the literature [2:83-85] was performed to determine the volume change of grouts after drying. The grout was poured in a plaster cup and its volume change, including cracking, was qualitatively evaluated over time. The support where the grout sets (i.e. plaster cup) is representative of a real case. The following additions were made in the test procedure: the test was performed both with dry and pre-wetted plaster; the test was performed in two rooms with a different relative humidity (50% ±5 RH 20°C; 99% RH 20°C) to check its influence. Grout A showed no cracks and a separation from the plaster cup walls of 0.5 mm in all the cases (dry/pre-wetted 50% RH; dry/pre-wetted 99% RH): it was classified as having *medium* drying shrinkage (for qualitative classification see reference 2). Grout B showed no cracks and a separation from the plaster cup walls of 0.5 mm just in the dry plaster cups (at both 50% and 99% RH): grout B in the wet cups were judged to have *no* drying shrinkage. Grout C1 and C2 had both the same results: dry cup-50% RH 0.5 mm wide cracks and 1.5 mm separation from the cup walls (*high* shrinkage); wet cup-50% RH no cracks and 0.5 mm separation from the cup walls (*medium* shrinkage); dry cup-99% RH no cracks and 1.5 mm separation from the cup walls (*high* shrinkage); wet cup-99% RH no cracks and no separation from the cup walls (*no* shrinkage). Grout A showed *medium* shrinkage also in the pre-wetted cups probably because of its high water content. Grout B showed *no* shrinkage in the pre-wetted cups probably because of its relatively low water content and its higher amount of fillers compared to the other two grouts. Grout C showed some shrinkage probably because of the very fine particle size of its filler. Even if qualitative, the test showed to be repeatable and reproducible.

Shrinkage was also calculated with a test developed in our labs having the following procedure: an empty mould of know volume is filled with grout; after the drying shrinkage is completed, mould+grout are weighed; the empty space between the grout and the edges of the mould is filled with a fine sand (0-90 µm); mould+grout+sand are weighed. The volume of the added sand (V_S) is calculated with $V_S = M_S/\rho$, where M_S is the weight of the sand added and ρ is the true density of the sand. The shrinkage (S) is calculated with $S = (V_S/V_M)\cdot 100$, where V_M is the volume of the mould. Results are listed in Table 3. The test showed to be repeatable, but not reproducible: grouts C1 and C2, indeed, have different shrinkage results. The sand chosen is probably not fine enough and it is possible that for grout C1 (having lower shrinkage) it did not completely fill the empty space inside the mould. In this test grouts A and B showed the same shrinkage.

3.2.2 Final setting time

The grouts final setting time was assessed with both a field test [2:87-89] and a lab test (Vicat needle –standard UNI EN 196-3). For the field test, both dry and pre-wetted plaster cups were used and the test was performed at both 50% and 99% RH. The setting time was checked making penetrate in the grout a 60 mL syringe (filled with 100 g granular material) having a needle attached to the tip. Results obtained by the same operator were consistent, but grouts C1 and C2 showed substantial differences. This is probably due to the difference of interpretation of the marks left on the grout surface by the needle (a blunt cannula would lead to a better and easier interpretation). In this case this test showed to be repeatable but not reproducible.

Final setting time was also assessed with the Vicat needle. The material was reduced from 200 mL to 40 mL to have a thickness which was more representative of a delamination to stabilise with a non-structural grout. Results are reported in Table 3. The test showed to be repeatable and quite reproducible (difference of one hour between grout C1 and C2). Grout C showed a shorter setting time because NHL-based. Grouts B and C showed a quite similar setting time with the Vicat needle, while a higher difference is noticed in the qualitative test at 99% RH with the pre-wetted cup. Grout A takes much longer to set because lime-based and with no hydraulic reaction involved.

Table 3 Shrinkage and final setting time

Mixture	**Shrinkage (%)**	**Setting time**				
		Dry cup/ 50% RH	**Wet cup/ 50% RH**	**Dry cup/ 99% RH**	**Wet cup/ 99% RH**	**Vicat needle**
A	5.7	7 h	20 h	48 h	> 15 days	53 h
B	5.7	9 h	49 h	47 h	9 days	50 h
C1	3.5	2 h	3 h	33 h	4 days	22 h
C2	4.2	1 h	6 h	47 h	5 days	23 h

3.3 Performance characteristics

3.3.1 Porosity

Total porosity (*n*), capillary porosity (U_E), and amount of air pores (*LP*) were determined following standard SIA 262/1**.** Results are reported in Table 4. Grout A, prepared with a higher water content, has 38.48% capillary porosity and 5.57% air pores. Grout B, prepared with a lower water content, has a lower capillary porosity (24.84%), but a higher percentage of air pores (10.41%), probably linked to pumice presence –porous itself. Grout C1 and C2 have very similar porosity, with a very high capillary porosity and a very low percentage of air pores. The test showed to be repeatable and reproducible.

3.3.2 Water vapour diffusion and capillary water absorption

The diffusion resistance factor (μ) of grouts was determined with standard UNI EN 12086. The lower the μ the easier the passage of the water vapour through the sample. Capillary absorption was determined following standard DIN 52617-A. The absorption as a function of time (w) is reported at 24 hours (w_{24h}) and at 30 minutes ($w_{30'}$). Results are reported in Table 4. While grout B has a low μ (easier passage of water vapour) probably due to the high percentage of air pores, grout A has a higher μ (accordingly, probably due to the lower air pores percentage). Grout C1 and C2 have a comparable μ. In terms of absorption at 24 hours (w_{24h}), grouts A, C1 and C2 have comparable values, while grout B a lower value –but still in the same range. The difference is more visible at 30 minutes ($w_{30'}$): grout A has the lowest value (it absorbs slower), a half compared to grout B one. Values of grouts C1 and C2 are much higher than grouts A and B values (grouts C1 and C2 absorb much faster). There is a difference, though, between $w_{30'}$ of grouts C1 and C2: according to that, grout C1 absorbs slower than grout C2.

Both tests are repeatable. As capillary absorption is linked to porosity and grouts C1 and C2 have a very similar porosity, the difference in $w_{30'}$ is probably linked to the different *size* of the pores in C1/C2 (pore size is not detectable with standard SIA 262/1). This may be the reason for the difference, rather the non-reproducibility of the test.

3.3.3 Mechanical strength

Compressive strength (σ_c) was tested following standard UNI EN 1015/11, and splitting tensile strength (σ_s) following standard UNI EN 12390/6 (samples are cylinders with diameter 50 mm and height 50 mm). Grout A is much stronger than grouts B and C1/C2. The results obtained by operator 1/operator 2 for all the grouts showed a high coefficient of variation. Moreover, the results of grouts C1 and C2 are considerably different, although the procedure followed was identical. In this case, it is not the test itself being not repeatable/reproducible: the differences/the high coefficient of variation are probably to be sought in: a) the samples size (cylinders: 50 mm Ø, 50 mm height– the smaller the sample the higher the potential error); b) the number of samples tested (to have a better average and reduce the coefficient of variation, it is useful to test many samples); c) the intrinsic characteristics of the grouts (e.g. pore size, which has an influence on mechanical strength).

Table 4

Mixture	**n (%)**	**U_E (%)**	**LP (%)**	**μ (-)**	**$w_{30'}$ (Kg/[$m^2\sqrt{t}$])**	**w_{24h} (Kg/[$m^2\sqrt{t}$])**	**σ_c (N/mm^2)**	**σ_s (N/mm^2)**
A	44.04	38.48	5.57	16	3.29	3.37	8.06	0.76
B	35.25	24.84	10.41	13	6.18	2.11	4.07	0.22
C1	43.12	40.57	2.56	14	17.53	3.43	1.74	0.06
C2	44.24	41.50	2.75	15	25.51	3.77	2.72	0.10

See the text for the meaning of symbols

4 Final discussion and conclusions

The results show that, as expected, the tests performed are repeatable (results of the same test repeated by the same operator are comparable among them).

As for reproducibility, the results show that the same test performed by different operators gives in most of cases comparable data. However, mechanical strength and capillary absorption speed gave different results when performed by the two different operators, although the procedures followed were identical. This is not due to the fact that the test is not reproducible, but it is due to the intrinsic properties of the grouts. Porosity is a property which has an influence on water vapour diffusion and capillary water absorption. It also has an influence on mechanical strength [3], in particular compressive strength. Although the two operators agreed the mixing procedure and the time of mixing, also small differences in grout mixing/sample preparation may lead to differences in porosity. As the porosity percentage of grouts C1 and C2 is very similar, the difference probably lays in the pore size. A different way to prepare mixtures and also to prepare samples (e.g. injecting them in the mould vs. pouring them, how the grout is compacted and so on) may lead to smaller/bigger pore size, in particular regarding air pores. Air pores, indeed, have an influence on absorption speed and on compressive strength.

5 References

1. Rickerby S, Shekede L, Fan Z, Tang W, Qiao H, Yang J and Piqué F (2010) Development and Testing of the Grouting and Soluble-Salts Reduction Treatments of Cave 85 Wal Paintings. In: N. Agnew, ed. Proceedings of the Second International Conference on the Conservation of Grotto Sites, Mogao Grottoes, Dunhuang, Conservation of Ancient Sites on the Silk Road, People‘s Republic of China, June 28–July 3, 2004. Los Angeles: The Getty Conservation Institute: 471–479

2. Biçer-Şimşir B and Rainer L (2013) Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces – A Manual of Laboratory and Field Test Methods. Los Angeles, The Getty Conservation Institute
3. Papayianni I and Stefanidou M (2006) Strength–porosity relationships in lime–pozzolan mortars. Constr. Build. Mater. 20: 700–705