RILEM TC 243-SGM report
grouting for historic architectural surfaces

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RILEM TC REPORT

RILEM TC 243-SGM report: grouting for historic architectural surfaces

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Abstract Historic Structures are commonly coated with mortar layers (plasters, renders, flooring) for protection and decoration. These well finished architectural stratigraphic surfaces often suffer from deterioration, such as lack of adhesion or detachment between support and mortar layers and even between mortar layers. Grouting and filling voids between delaminated layers can be an effective intervention if the layers are compact. This paper deals with aspects pertinent to the selection, design and implementation of a grout for the in situ stabilization and preservation of historic architectural surfaces. It presents the methodological approach, in accordance with the conservation principles, including sections on diagnosis, study and assessment of the deterioration phenomena, definition of grout requirements, selection of proper ingredients for custom-made or commercial grouts, as well as making and testing trial mixes, field testing and Assessment of the effectiveness of grouting. The aim of this paper, that is elaborated in the frame of RILEM TC 243 SGM, is to serve as a guide for users of lime-based grouts for the reattachment and reinstatement of historical architectural surfaces.

Keywords Historic architectural surfaces · Multilayers · Reattachments · Lime-based grouts · Methodological approach

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1 Introduction and definitions

Historic structures are commonly coated with mortar layers on the internal and external walls and occasionally on floors. These layers may have a high historical, archaeological and technological value, such as plasters of the Egyptian, Hellenistic, and Roman periods. They are often decorated or are of high cultural significance and may include wall paintings, sgraffiti, mosaics, ceramic tiles, and stuccoes. These wall and floor systems are characterized a stratigraphic nature composed of a primary support (made of stone, adobe, bricks, wood, mortar, concrete, etc.) and layers (plasters, renders, stucco, ceramic tiles, stone cladding, mosaics, etc.) with different features. Lack of adhesion between these components (Figs. 1, 2) is one kind of deterioration that can occur on this type of cultural heritage. Bulging (Fig. 3), bowing, blistering, disintegration and delamination can result in detachment and loss of historic finishing layers and often require stabilization [1, 2].

Grouting can be an effective intervention method when the layers are compact. Grout can fill the void which has resulted due to delamination between layers, or layers and the support and with a suitable material can restore the continuity of the support and the architectural surfaces.

Injection grouting aims to stabilize architectural surfaces by introducing a bulked fluid adhesive material (i.e. grout) into the delaminated area (Fig. 4). Grout is a mixture of binding material(s) with an adequate dispersing medium, that may or may not have fine aggregate and/or filler and admixtures, forming a fluid but stable injectable mixture. The layers which need re-adhesion may be very thin (some millimeters), as in the case of Hellenistic colored renders and plasters (Fig. 5), or may be of considerable thickness, usually one or two centimeters, such as in mosaics (Fig. 6).

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Specific challenges in the case of grouting intervention are that this operation is irreversible and that the problems (e.g. delamination, void) are often hidden and difficult to assess, both in terms of location and in terms of extent. In addition, evaluating the results of the grouting operation itself is complicated [3], because it is hard to verify where the grout mixture goes, what capacity it has to stabilize the system, and its performance over time. Therefore, planning for grouting should be carried out carefully with particular attention to the principles of minimal intervention and compatibility. Furthermore, grout performance must be controlled and recorded, and its effectiveness should be assessed periodically after intervention.

The main aim of this paper is to offer a methodological approach (guidelines) to design lime-based grouts or select commercial injection grouts, and implement grouting intervention for the in situ stabilization of archaeological or historic architectural surfaces.

2 Background

In the past, the most common intervention to solve the problem of delaminating plasters was to detach them and to relay them back in place or on a new support using an adhesive. This intervention often has proved to be destructive for the wall painting and when removed, resulted in the loss of its historical context.

In situ stabilization is commonly achieved by mechanical pinning to hold the plaster in place and injecting adhesives behind it. Many types of organic adhesives, such as natural water-soluble glues [4], acrylic resins and other polymers in dispersions [5, 6], as well as epoxy and polyester-based adhesives have been used unsuccessfully, mainly because these materials have physical, chemical, and mechanical properties very different from those of the layers that were re-adhered. Often, they created a hydrophobic, brittle layer with low durability problems [7, 8]. At the same time, inorganic binding materials, such as hydraulic lime, lime-clay mixtures and cement were used for grout formulations. One of the earliest published grouts designed for the conservation of architectural surfaces was the development of a hydraulic lime-based grout for conserving archaeological plasters, wall paintings, and mosaics by a team at ICCROM in 1979–83 [9, 10]. The grout was applied

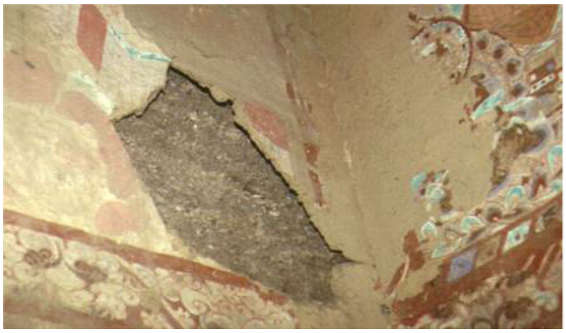

Fig. 1 Lack of adhesion (delamination) between earth-based plaster and rock-conglomerate support has caused loss of plaster in cave 89 at the site of Mogao, Dunhuang in Gansu province, China. photo by F.Piqué (©GCI)

Fig. 2 Delaminated lime-based render with loss of plaster on earth-block masonry in Santarem Building, Portugal. Photo by A. Velosa

Fig. 3 Detachment of wall-mosaics from a masonry wall due to bulging of the mortar layer because of compression forces within the layers, Monastery of Chios (1045–1056 A.D.), Greece. Photo archive: Lab of Building Materials AUTH

to stabilize architectural surfaces in nine Italian sites, including the House of Menander in Pompeii and the Church of San Lorenzo in Rome. The main reason hydraulic grouts were preferred over air lime-based grouts was the fact that grouts have very limited access to air and therefore, carbonation is expected to be very slow or may not occur in humid environments. Hydraulic lime-based grouts were also used for the conservation of eighteenth century lime plasters on stone [9]. A comprehensive list of early case studies, in which hydraulic lime grouts were used, is available in Ref [11], and a literature review of the materials and methods of evaluation of lime- and hydraulic lime-based grouts is available in Biçer-Şimşir et al. [12].

In the past three decades, the use of grouts based on inorganic binders has become common for the in-situ stabilization of architectural surfaces, while grouts based on organic binders have been abandoned (even if not completely) because of the problems related to incompatibility with the original materials.

3 Conservation principles and methodological approach for grouting intervention

The concepts of compatibility and irreversibility in conservation intervention [13, 14], were originally introduced as consequences of the Charter of Venice (1964) [15]. Grouting is an irreversible treatment and compatibility issues are fundamental. Therefore, in stabilizing detached surface layers by grouting operations, the primary objective is to save the original context (i.e. architectural surface and support) for the future, and minimize any further stabilization intervention. The intervention can be considered successful if, in addition to stabilizing the layers by providing adhesion between layers and to their support, no aesthetic alterations or secondary problems are caused to the original architectural surface.

Chemical compatibility refers to the whole system in context [16]. Physical and mechanical compatibility refers to the most deteriorated component. Grouting can be conducted between layers of similar material and composition or, more often, between different materials. A good understanding of the characteristics of the substrates and/or layers to be re-adhered is a necessary prerequisite to define technical requirements for the intervention. The fundamental properties, which can play a role as compatibility indicators

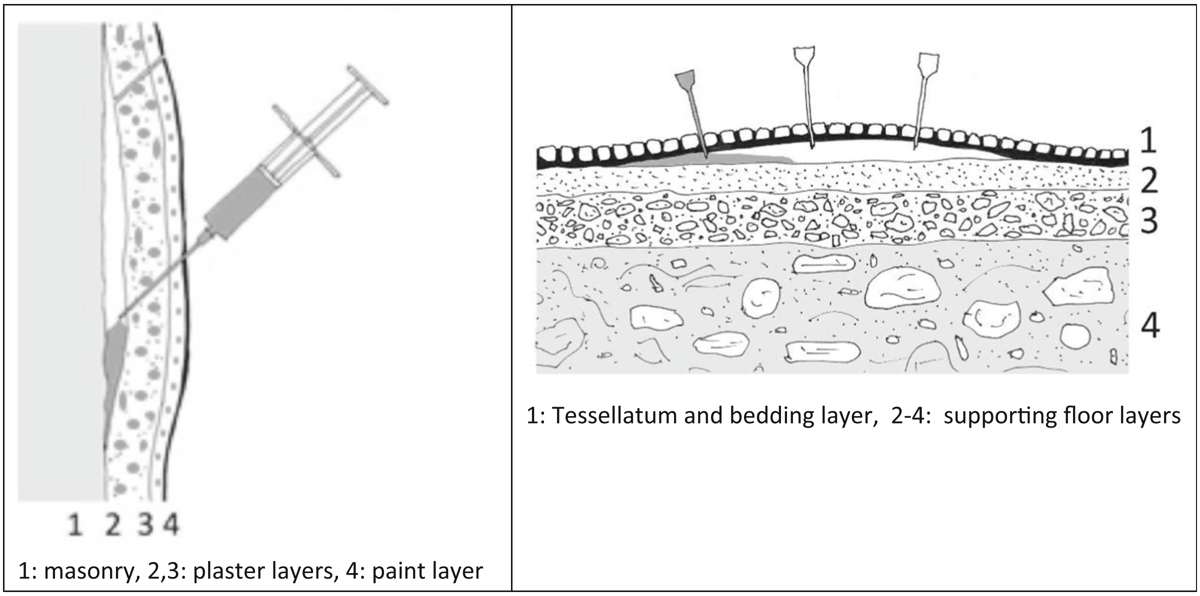

Fig. 4 Schematic diagrams showing injection of grout into a void in vertical and horizontal positions. Left: The plaster layers (2 and 3) and a paint layer (4) is detached from the support (e.g. masonry – 1). The grouting starts at the bottom. Right: Tessellatum and bedding layer (1) is detached from the supporting floor layers (2–4). Several injection holes are drilled to ensure a good distribution of the grout. Grouting starts at the sides and continues towards the middle. In both cases, it is important to provide an escape path for the air in order to ensure a good distribution of the grout, and the surface layer has to be supported against the pressure caused by the injected grout (Diagram by I. Valek and M. Dradcky)

Fig. 5 Detachment of a lime-based plaster from an internal plaster layer. Archaeological site of Dion, Greece (Roman period): Plaster consisting of two layers of a total thickness of 2.5 cm (external: 0.5 cm, internal: 2 cm) with the external layer decorated with wall paintings (Photo archive: Lab of Building Materials AUTH)

Fig. 6 Detachment due to deformation of substrate of floor mosaic layer (one part of the mosaic was detached due to subsidence another part was detached after cracking) (Galerius Palace, Greece, Roman period, photo archive: Ephorate of Classic Antiquities)

are: thermal and hygric coefficients of expansion and contraction, moisture transport, and mechanical characteristics including bond strength. Additionally, considering that often grout can leak outside the delamination, the grout should be easy to remove directly after application. In some cases, it is desirable that the color of the grout, after drying, matches that of the surrounding materials.

The development of a grouting operation requires a methodological approach. This approach should begin with a diagnostic investigation, including a thorough condition assessment, the characterization of the original and the added materials, and the development of an understanding of the deterioration causes and mechanisms (Fig. 7). This systematic planning is necessary to determine the compatibility criteria and the performance requirements for grouts, leading to the selection of the properties of an appropriate grout and to the overall development, implementation and control of the grouting intervention. Grout trial mixtures should be tested in the laboratory in fresh and hardened state. In situ applications and field tests will provide further guidance for selection. Furthermore, grout performance must be controlled and recorded, and its effectiveness should be monitored periodically after intervention.

4 Diagnostic Study

4.1 Condition assessment and investigation of causes of deterioration

Commonly, condition assessment includes the description of the layers affected by the problem, location (when possible the depth), extent and distribution of the problem, and an estimation of its severity. The assessment of the stratigraphy and bonding of the layers provides crucial information for the design or selection of a grout, which is expected to fill the voids and increase adhesion. The deterioration phenomena that can be stabilized by grouting include:

- **Cracks, fissures, and fractures** resulting from separation of one layer of the architectural surfaces from another
- **Deformation and bulging** where lack of adhesion is associated with deformation and creation of a void to be filled
- **Layering or delamination** (Fig. 8) where detachment affects laminated structures, corresponding to a physical separation of one or more layers

Deterioration problems may be related to the environmental conditions to which the outer plaster layer is exposed, or to pathology of the support on which the plaster has been applied, or a combination of these factors. Additionally, lack of adequate bonding due to improper technique of application and selection of materials, often leads to detachment problems. If the causes of deterioration are active, the problem will recur, and no grouting formulation can be effective over time. Therefore, in all cases, the cause of the damage should be considered and preventive approaches should be developed to address them prior to grouting intervention. The most common causes include:

- Causes of deterioration due to environmental conditions

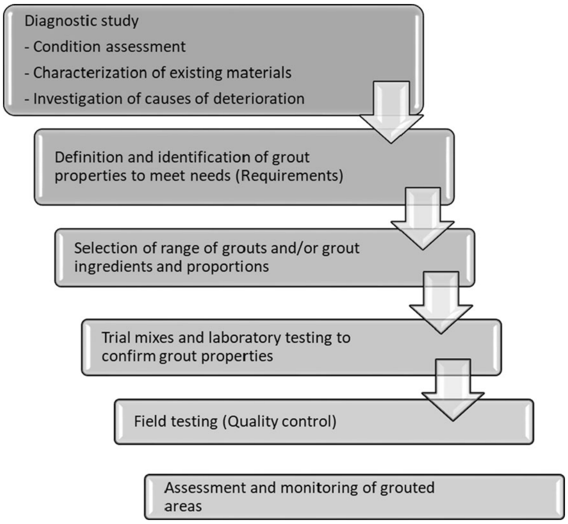

Fig. 7 Methodological approach for grouting historic architectural surfaces

Fig. 8 Detachment and loss of a lime-based plaster from the masonry support. Archaeological site of Dion, Greece (Roman period): Plaster consisted of two layers of a total thickness of 2.5 cm (external: 0.5 cm, internal: 2 cm), decorated with wall paintings. (Photo archive: Lab. of Building Materials AUTH)

- Shrinkage cracks of thin layers due to low relative humidity, heat (sun) and due to restraint of the support
- Wet-dry cycles by which the wall support and surface layer are deformed differently (i.e. lime plaster-adobe)
- Exposure to extreme temperatures (frost, fire)

- Causes of deterioration due to substrate
  - Differential deformation due to loading (static or dynamic) or hydrothermal loading cycling or to ground settlement in the case of floor mosaics
  - Salt concentration, crystallization and expansion, formed particularly at interfaces

- Causes of deterioration due to application technique
  - Cold joints between layers (timing of applying successive layers on top of each other or next to each other)
  - Inadequate workmanship of layers

- Causes of deterioration due to the previous inappropriate interventions

During condition assessment, the detached area is mapped in the design of the architectural surface and its borders are defined in order to select the points of grout injection by syringe. If the outer detached layer is very weak, a proper support should be provided. Methods often used for condition assessment are [17–20]:

- Tapping (tactile examination)
- Thermal imaging (passive or active)
- Endoscopy (to determine the number of layers, bonding)
- Acoustic tracing
- Laser speckle interferometry
- Ground penetrating radar (GPR)
- Portable 3D optical microscopy. This method enables detection and monitoring of different surface deposits (salts, biological growth), surface texture and cracks.
- Portable spectometers

Additionally, it is often necessary to measure the surface temperature, moisture rise, and salt or other contaminants content. These measures are not only essential for investigating the cause of deterioration, but understanding the environment in which the grout will be injected is also crucial for the selection or design of grouts.

4.2 Characterization of existing materials

Analysis of the existing layers and substrate materials is done in the laboratory after sampling, and provides valuable data, such as: chemical and mineralogical composition, porosity properties, hardness, toughness, modulus of elasticity, tendency to shrink or swell, as well as salt content or other inclusions (carbon grains or fibres). All these experimentally determined properties or values are needed for the selection of a compatible grout since they provide a basis on which the design of the grout will be made and further tested to confirm. The common instrumental methods used are mentioned below:

- Microscopic examination with stereoscope, petrographic microscope and SEM is used for the determination of the phases found in the microstructure (aggregates, binder matrix, salts, homogeneity, porosity, cracks, inclusions etc.).
- EDS, EDX, as well as XRD are used for quantitative chemical elemental composition and mineralogical analysis respectively.
- Thermal analysis DTA/TG/DSC are used for the quantitative identification of some mineralogical phases
- Wet chemical analysis after sieving and separation of aggregates analyzed by Atomic Absorption Spectrometry provides chemical composition of inorganic oxides
- Ion chromatography for determination of soluble salts
- IR, Raman and Mass spectrometry for identification of organic compounds, as well as oxalates, gypsum, and lime
- Non-destructive on site or semi-destructive (by taking small samples) methods for the

determination of mechanical characteristics of support and plaster layers

- Monitoring expansion and contraction, and deformations due to RH changes (by strain gages or LVDTs)
- Specific test methods for stone, fired brick, adobe, wood substrate may be added.

5 Requirements for grouting

Based on the diagnostic study, the specific technical requirements of the grout can be defined. Furthermore, preventive consolidation measures, as well as pre-treatment (desalination) of the support may be carried out to avoid premature failures. For example, fine cracks or small voids may require highly injectable grouts, while large-sized voids may require grouts with lower drying shrinkage. Location of delamination, for example at the ceilings, will require special attention to the bond strength. As stated earlier, requirements also depend on the causes of damage. In the case of an internal degradation (Fig. 9a), the treatment should not only improve the adhesion by grouting but also improve cohesion by consolidating the weakened parts. Special preparation steps to remove the debris may also be required. In plane or out of plane structural movements may cause detachment of plaster layers from the substrate (structure) (Fig. 9b). In this case, structural problems should be remedied before grouting. Repeated movements due

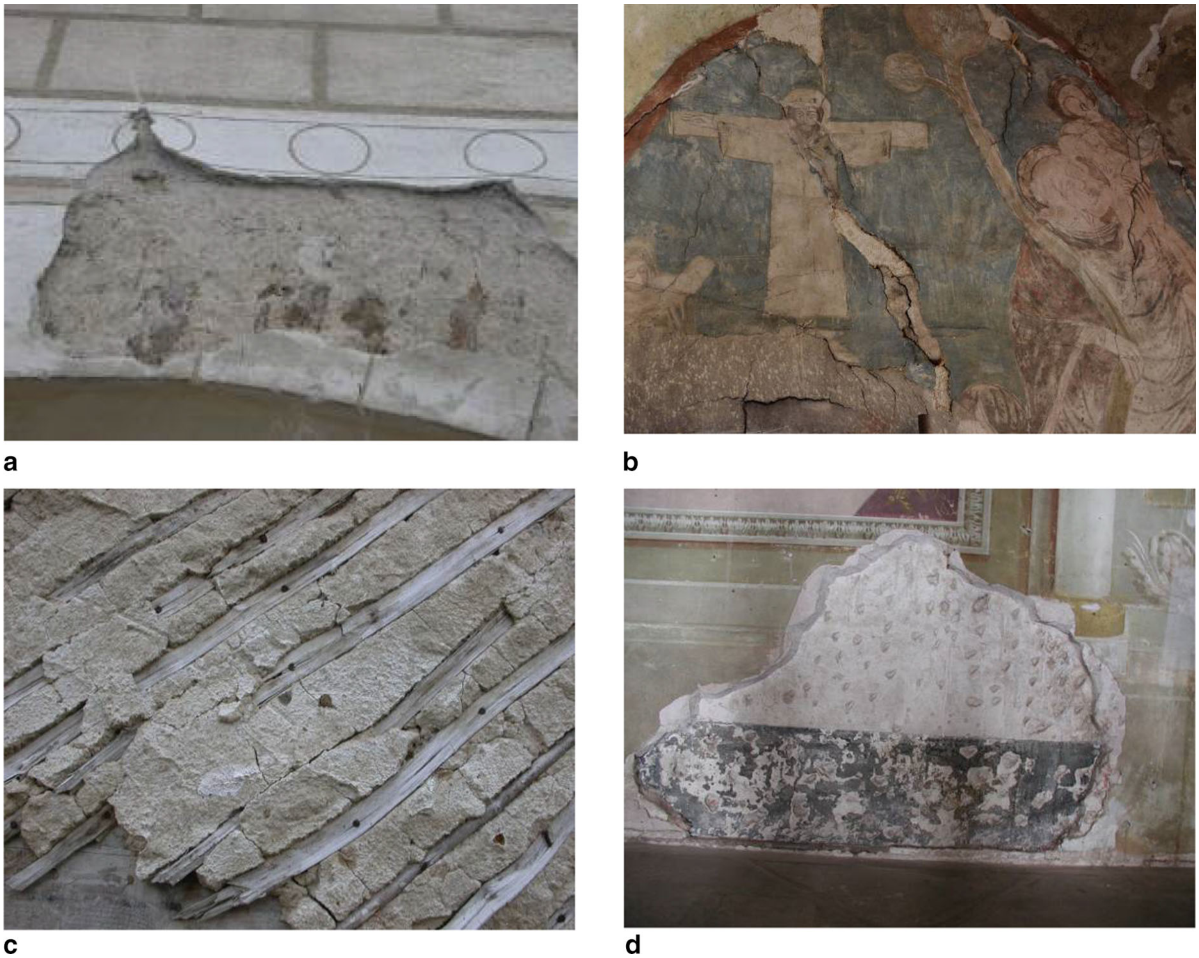

Fig. 9 Examples of delamination due to **a** Degradation and internal loss of cohesion, **b** Structural movements, **c** Differential thermal properties (as a result top plaster layer is completely missing), (d) insufficient bonding and construction related problems (photo archive: A. Velo sa)

to environmental exposures can cause cracks and delamination of plaster layers. Such movements are often not possible to be fully avoided and the repair treatment should take into account their future occurrence. Grouting should therefore respect the movements of individual parts and allow their dilatation. Different thermal properties and deformability of a coating layer and its substrate can also cause their detachment (as shown in Fig. 9c). In these cases the materials coexist together and grouting should have good adhesion and low modulus of elasticity to withstand expected deformations. Additional pinning could also be considered. Construction faults are also among the common problems of historic buildings (see Fig. 9d). Grouting could improve the bond and adhesion to the substrate, but in some cases, grouting may not be a solution (e.g. metal corrosion and swelling parts in the substrate).

5.1 Preparatory work

First of all, the necessary measures for strengthening the construction and reducing ongoing decay should be taken, such as pre-consolidation of some parts or specific treatment (e.g. structural stabilization, desalination) of layers or support before grouting.

Based on the mapped geometric characteristics (horizontal or vertical, depth, width, etc.) of cracks and voids, the method of grouting (i.e. by pressure or gravity) will be determined and a plan of locations of injection holes will be prepared. Additionally, the need for extra outer supporting media during grouting will be identified based on the state of deterioration.

Grouting is a highly specialized method and should be carried out by a conservator-restorer specialized in built heritage and decorated surfaces conservation. The technique of grouting is of high importance. For example, grouting with high pressure may increase existing detachment. Pre-wetting of surfaces with specific solutions (alcohol and water) may improve adhesion. In some cases, the addition of a diluted natural adhesive (i.e. seaweed, casein etc.) or a synthetic adhesive at high dilution 1:20 may be suggested [21]. When the presence of salts has been identified, minimal pre-wetting is preferable and the use of a dispersing agent alternative to water can be considered [22]. When pre-wetting is applied, time can be allowed between pre-wetting and grouting.

5.2 Technical requirements for grouts

The most important requirement for the design or selection of a grout is compatibility with the existing materials, including the properties and behaviour of the support system by matching chemical composition, as well as physical and mechanical properties, as long as these are not one of the causes of deterioration. However, it is often "de facto" impossible to design such a grout and the less risky alternative is adopted, fulfilling requirements, such as: (a) harmful chemical reactions between grout and surrounding materials should be avoided, (b) material introduced should be stable over time, resistant to biological attack and should not add soluble salt(s) to the system, (c) water transport properties (absorption, drying and hygroscopic behaviour) of a surface mortar-support combination should not change too much (e.g. more than 30%) with respect to those of the untreated system as a result of a grouting treatment, (d) stiffness of the grout should not surpass that of the surrounding materials, and (f) no visible change of colour due to deposit of leached grout should occur.

6 Grouts

When there is the need for grouting, there are two options that may be followed:

(a) the design of a custom-mixed grout which will be prepared with specific attention to site conditions and conservation issues, and (b) the selection of a commercial grout available on the market that meets more general requirements.

In both cases the characteristics and performance of the grout mixtures in fresh and hardened state must be tested, and their suitability for the specific case must be confirmed, even if commercial grouts usually are provided with technical data.

6.1 Custom-mixed grouts

Designing a grout, especially proportioning, requires good scientific technological understanding of binders, the role of water, admixtures, additives and fillers. For example, superplasticizers must be effective and compatible with the selected binding system and free of salts.

6.1.1 Grout ingredients

The constituents of custom mixed grouts should be selected according to the criteria defined for the specific application. The main components or ingredients of grouts, as mentioned before, are binders, aggregates or filler(s), mineral and organic admixtures, as well as some advanced technology materials added to improve grout properties or performance (see: ASTM C125 (Standard Terminology Relating to Concrete and Concrete Aggregates), ASTM C 11 (Standard Terminology Relating to Gypsum and Related Building Materials And Systems) about the terminology of the aforementioned materials). Binders are mostly powdered materials used in construction from the early ages and can be either natural (e.g. clay, pozzolan in combination with lime), or artificial (e.g. gypsum, lime, cement). Their role is to hold together all the grout ingredients, especially aggregates or fillers, and to ensure that the grout can adhere to the adjacent materials. Fillers, as defined in EN 12620, are inert, fine inorganic material, which pass through a 0.063 mm sieve and are added to improve certain properties, or to achieve special properties. Commonly used fillers are crushed limestone, powdered quartz, brick dust, marble powder, pozzolanic and other secondary (recycled) fine materials (a list of fillers used in grouts is given in Ref. [23]). Pigments used in grouts have to conform to EN 12878–2014 (Pigments for colouring building materials based on cement or lime). Admixtures are usually organic materials added in small quantities (indicated as percentage by mass of the binding system) during the mixing process, to modify the properties of fresh or hardened mixtures (mortar or grout). Superplasticizers, air entraining agents, adherence or viscosity modifiers are some widely used admixtures. As for all grout components, admixtures, should be free of sulfates (or other soluble salts) and should be effective at the pH of the binding system [24]. Currently, polycarboxylate ether-based admixtures are widely used in hydraulic binder grouts [25].

Inorganic binders can be grouped into air hardening (or non-hydraulic binders), such as clay, gypsum and hydrated lime that harden in air by drying or carbonation, and hydraulic binders, such as combinations of lime with pozzolan, hydraulic lime, Roman cement, Portland cement and blended type that contains cementitious and/or pozzolanic components. Pozzolanic components can have a natural origin (e.g. volcanic ash, trass (tuffstone), zeolites, diatomites), or an artificial one (e.g. crushed brick, silica fume, fly ash, metakaoline, calcined shale, blast furnace slag). Some of these pozzolanic materials have been used since antiquity with lime to obtain hydraulic binding systems. Today, the blended type binding systems incorporating cement and pozzolanic materials are particularly promoted in construction to address economic and sustainability issues. [39]

There are references to the use of nanoscale materials, such as nanolime and nanosilica to enhance interlayer bonding and adhesion [26]. Similarly, antifouling may also be added to increase the resistance of the grout to biological attack.

In the case of custom-mixed grouts, the selection of each grout component based on its properties and their quality control, is of great importance. The main countable criteria for the selection of the type of binder are mechanical properties. Typically, the first choice should be a binder of the same nature as that of the substrates to be re-adhered by the grout. Maximum grain size and grain size distribution of the aggregate/filler are important to achieve good injectability and packing, and to avoid bleeding. Fine aggregate/filler should be chemically compatible with the binder selected and the existing materials. It is important to identify whether the candidate filler has a hydraulic potential and therefore, a role in the hardening. Suspension media is selected based on the condition of the substrate (presence of salts) and type of binder.

It should be kept in mind that testing the quality and suitability of grout ingredients can be a costly and time-consuming process. For example, the reactivity of a pozzolanic material should be tested not only in combination with cement, as prescribed in EN 197, but also with lime, as prescribed in ASTM C 593. The content in reactive silica and fineness could also give an indication of the reactivity of pozzolan. If a filler is used, its specific surface area, water retention, and absorption properties should be determined so that its influence on the grout properties are taken into account.

Some of the most significant methods of testing properties of grout components are:

- Compositional analysis (Petrography (EN 12407, ASTM C 856, IN04B/IN04:C (SN670.115 (ATG

07A/B)), XRD, ESEM, TG/DSC, FTIR, Raman, wet chemical analysis, Atomic Absorption)
- Pozzolanic reactivity (modified Chapelle test, ASTM C 311, EN 196-5, other in-house methods, ASTM C 593)
- Particle size distribution (Sieve analysis (ASTM C 136, EN 1015–1, SIA 177.151), particle size analysis (ASTM C 810, EN933-1, EN 12620), sedimentation (ASTM D422, ASTM C110), particle size laser analysis
- Specific surface area (by BET), water absorption properties (ASTM C 67, RILEM TestNo.II.6) (EN 1015–18), water retention properties (DIN 18 555–7, ASTM C 1506, prEN 1015–8:2001)
- Salt content (Soluble salt content by Ion Chromatography (ASTM D 4327), Analytical strips and sets + photometry, Chromatography (EN 1744), EN 480–10 Water Soluble Chloride, EN 1744–5:2016 Tests for Chemical properties of Aggregates.

Most of the characteristics of the components available on the market are not provided by suppliers.

Once the type of binder and fine aggregates have been selected, following the criteria mentioned in the previous chapter 6.1.1, the proportioning of the grout mixture can be determined.

The binder/fine aggregate ratio is decided, based on experience and literature, to ensure cohesion and volumetric stability. This would be the 'prototype mixture' that is modified and refined through preliminary testing. The addition of admixtures (inorganic or organic) may improve and maintain cohesion, avoids segregation, and controls shrinkage (particularly in the case of rapid hydrating binders). In the case of lime-pozzolan systems, the hydrated lime to pozzolan ratio (by mass) is usually 1:1. Higher percentages of pozzolan may contribute to the increase of the total fine aggregate content. The ground pozzolans exhibit higher strength development. Brick dust is often used as pozzolanic material or filler in the grouting of architectural surfaces, especially those in the Byzantine style. Very fine pozzolanic materials, such as silica fume or metakaolin, increase the water demand of the grout mixture. [28]

Type and amount of suspension media is defined to obtain adequate injectability and other fluid state properties. The water/binder ratio must be kept as low as possible in relation to the required fluidity and penetrability, since it governs the strength characteristics and shrinkage deformations. Admixtures are selected to obtain the desired properties. A suitable superplasticizer may be used to adjust the water content. The water and filler content must finally be adjusted by preparing and testing trial mixes.

Preliminary tests are conducted with a variation of the 'prototype reference' mixture (defined above).

Once the composition is satisfactory, in terms of fresh state behaviour, the hardened state properties are evaluated. Through an iterative process, the harden state properties may indicate the need to change components and amounts. Changes are made and testing starts again from fluid state to harden.

In Tables 1, 2 and 3, indicative values of properties measured for acceptable custom-mixed and commercial grouts are given [25, 27, 28, 30, 35, 36].

6.2 Commercial grouts

The available grouts on the market may be grouped as:

(a) grouts based on polymer systems, which react with or without any organic solvent, and (b) grouts based on inorganic hydraulic binders.

Such grouts, as in group (a) are currently not recommended for repairing heritage structures due to the occurrence of a number of failures in the past [7, 8]. Commercial grouts are usually premixed, requiring only the addition of water. They can be easily transported in bags or buckets. Their quality (i.e. properties of constituents and their proportion) is standardized and controlled by the manufacturer. The main constituent is often hydraulic lime (natural or artificially produced), fillers and organic admixtures for improving properties. Grout properties, such as minimum values for mechanical strength, elasticity, water absorption coefficient etc. are guaranteed, if the suggested guidelines for mixing and preparation are followed. Manufacture-provided properties of these grouts should be compared with the technical requirements defined for the site to reduce the number of possible candidates. It is important to note that this comparison may not be straightforward, since manufacturers frequently use different standard test methods. It is suggested to confirm the properties of commercial grouts by preparing and testing trial mixes in the laboratory before making any decision.

Table 1 Field tests

Field test name	Test method
Injectability with syringe	Grout is poured into a vertically held syringe that is partially filled with granular material, and pressure is applied on the grout with the plunger
Flow with syringe	Grout is poured into a vertically held syringe that is partially filled with granular material, and the penetration of grout into the intergranular network is observed
Flow on plastered tile	A constant volume of grout is injected into a vertical crevice in plaster applied to a vertically placed tile, and the distance it flows is determined
Expansion and bleeding	Grout is placed in a graduated cylinder, and accumulation of bleed water and amount of expansion are measured
Wet density	Grout is weighed in a syringe, and the density is calculated
Properties during setting and curing	
Drying shrinkage	Dimensional changes, including cracks, of a grout specimen placed in a plastic or mortar cup are observed
Final setting time	The time of setting is determined by periodic insertion of a cannula into a cup filled with grout until solidification occurs
Hardened properties	
Capillary water absorption	Water absorption of a hardened grout specimen is measured by a gravimetric method, following the procedure for the laboratory test
Water vapor transmission rate by the wet cup method	The rate of water vapor transmission through a cured grout sample is determined gravimetrically in field conditions

7 Preparation and testing of grouts

7.1 Mixing procedure

The mixing parameters, such as the type of mixer (of low or high frequency rotation), the time of stirring, and the order of the addition of ingredients, affect the performance of the grout mixture (and must be adjusted to determine the best performance of the fresh grout (better homogeneity, less segregation, better injectability).

According to the literature [29], a high speed (2000–3000 rpm) stirrer is suggested for an efficient and effective grout mixture. In practice, different mixing procedures are followed, the effectiveness of which could be checked by measuring variation in bulk density or other properties of the grout. One procedure is the addition of all powders with ¾ of water mixing for about 2 min, addition of the remaining water with admixtures followed by another six minutes stirring [38]. However, according to the literature [22], this procedure was ineffective in using silica fume as a constituent. Another procedure suggested [31] is: the addition of pre-estimated water and superplastisizer, as well as additives into the mixer, and hand stir for 30 s. The premixed powders are subsequently added within 30 s., while mixing at low speed (200–300 rpm). Finally, mixing is continued at high speed for 4 or 5 min. Since there is no relevant standard for the mixing procedure of lime-based grouts, a criterion for adopting a procedure is checking the achieved homogeneity of the mixture in the place of use. In relation to commercial grouts, the mixing procedure suggested by the supplier should be followed.

7.2 Laboratory test methods

7.2.1 Methods for testing fresh state properties

A fresh commercial or custom-mixed grout mixture should exhibit adequate fluidity, penetrability, and volume stability for some time after water addition, and a reasonable setting-hardening time of the binding system.

The following test methods can be used to assess the performance of fresh grout, the first three (Fluidity, Volume stability, and Injectability) being the most important. For most grouts, modifications are needed to meet the particularities of each case study.

- Rheology measurements

  Flow by Marsh cone (ASTM C 393, UNI 11152, EN 196-3)

Table 2 Indicative properties of commercial grouts

	Type of grout	Constituents	Source	Characteristics in fresh state			Characteristics in hardened state		
				Fluidity	Volume stability bleedity %	Penetrability	28-d strength compressive/ flexural (MPa)	28-d Modulus of Elasticity (GPa)	Shrinkage %
1	Hydraulic LEDAN TB1	W/B = 1.25	*	EN 445 Marsh cone 7.05	Expansion: 3.3%	Sand column (EN 1771) 3.5 s	–	–	–
2	Hydraulic UNILIT B fluid 0/0	NHL + additives W/B = 0.7	[36]	good	good < 1%	Good: 22 (s)	1.53/1.27	–	–
3	Hydraulic PHLC (ASTM C1707)	Lime + metakaoline + cement + sand filler + SP	[28]	Marsh cone good: 13–25 (s)	Good: 1 h < 3%	adequate	10/(-)	–	28-d 0.06%
4	Hydraulic FASSA BORTOLO	W/B = 0.46	*	EN 445 Marsh cone 11.03	Reduction: 13.3%	Sand column (EN 1771) 2.47	4.78/1.92	0.925	–
5	ALBARIA	W/B = 0.53	*	EN 445 Marsh cone 10.31	Reduction: 6.7	Sand column (EN 1771) 3.52	5.09/1.92	0.76	–

Porosity Properties			Adhesion (MPa)
Porosity /Water absorption	Coeff. of capillary elevation	Water vapour transmission	
–	–	–	–
–	–	–	–
Water absorption 9.6%	-	1.9 g/m^2/h	Very good
–	–	–	–
–	–	–	–

*Report of the Laboratory of Building Materials, AUTH, in the frame of RILEM TSG Round Robin tests [38]

Viscometer (ASTM D 4016) (UNI 11152-05 by viscometer)

- Volume stability. Expansion and bleeding (ASTM C 940–03)
- Injectability: with sand columns (EN 1771–2005, NORM NFP 18–891, 1986*) and with syringe (Field testing procedure [31]).
- Water retention and release (DIN 18 555–7, ASTM C 1506–03, prEN 1015–8:2001)
- Wet density (ASTM C 185–02)
- Time of setting by Vicat needle (ASTM C 191). Measurements of setting with time passing.
- Initial adhesion (EN 1015–12:2000)
- Early shrinkage (ASTM C 490)

7.2.2 *Curing conditions*

Grouts are injected between layers and are influenced by environmental conditions, as well as by the interlayer substrate porosity and hydroscopic (hygric) properties.

A proper curing regime depends on the binder type. For example, for air-hardening (hydrated lime) air curing is suggested, while for hydraulic type binders (hydraulic lime, lime-pozzolan, cement-lime etc.), high relative humidity (RH > 90%) and temperature 20 ± 1 °C, are recommended according to preEN 998-–1, EN 1015-11. In practice, on-site measures are taken to prevent rapid loss of moisture by drying (such as prewetting holes for grout penetration) [37].

7.2.3 *Methods for testing properties of hardened grouts*

Although compressive strength constitutes a basic criterion that determines the quality of an inorganic binding system, in the case of grout, other properties such as adhesion, ability to fill voids, and hardening without much shrinkage are of great importance. Other target properties are: minimal physical and chemical alteration to the plaster and paint layer(s), as well as porosity, water vapour permeability, hydrothermal behaviour and mechanical strength similar to that of the existing materials, as well as durability and chemical stability.

The testing of properties is carried out in the laboratory on hardened grout specimens mainly for comparison of different grout mixtures. Among tests, the most important are those determining:

- Early and long-term shrinkage, ASTM C 1148
- Adhesion strength, EN 1015-12 (2000) (Fig. 10)
- Water absorption coefficient and water vapour permeability EN 1015-18 (2002), EN 1015-19 (1999)
- Compressive and flexural strength, EN 1015-11 (1999)

The adhesion test EN 1015–12 (2000) described in the literature [32, 33] (see Fig. 10), seems adequate for grouts used for reattachment, although the repeatability of the measured values is low. However, minimum required adhesion values, such as > 0.1 MPa, given in ref. [34], are achieved only in the case of grouts with hydraulic binders, while hydrated lime grouts usually exhibit lower values (0.01–0.08 MPa).

Durability of grouts In reality, durability refers to the ability of grouted layers to endure and maintain their stability after exposure to wet-dry, freeze–thaw and salt crystallization cycles, as well as to UV radiation or thermal deformation. The durability of grouts themselves may be checked comparatively on specimens 40 × 0× 160 (mm) following relevant test methods, or on specific specimens simulating conditions of the material to real in situ situation, such as small cylinders coming from sand column test [21, 25, 35]. It is proposed to follow the existing test methods (or the modified ones) after selection of the

Fig. 10 *Testing adhesion of grouts SIST EN 1015–12 (*photo archive: A. Padovnik*)*

Table 3 Indicative properties of custom-mixed grouts

	Type of grout	Constituents	Source	Characteristics in fresh state			Characteristics in fresh state		
				Fluidity	Volume stability bleeding %	Injectability	28-d strength compressive/flexural (MPa)	28-d Modulus of Elasticity (GPa)	Shrinkage %
1	Air-hardening	Lime + Limestone filler W/B = 1.08	[25]	adequate	1 h < 1%	good	1.08/0.8	–	–
2	hydraulic	NHL 2 W/B = 0.61	[25]	Marsh cone, ASTM C939 9.3 s	Reduction: 0.7%	sand column (EN 1771): 3.41 s	1.86/1.10	0.046	0.4
3	hydraulic	Lime + Nat. pozzolan + SP W/B = 1.22	[35]	Marsh cone ASTM C939 10 s	Reduction 1.2%	sand column (EN 1771): 2.10 s	28d. 1.14/ 0.04 90d 2.29/ 0.10	28d. 0.02 90d 0.023	28-d volume shrinkage 3.3
4	hydraulic	Lime + pozzolan + Limestone filler + SP W/B = 0.86	[25]	Marsh cone ASTM C939 10.5 s	1.3%	sand column (EN 1771): 7.5 s	2.49/1.13	0.051	–
5	hydraulic	Lime + pozzolan + 10% cement + SP W/B = 1.21	[25]	Marsh cone ASTM C939 10 s	Reduction 4.8%	sand column (EN 1771): 1.84 s	0.91/0.40	0.040	28-d 1.5

Porosity Properties			Adhesion (MPa)
Porosity/ Water absorption	Coeff. of capillary elevation	Water vapour transmission	
–	–	–	0.1–0.3
–	–	–	–
39%	2.29		-
–	–	–	–
44%	1.71		-

Type of grout	Constituents	Source	Characteristics in fresh state			Characteristics in fresh state		
			Fluidity	Volume stability bleedity %	Injectability	28-d strength compressive/ flexural (MPa)	28-d Modulus of Elasticity (GPa)	Shrinkage %
Hydraulic	Lime + pozzolan + brick dust cement 10% + SP W/B = 0.90	[35]	Marsh cone ASTM C939 10.2 s	1.0%	sand column (1.95 s)	1.72/0.84	0.028	–
Hydraulic	NHL5 + Lime stone filler + SP W/B = 0.8	[36]	adequate	1 h: 2%	adequate	2.82/1.90		
Hydraulic	NHL3.5 + Lime stone filler + SP W/B =	[36]	adequate		adequate	3.10/1.65		
Hydraulic	Lime putty + tuff + Limestone filler + SP W/B = 0.97	[23]	adequate	1 h: 2.8%	adequate	2.8/0.8	–	–
Hydraulic	Lime + trass + cement 30% + sil. fume + SP W/B = 0.98	[30]	adequate	acceptable	adequate	6.5/1.7	–	–

Table 3 continued

Porosity properties			Adhesion (MPa)
Porosity / Water absorption	Coeff. of capillary elevation	Water vapour transmission	
–	–	–	–
–	–	–	–
			–
–	–	–	–
–	–	–	Shear box τ_{max} 1.78

most significant environmental parameters for the given on site situations. For example, in Mediterranean countries, wet-dry, salt crystallization cycles, and UV radiation are of greater importance than freeze–thaw resistance. However, the existing standards for testing durability refers to concrete/cement mortars (such as, ASTM C666, RILEM TC 176-IDC Internal damage of concrete due to frost action), as well as natural stones, and they cannot be applied in the case of soft lime-based grouts. Most researchers use EN standards with modifications. These are EN 12371 (about frost resistance of natural stone), EN 12370 (about natural stone resistance to salt crystallization), and RILEM TC 25-PEM-1980 (recommended tests to measure the deterioration of stone and assess the effectiveness of treatment methods (test V.1a, test V.1b and test V.2)).

7.3 Field test methods

To control the quality of the grouting intervention, a system of on-site tests should be established, particularly in the case of large projects, and specialized technicians should be employed for their execution.

A list of field tests follows in the literature [31], where relevant photos and details of the procedures are included (Table 1).

According to reference [29, 31], laboratory tests (often modified standard tests) provide more precise and quantitative data, allowing comparison among grout formulations and among different laboratories, while field tests (no-standard tests) are easier, quicker and very helpful in adapting (especially in the case of working properties) a grout to local on-site conditions (Table 3).

8 Assessment and monitoring of grouted areas

The long-term stabilization of grouted areas may be checked by various test methods, from relatively simple to advanced technology-based. These methods could also be included in a monitoring program.

- Acoustic, ultrasonic or impact echo test methods can be used before and after grouting, showing the level/effectiveness of stabilization, since they detect the voids or, in general, discontinuities in a system.

- Laser Speckle Pattern Interferometry. It can be used for stress–strain measurements, vibration analysis and non-destructive testing.
- Infrared thermography: this method enables detection of defects (cracks, voids, delamination) within a depth of approximately 10 cm
- Clustering of simultaneously obtained GPR and thermographic data through the use of unsupervised fuzzy clustering methods: this method can be used when GPR and thermography are applied simultaneously
- Ground Penetrating Radar GPR[1] for detection of pipeline underground metallic and non metallic utilities leakages and voids
- 3D digital image correlation (DIC) photogrammetry, a method for the monitoring of surface crack propagation and render detachment

An extensive and methodologically correct case study report concerning the grouting of Case 85 Wall paintings in Mogao China is given in the Getty website: www.getty.edu The Mogao Cave 85 Project Report. Another short presentation of a grouting case study is given in Ref. [37].

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this manuscript.

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