Lime-based injection grouts for the conservation of architectural surfaces

Beril Biçer-Şimşir, Isobel Griffin, Bénédicte Palazzo-Bertholon and Leslie Rainer

Abstract

This paper reviews the literature on injection grouts used in the conservation of architectural surfaces including wall paintings, plasters, and mosaics. It presents the materials and techniques of grouting, and methods of evaluation, focusing on lime- and hydraulic lime-based grouts. This review indicates that a variety of materials have been investigated for use as injection grouts, and numerous commercial and custom-mixed grouts are available to conservators today. However, there are few standard or well-established methods to assess them, which have led to a wide range of test methods for their preparation, characterization, and evaluation. It is clear that a systematic study of the working and performance properties, test methods, and preparation and curing conditions is needed, as is an evaluation of products being used in the field.

Introduction

In 2004, the Getty Conservation Institute (GCI) initiated an interdisciplinary study involving the Field Projects and Science Departments to evaluate injection grouts used in the conservation of architectural surfaces, including wall paintings, plasters, and mosaics in situ.[1] The project aims to combine laboratory testing and field study to inform conservators about grouts currently in use, and to improve conservation practice. As an initial step, an extensive bibliography was compiled on the subject from a wide variety of published sources and a literature review was prepared.

This paper is on injection grouts used to reattach wall paintings, plasters, and mosaics to their original supports, and not structural grouts used in the strengthening of historic buildings and other structures. Even so, some references that include the evaluation of structural grouts and repair mortars are included when the information is considered relevant. This paper aims to provide a critical review of the literature on lime- and hydraulic lime-based injection grouts in particular to identify trends in the field, and potential areas for future research. Unpublished research, reports, and anecdotal experience are not included and therefore this review may not entirely reflect the situation of injection grouts being used in conservation. However, the authors believe that it can still provide a legitimate indication of current thinking and practice.

In this paper, injection grouting is defined as the introduction of a bulked fluid material injected behind a wall painting, plaster, or mosaic to fill cracks and voids and re-establish adhesion between delaminated layers. Grouting is an important method for the stabilization of architectural surfaces in situ. As conservation in situ has become established practice, researchers and conservators have recognized the need for injection grouts as an alternative to detaching wall paintings and mosaics, and this trend is clearly mirrored in the literature. The earliest published research on the development of grouts for wall paintings and mosaics is from the 1980s; Ferragni et al. report on the testing and implementation of injection grouts by the International Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM) [1, 2]. While publications relating to wall paintings continued to appear from this time onwards, articles relating to grouts for the conservation of mosaics in situ did not appear until almost a decade later [3–5].

This paper covers the materials used for grouts, their working properties and performance characteristics, preparation and curing, methods for evaluating grouts, and application techniques.

Materials for grouting

Selection of materials

The selection of materials for grouts generally depends on a range of factors, including the desired working properties and performance characteristics, and the availability of materials. Cost may also be an important factor, particularly for large-scale interventions.

In general, it is stated that grouts are selected to be compatible with the original material [6, 7]. Although the types of compatibility investigated – physical, chemical or mechanical – are not always clearly given, in most cases, researchers indicate that they select a principal binder for the grout that is similar to the original material [8, 9]. Some studies aim to develop compatible grouts by undertaking analysis of the original materials, following the example of repair mortars [1, 2]. For example, in order to develop conservation mortars for Hadrian's Wall in northern England, the Smeaton Project first analyzed original mortars from the wall [10, 11]. There are also studies analyzing original materials in order to try to match the measured properties of the original and repair mortars [12–14]. However, matching the composition of original mortars and plasters presents a problem in the case of injection grouts since it is probable that the same composition will not produce working properties that are desirable for an injection grout, such as flow. As the original mortar will often be weakened and deteriorated, a repair mortar or grout made to the same formula is likely to be stronger, which may be

[1] http://www.getty.edu/conservation/field_projects/grouts/index.html

undesirable [15]. Nonetheless, the issue remains a concern since the grout, as an intervention material, should be compatible with the original materials, capable of reinstating the integrity of the system without unintended consequences.

Binders

Hydrated lime is one of the most common binders used in injection grouts, since it is likely to be compatible with original lime-based materials. Ballantyne supports the use of traditional materials and argues that a simple hydrated lime putty and sand grout is adequate in many circumstances [16]. Asp [17] tested a number of grouts, including commercial grouts, and found that although the working properties of a basic hydrated lime and ground sandstone grout were not as good as some others, its long-term performance in situ was successful. Michoinovà [18] defends the idea that non-hydraulic lime-based grout can be used for wall paintings, but with additives such as polymer dispersions, fluidizers and water reducers. The most discussed disadvantage of using hydrated lime as a grout binder is that it requires exposure to carbon dioxide (CO_2) in the air to set and, with minimal exposure to air inside a wall, carbonation can only proceed very slowly, so the development of strength and durability will also be slow. Many authors therefore argue that hydrated lime should only be used as a binder if pozzolanic fillers are present to react with it, permitting a setting reaction in the absence of air [1, 2, 9, 19–21], while others have investigated ways of increasing the rate of carbonation. Baglioni et al. [9] studied additives such as ethyl carbamate and ammonium carbamate that might aid setting by producing CO_2 in an alkaline environment. Maryniak-Piaszczynski [22], and Strotmann [23] showed that injection grouts made with dispersed hydrated lime carbonate set much more quickly than non-dispersed hydrated lime, and produce a material with higher resistance to weathering. Other disadvantages of hydrated lime-based grouts may include high shrinkage [19] and poor injectability [24].

Hydraulic lime is commonly used for grouts because, like hydrated lime, it is likely to be compatible with original lime-based materials. Several studies state a preference for hydraulic lime over hydrated lime [1–3, 25, 26]. Others suggest that hydrated lime and hydraulic lime should be used in combination [27]; the advantage over hydrated lime is that it sets in the absence of air, and hence is particularly suitable for grouting internal voids. The development of strength is quicker and durability is higher than for a hydrated lime-based grout, and this makes it a good choice for situations where the grout will be used for deep voids, or will have a structural function and is likely to be exposed to freezing conditions [16, 27]. However, there can also be some disadvantages to using hydraulic lime binders. They can be excessively strong [28] and their performance varies dramatically depending on the type used [10, 29]. Sourcing a good hydraulic lime may be problematic, as some are manufactured artificially by adding cement or pozzolans to hydrated lime, and it has been suspected that some products described as natural hydraulic limes have included cement [29]. Other potential disadvantages of hydraulic lime-based grouts, as with grouts based on hydrated lime, may include high shrinkage [19] and poor injectability [24].

Investigations into non lime-based grouts include earth-based grouts for earthen (e.g. adobe) supports [13, 30, 31], non-aqueous grouts using synthetic organic resins with or without fillers [16, 32, 33], and cement-based grouts, which are mainly used for masonry consolidation in a structural context as opposed to reattachment of an architectural surface [34–38]. However, more recently, cement-based grouts have been considered for the conservation of modern wall paintings on cement-based supports or mosaics re-laid on concrete slabs. This paper is limited to a review of lime- and hydraulic lime-based grouts, and other binders are not further discussed.

Fillers

The fillers act as bulking materials, thereby reducing shrinkage and controlling mechanical strength. Fillers encountered in the literature are given in Table 1. By far the most commonly used inert filler is sand. It is inexpensive, easily obtainable, and has a long tradition of use. The particle size of the sand is important: a small particle size makes for a more easily injectable grout, but it has been shown that coarser sand produces stronger, stiffer grouts, which may also be desirable [39]. A broad particle size distribution is therefore recommended as long as the particle size remains fine enough to be injected. Some authors [17, 40] note that grouts containing sand have a tendency to segregate, and are fairly heavy, therefore light-weight fillers have also been investigated in the literature.

Some fillers such as pozzolans may function both as fillers and – through their reaction with lime – as binders. Pozzolans have been defined as 'materials which, though not cementitious in themselves, contain constituents which will combine with lime at ordinary temperatures in the presence of water to form stable insoluble compounds possessing cementing properties' [41]. The name, pozzolan, was originally given to vitreous pyroclastic material produced by volcanic action. However, the conventional use of the term pozzolana or pozzolan in a generic way prevails, and it is used here to describe both natural and man-made materials that react as described above.

The main advantage of using pozzolans is that the grout will set in the absence of air and under wet conditions, which is why they are commonly used for applications where carbonation would otherwise progress very slowly. Ferragni et al. state that they block the formation of insoluble calcium carbonate efflorescence since they bind free lime [1, 2]. Materials must be finely ground in order to function as pozzolans. It is noted that the use of ultrafine materials improves injectability and adds stability to the mixture, but may also increase viscosity and/or reduce workability [42, 43]. Natural pozzolans may be high in soluble salts. Their use was initially rejected by Ferragni et al. because of the high content of potassium ions. Their pozzolanic character affects mechanical properties. Griffin found that a

Table 1 Fillers

Fillers	*Reference*	*Comments*
Inert fillers		
Sand	[17, 39, 40]	
Marble dust	[25, 44, 45]	
Quartz filler	[44, 45]	
Powdered limestone	[45, 46]	
Graphite dust	[45]	
Crushed dolomite	[45, 47]	
Glass microballoons	[1, 6, 31, 37, 43, 44, 48]	Increased penetration; no segregation; improved stability.
Ceramic microspheres	[49]	Light-weight filler.
Pumice stone	[48]	Light-weight filler.
Fumed silica[a]	[17, 48, 50, 51]	Light-weight filler; good injectability and durability [50, 51]; but severe shrinkage [17].
Pozzolanic fillers		
Superventilata pozzolana	[1]	Natural pozzolan.
Santorini earth	[36, 40, 50, 51]	Natural pozzolan.
Skydra earth	[40]	Natural pozzolan.
Brick dust	[1, 2, 10, 20, 21, 28, 40]	Artificial pozzolan made from calcined clay; low content of soluble salts [1, 2, 20, 21].
Diatomaceous earth/dicalcite	[1, 2, 20, 21]	Material of organic origin; high porosity and low density [20, 21]; reduced injectability due to the thixotropic effect; need for high water content; high shrinkage upon setting [1, 2, 20, 21].
Trass	[12, 20, 21, 34]	Natural mineral; good performance but high soluble salt content [20, 21].
Crushed dolomite	[47]	Natural mineral; improved setting and hardening.
Granite dust	[45]	Natural stone; weak pozzolan.
Ceramic powder	[45]	Fired clay.
Bentonite	[35, 52, 53]	Fired clay.
Metakaolinite	[17]	Fired clays; found to set too quickly.
Fly ash	[17]	Industrial by-product; easily injected; does not set too rapidly, but poor adhesion in situ.
High temperature insulation material (HTI)	[10, 29]	Industrial by-product; does not perform as well or as consistently as brick dust.
Pulverized fuel ash (PFA)	[1, 27]	Industrial by-product; good flow; less tendency to separate than brick dust and HTI.
Ground granulated blast furnace slag (GBFS)	[20, 21, 52]	Industrial by-product; high strength, low porosity and low water vapor permeability if used alone [20, 52]; suggested to be mixed with other fillers.
Silica fume[a]	[34, 36]	Industrial by-product.

[a] Fumed silica is an exceptionally pure form of silicon dioxide made by reacting silicon tetrachloride in an oxy-hydrogen flame. It is mainly used to control flow properties and does not have noteworthy pozzolanic properties. Fumed silica is generally confused with silica fume. Silica fume is a by-product collected from electric arc furnaces in the production of silicon and ferrosilicon alloys. Its extreme fineness and high silica content are the reasons for its pozzolanicity.

natural pozzolan grout was too strong if the pozzolan was used alone, and suggested the addition of inert fillers [20, 21].

Brick dust is a pozzolan commonly used for grouting wall paintings, plasters, and mosaics [1, 2, 10, 20, 21, 28, 40]. Its pozzolanic properties depend on several conditions including the burning temperature (low-fired brick has high pozzolanicity) [2], the type and amount of clay, and particle size distribution. The Smeaton Project suggested that brick dust particles in the lower particle size range (<75 μm) act as a pozzolan, accelerating setting and creating a higher strength mortar, while particles in the higher particle size range (>300 μm) act as porous inert particulates aiding carbonation and improving resistance to frost and salt crystallization [10]. Brick dust may increase the amount of water required to obtain a grout with suitable working properties since it has a high surface area (being ultrafine) and the particles are porous so that they absorb water [28]. Brick dust improves the fluidity of the grout, but if the content is high, thixotropic behavior[2] is observed [40].

[2] Thixotropic behavior is the tendency towards gelling and showing resistance to flow without additional agitation.

Water

Typically, the liquid present in injection grouts is water. The main role of water in lime- or earth-based grouts is to act as a dispersive medium for the ingredients. In lime-pozzolan-, hydraulic lime-, and cement-based grouts it also contributes to the chemical reaction. The proportion present in the initial grout mix is important; a high water content gives improved flow and injectability [38, 54, 55], but also leads to less stable suspensions with segregation and bleeding [56] and greater volume change upon setting, with more potential for cracking [6]. High water content increases the total porosity and causes reduced mechanical strength once the grout has hardened. Fontaine et al. [39] found that high water content resulted in decreased mechanical strength but increased elasticity.

Additives

Various materials, other than the binder, filler(s) and water, are incorporated into grout mixtures in limited amounts to modify specific properties. For example, fluidizers/plasticizers are used to modify the flow properties, and accelerators and retarders to control setting. Additives discussed in the literature are given in Table 2.

The most common additives are synthetic organic materials, which are used as fluidizers/plasticizers. Traditionally, natural organic additives have been used for mortars but are not found in the literature for grouts, with the exception of casein [1, 2]. Some researchers conclude that the use of natural organic additives is risky since the source materials may be inconsistent and have very specific requirements for their preparation. Additionally, they may deteriorate and promote biological attack. Therefore, some researchers believe that the use of synthetic organic additives is preferable [57]. However, most fluidizers and plasticizers are manufactured for control of the fluidity of cement grouts, mortars and concrete rather than lime-based grouts. Availability and low cost are the two main reasons for their use in grouts for the conservation of architectural surfaces [56]. There is a need for the development of fluidizers/plasticizers specifically for lime-based grouts since the effectiveness of available products has not been tested for these systems.

Fluidizers/plasticizers improve the injectability of grouts, and reduce segregation [6] and the water content required to achieve the desired working properties. They may decrease the viscosity of the

Table 2 Additives

Name	*Reference*	*Comments*
Fluidizers and plasticizers		
Polynapthalene sulfonate calcium salt (PNSCS)	[9]	Surfactant.
Sulfonated melamine and/or napthalene formaldehyde	[34, 43, 54]	Surfactant.
Sodium gluconate solution	[1, 2, 6, 25, 28]	Surfactant.
Sodium salt of polycarboxylic ester polymer	[56]	Surfactant.
Methylhydroxyethylcellulose	[46]	
Methyl ethyl ketone	[33]	
Calcium caseinate compound	[58]	
Methyl cellulose	[58]	
Commercial fluidizers and plasticizers		
El Rey® Superior 200	[43, 49]	Acrylic emulsion.
Rhoplex™ E-330	[49]	Acrylic emulsion.
Primal™ AC 33	[1, 2, 7, 25, 43]	Acrylic emulsion.
Rheobuild® 716	[35, 53]	Sulfonated naphthalene with polyhydroxylated polymer.
Rheobuild® 561	[28]	Calcium naphthalene sulfonate.
Sikament® 10 ESL	[42]	Sulfonated vinyl copolymer and sodium salt.
Glenium® 27	[52]	Modified polycarboxylate ether.
Other additives		
Ethyl carbamate	[9]	Gas-producing agent; facilitates setting in absence of air; improves fluidity.
Ammonium carbamate	[9]	
Fluid coke	[1, 2]	Gas-producing agent; not tested; difficult to obtain.
Aluminum powder	[59]	Expansive grouts; low density; high porosity.
Dispersed pyrogen silica gel	[37]	Reduces sedimentation.
Bentonite	[32]	Reduces the separation of a pulverized fuel ash grout.
Barium hydroxide for lime-based grout	[59]	Reacts with carbon dioxide to form barium carbonate and binds the calcium hydroxide.
Gypsum	[19]	Accelerator.
Casein	[1, 2]	Retarder.
Sugar	[19]	Retarder.

grout at the start of the injection [58], improve water retention [40, 46], and improve adhesive properties [1, 2, 43, 49, 58]. They may also create a strong and lasting bond with the support [58], reduce the uptake of water by capillarity [43], and improve durability [9, 34]. A consequence of reduced water content is higher mechanical strength [1, 2, 9, 34, 58] and lower shrinkage [35]. Acrylic emulsions may produce less dense and more durable grouts due to foaming during mixing [49]. Disadvantages may include the degradation of polymers, which may also adversely affect performance of hardened grouts and cause the release of soluble salts. The latter danger can be minimized by using polycarboxylic ester polymers which have a lower concentration of ionic groups and polymers with a low concentration of cations of alkaline metals [56].

Air-entraining agents, which are used to introduce stable microscopic air bubbles to improve durability by reducing stresses caused by freezing water in pores, are commonly cited in mortar studies for improving freeze-thaw durability but literature on their use in grouts is limited [19]. Oldenbourg recommends foam grouts, based on a cement and gypsum anhydrite binder [33]. These flow very easily and have low water content, leading to low density, high porosity, and high durability. However they also have a high soluble salt content [33], which, in the authors' opinion, should be avoided.

Custom-mixed and commercial grouts

Custom-mixed grouts are defined as grouts that are formulated by the user and which contain both non-proprietary and proprietary materials. Examples include the ICCROM grout containing hydraulic lime, brick dust or superventilated pozzolana, sodium gluconate and an acrylic emulsion (Primal™ AC 33), developed for wall paintings and mosaics with lime-based supports [1, 2, 19, 25, 32, 37]. Matero and Bass developed a grout that contains hydraulic lime, ceramic microspheres, sand, and an acrylic admixture (El Rey® Superior 200) in water solution for the adhesion of lime plaster to earthen supports [43, 49]. Jerome et al. describe a grout developed at Columbia University, and modified for earthen structures [42], containing hydrated lime, fumed silica (Cab-O-Sperse® A3875) and a superplasticizer (Sikament® 10 ESL). There can be drawbacks in the use of custom-mixed grouts. These are typically related to inconsistencies in their preparation by different practitioners, and the difficulty of obtaining specific products used in the original custom mixes if they are not widely available, or if they are discontinued by the manufacturer.

Commercial grouts are generally found to be easy to prepare and have good working properties. However, disadvantages include undesirable performance characteristics, such as excessive strength and high soluble salt content. There appears to be a lack of confidence in commercial grouts, noted in the literature, related to manufacturers changing the composition of, or discontinuing, a product at any time.

In the thirty years since the first injection grouts were developed at ICCROM, a large number of commercial grouts have come on the market. Many of those in current use by conservators have not been comprehensively tested. However, there is an increasing interest in the evaluation and comparison of their properties as shown by recent publications [6, 60]. Commercial products mentioned in the literature include Ledan grouts (Ledan TB1 [24, 37, 61–65], Ledan TB03 [66], Ledan TC1 [66], Ledan D1 [17], Ledan D/F [17], Ledan Ital B1 [3], Ledan TC1 PLUS [67] and Ledan SM02 [68]); EMACO RESTO 1 [24]; Malta 6001 and Malta 6002 [64]; ICCROM commercial grout [2, 69]; Jahn M40 [42, 70]; and PLM grouts (PLM A [68] and PLM M [67]). Although modifications of commercial grouts are not recommended by the producer, there is published work that reports testing of various additives for Ledan grouts [33, 37, 48, 62].

Working properties and performance characteristics of grouts

Almost all studies touch upon the desirable working properties and performance characteristics of grouts. Some only mention them in passing or by implication, but many provide a list of the properties and characteristics of particular relevance. Most do not distinguish between working properties and performance characteristics, but the distinction is a useful one. Those that do make this distinction are usually following mortar studies [10]. For example, Penelis et al. note that 'the choice of a suitable grout for repairing old structures is not only dependent on the properties of set grout but on those of fresh grout which determine how effective it will be in situ', which also applies to the stabilization of architectural surfaces [40]. Working properties are defined as properties of the material in the state in which it is applied, measuring its practical ease of use, while the performance characteristics of the material relate to its long-term behavior in the wall. Criteria, such as the numerical specifications or recommendations used to evaluate working properties and performance characteristics obtained by a specific test method, are not provided in most of the studies.

Working properties

The working properties specified depend upon the context of the study. While there is general agreement on the desirable working properties of injection grouts for the conservation of architectural surfaces, it is rare to find suggested criteria used for the evaluation of working properties of grouts in the literature. Exceptions to this are Peroni et al. [28], who specify a maximum setting time of three days, while explaining that times of up to ten days may be tolerated in certain circumstances, and Ferragni et al. who specify a maximum setting time of 48 hours in the Vicat test [1, 2]. Where other authors have not set their own criteria they have referred to these values [20]. The most common desired working properties discussed in the literature are given in Table 3. The authors believe that injectability and penetration are

Table 3 Working properties

Desired Working Properties	*References*
Good injectability	[6, 8, 9, 13, 20, 21, 35–37, 43, 48, 49, 52, 53, 67, 71]
Low viscosity/good fluidity	[6, 8, 13, 14, 20, 21, 34, 35, 37, 40, 43, 49, 52, 53, 56, 67, 72]
Good penetration	[8, 14, 34, 43, 48]
Sufficient tackiness	[13, 17, 21, 25]
Good water retention	[44, 53]
No sedimentation/bleeding/segregation of components	[8, 34–37, 40, 43, 44, 48, 52, 56]
Reasonable setting time (laboratory and site conditions will differ for lime-based grouts)	[1, 2, 6, 8, 9, 13, 19–21, 29, 37, 49, 67]
Ability to set in the absence of air	[1, 2, 9, 19–21]
Ability to set in a wet or dry environment	[1, 2, 19–21, 28, 45]
Low toxicity and minimal health and safety implications for user	[13, 21, 20]
Good workability	[6, 29, 44]

essential working properties, and that other critical properties include flow, segregation, and setting time. Injection grouts should be fluid enough to be injected. During injection, they should retain their fluidity with minimal separation and clogging. They should set in a reasonable time in both dry and wet conditions, with or without contact with air.

Performance characteristics

As with working properties, the performance characteristics specified depend on the context of the study. For example, a desirable performance characteristic for a grout used to re-adhere large plaster detachments may be a low density [48], whereas a material with a low level of porosity and capillarity has been thought to give maximum stability under extreme weather conditions [43].

The validity of performance criteria and laboratory testing procedures designed to measure them is discussed in the literature, and some hold that conservation materials often cannot meet ideal criteria, and raise the question of what should be considered as acceptable [67]. The fundamental performance characteristic mentioned in nearly every study is that the grout should be compatible with the original materials. Many studies go on to discuss specific performance characteristics in more detail. Some define standards for specific applications by testing the historic materials for which they seek compatibility. However, others note the shortcomings of testing historic materials when it is difficult to obtain sufficiently large samples for destructive testing, and call for more research into non-destructive testing methods [37, 40, 47, 49].

Table 4 includes a list of performance properties and suggested criteria. It is rare to find actual numerical values suggested for performance characteristics. The main exception is Ferragni et al. [1, 2] who specify a compressive strength of 3–8 MPa, extractable alkaline ion content below 8 milliequivalents per kg of mixture, and volume change upon setting below 4%.

The general consensus on injection grouts for architectural surfaces appears to be that the mechanical strength of the grout should be similar to that of the original materials [1, 2, 19, 25, 37, 47], or possibly slightly lower [13, 20, 21] so that the grout will fail before the original materials. The rate of strength development is not generally discussed, but Penelis et al. suggest that it should be slow [40]. While some authors focus on the mechanical strength of grouts, others note that the bond strength is particularly important since the grout is required to bond cracked and exfoliated surfaces, and conclude that it is especially desirable for the adhesive properties to be similar to those of the original materials. However, only a few researchers measured bond strength [6, 20, 21, 37, 43, 49], while others stated it only as a theoretical criterion [27, 45, 48, 49].

The assumption made by some [6, 12, 13, 20, 21, 37, 48] that the ideal grout combines high water vapor permeability with low transportation of liquid water is challenged by Maryniak-Piaszczynski et al. [22]. They state that while materials with these properties may initially give good results, problems will arise if there is liquid water containing soluble salts present within the building structure. The water will diffuse through the areas of repair material in vapor form but the salts will crystallize in the parent material causing damage.

Methods for evaluating grouts

This section summarizes the most commonly used laboratory methods for the evaluation of injection grouts. In general, testing of the properties of grouts in the laboratory takes place before their practical use in situ [1, 2, 6, 13, 37, 49, 56, 72]. There are no standard tests specifically for injection grouts, and most test methods have been developed for other materials such as mortars, concrete, etc. This causes difficulties at every step of testing because the properties of injection grouts are quite different from the properties of materials for which these tests were designed. For example, if standard sized specimens for mechanical strength testing of mortar are used for injection grouts, the possibility of obtaining a sound, uncracked specimen is considerably reduced since injection grouts generally shrink more than mortars on drying. Furthermore, standard test procedures developed

Table 4 Performance characteristics

Desired Performance Properties	*References*	*Comments*
Minimal shrinkage	[1, 2, 6, 19–21, 25, 35, 37, 44, 45, 49, 52, 56, 67, 71]	
Low content of soluble salts/minimal potential for formation of salts	[6, 13, 19–21, 24, 26, 35, 37, 45]	No specific salts or ions.
	[1, 2, 25, 46, 56]	No sodium and potassium ions.
	[1, 2]	Low content of soluble calcium ions to avoid incrustations of soluble $CaCO_3$.
	[48]	Should contain no soluble salts.
Compressive strength	[1, 2, 19, 25, 37, 47, 52]	Similar to original materials.
	[13, 20, 21, 43, 52]	Similar to or less than original materials.
	[56]	Should be weaker than mosaic tessarae.
	[34]	Acceptable strength development in 90 days.
Young's modulus, modulus of elasticity	[6, 37, 48]	Similar to original materials.
	[43]	Similar to or less than original.
Adequate flexural strength or the modulus of rupture	[6, 47]	Similar to original materials.
	[13, 20, 21]	Reasonable flexibility.
Good adhesive strength/good shear strength	[6, 20, 21, 26, 37, 43, 45, 46, 48, 49, 52, 53, 67, 71]	
Thermal properties/coefficient of thermal expansion	[13, 20, 21]	Similar to original materials.
Coefficient of hygral expansion	[13, 20, 21]	Similar to original materials.
Good durability/resistance to sulfates/resistance to freeze-thaw	[12, 13, 20, 21, 46]	
Adequate porosity	[12, 13, 20, 21, 44, 47, 67]	Similar to original materials.
	[1, 2, 25]	Sufficient porosity to allow water evaporation.
	[43]	Porosity should be low.
	[56]	Relatively high porosity.
Good water vapor permeability/water vapor transmission rate	[6, 12, 13, 20, 21, 37, 48]	Similar to original materials.
	[43, 49]	Higher than original materials.
Low capillarity/water absorption	[43]	
Sufficient water resistance	[1, 2, 6, 25, 45]	Should be hydrophilic.
Good chemical stability	[13, 20, 21, 45, 49, 53]	
Good physical stability	[13, 20, 21, 48, 56]	
No promotion of microbiological growth	[13, 20, 21, 56]	
Low release of organic substances	[37]	
Low density	[6, 49, 48]	

for industry may be very costly or time-consuming [39].

Under these circumstances, it is not surprising that many different test procedures are encountered in the literature. National and international standard testing procedures are used, although these are frequently modified either to make them more suitable for testing grouts, or due to experimental constraints. Some test procedures for injection grouts have been developed specifically within the conservation field. These have been widely used following their publication [1, 2, 25, 49], although again with individual modifications.

Standards and widely accepted recommendations referred to in the literature include European Norms (EN), British Standards (BS), American standards (ASTM), Canadian standards (CSA), Italian standards (UNI and DM), German standards (DIN), French standards (CSTB), Dutch standards (NEN), Turkish standards (TS), NORMAL standards, RILEM recommendations, Building Research Establishment standards (BRE) and International Society for Rock Mechanics (ISRM) suggested procedures.

Examination and characterization of raw materials

Generally, information from manufacturers on the composition of the raw materials in the injection grouts is relied on, rather than undertaking any examination and characterization [11]. When they are examined, characterization includes determination of the particle size distribution of filler (sand and brick dust) and lime by sieving following ASTM C144 and C136 [1, 20, 21, 29, 49]; the apparent density and chemical composition of limes [73]; and the microscopic examination of pozzolans, because their reactivity is affected by particle size and morphology [13, 20]. Analysis of the extractable ion content of the raw materials has also been undertaken using ion chromatography and inductively coupled plasma atomic emission spectroscopy [20, 21], and by measuring the conductivity of aqueous extractions [17]. Thorough characterization is more likely to be undertaken if earthen materials are being used, as they are likely to be obtained on site rather than purchased with manufacturers' information.

Preparation and curing

Many studies do not describe the preparation of grouts, and even when this information is available, procedures vary widely. However, it is also known that the preparation procedure and curing conditions affect the final performance characteristics [74].

Standards have been developed for the preparation of cement mortars, including ASTM C305, BS 4551 and EN 196–1, but they are unsuitable for lime- or hydraulic lime-based grouts. It is suggested that more appropriate and relevant procedures are required following international standardization [29, 74] since studies show that the mixing method is an important factor affecting the performance of the grout [53, 55]. For example, an ultrasonic mixer [53, 55, 58, 60] facilitates better dispersion and wetting of the grout particles allowing the same penetration properties to be achieved with less water and superplasticizer. However, it is suggested that this method of mixing might induce an increase in internal microcracking. Both the stirring method and the mixing time have a vital influence on the consistency [6]. The longer the grout is mixed at high speed, the better the injectability and stability [6, 36]. Generally, the mixing has been done in the laboratory either by hand or using a commercial blender [1, 2, 25, 49].

Dry sieving of lime and/or filler such as sand and brick dust is often carried out to reduce particle size to within a 75–300 μm range in order to avoid clogging the needle when injecting the grout [1, 2, 20, 21, 29, 49]. Some studies recommend adding a pre-determined amount of water to the mix [6, 56, 67], or adding sufficient water to achieve a desired flow or viscosity [49]. The latter approach may result in the preparation of grouts with varying water content affecting not only their working properties but also their performance characteristics.

The curing period and environment will have a profound effect on the final performance characteristics of grouts. The authors find the lack of information in some studies, and the differing approaches of others, a cause for concern. Although the most commonly used curing period is cited as 28 days [2, 20, 21, 45, 49], it can vary greatly from as little as 72 hours [73] up to 120 days [10]. The curing environment can range from a relative humidity of 100% [47] to a 'dry indoor atmosphere' [22].

Assessment of working properties

The working properties typically assessed in the literature include injectability, viscosity, flow, setting time, segregation of components, and other parameters. Table 5 shows the test methods for assessing working properties, most of which have been adapted from test protocols for mortars.

Grouts require different flow characteristics depending on the application; therefore it is not surprising that one of the most discussed properties is fluidity or flow. In the literature, it is generally measured by various types of flow cone [24], of which the Marsh cone is the most frequently mentioned method [1, 2, 8, 25, 35, 40, 54, 73]. Other common tests in use are the flow table and mini-cone (Abrams cone). These are based on the fact that the flow of grout stops when the shear stresses in the sample become smaller than the plastic yield stress. Therefore, the shape and the size of the spread at the stoppage are directly related to the plastic yield stress and the time needed to reach the final spread value depends on the plastic viscosity. In general, their use in testing injection grouts with high fluidity is problematic. As the standard flow table is very small, the test is conducted without dropping the table and the speed of the removal of the mini-cone directly affects the shape and the size of the spread leading to issues of reproducibility [75].

Critics of the available standard flow cone tests include Van Rickstal [76], who states that the correlation between flow time and viscosity measurements is not very good, due in part to the very short outlet of the standard equipment. The authors have found that using a smaller nozzle size may give more representative results. However, the thixotropic behavior of some grouts remains an issue. It is suggested that equipment with a longer outlet would create a laminar flow and result in a better correlation between flow time and viscosity. Other researchers conclude that flow time value is not meaningful from the rheological point of view when the viscosity of the fluid is too low and the flow is not laminar [77]. Recently, viscosity and yield stress measurements of the grout with a rheometer have become increasingly popular [9, 35, 38, 54–56]. Over the past decade, studies on the dispersion and flow behavior of cementitious binders have advanced [75–77], and it is believed that this body of information will be beneficial to the development of injection grouts in conservation.

Assessment of performance characteristics

Most standard tests for performance characteristics require the preparation of standard-sized samples in non-porous containers. It is noted repeatedly that this is not representative of real conditions, and that samples prepared in contact with a porous material, to provide some suction effect, have higher strength and durability than those prepared in non-porous containers [20, 21, 47]. Therefore, samples are often prepared on porous surfaces such as brick, stone, and mortar, if only for qualitative or semi-quantitative measurements [13, 20, 21]. The standard sample sizes are generally determined for mortar, which experiences less shrinkage than grouts, and this may cause sample quality issues. Fontaine et al. note another problem with some standard tests that require samples to be removed from their moulds earlier than is desirable for lime-based materials [39].

Table 5 Working properties and related test methods

Properties	*Test Method*	*References*	*Comments*
Injectability	EN-NF 1771	[8, 25, 35, 36, 43]	Grout forced through a column filled with sand.
Penetration		[53]	Assesses penetration through the matrix of cracks of crushed bricks.
	Inclined plate test	[72]	Penetration of the grout along a simulated crack of decreasing width is measured.
	Penetrability meter test	[54]	The volume of the grout passing through different size filters under pressure until the filter is blocked is recorded.
Fluidity/flow	ASTM C939; ASTM C230; ASTM C937; ASTM D4016 and CRD C-78	[1, 2, 8, 25, 35, 40, 54, 73, 77]	Marsh cone.
		[76]	Afnor cup.
	DIN 4227, part 5	[34, 76]	Dropping ball and measuring the sinking time.
	ASTM C780	[39]	Flow table.
	DIN 18555	[34, 40]	Flow table.
		[13, 20, 21]	Simple qualitative assessments.
Segregation/bleeding/separation	ASTM C940C	[8, 35, 40, 72]	Observations of the grout mixture in a graduated cylinder.
		[35, 76]	Measures the buoyancy force to determine the stability of a grout.
		[38]	Gamma-densitometer bench containing a gamma-ray source moving along a column filled with fresh grout. Calibration is necessary to obtain density measurements.
Water retention capacity	DIN 18555, part 7	[46]	Percentage of water retained in the mix after it is absorbed by a filter paper.
Setting time		[10]	Penetrometer.
	ASTM C191; NF P18-362; UNI EN 196/3	[1, 2, 8, 28, 38]	Vicat needle.
		[9, 20, 21]	The time to reach constant mass.

Mechanical strength properties of grouts are the most commonly discussed performance characteristics in the literature. A large number of test methods are used to measure them, as given in Table 6. Different bond strength tests have been developed by researchers, whereby composite samples are prepared and the force required to pull them apart is measured [36], or assessed qualitatively [13, 25, 37, 49]. These tests often provide useful information about the relative strengths of the grout and the support material, showing which material is likely to fail first. However, bond strength tests have low reproducibility due to the high variation in the absorption capacity and surface texture of the porous substrate and the problems related to the experimental set-ups.

Soluble salt content is generally determined from the measurements of the extractable ion content including sodium (Na^+), potassium (K^+), calcium (Ca^{2+}), chloride (Cl^-), nitrate (NO_3^-), and sulfate (SO_4^{2-}). The methods used for analysis of the aqueous extractions are given in Table 6. Comparisons between studies of soluble salt content may be difficult if different extraction methods are used. For example, the grout sample can be crushed or whole, and the extraction can be performed at room or elevated temperature, with or without agitation. It has been argued that extractions at elevated temperatures and with agitation are inappropriate as they are not representative of natural conditions [78].

There is no mention in the literature of a standard for assessing the potential for the formation of salt efflorescence. Instead, some studies devise their own test method based on qualitative evaluation of the efflorescence area [1, 2, 24, 46]. Test methods used for determining the other performance characteristics are given in Table 6.

Less than 25% of the reviewed literature cites testing methods to assess performance characteristics. There is almost no cross-referencing for the discussion of results, due to the fact that few researchers are using the same test protocols. This once again shows the limitations and difficulty of comparing and discussing results because of the varying test methods used in the literature. The authors believe that establishing test methods specifically for injection grouts will address this problem.

In situ assessment

Less formally, the performance of grouts in situ is evaluated whenever they are used for fieldwork trials,

Table 6 Performance characteristics and related test methods

Properties	*Test Method*	*References*	*Comments*
Compressive strength	DIN 18555, part 3; ASTM C109/C109M; ASTM C942; ASTM C349; ASTM C780; EN 196-1 and CSA 179-94	[9, 34, 35]	
Young's modulus or the modulus of elasticity	Resonance method	[46]	
	Ultrasonic pulse velocity test	[34]	
	Modified versions of ASTM E447-92b and E111	[39]	
Flexural-tensile strength	ASTM C348-72; EN 196-1; ISRM suggested procedure	[13, 20, 21, 28, 34, 35]	
Split-tensile strength	ASTM C496-90	[43, 49]	
		[1, 2, 36, 56]	
Direct-tensile strength	Based on DIN 18555-5	[37]	
Shear strength	Casagrande shear set-up under 0.1, 0.3 and 1 MPa Normal stress	[35, 36]	
Bond strength	Pull-off test (Direct tensile test)	[6, 36]	
	Shear test; modified version of ASTM D905	[20, 21, 43, 53]	
	Developed method	[27]	Semi-quantitative measurement.
		[13, 25, 37, 49]	Qualitative evaluation.
Shrinkage		[56]	Measuring the length change of specimens (uni-directionally) and calculating the volume change using measured dimensions.
	ASTM C474	[1, 2, 49]	Calculating the volume change from weight measurements of the specimens in air and in kerosene.
Density		[13, 20, 21]	Calculated from mass and volume measurements.
Soluble salt content (extractable ion content)	Flame emission spectrometry	[56]	
	Inductively coupled plasma-atomic emission spectrometry	[20, 21]	
	Ion chromatography; NORMAL 13/83 [24]	[20, 21, 24, 67]	
	Absorption spectrophotometry	[28]	
Extent of carbonation	X-ray diffraction	[9]	
Effective porosity		[13, 20, 21]	Using the dry mass, saturated mass and volume of a grout.
	Thin sections	[13, 20, 21, 47]	
Total porosity	RILEM recommendation	[67]	
Pore size distribution	Mercury porosimeter; NORMAL F 4/80	[24, 25]	
Water vapor permeability or water vapor transmission	ASTM E96; RILEM 11-2; NORMAL 21/85 and CSTB	[7, 67]	Cup methods.
Capillary water absorption	RILEM 11-6; NORMAL 11/85	[10, 11]	
Absorption of water by total immersion and capacity of imbibition	NORMAL 7/81	[67]	

and it has also been noted that this should go hand in hand with laboratory testing [45, 79]. Although it is limited, assessment of the performance of various grouts in situ has also been undertaken [17, 58]. Reported examples include a visual examination, tapping to check for hollow areas [58], and pull tests to assess the adhesion of the grout to the substrate one year after treatment [22].

The need for in situ evaluations is emphasized in particular by those working on large-scale grouting operations in the context of building conservation. It is suggested that a combination of destructive (core-sampling) and non-destructive testing (radar, ultrasonic and electrical conductivity measurements) should be employed, and that there is a need for further development of non-destructive methods [53, 80].

Destructive testing, such as core-sampling and drilling resistance measurements [52], cannot usually be performed on wall paintings or mosaics. However, non-destructive methods have been employed to evaluate the effectiveness of treatments on wall paintings and plasters, and are being further developed.

Techniques for application of grouts

Many studies, particularly those describing the details of fieldwork, give advice on how grouts should be applied but none assess the techniques of application. In many cases voids are detected through visual evidence. However, where the presence of voids is merely suspected, studies recommend the simple non-destructive technique of knocking to identify hollow areas [27, 32]. More complex non-destructive techniques include radar, electrical conductivity measurements, examination using borescope fiber optics inserted into a cavity [32], ultrasonic measurements [6], and geo-electrical tomography [6, 52]. Zajadacz and Simon [6] explain that ultrasonic tomography requires three-dimensional access to the mock up, unlikely to be possible in situ.

Preparation typically begins with the clearing of loose debris and dust, usually by aspiration [1–3, 17, 19, 27]. Rinsing and pre-wetting with water, or water and alcohol, may then be undertaken to ensure good adhesion of the grout and to avoid rapid drying [1, 2, 19, 27, 31, 59, 82]. Pre-consolidation, depending on the condition of the original plaster, mosaic, and substrate, is sometimes advocated. Primal™ AC 33 (acrylic emulsion), diluted in water [1, 2, 19, 32] and polyvinyl butyral (acrylic resin), in a dilute solution in an organic solvent [31] have been used to pre-consolidate voids, as have Syton® W30 (a colloidal dispersion of silica in water) [83] and Gypstop® P (a dispersion of silica in water) [17]. Materials such as cotton wool or hessian have been suggested to seal holes through which the grout could leak [1, 2, 27]; fine cracks may be faced [31], and, if necessary, the edges of the area to be treated can be sealed with a lime mortar [17].

The technique utilized to introduce grout depends upon the size, accessibility and alignment of the void. If it can be accessed from the top, then a fairly liquid grout can be introduced at the upper edge and allowed to flow down inside the void. This is known as gravity grouting [27]. Instruments such as dental tools may be used to prod the mixture into place [1, 2, 27]. The grout should ideally be applied into an existing hole. If this is not possible, it should be injected through an area of loss, using as fine a needle as possible. Architectural conservators often recommend drilling a network of application holes [33, 80] to ensure that the void is completely filled, but this is clearly not viable for wall painting conservation. Many studies recommend applying the grout in stages to give each application time to lose water and begin to dry and harden [27, 46]. Supporting the wall painting or plaster while the grout sets may be advocated either to realign a deformed render, or to provide better contact and adhesion [1, 2, 13, 19–21, 32, 83]. There are recommendations for the surface to be covered after application [22, 48], and Asp [17] suggests repeated dousing of the area with water, which again may present problems for wall paintings. The technique used for the application of an injection grout will always be dependent on the context of the specific case.

Conclusions

In the majority of the reviewed literature, compatibility of the grout and the original material governs the selection of materials. Some studies define principles for their own specific application by testing the historic materials they seek compatibility with, and the authors consider this approach valuable. Although the type and level of compatibility investigated vary, in most cases researchers choose the principal binder of the grout to be similar to the original material. However, it is accepted that it is not necessarily appropriate to make the grout an exact match.

The differing expectations of researchers come across strongly in the studies on materials for grouting. Some studies are committed to the principles of using traditional materials and avoiding non-traditional additives, and are prepared to accept compromises in working properties to achieve these aims, while others embrace the use of commercial grouts and the use of additives. Specifications for desirable working properties and performance characteristics are only set in a few studies, representing less than a quarter of the literature, and show some agreement but by no means a final consensus.

A wide range of test methods for the preparation, characterization, and evaluation of grouts has been used. One reason for this is the lack of standard test methods developed specifically for injection grouts for the conservation of architectural surfaces. In most cases they were developed for other materials (e.g. mortars, epoxy resins, cement-based binders). As a result, diverse modifications of standard tests for evaluating injection grouts are common. Such modifications make it difficult to compare different studies and therefore have limited value when trying to compare results. Only general trends may cautiously be deduced, but specific conclusions may not. Furthermore, set criteria have limited use in situations where so many different test procedures are employed.

Internationally agreed protocols for the preparation and testing of injection grouts are still needed, as are criteria for desirable working properties and performance characteristics. In particular, the development of tests for durability and bond strength is required, as well as test methods for in situ evaluation.

In general, there is limited systematic research in the literature to guide conservators in evaluating different grouts in the laboratory and in the field. Published documentation by conservators working in the field describing grout preparation and conditions for curing would be a valuable addition to the literature. There is no comparative study on commonly used test methods for these grouts and for assessing application techniques. However, many studies include advice on how grouts should be applied, in particular those giving details of fieldwork. The importance of laboratory and field testing is clearly stated; it is suggested that the two types of investigation need to go hand in hand in order to obtain effective injection grouts.

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Authors

Beril Biçer-Şimşir graduated with a BS in Civil Engineering from the Middle East Technical University in Ankara, Turkey and a MS in Civil Engineering from the University of Illinois at Urbana-Champaign where she is continuing her research for a PhD specializing in the area of construction materials. She currently works as an assistant scientist at the Getty Conservation Institute. Her research interests include repair mortars and injection grouts. She is a member of ASTM Committee C07 on Lime and RILEM Technical Committee 203 on repair mortars for historical masonry.

Isobel Griffin studied wall painting conservation at the Courtauld Institute of Art, London and carried out research focussing on the use of pozzolans as additives for injection grouts, and the use of earth-based grouts in wall painting conservation. In 1999 she became the first Preventive Conservator for the National Museums of Scotland. She joined the National Trust for Scotland in 2001, first as Regional Conservator then as Head Conservator, and from 2006 onwards as the Special Projects Conservator. Isobel is currently studying for a PhD at the University of Edinburgh, investigating the deterioration and conservation of historic concrete structures.

Bénédicte Palazzo-Bertholon has a PhD in archaeology and is a specialist in ancient mortars, plasters and stuccoes. She has worked for several years on archaeometry of building materials and decorative elements from the European Middle Ages. She is an Associate at the Centre des Etudes Supérieures de Civilisation Médiévales (CESCM, CNRS-Université de Poitiers-France), and the Centre d'Etudes Médiévales d'Auxerre (CEM) where she studies the composition of medieval building materials, and the identification of mineral pigments used in wall paintings.

Leslie Rainer is a wall paintings conservator and senior project specialist at the Getty Conservation Institute where she manages the project 'Injection Grouts for the Conservation of Architectural Surfaces'. She received an Independent Master's Degree in the Conservation of Decorated Architectural Surfaces from Antioch University, a certificate from ICCROM in the Conservation of Mural Paintings, and was a recipient of the Rome Prize in Conservation and Historic Preservation.

Correspondence can be sent to:

Beril Biçer-Şimşir
Assistant Scientist
The Getty Conservation Institute
1200 Getty Center Drive, Suite 700
Los Angeles
CA 90049
USA
Email: bbicersimsir@getty.edu