



INJECTION GROUTS FOR STONE

Introduction to grouts

Lime-based hydraulic injection grouts are often used because of their compatibility with historic substrates in terms of their physical and chemical properties, mechanical strength, porosity, permeability.

Commercially produced grouts for conservation and custom/hand-mixed grouts formulated by conservators are most frequently of non-hydraulic lime with pozzolans or hydraulic limes, both mixed with sand/stone dust.

Non-hydraulic lime grouts require drying and access to carbon dioxide for carbonation to gain rigidity and are less frequently used as injection grouts because of their slow setting when used behind an architectural surface to fill voids or reattach a plaster, wall painting, or mosaic.

Grouting – Definition

An injection grout for the conservation of stone surfaces is a bulked fluid material that can be injected to fill cracks and voids and re-establish adhesion between delaminated layers upon setting.

Injection grouts are composed of one or more binders, aggregates, sometimes additives, and mixed with a fluid, typically water.

In many cases voids are detected through visual evidence. And where the presence of voids is suspected, the simple non-destructive technique of knocking on a surface will quickly identify hollow areas

Working practices

Preparation of the substrate prior to injecting the grout is critical to a successful grouting intervention.

Dirt and dust in the voids/cracks to be treated should be cleaned out with soft brushes, hand blowers, or battery vacuum cleaners with soft nozzles, and then followed by alcohol/water flushing.

Injecting Grout through a Syringe or Tube

The technique used to introduce grout into the void depends upon the size and accessibility.

Pre-wetting with alcohol or water is normal before application although care must be taken that no excess water is left as dilution or separation can occur of the grout's components. Excessive water can also potentially mobilise any salts in the stone and cause their crystallisation on drying.

If the void or crack can be accessed from the top, then a fairly liquid grout can be introduced at the upper edge usually by syringe and allowed to flow down inside the void, as in gravity grouting. Clay can be used to seal open sides to cracks and 50ml syringes with a 12 or 14 gauge cannula (needle) can be used to inject most grouts.

In the case of Juming Workshop samples they were pre-wetted with a 1:1 ethanol/water mix to reduce the potential for mobilising any salts.

For any large voids grouting needs to be in stages to allow for setting in between each injection. Sometimes area will need supporting or pressure applied to even out any distortion.

Following grouting, the surface should always be checked to ensure that no grout remains on the surface. Grout may flow and reappear on the surface in unexpected places.

During grouting, work should be monitored, and clean-up routines should be carried out immediately. Grout left on the surface is likely to set and will discolour a surface if it is not immediately cleaned off.

After grouting, colour-matched lime mortar fills are usually applied to cap the area above where the grout has been applied.

Working Properties and Performance Characteristics of Injection Grouts

To ensure the optimum performance of commercial and custom-made injection grouts, a wide range of working properties and performance characteristics needs to be evaluated including in its wet state, during setting, and after curing.

Commercially available grouts are often optimized for fundamental parameters, such as injectability, flow, and shrinkage. Both commercial and custom-made grouts should meet as many of the following performance criteria as possible:

- Grouts should be compatible with the original substrate and surface materials.
- Grouts should be fluid enough to be injected with no blocking up of the tube or needle. Wet grouts should not be too heavy to cause the collapse of the original material.
- Grouts should remain fluid long enough to fill the deepest part of the void/crack cavity before solidification, especially where repeated injections over several hours are necessary in order to completely fill voids.
- Grouts should have minimal separation of components. Grouts that are well formulated and properly proportioned should not visibly segregate or 'bleed'. Excessive segregation or bleeding of a grout will change its properties and cause clogging during injection.

- The volume shrinkage of a grout - from wet paste to hardened solid - should be minimal.
NB. The more water added for injectability, the greater the shrinkage.
- The concentration of soluble salts in the grout should be as low as possible.
- The mechanical strength (i.e., compressive, shear, or tensile strength) of the cured grout should be similar to, but less than, the original stone to avoid creating differential stresses.
- The capillary water absorption of the cured injection grout should be similar to that of the original material.
- The cured grout should allow water vapour in and out corresponding to the water vapour permeability of the original materials and should not create a vapour barrier.
- Grouts should provide an adequate bond, and the bond strength at the interfaces should be similar to but less than the strength of the original materials.



Terracotta head after grouting with Ledan TB1

Preparation of injection grouts

The preparation procedure for injection grouts is important since it affects their workability and their final performance characteristics.

Both the stirring method and the time of mixing have a vital influence on the consistency of the grout mixture.

In general, an injection grout mixed at high speed for a longer time produces a grout with better injectability and penetration and less separation of liquid and solid components.

For larger mixes a variable-speed power drill with a mixing attachment, a milkshake mixer, or a soup mixer can all be used. For small amounts vigorous hand-mixing should be done.

When a custom/hand-mixed grout is used, the grout ingredients should be measured accurately, and the mix proportions should be followed. Where absolute accuracy is required volume ratios can be converted into weight ratios to minimize measurement errors.

The amount of water used for injection grouts is an important factor that can modify flow, shrinkage, and bonding.

In order to obtain consistent grouts, the amount of water should be accurately measured at the time of mixing to avoid evaporation, and the water - grout ratio should be kept constant for each mixing.

It is often difficult to know the amount of water required until after several different ratios have been attempted, especially with handmade or custom-mixed grouts.

Once a ratio is established one example of a mixing procedure is:

1. Pour pre-measured water into the mixing bowl.
2. Add premixed dry ingredients within 30 seconds while mixing at low speed (mechanically if possible)
3. Increase mixing speed depending on the grout, and continue mixing for at least 4 minutes.
4. Following mixing, pass the fresh grout through a small sieve (eg. 300 micron to 1mm) to remove any clumps and to improve ease of injection.

Types of Grout:

There are many different grouts available ranging from commercial to handmade using non-hydraulic lime putty, hydraulic limes, ethyl silicates, acrylics etc

Ethyl silicate-based grouts have ethyl silicate binders (that is, silanes) with reportedly high silica gel deposits mixed with inert fillers - they include FUNCOSIL 500 STE and SYTON W30; non-hydraulic lime putty grouts are mixed with either inert fillers or inert and pozzolanic fillers (for faster setting); hydraulic grouts such as with NHL2 or NHL3.5 are mixed with aggregates including sand and stone dust with some additives for flow/fluidity improvement. Finally, commercial hydraulic grouts will have aggregates and often unspecified components such as in LEDAN TB1 or CalXnova.

Non hydraulic lime-based grouts

Non hydraulic lime grouts are atypically a mix of lime putty and fine sand. They need to carbonate slowly which can be problematic as the development of any strength and durability will also be slow. Many conservators will only use them by adding pozzolanic fillers such as brick dust or fired ceramic powders (eg. kaolin, trass) to chemically react in the absence of air. Other disadvantages of non hydraulic lime-based grouts may include high shrinkage and poor injectability.

Hydraulic lime based grouts

Hydraulic lime is compatible with original lime-based materials. Its advantage over non hydraulic lime is that it sets in the absence of air, and is particularly suitable for grouting internal voids.

The development of strength is quicker and durability is higher than for a non hydraulic lime 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 more extreme conditions.

However, they can sometimes be too strong and their performance can be variable. Other potential disadvantages of hydraulic lime-based grouts may include shrinkage and poor injectability.

Commercial variants of hydraulic limes such as Ledan TB1 are manufactured artificially and have additives such as small amounts of cement or pozzolanic material, or in the case of Ledan TB1 Silicate materials.

Acrylic grouts can include dispersions based on acrylic binders such as Primal B60A and Paraloid B72

In the workshop we wlooked at hydraulic lime based grouts using NHL 3.5, NHL2 and one commercial grout, Ledan TB1.

Fillers

Fillers act as bulking materials, thereby reducing shrinkage and controlling mechanical strength.

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.

A broad particle size distribution is therefore recommended as long as the particle size remains fine enough to be injected with around 75–300 μm is considered good for very fine cracks.

Sometimes grouts containing sand have a tendency to segregate, and are fairly heavy, therefore light-weight fillers have also been used.

Other fillers may be pozzolanic to effect a chemical set for a non hydraulic lime grouting - brick dust is a pozzolan commonly used for grouting wall paintings, plasters, and mosaics. Brick dust improves the fluidity of the grout, but if the content is high, it becomes thixotropic and resistant to flow.

Inert fillers include:

Sand

Marble dust

Quartz filler

Powdered limestone

Graphite dust

Glass microballoons - Increased penetration; no segregation; improved stability.

Ceramic microspheres - Light-weight filler.

Pumice stone - Light-weight filler.

Fumed silica (SiO_2) - Light-weight filler; good injectability and durability but severe shrinkage

Pozzolanic fillers include:

Brick dust

Diatomaceous earth

Trass

Crushed dolomite

Granite dust

Ceramic powder

Bentonite

Metakaolinite

Fly ash

Pulverized fuel ash (PFA)

Additives

Various materials are added to grout mixtures in limited amounts to modify specific properties.

For example, fluidizers or plasticizers are used to modify the flow properties, and accelerators and retarders to control setting.

Arguably some natural organic additives such as casein may deteriorate and promote biological attack so some conservators prefer to use synthetic organic additives as fluidizers/plasticizers.

Custom-mixed and commercial grouts

Custom-mixed grouts are defined as grouts that are formulated by the user and which may contain both non-proprietary and proprietary materials.

Examples include an early ICCROM grout containing hydraulic lime, brick dust, sodium gluconate (a sodium salt) and an acrylic emulsion (Primal B60A) developed for wall paintings and mosaics with lime-based supports.

Frank Matero at the Getty developed a water-based grout that contains hydraulic lime, ceramic microspheres, sand, and the acrylic Primal AC33 for the adhesion of lime plaster to earthen supports.

“Grout: 4 vol. Fine sieved Sand, 1 vol. Slaked Lime, 4 vol. microspheres, 4.5 vol. 10% Primal AC33, mixing properly, and applying by syringe.”

Handmade grouts include simple mixes using non-hydraulic or hydraulic limes, sand, stone dust, pozzolans etc with ratios such as 1:1, 2:1 and even 3:1 lime to aggregate, or as we used 1:2 lime and sand.

There can be drawbacks in the use of custom-mixed grouts such as potential inconsistencies in their components and methods of preparation by different practitioners.

Commercial grouts are generally found to be easy to prepare, consistent, and generally they have many good working properties. However, disadvantages include characteristics such as excessive strength and higher soluble salt content.

Many of the commercial grouts 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 with commercial products mentioned in the literature including the Ledan TB1 grout we tested.

Grout mixes for Juming tests

After clearing any dust or debris, it is normal to flush and pre-wet areas with a 1:1 ethanol/water mix

NHL2 mixes

Mixing hydraulic lime with sand <300 microns and with test mixes including glass microballoons added for better flow.

The samples we used in the workshop were based on a volume part of 20ml and used approximately 1 part liquid (20-25ml)

1:1 hydraulic lime and sand

1:1 hydraulic lime and sand/microballoons (1:1)

1:2 hydraulic lime and sand

1:2 hydraulic lime and sand/microballoons (1:1)

Water added using a measured container until fluid enough to inject (but not too wet).

A 50:50 water and ethanol mix could also be used to reduce the risk of mobilising salts if we were testing on the real Juming sculptures.

Ledan TB1 mixes

Ledan TB1 has hydraulic lime and <5% cement as binders and has quartz powder and slate dust as fillers. It also has some unknown antibacterial and plasticizer additives

Because the mobilisation and crystallization of soluble salts is one of the most important causes of stone deterioration, it is highly desirable that injection grouts should be free of these salts if possible.

Before use on any item of cultural heritage sample grouts would normally be tested using various mixes to find the most suitable for use. It is always the case that a balance needs to be made between the various characteristics offered by a grout.

For example, in the literature it is reported that Ledan TB1 has some salts and this is generally undesirable as conservation materials should not introduce soluble ions into porous materials.

However, Fig. 2 shows work at Hampton Court in London during grouting with Ledan TB1 on what are 500 year old and very fragile external terracotta sculptures. In tests Ledan TB1 displayed a high enough compressive strength and adhesion values and was able to penetrate very fine cracks without shrinkage, all of which were essential to hold together objects that were falling apart.

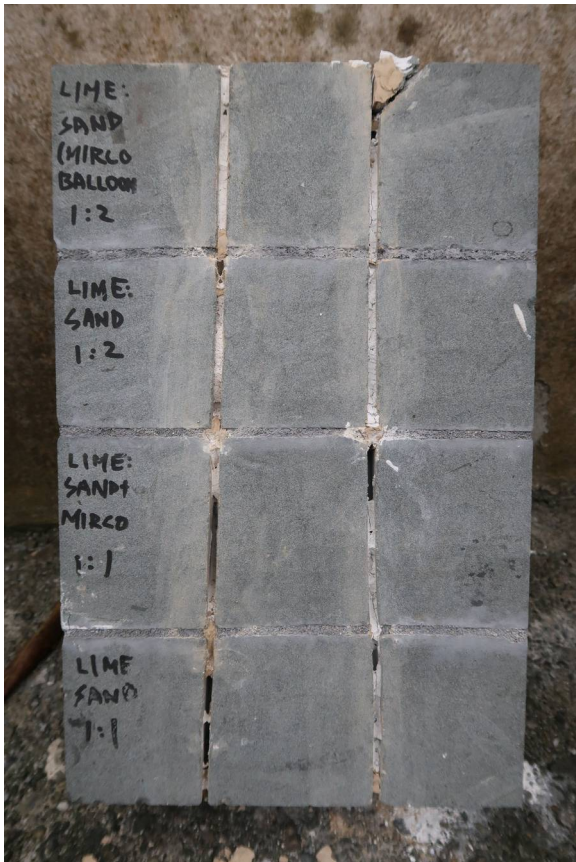
So with regard to the Juming sculptures, in any program of works a comparative salt analysis with the sandstone can be carried out by means of simple solution tests or with ion chromatography and if it is approx. equal or less to the salt content of the weathered Juming sculpture then it might be useful for fine crack grouting for the other characteristics it has.

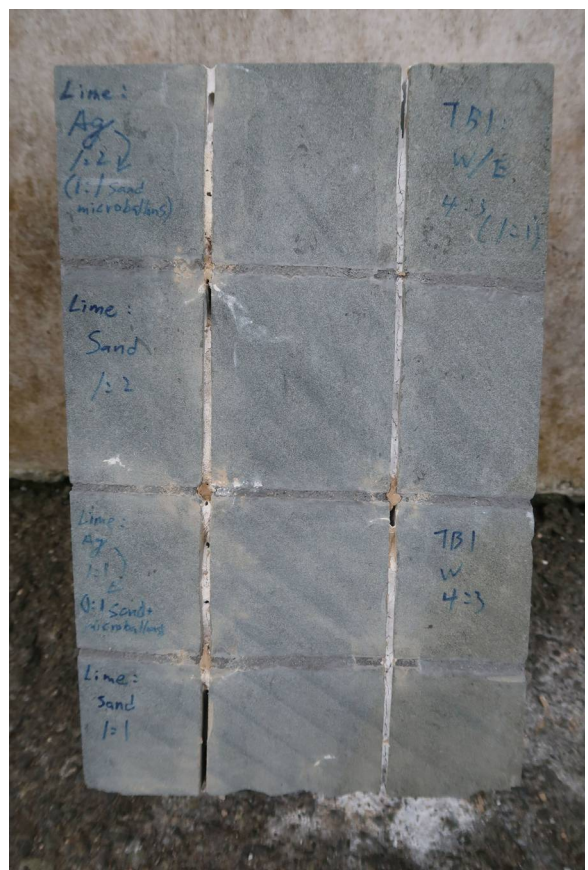
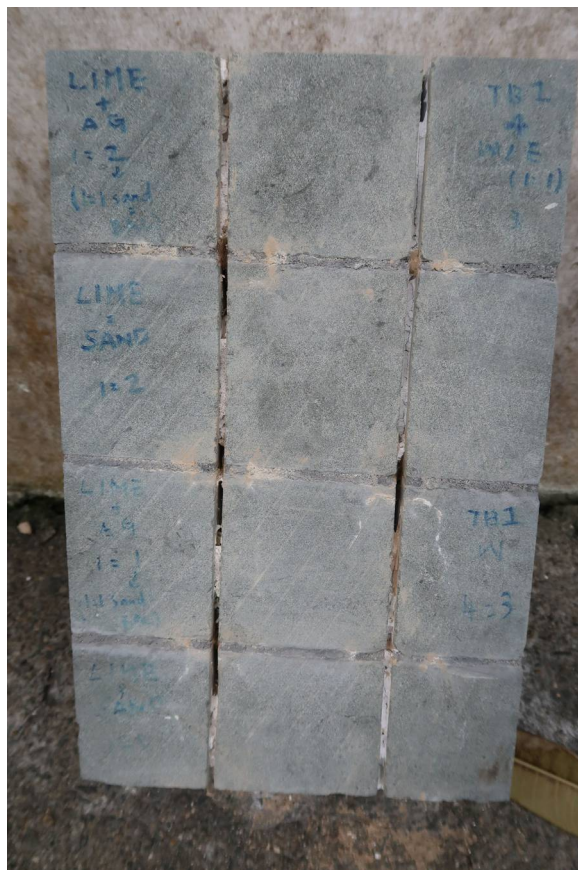
The samples we used in the workshop for Ledan TB1 were

100 parts Ledan TB1 in 75 parts deionised water (v/v)

100 parts Ledan TB1 in 75 parts ethanol/water (1:1) (v/v)

Results: The success of the applications varied between users and results were (expectedly) mixed:







The Ledan TB1 shows cracking on the upper surface probably because it was injected too quickly for the size of the 'void'. The lime and sand mixes are more successful and differences are probably due to user-variables such as: dampness of stone, injection speed, time in between injections, after care etc.

Dr Jonathan Kemp March/June 2018