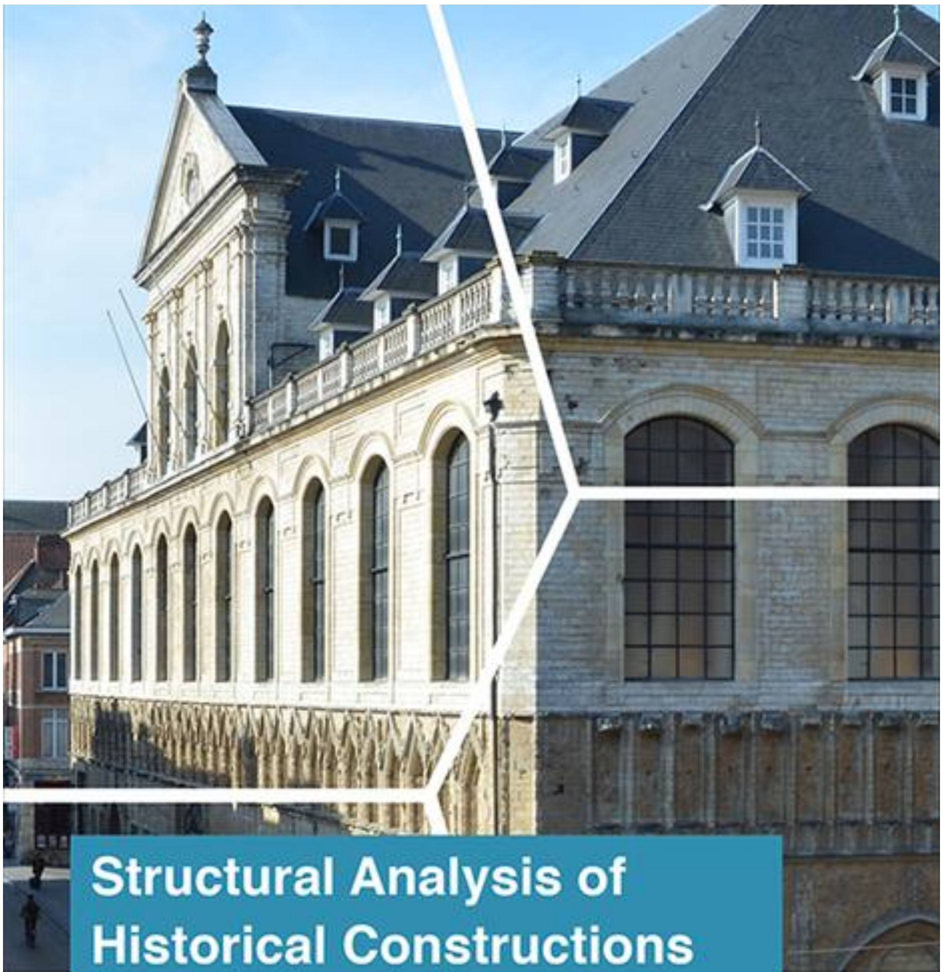

Structural Analysis of Historical Constructions

Anamnesis, diagnosis, therapy, controls

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Preliminary results for using micro-lime – clay soil grouts for plaster reattachment on earthen support

G. Schwantes & S. B. Dai
Architectural Conservation Laboratory, CAUP, Tongji University, Shanghai, China

ABSTRACT: This paper presents preliminary results of the ongoing research on grout formulations for the conservation of plaster on earthen support carried out at the Architectural Conservation Laboratory at Tongji University, Shanghai. Traditional building materials, clay and lime, with known physical and chemical compatibility are used. A risk of grout applications is often excessive water brought into the historical structure with the fresh grout. The presence of water may weaken historic clay plaster or dissolve and transport existing soluble salts. Micro-lime dispersed in propanol with a particle size range of 1-2 micrometer is therefore utilized in the described tests. Observations during the testing of grout formulations using micro-lime mixed with clay soil appear to show promising results in workability and performance; however, the grout performance is sensitive to climatic conditions.

The grouts have been preliminary tested in the laboratory, on mock-ups and on-site tests. The evaluation of the grout performance tests is not yet completed. Future testing will include the development of the grout performance over longer time periods and in varying on-site climatic conditions.

1 INTRODUCTION

Building surfaces often carry valuable information about the history and provide physical evidence of architecture on traditional materials and techniques. In situ conservation of historical plaster is therefore seen as a crucial practice. Although the reattachment of detached plasters with injection grouts has proven to be a successful way to preserve historic architectural surfaces in situ, it is also one of the most challenging conservation treatments conservators face. Grouting is not reversible. The grout will become an integral part of the treated object and will, in the future, suffer deterioration together with the historical materials. The grout should therefore show maximal chemical and physical compatibility with the original structure. At the same time, the most important criteria for a successful grouting treatment is that the grout serves its core purpose: to stabilize and provide a good adhesion between substrate and grout as well as plaster and grout.

With the most important characteristic in mind, the compatibility of the grout with the original fabric, it seems evident to concentrate on grouts containing a soil fraction. However, pure mud grouts often cannot fulfil the required working properties and performance characteristics: the main problems are shrinkage, poor adhesion and sensitivity to liquid water. Mud grouts are therefore modified with other materials. Earth and lime are some of the oldest building materials used in China (and the world) and there are many different ways these materials are applied together in China's heritage buildings from monuments to vernacular architecture. The use of lime as a stabilizer of earth structures is an ancient tradition and is, therefore, admissible as a non-synthetic although artificial technique (Warren, 1999, p. 114).

2 BACKGROUND

During the past five years, the Tongji Architectural Conservation Laboratory has worked on several projects investigating the application of grouts based on clay mixed with natural hydraulic lime (NHL) both for crack filling in earthen architectural structures and for reattachment of delaminated plaster (Dai 2013; Dai et al. 2012/ 2014). Initial tests with micro lime (ML) were included in the test series and all grouts showed positive results. Microscopic investigation shows that the adhesion between the NHL-grout and the historical plaster as well as to the substrate is satisfying. The adhesion of the ML- grout, however, is even better. The grout is homogeneous and shows no separation. Micro fissures in the grout and between grout and old materials are minimal and it appears that they do not have an impact on the overall stabilization effect. The tested NHL grout, even though it has performed well in tests carried out in Jinggangshan, Jiangxi, contains water, which may risk damage to the historical clay plaster or sensitive decorative finishes (Fig. 1).

On the basis of these results, research on ML grouts was expanded as described in this paper.

Figure 1. Application of water based NHL grout for reattachment of clay plaster at Liu's Family Ancestor Hall, Jinggangshan, Jiangxi. Water from pre-wetting and grout is transporting to the surface. (Photo: Dai Shi Bing)

This research will explore how the material characteristics of the fractions and their interactions can be utilized in favour of the requirement to create successful grouting treatment. A special challenge for using clay grouts for consolidation of earthen structures is the high variability in the chemical and physical composition of earthen materials. This variability is especially apparent with the quantity and quality of different clay minerals and in particular the presence of expansive clays has a great impact on the behaviour of the clay towards water and humidity. Testing therefore includes the comparison between applications of the grouting treatment with different clay soils. A problem with mud grouts and clay plaster is that no chemical bonding between substrates and repair material is achieved. It is known that lime can, under certain conditions in the presence of water, stabilize clayey soil. Rapid physiochemical reactions between the lime and clay minerals produce immediate changes in soil plasticity and workability and long-term soil-lime pozzolanic reactions result in the formation of cementing agents, which increase strength and workability (Greaves 1996, p.5-6). The lime content of the grout and the micro-lime solution used as mineral consolidation for existing clay structures may improve adhesion between grout and support.

While research on grouts often produces a large quantity of data and information about the performance of the grout in the laboratory, often little information is shared about on-site application and performance. Recording of on-site workability and successful performance such as distribution in voids will be an important part of the future evaluation of the mock-up tests and on-site tests.

3 RAW MATERIALS

Traditional building materials, clay soil and lime, are used in this study, whose compatibility and long-term performance are known. No synthetic or organic additives are added to the formulations.

3.1 *Micro-lime*

In the area of stone and lime plaster consolidation, calcium hydroxide nano-particles have shown good results to stabilize weakened historic fabric (Adolfs 2007; Ziegenbalg et al. 2010). However due to the nano-size of the particles (50-250nm), the stability of such fine particles is very limited. Therefore, lime hydrate ($Ca\ (OH)_2$) with particles sizes of 1-2 micrometer dispersed in isopropanol, micro-lime (ML), is used in the presented research. The micro-lime has a better stability and another advantage is that it can be produced locally at Shanghai DS Building Materials Co., Ltd. Two different kinds of mixtures were produced for the tests: 500g/l used in the injection grout formulations and 10g/l used for preparation of the substrate (pre-wetting before the application of grout). The advantages attributed to alcoholic lime dispersions are: compatibility of the consolidant with the original building material, no limitation of penetration due to the particle size, and no mobilisation of soluble salts (Daehne and Herm 2013, p.1). As mentioned above, compatibility can also be expected in the case of using lime in combination with earthen architecture.

3.2 *Properties and processing of the clay soil portion*

Two different soils were used in the grout formulations. A clay soil dug in Shanghai (SHS) and a clay soil from Jinggangshan area in Jiangxi Province (JGSS) dug in vicinity of the site where field tests are carried out (see Chapter 5). The soil was processed in the laboratory for use in the injection grout by wet and dry sieving. For ease of injection and stability of the wet grout, and to avoid segregation and sinking of large particles, the soil was sieved to particle size <0,125mm. Soil samples analysed for their mineral composition show a small amount of expansive clays (see Table 1).

Table 1. Mineral composition of clay soil samples from Jinggangshan and Shanghai (analyzed at the Nanjing Institute of Geology and Mineral Resources, China Geological Survey).

Sample No.	A1	A2	C1	C2
Location	Jinggangshan		Shanghai	
Quartz	75-80	70-75	55-60	55-60
Feldspar	-	-	5-10	10
Illite	10-15	15-20	10-15	10-15
Kaolin or Dicktite	5	-	-	-
Montmorillonite	<5	-	10-15	<5
Kaolin-Montmorillonite ML*	-	5	-	-
Chlorite- Serpentine ML*	-	-	<10	10

* ML= Mixed Layer. All values in %.

Samples of the unprocessed (S02/S04) and processed (S01/S03) soil samples of both clay soils (S01/02 Shanghai, S03/04 Jinggangshan) were analyzed for the particle distribution of the small fraction (Fig. 2). This analysis shows that the Jinggangshan soil has a higher amount of fine fractions.

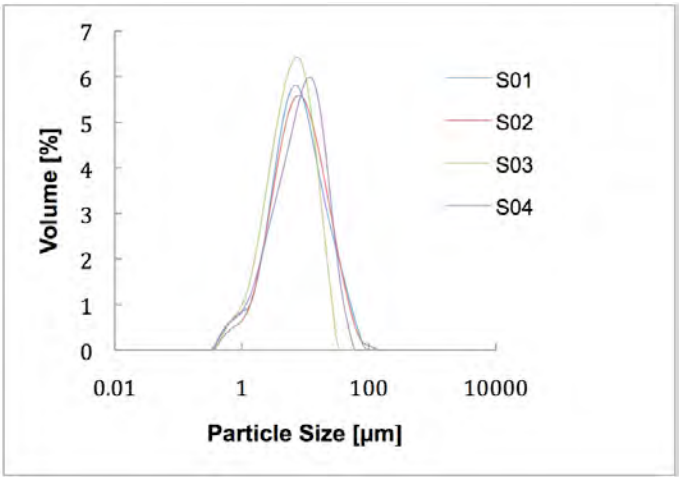

Figure 2. Particle size of small fractions of Shanghai (3/4) and Jinggangshan (1/2) (analyzed at the Department of Civil and Environmental Engineering, Strathclyde University, Glasgow UK).

3.3 *Additional Aggregate*

Limestone Powder ($CaCO_3$) (LSP) is used in the grout formulations as additional aggregate to reduce the shrinkage of the grout. The limestone powder is ground and sieved to particle size ≤ 0.125 mm in local laboratories.

Several formulations with fine quartz sand (particle size between 0,075 - 0,15mm) were tested, however the formulations did not show positive behaviour in the simple pre-tests set up for initial grout formulation determination. The formulations with sand showed higher shrinkage and comparatively poor flowability. Sand was therefore not used for further testing.

Some formulations with additions of 3-10% CaO were also tested with the idea that the expansion of the quicklime could work against the shrinkage of the grout. But all formulations cured into brittle material without cohesion and the expansion is not controllable. Apparently the expansion worked against stability and strength of the cured grout.

Due to the positive results with natural hydraulic lime (NHL) grouts in past research, several grout formulations were also tested adding a small percentage of NHL.

4 LABORATORY TESTS

Grouts were prepared in different admixtures of the mentioned components for testing of their workability and performance. A series of pre-tests included over 30 formulations and better performing mixtures were further tested until a final series of 8 grouts (4 formulations with the two different soils) was selected for the full test series. A control formulation without lime was included for comparison (see Table 2).

Table 2. Formulations of the grouts used for the workability and performance tests.

Grout No.	1+2	3+4	5+6	7+8	9+10
Soil %	50	50	50	55	50
LSP %	50	20	30	35	30
ML %	-	30	20	10	15
NHL%	-	-	-	-	5

In July 2014, a mock-up wall constructed from rammed earth (bottom) and adobe bricks (top) was built in an outdoor space of the laboratory. The wall is raised from the ground on a cement base and sheltered from direct rain. The rammed earth wall was built with local Shanghai soil mixed with sand and rammed into a wooden framework. The adobe bricks were made with the same clay soil mixed with a larger amount of sand, water and short cut straw, formed in wooden moulds and left to dry in the sun. They were then bricked using clay mortar.

4.1 *Workability of fresh grout*

All grout formulations were prepared by mixing the dry components first, then adding micro-lime and slowly adding additional isopropanol. The grouts were stirred for minimum 2 minutes with a laboratory mixer at 2000 rpm. It was observed that the grouts maintain a certain thixotropy in the beginning but at some point, sometimes only a matter of few milliliter of added isopropanol, reach an excellent flowability. The grouts are much smoother than mixtures of clay with water and fluidity is reached with a smaller volume of added liquid. The Jinggangshan soil mixtures generally required a higher amount of liquid volume to produce the desired flowability, which is most probably related to the higher amount of small clay fractions mentioned above.

Bleeding and Segregation

Following standard testing for grout formulations the bleeding and segregation of the grouts was planned to be examined. The test method is designed following *ASTM C 940—Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory.* Grout is placed in a graduated cylinder, and the change in total volume and the rate of accumulation of bleed water on the surface of the grout are measured over a period of time (Biçer-Simsir and Rainer 2011, p. 78). The volume of bleed water with respect to the total volume of grout is an indication of the separation of the liquid and solid phases. However, the test is designed for grouts containing water. Observations during the pre-testing show that bleeding and segregation does not seem to be an issue with the tested formulations. The grouts are stable for several hours if kept in a closed container. This gives enough time for application of the prepared grout.

Flowability Test

The flowability was tested by flow of the grouts over a porous surface using a board plastered with clay plaster. A syringe was filled with 4mm grout and poured on top of a clay plaster plate. The plate is positioned with an ankle of approximately 100 degrees and fine vertical channels scratched in the surface to cause the grout to flow vertically (for comparison purposes). All tested grouts were flowing with ease on the entire 30 cm length of the channels if just enough isopropanol was added to exceed thixotropic behavior. Grouts prepared with the soil from Jingganshan behaved slightly better (Fig. 3). The tests however show clearly that the pre-wetting of the surface is important for the adhesion. Pre-wetting was carried out with isopropanol containing 10% microlime. Pre-wetting reduces dust and loose particles on the surface and prevents rapid absorption of isopropanol into the substrate. The tested slightly more viscous grouts with fewer additional isopropanol separated from the substrate after curing, if the surface was not pre-wetted. All other grouts show good adhesion to the substrate.

From the results of this test, a decision was made to use grouts liquid enough to flow with ease on porous surfaces. Flowability was then used as a simple parameter during the preparation of the grouts. Isopropanol was added slowly to the mixture and approximately 4ml of the grouts were poured on blotting paper until the grout easily spread in a flat circle (instead of building small domes). This simple test is also easy to apply on site.

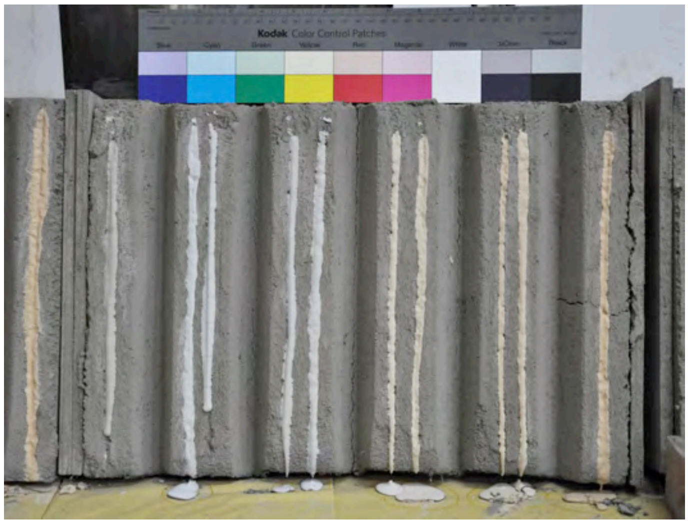

Figure 3. Flowability test on clay plaster substrate. (Photo: Gesa Schwantes)

4.2 *Performance of cured grout*

During the pre-testing clear differences in the hardness of the grouts was observed simply by comparing the effort needed to break the cured small grout plates produced in the flowability test. Mixtures with 10%, 20% and 30% ($Ca(OH)_2$) micro-lime particles showed noticeable differences in hardness of the cured grouts. Mixtures with more than 30% ($Ca(OH)_2$) were excluded from further testing. The differences in hardness were tested in more detail in the flexural strength tests and during the tests at the mock-up wall.

The most severe problem with the grout formulations is, as expected, the shrinkage. Especially as propanol evaporates faster than water, the shrinkage is expected to be more severe than in compared mixtures with addition of water. The shrinkage is mainly related to the amount of fluid in the fresh grout, the absorption of the substrate (fast reduction of the liquid portion), the type and amount of clay particles (relative to other mineral particles) and the sieve curve of the particles.

Shrinkage Test

An array of clay plaster cups was prepared on a wooden board to serve as porous substrate for testing of shrinkage. Two cups were filled per tested grout formulation. One of them was covered with plastic afterwards to avoid rapid evaporation and observe the influence of evaporation rate towards shrinkage (Fig. 4). In practical application, when a grout is injected in hollow spaces between plaster and substrate, the evaporation rate of the alcohol will also be limited.

The shrinkage is visible in fine cracks in all grout formulations. No clear difference was observed between the different soils (SHS/ JGSS). The mixtures with NHL appear to show slightly less shrinkage. All grouts appear to have good adhesion to the substrate.

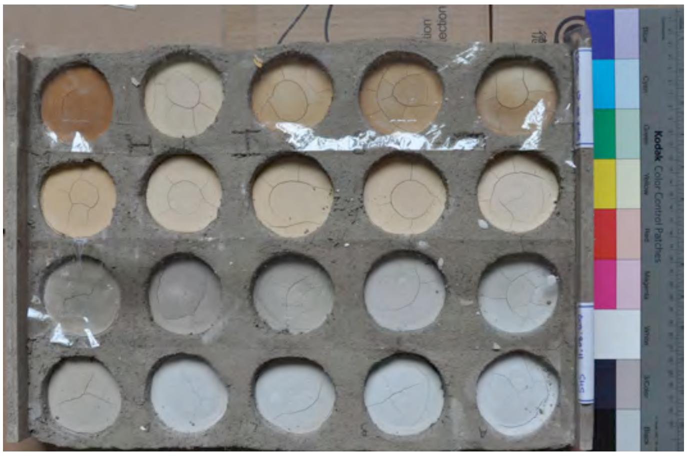

Figure 4. Shrinkage test on porous substrate (clay plaster) with and without covering with plastic to avoid rapid evaporation of the isopropanol. (Photo: Gesa Schwantes)

Flexural Strength Test

Over 200 rammed earth prisms were prepared using standard laboratory moulds. The prisms were dried in room temperature for two weeks. The dried prisms were broken in half in a three-point bending test machine. The 90 most even prisms with similar weight and similar break values in the three point bending test were used for the grout testing series: the prism halves were placed at approximately 2mm distance, the gap was sealed along the sides and then injected with grout. Nine prisms were prepared with the same grout formulation and will be tested in the in the same three point bending test machine after 30, 120 and 240 days of curing. It is expected that there will be measureable differences related to amount of lime and degree of carbonation between the different formulations. The test will be concluded in March 2016.

Mock-up Wall Test

In July 2015, small clay plaster tiles were prepared and attached to the surface of the mock-up wall by edging, leaving hollow spaces between the uneven wall surface and the back of the plaster tiles. The hollow spaces were filled with injection grouts of different formulations (Fig. 5). Several larger tiles with less sand but straw additive were prepared which showed fissures after drying but remained in one piece by the straw additive. The tiles were painted with red iron oxide pigment. The aim is to observe if micro-lime from the 10% solution used for pre-wetting of the substrate or micro-lime from the grout will migrate to the surface and cause discolouring of the surface.

The clay tiles will be detached in March 2016 and adhesion and distribution of the grouts will be studied. Crosscut samples will be prepared to study the contact zone between grout and substrate.

Figure 5. Mock-up wall tests. Different grout formulations were injected behind plaster tiles; after several month of curing adhesion and distribution in the void will be analysed/ recorded. (Photo: Gesa Schwantes)

5 SITE APPLICATION

Liu's Family Ancestral Hall located in Xiaqi county, Jinggangshan, Jiangxi Province is a 19th century traditional Chinese courtyard building combining rammed earth and adobe construction in the walls and plastered with mud plaster with a fine pure lime finish.

The Jinggangshan area is known as the birthplace of the Red Army and the cradle of the Chinese Revolution. The ancestor hall was used in the 1920s as a base for the Red Army and the presence of revolutionary slogans on the façade of the building are witness to this era. It is therefore the aim of the conservation plan to preserve the partly delaminated façade plaster to preserve the revolutionary slogans, which play an important role in the building's history and the history of the region (Fig. 6).

Figure 6. Red and grey revolutionary slogans on the west façade of Liu's Family Ancestral Hall, Xiaqi, Jinggangshan, Jiangxi. (Photo: Gesa Schwantes)

The walls of the building are built of rammed earth, and are plastered with clay plaster and finished with a lime whitewash. The plaster on the interior of the building is in very bad condition and does not hold valuable historic evidence (Fig. 7). A decision was made to replace the plaster (in the interior of the building) in a historical technique during the renovation of the building. In the meantime, these areas are used for testing the performance of the injection grouts (Fig. 8). This gives a rare opportunity for destructive testing under existing site conditions. The injected areas can be sampled extensively to investigate the adhesion between grout and substrate on cross sections under the microscope, and the plaster fragments can be removed to document the spread of the grout and behavior in the void.

Figure 7. Room on the west side of Liu's Family Ancestral Hall, with clay plaster fragments. (Photo: Gesa Schwantes)

5.1 *Site Tests*

Because of the unique situation to have a test area on site with very similar conditions to the actual area of conservation treatment, initial site tests were carried out before the laboratory tests are completed, but before the restoration works on site are continued.

Three grout formulations were selected for initial site tests in June 2015; two of the formulations which showed good performance in the laboratory pre-tests and one grout using a similar formulation, but a soil portion containing crushed and sieved aggregate of the original soil instead of limestone powder.

Three areas with detached plaster were selected and larger open areas were closed with lime-clay plaster with paper pulp fibres (the mixture shows ease of application, good adhesion in both wet and dry state and minimal cracking). The grout was prepared on site using a hand blender for mixing. The hollow areas were pre-wetted, with isopropanol containing ca. 10% micro-lime, via the edges and holes in the plaster. The amount of liquid injected varied and was estimated according to visual inspection of the area and the expected size of the treated void. The same holes were used for injection. In general, the grouts showed good on-site workability. The mixed grout was kept in a small bucket with a lid and did not show setting or separation of particles during the application time. All grouts appear to distribute well in the voids, however, the final result will be known after the destructive testing in summer 2016.

Figure 8. One of the 3 test areas after grouting of the delaminated plaster. Judging from percussion testing after curing the grout distributed well and re-established adhesion between plaster and substrate (Photo: Gesa Schwantes).

6 SUMMARY OF OBSERVATIONS/PRELIMINARY RESULTS

During the pre-testing of over 30 grout formulations, the following results were observed:

- The addition of different amounts of ($Ca(OH)_2$) micro-lime particles showed noticeable differences in hardness of the cured grouts.
- All grouts show good flowability in the laboratory test on porous substrate and the adhesion to the substrate, pre-wetted with 10% solution of micro-lime in isopropanol, appears to be good as well.
- All grouts show shrinkage but still maintain good adhesion to the substrate.
- The grouts show good workability for site application. If kept in a container with lid, the prepared grout was stable for over 4 hours and showed minimum to no setting of larger particles and no bleeding. The grouts show ease of flow and apparently good distribution in the voids (until now only judged by leaking of grout far from the injection area).

6.1 *Outlook*

It has been somewhat surprising that the water free grout formulations show positive properties, although water usually greatly influences the reactions in clay and of clay-lime mixtures. If the performance of water free grouts for reattachment of plaster can be proven to be successful, this would be a great advantage as water brought into historical structures during grouting treatments always bares risks.

Further evaluation of the grout performance will bring interesting conclusions on the application of alcohol based clay-lime grouts. Tests will also be carried out to investigate if micro-lime particles penetrating into the historic earthen structure can enhance the stability of the historic structure and improve the adhesion between plaster, grout and substrate. Microscopic investigations of the contact zone between grout and substrate and grout and plaster will be carried out on samples from the test site and the mock-up wall.

The reactions between the micro-lime and clay particles will also be investigated in order to determine if the stabilisation effect of the lime portion to the tested grouts is related to pozzolan reactions or a result of changes in grading of the fine fraction. Current tests were carried out in Shanghai and Jiangxi province, where the climate is relatively humid. Further research shall be completed in dry climate.

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REFERENCES

Adolfs, N. C. 2007. Die Anwendung von Calciumhydroxid-Sol als Festigungsmittel für historische Putze – erste Versuche und deren Überprüfung. Diplomarbeit. Institut für Restaurierungs- und Konservierungswissenschaften, Fakultät Kulturwissenschaften der Fachhochschule Köln.

Biçer-Simsir, B. & Rainer L. 2011. *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces. A Manual of Laboratory and Field Test Methods*. Los Angeles: The Getty Conservation Institute.

Daehne, A. & Herm C. 2013. Calcium hydroxide nanosols for the consolidation of porous building materials – results from EU-STONECORE. *Heritage Science* Vol. 1(11): 1-9.

Dai, S.B.; Wang J. H., Hu Y. & Zhang D.B. 2012. Materials and Practices for Surface Refitting of Cultural Heritage with lime-based Materials. Project Report. Tongji University, Shanghai CN-200092, PR China; China National Academy for Cultural Heritage, Beijing, Shanghai DS Building Materials Co Ltd., Shanghai.

Dai, S.B. 2013. Building limes for cultural heritage conservation in China. *Heritage Science*, Vol. 1:25.

Dai, S. B.; Fang X. N.; Han, J. & Hu Z. Y. 2014. Preliminary research on lime-based injection grouts for the conservation of earthen architectural heritage and ruins. In: Dai S.B., Lu D. & Zhang P. (Ed). *Architectural Conservation and Technology*, Tongji University Press, Chapter 1.13.

Greaves, H. M. 1996. An introduction to Lime Stabilisation. p. 5-12. In: *Lime Stabilisation*, ed.: C.D.F. Rogers, S. Glendinning and N. Dixon. London: Thomas Telford Publishing.

Warren, J. 1999. *Conservation of Earth Structures*. Oxford: Butterworth- Heinemann.

Ziegenbalg, G.; Bruemmer, K. & Pianski J. 2010. Nano-Lime – a New Material for the Consolidation and Conservation of Historic Materials”. Paper presented at the *2nd Historic Mortars Conference HMC2010 and RILEM TC 203-RHM Final Workshop*, 22-24 September 2010, Prague Czech Republic.