The Design and Evaluation of Injection Grouts for the Reattachment of Historic Plaster in St Mary's Church, Pöide, Estonia

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THE DESIGN AND EVALUATION OF INJECTION GROUTS FOR THE REATTACHMENT OF HISTORIC PLASTER IN ST MARY'S CHURCH, PÖIDE, ESTONIA

The research was inspired by the 13th century masonry building, the St. Mary's Church in Pöide, Estonia, where the large areas of ancient interior plaster are detached and loose, and require repair. The aim was to analyze and evaluate commercial and custom-mixed grouts in order to design a grouting mixture for use with medium and large size voids, as well as to reduce the cost of materials for restoration works. During the experiments two commercial grouts, Ledan TA1 and Vapo Injekt, and also thirty-five custom grout mixtures were analyzed and evaluated. The research began by the assessment of the plaster samples from St. Mary's Church in order to set the reference data for finding compatible injection grouts. The final results of this research conclude that the three selected custom grouts are suitable and recommended for the injection grouting of medium and large size voids and cracks. They are compatible with the historic plaster of the St. Mary's Church, and can reduce the cost of materials for conservation works. Selected custom grout mixtures, developed in this study could be used as well on the other sites with similar conservation problems and the characteristics of the historic plaster. The original recipes have been tested on several sites, such as: the chapel of the Sacred Heart at the Church of the Virgin Mary of the Rosary in České Budějovice, Czech Republic; the Koeru Mary Magdalene Church in Koeru, Estonia, and the Church of Padise Abbey in Estonia.

Introduction

Injection grouting is a conservation method used to fill voids and cracks and to reattach delaminated layers of plaster, wallpaintings, and mosaics to architectural surfaces. Grout itself is most commonly composed of binder, aggregate, admixtures, and fluid. There are many specific requirements for the grouting mixture. Most importantly it has to be sufficiently fluid, compatible with the original surface material, be able to mature in environments with the low CO_2 supply, and have the lowest concentration of soluble salts possible.[1]

The aim of this study, which was part of my MA thesis[2], was to find suitable injection grout for the conservation of the historic interior plaster in St. Mary's Church, Pöide, taking into account the composition, and the material characteristics of the historic plasters. In addition the aim was to reduce the cost of conservation materials by designing custom-mixed grouts. The more commonly used commercial products can have a significantly higher cost, especially for large areas of loose plaster as occur at St. Mary's Church.

The article gives an overview of the experiments carried out in this study and brings out the major results of the research. To give a closer look to the research process, the choice of the materials, and the laboratory experiments are described in the first half of the paper. In the second half the evaluation system for the test results is presented, and to conclude, the major results of the study are discussed.

Study of the historic plaster

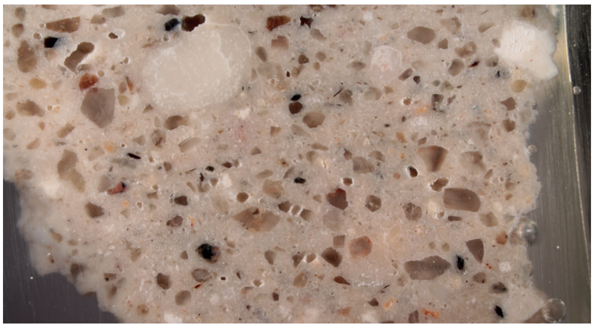
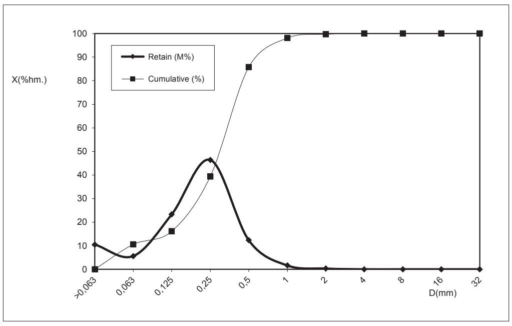
Beforehand to all the experiments with the injection grouts, the characteristics and the properties of the historic plaster samples from the St. Mary's Church were studied. It was done to be able to choose compatible materials for the custom-mixed injection grouts and to compare the properties with the ones of the commercial grouts. As well as to create an imitation mortar to prepare the necessary specimens for the laboratory testing. The wet silicate analysis and the grain size distribution test were used to determine the filler-binder ratio, and the size of the grains. In addition, cross sections were prepared for the microscopic examination using the optical microscope (Nikon, Optiphot 2-pol) and the scanning electron microscope (Tescan Mira 3 with EDS system Bruker). Ultrasonic pulse velocity measurement (device: Geotron-Elektronik, Rolf Kormholz, Pirna, D) was used to determine the L-wave velocity and the dynamic elastic modulus. Finally, the capillary water uptake was measured to evaluate porosity.

Already by the visual observation it was clear that the ancient plaster is lime-rich, relatively stiff, and consists of fine sand and big lumps of lime. Results of the analysis showed that the lime which was used to mix the mortar was with highly hydraulic properties, and the binder/

1 | Beril Biçer-Şimsir, Leslie H. Rainer, Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces: A Manual of Laboratory and Field Test Methods. Los Angeles: Getty Conservation Institute, 2013, pp. 2–3.

2 | Varje Õunapuu, The Design and Evaluation of Injection Grouts for the Reattachment of Historic Plaster in St Mary's Church, Pöide, Estonia. MA thesis. Tallinn: Estonian Academy of Arts Department of Cultural Heritage and Conservation, 2019. https://eka.entu.ee/public-thesis/entity-429186/ounapuu-varje-the-design-and-evaluation-of-injection-grouts-for-the-reattachment-of-historic-plaster-in-st-marys-church-poide, (accessed 06.01.2021).

Sample V-1-3: gothic plaster from the vaulting panel

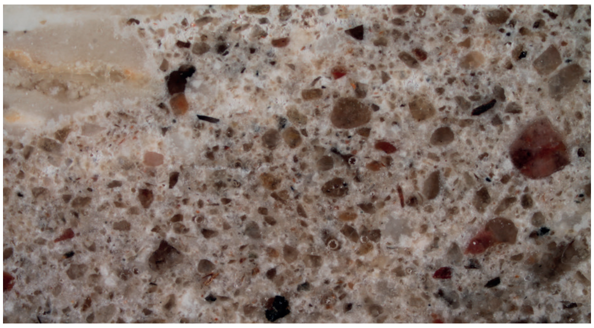
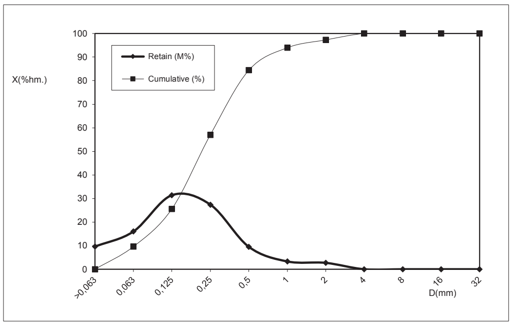
Cross section (magnification: x 20)	**Grain size distribution**
	

Water absorption coefficient (WAC): W = 31.2 $kg.m^{-2}.h^{-0,5}$

Wet silicate analysis	
Content of unsoluble part in HCl (M %)	35,62
Content of soluble part in HCl (% W.) (M %)	47,17
Ultrasonic pulse velocity (UPV) measurements	
VP (L-wave velocity) (km/s)	2,636
E-modulus (GPa)	7,476
Density (g/cm^3)	1,689

Tbl. 1 **Test results for the historic plaster sample from the vaulting panel.**

Sample W-1-1: gothic plaster from the lower part of the Northern wall

Cross section (magnification: x 20)	**Grain size distribution**
	

Water absorption coefficient (WAC): W = 7.1 $kg.m^{-2}.h^{-0,5}$

Wet silicate analysis	
Content of unsoluble part in HCl (M %)	64,38
Content of soluble part in HCl (% W.) (M %)	52,83
Ultrasonic pulse velocity (UPV) measurements	
VP (L-wave velocity) (km/s)	3,053
E-modulus (GPa)	8,233
Density (g/cm^3)	1,591

Tbl. 2 **Test results for the historic plaster sample from the Northern wall.**

aggregate weight ratio in the historic mortar was about 1:1. Compared to the plaster from the vaults, plaster from the wall contains a slightly higher amount of filler. Both analysed samples consisted mostly of fine and very fine quartz sand (grain size of 0–2 mm) and a small amount of granite.

The choice of materials

In order to establish a frame of reference within which to develop custom grouts, this research studied the properties of two commercially-available grouts which are: Ledan TA1 produced by Tecno Edile Toscana S.r.l., and Vapo Injekt produced by AQUA Obnova staveb s.r.o.

Ledan TA1 injection mortar was chosen because of its good working properties and common use in conservation of wall-paintings, especially for structural consolidation. It is chemically stable, with minor amount of water-soluble salts, finely ground powder with pure white colour and hydraulic properties.[3] Ledan products are also among the most well-known products used by conservators in Estonia, so I was familiar with its usefulness and characteristics. When filling medium to large size voids, a considerable amount of grout is required, as was discovered by researchers during the 2017 workshop.[4] In some cases, as much as 7 litres of grout were injected into only one larger void.[5] This can make the cost of the restoration works quite high. To lower the mechanical strength and the cost of Ledan products, the grout could be modified with the addition of an extra filler. In this study, limestone powder was added to Ledan TA1 in 1:1 weight ratio to evaluate the change in the working and mechanical properties of the injection mortar.

The second commercial grout Vapo Injekt, is developed and produced in the Czech Republic, and introduced in Estonia by Czech colleagues as a cheaper alternative to Ledan products. It is finely ground, yellowish white powder which is composed of calcium hydrate, calcined clay, limestone powders and organic additives.[6] Experiments with Vapo Injekt were done to better understand the material, and to compare its properties with Ledan TA1 and the custom injection grouts.

The choice of materials, for the custom mixtures, was inspired by the grout developed in the Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM), in the beginning of 1980's. The ICCROM grout consists of hydraulic lime, hydraulic filler, acrylic emulsion (Primal AC 33), sodium gluconate and water.[7] The more specific choices were done according to the mechanical properties of the historic plaster and the working properties of the commercial products.

For example, NHL5, which compressive strength after 12 months of curing is approximately 14,0 N/mm², was chosen to be the binder in order to reach the stiffness of the historic plaster.[8] Admixtures like sodium gluconate, Melment F10 (powder of a sulphonated polycondensation product based on melamine)[9], methyl hydroxyetyl cellulose,

3 | Ledan® TA 1 Leit 03, Technical Data Sheet, Kremer Pigmente, https://www.kremer-pigmente.com/media/pdf/31020e.pdf, (accessed 28.02.2018).

4 | "Matter and Meaning. Consolidation of Historic Plasters: Theoretical Issues, Resent Research and Conservation Methods", held in St. Mary's Church, Pöide, 2017. Final report can be found in: https://muinas.artun.ee/fotod/aruanded/konserveerimine/event_id-975, (accessed 08.01.2021).

5 | Final report of the international workshop *Matter and meaning. Consolidation of Historic Plasters: Theoretical Issues, Recent Research, and Conservation Methods*. Test are number 5. Tallinn, 2017, pp. 50–66.

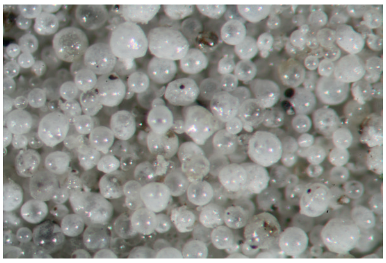
6 | Aqua® Vapo Injekt, Technical Data Sheet, https://www.aquabarta.cz/4_stahuj/tech_listy/VAPO_injekt_01.pdf, (accessed 28.02.2018).

7 | Daniela Ferragni, et. al., Injection Grouting of Mural Paintings and Mosaics. – Studies in Conservation 1984, vol. 29, no. sup. 1 January, p. 110.

8 | HYDRADUR® NHL5, Technical Data Sheet, https://cdn.shopify.com/s/files/1/1581/8535/files/hydradur_nhl_5_en.pdf?5927742592388195698, (accessed 28.02.2018).

9 | Melment® F 10, Technical Data Sheet, https://mychem.ir/uploads/tds/27513.pdf, (accessed 28.02.2018).

and acrylic emulsion were chosen to improve the fluidity and water retention of the grouts. Three variations of fillers were used as aggregates in the grouts: 1) limestone powder, 2) lightweight fillers like Poraver, Liaver and Fillite 160 W, and 3) the combination of limestone powder and lightweight fillers. The lightweight aggregates were analyze beforehand to make sure that the materials do not contain high amounts of water-soluble salts. The bulk content of water-soluble compounds, mainly soluble salts, was analyzed gravimetrically and by conductivity measurement.[10] Results showed that all the tested fillers are suitable for the injection grouting due to their relatively low water-soluble content.

Poraver 0,25–0,5 mm (mag. x 10)	**Liaver 0,25–0,5 mm** (mag. x 10)	**Fillite 160 W 5–180 µm** (mag. x 63)
		

Tbl. 3 **The optical microscope photographs of the three light-weight fillers used in the experiments.** Photos: courtesy of Romana Rajtárová

All together forty-one different grouting mixtures were tested in this study, and thirty-five of them were custom mixtures. After the first two experiments which were the funnel test and the filter paper test, the ten grouts were chosen for further testing. The rest of the grouts were eliminated due to poor working properties, or due to the choice of additives. For example, superplasticizer Melment F 10 was chosen instead of sodium gluconate, so the grouts with sodium gluconate were eliminated from the list. In this article the final ten mixtures are presented in the Tbl. 5 and 6. Two of the grouts are commercial, one is modified commercial, and the rest are the seven custom grouting mixtures.

Evaluation of the injection grouts

The following chapter is divided into three parts which describe the tests applied to the injection grouts. The first section covers the methods used to analyze the working properties of the grouts. The second section gives an overview of the methods used to analyze properties during the curing stage, and the third section describes the methods used to analyze the hardened grout samples.

The experiments have been mainly chosen by using two references: *A Manual of Laboratory and Field Test Methods*[11], published by the Getty Conservation Institute in 2013, and the article *Injection Grouting of Mural Paintings and Mosaics*[12] by the ICCROM team, published in the Studies in Conservation in 1984. Both of the resources cover

10 | Application Data Sheet of a conductivity meter by the Emerson Process Management, Theory and Application of Conductivity, January 2010, p.1.

11 | Beril Biçer-Şimsir, L. H. Rainer, Evaluation of Lime-Based, 2013.

12 | Daniela Ferragni, et. al., Injection Grouting, 1984, pp. 110–116.

information about materials used for grouts, including their working properties and performance characteristics, as well as methods used for evaluation. My research modifies some of the methods suggested in the manual and the article, adapting them to meet the specific needs of the study.

Working properties

The following tests were done to better understand the working properties of the injection grouts by comparing viscosity, and also water retention and release. First, the fluidity of all the grouting mixtures was measured by using a funnel test. The filter paper test was performed next to measure the water release/ retention. Finally the fluidity of the 10 selected injection grouts was examined on the plastered panel. The aim was to evaluate the influence of different additives to the workability of the injection grouts.

Funnel test

The funnel test[13] compared the fluidity of different grouting mixtures, and assessed the impact of the additives on the flow of the custom and modified grouts. This was done by timing the flow of each grout through the glass funnel. In order to select a grout which could be used as a reference for the custom, the fluidity and consistency of commercial grouts Ledan TA 1 and Vapo Injekt were examined first. 100 ml of grout was injected into the glass funnel with the tube diameter of 7 mm. The time of flow and/ or dripping were measured with the stopwatch. As a valuable information about the characteristics of the liquid grout, and for the comparison, both of the times were measured. Depending of the viscosity, some of the grouts were only dripping, only flowing, or flowing and dripping. At the end of each test, the funnel was photographed from the top to record how much grout had adhered to the surface of the glass. Tests were repeated four times with each grout, and the measurements were averaged. To evaluate the test results, marks from 0 to 4 were given to each grout to measure their performance level in the experiments. In this case the lowest marks were given to the grouts with the lowest viscosity, and the highest marks to the high viscosity grouts.

0	1	2	3	4
Very fluid	Fluid	Flowing in drips	Only dripping	Stuck inside the funnel

Tbl. 4 **Specification of the evaluation marks for the funnel test.**

Filter paper test

The aim was to observe and to compare the water release and retention properties of different grouts, and to measure the effects of the additives used in the formulas. 5 ml of grout was injected in the middle of filter paper with the diameter of 150 mm. The release of water out of the grout was documented by taking a photo at intervals of 1, 5, 10, 15, and 20 minutes. These photos were used to evaluate the amount of water released into the filter paper by measuring the wet area of filter paper, which was done with the image processing program.[14] Tests were simultaneously performed on two filter papers to see if there were differences in the test results. To evaluate the test results, the lowest marks were given to the grouts with the best water retention, observed as having the smallest measured area of water release.

Flow on plastered panel

This test is an adaptation of the test described in the Getty manual on pages 75–77.[15] According to the manual, the aim of this test was to compare the ability of different grouts to flow in vertical channels cut into the plaster. For this experiment Aeroc (now Bauroc) building blocks made of aerated concrete, in size of 250 x 300 x 50 mm, were covered with a 25 mm thick layer of the imitation mortar. Panels were matured in high relative humidity conditions (T = 15 ±1°C; RH = 91 ±1%) for one week, and then for approximately 8 weeks (55 days) in dryer conditions (T = 19 ±1°C; RH = 73 ±3%). After curing, 5 mm wide channels were cut in the plaster with a drywall hand saw. The space between the channels was about 15–20 mm. The depth of the channels was about 7–8 mm.

Pic. 1 **Plastered panel for the fluidity test.** Photo: Varje Õunapuu

13 | Daniela Ferragni, et. al., Injection Grouting, 1984, p. 114.

14 | ImageJ, An open platform for scientific image analysis, https://imagej.net/Welcome (accessed 01.11.2018).

15 | Beril Biçer-Şimsir, L. H. Rainer, Evaluation of Lime-Based, pp. 75–77.

To carry through the experiment the panel was placed vertically, and 5 ml of grout was injected into the top of the channel to measure the length of flow. Each grout was tested three times and the average of the results were calculated. Results were characterised by the length of flow, and were given an evaluation marks from 0 to 5 – the lowest marks were given to the grouts which flowed the longest distance, and therefore presented the best results.

Properties during curing

The experiment with the plaster cylinders was done to observe the volume shrinkage of the grouts, as well as the adhesion between the cylinder-shaped plaster specimen and the grouts. Plaster specimens were prepared out of the mortar imitating the properties of the historic plaster. The fresh mortar, consisting of NHL5 (250 g), sand (200 g), crushed marble (50 g), and water (200 g) was put in cylinder shaped detachable casts and let to cure for a few days, until the mortar was hardened enough to be removed from the casts. The inner surface of the specimen was coarsened before it was taken out of the cast, and left to cure for 28 days. The dimensions of the cylinder casts were 40 x 40 x 5 mm. To prepare the cylinders with higher volume, two specimens were glued on top of each other with the mixture of NHL5 and water. This created 80 mm high cylinders, where shrinkage would be more visible. Each grout was injected into two cylinders. After 21 days of curing, the specimens were cut in half to evaluate shrinkage. Specimens were documented by taking photos from the top, bottom, and after cutting in half. Test results were visually evaluated and each result was given a mark from 0 to 4. Lowest marks were given to the results with the least of visible shrinkage cracks. Adhesion between the cylinder and the grout was examined with the optical and scanning electron microscope.

Pic. 2 and 3 **Cylinders glued together with the mixture of NHL5 and water.** Photos: Varje Õunapuu

Pic. 4 and 5 **Casting the grouts inside the cylinders. The cured grout in the cylinder cut in halves.** Photos: Varje Õunapuu

Properties of hardened grout samples

The properties of the hardened grouts were examined to evaluate the compatibility of the commercial and custom grouts with the historic plaster. This group of experiments covers the measurements of capillary water absorption, mechanical properties like stiffness, density, compressive strength, and the adhesion test which measures the tensile strength of the selected grouts.

Capillary water absorption measurement

The aim of this test[16] was to evaluate the porosity of the injection grouts by measuring water absorption. The mass of the sample with dimensions of 15 x 15 x 40 mm was measured, and it was placed vertically on the petri dish with one side in the water. The mass gain of each sample was measured at 1-minute intervals. At the end of the test, the water absorption coefficient was calculated to compare the results with the historic plaster samples. The least absorbent grout according to the WAC value was evaluated with the mark 0 and the most absorbent grout was marked with 4.

The first measurements were done with the historic plaster samples and with the selected imitation mortar, to set the reference values for the injection grouts. The higher the coefficient number, the more absorbent/ porous is the material. The test results (see Tbl. 9) show that historic plaster from the lower part of the Northern wall is about four times less absorbent than the plaster from the vaulting panel, as well as the imitation mortar. This phenomena could be explained with the extreme moisture conditions which are present in St Mary's Church, especially on the lower parts of the walls. Possibly, over longer period of time it could condition the formation of the less porous layer of plaster.

Measurements done with commercial and custom grouts show that number 29 (custom grout) and 6 (modified Ledan TA1) (see the composition in Tbl. 5 and 6) have the highest coefficients, and the least absorbent, lower range grouts were number 5 (Vapo Injekt), 3 (Ledan TA1), 40 (custom mixed), and 36 (custom mixed). Vapo Injekt had the closest value to the historic plaster sample taken from the wall, and the modified Ledan grout had the closest value to the historic sample taken from the vault. The rest of the custom grouts (39, 37, 28 and 26) belong to the middle range of the test results. Experiments showed that the grouts containing only light-weight fillers, especially Poraver, were less absorbent than the grouts which contain limestone powder only.

16 | Beril Biçer-Şimsir, L. H. Rainer, Evaluation of Lime-Based, pp. 35–39.

Grout No.	Product name	Amount (g)	Extra filler	Amount (g)	Water (g)
3	Ledan TA 1	130	-	-	70
5	Vapo Injekt	150	-	-	60
6	Ledan TA 1	65	Lime-stone powder	65	70

Tbl. 5 **Formulas for the commercial grout mixtures which were studied in this research.**

Grout no.	Binder	(g)	Additives	(g)	Filler(s)	(g)	Water (g)
26	NHL5	45	Melment F10	0,18	Limestone powder	85	70,75
			5% Tylose MH 300	11,25			
28	NHL5	45	Melment F10	0,18	Limestone powder	85	62,75
			5% Tylose MH 300	11,25			
			5% K 9	8			
29	NHL5	45	Melment F10	0,18	Limestone powder	85	74
			5% K 9	8			
36	NHL5	45	Melment F10	0,18	Poraver 0,1–0,3 mm	40	50,75
			5% Tylose MH 300	11,25			
			5% K 9	8			
37	NHL5	45	Melment F10	0,18	Liaver 0,1–0,3 mm	36	50,75
			5% Tylose MH 300	11,25			
			5% K 9	8			
39	NHL5	45	Melment F10	0,18	Limestone powder	28,33	50,75
			5% Tylose MH 300	11,25	Poraver 0,1–0,3 mm	26,6	
			5% K 9	8			
40	NHL5	45	Melment F10	0,18	Limestone powder	28,33	50,75
			5% Tylose MH 300	11,25	Liaver 0,1–0,3 mm	23,74	
			5% K 9	8			

Tbl. 6 **Formulas for the seven selected custom grout mixtures (quantity of the grout according to this recipe is about 100ml). The final selection of grouts is marked.**

Ultrasonic pulse velocity (UPV) measurements

Ultrasonic pulse velocity method measures the time of travel of an ultrasonic pulse passing through the tested samples.[17] This method has been widely used for the condition assessment of concrete structures but also for cultural heritage objects.

During the testing the values of elastic modulus, L-wave velocity (VP), and density of the historic mortar, and also of the injection grouts, were measured and recorded. Results were used to compare the mechanical properties of the tested materials. The materials which had the highest values were the most stiff and dense, and were evaluated with the mark 0. Accordingly to the lowest values it was given the evaluation mark number 4.

17 | Ultrasonic Pulse Velocity Method, https://www.printfriendly.com/p/g/FxbtNZ (accessed 01.04.2019).

The device used for measurements was: Geotron UKS (Geotron-Elektronik, Rolf Krompholz, Pirna-Neundorf, D), according to the standard EN ISO 16823: 2014, frequency 250 kHz, UPG 250 kHz, UPE. Samples had been curing for 7 days in humid conditions (T = 15 ±1°C; RH = 91 ±1%) and 21 days in laboratory conditions (T = 19 ±1°C; RH = 73 ±3%) before the measurements.

Compressive strength

The aim was to measure the compressive strength values for the comparison of different grouting mixtures and their compatibility with the imitation mortar. Samples had matured for 7 days in humid conditions (T = 15 ±1°C; RH = 91 ±1%), and then 21 days in laboratory conditions (T = 19 ±1°C; RH = 73 ±3%) before the measurements. Matured samples were cut into dimensions of 35 x 31 x 25 mm. Measurements were done with the device FPZ100 (max. load 100 kN, crosshead speed 2,1–64 mm/min), according to the European standard recommended in *Methods of test for mortar for masonry – Part 11: Determination of flexural and compressive strength of hardened mortar* (EN 1015-11:1999). The results with the highest compressive strength values were evaluated with the mark 0 and the lowest with 4.

Adhesion test

The aim of the adhesion test was to measure the tensile strength of the selected injection grouts. Liquid grout was casted between the two specimens (40x40x20 mm) made from the imitation mortar. Cast samples matured in the humid conditions (T = 15 ±1°C; RH = 91 ±1%) for seven days and were then moved to the less humid conditions (T = 19 ±1°C; RH = 73 ±3%) for another 21 days. After 28 days solvent exchange drying method was applied to stop the hydration in the specimens. Samples were placed in a plastic container, soaked in isopropyl alcohol (C_3H_8O), and tightly sealed for two days. After two days, samples were removed from the solvent and left to dry in the laboratory ventilation hood until the solvent was evaporated. The time of evaporation varied depending on the size of the samples. Specimens were stored in airtight conditions until the measurements were carried out. The adhesion test was performed according to the standard ISO 4624:2016 or ČSN 73 2577. With the measurement device: FPZ100/1, VEB Thuringer Industriewerk, Rauenstein, loading with crosshead speed 2,1–64 mm/min. The test was applied only to the three selected grouts to control the adhesive capacity, and therefore evaluation marks were not given.

Pic. 6 **Prepared test specimen for the adhesion test. Cured injection grout between two pieces of imitation mortar.** Photo: Varje Õunapuu

Evaluation system and the final selection

To make conclusions of the test results it was necessary to create an evaluation system. In this study marks from 0 to 4 were given to each grout to measure their performance level in the experiments. The meaning of the given mark depended of the specific group of tests. For example the lowest score of marks in the tests evaluating working properties and the properties during curing was a good result. At the same time the lowest score in the tests measuring the properties of the hardened grouts could mean that the material is too stiff or dense – it is because the lowest score was given to the highest values. To give an overview of the formulas the commercial grouts are presented in Tbl. 5, and Tbl. 6 presents the seven selected custom grouts which were chosen for final evaluation.

The minimum sum of the given marks was 6 (grout with Ledan TA1 – no. 3) and the maximum was 33 (custom grout no. 29). Injection grouts with the lowest score were fluid, had good water retention, and were more stiff and dense than the rest of the grouts. The grout with the highest score has the opposite characteristics. Number 29 was evaluated with the highest mark in seven out of the nine experiments, which means that it had poor test results in workability and water retention. The grout was weak, and did not create strong bond with the imitation specimens in the shrinkage and adhesion tests.

Working properties				Properties during curing	Properties of hardened grouts					
Grout no.	Funnel test	Filter paper test	Flow on plaster panel	Shrinkage in cylinder	WAC	E-modulus	VP	Density	Compressive strength	**Total sum. of marks**
3	1	0	1	2	1	1	0	0	0	**6**
5	1	2	0	2	0	0	0	0	0	**5**
6	0	2	2	1	3	2	3	1	1	**15**
26	1	0	3	3	2	2	3	1	3	**18**
28	1	1	0	3	2	2	2	2	3	**16**
29	3	4	4	4	4	4	4	2	4	**33**
36	3	0	2	2	1	3	1	4	4	**20**
37	2	0	3	2	2	4	1	4	3	**21**
39	3	0	0	2	2	2	1	3	2	**15**
40	2	0	0	2	1	3	1	3	2	**14**

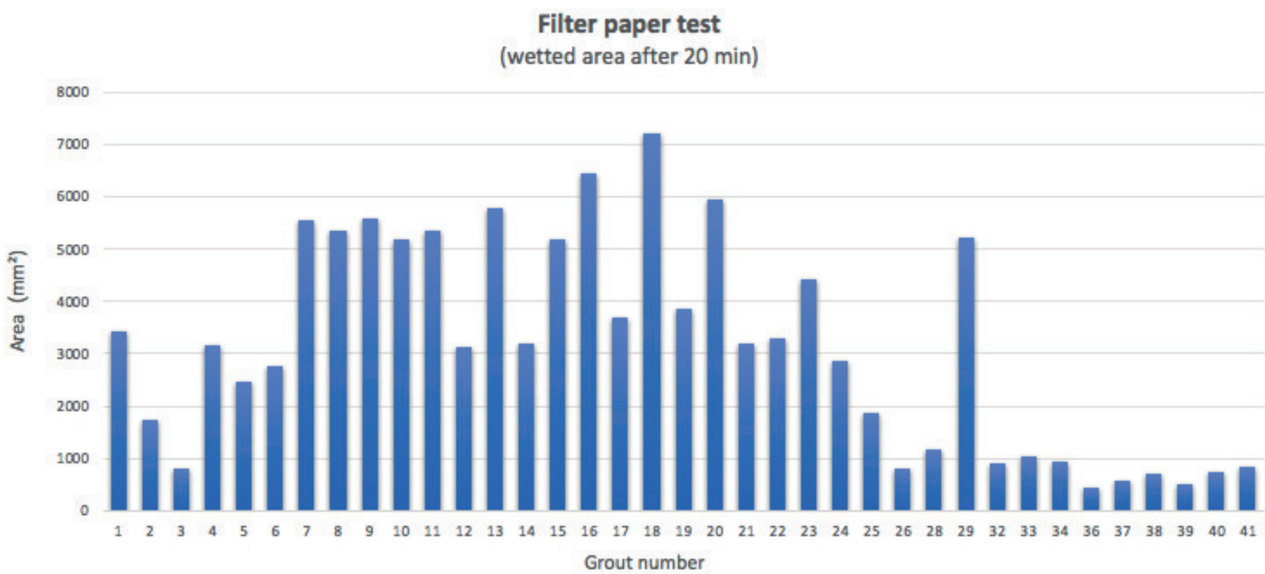
Tbl. 7 **Evaluation marks for the ten selected grouts. The results of the three final selected grouts (28, 39, 40) are marked.**

The final selection of the grouts was made from the custom grouts which scored in the middle range. These grouts had good workability and water retention, satisfactory results in the shrinkage test, were flexible, and had lower mechanical strength than the historic plaster samples.

The final three grouts, marked with numbers 28, 39 and 40, were consisting of the same amount of NHL5, organic additives and filler(s). The main difference of these mixtures was the type of the filler. Number 28 contains limestone powder, number 39 contains limestone powder and Poraver 0,1–0,3 in 1:1 weight ratio, and number 40 contains limestone powder and Liaver 0,1–0,3 in 1:1 weight ratio. Different fillers were used in order to test one grout which contains finer aggregate for re-adhering small (5–20 mm) and medium (20–40 mm) size voids, and one lightweight grout with the combined aggregate for use with large voids (≥ 40 mm). Two lightweight grouts were selected to test the workability of the two different expanded glass aggregates.

Results of the study

The major results of this research mainly describe the influence of the fillers and the additives which were used in the custom grouts. During the experiments with different additives it was noticed that the acrylic emulsion K9 had an unexpected influence on the working properties of the custom grouts. If it was the only additive in the grout the water release was almost the same as that of the grout which contained no additives (see Graph. 1, grouts no. 7 with no additives and 11 with only acrylic emulsion as additive). In combination with the fluidizers and methyl hydroxyethyl cellulose, K9 slightly increased the viscosity of the grout. The second additive, methyl hydroxyethyl cellulose was added to the custom grouts to increase water retention and tackiness, and the



Graph. 1 **Results of filter paper test. Comparison of the water release of grouting mixtures.**

Working properties				
Grout no.	Funnel test: time of flow and dripping in min.		Filter paper test mm²	Flow on plastered panel cm
	Flowing	Dripping		
3	01:14	00:03	795,6	26,7
5	02:06	00:47	2468,8	28,5
6	00:16	00:15	2773,7	25,2
26	03:04	00:25	788,3	22
28	02:57	01:18	1164,2	28,3
29	00:00	06:06	5216,5	10,7
36	08:45	00:00	432,3	24,5
37	04:06	00:08	577,7	21,8
39	00:00	10:57	517,3	28
40	07:00	00:00	745,1	28,5

Tbl. 8 **Test results of the funnel, filter paper and flow on plastered panel test. Results of the final chosen grouts are marked.**

Properties of hardened grouts						
Specimen name/ no.	WAC $kg.m^{-2}h^{-0,5}$	E-modulus GPa	VP (L-wave velocity) km/s	Density g/cm^3	Compressive strength MPa	Adhesion strength MPa
W-1-1 (wall)	7,5	8,233	3,05	1,591	nm	nm
V-1-3 (vault)	31,2	7,476	2,64	1,689	nm	nm
Imitation no 8	30,1	6,320	2,48	1,380	nm	nm
3	12,4	4,717	2,17	1,282	6,2	1,4*
5	7,3	5,760	2,29	1,372	5,4	nm
6	33,9	2,120	1,37	1,159	1,6	nm
26	24,8	1,914	1,38	1,163	1,1	nm
28	22,8	2,062	1,50	1,119	1,0	0,1
29	52,6	1,327	1,22	1,091	0,7	nm
36	15,5	1,614	1,64	0,680	0,5	nm
37	21,2	1,457	1,60	0,634	0,9	nm
39	18,1	1,803	1,57	0,825	1,3	0,5
40	15,5	1,633	1,53	0,802	1,4	≥0,2

* Value was taken from the Kremer Pigments Technical Data Sheet of Ledan TA1. nm – not measured

Tbl. 9 **Overview of the basic properties of the historic plaster samples (W-1-1 and V-1-1) hardened grouts. Results of the final chosen grouts are marked.**

experiments showed that it fulfilled this purpose. Besides that, increased water retention helps to slow down the drying process, and keeps the level of moisture which is necessary for the proper hydration of the injected grout. The amount of Tylose MH 300 was chosen according to the test results in the filter paper test. The aim was to reach similar characteristics as the commercial grout containing Ledan TA1 (grout number 3), and this was successful.

Soon, during the first workability tests it got clear that water retention plays an important role in the fluidity of the injection grouts. If the water retention is better, then the grout is more fluid in contact with absorbent materials like lime plaster. This was clearly visible with grout which was containing only fluidizer and acrylic emulsion (grout number 29) in the flow on plastered panel test. Due to poor water retention the grout flowed the least of all the tested grouts.

Tests with the modified Ledan product (grout number 6) gave valuable information mostly about the mechanical properties of the grout. Addition of the extra filler reduced the stiffness and compressive strength significantly. This could be evaluated as positive effect if the historic plaster has a lower compressive strength from the Ledan products. At the same time the grout was more absorbent and the water release in filter paper test was increased two times.

Test results were converted into the evaluation system, and three final injection grouts were chosen to carry out on the site testing (see Tbl. 7). The final selected custom grouts 28, 39, and 40 were all found to be suitable for the reattachment of the historic plaster in St. Mary's Church. Based on the *in situ* injection tests and the conservation works it is recommended to use the grout number 28 for small and medium size voids and cracks, and the lightweight grouts number 39 or 40, which difference is only in the filler product, for the medium and large voids. For the fine cracks and voids I recommend experimenting with replacing the filler in custom grouts to very fine or to use the commercial Ledan products.

In general can be concluded that custom grouts had lower compressive strength and were more flexible than historic plaster, and were able to create adhesive bonds similar to the Ledan products. Which means that the custom grouts are suitable for the reattachment of the historic plaster in the St. Mary's Church, Pöide, Estonia.

Suggestions for further research

The results presented in this study can hopefully contribute to a better understanding of commercial and custom injection grouts, and benefit the work of conservators active in the field of restoration and conservation. I believe that further research into other aspects of the topic could continue to develop our understanding. Some suggested topics for further study include:

- Clarifying the influence of the acrylic emulsion on the custom injection grouts.
- Possible methods for the reduction of the organic compounds in the custom grouts. For example, the use of the other types of methyl hydroxyethyl cellulose like Tylose MH 1000 P2 or Tylose Mh 30.000 YP4, which have a higher viscosity than Tylose MH 300 P2.
- Research of different lightweight aggregates available on the market and their suitability for injection grouting.
- Modification of the chosen custom grouts with the NHL2, and the evaluation of the mechanical properties to see if the custom grout could be compatible with the less stiff, historic aerial lime plaster.

Conclusion

The aim of this study was to evaluate custom injection grouts for conservation of the historic render in St. Mary's Church in Pöide, Estonia, and for the general improvement of the injection grouts used in the conservation field. This research looked for grouting materials which would be most suitable for filling the voids and re-adhering the masonry and plaster, not only in St. Mary's Church, but also in the conservation of other cultural heritage objects in similar condition.

The central work of this study was the design and evaluation of the grouting mixtures. The research evaluated the working properties, properties during curing and the properties of the hardened commercial and custom injection grouts.

The most important information from this study, which I hope will be of benefit to the reader and to the conservation field, in general, is as follows:

The binder NHL5 was selected for the grouts because it could match the stiffness of the historic plaster at St. Mary's Church. Limestone powder and expanded glass spheres, or a combination of both, were used as aggregates for the grout mixtures. In the beginning the focus was on workability. Experiments showed that to achieve better working properties, fluidizers like Melment F10 and admixtures like methyl hydroxyethyl cellulose are necessary. They ensure the fluidity of the grout when it is in contact with absorbent materials, like historic plaster. Acrylic emulsion K9 was added to the grout mixtures to improve water retention and to increase the tackiness. However due to its unexpected negative effect on the fluidity and water retention the purpose of the emulsion in the custom grout mixtures still needs further research.

The test results of the hardened grout properties showed that the mechanical strength of the commercial grouts is higher or equal to the strength of the historic plaster. The compressive strength and stiffness of Ledan TA1 could be reduced by modifying the grout with the addition of extra filler. The custom grouts showed lower compressive strength and more elasticity than the historic plaster, and reached a strong adhesive bond in the adhesion test. The custom grouts are compatible with the historic plaster, and can reduce the cost of the conservation works, especially in cases where there are large areas of detached plaster as occur in St. Mary's Church. However, commercial grout Ledan TA1 was still more useful when applied to fine cracks and small pieces of detached plaster.

Since the study was finished, selected grouts have been used to reattach the historic plaster in St. Mary's Church but as well on other heritage sites in Estonia and Czech Republic. Such as: the Chapel of the Sacred Heart at the Church of the Virgin Mary of the Rosary in České Budějovice, Czech Republic; the Koeru Mary Magdalene Church in Koeru, Estonia, and the Church of Padise Abbey in Estonia. The experiments and *in situ* testing has shown that it is possible to mix a custom injection grout with sufficient workability, low density and suitable mechanical properties. Therefore I encourage conservation specialists to continue studying and developing grout compositions that can fit the needs of different conservation works, and hope that this study has provided a useful contribution to such a process.

Acknowledgments

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RESUMÉ

NÁVRHY NA SLOŽENÍ INJEKTÁŽNÍCH MALT A VYHODNOCENÍ JEJICH POUŽITÍ PŘI KONSOLIDACI HISTORICKÝCH OMÍTEK V KOSTELE PANNY MARIE V PÖIDE V ESTONSKU

Cílem této studie bylo posoudit vhodnost speciálně připravených injektážních směsí pro potřeby konzervace historických omítek kostela Panny Marie v Pöide v Estonsku a celkově vylepšit kvalitu injektážních malt používaných v oblasti památkové péče. Projekt se zaměřil na vytvoření maltových směsí, které by byly nejvhodnější k vyplnění dutin a opravě omítky a zdiva nejenom v kostele Panny Marie, ale i při restaurátorských zásazích v objektech nacházejících se v podobném stavu.

Hlavní náplní práce bylo najít složení maltových směsí a vyhodnotit jej. Výzkum se věnoval vlastnostem běžně dostupných a speciálně namíchaných injektážních směsí během aplikace, schnutí a ve fázi po vytvrdnutí.

Pro výrobu maltových směsí bylo zvoleno hydraulické vápenné pojivo NHL5, neboť korespondovalo s tvrdostí původní omítky v kostele Panny Marie. Jako plnivo byla pro směsi použita vápencová moučka, kuličky expandovaného skla či kombinace obojího. Počáteční pozornost byla věnována aplikaci. Experimenty ukázaly, že k dosažení lepších vlastností je třeba do směsí přimíchat ztekucovací přísady, např. Melment F10, a přísady jako methyl-hydroxyethyl celulosa. Ty zaručují vhodné vlastnosti směsí při kontaktu s absorpčními materiály, např. původní omítkou. Ke zvýšení retence vody a přilnavosti byla přidána akrylová disperze K9. Vzhledem k negativním účinkům na fluiditu a retenci je ovšem třeba použití této disperze ve směsích podrobit dalšímu zkoumání.

Výsledky testů provedených na vyzrálé maltě ukázaly, že mechanická pevnost běžně dostupných směsí je vyšší či stejná jako v případě původní omítky. Pevnost v tlaku a tvrdost Ledanu TA1 lze snížit přidáním plniva. Speciálně namíchané směsi vykázaly nižší pevnost v tlaku a větší pružnost než původní omítka a v testu přilnavosti dosáhly vysokých hodnot. Speciálně připravené směsi jsou kompatibilní s původní omítkou a mohou snížit náklady na restaurátorský materiál, a to zejména v případech, kdy jsou poškozené velké plochy omítky, jak je tomu i v kostele Panny Marie. Komerční směs Ledan TA1 se nicméně ukázala jako vhodnější při vyplňování menších prasklin a dutin omítky.

Od ukončení projektu byly vybrané směsi použity ke konsolidaci původní omítky nejen v kostele Panny Marie, ale i na dalších místech v Estonsku a České republice. Byly mezi nimi kaple Nejsvětějšího Srdce Páně v kostele Panny Marie Růžencové v Českých Budějovicích, kostel sv. Máří Magdaleny v Koeru či v opatství Padise (obojí v Estonsku). Experimenty a testy přímo na místě prokázaly, že je možné namíchat injektážní maltovou směs s dobrou zpracovatelností, nízkou hustotou a vhodnými mechanickými vlastnostmi. Odborníkům na památkovou péči lze tedy doporučit, aby – i na základě této studie – dále zkoumali a vylepšovali složení injektážních směsí, tak aby odpovídaly konkrétním potřebám restaurátorských zásahů.