ESTONIAN ACADEMY OF ARTS
Faculty of Art and Culture
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Varje Õunapuu

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

Master Thesis

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Abstract:

This research analyzed and evaluated 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. The research was inspired by personal experience in the ongoing research and restoration works at St. Mary's Church in Pöide, Estonia. The church is one of the oldest in the country, dating back to the 13th century, and has been slowly restored from a ruined state over the last several decades. However, large areas of ancient plaster are still detached and loose, and require repair. Materials used in the process of injection grouting could be improved while also reducing conservation costs, and that has been the aim of this study.

Experiments analyzed and evaluated two commercial grouts, Ledan TA1 and Vapo Injekt, and also forty-one custom grout mixtures. The basic recipe for the design of the custom grouts was inspired by the grout mixture developed in 1981 by the International Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM), and also personal familiarity with the working properties of the commercial grouts which were selected for testing.

Test methods were selected based on the *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces: A Manual of Laboratory and Field Test Methods* by The Getty Conservation Institute, and also on the experience of the advisors of this study, MA. Jan Vojtěchovský Ph.D. and MSc Karol Bayer from the Faculty of Restoration, University of Pardubice.

The research began by evaluating plaster samples from St. Mary's Church in order to set the reference data for finding compatible injection grouts. Experiments included a funnel test, filter paper test, and a flow on plastered panel test to evaluate the workability of the grouting mixtures, and to determine the quantities of admixtures. Tests also evaluated the volume shrinkage of the grouts. Capillary water absorption, compressive strength, adhesion, and ultrasonic pulse velocity were measured, and the matrix structure of the hardened grouts was examined by the SEM-EDS analysis.

The results of these experiments determined that three of the custom grouts were most suitable for testing in real working conditions. Further on-site testing was carried out at St. Mary's Church, and also in the Chapel of the Sacred Heart at the Church of the Virgin Mary of the Rosary in České Budějovice, Czech Republic.

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 more compatible with the historic plaster, and can reduce the cost of materials for conservation works. However, commercial grout Ledan TA1 was still more useful when applied to fine cracks and small pieces of detached plaster.

Suggestions for further research and design of custom grouts is offered at the end of the paper.

Number of illustrations: 74

References: 74

Sources: 32

Key words: St. Mary's Church, conservation, ancient plaster, injection grouting, custom-mixed injection grouts, evaluation.

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Varje Õunapuu

Introduction

St. Mary's Church is an extraordinary monument on the island of Saaremaa in the Western part of Estonia, situated in the small village of Pöide. The church is one of the oldest in the country, dating back to the 13th century. Because of its importance in the local history, it has been of interest to many local art historians, researchers, and restorers throughout time. The church differs from other medieval churches in Estonia due to its massive, one-nave shape and its unique artworks, including medieval paintings and stone carvings.

In 2012, I was part of the research team on the project *Sustainable Management of Historic Rural Churches,* which mapped the condition and the layers of the historic facade and interior plaster in St. Mary's Church. It was while working on this team that I became more interested in the conservation of the historic render in St. Mary's church, and I continued my study of the church with two more workshops in 2016 and 2017. In 2016, I participated in a one week course at the church, called *Nano-lime for Conservation of Stone, Plaster and Architectural Surfaces*, and in 2017, I joined a grouting workshop about the practical methods and materials for the consolidation of historic plaster. Through all of these experiences I developed a personal interest which inspired me to do my thesis research on the reattachment of the historic plaster at this site.

The aim of this thesis research was to find suitable injection grouts for the conservation of the historic interior plaster in St. Mary's Church, Pöide, taking into account the inner climate of the church, as well as the composition and characteristics of the historic plaster. In addition, I wanted 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.

This paper is divided into three parts. The first part gives an overview of the church itself, including the history of its construction and conservation, the technical condition of the interior render, and the various factors that contributed to its decay. This section provides a frame of reference with which to introduce the central concern of this paper, which is an assessment of various injection grouts and grouting methods for the re-adhesion of historic plaster in St. Mary's Church.

The second part presents the methods used for the evaluation of the historic plaster, and also for the design and evaluation of the injection grouts. It gives an overview of the composition of the grouts, the test methods used to evaluate the grouts, and the test results. Test methods were chosen based on the *Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of*

Architectural Surfaces. A Manual of Laboratory and Field Test Methods by The Getty Conservation Institute[1], and also on the experience of Jan Vojtěchovský and Karol Bayer from the Faculty of Restoration, University of Pardubice, who served as advisors for this study.

The third part describes the *in situ* experiments, and presents a final discussion and conclusions. I did the on-site experiments at St. Mary's Church myself, and further experiments were done in the Chapel of the Sacred Heart at the Church of the Virgin Mary of the Rosary in České Budějovice, Czech Republic by the students of the Studio of Restoration and Conservation of Wall Paintings and Sgraffito from the University of Pardubice.

[1] Biçer-Şimşir, Beril, and 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.

1. A brief history of St. Mary's Church

St. Mary's Church in Pöide is the biggest single-nave church in Estonia. The church was built by the Livonian Order during the 13th century, together with the fortress connected on the Northern side of the building.[2] Throughout the centuries the church has caught fire several times. The most recent fire occurred in 1940 when the spike of the tower was struck by lightning. The lightning strike damaged the tower vaulting, the steeple was totally burnt down, and a massive crack formed along the western wall. Later, during the Second World War, the interior of the church was destroyed by the Soviet military and the space was used as storage.[3] The condition of the Church deteriorated dramatically during the decade after the war, and in 1955 was officially estimated as critical by the architect Toivo Kallas, who compiled the report about the technical conditions of the church.[4] Practically, only ruins remained.

1.1 Construction history

From 1958 to 1961, the first major field and restoration works done since 1904 saved the church from further disintegration. These projects were led by the Estonian architectural and art historians Kalvi Aluve (1929-2009) and Villem Raam (1910-1996). Researchers determined that the construction history of the church could be divided into three stages – the Romanesque church (I), the Gothic church (II), and the construction of the tower (III). I participated in a study of historic plasters led by the wall-painting conservator Eva Mölder in 2012, which further detailed the timeline of the church into seven stages – the Romanesque church (I), extension against the western wall of Romanesque church, which later became the basis for a tower (II), the Gothic rebuilding stage (III), the heightening of the tower in two stages (IV), the construction of the sacristy (V), the 17th-18th century renovation works on the façade and interior (VI), and the building of the partition wall between the tower vaulting and the nave, together with the organ loft in 1852 (VII).[5]

The Romanesque church was possibly built during the 1230's, and although its exact dimensions are not known, its original portals and window remain even today in the central part of the building.[6]

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[2] Üldtoimetaja Villem Raam, Eesti Arhitektuur 2. Lääne-,Saare-, Hiiu-, Pärnu-, Viljandimaa. Tallinn: „Valgus", 1996, pp. 77-78.

[3] Eesti kirikud andmebaas, Pöide kirik, konserveerimise ajalugu, http://kirikud.muinas.ee/?page=3&subpage=134&id=136 (accessed 04.04.19)

[4] Archive of the National Heritage Board of Estonia (Muinsuskaitse arhiiv, henceforth MA), P-11830: T. Kallas, Tehnilise seisukorra akt Saaremaal, Orissaare rajoonis asuva Pöide kiriku kohta, Tallinn, 1955.

[5] J. Kilumets, E. Sova, Pöide kiriku fassaadi ja interjööri restaureerimise põhiprojekt. Tallinn, 2013, pp. 22-23.

[6] Üldtoimetaja Villem Raam, Eesti Arhitektuur 2. ... 1996, pp. 77-78.

The renovation of the church to Gothic style happened during the last quarter of the 13th century. The walls were built higher and a travis was added to the Eastern and Western sides of the building. The ceiling was also arched, and Gothic windows and portals were installed and decorated with stone carvings and paintings. Probably no later than the 15th century, the western vaulting was elevated to form a massive tower.[7]

During the large scale renovation works in 17th-18th century, the façade was plastered. The interior was also renewed with lime-wash, and decorated with paintings.

This is the most current understanding of the construction history of St. Mary's Church at the time of my research.

1.2 The main restoration works and study

1.2.1 Field and restoration works in St. Mary's Church 1958–1961

Estonian art historians and restorers Kalvi Aluve and Villem Raam led a field project in St. Mary's Church between 1958-1961. They sought to discover the historical construction process of the building, and to design a conservation and restoration project based on this new understanding. To reveal the joints in the masonry, seventeen opening were cut through the historic plaster layers. Each opening had a size of 1 m² or more.[8] This method helped researchers answer questions about construction history, but was destructive to the architectural surfaces of the interior and the façade. It seems possible that during that period researchers may have overlooked the value of historic plasters that did not bear a painting, and were more willing to damage undecorated areas in order to further their studies.

In addition to this research, during these years restoration was being done to begin recovering the church from its ruined state. Works included repair of the vaults and the building of a new roof.[9]

1.2.2 Restoration of the choir and sacristy 1994-1998

From 1994-1998, projects were undertaken to restore the sacristy and choir, so that the congregation of St. Mary's Church could use the space for services. Windows, doors, historic

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7 Üldtoimetaja Villem Raam, Eesti Arhitektuur 2. ... 1996, pp. 77-78.

8 MA, P-359: K. Aluve. V. Raam. Pöide kirikus teostatud väliuurimistööd. Köide I. Tallinn, 1962, p. 3.

9 MA, P-360: K. Aluve. Aruanne 1958-1961. a. teostatud väliuurimiste kohta.Tallinn, 1962, pp. 5-6.

plaster, wall-paintings, and the altar were restored. Works in the sacristy were led by a company called OÜ Frantsiskus. With these restorations, masonry was repaired, and the walls were plastered and painted.[10] The final report of these projects describe that restorers used local sand, lime, and limestone for reconstruction and repair. This was a new restoration technique influenced by methods and materials used in Northern countries. The concept was to make as few changes as possible, and to use traditional materials to technically and aesthetically match with the original materials.[11] Even though original fragments of plaster were preserved, no documentation about the scale, condition, or the conservation methods used at the time currently exist.

The same company continued works in the choir, restoring the altar and the floor, keeping to the same restoration concept of using natural materials as was applied in the sacristy. At the same time, the company AS KAR Grupp studied and conserved the historic plaster and wall-paintings.[12] During conservation works, detached and loose layers of plaster were re-attached to the walls. Paintings were cleaned and consolidated, and lacunas were filled and retouched. The final report describes that layers of plaster were attached by injection method, and that the grout used was based on lime. According to the memories of Anneli Randla, the head researcher at the Estonian Academy of Arts, the grouts were most likely based on lime putty. This method was probably used for bigger, more unstable voids. For lime-wash layers and thinner pieces of plaster, re-adhesion was done using Ledan TB 03.[13]

The condition of the plaster in the choir has not been studied since these projects, so the efficiency of injection grouting done during this period of restoration is unclear. It is only known from the report that the conservation of the walls and the paintings in the choir was done in outdoor climate conditions, because at that time there were no windows.[14] This means that unstable climate conditions could influence the results of the restoration work.

Lastly, new stained-glass windows designed and made by Andrei Lobanov were installed in 1997 -1998.[15]

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10 MA, A-3922: T. Parmakson. T. Sepp. Pöide kirik. Restaureerimistööde aruanne 1995-1998. Tallinn, 1998, p. 2.

11 MA, A-3922: T. Parmakson. T. Sepp. Pöide kirik. ... p. 2.

12 MA, A-5535: AS KAR Grupp. Pöide kiriku siseviimistluse sondeerimistööde aruanne. Tallinn, 1994.

13 MA, A-5535: AS KAR Grupp. Pöide kiriku kooriruumi maalingute konserveerimis - uurimistööde aruanne.Tallinn, 1995, p. 2.

14 AS KAR Grupp. Pöide kiriku kooriruumi maalingute ... p.1

15 AS KAR Grupp. Pöide kiriku kooriruumi maalingute ... p.1

1.2.3 Conservation, restoration, and research from 2000-2016

At the beginning of the 2000's, Jaak Mäll led archaeological studies on the northern side of the church in the area near the fortress. The aim of the studies was to finish the works held in 1989-1991 by removing the oldest layers of the excessive soil from the ruins of the fortress. Later in 2007, OÜ Rändmeister surveyed and studied the ashlar windows of the church.[16] The dimensions and deterioration of the ashlar window frames were documented to compile an action plan for the restoration works.

The next large research project at St. Mary's Church, *The Sustainable Management of Historic Rural Churches in the Baltic Sea Region (SMC),* took place in 2012. This project mapped the condition of the historic layers of plaster. In addition, the researchers tested lime-based repair mortars *in situ*, and also made separate tests of the conservation mortars in a laboratory. With the aim of applying a holistic approach to the management of rural churches, the team also researched the indoor climate conditions, the moisture content in the massive limestone walls, as well as bio-deterioration in the building.[17]

Additional research looked into the previous records related to the history of the church's construction. Researchers created a graphic documentation, which gave an overview of the different historic plaster layers and its condition (see ill. 1). Based on the collected information about different types of masonry mortar and rendering plaster, art historians and researchers agreed on the new construction timeline.[18]

Visual examination determined eleven different types of plaster. The oldest belonged to the Romanesque church, and the latest, used to repair the render of the walls, presumably belongs to the first half of the 20th century. Six kinds of decay were identified in the conditions of the plaster:[19]

1. Hollows - plaster slightly detached from the wall
2. Bigger hollows and moving/ detached plaster
3. Loss of the surface
4. Sanding – loss of cohesion
5. Bigger cracks in the render

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[16] Eesti kirikud database,... (accessed 04.12.17)

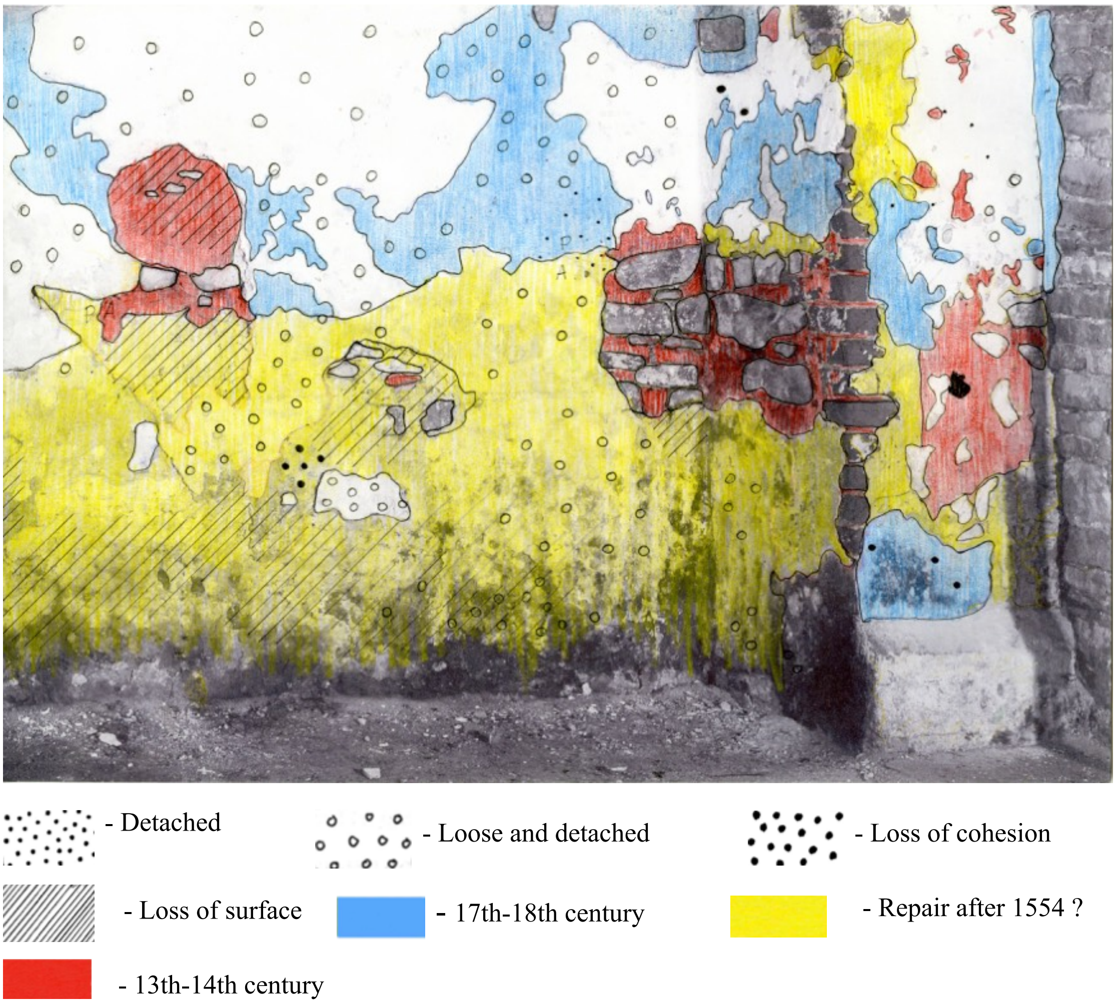
[17] The final report of the project *Sustainable Management of Historic Rural Churches in the Baltic Sea Region (SMC)* (Kalamees T., editor). Tallinn-Visby, Estonia-Sweden, 2013.

[18] J. Kilumets, E. Sova, Pöide kiriku fassaadi ja interjööri restaureerimise põhiprojekt. Tallinn, 2013, pp. 22-23.

[19] E. Mölder, Pöide kiriku fassaadi ja interjööri seisukorra uuringud. Uuringute aruanne I. Välis- ja siseviimistlus. Tallinn, 2013, pp. 85-86.

6. Deeper holes in the render

1. A fragment of a graphic documentation done during the SMC project in 2012. Fragment from the Southern wall of the nave. Foto: Peeter Säre. Graphic documentation: Varje Õunapuu, Eva Mölder.



For my research, plaster from the Gothic construction period, which is the second building stage, was chosen as the reference for further tests. The first layer of the remaining plaster on the vaults and the walls, and also the painted decorations, belong to the 13^{th}-14^{th} century when the romanesque style church became gothic.[20]

The 2012 SMC project made a study of paint layers in addition to studying the plaster. Researchers discovered painted decorations on all three vaultings and the choir of the church. Underdrawings of the decorations had been pressed inside the fresh mortar, and can be still seen with close visual examination. This is the main proof that the paintings belong to the Gothic construction period, together with the interior plaster which covers the vaults and the walls, as well as the masonry of the vaults.

[20] E. Mölder, Pöide kiriku fassaadi…, Tallinn, 2013, pp. 5.

Paintings in St. Mary's Church can be described as architectural decorations. On the vaults of the choir, painted groins are combined with the limestone ribs and finely carved polychrome stone bosses in the centre of the vault to create more ceremonial look. On the northern wall, researchers discovered a fragment of a painted rose window. In the nave, the fragment of an underdrawing of a smaller rose window was revealed. The general decoration scheme for the nave and the vault under the tower is characterized by groins and bosses marked with modelled and painted plaster.[21] From the three painted vault bosses, the most well preserved is the one on the vault under the tower. It depicts a schematic drawing of a man's face, surrounded by the Fleur-de-lis motifs which mark the four groins (see ill. 2).[22]

Restoration of the roof began immediately following the SMC projects in 2012, and finished by 2014. This eliminated extensive rainwater leakages, and soon after the interior of the church, mainly the vaults, began to dry. Lastly, restoration and conservation of the portals and windows was carried out in 2015-2016.

2. Man's face surrounded by the Fleur-de-lis motifs in St. Mary's Church. Photo: Peeter Säre

[21] A. Randla, H. Hiiop, Mõningaid uusi andmeid vanast dekoratiivsest seinamaalist [New Finds of Church Murals in Estonia], Kustiteduslikke Uurimusi [Studies on Art and Architecture], Estonian Society of Art Historians and Curators, Tallinn, 2016, pp.145-163

[22] H. Hiiop, J. Kilumets, A. Randla, Kes vaatab Pöide võlvilt? [Who is Looking Down from the Vault of St. Mary's Church in Pöide], Järelevastamine. Kaur Alttoale, Eesti Kunstiakadeemia muinsuskaitse ja konserveerimise osakond: 2017, p. 280.

1.2.4 International workshops in 2016 and 2017

The conservation department of the Estonian Academy of Arts organised international workshops at St. Mary's Church during two consecutive years. In 2016, the workshop *Nano-lime for Conservation of Stone, Plaster and Architectural Surfaces* reviewed various nanoparticle lime products (CaLoSil E 25; CaLoSil Paste-like; CaLoSil Micro), and tested their application methods on site. Nano-lime is suitable for the consolidation of historic plaster which has poor cohesion, as is found in St. Mary's Church due to the long-term excessive moisture in the limestone walls.[23]

In 2017, the second workshop was held in Pöide, this time focusing on the re-adhesion of the architectural surfaces. The combination of theory and practice offered an excellent opportunity for participants to learn about hydraulic lime-based injection grouting mixtures, and to actually go through the injection process by practicing on the original plaster of St. Mary's Church.

1.2.5 Conclusions

The restoration and research projects done in Pöide demonstrate different approaches to the preservation of this valuable monument. Works done at the end of the 1950's improved the technical condition of the church considerably, and increased understanding about its different construction stages. This information provided a valuable basis for later research. During the SMC project in 2012, the construction stages were reviewed and made more complete based on the study of the historic render.

The choir and sacristy are the only parts of the church which have been fully restored, during the 1990's. It would be useful to re-evaluate this work in order to see how the restoration materials have resisted the rather unstable indoor climate at the church.

In recent years, the condition of the church has improved in many ways. In 2014, the building of a new roof was finished, and the church started to dry. A project in 2015 restored the portals and stained-glass windows, and helped to stabilize the indoor climate. The latest workshops in 2016 and 2017 gave inspiration and ideas about suitable methods and materials for the conservation of the interior render.

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[23] Nano-lime for Conservation of Stone, Plaster and Architectural Surfaces. Final Report. Tallinn, 2016, pp. 4-12.

2. The causes of deterioration of the church and the interior render

To better understand the extent of conservation work which can still be done in St. Mary's Church, an awareness of the technical conditions of the church as a whole can be useful. Like other medieval churches in the region, the church is unheated and the indoor climate depends on the outdoor climate. The concerns related to the indoor and outdoor climate are as follows:[24]

- Decay of waterlogged constructions caused by freezing and melting cycles
- Condensation of aqueous vapour on and inside architectural surfaces
- Excessive moisture caused by unregulated indoor climate
- Microbiological decay – algae, fungi, bacteria
- Recrystallization of water-soluble salts

Excessive moisture is mainly conditioned by different factors, including:[25]

- Absence of rain gutters
- Rain falling down at an angle and wetting the façade of the building
- Water pouring down on the façade and entering inside from the cracks and damaged areas
- Condensation of aqueous vapour on cold surfaces in the interior
- Water dripping down from the eaves and splashing onto the lower part of the wall
- Capillary rise of water inside the soil and walls of the church

Through the centuries, the roof and tower of St. Mary's Church have had problems with leakage, and during the first half of the 1900's were renovated regularly. In 1940, the spire of the tower burnt down. During and after World War II, and until the restoration works in the beginning of 1960's, the church was in extremely deteriorated conditions. Also preceding the restoration of the roof in 2012-2014, there were problems with leakage for several decades.[26] Constant moist conditions in the church have been extremely destructive to the interior render, resulting in green algae almost fully covering the walls and vaulting, and also decay to the historic plaster. Large areas

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[24] P. Klõšeiko, T. Kalamees. Eesti uuritud kirikute ehitustehniline seisund. In: The final report of research of project "Sustainable Management of Historic Rural Churches in the Baltic Sea Region (SMC)" (Kalamees T., editor). Tallinn-Visby, Estonia-Sweden, 2013, p 7.

[25] P. Klõšeiko, T. Kalamees. Eesti uuritud ..., p. 8.

[26] Eesti kirikud database,…(accessed at 04.04.19).

of the plaster are dangerously detached from the masonry. The SMC project in 2012 estimated that only about 60% of the interior render remains, and large areas of it are loose and have big voids.[27]

Although restoration of the roof stopped leakages by 2014, excessive moisture can still find its way into the masonry through the soil with capillary rise, as well as due to the absence of rain gutters. More stable indoor conditions are needed to achieve durable conservation results. Conservation of interior plasters and paintings might not be first on the list of works to be done to preserve the church, however research and testing for finding suitable methods and materials is necessary.

2.1 Challenges for plaster conservation in St. Mary's Church

Several characteristics of St. Mary's Church complicate conservation of the interior render. The surface area to be conserved is quite large, requiring extensive materials, time, and money for the restoration work. The condition of the historic render is also poor, and large areas of it have been destroyed. Further, there are several other problematic conditions. Studies of the plaster in 2012 distinguished several types of decay:[28]

1. Slight detachment: Plaster is still stable if it has good cohesion and is not cracked. Therefore injection grouting is generally not needed. Almost every piece of plaster in the interior is slightly detached from the masonry.

2. Extensive detachment: Plaster is detached several centimeters from the ground surface, and has deformed. The area is usually cracked and is moving. There are loose pieces of crumbled plaster in the voids which makes consolidation of these areas more difficult. This type of decay is more common in the western part of the nave on the northern and southern wall at the level of the springers, and on the southern panels of the vaulting. Due to the large size of the voids, the use of lightweight injection grout could be considered in these situations.

3. Friable plaster: Sanding off, powdering. The historic plaster has lost cohesion or is lacking of it. This type of decay is more often found on the middle and lower parts of the walls in areas where the presence of excessive moisture has been constant, usually due to the capillary rise of water from the soil. Crystallization of soluble salts, and also freezing and melting cycles, have

[27] J. Kilumets, E. Sova, Pöide…, 2013, p. 25.

[28] E. Mölder, Pöide kiriku fassaadi…, ja interjööri seisukorra uuringud. Uuringute aruanne I. Välis- ja siseviimistlus. Tallinn, 2013, pp. 13-14.

destroyed and weakened the structure in the plasters, so structural consolidation is needed before injection grouting can be done.

4. Loss of surface: The loss of surface of the plaster can be seen on all the walls and on all the levels. It is not a big problem if the plaster has retained cohesion, but if the plaster is friable and detached, consolidation of the structure is required before injection grouting.

5. Petrification: Formation of a hardened crust on the surface of the plaster, together with the traces of lime wash layers, forms in areas which have been wet for longer periods of time. It is common on the vaults and springers, and on the walls near these areas.

6. Bio deterioration: During the 2012 study, three types of microorganisms were found on the surface of the walls: algae, invisible surface mould, and bacteria (pink coloration).[29] The growth of algae in St. Mary's Church was extensive. Moist conditions and the leaking roof created a suitable environment for algae colonies to grow and spread. Damage is extensive in the areas of the springers. After the construction of the new roof was finished in 2014, areas with algae colonies were able to dry, inhibiting the growth of algae. Visual examination shows that areas on the springers are not dark green anymore, and there are no signs of formation of new colonies. However, on the socle area conditions are more unstable. In these areas excessive moisture is mainly caused by capillary rise and rainy weather conditions, which prevent the lower areas of the walls from drying and allows for continued algae growth.

7. **Delamination:** The loss of adhesion between different plaster layers occurs most often on the walls where there are several layers of plaster.

8. **Bigger cracks and deeper holes** in the plaster are found on walls and vaultings.

3. Types and extent of decay of the historic interior plaster.[30]

	TYPE OF DECAY	EXTENT OF DECAY*
1.	Loss of render	• About 60%** of render remains on the walls and vaulting
2.	Slight detachment from the masonry	• About 50% of remaining plaster • In most cases plaster is still stable
3.	Extensive detachment from the masonry Plaster is loose and moving	• About 50% of remaining plaster • All over the walls and vaultings

[29] U. Kallavus. Biodeterioration, Salt Distribution and Damage to Plaster and Render/ Extent and Reasons for Salt, Mould and Rot Damage. In: The final report of research of project “Sustainable Management of Historic Rural Churches in the Baltic Sea Region (SMC)” (Kalamees T., editor). Tallinn-Visby, Estonia-Sweden, 2013, p 59.

[30] J. Kilumets, E. Sova, Pöide ...2013, p. 25.

4.	Friable plaster – loss of cohesion	• About 20% of remaining plaster • Characteristic decay mostly to the plaster found on the lower parts of the walls
5.	Loss of surface	• About 30% of remaining plaster • Decay can be found on both walls and vaultings at all the levels
6.	Petrification of the surface	• About 40% of remaining plaster. • On the upper parts of walls and vaultings • Especially areas where there has been water leakage
7.	Bio-deterioration (algae, un-visible surface mould, bacteria)	• About 40% of remaining render is covered with algae • The reach of bacteria and mould is not documented
8.	Delamination of different layers of plaster	• About 10% of remaining plaster • Occurs mostly on the middle and lower parts of the walls where there are different types of plasters on top of each other
9.	Bigger cracks and deeper holes in the plaster	• About 10% of remaining plaster

* Information about the extent of decay is based on graphical documentation from the 2012 study.[31]
**The 60% only includes render conditions in the nave and tower vaulting. The choir was excluded from study because it had already been fully restored in 1995.

2.1.1 Conclusions

The main types of decay necessitating injection grouting are the loose and moving plasters which have extensively detached from the walls, as well as layers of plaster which have been delaminated. The work which requires the greatest time and material is the filling of large voids which cover about 50% of the remaining render on both the walls and the vaulting. In the case of friable plaster, structural consolidation is needed before the application of injection grouting for reattachment.

[31] E. Mölder, Pöide ...2013, lk 62-77.

3. Injection grouting

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 fluid, compatible with the original surface material, be able to cure in environments with low levels of CO_2, and have the lowest concentration of soluble salts possible.[32]

In 1979, a group of researchers comprised of former course participants from the International Centre for the Study of the Preservation and Restoration of Cultural Property (ICCROM) began carrying out laboratory and field tests of grouting materials and techniques suitable for wall-paintings and mosaics. Their goal was to discover a less destructive method for conservation of mural decorations. Detachment of murals from the original support in order to transfer them to a new support is a method precisely described in *Conservation of Wall Paintings*, by Paolo and Laura Mora, and Paul Philippot, published in 1984. Even though the authors of the book conclude that these methods are destructive and irreversible and should be avoided if possible, it seems that detachment methods were still commonly used in Italian conservation practice at that time.[33]

The ICCROM team published their first series of results in 1981. At that time, materials such as lime casein, synthetic resins and cement were used to reattach plaster. However, these materials did not fulfill the requirements for the injection grouts presented by the research team, which included:[34]

1. The mortar must set in a reasonable time.
2. Volume shrinkage should be as small as possible.
3. Mechanical strength should not be too much above that of historic mortars.
4. The mortar should allow the passage of water vapour.
5. The soluble salt content in the mortar should be kept as low as possible.
6. Grout should develop some tackiness soon after injection because this quickly secures dangerously detached superficial fragments.

[32] B. Biçer-Şimşir, L. H. Rainer, Evaluation of Lime-Based, 2013, pp. 2-3.

[33] P. Mora, L. Mora, P. Philippot, Conservation of Wall Paintings. Glasgow: Butterworths, 1984, pp. 245-246.

[34] D. Ferragni, M. Forti, J. Malliet, P. Mora, J. M. Teutonico, G. Torraca, Injection Grouting of Mural Paintings and Mosaics. – Studies in Conservation 1984, vol. 29, no. sup. 1 January, p. 110.

Laboratory and field testing resulted in the development of ICCROM grout. The ingredients of the ICCROM grout formula are shown in the following table (see ill. 4):[35]

4. Composition of the ICCROM grout.

Hydraulic lime	1 volume part
Hydraulic filler	1-2 volume parts
5% Primal AC 33	10% vol. to lime
10% solution of sodium gluconate	1% vol. to lime
Water	2-9 volume parts

The choice of hydraulic lime as a binder ensured the curing of the grout in low-CO_2 conditions, as occur in voids between masonry and plaster. The filler in the grouting mixture reduces shrinkage and increases mechanical strength. The ICCROM team preferred to use reactive fillers like pozzolana and crushed brick powder because of their ability to react with calcium hydroxide at room temperature, and therefore to prevent soluble calcium from transforming into insoluble calcium carbonate efflorescence, which could damage the paint layer. The addition of sodium gluconate as a fluidizer increased the workability of the grout and minimised the amount of water in the mixture, reducing shrinkage. Another ingredient, Acrylic emulsion Primal AC33, reduced water loss to the adjacent pores and gave some tackiness to the mixture.[36]

A Manual of Laboratory and Field Test Methods, published by the Getty Conservation Institute in 2013, gives a precise overview of test methods which could be used to evaluate the commercial and custom injection grouts. Similar to the article by the ICCROM team, it covers information about materials used for grouts, including their working properties and performance characteristics, as well as methods used for evaluation.[37] I have used this manual as a resource when designing the test methods for this research. My research modifies methods suggested in the Getty manual, adapting them to meet the specific needs of the study. Some of the tests were designed based on the experience of the supervisors of this thesis, Jan Vojtěchovský and Karol Bayer.

The following table gives an overview of test methods which were used in the ICCROM research, and which were suggested in the Getty publication, and which I used in this study (see ill. 5).

[35] D. Ferragni, et. al., Injection Grouting, 1984, pp. 111-112.
[36] D. Ferragni, et. al., Injection Grouting, 1984, pp. 111-112.
[37] B. Biçer-Şimşir, L. H. Rainer, Evaluation of Lime-Based, 2013.

5. Overview of test methods used in the ICCROM research[38], suggested in Getty publication,[39] and used in this study:

PROPERTIES	ICCROM	GETTY	THESIS WORK
Working properties	• Flow cone test by using Marsh cone • Sand column test	• Sand column test • Expansion and bleeding test • Wet density • Water retention and release • Rheological measurements • Inject-ability and flow with syringe • Flow on plastered tile	• Glass funnel test • Filter paper test • Flow on plastered panel
Properties during setting and curing	• Time of setting by Vicat needle • Drying shrinkage (ASTM C474-67)	• Time of setting by Vicat needle • Drying shrinkage (ASTM C474-05) • Final setting time	• Drying shrinkage in cylinder (visual examination)
Hardened properties	• The Brazilian test - tensile strength • Measurement of pore size distribution by the mercury porosimeter • Measurements of electrical conductivity and concentration of sodium, potassium and calcium	• Splitting tensile strength • Soluble salt content by ion chromatography • Capillary water absorption • Water vapour transmission by the wet cup method • Shear bond strength	• Compressive strength • Ultrasonic Pulse Velocity measurements (e-modulus, density, velocity of propagation) • Capillary water absorption • Adhesion test • Measurements of electrical conductivity of the extraction of lightweight fillers

[38] D. Ferragni, et. al., Injection Grouting, 1984, pp. 112-115.

[39] B. Biçer-Şimşir, L. H. Rainer, Evaluation of Lime-Based, 2013, pp. 4-6.

4. Evaluation of the historic plaster samples

The following chapter gives an overview of test methods used to analyze the historic plaster from the Gothic construction period in St. Mary's Church. It also describes the process of designing the imitation mortar which was used in the laboratory testing of the injection grouts, and offers some concluding ideas about the results of the research.

4.1 Historic plaster from the Gothic construction period

Plaster from the Gothic construction period during the 13th-14th century forms the first layer of render on the vaults and walls of the church. I selected samples from this area to serve as the reference material for the injection grout research. Three samples were taken from the Southern vaulting panel of the tower, referred as TV-V-1, TV-V-2 and TV-V-3. One sample was taken from a second test area in the tower vaulting, located on the lower part of the Northern wall, referred to as TV-W-1.

4.2 Characteristics and composition of the historic plaster

4.2.1 Description of the test methods to examine historic plaster

I used the wet silicate analysis and the grain size distribution test to determine the filler-binder ratio and the size of the grains. In addition, cross sections were prepared for visual examination and SEM-EDS analysis of the samples. Ultrasonic pulse velocity tests measured the mechanical properties of the samples. This helped to determine the level of the natural hydraulic lime (2; 3,5 or 5)[40], which was used as a binder in the imitation mortar and the injection grouts. The aim was to reach compression strength compatible with the historic plaster. Finally, the water absorption coefficient was measured to evaluate porosity.

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[40] Hydraulic strength of the Natural Hydraulic Lime in MPa. For example 2, 3,5 and 5 MPa. Source: A. Costigan, S. Pavia, Influence of Mechanical Properties of Lime Mortar on the Strength of Masonry: 2nd Historic Mortar Conference HMC 2010 and RILEM TC 203-RHM Final Workshop, Prague, 22-24 September 2010, p. 459.

4.2.1.1 Mortar analysis. The simple method Wet silicate analysis

Test method is described in *A Laboratory Manual for the Architectural Conservators* published by ICCROM in 1988.[41]

The aim

This test analyzed the binder/aggregate ratio in the historic mortar samples.

Test procedures

1. The historic plaster sampled were weighed.
2. Samples were crushed into smaller pieces with a pestle. Grinding had to be gentle to avoid crushing the aggregate into smaller pieces, since this would give inaccurate results in the grain size distribution of the sand (see ill. 6).

6. Weighting and grinding of historic plaster samples.

3. The crushed samples were put into a 600 ml glass container and weighed again.
4. The crushed samples were covered with a 35% concentration of hydrochloric acid (HCl) and left to react overnight.
5. After the acid had completely dissolved the calcium carbonate, the remaining sand was filtered out from the liquid and washed with distilled water repetitively until the wash water was clear (see ill. 7).

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[41] . M. Teutonico, A Laboratory Manual for Architectural Conservators, Rome: ICCROM, 1988, p 113-116.

7. Crushed sample in hydrochloric acid and the filtration of the sand.

6. After filtering, the filter paper together with the sand was placed on a petri dish and dried in the oven at 45°C for two hours.
7. The sand was carefully removed from the filter paper to document the weight of the dry sand (see ill. 8).

8. Dried sand on a petri dish.

8. To calculate the percentage of the sand the total weight of the sand (2nd weight) was subtracted from the weight of the crushed sample (1st weight) and the result was multiplied with 100. The percentage of dissolved binder is equal with the percentage of the sand subtracted from the 100%.[42]

$$\% \text{ of sand} = (2^{nd} \text{ weight} / 1^{st} \text{ weight}) \times 100$$

% of dissolved binder = % of sand subtracted from 100%

9. Later the same sand was passed through a stack of ASTM[43] standard sieves, with the mesh diameter minimum of 0,063 mm, and the maximum of 4 mm, to measure the grain size distribution (see ill. 9).

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42 J. M. Teutonico, A Laboratory Manual, 1988, p 113-116.

43 American Society for Testing and Materials

9. The list of standard sieves used in the experiment.

Sieve no.	**Mesh diameter** D (mm)
5	4
10	2
18	1
35	0,5
60	0,25
120	0,125
230	0,063

4.2.1.2 Grain Size Distribution

This test measured the granulometry of the aggregates in the historic mortar, as well as in the imitation plaster.

Test Procedures

1. 100g of the sand was sieved through a stack of standard sieves with the mesh diameter from 0,063 mm - 4 mm to separate different sizes of sand particles.
2. After the sand particles had been separated, the weight of each group was measured.

Note:

- In the case of the historic plaster samples, the total amount of sand depended on the size of the samples which were available for collection from the site.

4.2.1.3 Preparation of cross sections[44]

The aim

Cross sections of the historic plaster samples were made for examination with the optical and scanning electron (SEM-EDS) microscopes.

[44] J. M. Teutonico, A Laboratory Manual, 1988, p 142-144

Test Procedures

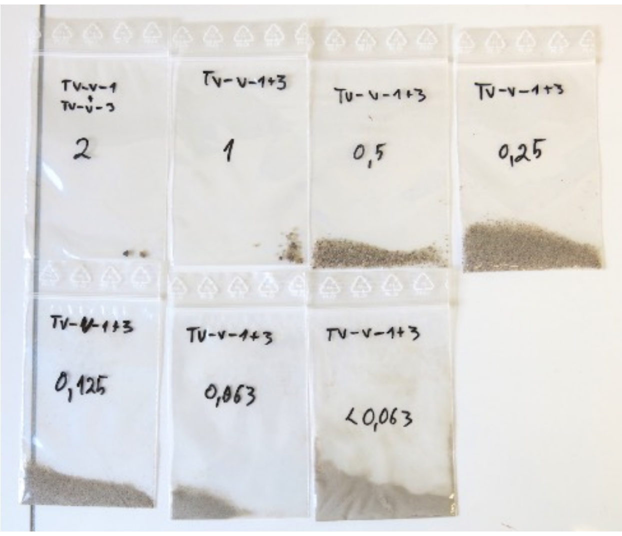
1. Selected plaster samples were placed in a silicon cast and soaked in transparent, low-viscosity epoxy resin (Araldite 2020/A - resin + Araldite 2020/B - hardener).
2. Samples were placed in a vacuum desiccator to prevent the formation of air bubbles in the resin.
3. When the resin was dry, then the cross section was prepared. One side of the sample was machine-polished using fine and very fine sandpapers to gradually remove scratches in the resin which could disturb the viewing of the cross section under the microscopes.

4.2.2 Test results

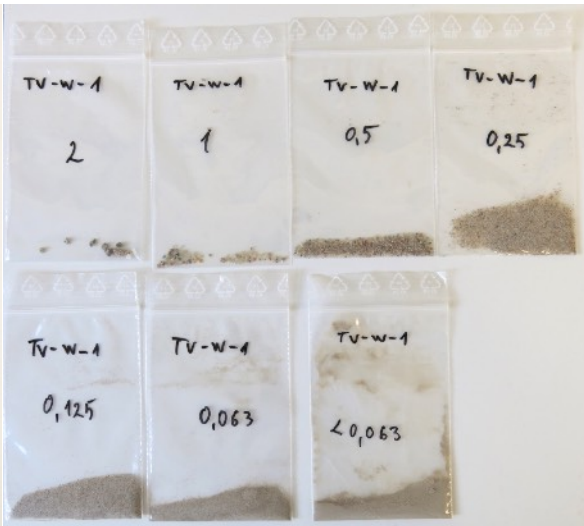
4.2.2.1 Results of the grain size distribution

Results of the grain size distribution analysis show a high content of fine and very fine sand in both historic mortar samples. The size of the grains is from 0-2 mm. See illustration 10, 11, 12 and 13.

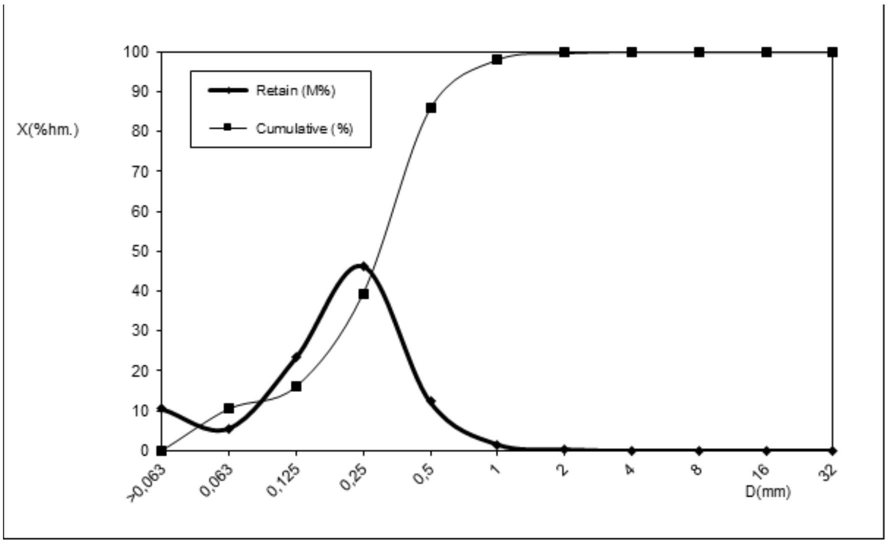
10. Sand divided by the grain size (crushed samples TV-V-1+TV-V-3 were tested together).



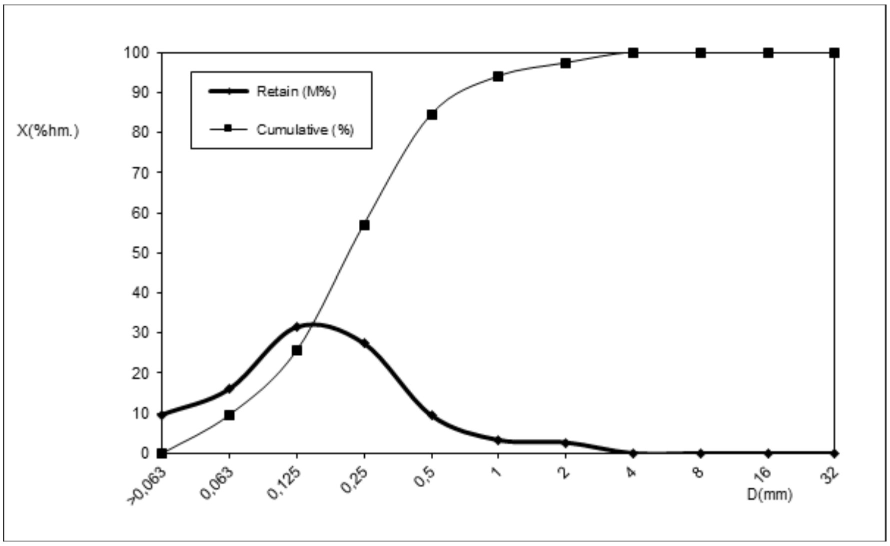
11. Sand divided by the grain size (sample TV-W-1).



12. Grain size distribution shown on chart (sample TV-V-1).



13. Grain size distribution shown on chart (sample TV-W-1).



4.2.2.2 Results of the ultrasonic pulse velocity measurements

The first three samples are taken from the vault and the fourth sample from the lower part of the Norther wall.

Measurements describe the mechanical properties of the historic plaster samples and show that the plaster from the wall is more stiff than the ones from the vault. In general the e-modulus of all the historic plaster samples are relatively high. This means that the plaster is hard and stiff in both of the cases.

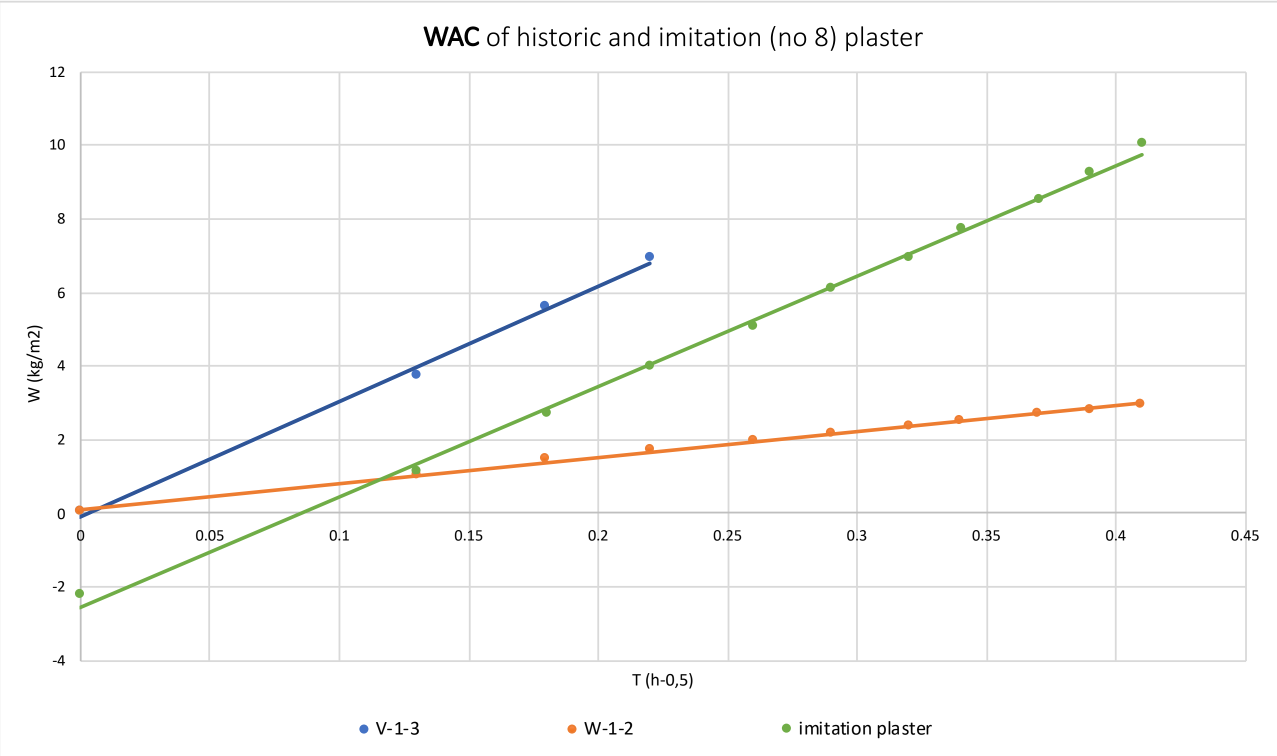
14. Ultrasonic pulse velocity (UPV) measurements for the historic plaster samples.

Sample no.	Vault V-1-1	Vault V-1-2	Vault V-1-3	Wall W-1-1
VP (Velocity propagation) (km/s)	2,96	2,618	2,636	3,053
E-modulus (kN/mm²)	5,886	5,868	7,476	8,233
Density (g/cm³)	1,054	1,181	1,689	1,591

4.2.3 Water absorption coefficient of historic plaster from the St. Mary's Church, Pöide

Water absorption through capillary action depends on the porosity of the plasters. More porous samples have a higher water absorption coefficient, drawing larger quantities of water within a shorter period of time. The test results show that the historic plaster sample taken from the vault is more porous than the one taken from the lower part of the Northern wall (see ill.15).

15. Water absorption coefficient of historic plasters from the vault (V-1-3), wall (W-1-2) and the plaster imitation number 8 (chosen to carry out laboratory testing of injection grouts).



Plaster from the vault (V-1-3)	Plaster from the wall (W-1-2)	Plaster imitation (no. 8)
W = 31.2 $kg.m^{-2}\cdot h^{-0,5}$	W = 7.1 $kg.m^{-2}\cdot h^{-0,5}$	W = 30.1 $kg.m^{-2}\cdot h^{-0,5}$

4.2.4 Characteristics of the historic plaster

Already by visual observation it was clear that the plaster is lime-rich, relatively hard, and consists of fine sand and big lumps of lime.

Results of the wet silicate analysis showed that the binder/aggregate weight ratio in the Gothic mortar was about 1:1. Compared to the plaster from the vaults, plaster from the wall contains a slightly higher amount of filler (see ill. 16).

16. Results of the wet silicate analysis.

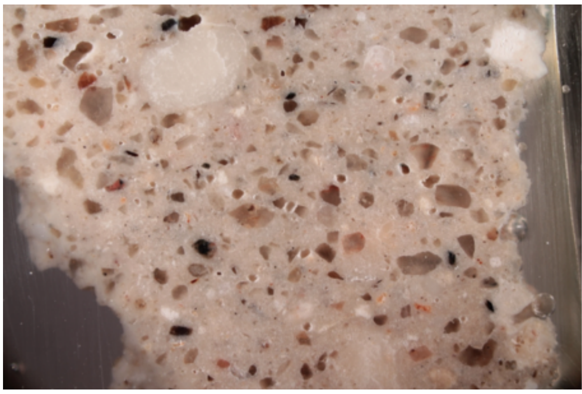
Sample no.	Content of unsoluble part in HCl (M %)	Content of soluble part in HCl (% W.) (M %)
TV-V-1 + TV-V-3	35,62	64,38
TV-W-1	47,17	52,83

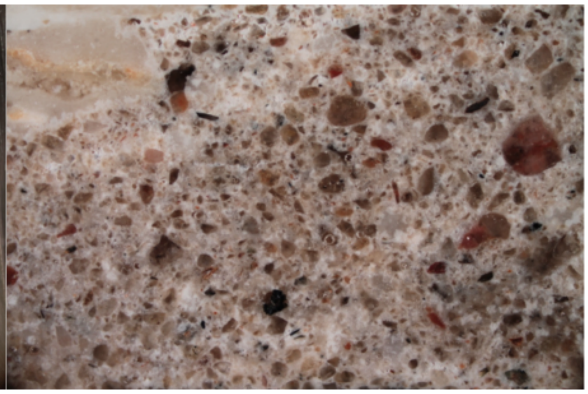
The grain size distribution analysis showed that it consists mostly of fine and very fine quartz sand (grain size of 0-2 mm) and a small amount of granite. Both plasters are compact and stiff. Cross sections were prepared to examine the samples under the optical and the scanning electron microscope.

The composition of the plasters from the vault and the wall are very similar, with the only difference being that the concentration of sand in the binder/filler ratio is more dense in the historic plaster sample from the wall. This can be clearly seen on the optical microscope photographs (see ill. 17 and 18):

17. Gothic plaster from the vaulting panel (magnification: x 20). Designation of the sample is TV-V-3.

18. Gothic plaster from the lower part of the Northern wall (magnification: x 20). Designation of the sample is TV-W-1.





Fine particles of quartz (transparent/white) and granite (light red) sand can be seen in these optical microscope photographs, taken from the sand of both samples, TV-V-1 and TV-W-1 (see ill. 19 and 20).

19. TV-V-1. Fraction of the sand is 0,25mm. Magnification: x10.

20. TV-W-1. Fraction of the sand is 0,25mm. Magnification: x10.

4.3 Design and preparation of the historic mortar imitations

I designed nine different mixtures with a variety of binder/aggregate and filler/filler ratios. I began by looking for suitable aggregates. A grain size distribution test was carried out with five different sands (see ill. 21) to find sands that matched the historic mortars. Two sands seemed most compatible. The first, and most compatible, was sand number 5, originating from Kostelecke Horky sand pit in Czech Republic, due to the wide variation of grain sizes (see ill. 22), and the second was sand number 4, from the Strelec sand pit in Czech Republic, due to its high amounts of very fine sand particles (see ill. 22). Because the range of the sand particles in the historic plaster was 0-2 mm, sand number 5, which originally had fractions from 0-4 mm, was sieved to the same range before being used in the imitation mortar samples. Sand number 4 was sieved to the range of particles from 0-0,063 mm to compensate for the lack of very fine particles in sand number 5. The percentage of the very fine sand in the nine mixtures varied from 3%, 7%, and 12%. Crushed marble was added to three of the formulas to imitate the lime lumps found in the historic plaster. The binder/aggregate ratio in the samples was 1:1 and 1:2 (see ill. 23).

All the mortars were cast and allowed to cure for 28 days before continuing with further analysis of the mechanical properties of the nine samples.

21. The grain size and the origin of the sands used to prepare the mortar.

Sand no.	1	2	3	4	5
Grain size	0-4 mm	0-0,5 mm	0-4 mm	0-0,5 mm	0-4 mm
Origin	Kostelecke Horky sand pit, Czech Republic.	Strelec sand pit white silicate sand, Czech Republic.	Sand from unknown source, Czech Republic.	Strelec sand pit, Czech Republic.	Kostelecke Horky sand pit, Czech Republic.
Notes	Sand has been previously sieved for other purposes.	-	-	White silicate sand. High amount of very fine fractions.	Un-sieved version of sand number 1.

22. Particle-size distribution for five sands tested for the mortar mixture (100g of each sand was used for sieving).

D (mm)	Sand no 1 (g)	Sand no 2 (g)	Sand no 3 (g)	Sand no 4 (g)	Sand no 5 (g)	Crushed marble (g)
4	0,31	-	3,48	-	4,29	-
2	0,60	-	11,81	-	6,25	3,04
1	10,26	-	13,43	-	6,65	62,37
0,5	48,31	5,63	22,40	1,15	16,09	12,08
0,25	32,22	82,33	29,78	9,50	42,91	6,9
0,125	6,67	10,61	9,47	43,40	21,75	5,13
0,063	1,09	0,31	1,11	34,77	1,41	3,42
< 0,063	0,43	0,09	2,12	10,73	0,54	6,9

4.3.1 Formulas of the 9 imitation mortars

The design process resulted in three different groups of imitation mortars. Mortars were divided into three groups, each with a specific concentration of very fine sand (0-0,063mm) – 3%, 7%, and 12 wt%, respectively (see ill. 23). The first mortar recipe in each group had a binder/

aggregate ratio of 1:1. The second contained 20 wt% of crushed marble to imitate the lime lumps found in the historic plaster. The last mixture had a binder/aggregate ratio of 1:2 (see ill. 24).

23. The amount of very fine sand added to mortar mixtures.

Formulas nr 1- 3	Formulas nr 4- 6	Formulas nr 7- 9
• **3 mass %** of sand no. 4 (0-0,063 mm) was added to sand no. 5 (0-2 mm)	• **7 mass %** of sand no. 4 (0-0,063 mm) was added to sand no. 5 (0-2 mm)	• **12 mass %** of sand no. 4 (0-0,063 mm) was added to sand no. 5 (0-2 mm)

24. The nine imitation mortar formulas.

Mortar No.	1	2	3
Binder aggregrate ratio (weight ratio)	1:1	1:1	1:2
NHL5 (g)	500	360	150
Sand (g)	500	300	300
Crushed marble (g)	-	60	-
Water (g)	434	288	140

Mortar No.	4	5	6
Binder aggregrate ratio (weight ratio)	1:1	1:1	1:2
NHL5 (g)	250	250	150
Sand (g)	250	200	300
Crushed marble (g)	-	50	-
Water (g)	200	200	120

Mortar No.	7	8 (chosen)	9
Binder aggregrate ratio (weight ratio)	1:1	1:1	1:2
NHL5 (g)	250	250	150
Sand (g)	250	200	300
Crushed marble (g)	-	50	-
Water (g)	200	200	120

4.3.2 Final selection of imitation mortar

The final imitation mortar was chosen based on the composition, and the ultrasonic pulse velocity, and capillary water absorption measurements after 28 days of curing the samples. Test results for all the recipes were similar. Mortar number 8 was chosen because of the highest amount of very fine sand, and because the mixture was containing crushed marble which was used to imitate the lime lumps.

25. Ultrasonic pulse velocity measurements for the nine imitation mortars.

Sample No.	1	2	3	4	5	6	7	8	9
VP (km/s)	1,592	1,695	1,438	1,608	1,470	1,474	1,331	1,601	1,678
E-modulus (kN/mm^2)	2,963	3,735	3,129	3,115	2,517	3,78	2,09	2,976	3,608
Density (g/cm^3)	1,694	1,769	2,028	1,699	1,686	1,974	1,589	1,685	1,873

*The size of each specimen was about 15x15x40mm.

4.3.3 Preparation of the selected imitation mortar

Both sands were sieved before preparing the mortar. Sand number 5 from Kostelecke Horky had a wide range of particles. In order to have similar particle size distribution as the original plaster, it was sieved with the no. 10 sieve, with a nominal sieve opening of 2 mm, before being added to the mortar mixture. To simulate the high amount of fine particle found in the historic plaster, 12 % mass of sand number 4 from Strelec sand pit was sieved with the no. 230 sieve, with a nominal sieve opening 0,063 mm, and added to the mixture. Finally, 20% mass of crushed marble [45] with particles in fractions of 0,5-1 mm was added to the mortar for the purpose of simulating the lime lumps of different sizes found in the historic mortar (see ill. 26).

When the NHL5 binder and the aggregates had been mixed together, water was added and the mortar was mixed by hand for about 1-2 minutes. Casts measuring 40x40x160 mm were filled with the mortar halfway to create samples of 20x40x160 mm size. The mortar was left uncovered in

[45] Product name: CAROLITH 0,5-1. Producer: AQUA Obnova staveb s.r.o, CZ

the cast for a maximum of one day, and then sealed with a plastic covering. After two days, the samples were taken out of the cast and left to cure for a minimum of 28 days.

26. Recipe for the chosen imitation mortar.

Ingredient	**Ratio/ amount** (g)
Binder : aggregrate ratio by weight	1:1
NHL5	250
Sand (consists of 12% of Strelec sand (0-0,063 mm) by weight + 88 weight % of sand from Kostelecke Horky (0-2 mm))	200
Crushed marble 20% by weight (g)	50
Water*	200

5. Design and evaluation of the injection grouts

5.1 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. Producers of commercial grouts have made in-depth research over long periods of time to test their products, so the working properties of the grouts are usually very good. However, the commercial products tend to be harder and less elastic than traditional mortars.[46]

Ledan TA1 was chosen for this study because of its good working properties and common use in conservation of wall paintings. 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. In some cases, as much as 7 litres of grout were injected into only one larger void.[47] This can make the cost of the restoration works quite high. For example,

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[46] According to the Ferragni et. al. in *Injection Grouting of Mural Paintings and Mosaics* the compressive strength of traditional mortars is in the range of 3-8 MPa.

[47]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.

in the case of recipe number 3 (see ill. 29) the cost of 7 litres of grout would be €73 if 1,3 kg of the dry powder is used per 1 litre of grout. The cost of Ledan TA1 is currently €8.03/kg at the *Kremer Pigments* webpage[48]. To lower the mechanical strength and the cost of Ledan products, the common practice is to modify the grout with the addition of an extra filler. In this study, limestone powder was added to Ledan TA1 in 1:1 weight ratio.

The second commercial grout I chose for testing was Vapo Inject, developed and produced in Czech Republic, and introduced in Estonia by Czech colleagues as a cheaper alternative to Ledan products. In the case of Vapo Inject, the modified version of the product was not tested. Experiments with Vapo Inject were done to better understand the material, and to compare its properties with Ledan TA1 and the custom injection grouts.

Materials for the custom grouts were chosen according to the mechanical properties of the historic plaster and the working properties of the commercial products. Natural hydraulic lime number 5 with the compressive strength of 5 MPa, was chosen to be the binder in order to reach the hardness of the historic plaster. From the natural hydraulic limes NHL 5 is the hardest which is available on the market. Admixtures like sodium gluconate, Melment F10, methyl hydroxyetyle cellulose, 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, and 3) the combination of limestone powder and lightweight fillers.

27. The cost of commercial products.

Product	Amount	Cost for 1kg (€)	Source of information
Ledan TA1	1 kg	8,03	https://www.kremer-pigmente.com/en/ (seen 09.04.19)
Vapo Injekt	1 kg	4,68	http://www.aquabarta.cz/4_stahuj/cenik.pdf (seen 09.04.19)

[48] https://www.kremer-pigmente.com/de/ledan-ta-1-leit-03-31020.html (seen 01.04.2019)

5.1.1 Lightweight aggregate

Three types of lightweight aggregates were used in this study - Poraver[49], Liaver[50] and Fillite 160W[51]. Products were chosen according to the availability and based on the personal preferences. Poraver and Liaver are both spherical expanded glass granulates, produced from the recycled glass. The particles are closed cells with tiny pores inside. Two granular sizes of these products were chosen for this study 0,1-0,3 mm and 0,25-0,5 mm. Fillite ceramic microspheres are glass hard and hollow. Product is produced by extracting the pulverised fuel ash from coal-fired power stations. The granular size of Fillite 160 W is in range of 5-180 µm.[52]

5.1.1.1 Gravimetrical analysis and conductivity measurements

Aggregates were analyzed beforehand to make sure that 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 (see ill. 28).[53] Conductivity measurements determine the ionic strength of the solutions but are not specific. It measures the total concentration of ions in solution. The ions responsible for the conductivity come from the electrolytes dissolved in the water and some of the salts are electrolytes.[54]

The lowest conductivity and the water soluble content had Fillite 160W, and the next one was Liaver 0,1-0,3 mm. Poraver in size of 0,1-0,3 mm, and especially Poraver 0,25-0,5 showed the highest percentage of the water soluble content from the three chosen light weight fillers. In conclusion, all the light weight fillers used in this study are suitable for the injection grouting.

Test Procedures:[55]

1. Extraction of the lightweight fillers was prepared by soaking 1g of lightweight filler in 50 ml of demineralised water.

[49] Poraver expanded glass, https://www.poraver.com/us/, (accessed 02.03.19).

[50] Liaver Expanded Glass Technologies, http://www.liaver.com/en/home/, (accessed 02.03.19).

[51] OMYA, https://www.omya.com/fillite, (accessed 02.03.19).

[52] micrometer

[53] J. M. Teutonico, A Laboratory Manual for Architectural Conservators. Rome: ICCROM, 1988, pp. 68-69.

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

[55] E-mail consultation with MSc Karol Bayer, University of Pardubice, (08.04.2019).

2. Filtration of the aggregate.
3. Conductivity measurement with the conductivity meter GHM 3430 Greisinger electronic GmbH.
4. Gravimetrical determination of bulk soluble content: 10ml of filtered extract was placed into a Petri dish and dried in the laboratory oven at 100°C to constant weight.
5. The bulk soluble content was calculated in relation to the weight of the lightweight filler.

28. Results of gravimetrical and conductivity measurements of lightweight fillers.[56]

Lightweight filler	Grain size (mm)	Bulk density (g/cm^3)	Water soluble content (wt%)	Conductivity (µS/cm)	pH
Poraver	0,1-0,3	0,47	0,47	165,1	7
	0,25-0,5	0,34	1,37	298	6
Liaver	0,1-0,3	0,4	0,27	106,4	6
	0,25-0,5	0,3	0,70	115,5	6
Fillite 160W	0,005-0,2	0,47	0,20	98,6	6

5.2 Formula design of commercial injection grouts

The following table presents the recipes for six varieties of commercial grouts which were prepared from Vapo Injekt and Ledan TA1 (see ill. 29). First, two mixtures were prepared based on the recommended water/product ratios found in the technical data sheets of the commercial products. In the case of Ledan TA1, the recommended ratio for strengthening of masonry and arches was 8 litres of water to 10 kg of Ledan TA1.[57] In the case of Vapo Inject, the recommended ratio was 3 parts dry powder to 1 part water. As a result, the mixture with Ledan was too liquid for the injection of larger voids, and the mixture with Vapo Injekt too thick for any injection grouting at all. The technical data sheet of Vapo Inject recommends adjusting with a 3:1 ratio by adding water

[56] R. Rajtárová, Reštaurovanie maľby sv. Vojtecha na severozápadnej fasáde Suchardovho domu v Novej Pake. Injektážne malty modifikované ľahčenými plnivami na vyplnenie dutín vo vápenných omietkach [Restoration of the wall painting St. Vojtech on the northwestern facade of Suchard‘s house in the town of Nová Paka. Injection grouts modified by lightweight aggregates to the fill cavities in lime plaster]. MA thesis. Litomyšl: University of Pardubice Faculty of Restoration, 2019, p. 98.

[57] Technical data sheet of Ledan TA1 from the Kremer Pigments website (http://shop.kremerpigments.com/en/) (accessed 09.04.2019).

as needed, but warns that too much liquid will cause sedimentation of the solid particles.[58] This occurred in the case of grout mixture number 4.

In the beginning of the study, I redesigned the ratio of mixtures with grouts number 3 and 5 to have a viscosity similar to honey. The aim was to create a mixture with good working properties, but with as little water as possible, to minimise the shrinkage of the drying grouts.

Grout number 6 is a modification of Ledan TA1. The aim was to see how much the compressive strength and elastic modulus of Ledan TA1 could be lowered by adding extra filler to the mixture, and also to evaluate the influence on the working properties and shrinkage volume.

Modification of the commercial product to reduce the compressive strength and bonding force is also recommended in the Ledan TA1 technical data sheet.

29. Commercial grout recipes and preparation notes.

Grout No.	**Product name**	**Amount** (g)	**Extra filler**	**Amount** (g)	**Water** (g)	**Notices**
1	Ledan TA 1	100	-	-	80	Ratio is recommended in product's technical data sheet. Grout was **too liquid** to inject voids bigger than few millimetres.
2	Vapo Injekt	90	-	-	30	Ratio is recommended in product's technical data sheet. Grout was **too thick** to inject.
3	Ledan TA 1	130	-	-	70	Consistency like honey. **Workability** and **water retention** of this grout was set as a positive example to custom-made grouts.
4	Vapo Injekt	130	-	-	70	Grout was created based on the same water : product ratios as in test no 3 to compare fresh mortars from Vapo Injekt and Ledan TA 1. Grout was **very liquid** and **fast sedimentation** was noticed.
5	Vapo Injekt	150	-	-	60	Consistency of this mixture was subjectively considered as good grout. **Consistency like honey***.

[58] Technical data sheet of Vapo Inject by AQUA obnova staveb s.r.o., Prague, Czech Republic.

Grout No.	Product name	Amount (g)	Extra filler	Amount (g)	Water (g)	Notices
6	Ledan TA 1	65	Lime-stone powder	65	70	Limestone powder was added to reduce excessive mechanical strength and the cost of Ledan TA1. Mixture had good consistency.

* *If the fresh grout has a consistency like honey the water : commercial product ratio is suitable for the injection of medium and large voids (starting from 5 mm).*

5.3 Formula design of custom injection grouts

5.3.1 Composition of the custom injection grouts

All the custom injection grouts consist of binder, additives, different kinds of fillers, and regular tap water (see ill. 32). NHL 5[59] was used as binder to match the hardness of the historic plaster.

To enhance the workability of the grouts, different additives were tested, including fluidizers, acrylic emulsion, and methyl hydroxyethyl cellulose. Acrylic emulsion and methyl hydroxyethyl cellulose were added to the grout mixtures to improve water retention and to increase the tackiness of the grouting mixtures.[60] Acrylic emulsion K9 was chosen to replace Primal AC 33, which was used in the ICCROM grout but is not in production anymore. Limestone powder and three types of lightweight fillers were tested as aggregates. Lightweight fillers were chosen to reduce the weight of the grouts. Products were chosen for their low soluble salt content and their availability on the market (see ill. 30).

[59] Otterbein, Calcidur NHL5

[60] D. Ferragni, et. al., Injection Grouting of Mural Paintings and Mosaics. – Studies in Conservation 1984, vol. 29, no. sup. 1 January, p. 112.

30. Overview of products used in this research.

Ingredients	Product name	Producer/ distributor	Reason why used in the grouts
Binder (Natural Hydraulic Lime 5)	HYDRADUR® NHL5 (Natural Hydraulic Lime EN 459-1)	OTTERBEIN	• Ability to harden by the reaction with water. • Hardness is compatible with historic lime plaster from the church.
Filler (limestone powder)	OMYACARB 15 VA (average size of the particles is 12 µm)	• Omya	• Used as filler. • Reduces shrinkage and makes the grout mechanically stronger.
Lightweight fillers	• Poraver (0,1-0,3mm; 0,25-0,5mm) • Liaver (0,1-0,3mm; 0,25-0,5mm) • Fillite 160W (5-180 µm)	• Dennert Poraver GmbH • Liaver GmbH & Co. KG • Omya	• Used as lightweight filler. • To reduce the weight of the fresh and hardened grout.
Fluidizers	• Melment® F 10 • Sodium gluconate	• BASF Construction Polymers GmbH •	• Used to make the grout more fluid. • Due to the effect of fluidizer the amount of water can be reduced. This in turn reduces the shrinkage.
Admixture (methyl hydroxyethyl cellulose)	Tylose® MH 300	• Kremer Pigmente GmbH & Co. KG (vendor)	• Used to raise the water retention of the grout. • To improve the fluidity of the grout in contact with absorbent materials. • To give some tackiness to the grout.
Admixture (acrylic emulsion)	Dispersion K9	• Kremer Pigmente GmbH & Co. KG (vendor)	• To reduce water loss during injection. • To give some tackiness to the grout.

31. The cost of the products used to prepare the custom made injection grouts.

Product	Amount	Cost with tax (€)	Approximate cost for 1kg (€)	Source of information
Otterbein Hydradur® NHL 5	25 kg	11.07	0,44	https://www.deffner-johann.de/ (seen 09.04.19)
OMYACARB 15 VA	1 kg	-	0,95	http://www.aquabarta.cz/4_stahuj/cenik.pdf (seen 09.04.19)
Poraver (0,1-0,3mm; 0,25-0,5mm)	55 l (12,5 kg)	29,19	2,34	https://www.poraver.com/en/ (seen 09.04.19)
Liaver (0,1-0,3mm; 0,25-0,5mm)	60 l (18 kg ± 15 %)	42,90	2,38	https://www.moertelshop.eu/ (seen 09.04.19)
Melment F10	-	-	-	-
Tylose® MH 300	1 kg	-	28,56	https://www.kremer-pigmente.com/en/ (seen 09.04.19)
Dispersion K9	1 L	-	10,71	https://www.kremer-pigmente.com/en/ (seen 09.04.19)

32. Formulas for all the custom grouts. Previous six recipes are based on the commercial grouts (see ill. 29).

Grout no.	Binder	(g)	Additive	(g)	Filler	(g)	Water (g)	General notes about funnel test which describe the workability
7	NHL5	45	-	-	Lime-stone powder	85	82	Without additives grout is not injectable.
8	NHL5	45	Sodium gluconate	0,18	Lime-stone powder	85	82	Better fluidity than in previous test but still not enough for successful injection.
9	NHL5	45	Sodium gluconate	0,9	Lime-stone powder	85	82	Flowing in continuous line. Less grout stays on the surface of the funnel.

10	NHL5	45	3% Tylose MH 300	4,5	Limestone powder	85	77,5	Grout is only dripping through the funnel but has better fluidity than the grout no 11.
11	NHL5	45	3% K 9	4,5	Limestone powder	85	77,5	Addition of K9 decreased the fluidity. Grout was difficult to pull into syringe and it stuck inside the funnel.
12	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5	Grout had considerably good fluidity. Consistency like honey.
			3% Tylose MH 300	4,5				
13	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5	Flowing in continuous line but quite a lot of grout stuck in the funnel.
			3% K 9	4,5				
14	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5	Consistency like honey, and good workability.
			5% Tylose MH 300	4,5				
15	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5	A lot of grout was stuck in the funnel.
			5,26% K 9	4,5				
16	NHL5	45	Melment F10	0,9	Limestone powder	85	82	Grout was very liquid and quick sedimentation of the suspension was noticed.
17	NHL5	45	Melment F10	0,9	Limestone powder	85	77,5	Grout was a bit too liquid and fairly quick sedimentation was noticed.
			3% Tylose MH 300	4,5				
18	NHL5	45	Melment F10	0,9	Limestone powder	85	77,5	Too liquid to work with. Quick sedimentation of the grout was noticed.
			3% K9	4,5				
19	NHL5	45	Melment F10	0,18	Limestone powder	85	77,5	Grout is flowing in continuous line.
			5% Tylose MH 300	4,5				
20	NHL5	45	Melment F10	0,18	Limestone powder	85	77,5	Grout is thicker than the previous mixture (grout no. 19).
			5% K 9	4,5				
21	NHL5	45	5% Tylose MH 300	4,5	Limestone powder	85	73	Grout is thick and not fluid. Was not possible to suck in the syringe.
			5% K 9	4,5				
22	NHL5	45	Melment F10	0,18	Limestone powder	85	73	Compared to the previous grout the fluidity is much

			5% Tylose MH 300	4,5				better.
			5% K 9	4,5				
23	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5	Grout is flowing in continuous line.
			50,5% K9	4,5				
24	NHL5	45	Melment F10	0,18	Limestone powder	85	75,25	Grout is only dripping through the funnel.
			5% Tylose MH 300	6,75				
25	NHL5	45	Melment F10	0,18	Limestone powder	85	73	Better fluidity than on previous grout. Water retention is increasing.
			5% Tylose MH 300	9				
26	NHL5	45	Melment F10	0,18	Limestone powder	85	70,75	Good fluidity and similar water retention to Ledan TA1.
			5% Tylose MH 300	11,25				
28	NHL5	45	Melment F10	0,18	Limestone powder	85	62,75	Similar fluidity to the grout no 26. Only the time of dripping was higher in funnel test (probably the influence of K9).
			5% Tylose MH 300	11,25				
			5% K 9	8				
29	NHL5	45	Melment F10	0,18	Limestone powder	85	74	Grout is only dripping through the funnel and is difficult to pull into the syringe.
			5% K 9	8				
30	NHL5	45	Melment F10	0,18	Poraver 0,1-0,3 mm	40	62,75	-
			5% Tylose MH 300	11,25				
			5% K 9	8				
31	NHL5	45	Melment F10	0,18	Poraver 0,1-0,3 mm	50	70,75	Has shorter time of flow than the same grout with limestone powder as filler (no 26).
			5% Tylose MH 300	11,25				
32	NHL5	45	Melment F10	0,18	Liaver 0,1-0,3 mm	50	70,75	Slight sedimentation of the filler – bigger particles float on the top. Has longer flow time than the same grout with Poraver as filler.
			5% Tylose MH 300	11,25				
33	NHL5	45	Melment F10	0,18	Fillite SGHA	50	70,75	-

			5% Tylose MH 300	11,25				
34	NHL5	45	Melment F10	0,18	Poraver 0,1-0,3 mm	50	62,75	-
			5% Tylose MH 300	11,25				
			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	Grout had higher viscosity than the grout without K9 (no 32).
			5% Tylose MH 300	11,25				
			5% K 9	8				
38	NHL5	45	Melment F10	0,18	Fillite 160 W	38	50,75	Grout is only dripping through the funnel.
			5% Tylose MH 300	11,25				
			5% K 9	8				
39	NHL5	45	Melment F10	0,18	Limestone powder	28,33	50,75	Grout is only dripping and has the longest measured average time in funnel test.
			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	Grout is flowing in drips out of the funnel.
			5% Tylose MH 300	11,25	Liaver 0,1-0,3 mm	23,74		
			5% K 9	8				
41	NHL5	45	Melment F10	0,18	Limestone powder	28,33	50,75	Grout is flowing in drips out of the funnel.
			5% Tylose MH 300	11,25	Fillite 160 W	25,26		
			5% K 9	8				

* Test results of the grouts marked with the same colour can be compared.

5.4 Preparation of the grouts for laboratory testing

5.4.1 The mixing procedure

The mixing procedure for the grouts was as follows:

1. Water was measured and mixed with additives in a magnetic stirrer for about 3 minutes at 250rpm.
2. The dry ingredients were measured and added to the liquid.
3. The mixture of all ingredients was hand mixed with a spoon for 10 to 15 seconds to prevent the creation of lumps, and then placed in the magnetic stirrer for 5 min at 250rpm. The temperature on the mixer was set to 20ºC, but because of the influence of the indoor climate and the increasing warmth of the mixer as it functioned, the actual temperature was between 23-30ºC.

5.4.2 Casting of the grouts

Grouts were cast in high-density fibreboard (HDF) moulds with dimensions of 40x40x40 mm and 160x40x20 mm, and allowed to cure for 7 days in high relative humidity conditions (T= 15 ±1°C; RH= 91 ±1%). Then the specimens were removed from the casts or transferred with the casts to laboratory conditions with lower relative humidity (T= 19 ±1°C; RH= 73 ±3%), for another 21 days.

5.4.2.1 Problems with the removal of the grouts from the moulds

Problems occurred with the removal of custom from the high-density fibreboard moulds. Fibreboards contain Urea-formaldehyde resin, a polar polymer with no hydrophobic properties. So materials like modified lime mortars can potentially adhere on the surface of the HDF boards.[61] As a result, the grouting mixtures adhered to the surface of the casts, and shrinkage cracks formed throughout the specimens (see ill. 33). It was not possible to open the casts without breaking the samples. To solve the problem within the limited time available, moulds were covered with silan/siloxane solution (Funcosil SNL, Remmers). The intention was to create a hydrophobic coating to prevent mortars from adhering to the HDF casts. Unfortunately, this method did not work, and it

[61] E-mail consultation with MSc Karol Bayer, University of Pardubice, (08.04.2019).

was not possible to remove the samples from the moulds in a single piece. In order to perform the necessary tests, some samples were resized from the larger surviving pieces. This meant that compression strength was measured with the specimens in sizes of 31x35x25 mm, instead of 40x40x40 mm^3 as recommended by the European standard.[62]

33. Example of the shrinkage cracks which formed in the moulded grouts.

5.4.3 Solvent exchange drying method for the cured grout specimens

5.4.3.1 The aim

The aim of the solvent exchange treatment was to stop hydration in grout samples by replacing water in the sample pores with solvent which could later evaporate. The hydration was stopped after grout specimens had cured for 28 days. The aim was to obtain the precise degree of hydration necessary to be able to carry out all the necessary measurements over a longer period of time.[63]

5.4.3.2 Description of the test

1. Samples were placed in a plastic container, soaked in isopropyl alcohol (C_3H_8O), and tightly sealed for two days.

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[62] Methods of test for mortar for masonry. Determination of flexural and compressive strength of hardened mortar. European standard: EN 1015-11:1999

[63] J. Zhang, G. W. Scherer, Comparison of Methods for Arresting Hydration of Cement, Cement and Concrete Research 41, 2011, p. 1024.

2. 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.
3. The dried samples were wrapped in plastic and stored in airtight containers to minimise carbonisation.

Note:

- The hardening of natural hydraulic lime is a combination of hydration and carbonisation.

6. Test methods for the 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.

6.1 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.

6.1.1 Funnel test

6.1.1.1 The aim

The funnel test compared the fluidity of different grouting mixtures, and analyzed the influence 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.

6.1.1.2 Equipment and materials:

- Glass funnel with the tube diameter of about 7 mm
- Tripod to support the funnel
- Two glass containers to mix and collect the grout
- Magnetic stirrer
- Syringe of 150 ml
- Stopwatch for measuring the time of flow and dripping
- Citric acid solution to clean the funnel from lime between the tests
- Camera

6.1.1.3 Test Procedures

1. 100 ml of grout was injected into the glass funnel – not directly inside the opening of the funnel but to the side of it to decrease the influence of the application to the speed of the flow.
2. The time of flow and/or dripping were measured. A stopwatch was used to measure the time between the moment when the grout reached the tip of the funnel tube and when the grout finished flowing and/or dripping through the funnel.
3. 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.
   - For a more precise result of the funnel test, measure the weight of the funnel before and after the process to note the amount of grout which adhered to the surface.
4. Tests were recorded in video to be able to observe again if necessary.

6.1.1.4 Notes

1. The grout was prepared immediately before each cycle of testing.
2. The glass funnel used for tests was not standardised, therefore the same funnel was used for all the tests to maintain consistent measurements.
3. After each test, the funnel was washed in citric acid solution to remove the traces of lime from the glass which could affect the speed of flow.
4. Tests were repeated four times with each grout, and the measurements were averaged.

6.1.2 Filter paper test

6.1.2.1 The aim

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.

6.1.2.2 Equipment:

- Stopwatch
- Filter papers with a diameter of 150 mm
- Flat glass dishes to place under the filter papers
- Syringe of 50 ml
- Camera

6.1.2.3 Test Procedures

1. 5 ml of grout was injected in the middle of filter paper.
2. A stopwatch was used to track the time.
3. The release of water out of the grout was documented by taking a photo at intervals of 1, 5, 10, 15, and 20 minutes.
4. Photos taken during the test were used to evaluate the amount of water released into the filter paper by measuring the wet area of filter paper. This was done with the ImageJ image processing program.[64]

6.1.2.4 Note

1. Tests were simultaneously performed on two filter papers to see if there were differences in the test results.

6.1.3 Flow on plastered panel

6.1.3.1 The aim

This test is an adaptation of the test described in the Getty manual on pages 75-77.[65] 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.

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[64] ImageJ, An open platform for scientific image analysis, https://imagej.net/Welcome (accessed 1st November 2018).

[65] B. Biçer-Şimşir, L. H. Rainer, Evaluation of Lime-Based, pp. 75-77.

6.1.3.2 Preparation of the plastered panel

For this test, imitation mortar number 8 was chosen to cover the support.

34. Formula for the imitation mortar.

Binder	Natural Hydraulic Lime (NHL 5)	250 g
Aggregrate (sand from Czech Republic and crushed marble)	Sand: • Strelec sand pit (0 - 0,125 mm) - 12 wt % • Kostelecke Horky sand pit (0-2 mm) - 88 wt % Crushed marble (0-2 mm) - 20 wt %	200 g 50 g
Liquid	Regular tap water*	100 g

** To cover the panels, the amount of water in the mortar was reduced from the original 200g to 100g.*

1. Aeroc building blocks, cut to 250 x 300 x 50 mm were used as a support.
   - Before the plaster application, panels were soaked in water overnight.
   - The next day, panels were covered with 25 mm thick layers of mortar.
2. Panels were cured 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%).
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.
4. After the channels were cut, dust was removed from the gaps with compressed air.

6.1.3.3 Used equipment and materials:

- Support panels for the plaster
- Syringe of 10 ml
- Trowels
- Hand saw
- Bowl for mixing the grout
- Drill mixer
- Lime mortar
- Camera

6.1.3.4 Test Procedures

1. The panel was placed vertically on the table, and 5 ml of grout was injected into the top of the channel.
   - Injection was done slowly so that the grout could flow slowly inside the gap. If done too quickly, there was a likely chance that the grout would be pushed out of the gap by the pressure, disrupting the test measurements.
   - Injection speed was measured with a stopwatch.
2. To check the influence of the pre-wetting on the speed of flow, 10 ml of regular tap water was injected in the channel immediately before the injection of the grout. This was done with the three selected grouts only - numbers 28, 39 and 40.
3. At the end of the test, the length of the flow was measured, and averages were calculated from three results.
4. Results were characterised by the length of flow, and were divided into 5 groups.

6.1.3.5 Note

1. The test was performed only with the ten selected grouts - numbers 3, 5, 6, 26, 28, 29, 36, 37, 39, and 40.

6.2 Properties during curing

6.2.1 Shrinkage in plaster cylinder

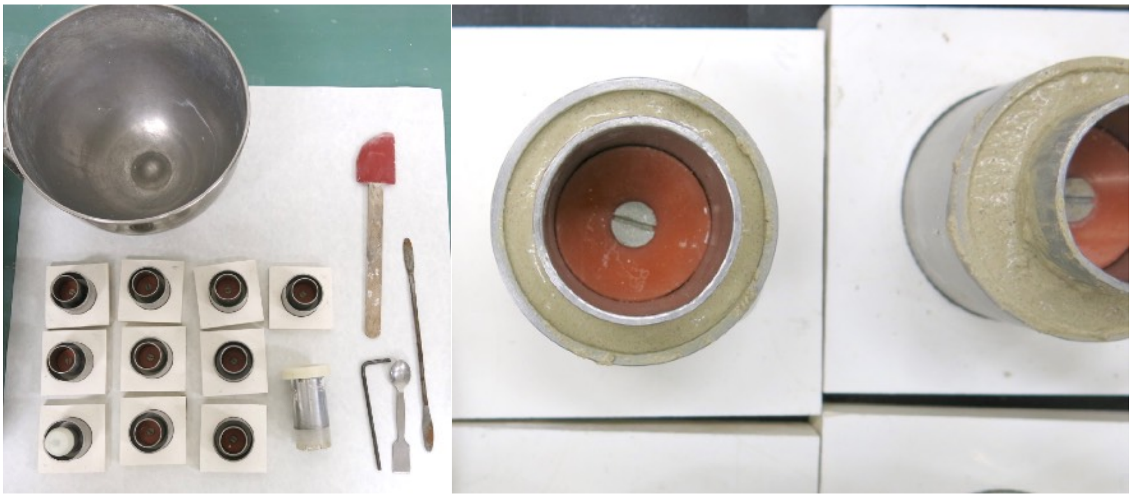
6.2.1.1 The aim

This experiment was done to observe the volume shrinkage of the grouts, as well as the adhesion between the cylinder-shaped plaster specimen and the grouts.

6.2.1.2 Preparation of cylinder specimens

1. Fresh mortar 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 (see ill. 35).

35. Casting of the mortar.



2. 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 specimens were 40x40x5 mm (see ill. 36).

36. Coarsened inner surface and the cured cylinders.

3. Two cylinders were glued together with the mixture of NHL 5 and water (see ill. 37).

37. Cylinders glued together.

4. Two cylinders were used for each grout. After 21 days of curing, the specimens were cut in half to evaluate shrinkage (see ill. 38).

38. Casting the grouts inside the cylinders. The cured grout in the cylinder cut in halves.

6.2.1.3 Test Procedures

5. Cylinder shaped plaster specimens were prepared out of imitation mortar.
   - The diameter of the cylinder casts was 40x40x5 mm.
   - To have 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.
6. Each grout was injected into two cylinders.
7. For the first seven days, cylinder specimens were kept in high humidity conditions (T=15 ±1°C; RH= 91 ±1%), and next 21 days in laboratory conditions T= 19 ±1°C; RH= 73 ±3%.
8. After the grouts had cured for 21 days, the cylinders were cut in half with a hand saw to examine the shrinkage.
9. Specimens were documented by taking photos from the top, bottom, and after cutting in half.
10. Test results were visually evaluated.
11. Adhesion between the cylinder and the grout was examined with the optical and scanning electron microscope (SEM).

Note:

- The same SEM samples were used to observe the cohesion of the filler and the matrix.

6.3.1 Capillary water absorption measurement

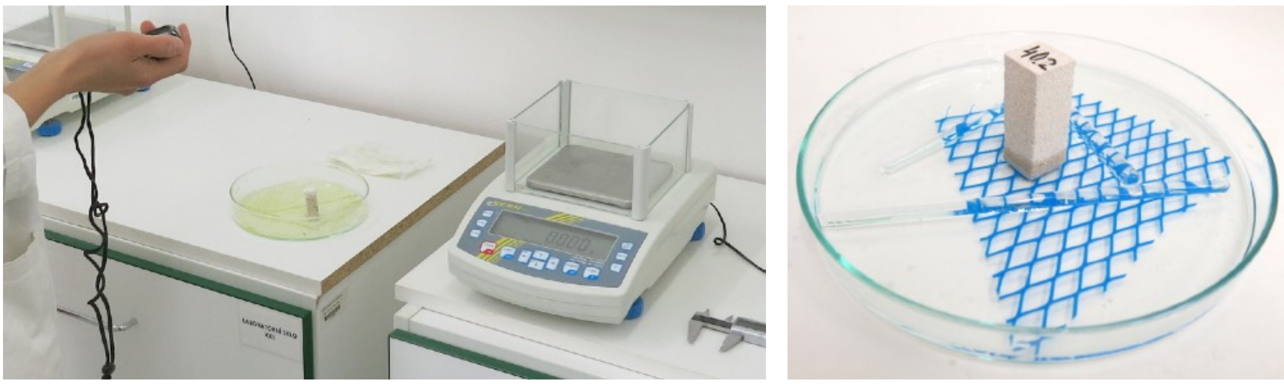
6.3.1.1 The aim

The aim of this test was to evaluate the porosity of the injection grouts by measuring water absorption. At the end of the test, the water absorption coefficient was calculated to compare the results with the historic plaster samples.

6.3.1.2 Test Procedures

1. The bottom of the 150 mm petri dish was covered with a piece of plastic net to lift the sample above the bottom of the dish.
2. Distilled water was poured in the petri dish, to about 3 mm above the plastic net.
   - The exact level of the water was measured with the plastic calliper.
3. 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.
4. The mass gain of the sample was measured every minute.
   - The maximum duration of the test was 10 minutes. The time of measurements depended on the absorption speed and the size of the sample.
5. Before the sample was put on the scale to measure the gain in weight, the wet side was wiped on moist paper tissue to eliminate extra drips of water.
6. The water absorption coefficient was calculated and recorded.

39. Capillary water absorption measurement of a grout sample.



6.3.2 Ultrasonic pulse velocity (UPV) measurements

Ultrasonic pulse velocity method measures the time of travel of an ultrasonic pulse passing through the tested samples.[66] This method has been widely used for the condition assessment of concrete structures but also for cultural heritage objects.

6.3.2.1 The aim

The ultrasonic pulse velocity test measured elastic modulus, velocity propagation, and density of the historic mortar, and also of the injection grouts. Results were used to compare the mechanical properties of the tested materials.

6.3.2.3 Test Procedures

1. 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.
2. 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.
3. All the samples were cut to the standard size - 15 x 15 x 40 mm.
4. The exact length, diameter, and weight of the samples were measured.

6.3.2.4 Notes

1. Measurements were done according to the recommendations of the European standard *Non-Destructive testing - Ultrasonic testing - Transmission technique* (EN ISO 16823: 2014)
2. The sizes of the grout samples were chosen according to the sizes of the historic plaster samples.

6.3.3 Compression strength

6.3.3.1 The aim

The aim was to measure the compressive strength values for the comparison of different grouting mixtures and their compatibility with the imitation mortar.

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[66] Ultrasonic Pulse Velocity Method, https://www.printfriendly.com/p/g/FxbtNZ (accessed 1st April 2019).

6.3.3.2 Test Procedures

1. Samples had cured 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.
2. Cured samples were cut into dimensions of 35x31x25 mm.
3. Measurements were done with the device FPZ100 (max. load 100 kN, crosshead speed 0,63 mm × min-1), 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).

Notes:

1. The size of the tested samples was irregular due to problems which occurred with the removal of the grouts from the moulds. The standard size of the samples according to the reference manual should be 40x40x40 mm^3.
2. However, measurements done with the non-standard samples were still suitable for the comparison of different grouts tested in this research.

6.3.4 Adhesion test

6.3.4.1 The aim

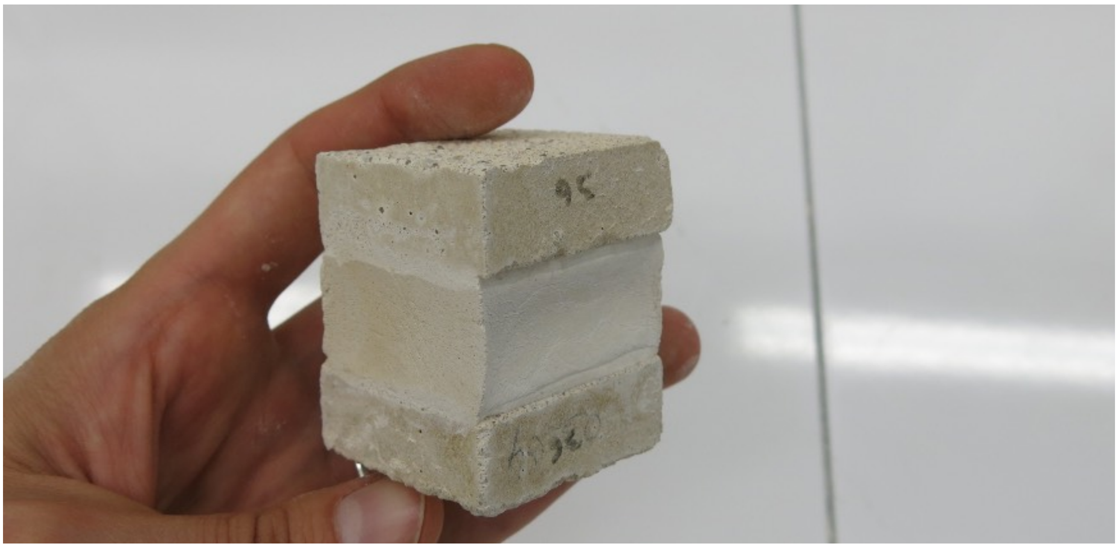
The aim of the adhesion test was to measure the tensile strength of the selected injection grouts.

6.3.4.2 Test procedures

1. Beforehand the test specimens in size of 40x40x20 mm were prepared from the imitation mortar.
2. One side of the specimen was coarsened with the sand paper number P 180.
3. Liquid grout was casted between the two specimens. Procedure was repeated two times to each grout (see ill. 40).
4. Cast samples cured 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.
5. After 28 days solvent exchange drying method was applied to stop the hydration in the specimens.

6. Specimens were stored in air-tight conditions until the measurements were carried out.
7. 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.

40. Prepared test specimen for the adhesion test. Injection grout between two pieces of imitation mortar.



6.3.5 Optical and scanning electron microscope (SEM) analysis

Small samples were taken from the cylinder specimens filled with hardened grout to examine the structure of the matrix, its bond with the filler and the bond between the cylinder and the grout.

7. Test results

7.1 Evaluation system for the test results

Marks from 0 to 4 were given to each grout to measure their performance level in the experiments.

- In the **funnel test** the lowest marks were given to the grouts with the lowest viscosity, and the highest marks to the high viscosity grouts (see ill. 41).

41. Specification of the evaluation marks for funnel test.

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

- In the **filter paper test**, the lowest marks were given to the grouts with the best water retention, observed as having the smallest measured area of water release, recorded across the following range (see ill. 42):

42. Specification of the evaluation marks for the filter paper test.

0	1	2	3	4
432,3–892,4 mm²	947,7–1871,6 mm²	2468,8–3295,6 mm²	3438,1– 4434,7 mm²	5180,8–7219,8 mm²

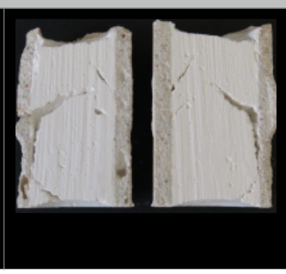
- In the **flow on plastered panel test**, the lowest marks were given to the grouts which flowed the longest distance (see ill. 43).

43. Specification of the evaluation marks for the flow on plastered panel test.

0	1	2	3	4
28-28,5 cm	26,7 cm	24,5 - 25,2 cm	21,8 - 22,7 cm	10,7 cm

- In the **shrinkage in cylinder test**, the lowest marks were given to the grouts with the least shrinkage cracks (see ill. 44).

44. Specification of the evaluation marks for the shrinkage in cylinder test.

0	1	2	3	4
No grout was given this mark				

- For the experiments measuring the **properties of the hardened grouts** (e-modulus, VP, density, compressive strength), the lowest mark was given to the result with the highest value (see ill. 45).

45. Specification of the evaluation marks for the UPV and the compressive strength measurements.

	0	**1**	**2**	**3**	**4**
Elastic modulus (kN/mm^2)	5,760	4,717	1,803 - 2,120	1,614 - 1,633	1,327
VP (km/s)	2,17 - 2,29	1,53 - 1,64	1,50	1,37 - 1,38	1,22
Density (g/cm^3)	1,282 - 1,372	1,159 - 1,163	1,091 - 1,119	0,802 - 0,825	0,634 - 0,680
Compressive strength (MPa)	5,4 - 6,2	1,6	1,3 - 1,4	0,9	0,5 - 0,7

The ten grouts chosen for further testing after the first two experiments are marked with a light orange colour in the "summary of marks" cell in table 46. 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.

The only grout which showed poor properties, observed already in the funnel test, but which was still selected for the further testing was number 29, since it contained acrylic emulsion K9. The aim was to examine the influence of the acrylic emulsion K9 to the working, setting, and hardened properties of the grout. Later it was possible to compare the results with the grouts containing methyl hydroxyethyl cellulose Tylose MH 300.

46. Evaluation marks for all the tested commercial and custom made grouts.

Marks for working properties and properties during curing						**Marks for properties of hardened grouts**				
Grout no.	Funnel test	Filter paper test	**Sum. of marks**	Flow on plastered panel	Shrinkage in cylinder	WAC	E-modulus	VP	Density	Comp-ressive strength
1	0	3	3	nm	nm	nm	nm	nm	nm	nm
2	4	1	5	nm	nm	nm	nm	nm	nm	nm
3	1	0	1	1	2	1	1	0	0	0

Marks for working properties and properties during curing						Marks for properties of hardened grouts				
Grout no.	Funnel test	Filter paper test	**Sum. of marks**	Flow on plastered panel	Shrinkage in cylinder	WAC	E-modulus	VP	Density	Comp-ressive strength
4	0	2	2	nm	nm	nm	nm	nm	nm	nm
5	1	2	3	0	2	0	0	0	0	0
6	0	2	2	2	1	3	2	3	1	1
7	3	4	7	nm	nm	nm	nm	nm	nm	nm
8	2	4	6	nm	nm	nm	nm	nm	nm	nm
9	0	4	4	nm	nm	nm	nm	nm	nm	nm
10	3	4	7	nm	nm	nm	nm	nm	nm	nm
11	4	4	8	nm	nm	nm	nm	nm	nm	nm
12	0	2	2	nm	nm	nm	nm	nm	nm	nm
13	0	4	4	nm	nm	nm	nm	nm	nm	nm
14	0	2	2	nm	nm	nm	nm	nm	nm	nm
15	0	4	4	nm	nm	nm	nm	nm	nm	nm
16	0	4	4	nm	nm	nm	nm	nm	nm	nm
17	0	3	3	nm	nm	nm	nm	nm	nm	nm
18	0	4	4	nm	nm	nm	nm	nm	nm	nm
19	0	3	3	nm	nm	nm	nm	nm	nm	nm
20	0	4	4	nm	nm	nm	nm	nm	nm	nm
21	4	2	6	nm	nm	nm	nm	nm	nm	nm
22	0	2	2	nm	nm	nm	nm	nm	nm	nm
23	0	3	3	nm	nm	nm	nm	nm	nm	nm
24		2		nm	nm	nm	nm	nm	nm	nm
25		1		nm	nm	nm	nm	nm	nm	nm
26	1	0	1	3	3	2	2	3	1	3
27	4	nm	-	nm	nm	nm	nm	nm	nm	nm
28	1	1	2	0	3	2	2	2	2	3
29	3	4	7	4	4	4	4	4	2	4
31	1	nm	-	nm	nm	nm	nm	nm	nm	nm
32	1	0	1	nm	nm	nm	nm	nm	nm	nm
33	1	1	2	nm	nm	nm	nm	nm	nm	nm

Marks for working properties and properties during curing						Marks for properties of hardened grouts				
Grout no.	Funnel test	Filter paper test	**Sum. of marks**	Flow on plastered panel	Shrinkage in cylinder	WAC	E-modulus	VP	Density	Comp-ressive strength
34	1	1	2	nm	nm	nm	nm	nm	nm	nm
36	3	0	3	2	2	1	3	1	4	4
37	2	0	2	3	2	2	4	1	4	3
38	3	0	3	nm	3	nm	nm	nm	nm	nm
39	3	0	3	0	2	2	2	1	3	2
40	2	0	2	0	2	1	3	1	3	2
41	2	0	2	nm	3	nm	nm	nm	nm	nm

** nm- not measured*

7.2 The results and evaluation of the funnel test

The funnel test measured the fluidity of the grouts, and allowed a comparison of the influence of different additives and filler combinations by measuring the speed of flow. The main characteristics measured by the funnel test are:

- The speed of flow through the funnel (see ill. 47)
- The consistency and sedimentation of the grout by visual examination
- The influence of the various additives and fillers on the speed of flow (see ill. 47)
- The amount of grout which adhered to the funnel by visual examination (see ill. 49; 50; 51)

The first phase of tests examined fluidity, adding as little water as necessary to the mixture in order to design a grout which is suitable for injection into larger voids (>2 cm) with minimal volume shrinkage. Grouts number 7 to 41 are custom made grouts. Test results showed that the fluidity was the most influenced by the fluidizers, as well other admixtures used in the custom-made grout recipes, which are acrylic emulsion K9, and methyl hydroxyethyl cellulose Tylose MH 300.

Acrylic emulsion K9 had a negative effect on the fluidity. It made the grout mixtures thick and did not enhance the working properties. This was especially the case if K9 was used as the only additive, but also if combined with the fluidizer. Looking at the results of grouts number 11 and 13, number 11, which contained only K9 as an additive, got stuck inside the funnel and failed the test. Number 13, with the same amount of K9 and the addition of sodium gluconate, performed well in the test, and was classified as very fluid (see ill. 48). Different amounts and concentrations of K9 were tested, starting from the 10 wt% of K9 to the binder in concentration of 3%, and up to 17,7 wt% of K9 in concentration of 5%. The negative effect of K9 on the fluidity was more apparent if the concentration of additive was higher. For example grout number 29 (see ill. 48).

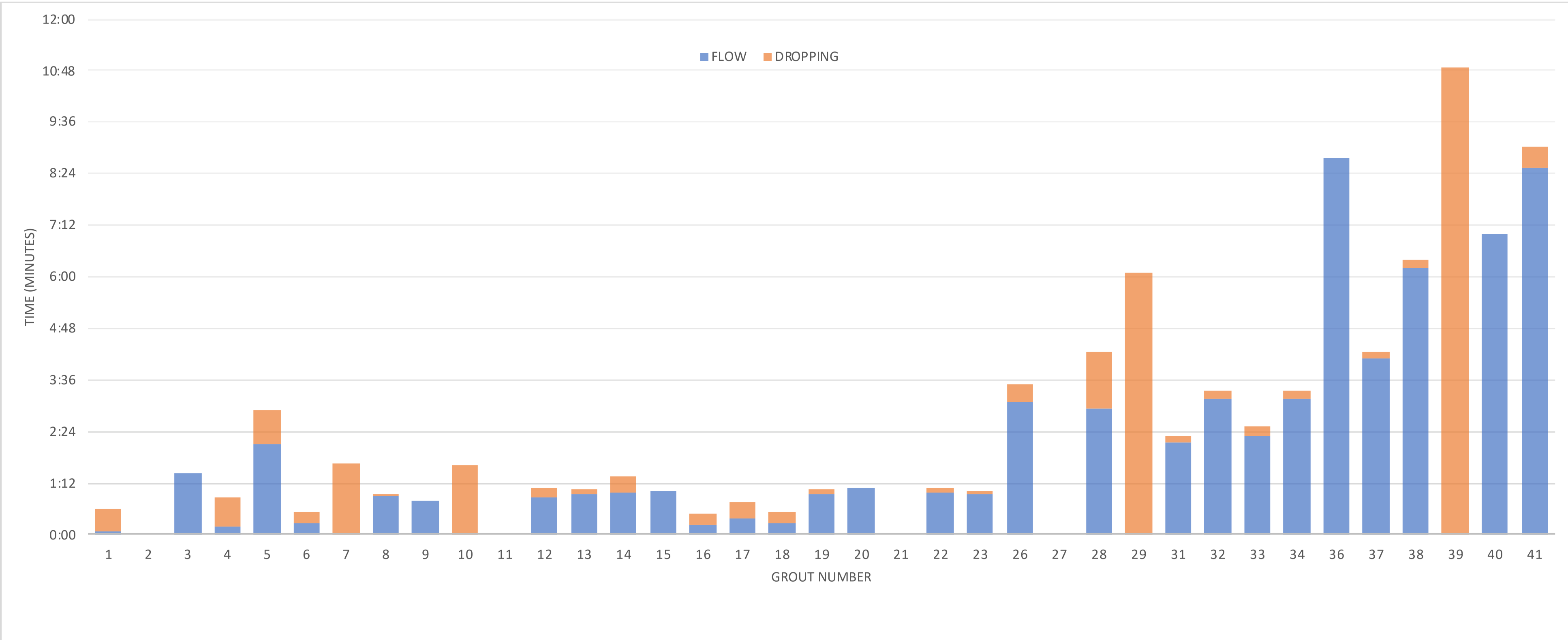
The second additive used was methyl hydroxyethyl cellulose. The test results showed that the viscosity of the grouts gets noticeably higher when methyl cellulose is added to the mixtures. This can be clearly seen on the chart 47. Starting from grout number 26, 25 wt% to the binder of 5% Tylose MH 300, was added to the mixtures.

Two different fluidizers were tested. In the first test, based on the ICCROM grout, sodium gluconate was added in concentrations of 0,4 wt% to 2 wt% to the binder. The amount of 2 wt% was found to be most suitable (see grout no 9 on the ill. 47). Tests with the fluidizer Melment F10 were begun with a measure of 2 wt% of Melment added to the binder. Due to the strong effect, the amount was reduced to 0,4 wt%. I decided to keep using Melment F10 in the rest of the mixtures because of its stronger effect and better commercial availability than sodium gluconate.

7.2.1 Summary

- Fluidity of the grouts was most influenced by the fluidizers (sodium gluconate, Melment F 10), acrylic emulsion (K9), and methyl hydroxyethyl cellulose (Tylose MH 300).
- Acrylic emulsion made the grout mixtures thick, and did not enhance the working properties.
- The viscosity of the grouts increased noticeably if 25 wt% (to the binder) of Tylose MH 300 in concentration of 5% was added to the grout mixtures.
- From the two tested fluidizers, Melment F 10 was chosen due to its stronger effect. Based on the results 0,4 wt% of Melment F10 was added to the binder.

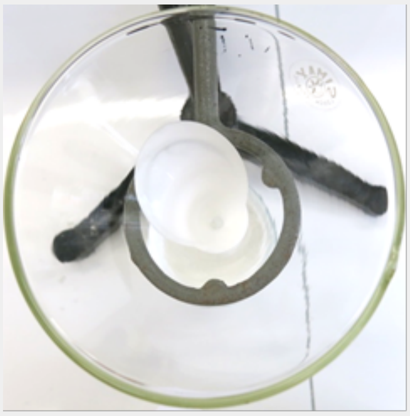
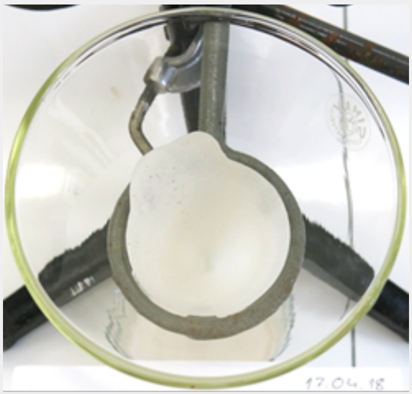
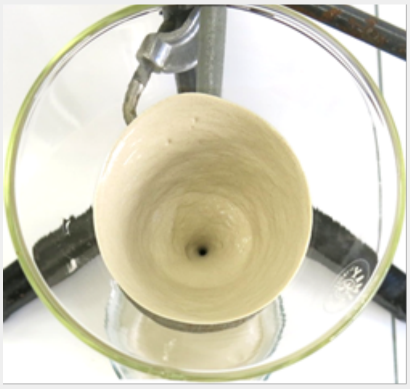
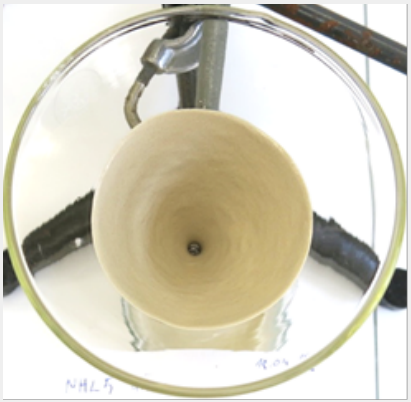
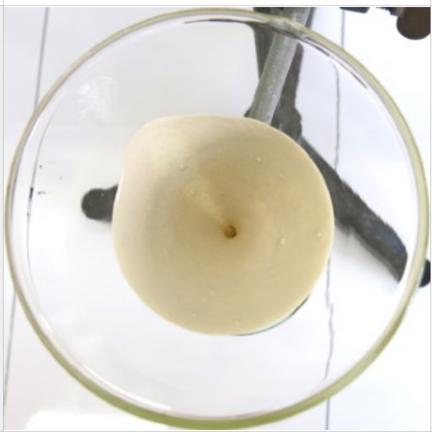
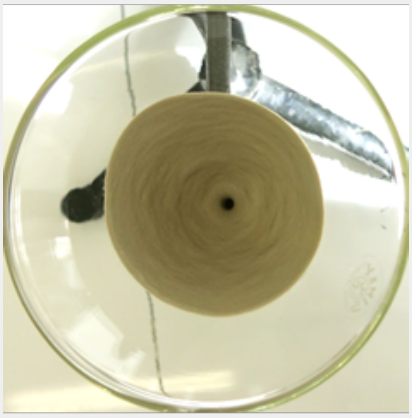
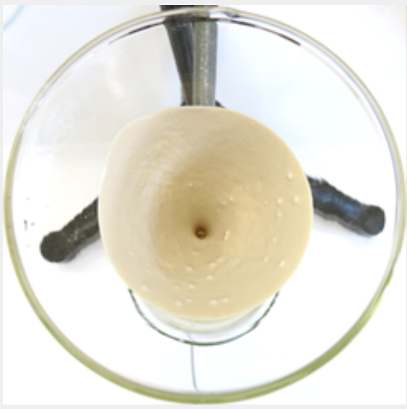
47. Fluidity of the grouts by the funnel test. Speed of flow and dripping through the funnel.

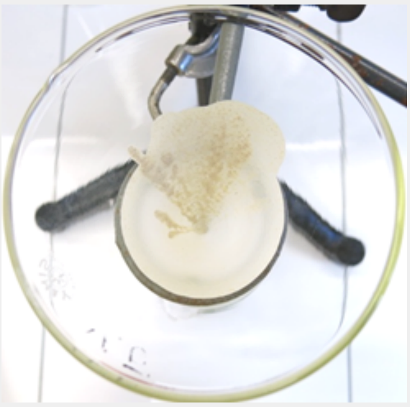
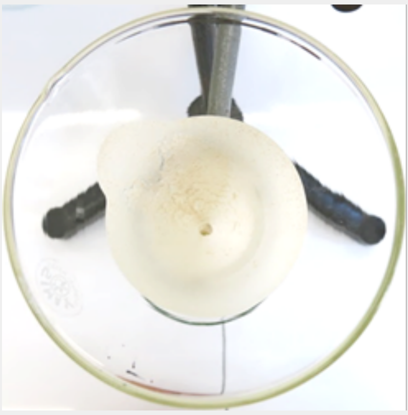
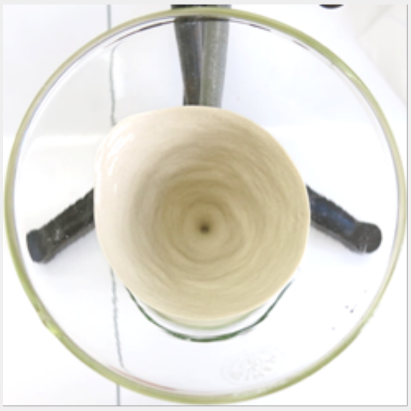
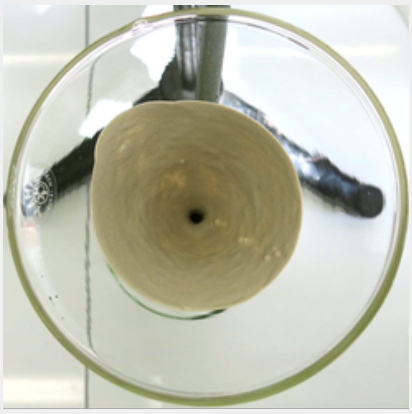
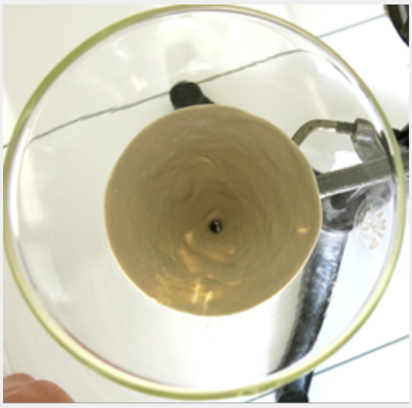


48. Evaluation system for funnel test.

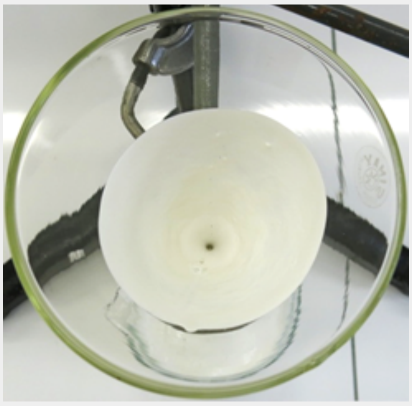
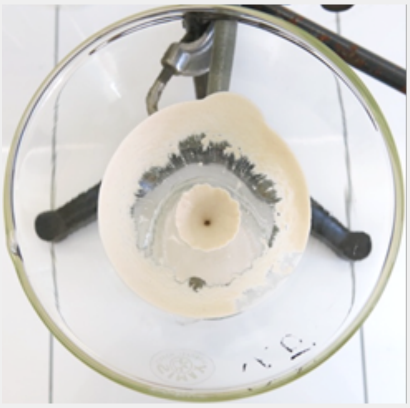
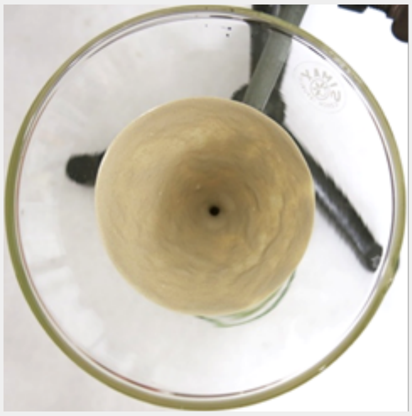
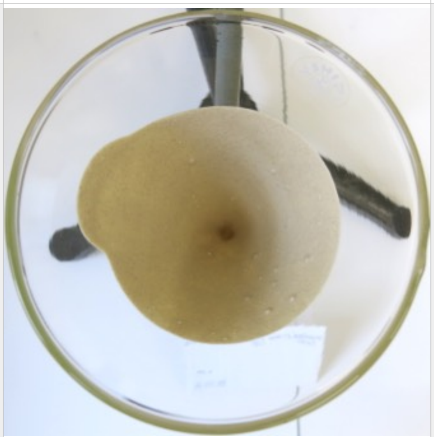
Evaluation system for funnel test									
0		**1**		**2**		**3**		**4**	
VERY FLUID 0:00 – 1:12 min		FLUID 1:12 – 3:36 min		FLOWING IN DRIPS 00:53 – 8:33 min		ONLY DRIPPING 1:40 – 10:52 min		STUCK IN THE FUNNEL 00:00 – 00:00	
Grout no.	T (min)	Grout no.	T (min)	Grout no.	T (min)	Grout no.	T (min)	Grout no.	T (min)
1	00:05	**3**	01:25	**8**	00:53	**7**	01:40	**2**	00:00
4	00:12	**5**	02:06	**37**	04:06	**10**	01:37	**11**	00:00
6	00:16	**26**	03:04	**40**	07:00	**29**	06:06	**21**	00:00
8	00:53	**28**	02:57	**41**	08:33	**36**	08:45	**27**	00:00
9	00:48	**31**	02:09			**38**	06:12		
12	00:51	**32**	03:10			**39**	10:52		
13	00:56	**33**	02:18						
14	00:58	**34**	03:10						
15	01:01								
16	00:14								
17	00:23								
18	00:15								
19	00:56								
20	01:05								
22	00:59								
23	00:56								

49. Grouts classified as **very fluid (evaluation mark: 0).** Each photo is marked with the grout number.

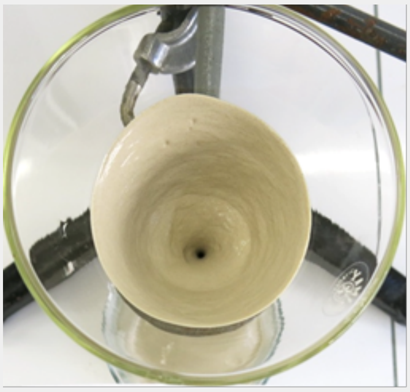
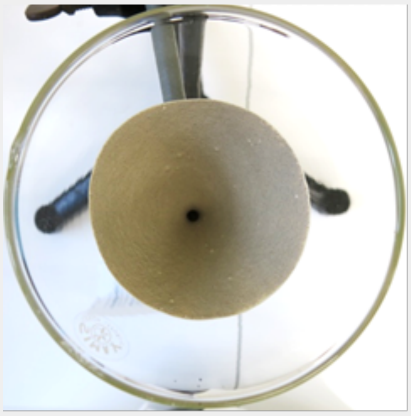
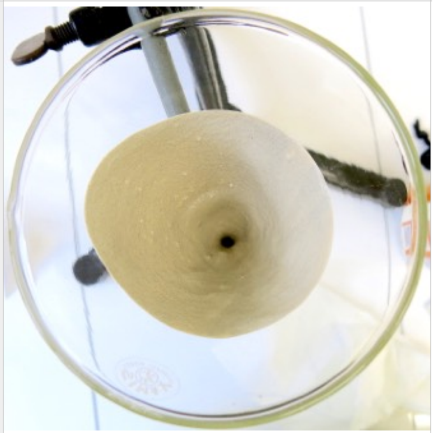
1	4	6
		
8	9	12
		
13	14	15
		

16	17	18
		
19	20	22
		
23		
	-	-

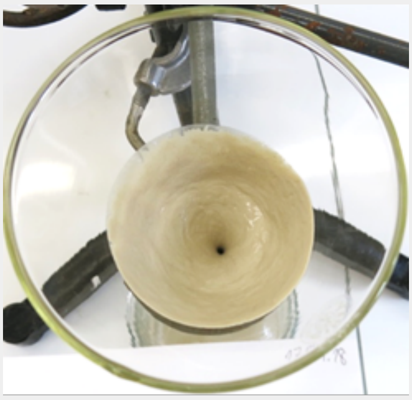
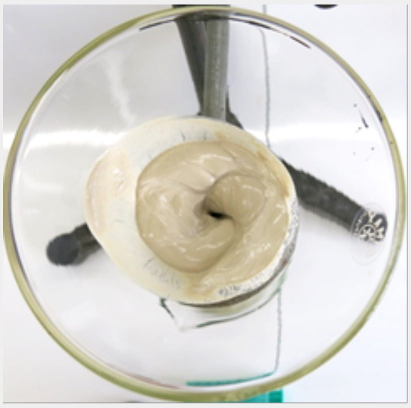
50. Grouts classified as **fluid (evaluation mark: 1)**:

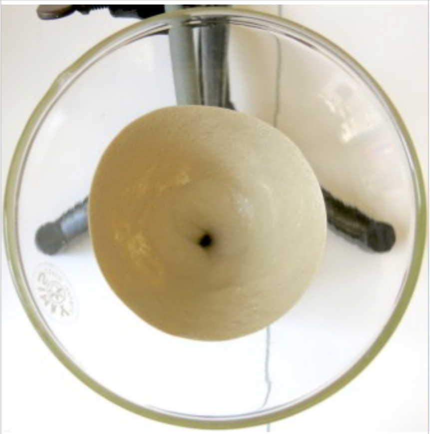
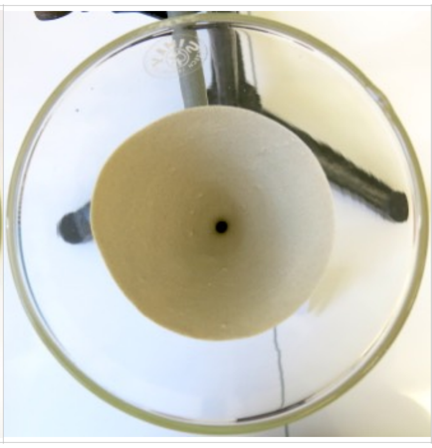
3	5	26
		
28	31	32
		
33	34	
		-

51. Grouts classified as **flowing in drips (evaluation mark: 2)**:

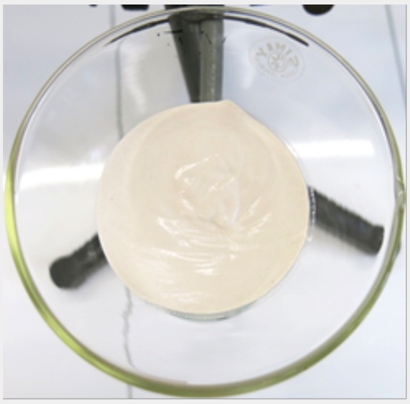
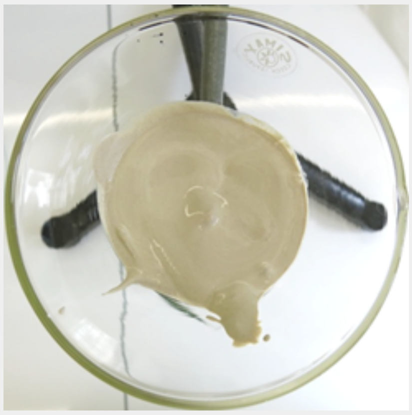
8	37	40
		
41		
	-	-

52. Grouts classified as **only dripping (evaluation mark: 3)**:

7	10	29
		
36	38	39

53. Grouts classified as **stuck in the funnel (evaluation mark: 4)**:

2	11	21
		

7.3 Results and evaluation of the filter paper test

The filter paper test results can be divided into three groups. The first group contains the grouts which had the best water retention, identified by the smallest amount of water release into the filter paper. For these grouts, the measurements of water absorption into the filter paper after 20 min were between 432,3 and 1164,2 mm². Mixtures number 26, 28, 32, 33, 34, 36, 37, 38, 39, 40, and 41 belong to this group. All of these grouts consist of 25 wt% of Tylose MH 300 to the binder in concentration of 5%. Among the commercial grouts, number 3, which contains Ledan TA1, belongs to the first group. The measurement for this grout was 795,6 mm². Grouts number 36 and 39, which both contain the lightweight filler Poraver (0,1-0,3 mm), had the lowest water release - 432,3 and 517,3 mm².

The second group contains grouts whose measurements were in the middle range of results, between 1744 and 3875,9 mm². In this group, all the custom mixtures contain Tylose MH 300 in combination with sodium gluconate, Melment F10, and K9 (grouts 12, 14, 17, 19, 21, 22, 24, 25). Five out of the six also commercial grouts belong to this range, (grouts 1, 2, 4, 5, 6).

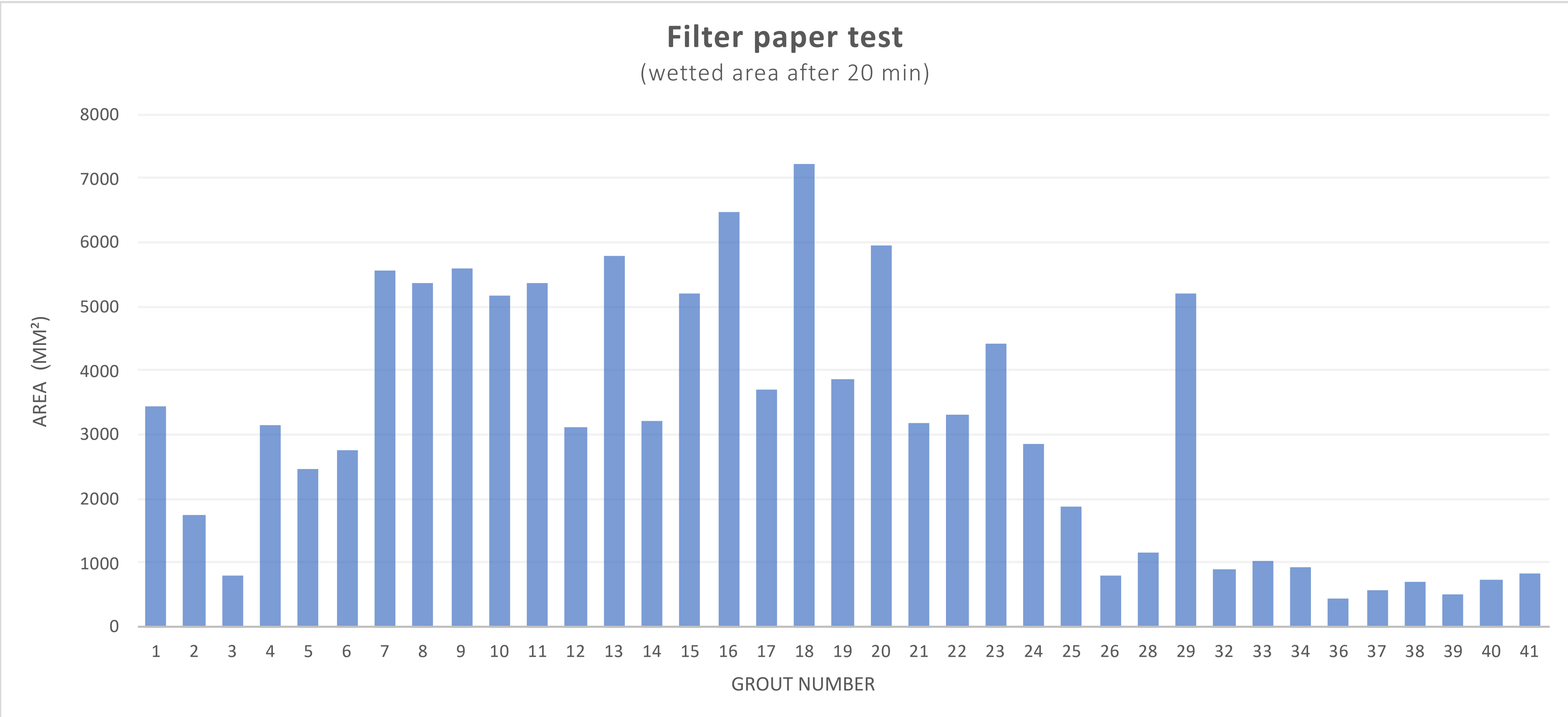
The third group contains the grouts with the highest amount of water released, from 4434,7 to 7219,8 mm². High water release in these grouts is clearly related to the use of fluidizers and acrylic emulsion K9. Exceptions are grout number 7 and 10. In the case of number 7, no additives were used, and in number 10 only Tylose MH 300 was used in low concentration (3% solution, 10 wt% to binder). Four of the grouts in this range consist of only sodium gluconate (grouts 8, 9), only Melment F10 (grout 16), or only K9 (grout 11). Grouts number 13, 15, 18, 20, 23 and 29 consisted of a combination of the fluidizer and acrylic emulsion.

For the visual test results see the appendix 1 at the pages 117 - 123.

7.3.1 Summary

- The grouts with the best water retention consisted of 25 wt% of Tylose MH 300 to the binder, in a concentration of 5%.
- High water release appeared in custom-made grouts if:
  - no additives were used
  - only fluidizer or acrylic emulsion was used
  - only methyl hydroxyethyl cellulose was used in lower concentration (3% solution, 10 wt% to binder)
  - the fluidizer and the acrylic emulsion were used together
- Slightly higher water retention occurred if higher amounts of acrylic emulsion were used (see grouts number 20 and 29 on the chart 54).
- In all cases, the water release of the grout slowed down noticeably after about 15 to 20 minutes.

54. Results of filter paper test. Comparison of water release of different grout mixtures.



7.4 Results and evaluation of the flow on plastered panel.

This test was applied to 10 selected grouts, 3 of them commercial, and 7 custom made. Results (see ill. 55) show that the maximum distance of flow (28-28,5 cm) occurred with the commercial grout number 5 and the custom made grouts number 28, 39 and 40, which all contained Tylose MH 300 (5% solution, 25 wt% to binder), K9 (5% solution, 17,8% to binder) and Melment F10 (0,4 wt% to binder).

The least fluid grout measured was number 29, with 11 cm of flow distance (see ill. 55). Additives in this mixture were Melment F10 (0,4 wt% to binder) and acrylic dispersion K9 (5% solution, 17,8% to binder). Similar to the funnel test, this result shows that the inclusion of acrylic dispersion does not improve the fluidity.

Grouts number 6, 26, 36, and 37 had medium range results. Grout number 6 is a modified commercial grout with the addition of limestone powder to Ledan TA1 (weight ratio 1:1). It had the lowest result (25 cm) compared to the two other commercial grouts, number 3 and 5. The difference in the distance could be related to the added filler, which increased the water release and therefore made the grout less fluid on the plastered panel. Number 26 measured 6 cm less in flow, compared to number 28. The only difference in the recipes was that grout 28 contained acrylic emulsion in addition to the fluidizer and methyl hydroxyethyl cellulose.

Grouts number 36 and 37 contained the same amount of additives as the mixtures with the maximum results, but included different fillers. The light weight of the Poraver and Liaver fillers seems to have influenced the distance of flow.

Notes:

1. The time spent on emptying the syringe in plastered panel test was from 7 to 35,7 seconds depending on grout's composition and properties.
2. The channels where the grouts were injected had a square shape bottom. This may have complicated the injection process. The Getty manual recommends to cut V-shaped channels. This helps the grout to flow in the gap easier, and ensures more accurate test results.

7.4.1 Summary

- Commercial grout number 5 (Vapo Injekt) showed the maximum distance of flow (28-28,5 cm). Custom grouts number 28, 39, and 40, which all contained Tylose MH 300 (5% solution, 25 wt% to binder), K9 (5% solution, 17,8% to binder) and Melment F10 (0,4 wt% to binder) performed the same result.

- The least fluid grout was number 29 (11 cm).
- Modification of the commercial product Ledan TA1 with limestone powder slightly reduced the distance of flow compared to the pure Ledan TA1.
- The use of lightweight fillers Poraver and Liaver reduced the distance of flow due to their lighter weight and coarser filler.

55. Evaluation of the flow on plastered panel test. Evaluation marks are marked with red.

3	5	6	26
1	0	2	3

28	29	36	37
0	4	2	3

<table>
<tr><th>39</th><th>40</th></tr>
<tr><td>0</td><td>0</td></tr>
<tr><td></td><td></td></tr>
<tr><td></td><td></td></tr>
<tr><td></td><td></td></tr>
</table>

7.5 Results and evaluation of shrinkage in plaster cylinder

The volume shrinkage test examined the characteristics of the shrinkage cracks formed during the setting of the injection grouts, as well as adhesion between the cylinder specimen and the grout. Results of shrinkage were evaluated only visually, but clearly observed the general shrinkage tendencies of different grouts. Adhesion between the cylinder plaster and the grout was examined with the SEM, which revealed if there are any micro cracks. The least volume shrinkage appeared on grout number 6 (see ill. 56), which also had good adhesion with the cylinder specimen. The most shrinkage was seen in grout number 29, which also had almost no adhesion to the cylinder. Among the custom made grouts, the least shrinkage cracks appeared with mixtures number 36, 37, and 40, which all contain lightweight fillers in grain size of 0,1-0,3 mm. Similar shrinkage cracks appeared in all three cases, forming straight along one side between the cylinder and the grout.

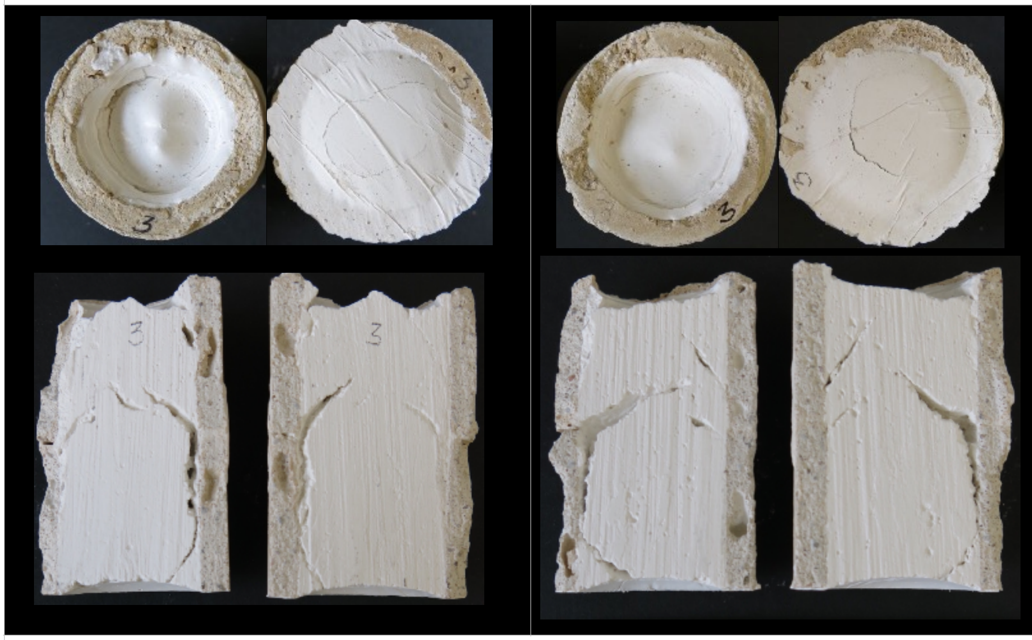
7.5.1 Summary

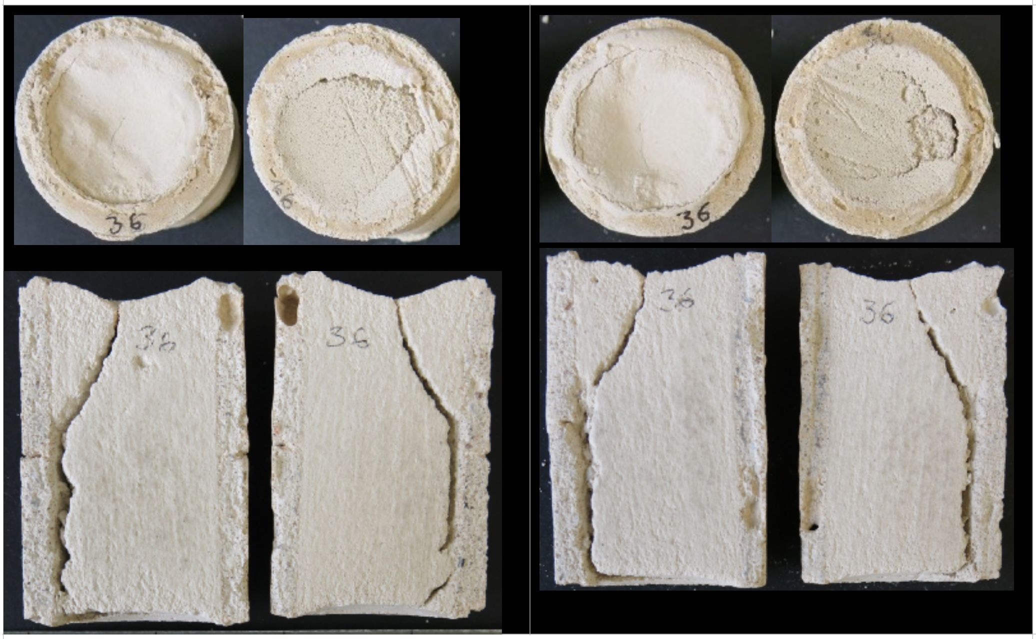
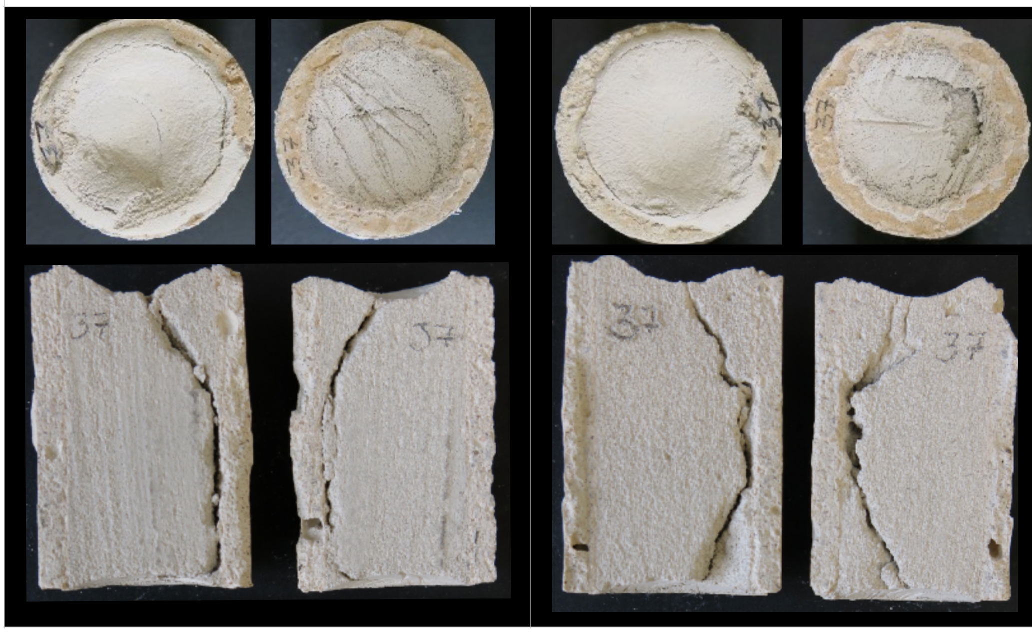
- The least volume shrinkage appeared in modified grout number 6.
- The most volume shrinkage and least adhesion to the cylinder specimen appeared in grout 29.
- All the grouts except number 29 had satisfactory results in volume shrinkage.

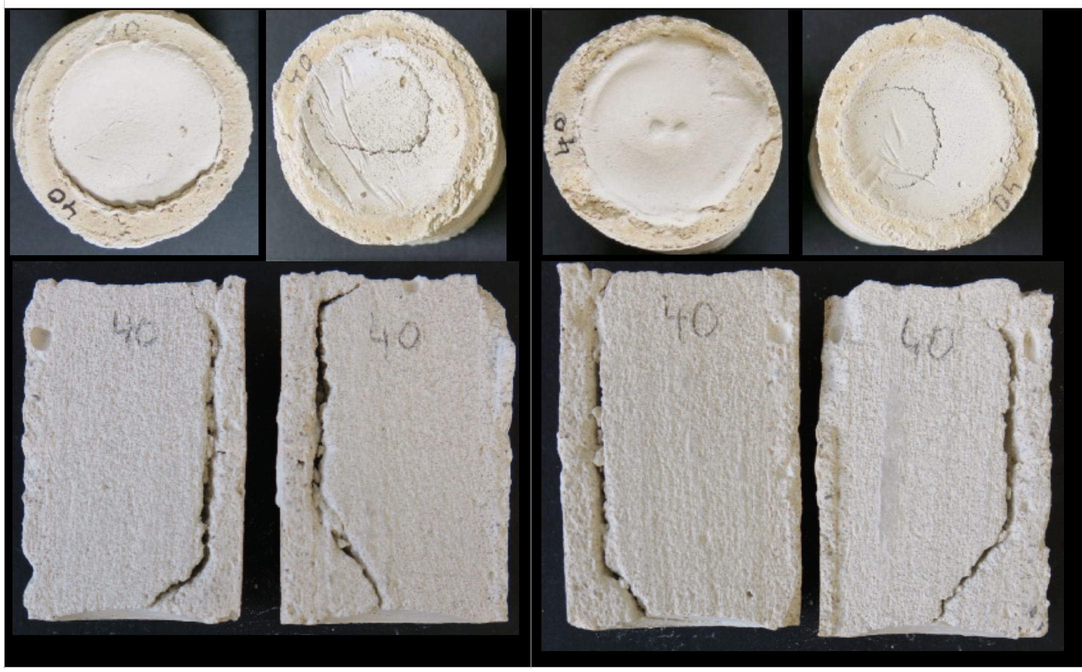
56. Shrinkage in cylinder test. Grouts with the evaluation mark number 1. Specimens are photographed after 21 days of curing, from the top, bottom and when cut in halves. Each grout was tested in two cylinders which are both presented in the table.

Grouts with evaluation mark no 1	
Grout no 6	**Notes**
	-

57. Shrinkage in cylinder test. Grouts with the evaluation mark number 2.

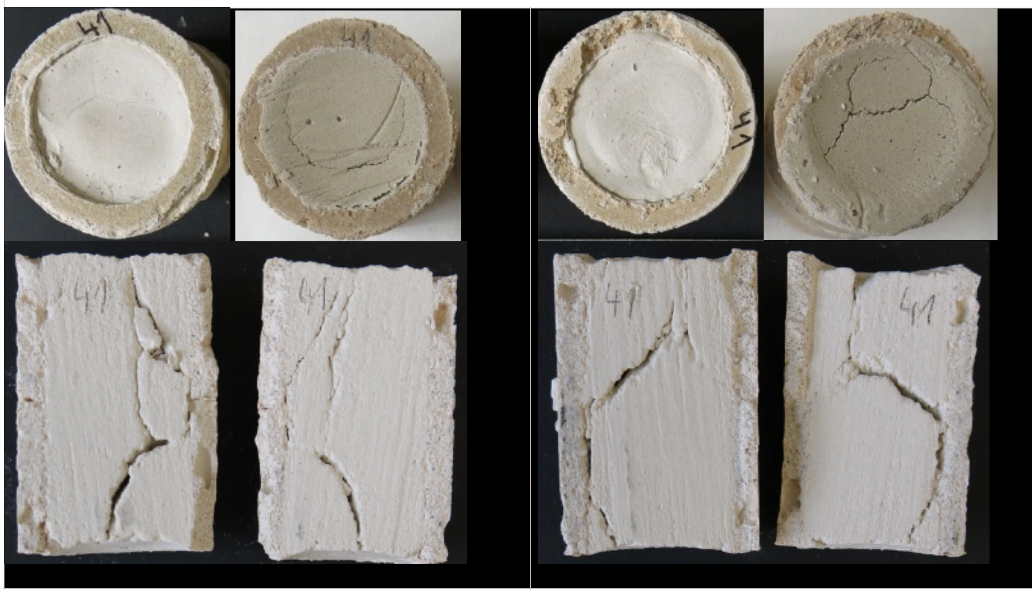
Grouts with evaluation mark no 2	
Grout no 3	**Notes**
	-
Grout no 5	**Notes**
	-

Grout no 36	Notes
	-
Grout no 37	**Notes**
	-

Grout no 39	Notes
	The specimen broke during cutting it in halves.
Grout no 40	**Notes**
	-

58. Shrinkage in cylinder test. Grouts with the evaluation mark number 3.

<table>
<tr><th colspan="2">Grouts with evaluation mark no 3</th></tr>
<tr><th>Grout no 26</th><th>Notes</th></tr>
<tr><td>
</td><td>One of the specimens broke during cutting it in halves.</td></tr>
<tr><th>Grout no 28</th><th>Notes</th></tr>
<tr><td>
</td><td>One of the specimens broke during cutting it in halves.</td></tr>
</table>

Grout no 38	Notes
	-
Grout no 41	**Notes**
	

59. Shrinkage in cylinder test. Grouts with the evaluation mark number 4.

Grouts with evaluation mark no 4	
Grout no 29	**Notes**
	One of the specimens broke during cutting it in halves. Gout did not adhere well to the cylinder.

7.6 Test results of the mechanical properties

This section presents the test results measuring capillary water absorption, compressive strength, tensile strength, and ultrasonic pulse velocity measurements including information about elastic modulus, velocity propagation, and density. The test results show the absorbency, pushing and pulling force values, and stiffness of the tested materials.[67]

7.6.1 Water absorption coefficient (WAC)

The first measurements were done with the historic plaster samples and with the selected imitation mortar number 8 to set the reference values for the injection grouts. As a result of this test, the water absorption coefficient was calculated for each tested plaster or grout sample and recorded in the chart. The higher the coefficient number, the more absorbent/porous is the material. The test results show that historic plaster from the lower part of the Northern wall is less absorbent than the plaster from the vaulting panel, as well as the imitation mortar.

Measurements done with commercial and custom grouts show that number 29 and 6 have the highest coefficients, which means that these grouts were the most absorbent. The highest

[67] C. Newey, R. Boff, V. Daniels, M. Pascoe, N. Tennant, Science for Conservators: Volume 3 Adhesives and Coatings. Conservation Science Teachings Series. London, New York: Museums & Galleries Commission, 1992, pp. 68, 69.

coefficient seen in number 29 can presumably be due to the fluidizer and acrylic dispersion used in the mixture, which increased porosity during the curing process.

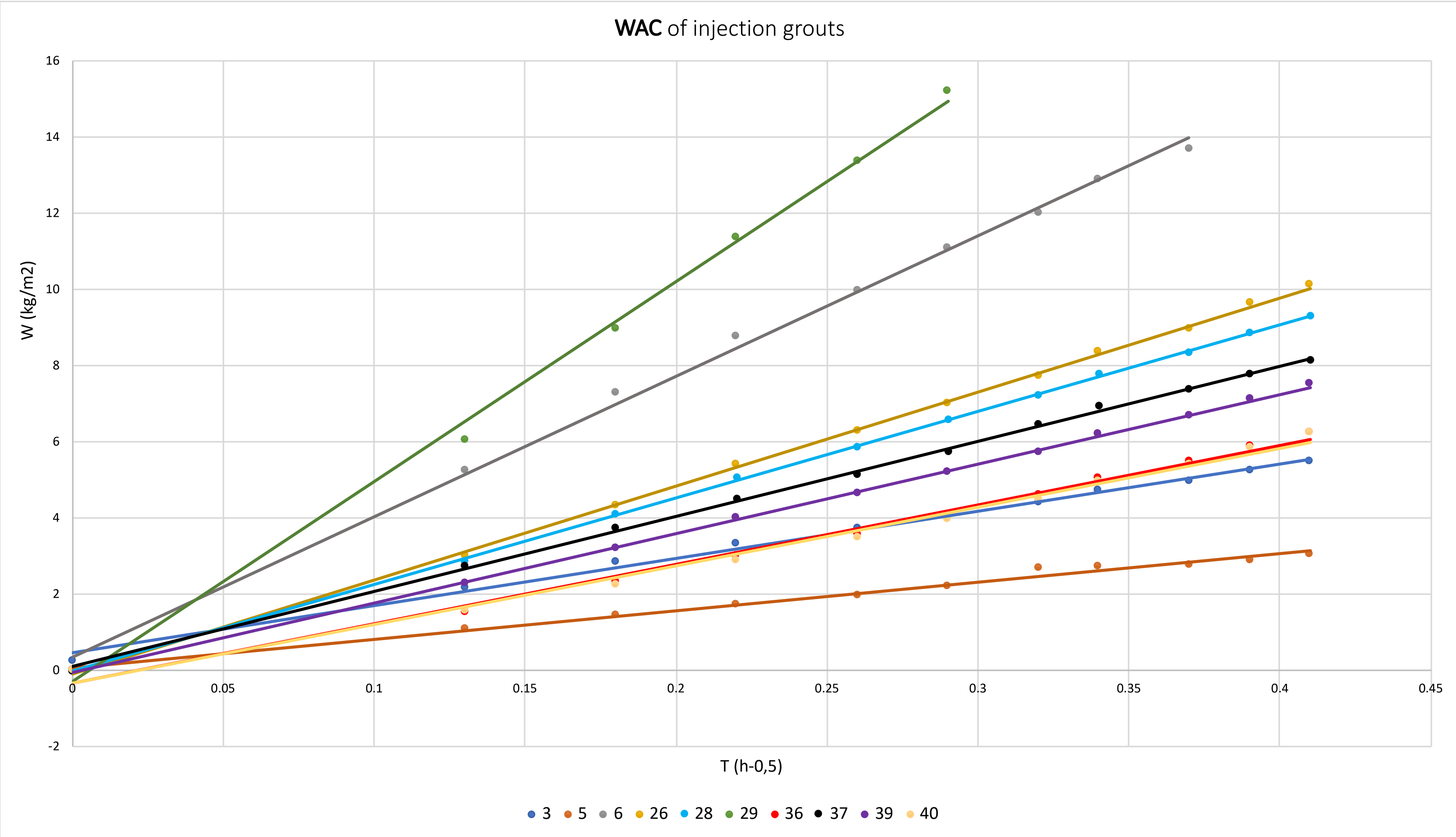
The least absorbent, lower range grouts were number 5, 3, 40, and 36. Number 5 had the closest value to the historic plaster sample taken from the wall, and number 6 had the closest value to the historic sample taken from the vault. The rest of the grouts (39, 37, 28 and 26) belong to the middle range of the test results (see ill. 60).

The fillers influenced the water absorption coefficient of the custom made grouts. See numbers 36 and 37 on the Chart 6 which both contain only lightweight fillers. These grouts are less absorbent than number 26 and 28, which contain limestone powder only. At the same time, number 37 contains Liaver (0,1-0,3 mm), and has a higher water-absorption coefficient than grout number 36, which contains Poraver (0,1-0,3 mm). This could mean that the use of Poraver made the grout less absorbent than the limestone powder or Liaver.

7.6.1.1 Summary

- Historic plaster from the lower part of the Northern wall is about 4,4 times less absorbent than the plaster sample from the vault.
- The closest value to the historic plaster sample from the wall (W-1-2) was the grout number 5 made from Vapo Injekt. The closest value to the sample taken from the vault (V-1-3) was grout number 6, which is a modified version of Ledan TA1.
- Injection grouts number 29 and 6 were the most absorbent.
- The least absorbent grouts were number 5, 3, 40 and 36.

60. Water absorption coefficient of injection grouts.



7.6.2 Ultrasonic pulse velocity measurements

The ultrasonic pulse velocity test measures the time of travel of an ultrasonic pulse passing through the grout and plaster samples.[68] Results are presented in the velocity propagation (VP) chart (see ill. 62), in the elasticity modulus chart (see ill. 61), and in the density chart (see ill. 63). The measurements were made after 28 days of curing, before the grouts had reached the final setting state. Therefore the test results rather give an overview of the tendencies of the mechanical properties.

The value of the elastic modulus describes the stiffness of the grout specimens.[69] Commercial grouts with Ledan TA1 and Vapo Injekt had the highest values in the test result, which means that these grouts are more stiff than the modified commercial grouts and custom grouts. The high stiffness of the injection grouts can cause damage to the historic rendering if the masonry shifts. Because of this, grouts number 6, 26, 28, 29, 36, 37, 39, and 40 are much more elastic and suitable for injection grouting.

Values presented in the chart 63 show that the density of the grouts depends on the type of the aggregate. Mixtures with a higher amount of fine and very fine aggregate are more dense than the mixtures containing lightweight fillers. Grouts number 26, 28, and 29 contained only limestone powder as filler, and grout number 6 was modified with limestone powder 1:1. These grouts have values from 1,091 to 1,163 g/cm^3. Grouts 36 and 37 contain only lightweight fillers which consist of spherical particles. Poraver particles are like pieces of solid foam, and Liaver particles are hollow inside.[70] Therefore these grout samples were not as dense compared to the rest of the samples, due to the air inside the spheres. The values are respectively 0,680 and 0,634 g/cm^3. Grouts number 39 and 40 contained a combination of the limestone powder and lightweight fillers. The density values are 0,825 and 0,802 g/cm^3. Commercial grouts number 5 and 3 and the imitation mortar have the highest values - 1,372 and 1,282 g/cm^3.

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[68] Ultrasonic Pulse Velocity Method, https://www.printfriendly.com/p/g/FxbtNZ (accessed 1st April 2019).

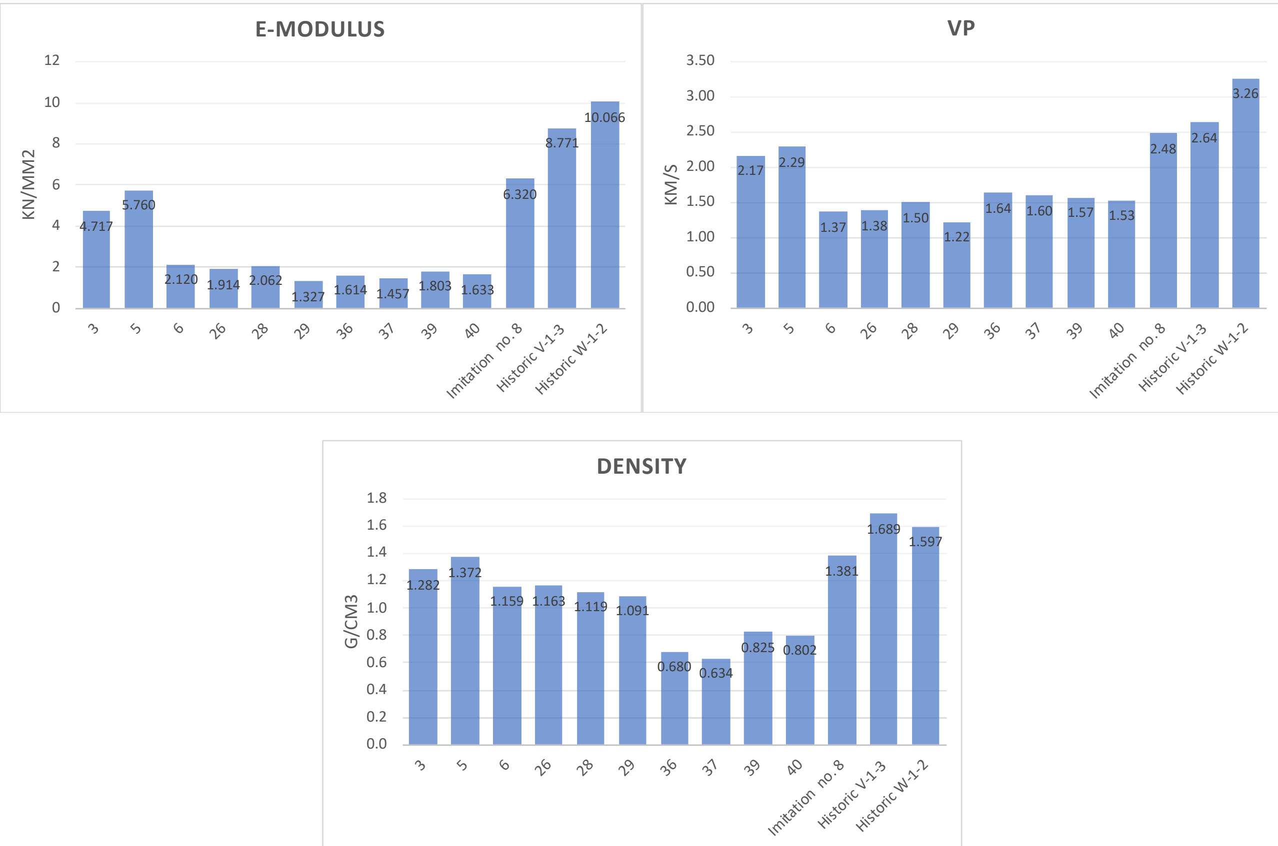
[69] C. Newey, et. al., Science for Conservators, p. 74.

[70] R. Rajtárová, Reštaurovanie maľby sv. Vojtecha na severozápadnej fasáde Suchardovho domu v Novej Pake. Injektážne malty modifikované ľahčenými plnivami na vyplnenie dutín vo vápenných omietkach [Restoration of the wall painting St. Vojtech on the northwestern facade of Suchard's house in the town of Nová Paka. Injection grouts modified by lightweight aggregates to the fill cavities in lime plaster]. MA thesis. Litomyšl: University of Pardubice Faculty of Restoration, 2019, pp. 195-198.

7.6.2.1 Summary

- If the injection grouts is too stiff it may cause damage to the historic plaster.
- Modification of Ledan TA1 with limestone powder in 1:1 weight ratio reduces the elastic modulus by half.
- Grouts containing lightweight fillers are less dense.
- Commercial grouts number 5 and 3 had the highest density.

61, 62, and 63. Results of UPV measurements.



7.6.3 Compressive strength

The compressive strength of the tested grouts was measured after 28 days of curing, before the samples had reached their final mechanical strength. All the custom made grouts were based on the natural hydraulic lime. The final strength of NHL 5 can be reached, in 12 months of curing (approximately 14,0 MPa), according to the product's technical data sheet (see ill. 64). So the results do not show the final compressive strength of the grout samples, but rather give an overview of the tendencies.

The results showed that in four weeks commercial products Ledan TA1 and Vapo Inject are able to reach about a 4-5 times higher compressive strength than custom grouts. Modification of the Ledan TA1 reduced the strength almost 5 times. Grouts number 39 and 40 had the highest compressive strength among the custom grouts, measuring 1,3 and 1,4 MPa, which is close to the strength of modified commercial grout number 6 which had a value of 1,6 MPa.

64. Compressive strength of NHL 5.

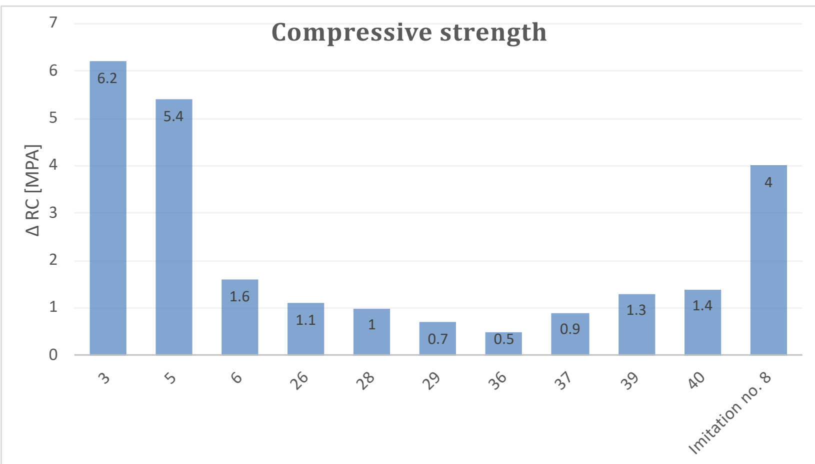
Compression strength of NHL5 (acc. to EN 459-2):	
28 days	approx. 6,5 MPa
6 months	approx. 11,0 MPa
12 months	approx. 14,0 MPa

** Information is taken from the technical data sheet of HYDRADUR® NHL5 by OTTERBEIN.*

7.6.3.1 Summary

- Results give an overview of the tendencies in compressive strength, after 28 days of curing.
- Ledan TA1 and Vapo Injekt are able to reach about 4-5 times higher compressive strength than custom grouts.
- Modification of the Ledan TA1 with the limestone powder, reduced the compressive strength almost 5 times.

65. Results of compressive strength.



7.6.4 Adhesion test

Test was applied on the three injection grouts, which were chosen for the on-site testing. Results show that the adhesive capacity of tested custom grouts is on the level of Ledan TB1 and Ledan TC1 plus but probably lower than Ledan TA1. The adhesive capacity of the mortars 39 and 40 is can be actually higher than the measured value because the samples cracked in the plaster layer of the test specimens not in the mortar layer or on the border of the mortar and the plaster layer (see ill. 66).[71]

66. Results of the adhesion strength measurements.

Sample no.	Strength (N)	Adhesion strength (MPa)	Additional comments
28-1	-	App.0,1	Samples with cracks in the injection layer, de-cohesion in the injection layer due to the cracking along the cracks, decohesion in the injection layer
28-2	130,3	0,1	
39-1	823,1	0,5	Very strong, no cracks, homoge-nous, decohesion in the injection layer (1), sample (2) within the layer of the epoxy glue
39-2	731,8	≥0,5	
40-1	304,3	≥0,2	Decohesion within the layer of the epoxy glue
40-2	284,4	≥0,2	

7.7 Final selection of the custom grouts based on test results

Table 29 presents all the evaluation marks given to the 10 selected grouts. The minimum sum of the given marks was 6 (number 3) and the maximum was 33 (number 29). Injection grouts with the lowest score were fluid, had good water retention, and were more stiff and dense than the

[71] E-mail consultation with MSc Karol Bayer, University of Pardubice, (08.04.2019).

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 a bond with the imitation specimens in the shrinkage and adhesion tests.

The final selection of the grouts was made from the custom grouts which scored in the middle range (see ill. 67). These grouts had good workability and water retention, satisfactory results in the shrinkage test, were elastic, and had lower mechanical strength than the historic plaster samples.

Finally, the three selected grouts were number 28, 39, and 40. The main difference in the composition of these grouting mixtures was 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. These grouts were selected in order to test one grout which contains finer aggregate for re-adhering medium size voids, and one lightweight grout with the combined aggregate for use with large voids. Two lightweight grouts were selected to test the workability of the two different expanded glass aggregates.

67. Evaluation marks for the ten selected grouts.

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	Comp-ressive 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**

8. On-site testing of the custom injection grouts

As a final part of the research, three selected grouts were tested on-site in St. Mary's Church, and also in the chapel of the Sacred Heart at the Church of the Virgin Mary of the Rosary in České Budějovice, Czech Republic. The aim was to evaluate the working properties of the injection grouts by testing the mixtures in actual working conditions which are difficult to imitate in the laboratory.

8.1 In situ tests in St. Mary's Church, Pöide

Two test areas were chosen in St. Mary's Church. One of the areas is situated on the vault, and the other on the lower part of the Northern wall in the vaulting under the tower.

For further understanding the size of the voids is described in the following table:

68. The sizes of the voids and cracks.

Fine	Small	Medium	Large
≤ 5mm	5-20 mm	20-40 mm	≥ 40 mm

8.1.1 Test area number 1

Test area number 1 is situated on the vaulting panel under the tower. There is only one type of plaster in the test area. According to the 2012 SMC plaster research report, it originates from the Gothic construction period in the 13th-14th century, the time when the western vaulting was built.[72] About 70% of the remaining plaster in this area is slightly detached, and the rest is loose. There are a few bigger cracks, and algae covers the whole surface of the test area.

[72] E. Mölder, Pöide…, pp. 85-86.

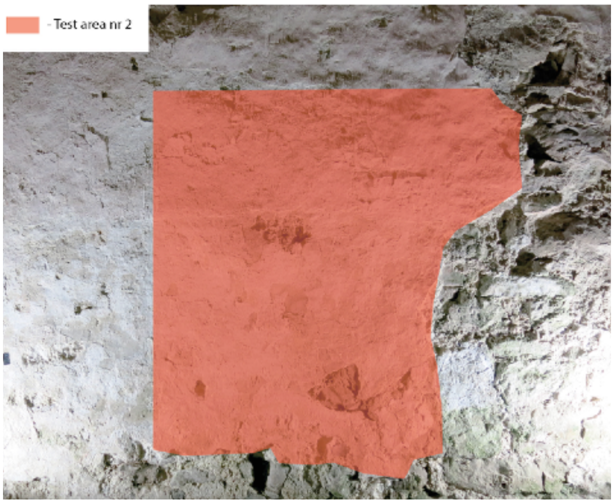
69. The size of the test area is126x110x124 cm.

70. The condition mapping of the test area number 1.

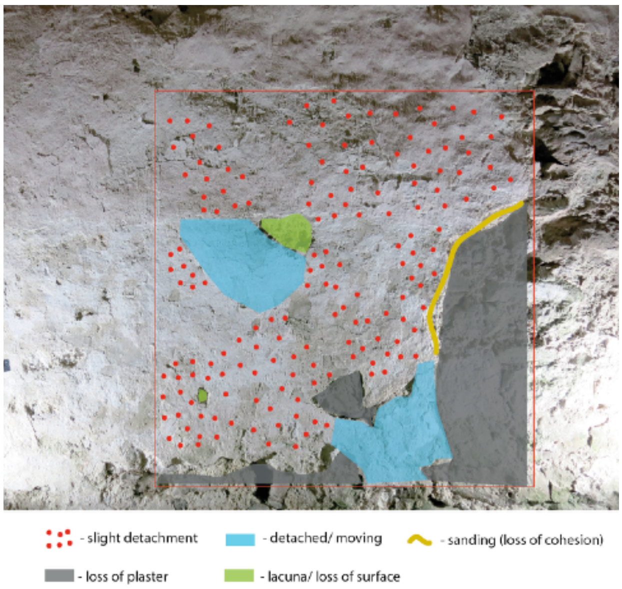
8.1.2 Test area number 2

Test area number 2 is located on the lower part of the northern wall. In that area three types of historic plaster were found. First, the plaster from the Gothic construction stage, then the repair mortar, which was applied after the fire in 16th century, and finally the plaster from 17th or 18th century.[73] Climate conditions in that area are of the wall are poor, since there is excessive moisture in the lower part of the masonry throughout the year. [74]

71. The size of the test area is 130x148 cm.



72. The condition mapping of the test area number 2.



[73] E. Mölder, Pöide ..., p. 85.

[74]L. Kurik, Pöide kiriku seinte niiskusmõõtmised, Tallinn, 2018, pp. 8-12, http://kirikud.muinas.ee/pdf/ebgnh5bf1a992e0ea2_2018.pdf (accessed 30 March, 2019).

8.1.3 Results of the *in situ* experiments

8.1.3.1 Injection grout number 28:

73. Recipe of the grout number 28. The total quantity of the grout according to this recipe is about 100 ml.

Grout no.	Water	NHL 5	Limesone powder	5% Tylose MH 300	Melment F10	5% K9
28	62,75 g	45 g	85 g	11,25 g	0,18 g	8 g

Preparation and application

Grout number 28 was tested on the vaulting panel in St. Mary's Church to fill smaller voids which were detached about 5 to 10 mm. About half a litre of the grout was prepared at once. Dry ingredients were added to the water which was premixed with the additives, Melment F10, K9, and Tylose MH 300. The grout was mixed with a drill mixer for about two minutes until it was homogenous. The void was pre-wetted with the 1:1 solution of ethanol and water to ensure better adhesion between the grout, the historic plaster, and the masonry. Injections were done through small tubes with an inner diameter of 3 mm, which fit the tip of the 60 ml syringe. In some cases injections had to be done by applying quite a lot of pressure to the syringe to push the grout into small voids (about 5 mm or less). This is not convenient because it can be physically tiring for the restorer over time. Due to this inconvenience, I think that the grout is not suitable for very small voids and cracks (< 5 mm). Because of the relatively high amount of Tylose MH 300 (5% Tylose MH 300, 25 wt% to binder) in the grout, it has a level of tackiness and viscosity which prevents the grout from flowing in very small cracks and voids. However, for larger voids that amount of Tylose MH 300 ensures the necessary water retention, and helps the grout to flow further inside the void. Because of its good working properties, Ledan TA1 was used instead to inject the fine and very fine cracks and voids. I do not recommend increasing the volume of the water in the original recipe of grout number 28. This creates imbalance in the ratio of water and methyl hydroxyethyl cellulose, which can cause the sedimentation of the solid particles, and decreases water retention. As a result the grout is less fluid when in contact with the historic plaster.

8.3.2 Injection Grout number 39 and 40:

74. Recipes of the grouts number 39 and 40.

Grout no.	Water	NHL 5	Lighweight filler	Limesone powder	5% Tylose MH 300	Melment F10	5% K9
39	50,75 g	45 g	Poraver 0,1-0,3 mm	28,33 g	11,25 g	0,18 g	8 g
			26,6 g				
40	50,75 g	45 g	Liaver 0,1- 0,3 mm	28,33 g	11,25 g	0,18 g	8 g
			23,74				

Preparation and application

Injection grouts number 39 and 40 were evaluated together. Mixtures were both used to fill medium or large voids (starting from 2 cm). The only differences in the recipes are the lightweight fillers, Poraver or Liaver. The grouts were prepared similarly to grout number 28. Dry ingredients were added to water which was already premixed with the additives. About two litres of grout were prepared, and mixed with the drill mixer for about 2-3 minutes. During injection work the grout was regularly hand-mixed with the spatula, before filling the syringe, to keep the mixture homogenous. Sedimentation of the solid particles was not noticed during these tests.

The grout was injected through tubes with an inner diameter of 10 mm, using 100 ml syringes. The void was pre-wetted with the ethanol and water in a 1:1 solution. The fluidity of the grouts was suitable for the injection with a bigger, 100 ml syringe. The grouts were also suitable for the reattachment of the loose fragments of plaster. In practice, there was no noticeable difference between the working properties of these two grouts. Either of the grouts could be suitable for the injection of medium and large voids.

8.2 In situ tests in the chapel of the Sacred Heart

This section is contributed by the students from the Faculty of Restoration of the University of Pardubice under the supervision of MA. Jan Vojtěchovský, Ph.D. and MA. Barbora Viková.

In October 2018, grouting mixtures, which were designed in the framework of Varje Õunapuu's diploma thesis at Eesti Kunstiakadeemia in Tallinn in cooperation with the Faculty of Restoration of the University of Pardubice, were tested in the chapel of the Sacred Heart at the Church of the Virgin Mary of the Rosary in České Budějovice, Czech Republic. In the framework of the testing, three selected mixtures were investigated and slightly modified for the practical use. Information from the experiments are following.

Experiments

1. Injection grout no. 40

Original formula

Water	*NHL 5*	*Liaver 0,1-0,3*	*Limestone powder*	*5% Tylose MH 300*	*Melment F10*	*5% K9*
50,75 g	*45 g*	*23,74 g*	*40 g*	*11,25 g*	*0,18 g*	*8 g*

Comments to the application with a modification of the grout

The task of this part of the experiment was to fill a larger crack in the eastern corner of the chapel. The grout was first prepared according to the original formula, with one exception, that a Liaver compound with a bigger grain size of 0.25–0.5 mm was used due to the size of the crack. In addition, larger amounts of the grout were prepared due to the large volume of the cavity.

The mixed grout was too viscous, which was caused by a mistake in the original formula. In the original formula, the amount of lime powder was mistaken. Therefore, water was gradually added to the consistency which appeared to be suitable for injection. In the final, 20 % more water was used than in the original formula. The mixture was applied via funnel to 1 cm wide plastic tube. After the dilution, the viscosity of the mixture for the particular application method was optimal. However, the mixture had to be mixed frequently, as the conventional filler (limestone powder) was setting rapidly. The rate of settling was comparable, for example, to the Vapo Injekt commercial mixture, although the rate of settling was somewhat higher.

Due to the large crack volume, the filler amount had to be further adjusted, especially for economic reasons. Therefore, the share of limestone powder and Liaver was replaced with finely sieved quartz *sand (from the sand pit Kostelecké Horky). The mixture thus prepared was not necessary to dilute according to the original formulation, but the mixture had to be thoroughly mixed prior to each application to avoid sand deposition.*

Another modification of the grout

The formula of the mixture was further modified in terms of simplification of preparation and error in the original recipe. The proportion of water decreased due to the lower concentration of Tylose MH 300, namely 1% instead of 5%. The modified composition of the grout was as follows:

Water	***NHL 5***	***Liaver 0,1-0,3***	***Limestone powder***	***1% Tylose MH 300***	***Melment F10***	***5% K9***
5,75 g	*45 g*	*23,74 g*	*28,3 g*	*56,25 g*	*0,18 g*	*8 g*

Grouting was performed via syringes through a PVC tube with an internal diameter of 3 mm. This mixture was of a slightly thicker consistency but worked without any problems or the need to use more force on the piston. The force needed to inject the mixture through the hose was more likely derived from the position of the tube to the wall. During the work, we did not notice any excessive setting, blocking of the tube or other negative properties of the mixture.

2. Injection grout no. 39

Original formula

Water	***NHL 5***	***Poraver 0,1-0,3***	***Limestone powder***	***5% Tylose MH 300***	***Melment F10***	***5% K9***
50,75 g	*45 g*	*26,6 g*	*40 g*	*11,25 g*	*0,18 g*	*8 g*

Comments to the application

The amounts of all individual components were multiplied by a uniform coefficient according to the desired amount relating to the volume of the injected void.

The mixed mixture was too viscous for pressure injecting, therefore 20 % more water was added to it. Probably the biggest problem was the above mentioned mistaken amount of limestone powder in the formula, which should be 28.33 g. More complicated application was also caused by the particle size of the Poraver filler because the injection needle of the diameter of 1.8 mm, was blocked after a moment of grouting and it was not possible to get the grout to the crack anymore.

The mixture was therefore filtered through a fine sieve, but this resulted in a situation, that part of the filler did not pass through the sieve. It was obviously caused by clusters of limestone powder and perhaps also by too high concentration of Tylose MH 300. The mixture should have been mixed prior to each filling in the syringe, as limestone powder was depositing at the bottom of the vessel. The commercial injection grout Vapo Injekt behaved similarly to our grout, including the need to be mixed before filling into the syringe, however, the commercial grout did not clog the needle so often.

3. Injection grout no. 28

Original formula

Water	***NHL 5***	***Limestone powder***	***5% Tylose MH 300***	***Melment F10***	***5% K9***
62,75 g	*45 g*	*85 g*	*11,25 g*	*0,18 g*	*8 g*

Comments to the application with a modification of the grout

As in the case of the grout no. 40, this formula was further modified to simplify preparation. Instead of a 5% solution of Tylose MH300, a 1% solution was used, resulting in a reduction in the proportion of water. The modified composition of the blend was as follows:

Water	***NHL 5***	***Limestone powder***	***1% Tylose MH 300***	***Melment F10***	***5% K9***
18 g	*45 g*	*85 g*	*56,25 g*	*0,18 g*	*8 g*

With a grout mixed in a quantity according to the desired void size, after the pre-sieving of the materials (due to their lumpiness) and the extensive mixing, the resulting mixture worked very well. For injection with a needle of 1.8 mm diameter, the consistency was sufficient and the needle was never blocked. The mixture should be mixed well before each filling into the syringe as the heavier components of the mixture are settling at the bottom of the vessel. Compared to the Vapo Injekt injection grout, our grout was very similar, and it behaved similarly in terms of deposition of heavier particles.

If bulk materials were not sieved before mixing, the needle was blocked (after each application of about 10 ml), which made the work significantly more difficult. Even significant addition of water (about 30%) and re-mixing was not effective. In the attempt to filter or the mixture from biggest clusters (or to break them), the material with larger particles remained on the sieve, so that the original density of the mixture was significantly lowered. A similar behaviour in the straining of the *already mixed grout was also observed with the Vapo Injekt. Thus, it is quite clear from this part of the experiment that it is convenient to carry out the sieving of the grout or its bulk components prior to mixing with the liquid part.*

The team of the Faculty of Restoration, University of Pardubice represented by:

Mgr. art. Jan Vojtěchovský, Ph.D.

8.3 Summary of the on-site experiments

- Grout number 28 was suitable for the small and medium voids and cracks.
- In case of the grout number 28 it is possible to use a needle instead of the tube. To avoid the blockages, sieving of the aggregate was recommended prior the preparation of the grout.
- Grouts number 39 and 40 were suitable for the injection of medium and large voids.
- Custom grouts had enough tackiness to glue back loose pieces of plaster.
- Ledan TA1 was used to inject fine and small voids and cracks. The grouting mixture was diluted according to the need.
- The proportion of water in custom grouts was decreased to lower the concentration of the Tylose MH 300.

9. Practical suggestions

The following practical suggestions are mainly based on the *in situ* experiments and on the subjective observations made during the laboratory testing.

- While preparing the injection mortar, it is recommended to add the dry ingredients to the liquid compound. This way it is easier to mix the grout without creating lumps. Mix the grout throughly for about 2 minutes with the drill mixer, and from time to time during the grouting work.
- In the initial recipe, 5% of Tylose MH 300 was used, but due to its thick consistency it was found to be more convenient to prepare and add the 1% solution to the mixture instead. The binder/additive ratio was kept the same.

- The type of aggregate can be changed in the custom injection grouts, according to specific needs. For example if the coarser aggregate is needed. The binder/aggregate/water ratio should be kept similar to the initial recipes to avoid the sedimentation of the solid particles and the reduction of the water retention.

10. Final Results

The major results of this research are as follows:

- Acrylic emulsion K9 had an unexpected influence on the working properties of the custom grouts. If it was the only additive in the grout, fluidity was decreased. However, no change in the water retention was observed. The water release of the grout was the same as that of grout number 7, which contained no additives. In combination with the fluidizers and methyl hydroxyethyl cellulose, K9 slightly increased the viscosity of the grout.
- 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 number 29 in the fluidity on plastered panel test. Due to poor water retention the grout flowed the least of all the tested grouts.
- Methyl hydroxyethyl cellulose was added to the custom grouts to increase water retention and tackiness, and the experiments showed that it fulfilled this purpose. 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 commercial grout number 3, and this was successful.
- Modification of the Ledan TA1 with an extra filler reduced the stiffness and compressive strength significantly. The grout was more absorbent and the water release in filter paper test was increased two times.
- Custom grouts had lower compressive strength and were more elastic than historic plaster, and were able to create adhesive bonds similar to the Ledan products.
- Custom grouts number 28, 39 and 40 are more elastic than Ledan TA1 but can have adhesive capacity on the level of Ledan TB1 and Ledan TC1 plus.
- 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, I recommend number 28 for small and medium size voids and cracks, and the lightweight grouts number 39 or 40 for the medium and large voids. For the fine cracks and voids I recommend commercial Ledan products.

11. 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.

Conclusion

The aim of this study was to evaluate custom injection grouts for conservation of the historic render in St. Mary's Church, 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 grout developed by the ICCROM team in 1981 inspired the choice of ingredients for the custom grouts in this study. The binder NHL5 was selected for the grouts because it could match the hardness 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, fluidizer 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. The influence of acrylic emulsion K9 still needs further research due to its unexpected negative effect on the fluidity and water retention.

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 more 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.

I am pleased with the outcome of the custom grouts developed in this study, and I am confident that further useful developments could be made through continued research into other possible additives and fillers. 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.

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Resümee

“Injekteerimismörtide hindamine ja kasutus Pöide Maarja kiriku näitel”

Varje Õunapuu

Magistritöö uurib ajaloolise krohvi kinnitamiseks mõeldud injekteerimismörte, et leida sobivad materjalid Pöide Maarja kiriku siseviimistluskrohvi ning ühes sellega keskaegsete maalingute säilitamiseks. 13. sajandil rajatud kirik kuulub Eesti vanimate hulka. Viimase aastakümne jooksul on kirikus toimunud mitmed uurimisprojektid ning restaureerimistöid. Paigaldatud on uus katus ning restaureeritud avatäited. Interjöörikrohvid on endiselt kriitilises seisukorras - suures mahus müürist irdunud ja kohati varisemisohtlikud. Uurimustöö põhieesmärk oli valmistada originaalkrohviga kokkusobituv injekteerimismört ning muuta restaureerimistöid majanduslikult soodsamaks.

Magistritöö on jaotub kolmeks osaks. Esmalt antakse lühiülevaade kiriku ajaloost ja restaureerimistöödest ning tehnilisest seisukorrast. Töö teises, ehk põhiosa, kirjeldatakse ajalooliste krohviproovide analüüsimeetodeid ja tulemusi. Selgitati välja vana krohvi omadused ja koostis. Seejärel kirjeldatakse injekteerimismörtide koostist ning mördiretseptide koostamise protsessi. Katsetusi alustati kahe kommertstoote, Ledan TA1-e ja Vapo Injekti, omaduste analüüsimisega. Eesmärk oli uurida vedela mördi voolavust ja rakendatavust injekteerimisel, et selle põhjal luua referents. Hüdraulilisel lubjal põhinevad mördisegud, koosnesid lisaks sideainele erinevatest lisa- ja täiteainetest ning veest. Töö teise osa lõpetavad uurimuses kasutatud katsemeetodite kirjeldused.

Kolmas osa koosneb katsetulemuste analüüsist, objektil kohapeal teostatud katsetuste tulemustest ning uurimustulemuste kokkuvõtetest ja järeldustest.

Katsemeetodite valikul kasutati lähteallikana Getty Konserveerimisinstituudi väljaannet “Evaluation of Lime-Based Hydraulic Injection Grouts for the Conservation of Architectural Surfaces: A Manual of Laboratory and Field Test Methods”. Lisaks tugineti meetodite väljaarendamisel magistritöö juhendajate M.A. Jan Vojtěchovský Ph.D. ja MSc Karol Bayeri kogemustele. Katsemeetodid jaotusid vastavalt uuritavatele omadustele kolme gruppi: meetodid mörtide reoloogiliste omaduste ehk rakendatavuse uurimiseks, mörtide mahukahanemise uurimiseks ning juba kivistunud mörtide omaduste uurimiseks.

Katsetuste tulemusel valiti välja kolm injekteerimismörti, mis osutusid sobilikuks keskmiste ja suuremõõtmeliste tühimike täitmisel. Valitud segude koostis erineb ainult täiteaine tüübi poolest. Esimesl juhul kasutati lubjakivipulbrit ning kahes viimases mördis kergekaalulisi

täiteaineid - Poraveri ja Liaveri. Peenete pragude ja väiksemate irdunud krohvitükkide kinnitamiseks kasutati endiselt kommertstoodet Ledan TA1.

Usun, et uurimuse tulemusi saab laiemalt rakendada ka teiste Eesti keskaegsete kirikute, sise- ja välisviimistluse säilitamisel. Julgustan konservaatoried magistritöös välja pakutud injekteerimissegusid katsetama ning vastavalt vajadusele edasi arendama.

Appendices

Appendix 1

Test results

1. Results of the funnel test

Tables present the time of flow and dripping of all the tested grouts.

Grout number 1:

Test nr	Flow (min)	Dripping (min)
1	00:06	00:39
2	00:05	00:42
3	00:06	00:27
4	00:06	00:27
Average of 1, 3 and 4	00:05	00:31

Grout number 2:

Test nr	Flow (min)	Dripping (min)
1	00:00	03:40
2	00:00	00:00
3	00:00	00:00
4	00:00	00:00
Average:	00:00	00:00

Notes
Thick and flowing in drips. Most of the grout stuck in funnel. Tests were not repeated.

Grout number 3:

Test nr	Flow (min)	Dripping (min)
1	01:13	00:00
2	01:08	00:00
3	02:00	00:00
4	01:21	00:09
Average of 1, 2 and 4	01:14	00:03

Grout number 4:

Test nr	Flow (min)	Dripping (min)
1	00:11	00:43
2	00:12	00:37
3	00:13	00:37
4	00:11	00:31
Average 1, 2 and 3:	00:12	00:39

Grout number 5:

Test nr	Flow (min)	Dripping (min)
1	02:10	00:21
2	01:56	01:35
3	02:52	01:34
4	02:12	00:25
Average of 1, 2 and 4:	02:06	00:47

Grout number 6:

Test nr	Flow (min)	Dripping (min)
1	00:16	00:14
2	00:16	00:16
3	00:15	00:18
4	00:17	00:15
Average of 1, 2 and 4:	00:16	00:15

Grout number 7:

Test nr	Flow (min)	Dripping (min)
1	00:00	01:32
2	00:00	01:23
3	00:00	01:43
4	00:00	01:45
Average of 1,3 and 4:	00:00	01:40

Notes
A lot of grout stuck inside the funnel - about 29g.

Test nr	Flow (min)	Dripping (min)
1	00:54	00:00
2	00:54	00:06
3	00:53	00:00
4	00:53	00:05
Average of 1, 2 and 3:	00:53	00:01

Grout number 8:

Test nr	Flow (min)	Dripping (min)
1	00:49	00:00
2	00:43	00:00
3	00:48	00:00
4	00:48	00:00
Average of 1, 3 and 4:	00:48	00:00

Grout number 9:

Test nr	Flow (min)	Dripping (min)
1	00:00	01:38
2	00:00	01:36
3	00:00	01:38
4	00:00	01:46
Average of 1, 2 and 3:	00:00	01:37

Grout number 10:

Test nr	Flow (min)	Dripping (min)
1	00:00	00:00
2	00:00	00:00
3	00:00	00:00
4	00:00	00:00
Average:	00:00	00:00

Notes
Grout did not flow through the funnel.

Grout number 11:

Grout number 12:

Test nr	Flow (min)	Dripping (min)
1	00:49	00:16
2	00:52	00:15
3	00:44	00:17
4	00:52	00:14
Average of 1, 2 and 4:	00:51	00:15

Grout number 13:

Test nr	Flow (min)	Dripping (min)
1	00:54	00:09
2	00:57	00:09
3	00:57	00:05
4	00:52	00:08
Average of 1, 2 and 3:	00:56	00:07

Grout number 14:

Test nr	Flow (min)	Dripping (min)
1	01:08	00:23
2	01:04	00:22
3	00:54	00:24
4	00:57	00:24
Average of 2, 3 and 4:	00:58	00:23

Grout number 15:

Test nr	Flow (min)	Dripping (min)
1	01:04	00:00
2	01:03	00:00
3	00:58	00:00
4	00:53	00:09
Average of 1,2 and 3:	01:01	00:00

Grout number 16:

Test nr.	Flow (min)	Dripping (min)
1	00:10	00:14
2	00:15	00:16
3	00:14	00:15
4	00:15	00:15
Average of 2,3 and 4:	00:14	00:15

Grout number 17:

Test nr	Flow (min)	Dripping (min)
1	00:23	00:21
2	00:31	00:21
3	00:24	00:18
4	00:23	00:29
Average of 1, 3 and 4:	00:23	00:22

Grout number 18:

Test nr	Flow (min)	Dripping (min)
1	00:14	00:17
2	00:18	00:16
3	00:13	00:15
4	00:18	00:23
Average of 1,2 and 3:	00:15	00:16

Grout number 19:

Test nr	Flow (min)	Dripping (min)
1	00:54	00:09
2	00:59	00:07
3	00:57	00:10
4	01:09	00:08
Average of 1, 2 and 3:	00:56	00:08

Grout number 20:

Test nr	Flow (min)	Dripping (min)
1	00:58	00:00
2	01:09	00:00
3	01:09	00:00
4	00:59	00:00
Average of 2, 3 and 4:	01:05	00:00

Grout number 21:

Test nr	Flow (min)	Dripping (min)
1	00:00	00:03
2	00:00	00:00
3	00:00	00:00
4	00:00	00:00
Average:	00:00	00:00

Notes
Grout got stuck inside the funnel. Was not possible to measure the test.

Grout number 22:

Test nr	Flow (min)	Dripping (min)
1	00:58	00:08
2	00:58	00:08
3	01:06	00:07
4	01:03	00:06
Average of 1, 2 and 4:	00:59	00:07

Grout number 23:

Test nr	Flow (min)	Dripping (min)
1	00:58	00:05
2	00:55	00:06
3	00:51	00:02
4	00:56	00:03
Average of 1, 2 and 4:	00:56	00:04

Grout number 24:

Test nr	Flow (min)	Dripping (min)
1	00:00	05:36
2	00:00	00:00
3	00:00	00:00
4	00:00	00:00
Average:	00:00	00:00

Grout number 26:

Test nr	Flow (min)	Dripping (min)
1	03:07	00:35
2	03:06	00:23
3	02:55	01:47
4	03:01	00:19
Average of 1, 2 and 4	03:04	00:25

Grout number 28:

Test nr	Flow (min)	Dripping (min)
1	02:53	01:45
2	03:19	00:26
3	02:45	01:39
4	03:14	00:30
Average of 1, 3 and 4	02:57	01:18

Grout number 29:

Test nr	Flow (min)	Dripping (min)
1	00:00	05:39
2	00:00	00:00
3	00:00	12:41
4	00:00	10:58
Average of 1, 3 and 4	00:00	06:06

Grout number 31:

Test nr	Flow (min)	Dripping (min)
1	02:04	00:17
2	02:18	00:05
3	02:07	00:03
4	01:45	00:55
Average of 1, 2 and 3:	02:09	00:08

Grout number 32:

Test nr	Flow (min)	Dripping (min)
1	03:26	00:14
2	01:56	00:04
3	03:02	00:07
4	03:02	00:10
Average of 1, 2 and 4:	03:10	00:10

Grout number 33:

Test nr	Flow (min)	Dripping (min)
1	02:15	00:18
2	02:20	00:11
3	03:13	00:04
4	02:20	00:10
Average of 1, 2 and 4:	02:18	00:13

Grout number 34:

Test nr	Flow (min)	Dripping (min)
1	03:26	00:14
2	01:56	00:04
3	03:02	00:07
4	03:02	00:10
Average of 1, 3 and 4:	03:10	00:10

Grout number 36:

Test nr	Flow (min)	Dripping (min)
1	07:35	00:08
2	08:43	00:00
3	09:04	00:00
4	08:28	00:00
Average of 2, 3 and 4:	08:45	00:00

Grout number 37:

Test nr	Flow (min)	Dripping (min)
1	03:30	00:08
2	04:15	00:09
3	05:12	00:12
4	04:33	00:08
Average of 2, 3 and 4:	04:06	00:08

Grout number 38:

Test nr	Flow (min)	Dripping (min)
1	06:00	00:11
2	04:00	00:18
3	06:37	00:20
4	05:59	00:07
Average of 1, 3 and 4 :	06:12	00:12

Grout number 39:

Test nr	Flow (min)	Dripping (min)
1	00:00	12:32
2	00:00	10:48
3	00:00	10:54
4	00:00	10:55
Average of 2, 3 and 4:	00:00	10:52

Grout number 40:

Test nr	Flow (min)	Dripping (min)
1	06:27	00:00
2	06:49	00:00
3	07:44	00:00
4	09:20	00:00
Average of 1, 2 and 3:	07:00	00:00

Grout number 41:

Test nr	Flow (min)	Dripping (min)
1	08:00	00:24
2	08:41	00:28
3	09:00	00:33
4	11:10	00:05
Average of 1, 2 and 3:	08:33	00:28

2. Flow on plastered panel test

Measured lengths of flow on plastered panel.

Grout nr.	3	5	6	26	28	29	36	37	39	40	42
1.	24	28,5	23	21	28,5	10,5	25,5	22,5	28	28,5	24
2.	28,5	28,5	25	22	28,5	10	25	22,5	28	28,5	22
3.	27,5	28,5	27,5	23	28	11,5	23	20,5	28	28,5	22
AVERAGE	26,7	28,5	25,2	22	28,3	10,7	24,5	21,8	28	28,5	22,7

3. Filter paper test

Recipes for commercial and modified grouts.

Grout No.	Product name	Amount (g)	Extra filler	Amount (g)	Water (g)
1	Ledan TA 1	100	-	-	80
2	Vapo Injekt	90	-	-	30
3	Ledan TA 1	130	-	-	70
4	Vapo Injekt	130	-	-	70
5	Vapo Injekt	150	-	-	60
6	Ledan TA 1	65	Lime-stone powder	65	70

Recipes for the custom-mixed grouts.

Grout no.	Binder	(g)	Additives	(g)	Fillers	(g)	Water (g)
7	NHL5	45	-	-	Lime-stone powder	85	82
8	NHL5	45	Sodium gluconate	0,18	Lime-stone powder	85	82
9	NHL5	45	Sodium gluconate	0,9	Lime-stone powder	85	82
10	NHL5	45	3% Tylose MH 300	4,5	Limestone powder	85	77,5
11	NHL5	45	3% K 9	4,5	Limestone powder	85	77,5
12	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5
			3% Tylose MH 300	4,5			
13	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5
			3% K 9	4,5			
14	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5

			5% Tylose MH 300	4,5			
15	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5
			5,26% K 9	4,5			
16	NHL5	45	Melment F10	0,9	Limestone powder	85	82
17	NHL5	45	Melment F10	0,9	Limestone powder	85	77,5
			3% Tylose MH 300	4,5			
18	NHL5	45	Melment F10	0,9	Limestone powder	85	77,5
			3% K9	4,5			
19	NHL5	45	Melment F10	0,18	Limestone powder	85	77,5
			5% Tylose MH 300	4,5			
20	NHL5	45	Melment F10	0,18	Limestone powder	85	77,5
			5% K 9	4,5			
21	NHL5	45	5% Tylose MH 300	4,5	Limestone powder	85	73
			5% K 9	4,5			
22	NHL5	45	Melment F10	0,18	Limestone powder	85	73
			5% Tylose MH 300	4,5			
			5% K 9	4,5			
23	NHL5	45	Sodium gluconate	0,9	Limestone powder	85	77,5
			50,5% K9	4,5			
24	NHL5	45	Melment F10	0,18	Limestone powder	85	75,25
			5% Tylose MH 300	6,75			
25	NHL5	45	Melment F10	0,18	Limestone powder	85	73
			5% Tylose MH 300	9			
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	powder		
			5% K 9	8			
29	NHL5	45	Melment F10	0,18	Limestone powder	85	74
			5% K 9	8			
30	NHL5	45	Melment F10	0,18	Poraver 0,1-0,3 mm	40	62,75
			5% Tylose MH 300	11,25			
			5% K 9	8			
31	NHL5	45	Melment F10	0,18	Poraver 0,1-0,3 mm	50	70,75
			5% Tylose MH 300	11,25			
32	NHL5	45	Melment F10	0,18	Liaver 0,1-0,3 mm	50	70,75
			5% Tylose MH 300	11,25			
33	NHL5	45	Melment F10	0,18	Fillite SGHA	50	70,75
			5% Tylose MH 300	11,25			
34	NHL5	45	Melment F10	0,18	Poraver 0,1-0,3 mm	50	62,75
			5% Tylose MH 300	11,25			
			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			
38	NHL5	45	Melment F10	0,18	Fillite 160 W	38	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			
41	NHL5	45	Melment F10	0,18	Limestone powder	28,33	50,75
			5% Tylose MH 300	11,25	Fillite 160 W	25,26	
			5% K 9	8			

Approximate volumes for the ingredients used in the custom grouts.

Binder/fillers	volume	mass
NHL	**80 ml =**	**45g**
Limestone powder	80 ml =	85g
Poraver 0,1-0,3	80 ml =	40g
Fillite 160W	80ml =	38g
Liaver 0,1-0,3	80ml =	36g

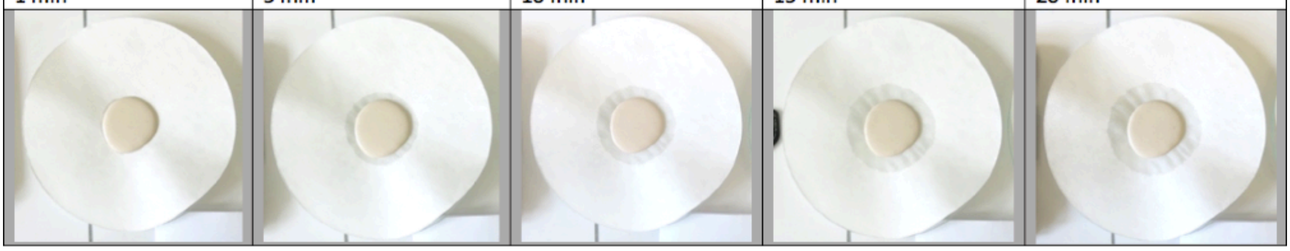
Test results on filter paper

1

1 min | 5 min | 10 min | 15 min | 20 min

2

1 min | 5 min | 10 min | 15 min | 20 min



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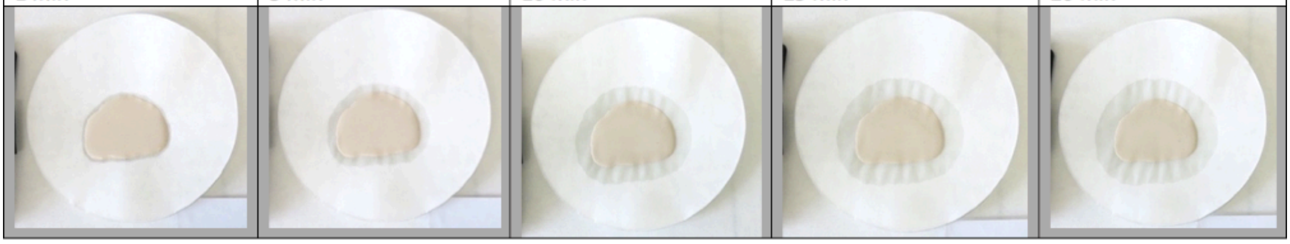
1 min	5 min	10 min	15 min	20 min



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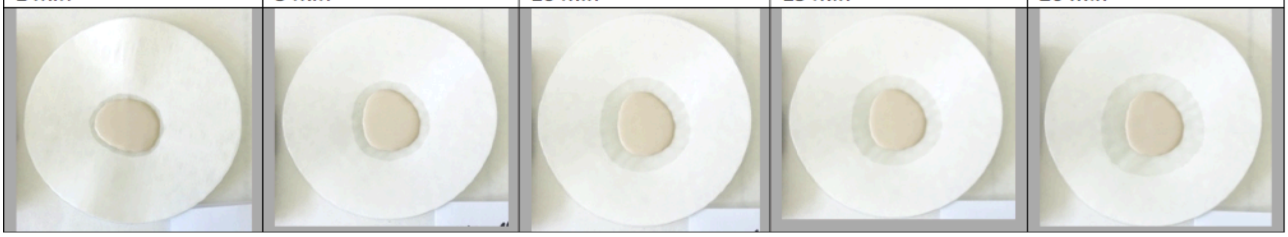

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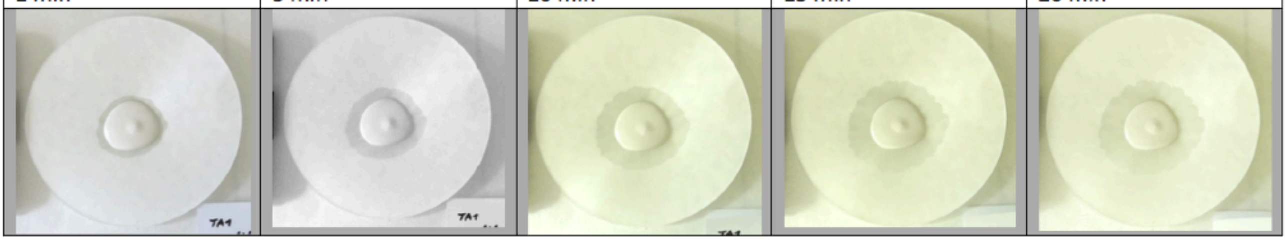

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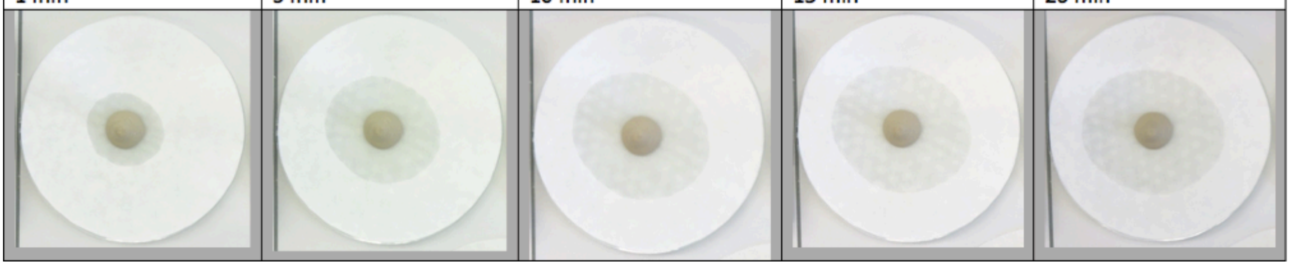

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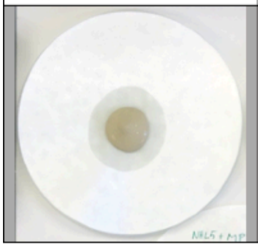			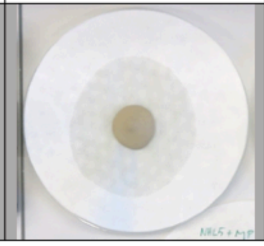	

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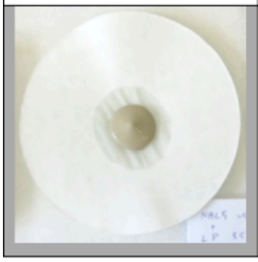	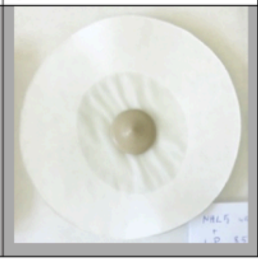	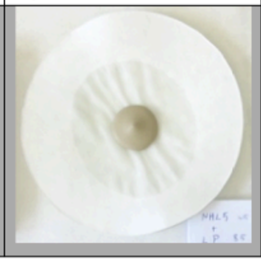		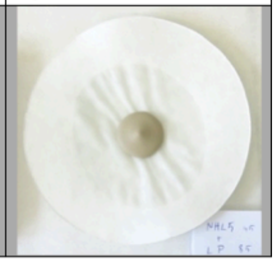

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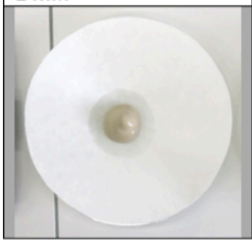				

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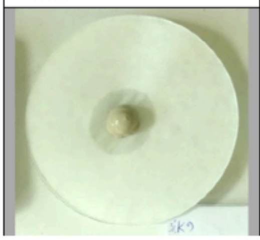	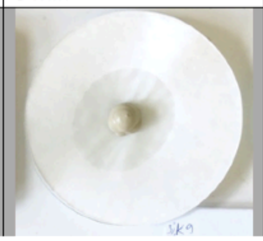	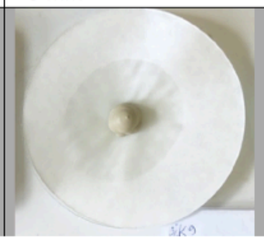	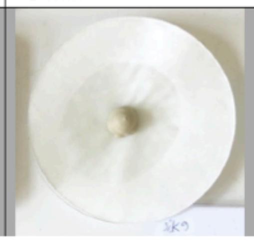	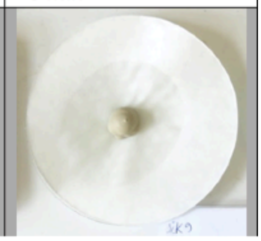

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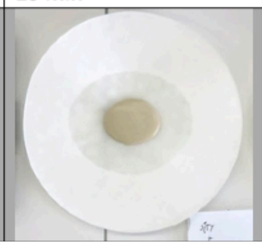
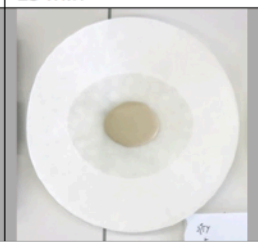
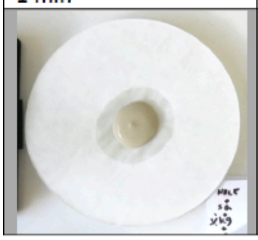
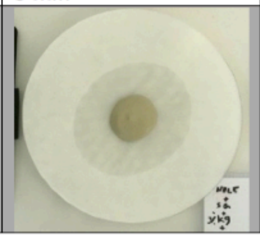
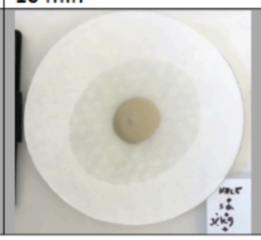
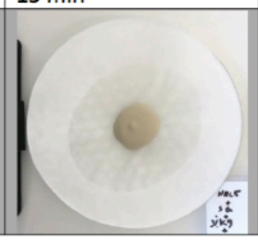
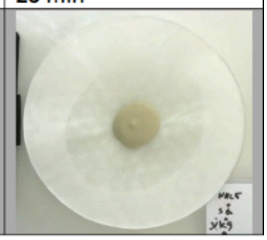
				

13				
1 min	5 min	10 min	15 min	20 min
				

17				
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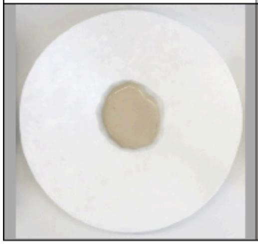
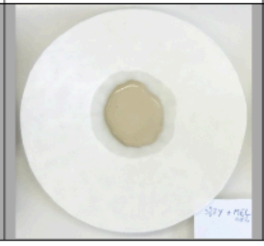
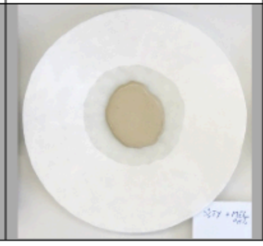
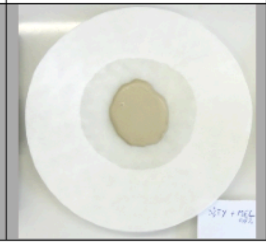
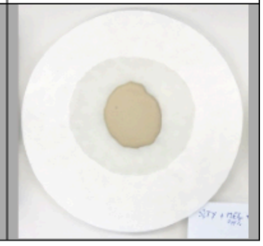
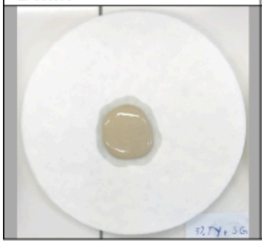
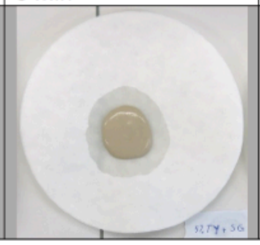
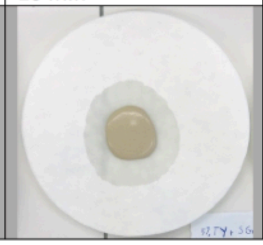
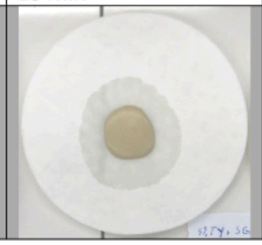
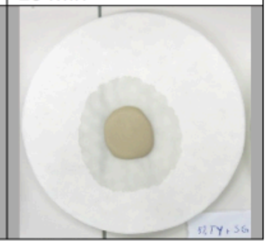
				

14				
1 min	5 min	10 min	15 min	20 min
				

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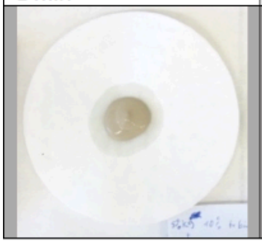	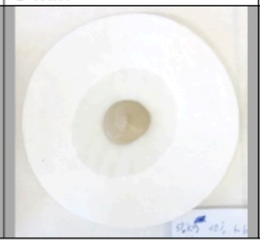			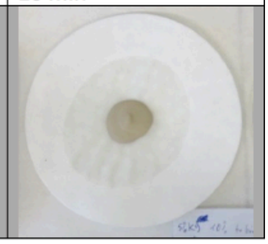

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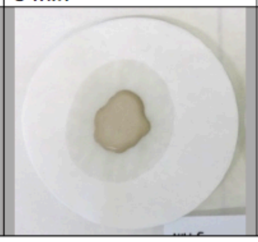
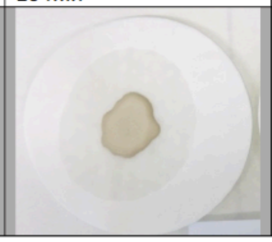
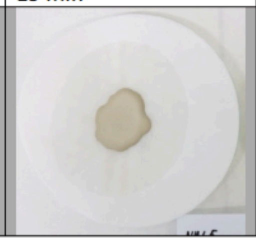
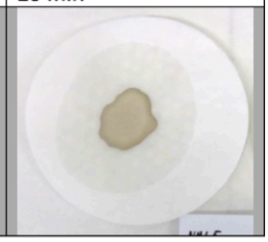
1 min	5 min	10 min	15 min	20 min
				

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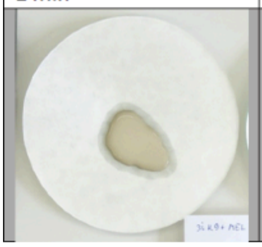
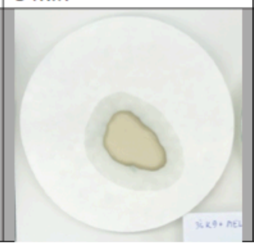
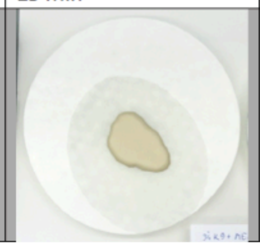
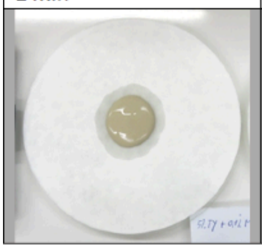
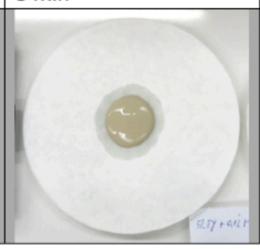
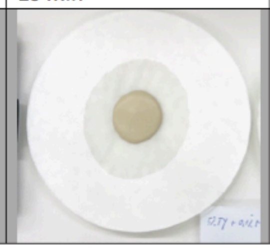
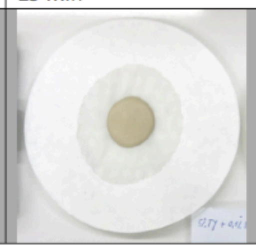
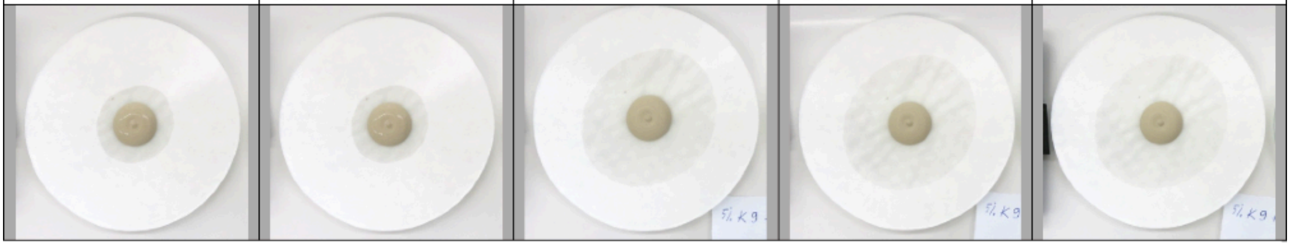
				

19				
1 min	5 min	10 min	15 min	20 min
				

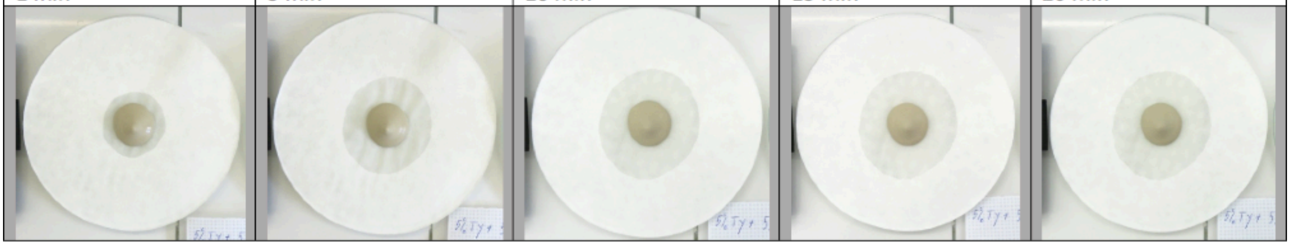
20

1 min	5 min	10 min	15 min	20 min



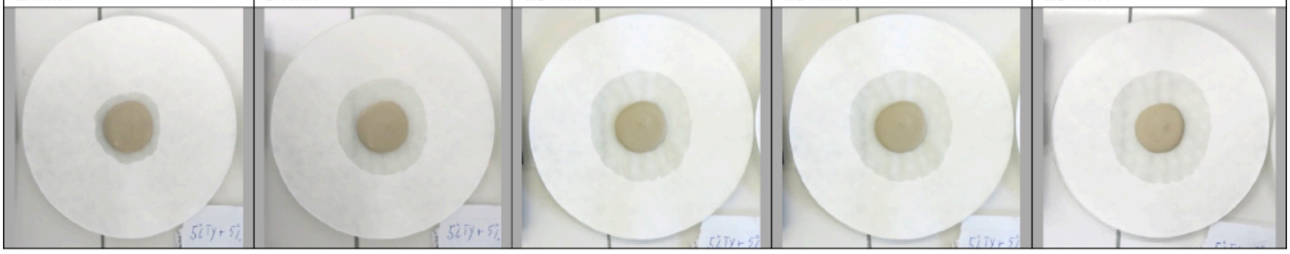
21

1 min	5 min	10 min	15 min	20 min



22

1 min	5 min	10 min	15 min	20 min

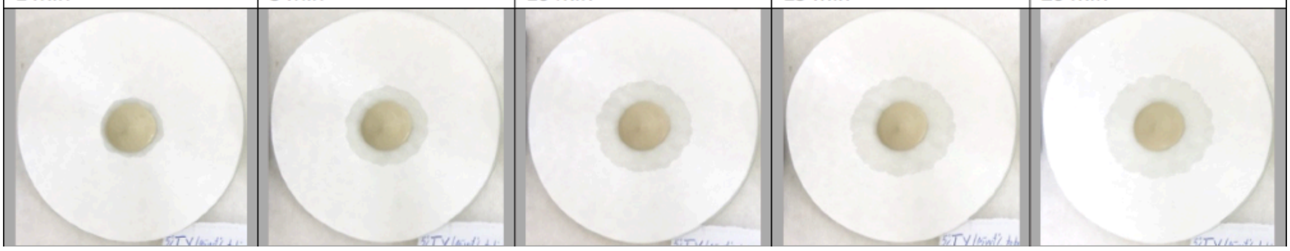


23

1 min	5 min	10 min	15 min	20 min

24

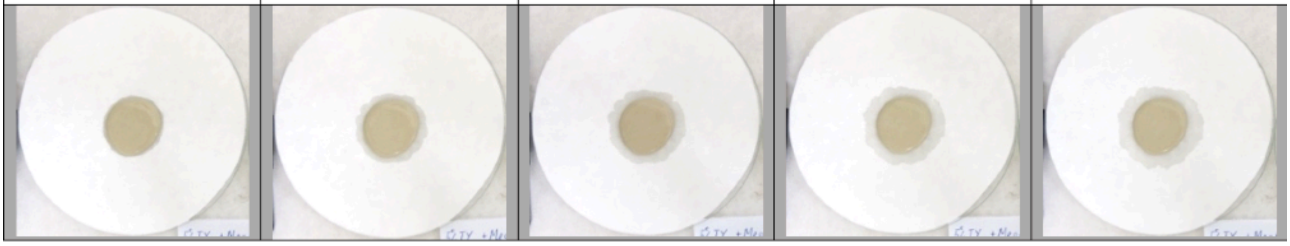
1 min	5 min	10 min	15 min	20 min



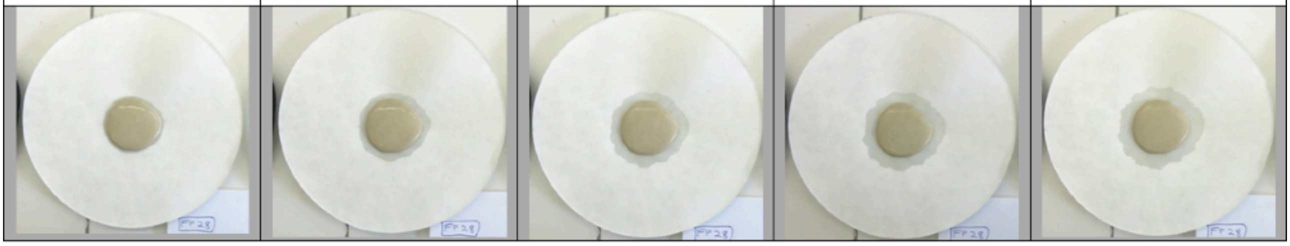
25

1 min	5 min	10 min	15 min	20 min

26				
1 min	5 min	10 min	15 min	20 min



28				
1 min	5 min	10 min	15 min	20 min

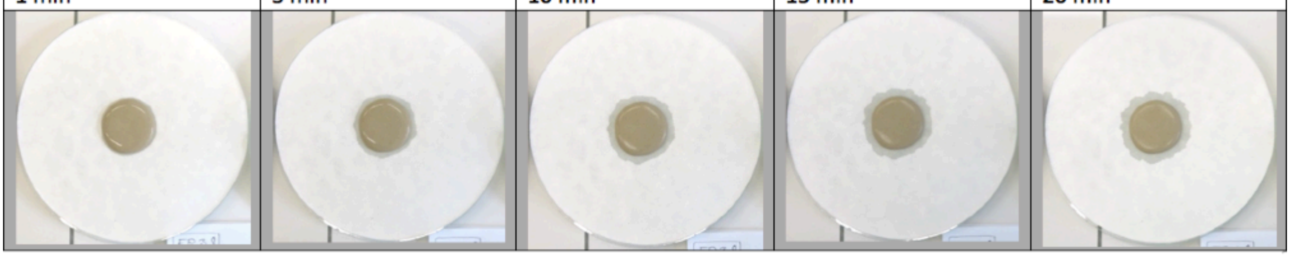


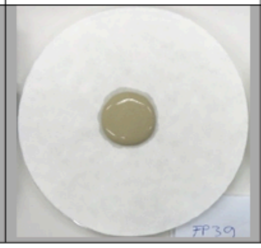
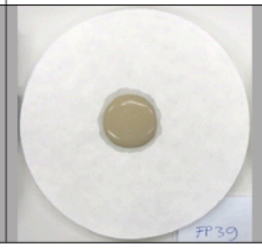
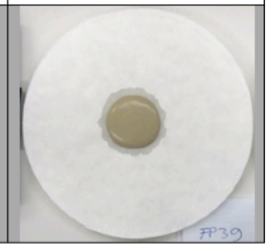
29				
1 min	5 min	11 min	15 min	20 min

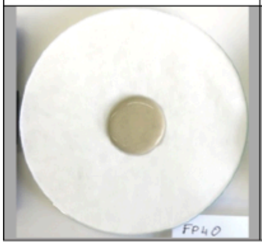
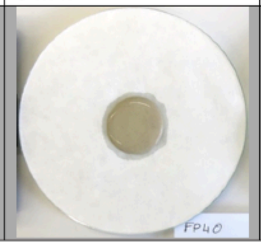
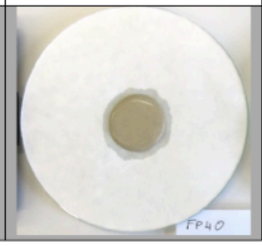
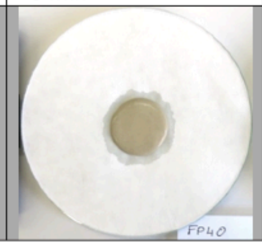
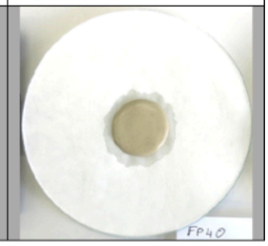
36				
1 min	5 min	10 min	15 min	20 min

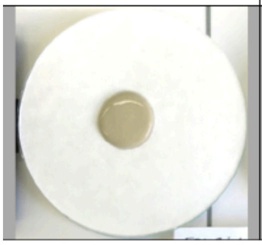
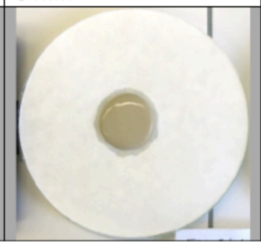
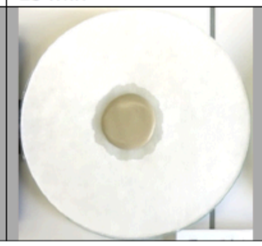
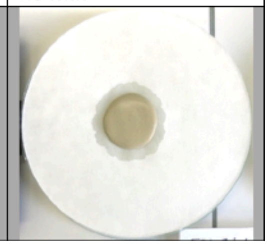
37				
1 min	5 min	10 min	15 min	20 min

38				
1 min	5 min	10 min	15 min	20 min



39				
1 min	5 min	10 min	15 min	20 min
				

40				
1 min	5 min	10 min	15 min	20 min
				

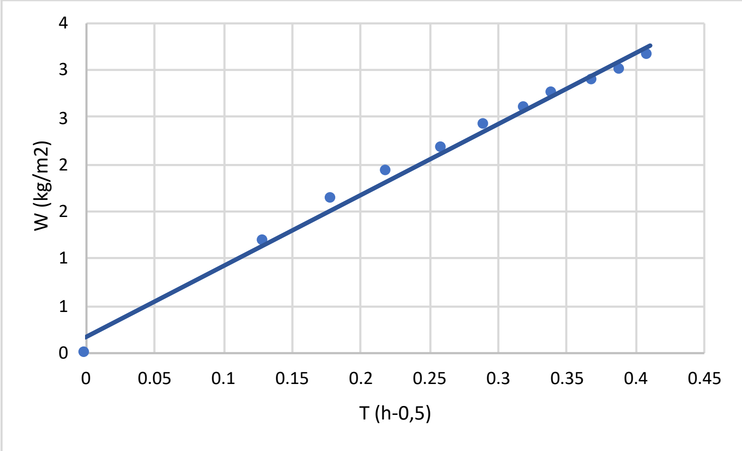
41				
1 min	5 min	10 min	15 min	20 min
				

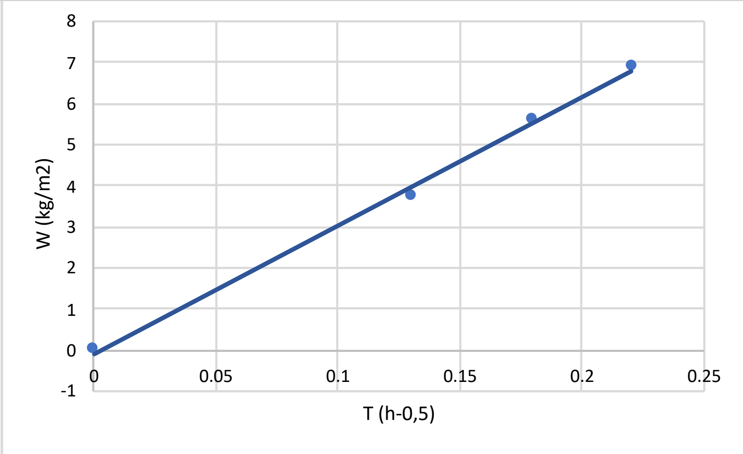
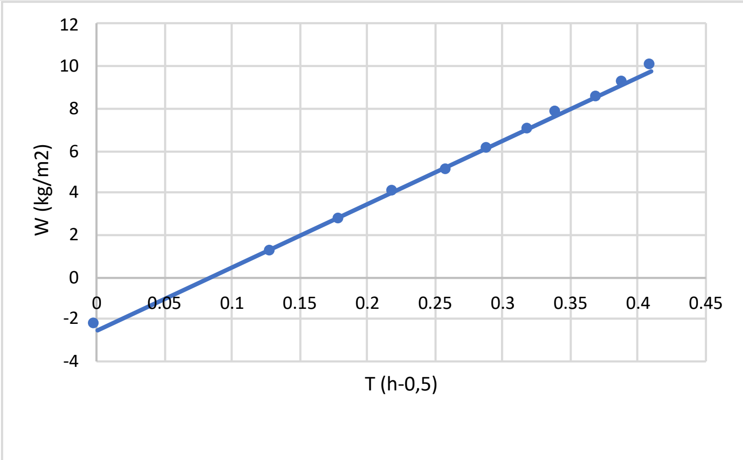
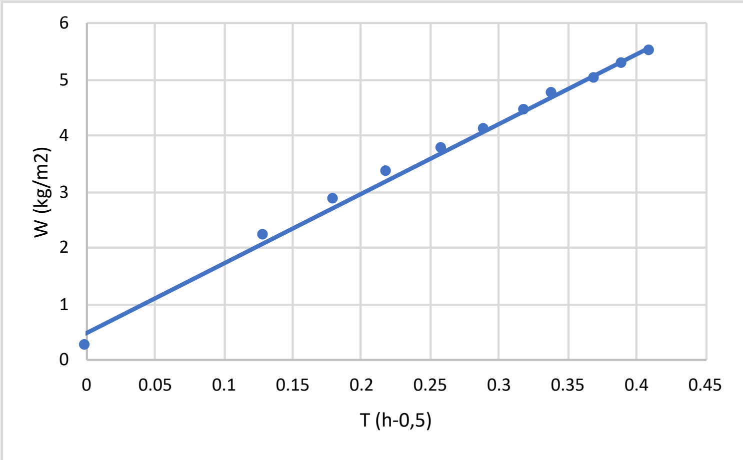
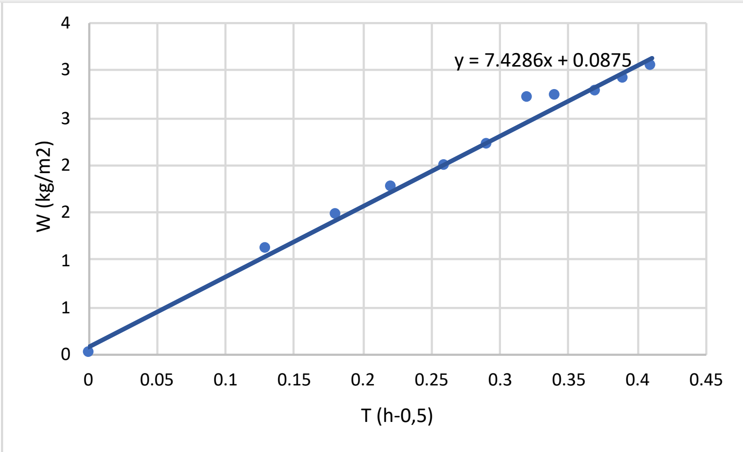
4. Water absorption test results (WAC)

<u>Historic plaster from the wall</u> (lower area of the Northen wall in the tower vaulting St Mary's Church, Pöide).

Sample W-1-1

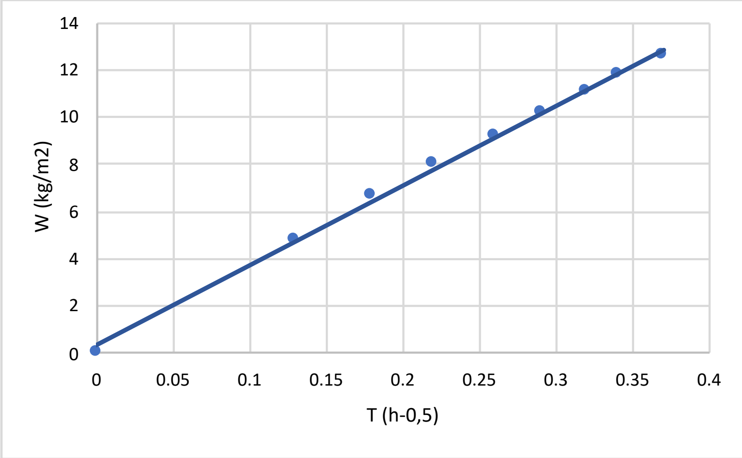
W = 7.5 kg.m^{-2}áh$^{-0,5}$



Historic plaster from the vault (tower vaulting St Mary’s Church, Pöide). Sample V-1-3 **W = 31.2 kg.m-2áh-0,5**	
Imitation plaster no 8 **W = 30.1 kg.m^{-2}áh$^{-0,5}$**	
Grout no 3 **W = 12.4 kg.m^{-2}áh$^{-0,5}$**	
Grout no 5 **W = 7.3 kg.m^{-2}áh$^{-0,5}$**	

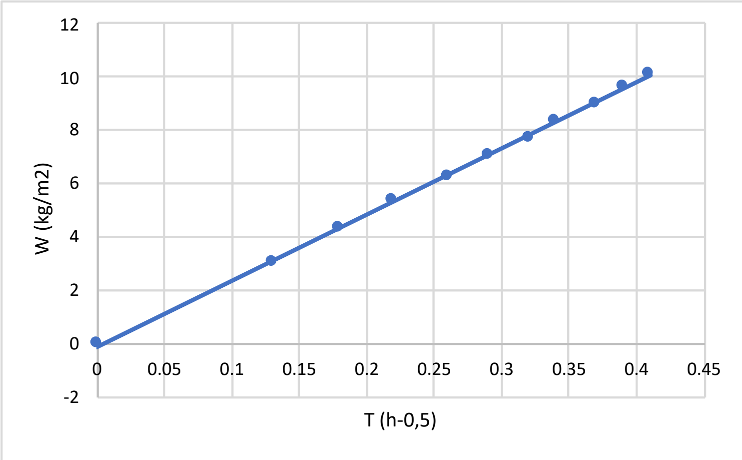
Grout no 6

W = 33.9 kg.m^{-2}áh$^{-0,5}$



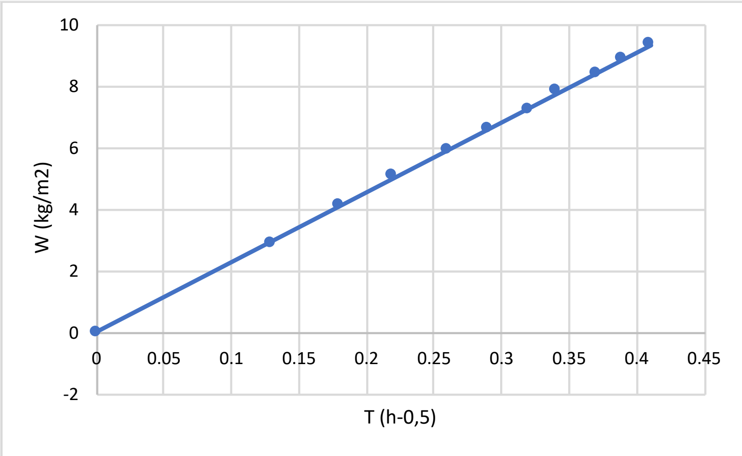
Grout no 26

W = 24.8 kg.m^{-2}áh$^{-0,5}$



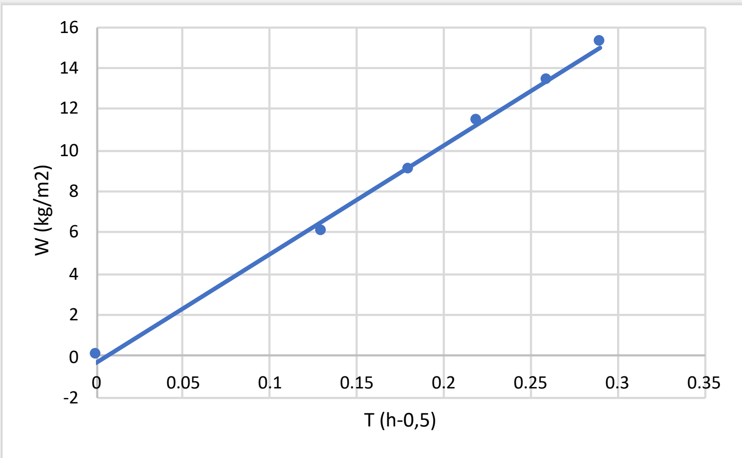
Grout no 28

W = 22.8 kg.m^{-2}áh$^{-0,5}$



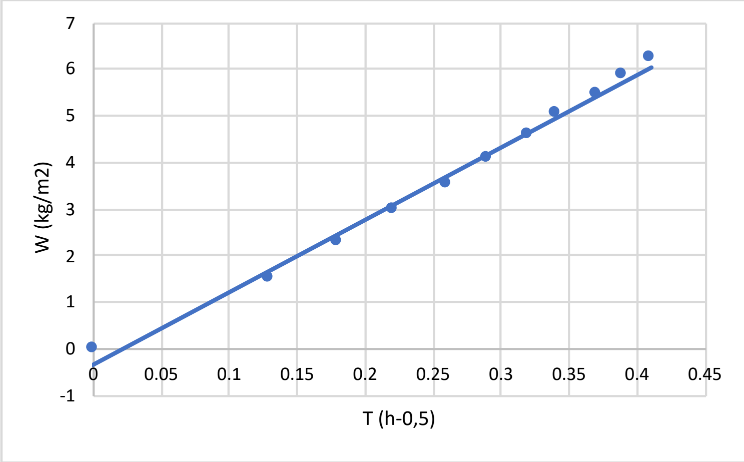
Grout no 29

W = 52.6 kg.m^{-2}áh$^{-0,5}$



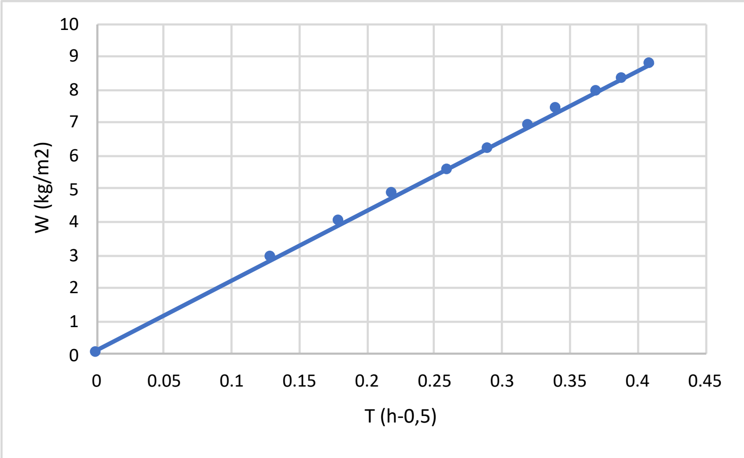
Grout no 36

W = 15.5 kg.m^{-2}áh$^{-0,5}$



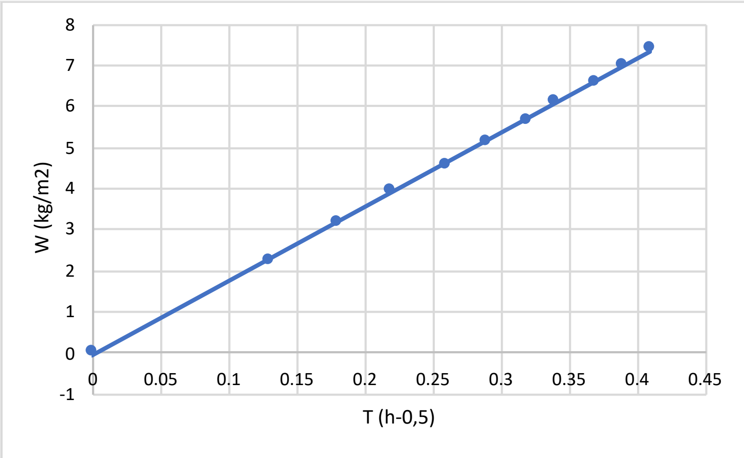
Grout no 37

W = 21.2 kg.m^{-2}áh$^{-0,5}$



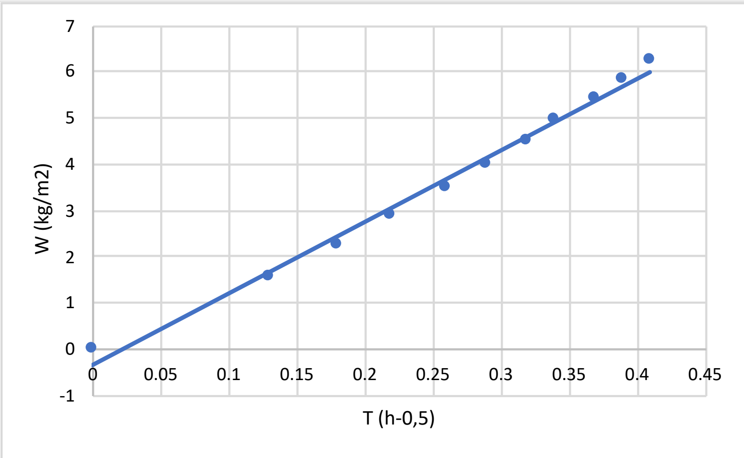
Grout no 39

W = 18.1 kg.m^{-2}áh$^{-0,5}$



Grout nr 40

W = 15.5 kg.m^{-2}áh$^{-0,5}$



5. Compressive strength measurements of the imitation mortar number 8 and the custom grouts

Grout no.	Surface			Compressive strenght	
	b [mm]	h [mm]	F [N]	Rc[MPa]	Δ Rc [MPa]
8A	36,21	31,15	4116,62	3,7	4,0
8B	36,26	31,85	4388,59	3,8	
8C	35,6	30,8	4837,44	4,4	
3-1	35,27	31,68	7499,92	6,7	6,2
3-2	36,63	31,48	6696,75	5,8	
3-3	35,12	31,85	6816,07	6,1	
5-1	35,95	31,9	6541,42	5,7	5,4
5-2	35,38	31,65	5706,78	5,1	
5-3	36,52	31,49	6293,87	5,5	
36-1	35,77	31,88	1397,59	1,2	0,5
36-2	36,02	31,4	122,68	0,1	
36-3	35,87	31,91	122,57	0,1	
37-1	36,05	30,97	132,74	0,1	0,9
37-2	35,4	31,68	1324,95	1,2	
37-3	35,42	32,43	1623,99	1,4	
6-1	35,4	30,92	1600,23	1,5	1,6
6-2	35,59	31,26	2033,11	1,8	
6-3	34,88	31,12	1663,31	1,5	
39-1	35,73	31,51	1363,81	1,2	1,3
39-2	35,84	32,05	1455,62	1,3	
39-3	36,13	32,02	1631,83	1,4	
28-1	35,47	32,15	1133,77	1,0	1,0
28-2	35,64	31,67	1363,86	1,2	
28-3	35,56	32,22	1073,79	0,9	
40-1	35,95	31,7	1473,44	1,3	1,4
40-2	35,92	31,85	1686,4	1,5	
40-3	35,5	31,58	1652,04	1,5	
26-1	35,31	30,9	1095,58	1,0	1,1
26-2	35,27	31,24	1149,2	1,0	
26-3	35,55	31,5	1311,63	1,2	

41-1	35,23	31,64	1305,69	1,2	1,2
41-2	35,63	31,77	1358,67	1,2	
41-3	36,32	31,79	1307,26	1,1	
29-1	35,29	30,22	783,52	0,7	0,7
29-2	35,5	31,28	802,5	0,7	
29-3	35,88	30,91	689,71	0,6	

6. Density of the custom grouts

Grout no.				Weight [g]	Density [kg · m^{-3}]	Δ OH [kg · m^{-3}]
	l [mm]	b [mm]	h [mm]			
36-1	39,71	15,54	15,48	6,208	650	665
36-2	39,44	16,34	15,3	6,722	682	
36-3	39,13	15,75	15,42	6,295	662	
39-1	39,57	15,78	16,18	8,014	793	813
39-2	39,81	15,69	15,68	8,173	834	
39-3	39,68	16,83	15,42	8,358	812	
8-1	39,81	15,81	15,88	14,489	1450	1412
8-2	39,91	15,84	15,81	13,485	1349	
8-3	39,79	15,86	15,48	14,028	1436	
29-1	39,11	15,95	15,92	11,065	1114	1098
29-2	38,76	16,18	15,88	10,648	1069	
29-3	39,3	15,76	15,67	10,792	1112	
6-1	40,16	15,59	15,16	11,126	1172	1181
6-2	39,97	15,83	15,32	11,545	1191	
6-3	40,01	15,72	15,28	11,345	1180	
5-1	39,65	16,31	16,17	13,854	1325	1357
5-2	39,48	16,07	15,85	13,437	1336	
5-3	39,54	15,88	16,03	14,179	1409	
3-1	39,92	16,34	16,04	12,899	1233	1220
3-2	40,16	15,94	16,33	12,586	1204	
3-3	40,09	16,53	16,47	13,35	1223	
37-1	39,79	16,07	16,25	6,358	612	605
37-2	39,53	16,21	15,75	6,115	606	
37-3	40,17	16,07	16,32	6,286	597	
28-1	39,08	15,87	15,51	11,132	1582	1592

28-2	38,94	16,12	15,87	11,35	1583	
28-3	39,17	16,24	15,22	10,932	1611	
40-1	40,12	16,04	15,42	8,037	1515	1541
40-2	39,42	16,21	15,99	7,765	1569	
40-3	39,71	16,52	16,2	8,393	1539	
26-1	39,63	15,21	15,86	10,592	1524	1523
26-2	38,83	15,85	15,43	10,454	1508	
26-3	38,7	14,83	15,95	10,71	1536	

7. Ultrasonic pulse velocity (UPV) measurements of selected injection grouts and the imitation mortar samples

Measured at 13.07.18

Specimen no	Length (mm)	Diameter (mm)	Weight (g)	E-modulus	Average	VP km/s	Average	Density g/cm3	Average2
3.1	40	16	12,974	4,651		2,197		1,267	
3.2	40	16	12,779	4,573		2,159		1,248	
3.3	39	16	13,279	4,928	4,717	2,139	2,17	1,33	1,282
5.1	40	16	13,837	5,637		2,232		1,35	
5.2	39	16	13,604	5,628		2,289		1,363	
5.3	40	16	14,353	6,015	5,760	2,35	2,29	1,402	1,372
6.1	40	15	11,119	2,287		1,407		1,235	
6.2	40	16	11,591	2,075		1,359		1,132	
6.3	40	16	11,356	1,997	2,120	1,346	1,37	1,109	1,159
26.1	40	15	10,703	1,987		1,373		1,189	
26.2	38	15	10,281	1,93		1,395		1,202	
26.3	38	16	10,686	1,826	1,914	1,379	1,38	1,098	1,163
28.1	38	16	11,033	2,006		1,517		1,134	
28.2	39	16	11,274	2,09		1,498		1,129	
28.3	39	16	10,937	2,09	2,062	1,481	1,50	1,095	1,119
29.1	35	16	9,92	1,341		1,229		1,107	
29.2	38	16	10,448	1,243		1,193		1,074	
29.3	38	16	10,612	1,396	1,327	1,228	1,22	1,091	1,091
36.1	40	15	6,343	1,734		1,679		0,704778	
36.2	39	16	6,887	1,612		1,596		0,689804	
36.3	39	16	6,454	1,497	1,614	1,636	1,64	0,646434	0,680
37.1	40	16	6,543	1,505		1,638		0,638967	
37.2	39	16	6,267	1,444		1,577		0,627704	
37.3	40	16	6,497	1,421	1,457	1,579	1,60	0,633984	0,634
38	nm								
39.1	39	16	8,206	1,761		1,577		0,821915	
39.2	40	16	8,355	1,797		1,555		0,815918	
39.3	40	16	8,575	1,85	1,803	1,579	1,57	0,837402	0,825
40.1	40	16	8,172	1,599		1,519		0,798047	
40.2	40	16	7,885	1,441		1,469		0,770019	
40.3	40	16	8,594	1,858	1,633	1,598	1,53	0,839258	0,802
41	nm								
8.1	40	16	14,567	6,775		2,528		1,423	
8.2	40	16	13,63	5,924		2,435		1,331	
8.3	40	16	14,225	6,261	6,320	2,481	2,48	1,389	1,38

8. Second UPV measurement of the injection grouts and the imitation mortar samples

Samples were measured after 4 month had passed from the solvent exchange drying method.

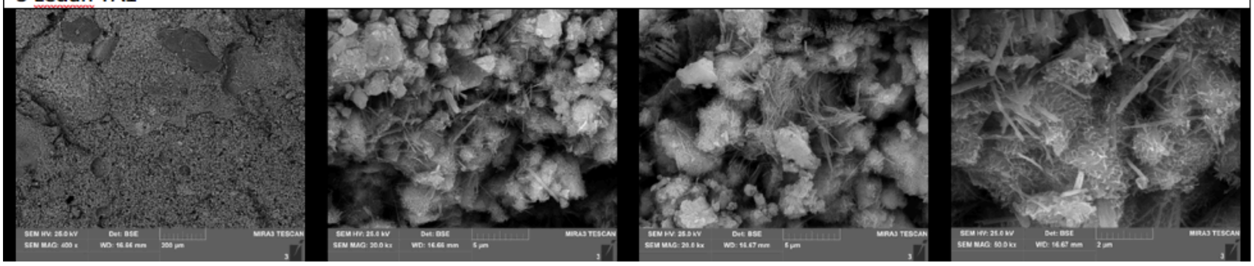
Measured at 13.11.18

Specimen nr	Length (mm)	Diameter (mm)	Weight (g)	E-modulus kN/mm2	Average	VP km/s	Average2	Density (g/cm3)	Average3
3.1	40	16	12,792	4,397		2,081		1,249	
3.2	40	16	12,616	4,268		2,018		1,232	
3.3	39	16	13,088	4,574	4,413	2,104	2,07	1,311	1,264
5.1	40	16	13,888	5,844		2,232		1,356	
5.2	39	16	13,49	5,963		2,33		1,351	
5.3	40	16	14,237	6,23	6,012	2,35	2,30	1,39	1,366
6.1	40	15	11,042	2,164		1,407		1,227	
6.2	40	16	11,49	1,957		1,332		1,122	
6.3	40	16	11,274	1,944	2,022	1,393	1,38	1,101	1,150
26.1	40	15	10,699	2,062		1,407		1,189	
26.2	38	15	10,288	2,03		1,395		1,203	
26.3	38	16	10,685	1,833	1,975	1,41	1,40	1,098	1,163
28.1	38	16	11,044	2,095		1,459		1,135	
28.2	39	16	11,274	2,071		1,416		1,129	
28.3	39	16	10,98	2,143	2,103	1,539	1,47	1,1	1,121
29.1	35	16	9,904	1,351		1,175		1,105	
29.2	38	16	10,45	1,323		1,228		1,074	
29.3	38	16	10,609	1,483	1,386	1,277	1,23	1,091	1,090
36.1	40	15	6,332	1,59		1,722		0,703556	
36.2	39	16	6,879	1,741		1,678		0,689002	
36.3	39	16	6,445	1,638	1,656	1,723	1,71	0,645533	0,679
37.1	40	16	6,568	1,554		1,638		0,641406	
37.2	39	16	6,275	1,514		1,616		0,628506	
37.3	40	16	6,491	1,466	1,511	1,598	1,62	0,633887	0,635
38	NM								
39.1	39	16	8,208	1,821		1,515		0,822115	
39.2	40	16	8,359	1,734		1,658		0,816308	
39.3	40	16	8,568	1,93	1,828	1,579	1,58	0,836719	0,825
40.1	40	16	8,172	2,14		1,555		1,016	
40.2	40	16	7,881	1,93		1,502		0,979922	
40.3	40	16	8,592	2,31	2,127	1,502	1,52	1,068	1,021
41	NM								
8.1	40	16	14,554	8,626		2,481		1,81	
8.2	40	16	13,594	7,223		2,435		1,69	
8.3	40	16	14,161	7,42	7,756	2,35	2,42	1,761	1,754

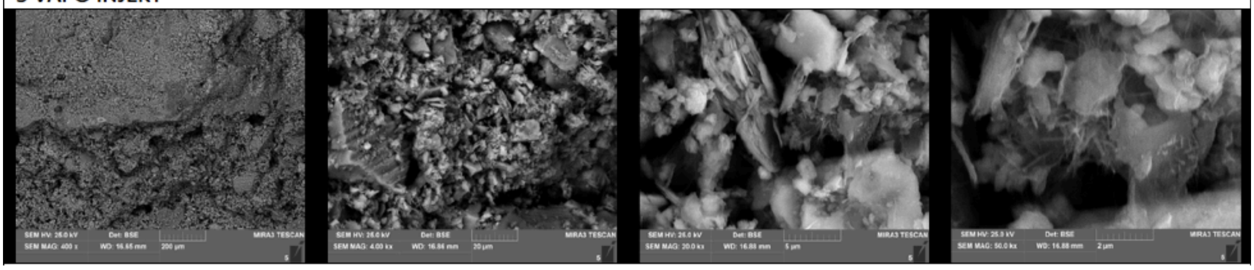
9. Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS)

Following tables contain the SEM images taken from the injection grouts. First photograph in each line shows the border between the grout (below) and the imitation mortar (above). The rest of the images show the bonding of the matrix to the aggregate and the needle shaped crystals formed in the matrix during the hydration process.

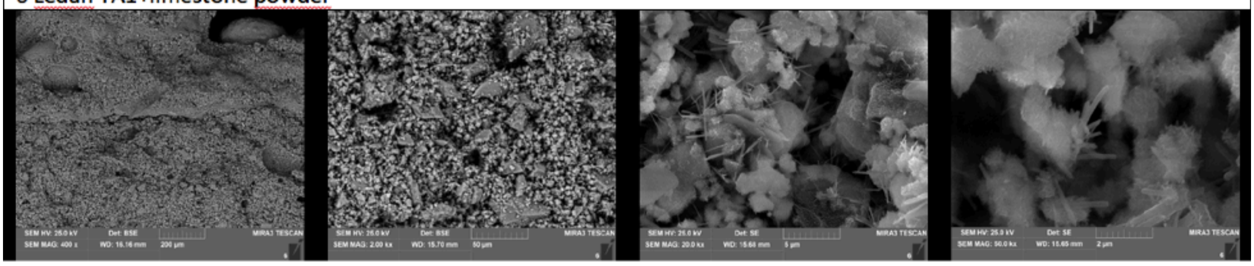
3 Ledan TA1



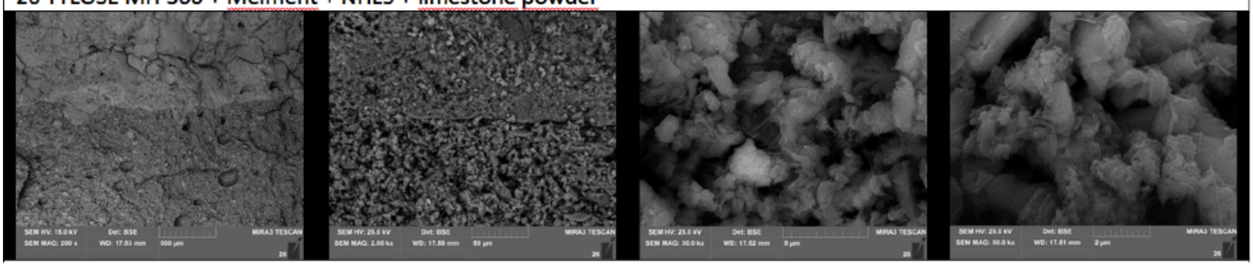
5 VAPO INJEKT



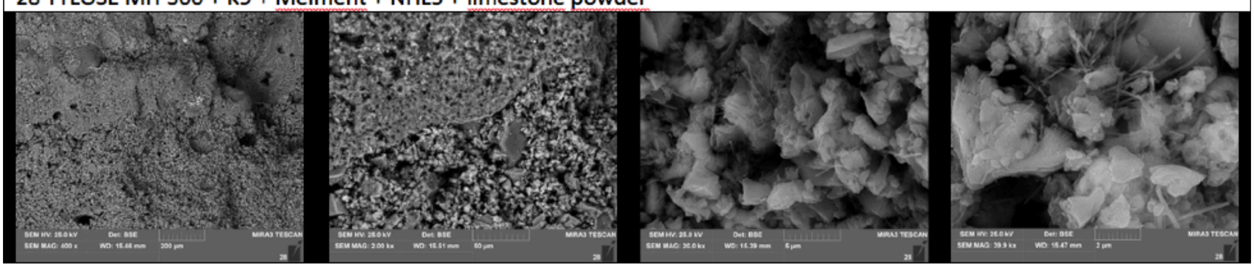
6 Ledan TA1+limestone powder



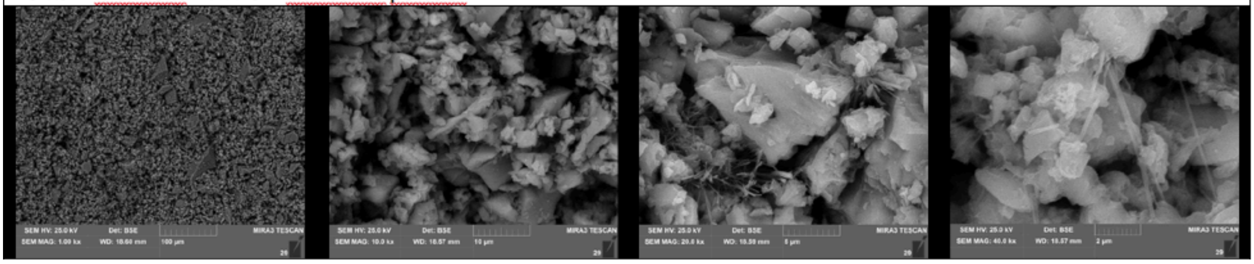
26 TYLOSE MH 300 + Melment + NHL5 + limestone powder



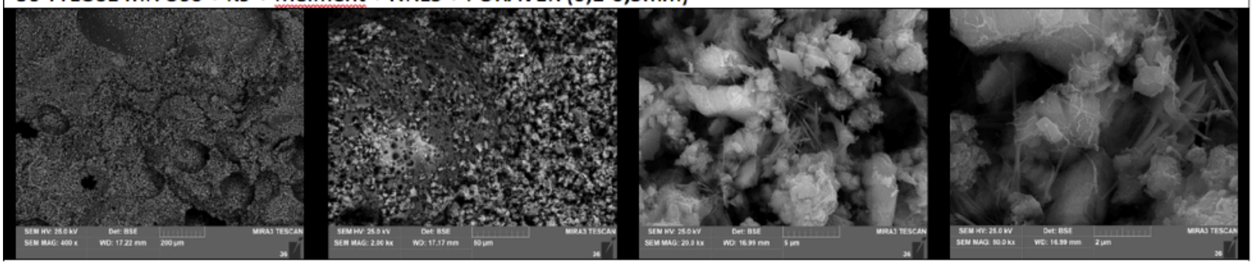
28 TYLOSE MH 300 + K9 + Melment + NHL5 + limestone powder



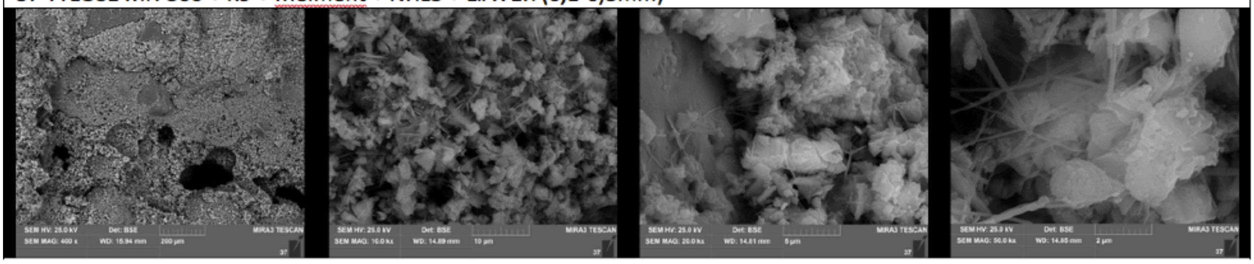
29 K9 + Melment + NHL5 + limestone powder



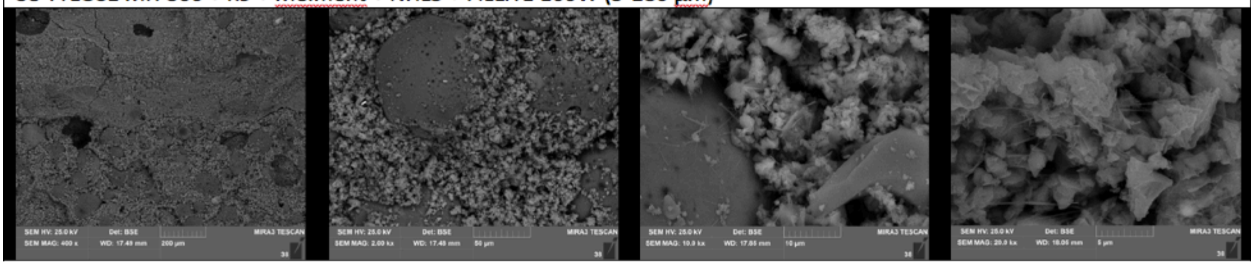
36 TYLOSE MH 300 + K9 + Melment + NHL5 + PORAVER (0,1-0,3mm)



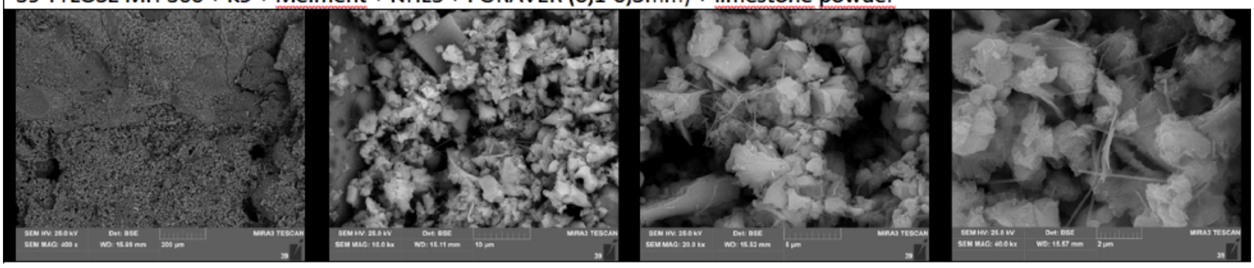
37 TYLOSE MH 300 + K9 + Melment + NHL5 + LIAVER (0,1-0,3mm)



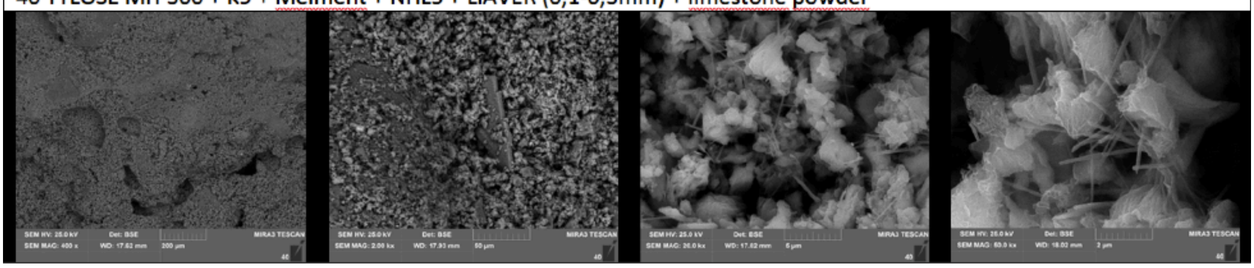
38 TYLOSE MH 300 + K9 + Melment + NHL5 + FILLITE 160W (5-180 µm)



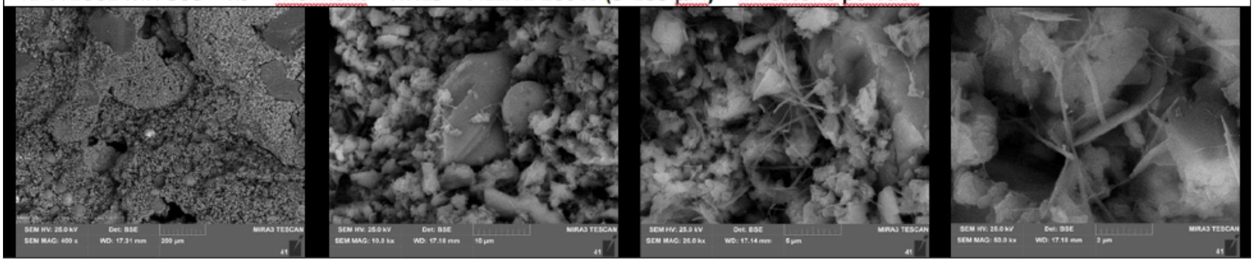
39 TYLOSE MH 300 + K9 + Melment + NHL5 + PORAVER (0,1-0,3mm) + limestone powder



40 TYLOSE MH 300 + K9 + Melment + NHL5 + LIAVER (0,1-0,3mm) + limestone powder

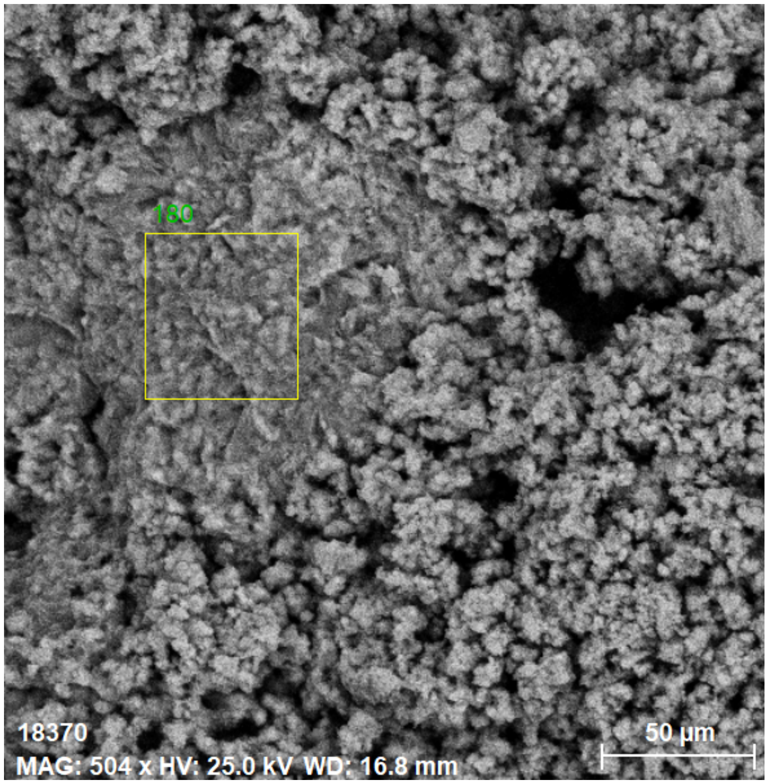


41 TYLOSE MH 300 + K9 + Melment + NHL5 + FILLITE 160W (5-180 μm) + limestone powder

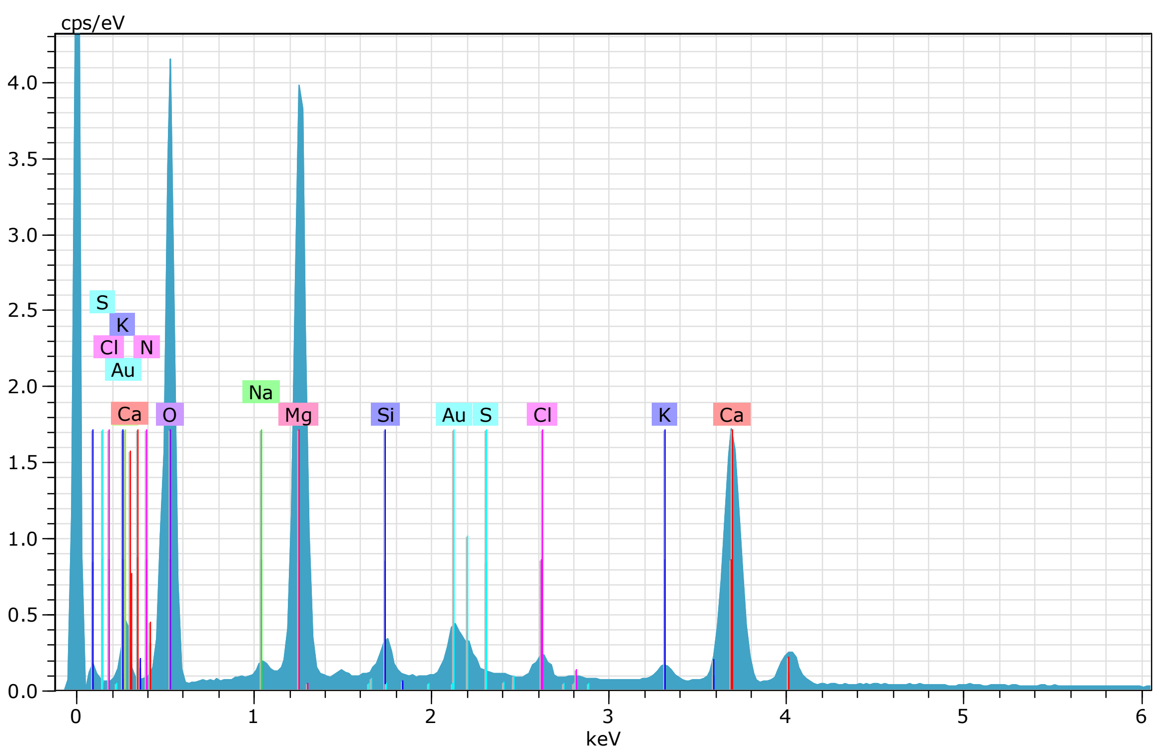


10. EDS elemental analysis of the historic paster samples from the St. Mary's Church, Pöide

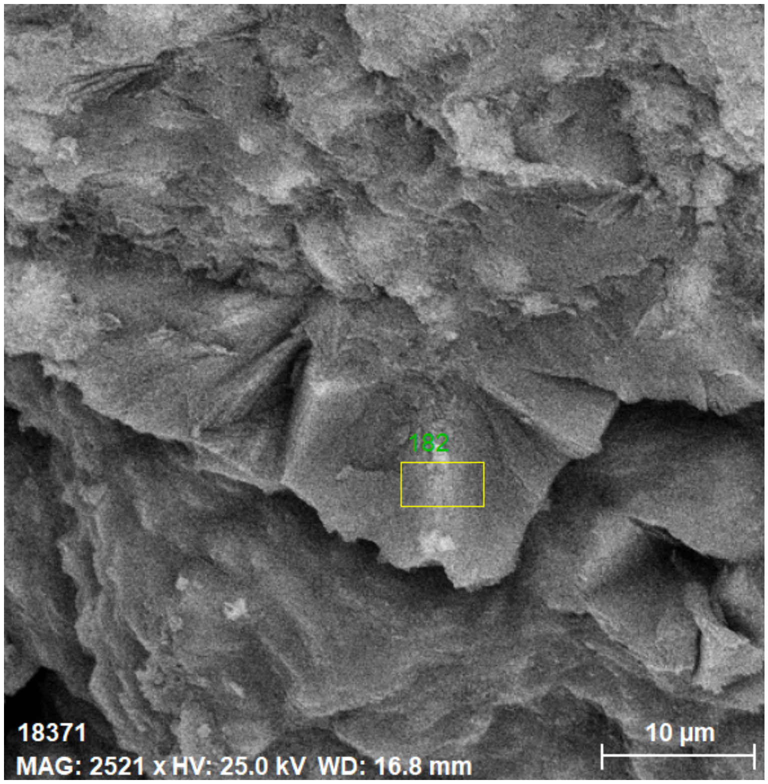
Samples: TV-V-1; TV-W-2; TV-V-3



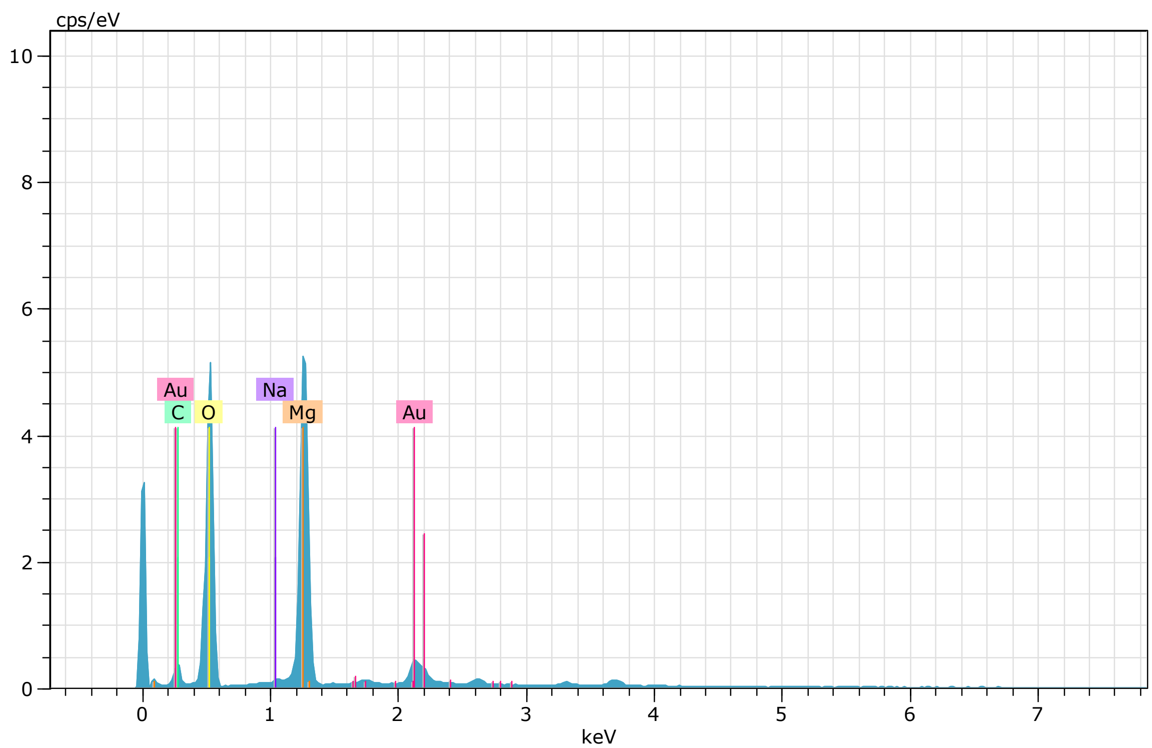
180
18370
MAG: 504 x HV: 25.0 kV WD: 16.8 mm
50 µm



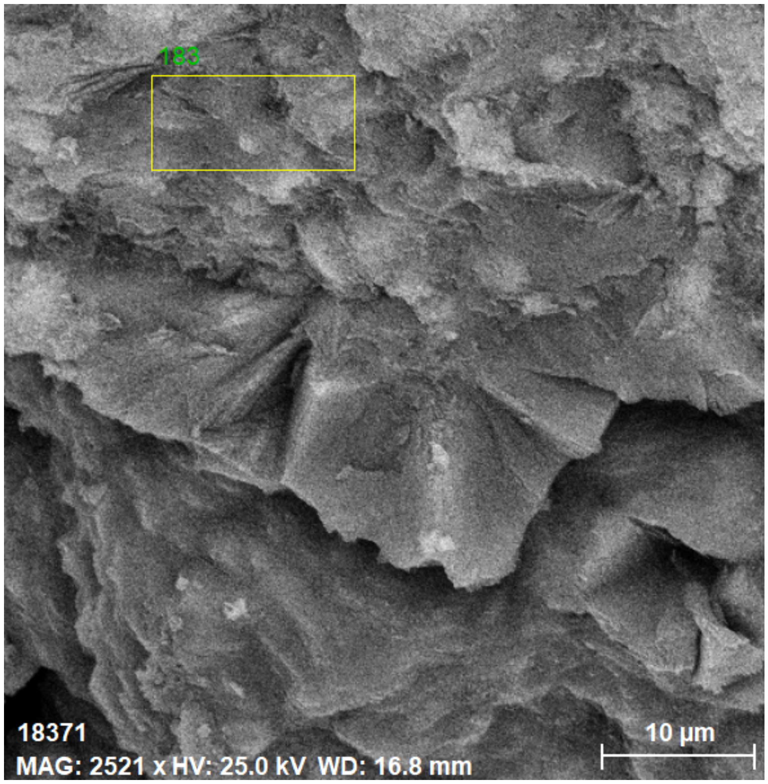
cps/eV
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
S
K
Cl
N
Au
Ca
O
Na
Mg
Si
Au
S
Cl
K
Ca
0
1
2
3
4
5
6
keV

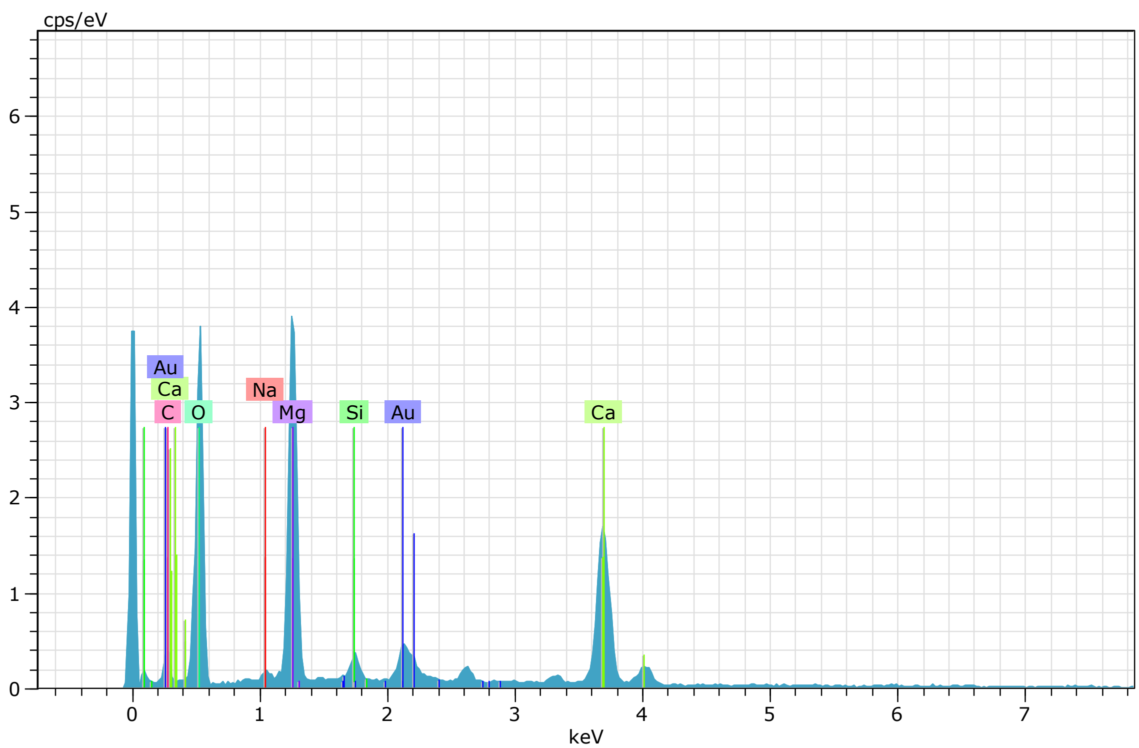


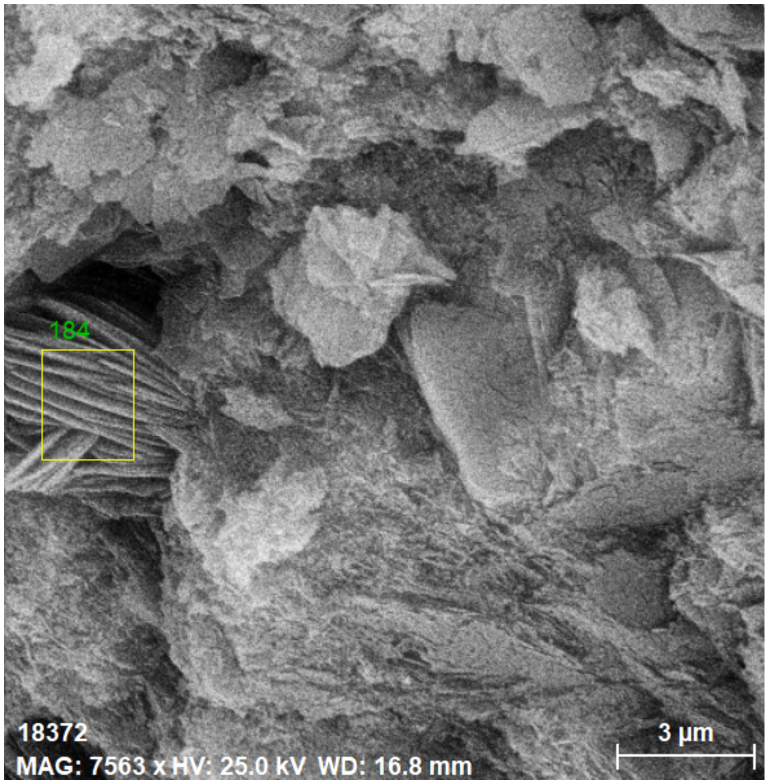
182
18371
MAG: 2521 x HV: 25.0 kV WD: 16.8 mm
10 µm

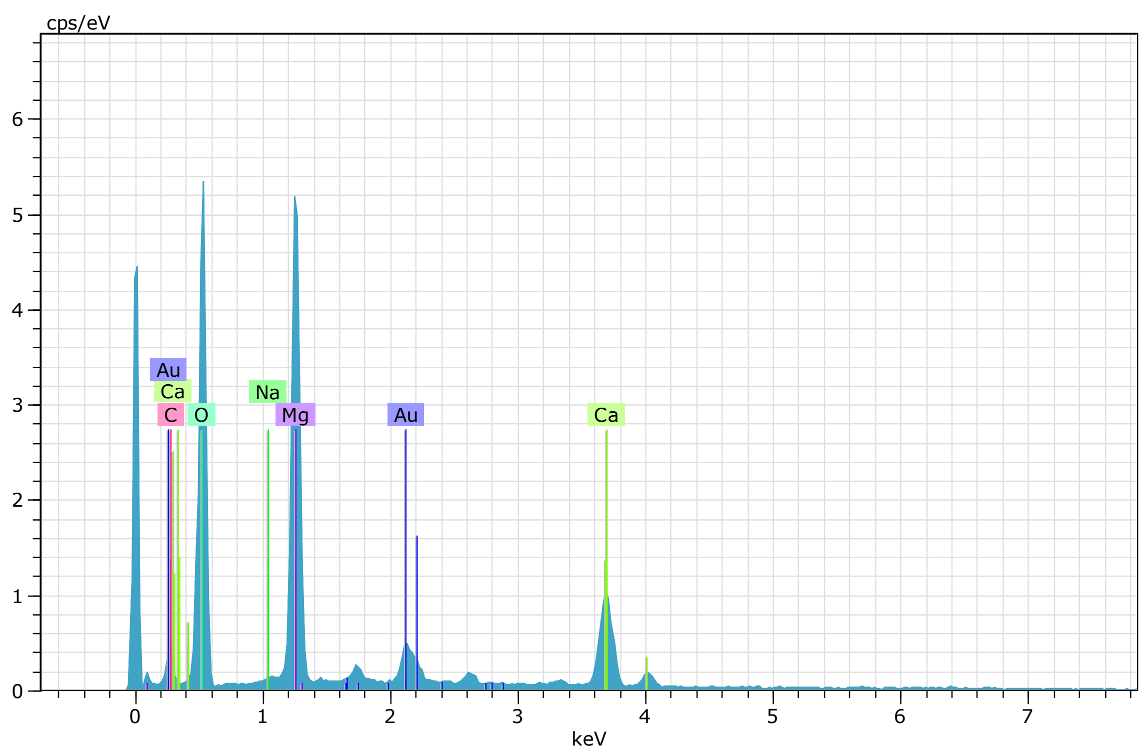


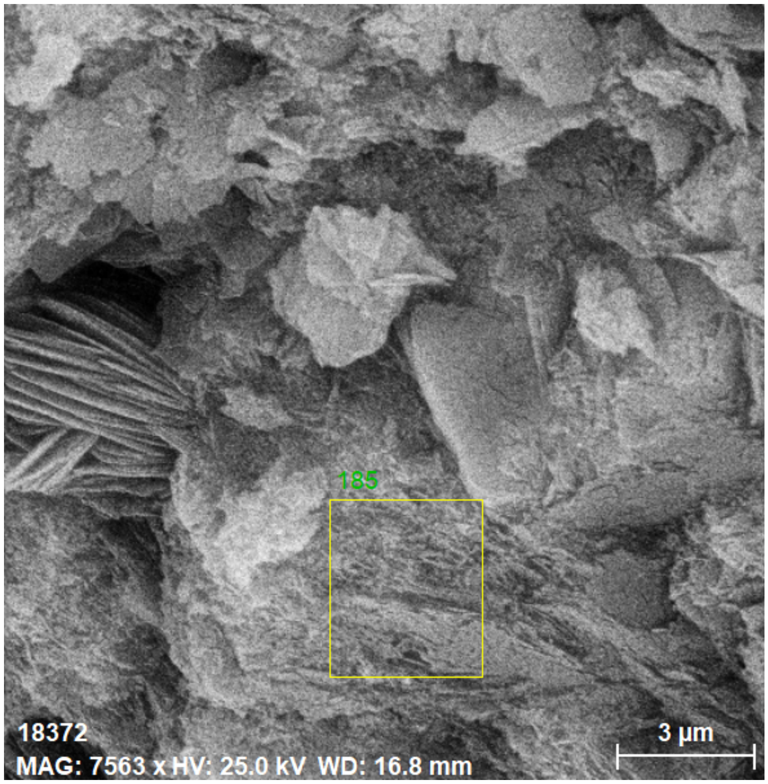
cps/eV
10
8
6
4
2
0
Au
C
O
Na
Mg
Au
0
1
2
3
4
5
6
7
keV

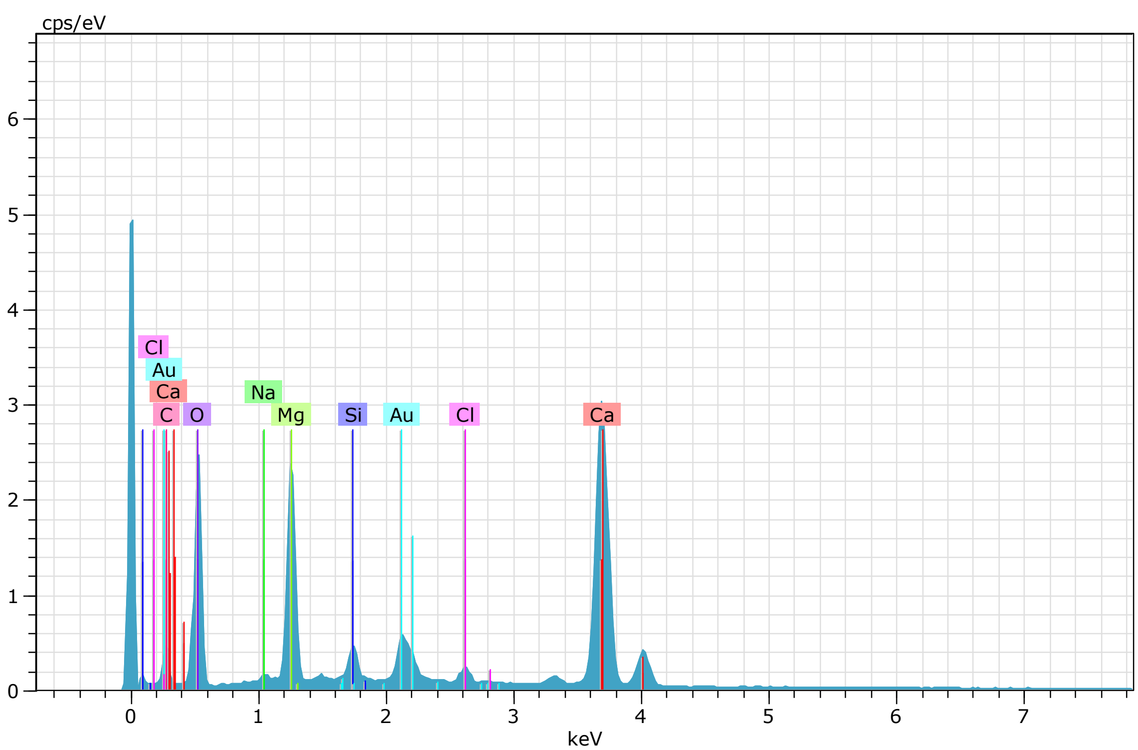

183
18371
MAG: 2521 x HV: 25.0 kV WD: 16.8 mm
10 µm

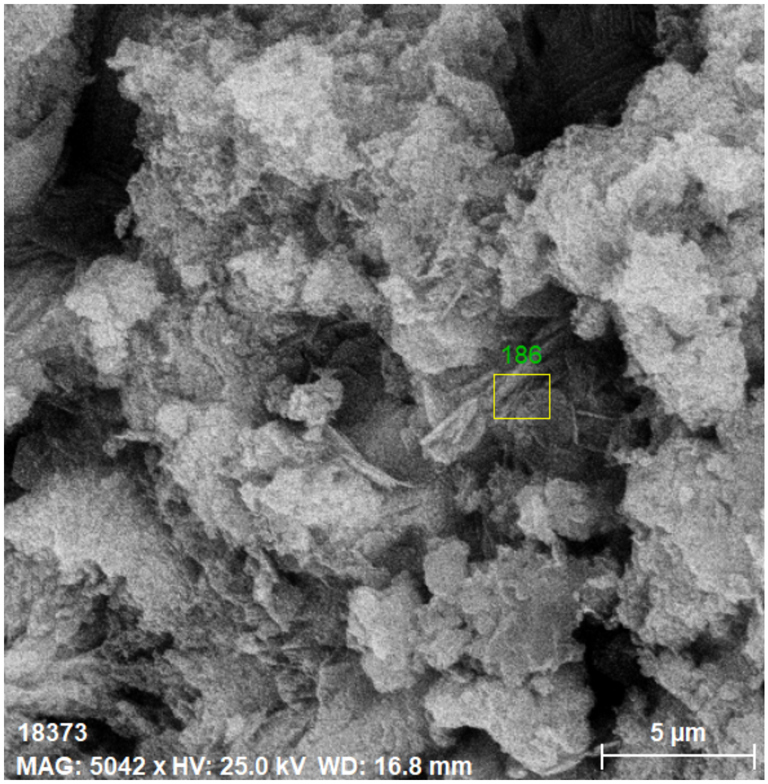

cps/eV
keV
Au
Ca
C
O
Na
Mg
Si
Au
Ca

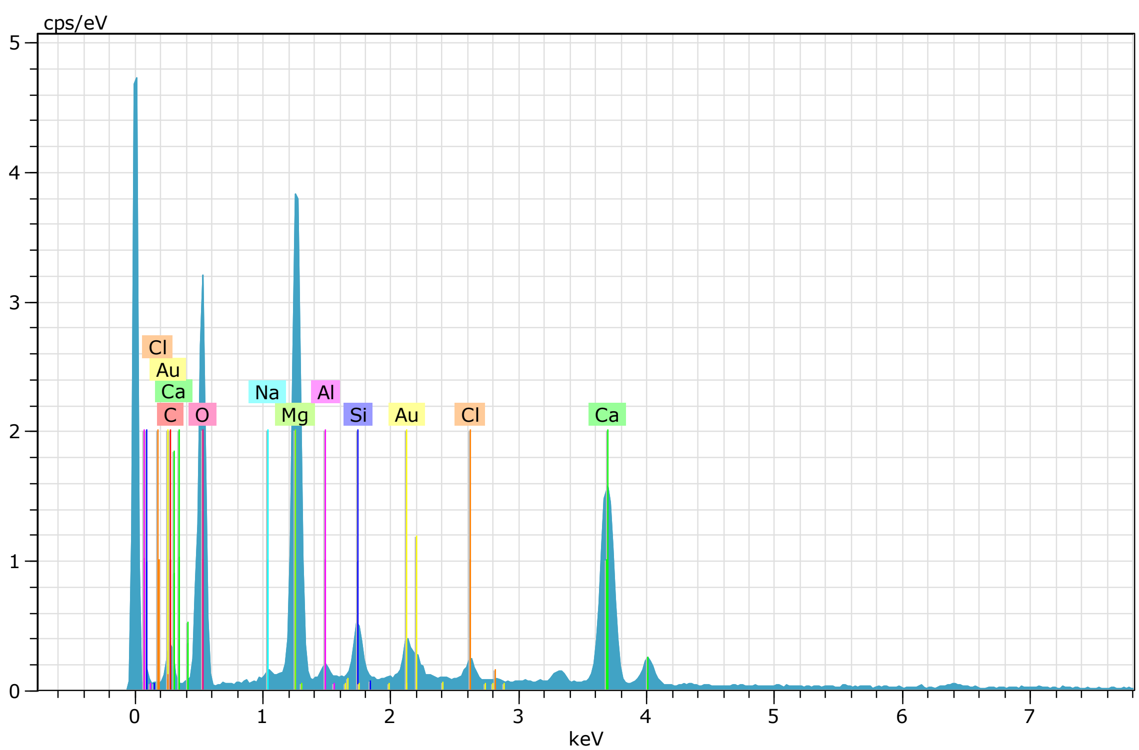

184
18372
MAG: 7563 x HV: 25.0 kV WD: 16.8 mm
3 µm

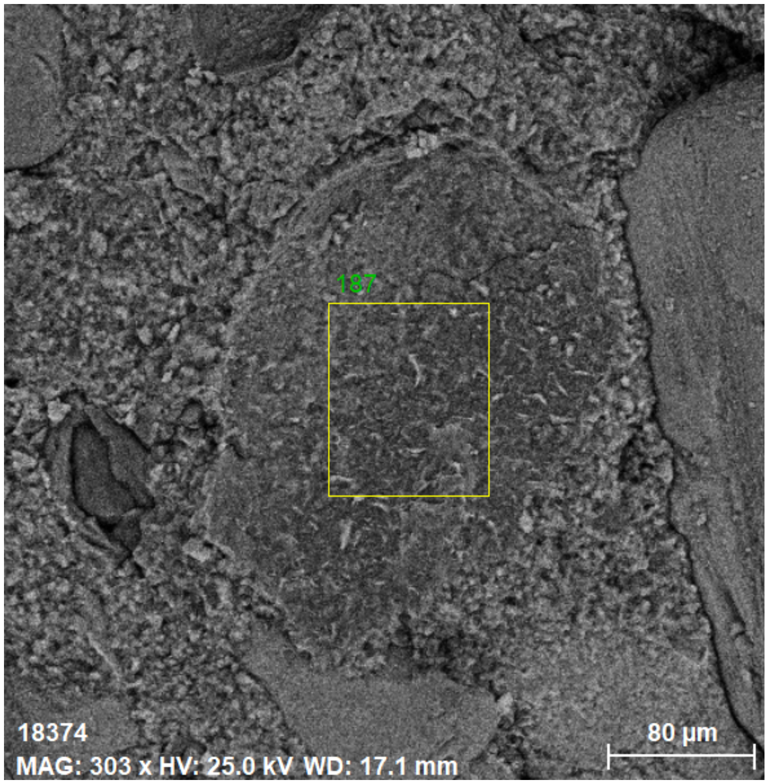

cps/eV
6
5
4
3
2
1
0
Au
Ca
C
O
Na
Mg
Au
Ca
0
1
2
3
4
5
6
7
keV

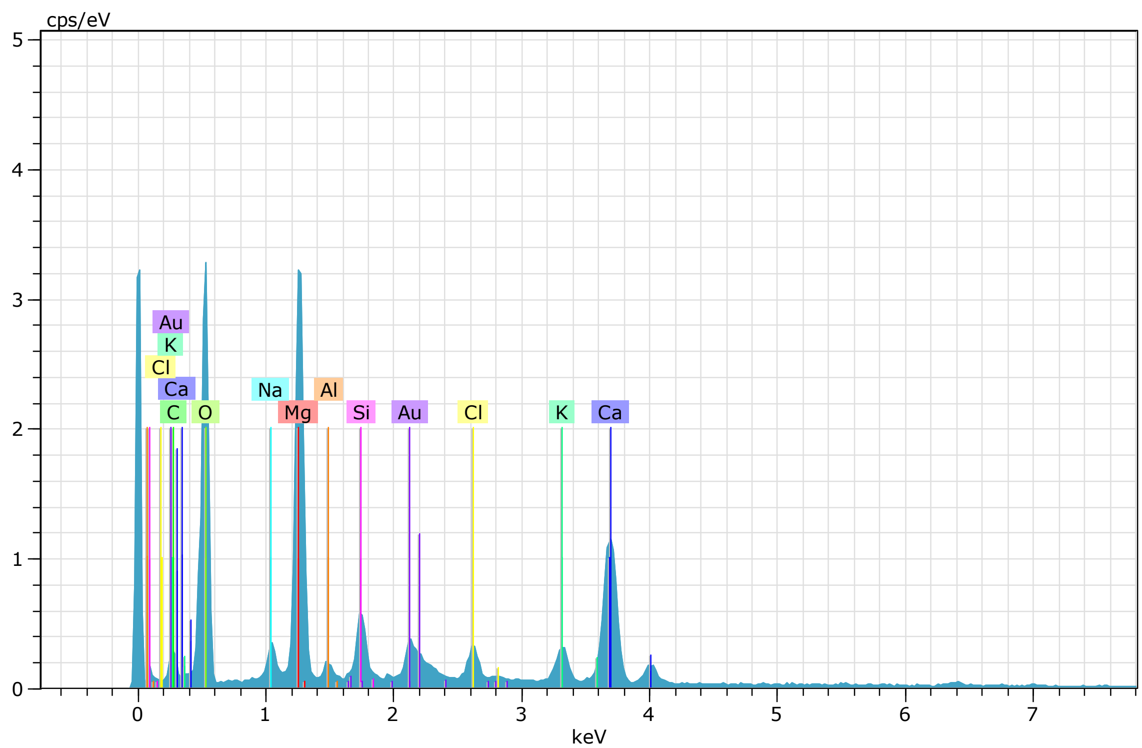

185
18372
3 µm
MAG: 7563 x HV: 25.0 kV WD: 16.8 mm

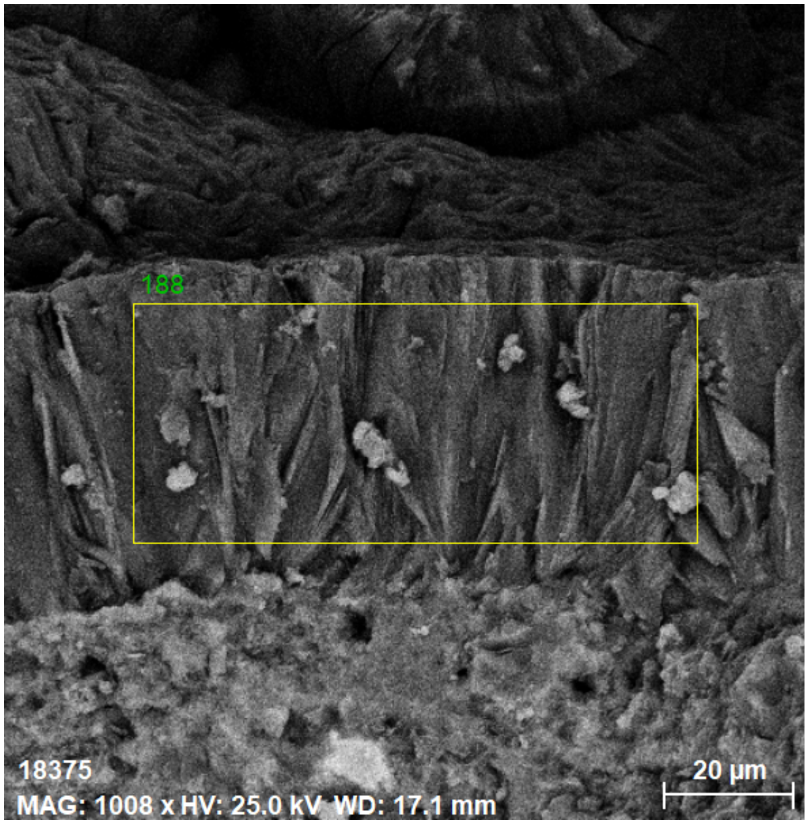

cps/eV
Cl
Au
Ca
C
O
Na
Mg
Si
Au
Cl
Ca
keV

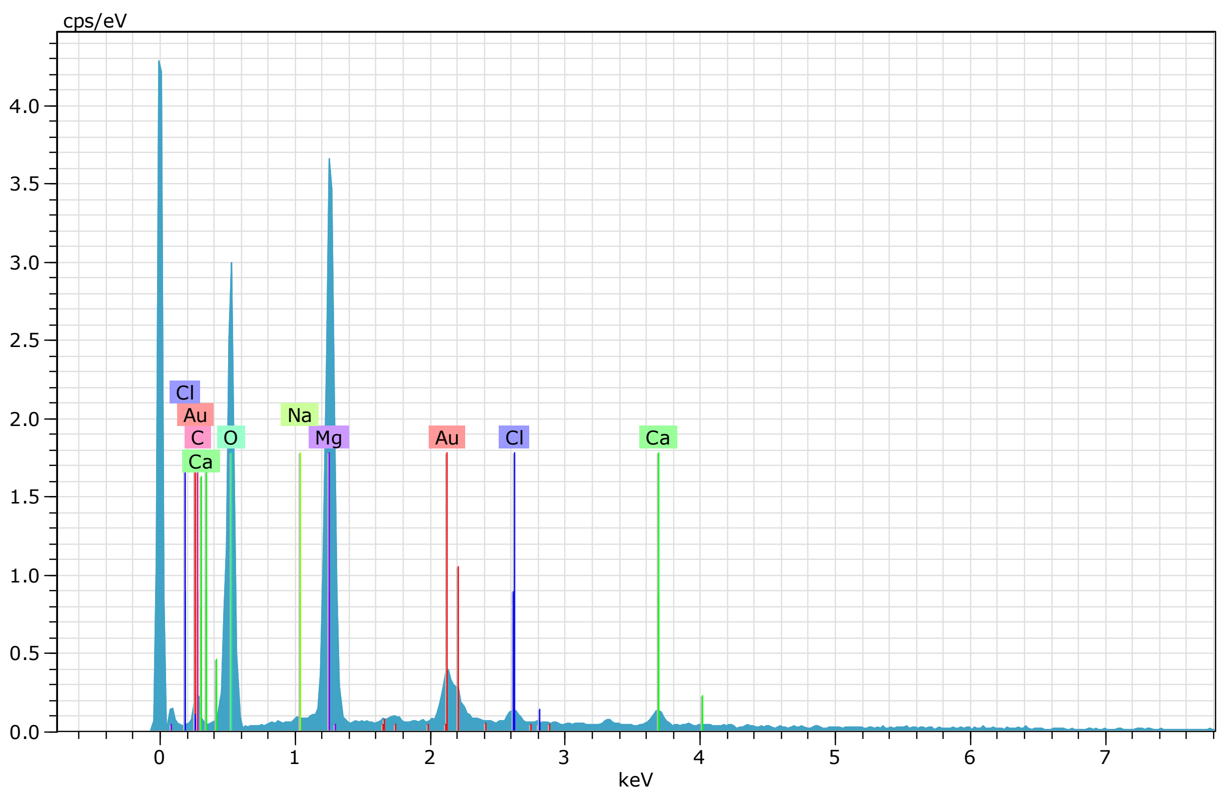

186
18373
MAG: 5042 x HV: 25.0 kV WD: 16.8 mm
5 µm

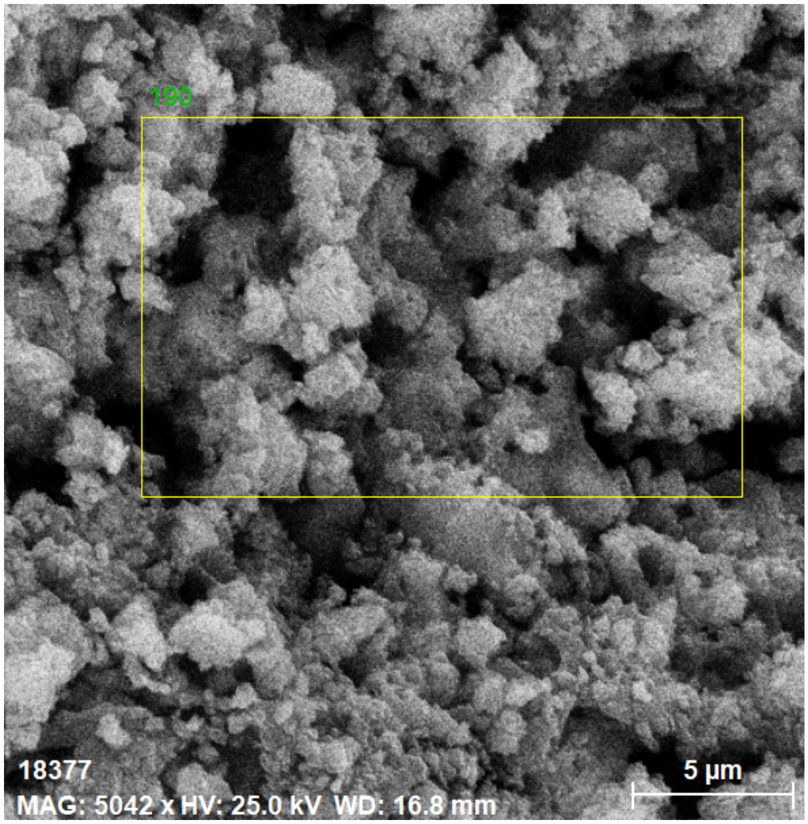

cps/eV
5
4
3
2
1
0
Cl
Au
Ca
C
O
Na
Mg
Al
Si
Au
Cl
Ca
0
1
2
3
4
5
6
7
keV

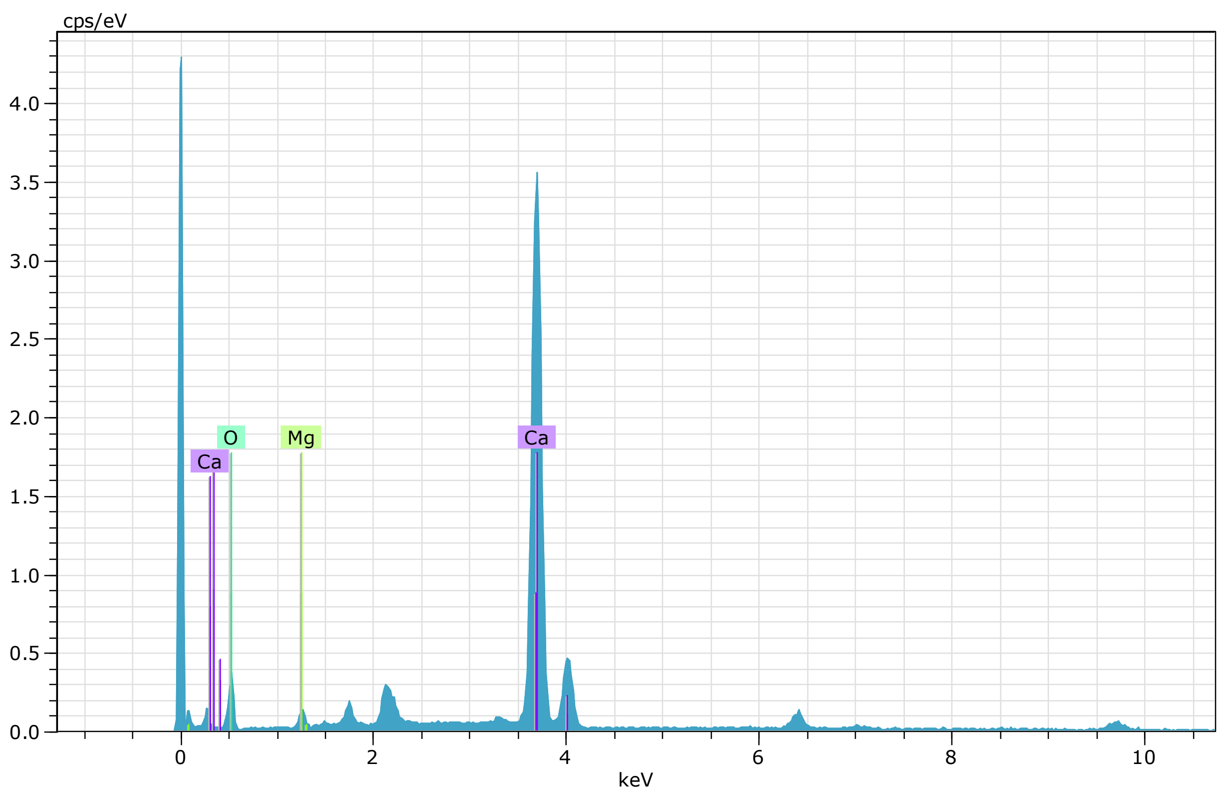

187
18374
MAG: 303 x HV: 25.0 kV WD: 17.1 mm
80 µm

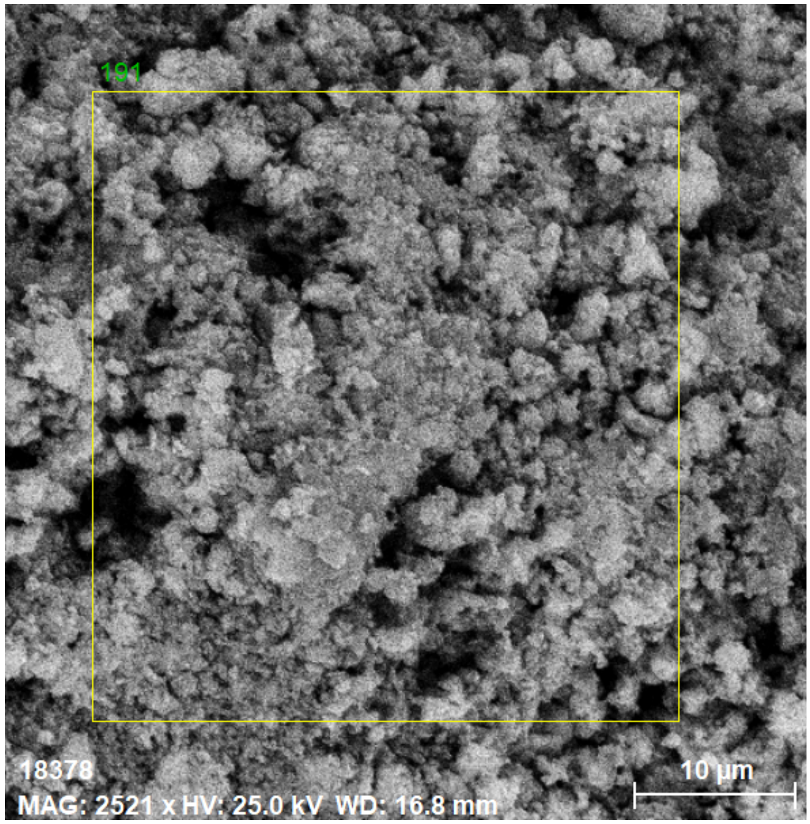

cps/eV
5
4
3
2
1
0
Au
K
Cl
Ca
C
O
Na
Al
Mg
Si
Au
Cl
K
Ca
0
1
2
3
4
5
6
7
keV


188
18375
MAG: 1008 x HV: 25.0 kV WD: 17.1 mm
20 µm

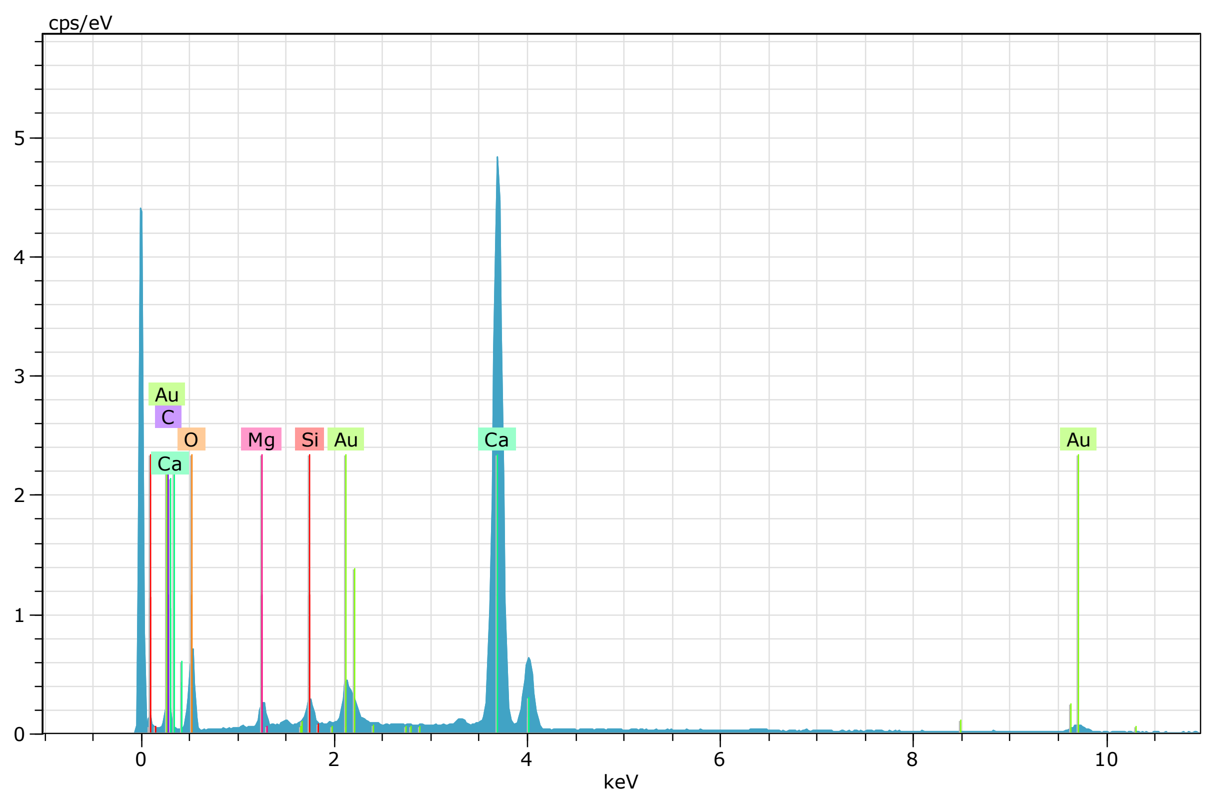

cps/eV
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Cl
Au
C
O
Ca
Na
Mg
Au
Cl
Ca
0
1
2
3
4
5
6
7
keV


190
18377
MAG: 5042 x HV: 25.0 kV WD: 16.8 mm
5 µm

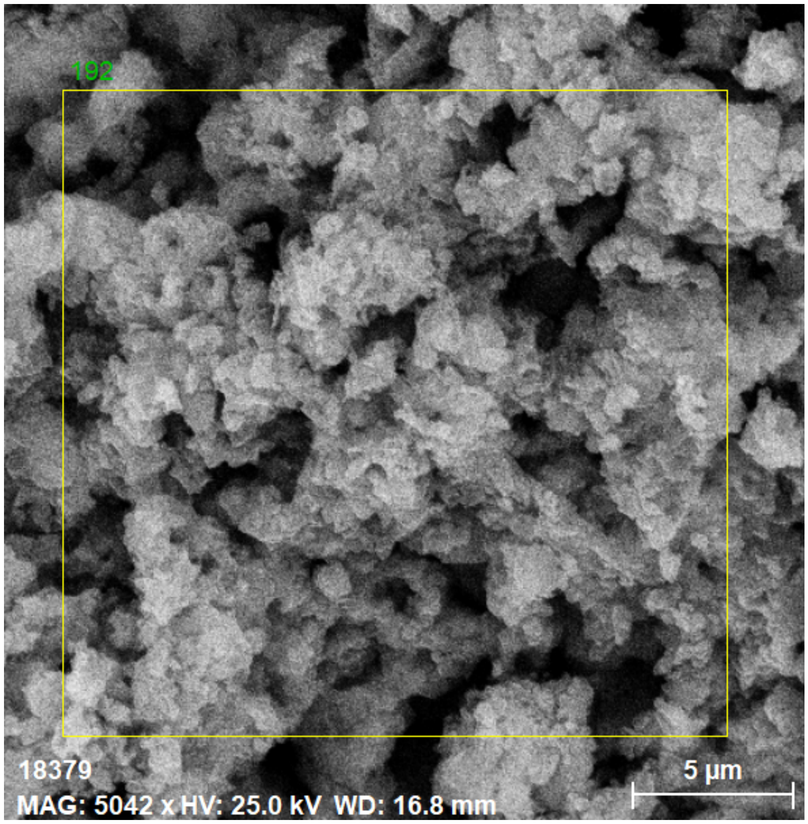

cps/eV
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
Ca
O
Mg
Ca
0
2
4
6
8
10
keV

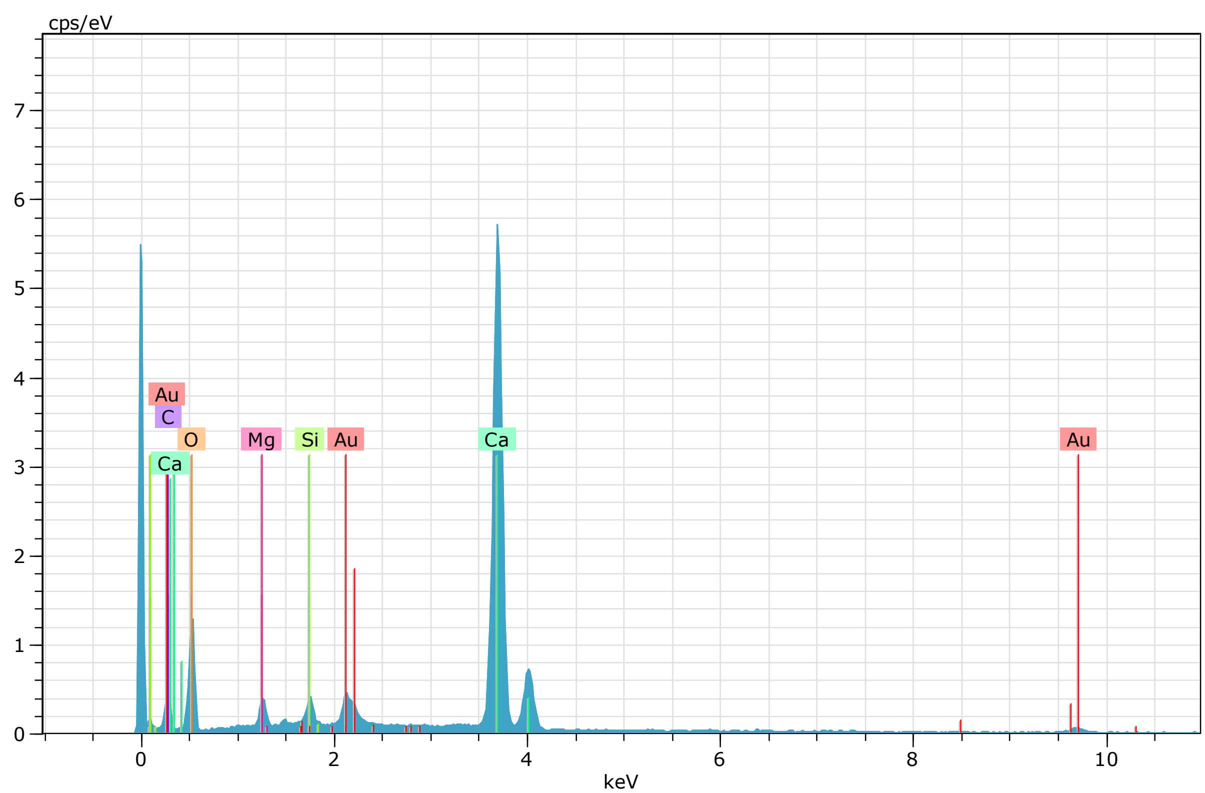


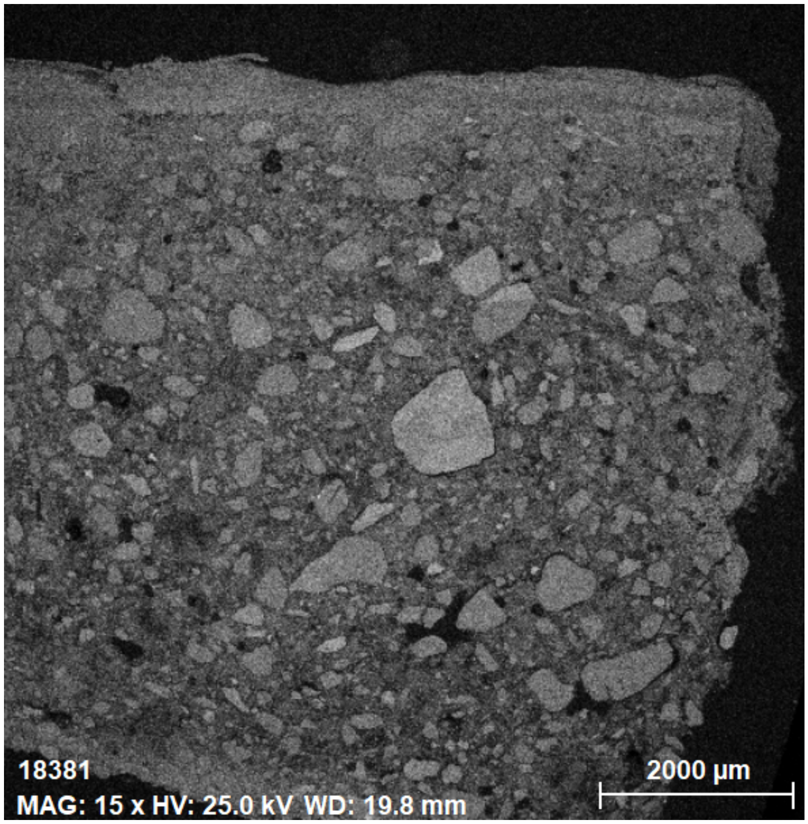
191
18378
MAG: 2521 x HV: 25.0 kV WD: 16.8 mm
10 µm

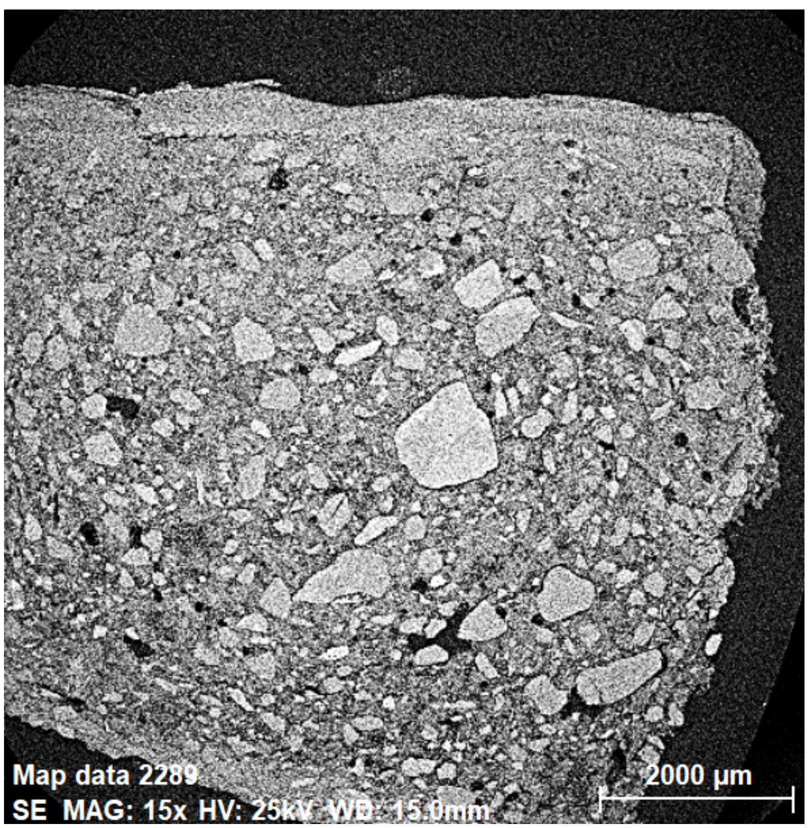


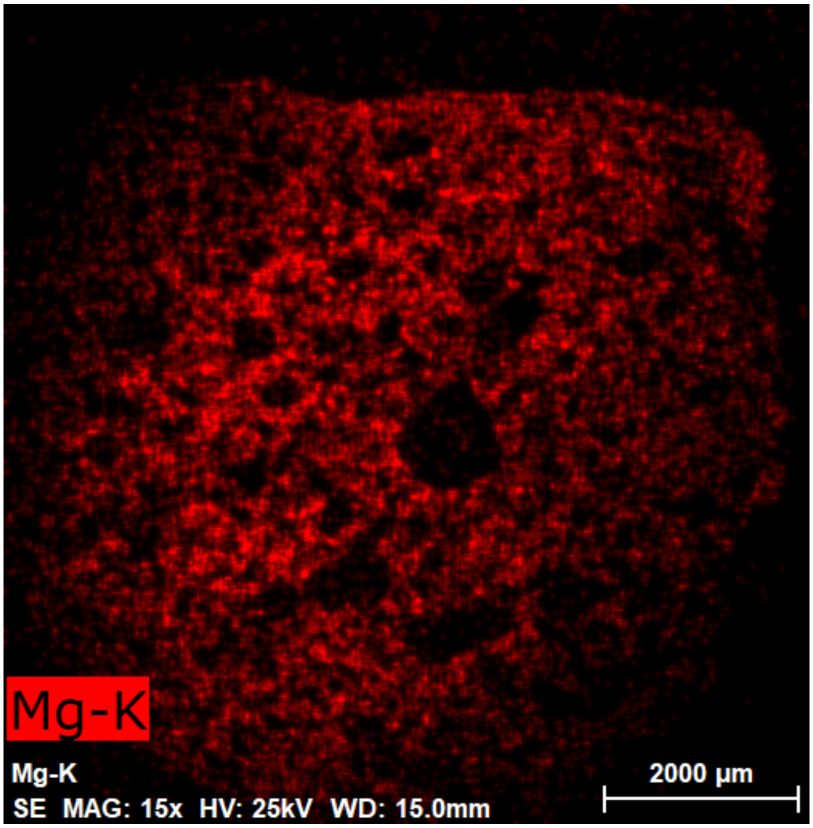
cps/eV
Au
C
O
Mg
Si
Au
Ca
Au
Ca
keV

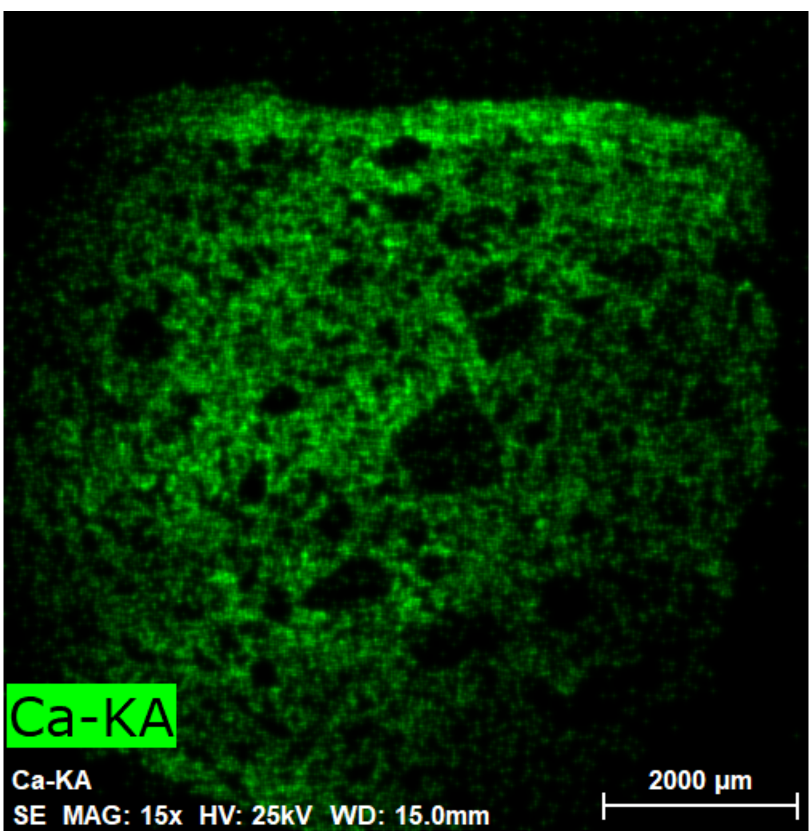

192
18379
MAG: 5042 x HV: 25.0 kV WD: 16.8 mm
5 µm

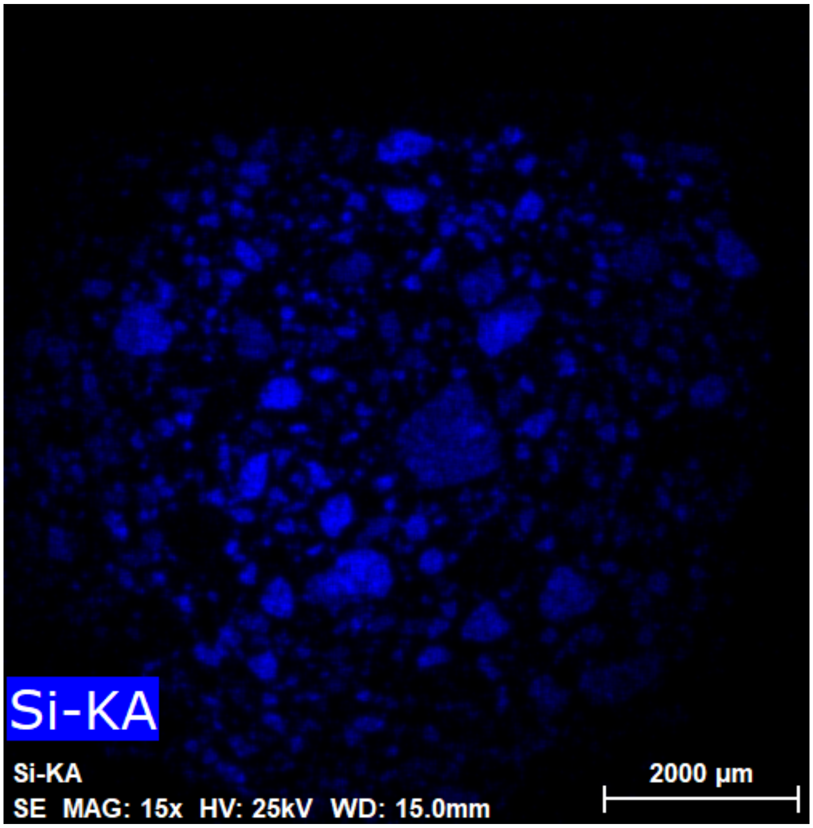

cps/eV
Au
C
O
Ca
Mg
Si
Au
Ca
Au
keV

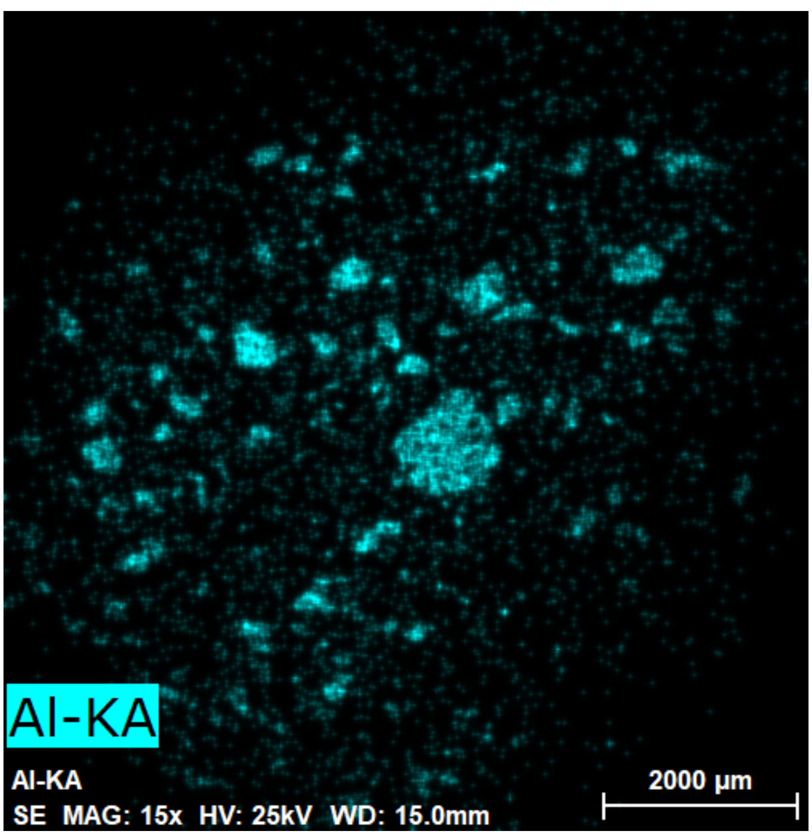

18381
2000 µm
MAG: 15 x HV: 25.0 kV WD: 19.8 mm

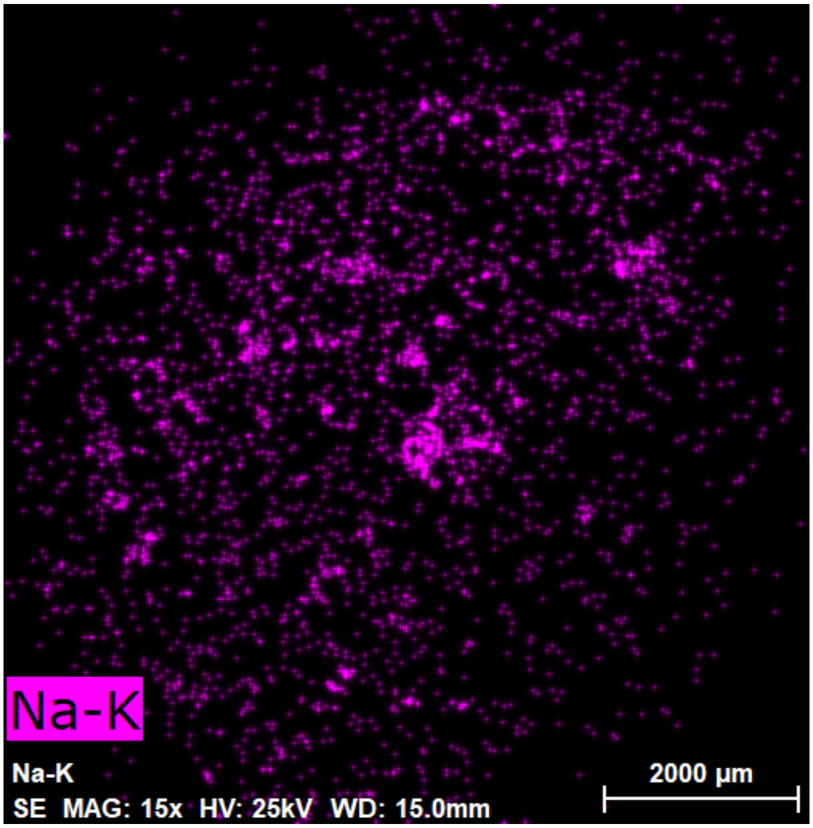

Map data 2289
2000 µm
SE MAG: 15x HV: 25kV WD: 15.0mm

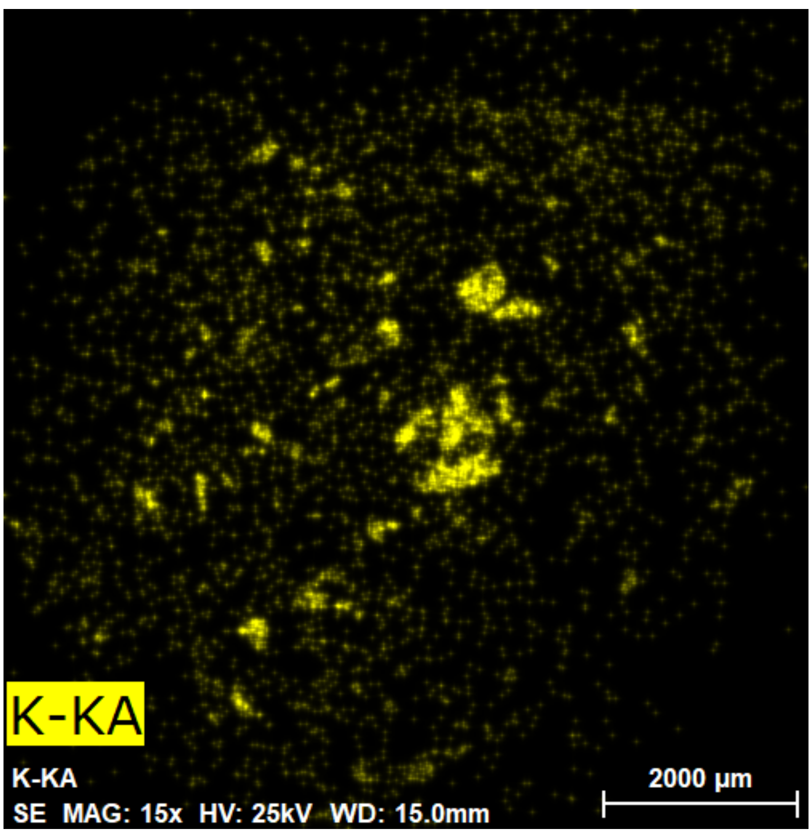

Mg-K
Mg-K
SE MAG: 15x HV: 25kV WD: 15.0mm
2000 µm

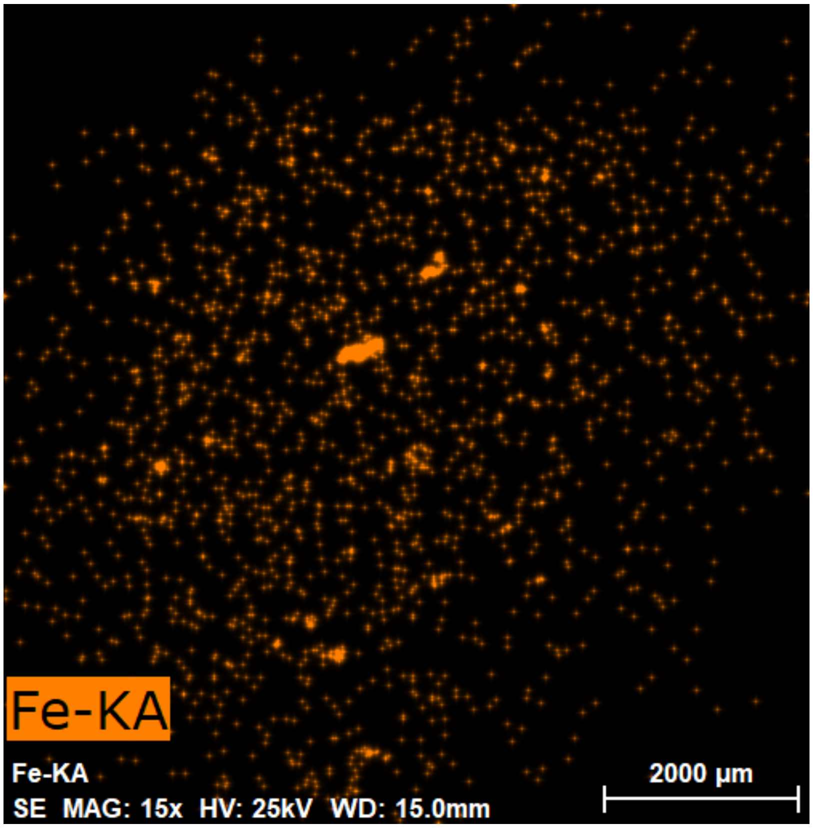

Ca-KA
Ca-KA
SE MAG: 15x HV: 25kV WD: 15.0mm
2000 µm

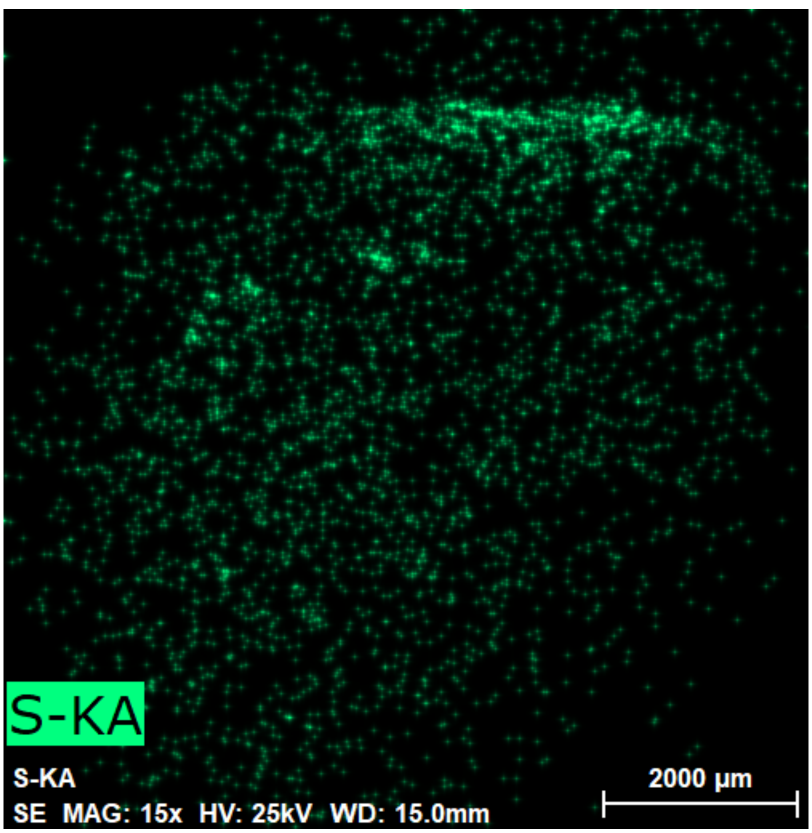

Si-KA
Si-KA
SE MAG: 15x HV: 25kV WD: 15.0mm
2000 µm

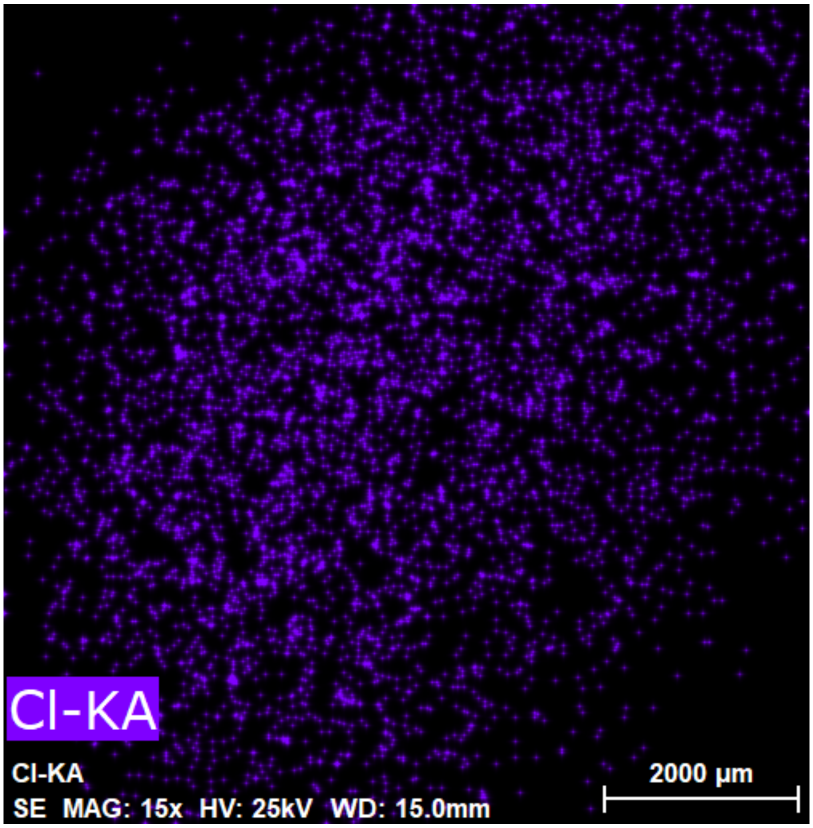

Al-KA
Al-KA
SE MAG: 15x HV: 25kV WD: 15.0mm
2000 µm


Na-K
Na-K
2000 µm
SE MAG: 15x HV: 25kV WD: 15.0mm


K-KA
K-KA
2000 µm
SE MAG: 15x HV: 25kV WD: 15.0mm


Fe-KA
Fe-KA
SE MAG: 15x HV: 25kV WD: 15.0mm
2000 µm


S-KA
S-KA
SE MAG: 15x HV: 25kV WD: 15.0mm
2000 µm


Cl-KA
Cl-KA
SE MAG: 15x HV: 25kV WD: 15.0mm
2000 µm

TV-W-1

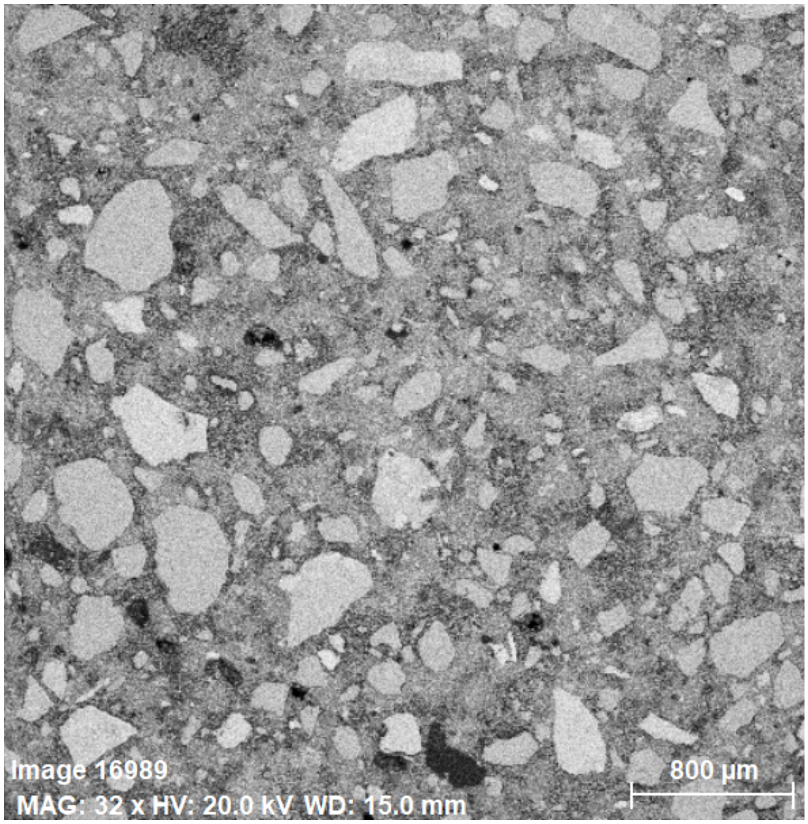
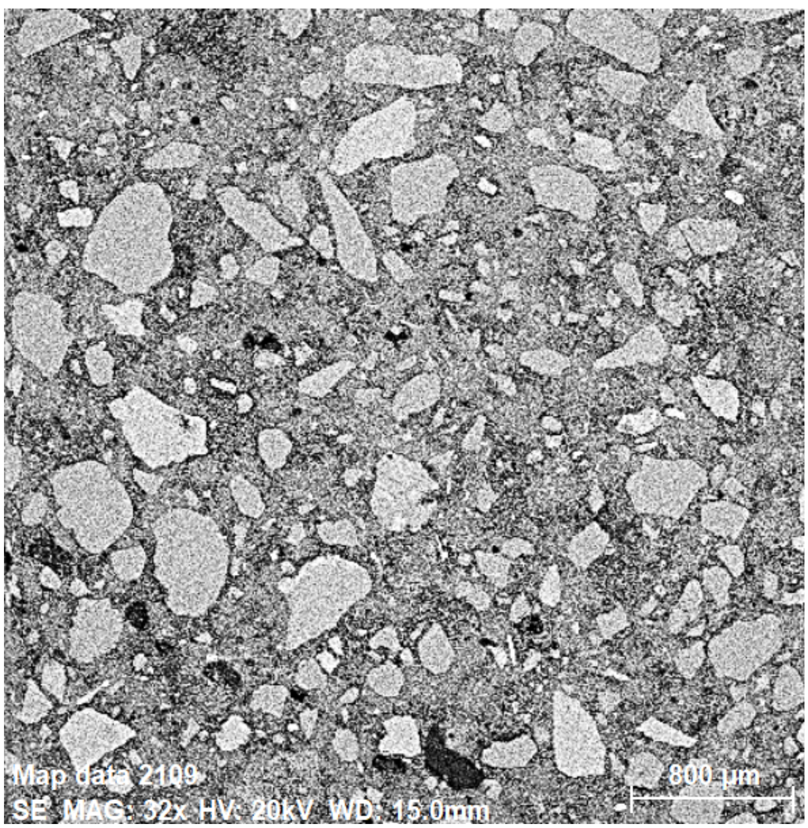
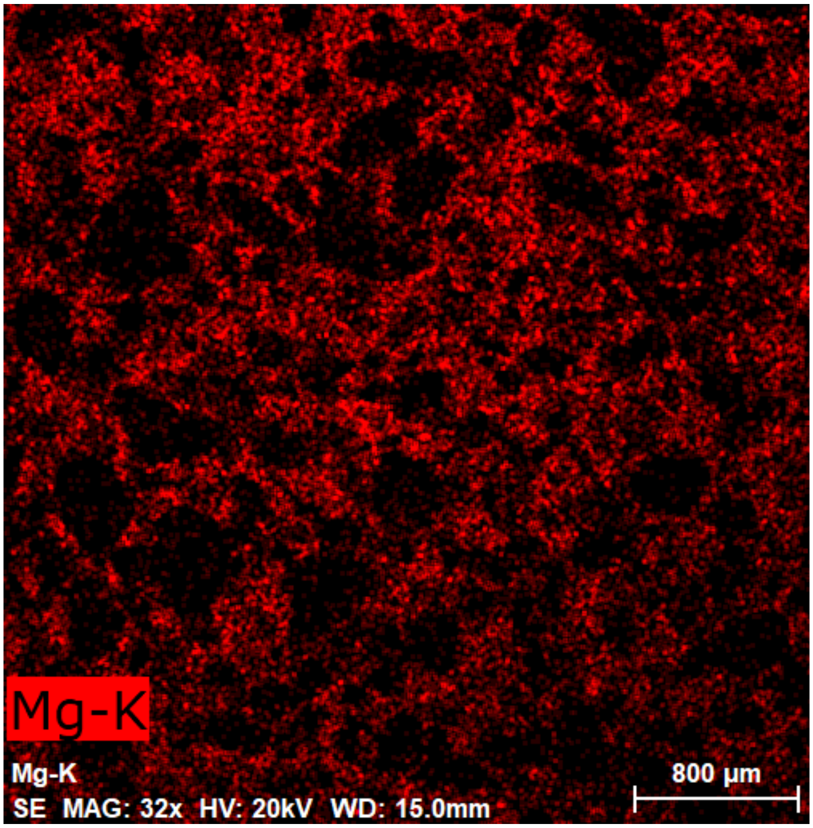
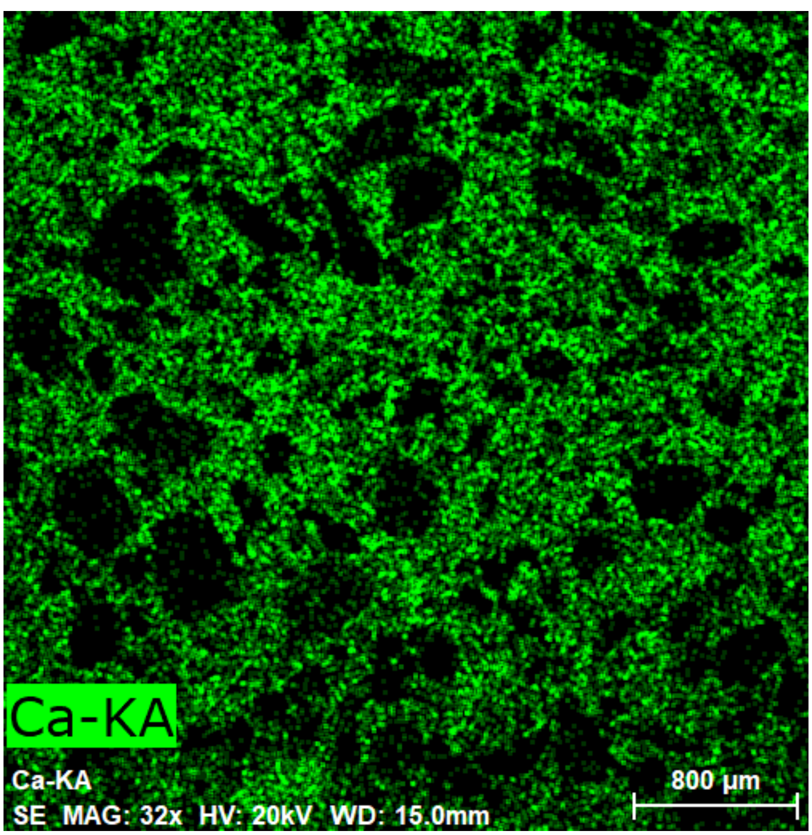
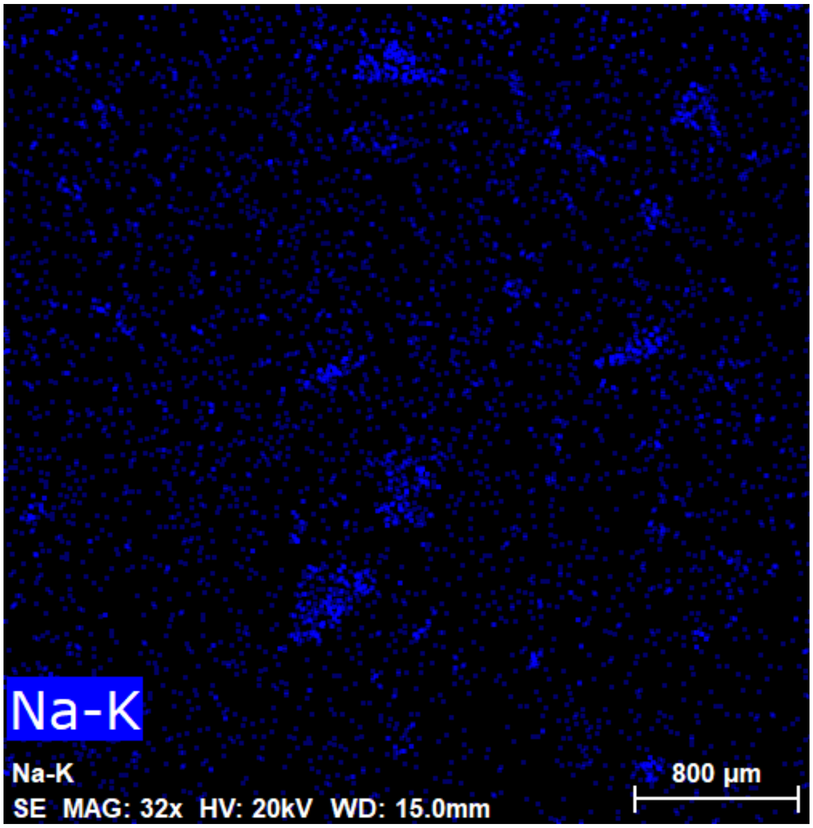


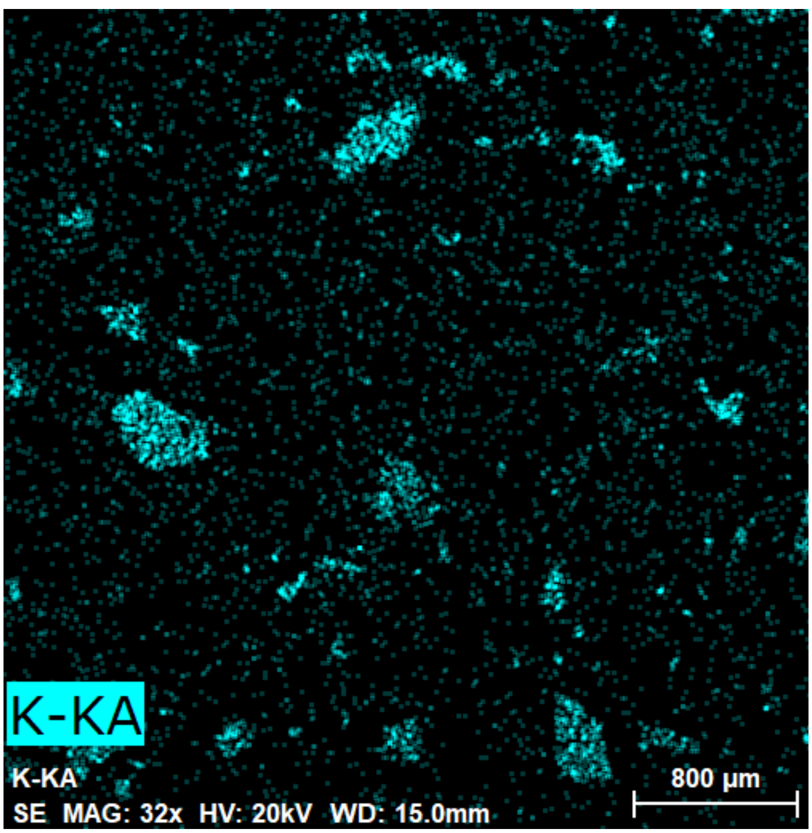
Image 16989
MAG: 32 x HV: 20.0 kV WD: 15.0 mm
800 µm

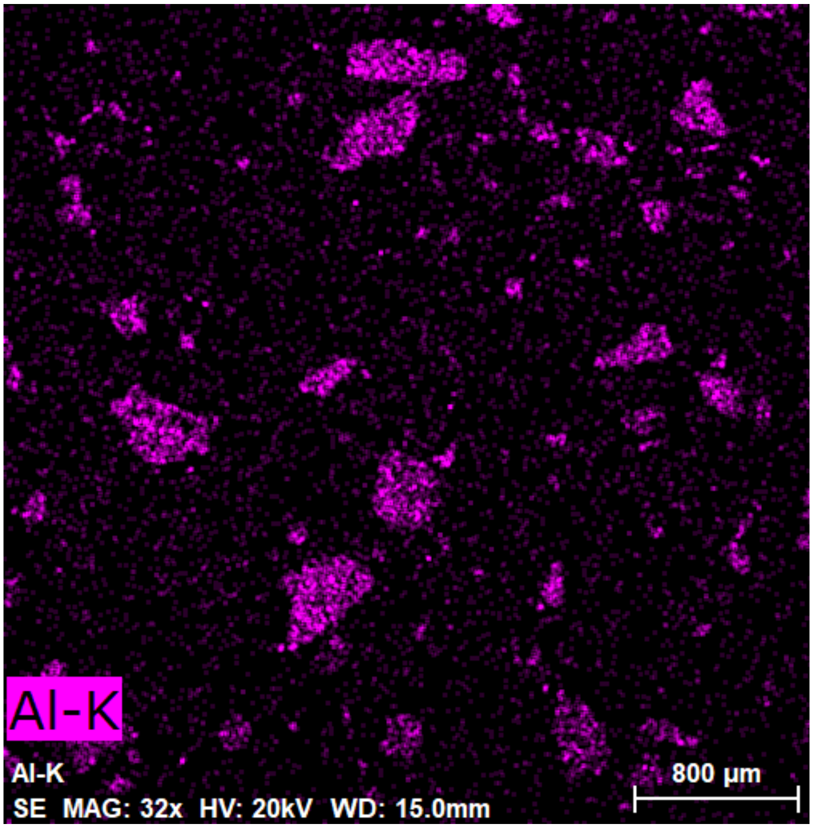

Map data 2109
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm

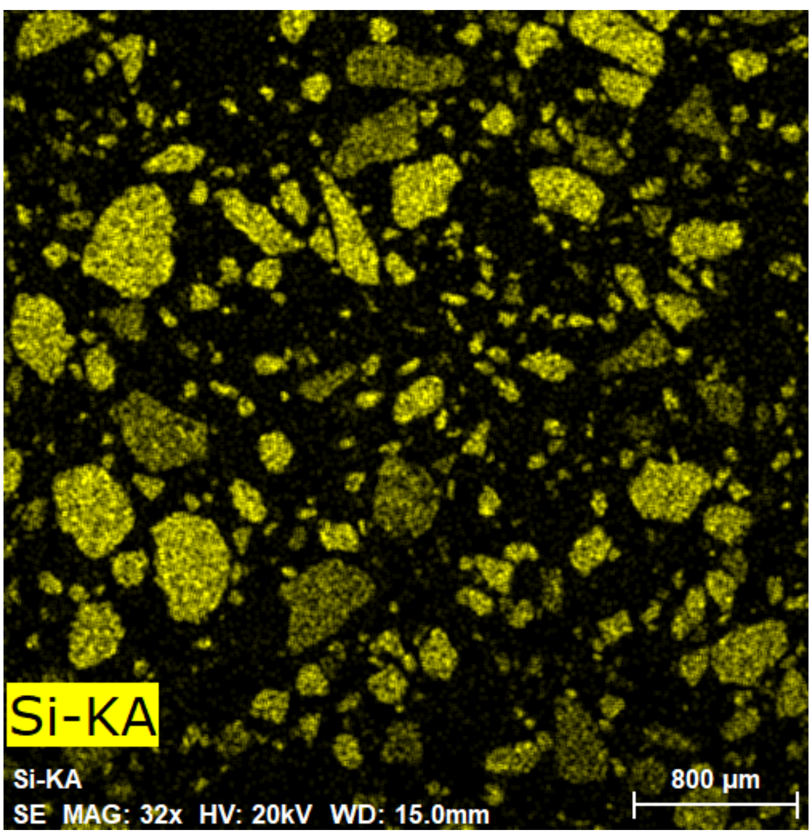

Mg-K
Mg-K
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm

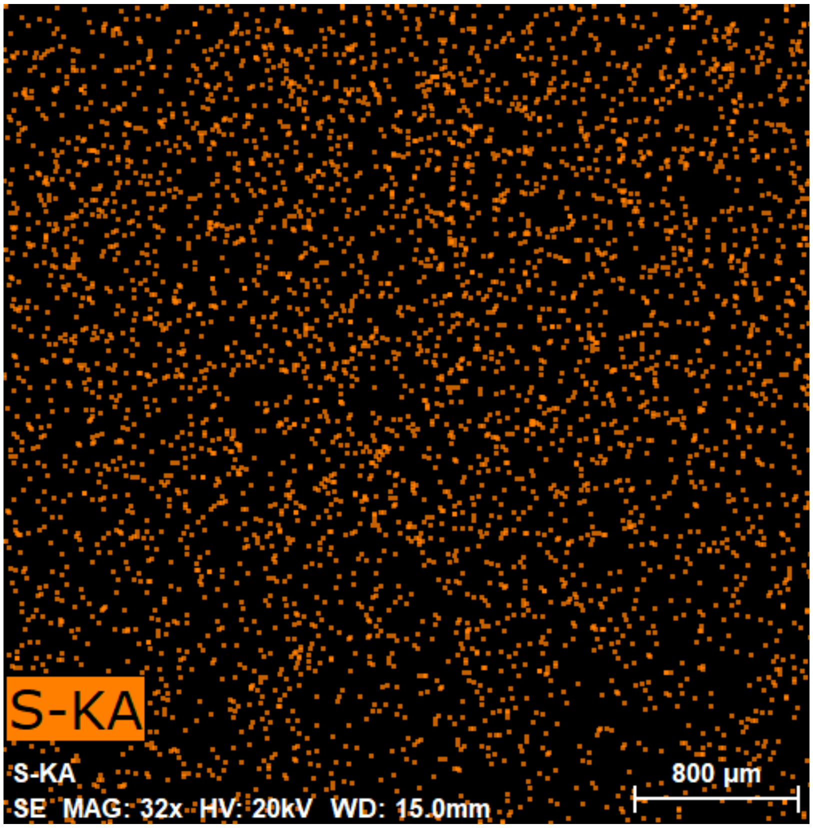

Ca-KA
Ca-KA
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm

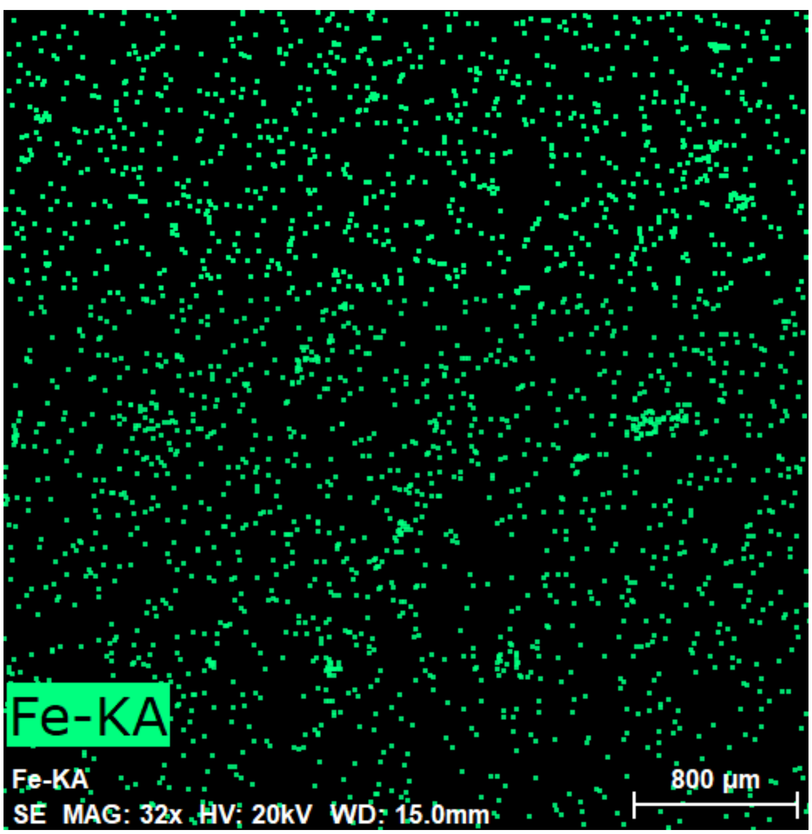

Na-K
Na-K
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm

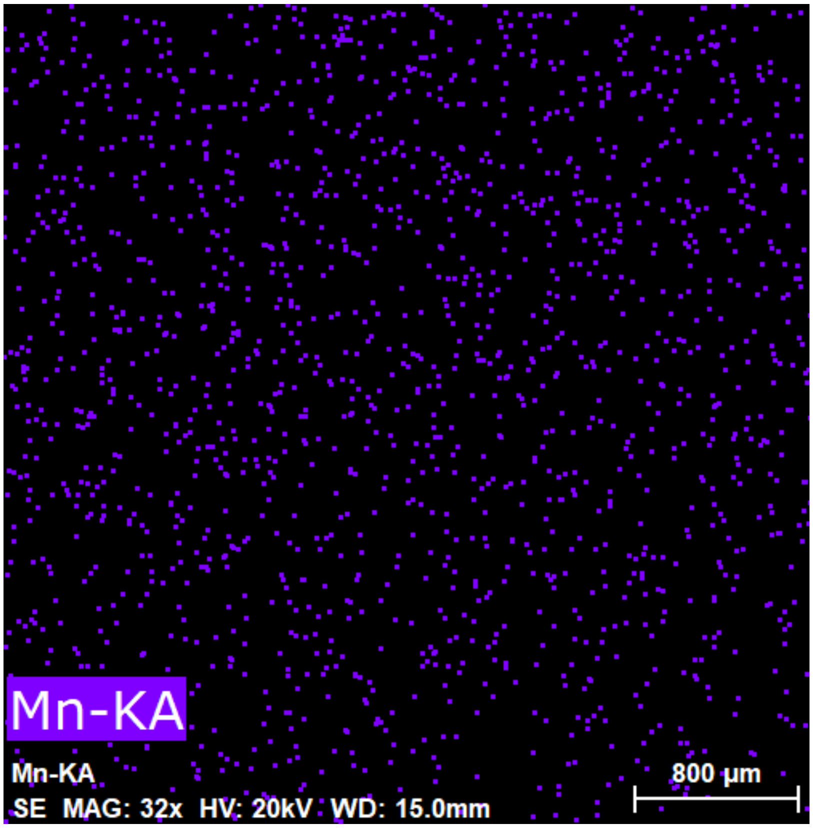

K-KA
K-KA
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm


Al-K
Al-K
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm


Si-KA
Si-KA
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm


S-KA
S-KA
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm


Fe-KA
Fe-KA
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm

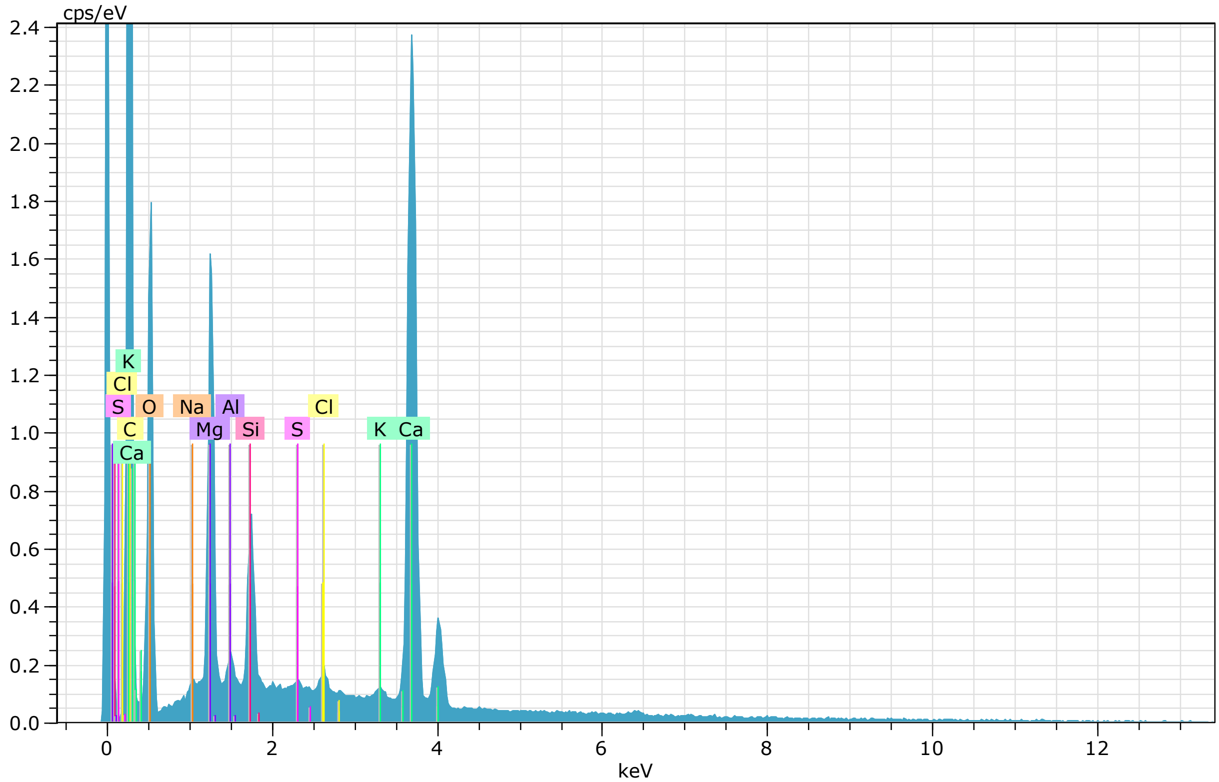

Mn-KA
Mn-KA
SE MAG: 32x HV: 20kV WD: 15.0mm
800 µm

Spectrum: 214

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Calcium	K-series	22.34	61.15	50.75	0.69
Magnesium	K-series	7.95	21.76	29.79	0.47
Silicon	K-series	2.57	7.05	8.35	0.14
Sulfur	K-series	0.42	1.16	1.20	0.05
Chlorine	K-series	1.05	2.88	2.70	0.07
Potassium	K-series	0.58	1.60	1.36	0.05
Sodium	K-series	0.73	2.01	2.90	0.08
Aluminium	K-series	0.88	2.40	2.96	0.08
	Total:	36.54	100.00	100.00	

Spectrum: 214

Element	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Compound	norm. Comp. C [wt.%]	Error (3 Sigma) [wt.%]
Calcium	17.91	40.30	24.41	CaO	56.39	1.66
Magnesium	6.78	15.26	15.24	MgO	25.31	1.21
Silicon	1.95	4.39	3.79	SiO2	9.38	0.34
Sulfur	0.25	0.57	0.43	SO3	1.42	0.11
Chlorine	0.78	1.75	1.20		1.75	0.17
Potassium	0.37	0.84	0.52	K2O	1.01	0.12
Sodium	0.67	1.50	1.58	Na2O	2.02	0.23
Aluminium	0.64	1.44	1.30	Al2O3	2.73	0.19
Oxygen	15.09	33.96	51.53		0.00	5.93
Total:	44.43	100.00	100.00			



Spectrum: 215

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Carbon	K-series	61.06	56.30	67.55	7.20
Oxygen	K-series	31.50	29.05	26.16	4.16
Calcium	K-series	10.53	9.71	3.49	0.34
Magnesium	K-series	3.67	3.39	2.01	0.23
Silicon	K-series	0.98	0.90	0.46	0.07
Sulfur	K-series	0.04	0.04	0.02	0.03
Chlorine	K-series	0.19	0.18	0.07	0.03
Sodium	K-series	0.17	0.15	0.10	0.04
Potassium	K-series	0.11	0.10	0.04	0.03
Aluminium	K-series	0.20	0.19	0.10	0.04
	Total:	108.44	100.00	100.00	

Spectrum: 215

Element	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Compound	norm. Comp. C [wt.%]	Error (3 Sigma) [wt.%]
Oxygen	15.48	34.59	52.10		0.00	6.17
Calcium	17.84	39.87	23.98	CaO	55.79	1.66
Magnesium	7.06	15.79	15.66	MgO	26.18	1.26
Silicon	2.24	5.00	4.29	SiO2	10.70	0.38
Sulfur	0.21	0.46	0.35	SO3	1.16	0.11
Chlorine	0.49	1.10	0.75		1.10	0.14
Sodium	0.49	1.09	1.15	Na2O	1.47	0.20
Potassium	0.24	0.53	0.33	K2O	0.64	0.11
Aluminium	0.70	1.57	1.40	Al2O3	2.96	0.20
Total:	44.75	100.00	100.00			

Appendix 2

Technical data sheets

Ledan TA1

Vapo Injekt

HYDRADUR NHL 5

OMYACARB limestone powder

Poraver

Liaver

Fillite 160 W

Tylose MH 300

Dispersion K9

Melment F 10

31020 Ledan® TA 1 Leit 03

Injection mortar to strengthen the surface of masonry walls, in pillars, ceilings and below surfaces with fresco paintings; at cavities and cracks of max. 5 mm.

Special Properties:

Excellent flowing ability, the underground does not have to be wetted; compatible and chemical-physical similar to lime and hydraulic lime; high mechanic stability, no blooming even in very humid surroundings and with lime and plasters.

Application:

Ledan® TA 1 can be applied to strengthen the surface of masonry wall structures. Further fields of application are:

- to strengthen masonry arches
- to strengthen masonry pillars
- to strengthen archeological walls
- to strengthen masonry walls with paintings

Composition of mortar:

Special chemically stable hydraulic binding agent components with minor amount of salts, silical powder, very fine Terra Pozzuoli which contains a special additive mixture to improve the fluidity, water containment and pore formation. This mixture is finely grounded and mixed. Ledan® TA 1 is pure white.

Mixture:

Mix Ledan® TA 1 thoroughly with demineralized water for about 3 minutes. It is recommended to filter the obtained pulp to remove possible clots.

Mixing recommendations:

Application	Ledan® TA 1	Water
Strengthening of loose surfaces:	10 kg	16 liter
Strengthening of masonry and arches:	10 kg	8 liter

Ledan® TA 1 can be mixed with sand. Not very absorbent marble dust or quartz powder is mixed with Ledan® TA 1 in a ratio of 1 : 1. This mixture can be further mixed in a ratio of 1 part Ledan® TA 1 with 7 parts of sand when the strength and bonding force should be reduced.

Recommended applications:

1. Close possible discharge openings of mortar with a reversible material.
2. Make suitable boreholes to apply the strengthening layer of Ledan® TA 1. The holes should have a diameter of at least 8 mm, the distance between the boreholes should not be greater than 50 cm. The depth of the boreholes should be at least 2/3 of the masonry.
3. The boreholes have to be cleaned thoroughly by blowing or exhausting.
4. The injection can be carried out by continuous application.

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kremer-pigmente@t-online.de, www.kremer-pigmente.com

After carefully making the boreholes and after removing all loose parts, Ledan® TAB 1 can be infused in the hollow sites without previous wetting or without any pressure.

Limited Areas of Application:

Ledan® TA 1 is a binding agent containing lime, thus the temperatures should not be below 5°C and not above 35°C when working with this mortar.

Properties:

	Value
Specific weight	1.40 g/cm³
Waiting time	5 min.
Start of hardening:	45 min.
End of hardening:	60 min.
Processability:	40 min.
Compressive strength:	13 N / mm²
Bending strength:	3.5 N / mm²
Adhesive strength:	1.4 N / mm²
Weeping:	insignificant
Vapor permeability:	9 μ
Elasticity modul:	11000 N / mm²
Retention of water:	0.6 %
Absorption capacity:	5.1 %

Blooming

Ledan® TA 1 does not cause blooming according to the Italian Standard RAL 544/3.

Stability

Stability tests have been carried out with samples of Ledan® TA 1 which correspond to an aging process of about 20 years. These tests showed a change of properties of less than 5 %.

References

The injection mortar Ledan® TA 1 has been used since 20 years in Italy, Germany and other European countries. The department of historical monuments in Materia (Church of Rupestri) and that of Etruria (Nekrople of Tarquinia) in Italy have successfully used Ledan® TA 1 and recommend this injection mortar for the applications listed above.

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TECHNICAL SHEET

VAPO INJEKT

LIME MIXTURES SYSTEM FOR RESTORATION OF PLASTERS
CZECH PRODUCT

Recommended use
Injection mixture for restoration of historic buildings.
Created for filling of thin cracks and hollows in and under plasters or within stone blocks, as well as gluing loose layers of lime paint, or stucco.

Characteristic properties
The injection mixture is based on a hydraulic lime bonding, micro milled and crushed limestone with grain size up to 2mm, and organic additive up to 1% volume.
The injection mixture does not segregate from the water, maintaining sufficient liquidity in order to flow freely into very narrow cracks. Does not change volume when drying and solidifying, does not produce any harmful salts.

Technical data
pH (in water) - 13
Weight (dry) - about 1000g/l
The mixture is sold in a plastic container preventing direct contact of the mixture with the air humidity. The longevity in unopened package is guaranteed up to 6 months.

Surface preparation
Prepare the surface by wetting with highly diluted injection mixture, lime water or clean water in order to get rid of the mild dust and dirt which would lower the adhesion quality. Apply on a wet surface. In a case of need, we recomment reinforcing the surface with POROSIL ZTS (in combination with the lime water) , or POROSIL Z from our production line.

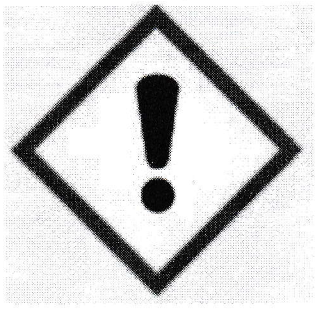
Application manual
Mix the dry mixture with the water in a 3:1 ratio, and stir for five minutes thoroughly. Adjust liquidity by adding water per need. Warning! Too much diluting will cause sedimentation of the solid particles.
Remove any leaks outside of the intended area of use immediately.
The workability of the mixture is about 4 hours at the temperature of 20°C

Climatic conditions
Use in temperatures above 5°C, stay clear of the direct sunlight. It is necessary to wet the treated surface after application, once per 24 hours.

Tools maintenance
Clean by water immediately.
Let the remaining material dry up and then dispose of freely.

Safety, handling and storage

Dangerous materials: calcium hydroxide CAS 1305-62-0
Pictogram: Xi Irritant



H315	Causes skin irritation
H318	Causes serious eye damage
P102	Keep out of reach of children
P305+P351+P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P280	Wear protective gloves/protective clothing/eye protection/face protection

Informations based on the current knowledge of the producer, proper laws and regulations must be adhered to at user's own responsibility.

Made and sold by AQUA obnova staveb s.r.o., Grafická 12,150 00 Praha 5, tel.: 257 312 636
www.aquabarta.cz **email: aquabarta@aquabarta.cz**

In Prague 09.02.2009

Dr. Ing. Jiří Rathouský, DrSc.
Ing. Zuzana Slížková
Ing. Arch. Jan Bárta

FASZINATION KALK ...

OTTERBEIN

... SEIT JAHRTAUSENDEN BEWÄHRT ®

Wer auf die Natur setzt,
kann auf uns bauen.

Technical Data Sheet

HYDRADUR® NHL 5

Natural Hydraulic Lime
EN 459-1 NHL 5

Mixing ratio for standard-type mortar in room sections:

Mortar for stonework		
Mortar group	HYDRADUR NHL 5	Sand
I	1	4.5
II	1	3
Plastering mortar		
PIIa	1	3 to 4

Composition
HYDRADUR® NHL 5 is a natural hydraulic lime acc. to EN 459-1. It is produced through burning and staking a specifically selected shell limestone. The stabilizing process of HYDRADUR® NHL 5 is effected through carbonation and hydraulic hardening.
HYDRADUR® NHL 5 is free from cement!

Properties
- historic, cement- and gypsum-free natural hydraulic binder
- high sulphate resistance
- low-tension hardening process
- very good subsequent hardening properties
- no penetration of damaging salts into the masonry
- high elasticity and water retention capacity in mortar produced
- very good side adhesion at stone
- low elasticity module

Application
For the production of plaster- and brick mortar in ecological, biological building construction, in the restoration and preservation of historic buildings.

Delivery
In 25 kg bag
In big bag
In silo vehicles

Storage
Dry, if possible on wooden shelves and protected against draft. Storage time shall not exceed 6 months.

Technical data

Bulk density:	approx. 0.55 kg/dm^3
free CaO:	approx. 30 %
SO_3:	approx. 0.5 %

Compression strength (acc. to EN 459-2):

28 days:	approx. 6.5 N/mm^2
6 months:	approx. 11.0 N/mm^2
12 months:	approx. 14.0 N/mm^2

Safety instructions
HYDRADUR® NHL 5 reacts strongly alkaline with water, thus: Protect skin and eyes, rinse thoroughly with water in case of contact, immediately contact doctor in case of eye contact.

Quality-monitored production
HYDRADUR® NHL 5 is continuously tested in our plant laboratory within the scope of our in-house monitoring with respect to the fulfilment of composition and properties. This will ensure a uniform quality of the product. HYDRADUR® NHL 5 has the quality certificate „Building lime“ and is certified acc. to EN 459-1.

OMYACARB 2 VA, 5 VA, 15 VA

Chemical name - $CaCO_3$

Form - fine powder of white colour

Physico-chemical properties - Carbonate filler Omyacarb is clearly white marble powder acquired by grinding, milling and sorting of a natural crystallic calcite.

Usage - It is used in production of paints, bindings, rubber and plastic mixtures, building materials, disperse and mineral plasters, fine ceramic etc.

Packing - 2VA - 40 kg, 5VA - 50 kg, 15VA - 50 kg paper bags, 1 pallet / 1200 kg

Technical characteristic / Technická charakteristika		2VA	5VA	15-VA
$CaCO_3 + MgCO_3$	% min	98,5	98,5	98,5
from that $MgCO_3$ / z toho $MgCO_3$	% max	0,8	0,8	0,8
SiO_2	% max	0,8	0,8	0,8
$Al_2O_3 + Fe_2O_3$	% max	0,4	0,4	0,4
from that Fe_2O_3 / z toho Fe_2O_3	% max	0,1	0,1	0,1
Insoluble residue in HCl / nerozpustný zbytek v HCL	% max	1,5	1,5	1,5
MnO	% max	0,01	0,01	0,01
SO_3	% max	0,05	0,05	0,05
Whiteness Ry / Bělost Ry	% min	93,7 ±1,2	92±2	88-93
Average particles size / Průměrná velikost částic	µm	2	5	12
Moisture / Vlhkost	% max	0,2	0,3	0,5

OMYACARB 2 VA, 5 VA, 15 VA

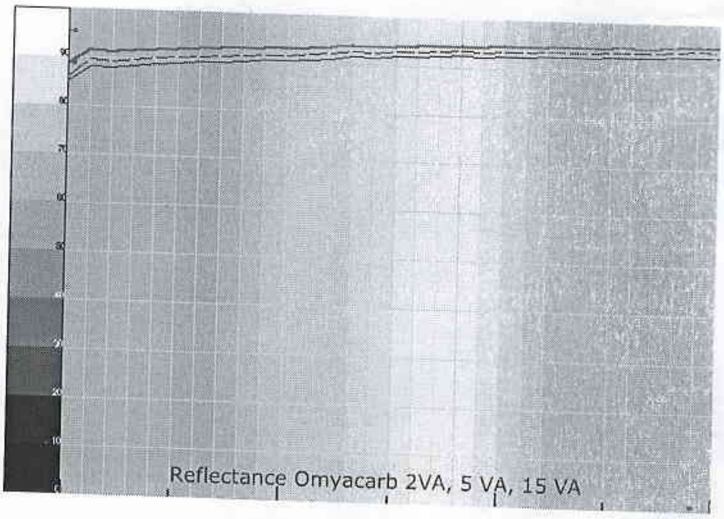
Chemický vzorec - $CaCO_3$

Forma - jemný prášek bílé barvy

Fyzikálně-chemické vlastnosti - Karbonátové plnidlo Omyacarb je čistě bílá mramorová moučka získaná drcením, mletím a tříděním přírodního krystalického vápence.

Použití - Používá se k výrobě barev, tmelů, gumárenských a plastikářských směsí, stavebních hmot, disperzních a minerálních omítek, jemné keramiky atd.

Balení - 2VA - 40 kg, 5VA - 50 kg, 15VA - 50 kg papírové pytle, 1 paleta / 1200 kg



Reflectance Omyacarb 2VA, 5 VA, 15 VA

poraver® expanded glass

TECHNICAL DATA SHEET

Lightweight aggregate according to DIN EN 13055-1

Poraver® expanded glass	BASIC GRANULAR SIZES					SPECIAL GRANULAR SIZES	
Granular size in mm	**0.1-0.3**	**0.25-0.5**	**0.5-1**	**1-2**	**2-4**	**0.04-0.125**	**0.5-1.25**
Bulk density in kg/m^3	400 ± 60	340 ± 30	270 ± 30	230 ± 30	190 ± 20	530 ± 70	260 ± 30
Particle density in kg/m^3	950 [1)] ± 150	700 [1)] ± 80	500 [1)] ± 80	400 [1)] ± 60	320 [2)] ± 40	1400 [3)] ± 300	490 [1)] ± 80
Crushing resistance in N/mm^2 according to DIN EN 13055-1 [4)]	≥ 4.5	≥ 2.6	≥ 2.0	≥ 1.6	≥ 1.4	-	≥ 1.9
Oversize % by mass	≤ 10						
Undersize % by mass	≤ 15						
pH value	8 - 12						
Moisture content % by mass	≤ 0.5						
Water absorption % by volume 5 min. [5)]	33	15	9	7	4.5	- [6)]	10
Water absorption % by mass 5 min. [5)]	35	21	18	19	14	- [6)]	20
Softening point	approx. 700°C						
Colour	creamy white						
Thermal conductivity W/(m·K)	-	-	-	-	0.07 [7)]	-	-
CE according DIN EN 13055-1	•	•	•	•	•	-	•
Approval Z-3.42-1894	•	•	•	•	•	-	•
Approval Z-23.11-114	-	-	-	-	•	-	-

1) Test according to DIN V 18004, calculation of apparent (relative) density see method DIN EN 1097-6
2) Apparent (relative) density according to EN 1097-6
3) Density of filler according to EN 1097-7
4) Values according to DIN V 18004 on request
5) Approximate values due to possible measurement tolerances
6) Values not determinable due to the fineness of the material
7) Calculated values DIBt according to Approval Z-23.11-114 (Thermal insulating material, non combustible according to construction material class DIN 4102-A1)

The strength grades may vary within the tolerance range of bulk densities. The availability and delivery conditions for special grain sizes will be agreed on an individual basis.

CHEMICAL ANALYSIS

Constituent	Applied to the sample dried at 105°C	Analysis method
SiO_2	70 - 75 %	DIN EN ISO 12677 measured with XRF
Na_2O	10 - 15 %	
CaO	7 - 11 %	
Al_2O_3	0.5 - 5 %	
MgO	0 - 5 %	
K_2O	0 - 4 %	
Loss on ignition	< 0,1 %	

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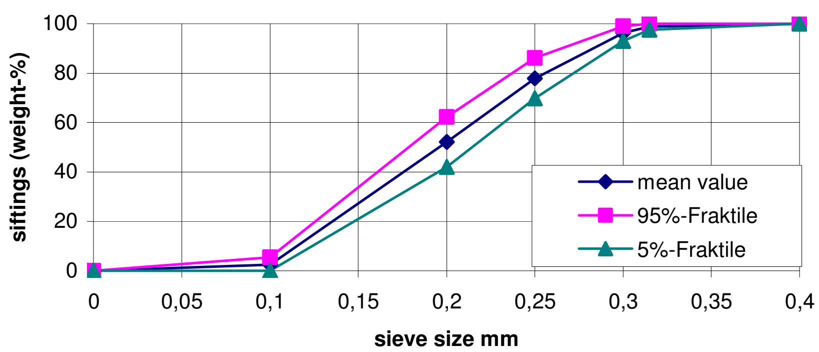
Data Sheet

Type	Liaver 0.1-0.3mm	Declaration siftings 100% < 0.4mm 90% < 0.3mm
Main properties		
Kind of material	Expanded glass granules	Leightweight aggregate
Shape	spheric, closed surface	according to EN 13055-1
Geometrical properties		**Test method**
Grain size	0-0.5 mm	EN 933-1
Dust (< 0.063mm)	< 1 %	EN 933-1
Physical properties		
Loose bulk density	450 ± 15% kg/m³	EN 1097-3
Particle density	800 ± 15% kg/m³	DIN V 18004
Crushing resistance	≥ 3.5 N/mm²	following EN 13055-1 Annex A
Chemical properties		
Chloride	< 0.01 weight-%	DIN EN 1744-1
Acid soluble sulfat	< 0.1 weight-%	
Total sulfur	< 0.1 weight-%	
Chemical Composition		
SiO_2	71 ± 2 weight-%	DIN 51001
Al_2O_3	2 ± 0.3 weight-%	
Na_2O	13 ± 1 weight-%	
Fe_2O_3	0.5 ± 0,2 weight-%	
CaO	8 ± 2 weight-%	
MgO	2 ± 1 weight-%	
K_2O	1 ± 0.2 weight-%	
Trace elements	< 0.5 weight-%	
Other properties		
Classification of fire	A1	DIN 4102-4
VOC-class (French regulation of VOC from constuction products Decret No. 2011-321)	A+ (very weakly emission)	ISO 16000; DIN EN 717-1

Particle size distribution (statistics of the year 2013)



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Liaver®
expanded-glass
technologies

01/2014 St

Fillite Standard Grades

Hollow ceramic microspheres

FILLITE is a glass hard, inert, hollow silicate sphere. **FILLITE** is primarily used to reduce the weight of plastics, rubbers, resins, cement, etc., but also imparts further benefits in many situations. Many of the advantages from the use of **FILLITE**, including increased filler loading and improved rheology, are directly attributed to the spherical nature of the mineral.

Properties

- Reduced weight.
- Increased filler loading.
- Better flow characteristics due to the spherical shape.
- Improved physical properties in moldings, castings and laminates.
- Reduced water absorption.
- Improved flame retardance.
- Improved chemical resistance.
- Good crush strength.
- Less shrinkage and warping.
- Grey colour.

Fields of application

- Refractories.
- PVC flooring.
- Brake linings.
- Phenolics and epoxies.
- Cast polyesters.
- Synthetic marbles.
- Syntactic foams.
- BMC, SMC and FRP.
- Low density, shotcrete.
- Wallboard joint compounds.
- Automotive sound-damping sheets.
- Oil well cements.

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63600 - Tylose® MH 300

1. Identification of the Substance/Preparation and of the Company/Undertaking

Identification of the Product

Product Name:	Tylose® MH 300
Article No.:	63600
Use of the Substance/Preparation:	Artists´ and Restoration Material

Company

Company:	Kremer Pigmente GmbH & Co. KG
Address:	Hauptstrasse 41-47, D 88317 Aichstetten
Tel/Fax:	Tel +49 7565 914480, Fax +49 7565 1606
Internet:	www.kremer-pigmente.de, kremer-pigmente@t-online.de
Emergency No.:	+49 7565 914480, Mon-Fri 8:00 - 17:00

2. Hazard Identification

Hazard designation:	None known.

3. Composition/Information on Ingredients

Chemical Characterization:	Methyl hydroxyethyl cellulose CAS No. 9032-42-2

4. First Aid Measures

After inhalation:	In case of complaints consult a physician.
After skin contact:	Wash with soap and rinse with plenty of water.
After eye contact:	Rinse open eyes with plenty of water. In case of discomfort seek medical help.
After ingestion:	If symptoms persist consult physician.

5. Fire-Fighting Measures

Suitable extinguishing media:	Foam, water jet.

6. Accidental Release Measures

Personal precautions:	Together with water product causes slippery surfaces.
Environmental precautions:	Prevent contamination of soil, drains and surface waters.
Methods of cleaning/absorption:	Clean up mechanically.

7. Handling and Storage

Handling

Instructions on safe handling:	Avoid dust formation.
Information on fire and explosion protection:	Risk of dust explosion.
Dust explosion class:	ST1

Storage

Storage conditions:	Store in a cool and dry place.
Storage class (VCI):	11; Combustible solids

8. Exposure Controls/Personal Protection

Additional information about design of technical systems:	Provide adequate ventilation.
Components with workplace control parameters (Germany):	TRGS 900
	MAC-Value: 10 mg/m3 general dust limit inhalable fraction.

Personal protective equipment

General protective measures:	The usual precautionary measures are to be adhered to when handling chemicals.
Respiratory protection:	Dust mask.
Hand protection::	Protective gloves made of rubber.
Eye protection:	Safety glasses

9. Physical and Chemical Properties

Form:	powder
Color:	whitish
Odor:	odorless
Melting temperature:	not applicable
Boiling temperature:	not applicable
Ignition temperature:	> 360°C
Self-ignition:	> 240°C
Explosion risk:	not applicable
Burning class:	5
Density:	1.28 - 1.30 g/cm3 (20°C)
Bulk density:	ca. 400 kg/m3
Solubility in water:	soluble
pH-Value:	neutral
Coefficient of variation (n-Octanol/Water):	< 3

10. Stability and Reactivity

Thermal decomposition/Conditions to be avoided:	> 200°C
Hazardous reactions:	No information available.

11. Toxicological Information

Acute toxicity

LD50, oral:	> 2000 mg/kg (rat; OECD 401)
LD50, inhalation:	No lethality (OECD 403).

Primary effects

Irritant effect on skin:	Non irritating (rabbit)
Irritant effect on eyes:	Non-irritating to eyes (rabbit)
Further toxicological effects:	Result based on a product with similar composition.

12. Ecological Information

Elimination (Persistency and Degradability):	10-30 % (Method: OECD 302B)
Ecological effects	
Aquatic toxicity:	:
- Fish toxicity:	LC50: > 500 mg/l (96h, Brachydanio rerio) (OECD 203)
- Daphnia toxicity:	EC50: > 100 mg/l (48h, Daphnia magna) (OECD 202)
- Bacteria toxicity:	EC50: > 1000 mg/l (OECD 209)
- Algae toxicity:	EC50: > 100 mg/l (72h, Scenedesmus subspicatus) (OECD 201)
Further ecological effects:	Organic carbon (TOC): 450 mg/g Chemical oxygen demand (COD): 1200 mg/g Result based on a product with similar composition.
Further information	
Water hazard class:	1

13. Disposal Considerations

Product:	In accordance with current regulations, product may be taken to an incineration plant.
Uncleaned packaging:	Recycling is possible when packaging is clean. Completely empty packaging can be disposed of with the regular waste.

14. Transport Information

Further information:	Not classified as a dangerous good under transport regulations.

15. Regulatory Information

Designation according to EC guidelines:	The material is not subject to classification according to EC lists.
Water hazard class:	1, slightly hazardous for water

16. Other Information

This product should be stored, handled and used in accordance with good hygiene practices and in conformity with any legal regulations.

This information contained herein is based on the present state of knowledge and is intended to describe our product from the point of view of safety requirements. It should be therefore not be construed as guaranteeing specific properties.

75367 Dispersion K 9

Dispersion K 9 acrylic emulsion has been designed for the formulation of environmentally friendly, low odour, interior wall paints. It has exceptional pigment binding capacity which permits formulation of flat wall paints at high pigment volume concentration while maintaining good scrub resistance. Thus, dispersion K9 can be formulated economically in solvent free flat paints compared to competitive technologies.

High PVC wall paints based on dispersion K9 exhibit excellent wet colour rub, particularly observable in deep tones, which further distinguish if from other solvent free products and allow the formulation of superior coatings.

In addition, dispersion K9 allows the manufacture of interior sheen paints with good resistance to block and dirt pick up. Its all acrylic composition permits use in exterior coatings for mineral substrates such as masonry and on wood.

Environmental Properties

- Film formation without the need for coalescents and solvents.
- Ammonia free
- Formaldehyde free
- Alkyl phenol ethoxylate free
- Low residual monomer levels and low odour

Characteristics of the Product

- High pigment binding capacity affording economical interior wall paints
- Excellent mechanical stability under high shear conditions
- Excellent scrub resistance
- Easy removal of stains
- Exterior durability

Typical Physical Properties (Not to be used as specifications)

Solids Content	49.5 – 50.5 %
pH	8.0 - 9.0
Viscosity (Brookfield LV spindle 3 at 60 rpm)	< 500 mPa.s
Minimum Film Formation Temperature	1°C

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Construction Polymers

Technical Data Sheet

Melment® F 10

Chemical Nature	**Melment® F 10 is free-flowing spray dried powder of a sulphonated polycondensation product based on melamine. Superplasticizer for cement and calcium sulphate based materials.**

Properties

Typical Properties

Physical shape	powder
Appearance	characteristic, white to slightly colored
Drying loss	max. 4.0%
Bulk density	500 – 800 kg/m³
Dosage recommendation by weight of cementitious materials	0.20 – 2.00%
pH value at 20 ˚C, 20% solution	9.0 – 11.4

Applications

Fields of application

Melment® F 10 is especially optimized for plastification and water reduction of cement and calcium sulphate based materials; including the following:

- Self-leveling underlayments (SLU)
- Feather edge products
- Cementitious floor screeds
- Dry-mix concrete
- Repair mortars
- Non-shrink grouts
- Cementitious self leveling floor screeds
- Tile adhesives and joint fillers

Safety

General

The usual safety precautions when handling chemicals must be observed. These include the measures described in Federal, State and Local health and safety regulations, thorough ventilation of the workplace, good skin care and wearing of protective goggles.

Material Safety Data Sheet

All safety information is provided in the Material Safety Data Sheet for Melment® F 10.

Transport Regulation

Not known as a dangerous good according to transport regulations.

Storage

Melment® F 10 has a shelf life of 2 years. It is to be stored in its unopened original packaging, dry and cool (not exceeding (40˚C / 104˚F).

Packaging 25 kg / 55 lb paper bag

Important

The descriptions, designs, and data contained herein are presented for your guidance only. Because there are many factors under your control which may affect processing or application/use it is necessary for you to make appropriate tests to determine whether the product is suitable for your particular purpose prior to use. **NO WARRANTIES OF ANY KIND, EITHER EXPRESS OR IMPLIED, INCLUDING WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, ARE MADE REGARDING PRODUCTS DESCRIBED OR DESIGNS, OR INFORMATION SET FORTH, OR THAT THE PRODUCTS, DESIGNS, OR DATA MAY BE USED WITHOUT INFRINGING THE INTELLECTUAL PROPERTY RIGHTS OF OTHERS. IN NO CASE SHALL THE DESCRIPTIONS, DATA OR DESIGNS PROVIDED BE PRESUMED TO BE A PART OF OUR TERMS AND CONDITIONS OF SALE**. Further, you expressly understand and agree that the descriptions, designs, and data furnished by BASF hereunder are given gratis and BASF assumes no obligation or liability for same or results obtained from use thereof, all such being given to you and accepted by you at your risk.

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