Recent progress in the consolidation of calcareous materials

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Litomyšl, Czech Republic

Book of Abstracts

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Program

The original program was changed due to the closure of the airspace in Europe caused by the ash cloud of the volcano Eyjafjallajökull. The cancelled presentations are marked in grey.

Wednesday, 21. April 2010

08:30 - 10:00 Registration

10:00 - 10:15 Opening

10:15 - 10:30 Gerald Ziegenbalg, IBZ-Freiberg, Germany
STONECORE - A European project funded in the 7th Framework Programme

10:30 - 10:55 Gerald Ziegenbalg, IBZ-Freiberg, Germany
Nano-particles for stone conservation – state of the art, characteristics and recent developments

10:55 - 11.20 Zuzana Slížková, Dita Frankeová, Claire Moreau, Miloš Drdácký, Libor Nosál, ITAM, Prague, Czech Republic
Consolidation of porous limestone with suspensions of calcium hydroxide nano-particles in alcohols

11:20 - 11:45 Karol Bayer, Blanka Kolinkeova, Machacko Lubos, University Pardubice
Results of laboratory testing of structural consolidation of Kutna Hora limestone with lime nanosols

Lunch Break

13:00 - 13:25 Gottfried Hauff; University of Applied Sciences Potsdam, Germany
Surface protection of 4 marble types from the "Marblepalace" of Potsdam – Comparison of selected water repellents in 10 years outdoor exposure

13:25 – 13:50 Miloš Drdácký, ITAM, Prague, Czech Republic
Peeling test application introduction guide

Jadwiga W. Łukaszewicz, Uniwersytet Mikołaja Kopernika, Toruń, Poland
The effectiveness of natural stone reinforcement by means of CaloSiL-Impregnates

Pete Askew, IMSL, United Kingdom
Fungal and Algal Growth on Stone and its Remediation

13:50 – 14:15 Malgorzata Musiela, Restauro, Poland
Possibilities for using CaLoSiL E-25 for the conservation of disintegrated lime mortars exemplified on two restoration projects: Conservation of the cellars of the middle castle in Malbork and the façade of the church of the visitation order in Warsaw

Maria Stefanidou, Ioanna Papayianni; Civil Engineering Department, Aristotle University of Thessaloniki

The role of nano- particles to water repellency of lime-based mortars

Coffee Break

14:45 – 15:15 Claire Moreau, Zuzana Slížková, Dita Frankeová, Miloš Drdácký, ITAM, Prague, Czech Republic
Effects of impregnation of lime mortars with nano lime on their physical characteristics

15:15 – 15:45 Elisabeth Ghaffari, Johannes Weber; University of Applied Arts Vienna
Penetration, distribution and precipitation of CaLoSiL in laboratory tests on different sand fractions by means of optical microscopy and SEM

15:45 – 17:00 Doubal Jakub, Kolinkeova Blanka, Machacko Lubos, Vojtechovsky Jan, University of Pardubice, Czech Republic,
Practical demonstration of lime nanosol application on limestone and imitated corroded mortar samples

Poster Session

S. ABD EL AAL, M. ALI , A.TUROS, A.KORMAN, A.STONERT, F.MUNNIK, G.MAHGOUB, S.ABD EL AZEEM Fayoum University, Faculty of Archaeology, Conservation, Cairo University, Faculty of Archaeology, Restoration&Conservation, Soltan Institute for Nuclear Studies, Swierk/Otwock, Poland, Forschungszentrum Dresden, Dresden, Germany, National Research Center,Cairo ,Egypt.

Non-destructive analysis and identification of ancient Egyptian pigments

Martina Lesar Kikelj , Ana Mladenović, Alenka Mauko, Maja Uroševic, Michele Macchiarola, Jelka Kuret, Sabina Kramar; Institute for the Protection of Cultural Heritage of Slovenia, Slovenian National Building and Civil Engineering Institute; University of Granada, Faculty of Science, (Spain), CNR-ISTEC, Faenza (RA), Italy
Consolidation of wall paintings with nanolime: in situ and laboratory evaluation
+ Last minute Posters

20:00 Conference Dinner

Thursday, 22. April 2010

09:30 - 09:55 Elisabeth Ghaffari; Georg Hilbert; University of Applied Arts Vienna (Austria), Remmers Fachplanung (Germany)
Consolidation of complex mortar structures

09:55 – 10:20 Martin Pracher, Consolidas; Kunst & Kulturgut GmbH "Alte Ziegelei"; Scheßlitz / Bamberg; Germany
The concept of full consolidation of stone- from acrylic resin to functional Silanes

10:20 – 10:55 Ewa Piaszczynski, Strotmann&Partner, Germany
The combination of nano-lime and silicic acid esters - a new possibility for the structural consolidation of scaling and peeling surfaces

Coffee Break

11:20 – 11:50 Last minute contributions

Arnulf Dähne, University of fine Arts Dresden, Germany
Consolidation of Mortars Calcium hydroxide and Calcium sulphate nanosols – Laboratory Studies

Tabitha Mifsud & Joann Cassar; Department of the Built Heritage, Faculty for the Built Environment, University of Malta, Malta
The performance of an induced calcium oxalate surface on Globigerina Limestone

11:50 – 12:15 Rolf Krompholz,; Geotron, Germany
Nondestructive Ultrasonic Testing

12:15 – 12:40 Claudio Patriarca, Evert Slob, Technical University Delft, The Netherlands
Non-destructive characterization of natural and artificial materials using electromagnetic methods

Lunch Break

13:45 – 14:05 Klisthenis Dimitriadis, Geoservice, Greece
Ground Penetrating Radar: A non-invasive geophysical method for the quantitative evaluation of stone damage

14:05 – 14:30 Claudio Patriarca, Evert Slob, Technical University Delft, The Netherlands
Electromagnetic characterization of materials destined to refurbishment activities

14:30 – 14:50 Demosthenes Giraud, Greek Ministry of Culture

Preliminary report on trial treatments at the Ancient Theatre of Megalopolis

Abd El-Hady, M.M. Dept. of Conservation Science, Faculty of Archaeology, Cairo University – Giza – Egypt.
Investigation and Conservation of Mural Paintings in Some Pharaonic Tombs in Egypt

Pete Askew; IMSL, United Kingdom
Development of an online knowledge base and management system for the characterisation and restoration of historic objects (demonstration during coffee break)

Jadwiga W. Łukaszewicz; Uniwersytet Mikołaja Kopernika, Toruń, Poland
Reinforcement of historical limestone objects by means of tetraethoxysilane-based treatments

14:50 Gerald Ziegenbalg, IBZ-Freiberg, Germany
Summary and outlook

STONECORE - A European project funded in the 7th Framework Programme of the European Union

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Six SME's, four universities, one public research organisation and one public body, from seven countries have joined together in project STONECORE in order to find a new approach for refurbishment of stones, mortars and plasters used in the construction of historic monuments. The idea is to develop and test nano-materials compatible to those used during construction together with new non-destructive methods for the assessment of stone. The following subjects were at the centre of interest in the first half of the project:

- Fundamental investigations into the synthesis of different types of nano-materials.
- Characterisation of the basic properties of different types of nano-sols.
- Determination of the applicability of different nano-materials to natural and artificial stones, mortars and plasters.
- Documentation, sampling and characterisation of the objects foreseen for trial testing and demonstration.
- Characterisation of fungal and algal growth on different stones, mortars and plasters.
- Methodology development for non-destructive assessment of stone.

Due to their importance, mainly sols based on calcium hydroxide were characterised at that time. It could be proven that a significant strengthening of treated materials can be achieved. The penetration depths depend on the used nano-lime suspensions and the characteristics of the materials that have to be treated.

Identification of fungal and algal growth was performed on many objects. During the main isolation phase of the study a total of 53 fungal and 25 algal strains were isolated and purified. This has yielded at least 25 unique species.

Ground penetration radar, ultrasonic measurements and advanced innovative methods for assessing surface degradation were at the centre of the investigations for the development of new non-destructive stone assessment methods. Significant progress was made in all three subjects.

Trial testing and demonstration, both of the nano-materials and the non-destructive damage assessment methods will be the main subject of the second half of the STONECORE project. For that, 17 different objects were already selected and documented in detail.

Nano-particles for stone conservation – state of the art, characteristics and recent developments

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Nano-materials have proven their advantages in many areas, for example in self cleaning surfaces, as antibacterial agent, as catalyst or in medical applications. Up to now, however, only few investigations known dealing with the use of nano-particles for the conservation and restoration of the cultural heritage.

Consolidation and conservation of calcareous materials requires products which are compatible with the components originally used. The application of colloidal calcium hydroxide sols offers such a possibility. Lime nano-particles are stable dispersed in different alcohols. These are produced by a novel synthesis way. The particles have sizes ranging between 50 and 250 nm. Typical concentrations are between 5 and 50 g/L. Ethanol, n-propanol and iso-propanol are used as solvent. Treatment of mortar, limestone or marble with nano-lime results after evaporation of the alcohol in the formation of solid calcium hydroxide, which converts into $CaCO_3$ (calcite) in a way similar to traditional lime mortars by reaction with atmospheric carbon dioxide. The solvent evaporates without any residues. Compounds deteriorating the mortar are not formed. All typical techniques for the application of traditional stone consolidants can be used also for the $Ca(OH)_2$ nano-sols.

Alcoholic sols containing lime nano-particles can also be used as biocide free agent for stopping fungal and algal growth. Safe mould removal is combined with the creation of alkaline conditions as well as the strengthening of the treated area.

Further nano-materials are under development, for example sols based on $CaSO_4$ for the consolidation of gypsum stucco or suspensions of $CaCO_3$ nano-particles for using as neutralisation agent for acidic areas.

Nano-particles offer the following advantages in stone, mortar and plaster consolidation:

- Possibility to penetrate deep into damaged zones, no limitations due to the particle size,
- High reactivity and fast reactions (such as carbonatisation) in the treated zones,
- High purity and defined composition.

Consolidation of porous limestone with suspensions of calcium hydroxide nano-particles in alcohols

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ABSTRACT

The main idea of the STONECORE project is to develop nano materials for the use in refurbishment and conservation of buildings and monuments. One of the solved tasks has been focused on consolidation of porous limestone by means of an application of a commercially available product CaLoSiL, which is suspension of calcium hydroxide nano particles in different alcohols.

4 types of CaLoSiL having variable concentration of $Ca(OH)_2$ (25 g/ litre or 50 g/ litre) and different alcohol as suspension mediums (ethyl-alcohol, isopropyl-alcohol or n-propyl-alcohol) were used for evaluation of the treatment effect on different types of natural porous sedimentary limestone or carbonatic rock: Kutná Hora limestone, Maastricht limestone, Pińczów limestone and Prague Opuka, which is characterized as carbonatic siltite. Mineralogical composition, open porosity and distribution of pores' size were determined and considered as key characteristics of stones. Particularly the mono-mineralogical and highly porous limestone from Maastricht (open porosity up to 53 %, diameter of the most frequent pore of 48 μm) was selected as a substrate suitable for wide laboratory testing.

With respect to a relatively high open porosity of all selected stones the technique of structure consolidation was adopted in this study aiming at impregnation and strengthening of 3 -5 cm thick layers of stone. The CaLoSiL products were applied on the top surface of stone specimens by pipette or by pouring the surface for so long until the wetting front reached the bottom of a specimen. Action effects of one or more cycles of the treatment were investigated experimentally by observation the following characteristics: ultrasonic velocity in depth profile, mechanical characteristics including bending strength in depth profile, porosity, pore size distribution, sorption characteristics, white haze formation and course of carbonation after treatment.

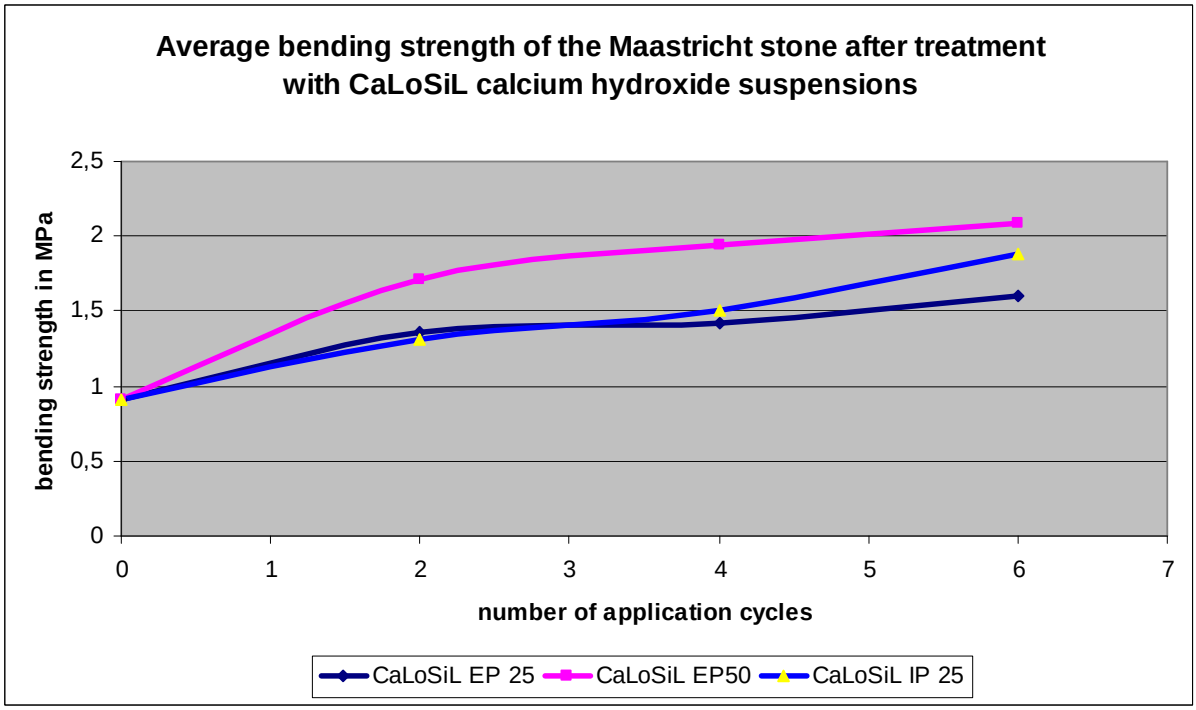
Maastricht limestone presents a high porosity, composed of big pores. Moreover, the water uptake coefficient is high. The US velocities in depth profile show higher values on the first and the last 10mm. The consolidating effects are greater near the top surface of the specimens, and at the opposite side. During treatment, there is an accumulation of particles at the surface where the product is applied and at the bottom, probably due to gravity forces. This phenomenon can be related with the white stains that appear on the bottom of the specimens. The increase of ultrasonic velocities near the top surface varies between 26 and 52 % for CaLoSiL E50, whilst it

varies between 10 and 33 % for CaLoSiL E25 and CaLoSiL IP 25. The increase of the bending strength due to impregnation ranges between 36 – 220 % and it depends on the number of applied cycles (from 2 to 6) and concentration of active substance (from 25 to 50 g/l). The porosity decreases with the number of applications for each type of sols: for E25 and IP25 decreases from 49.5% for two applications to 47.4 % for six applications and for E50 from 48.5 % after 2 applications to 45.5 % after 6 applications.

Pińczów limestone has a high porosity, but it is composed of a broad range of pores. The presence of small pores might affect the penetration of the nano sols, if they are connected to bigger pores. Based on the bending strength tests carried out on the Pińczów limestone treated with CaLoSiL E 25, IP25 and NP25, the effectiveness of applications can be assessed. For samples treated one and three times, the increase in bending strength is above 20% for all types of sols. After six impregnation processes, the strength increase is about 70% for the E25 and it is approximately 40% for IP25 and NP25.

Kutná Hora limestone and particularly more porous type of the stone seems to be a suitable material for impregnation by nano sols. Bending strengths after application of 5 respectively 10 cycles of CaLoSiL E 25 or IP 25 represent values 20 - 50 % higher than those of non-treated stones.

The pores of Opuka stone are too small to allow penetration of the nano sols. However, the weathering of Opuka stone produces surface disintegration as sugaring and delamination, and nano sols help to “re-attach” the scales formed on the surface of the materials.



Bending strength of Maastricht limestone consolidated by CaLoSiL products

Results of laboratory testing of structural consolidation of Kutna Hora limestone with lime nanosols

Bayer Karol, Kolinkeova Blanka, Machacko Lubos, University of Pardubice

The paper is focused on the summarization of laboratory tests on structural consolidation of one of the organodetritic types of limestones quarried historically in the Kutná Hora's region. This type of rock was frequently used as a building and sculpting material in Kutná Hora and its surrounding in the past. A type with high porosity and a coarse porous system was selected for the testing.

Basic preliminary tests oriented on fundamental properties and penetration ability of the suspensions were done in the starting period of the project. Two types of lime nanosuspensions (CaLoSiL®E25 a CaLoSiL®IP25) were selected for further testing on the basis of these tests.

The samples were impregnated by dropping the same amount of the consolidant with pipette on their top surface in 5 resp. 10 cycles. The amount of the absorbed consolidant, the penetration depth, absorbability, colour scheme, ultrasound transmission, drilling resistance and mechanical properties were observed as comparative parameters.

Same trials and comparisons were done also on the samples of corroded mortar's imitation, prepared from crushed limestone (St.Margarethen, 0 - 4 mm, A) and local quartz sand.

Results of the tests and their comparison may be summarized in a following way:

Both of the preparations CaLoSiL®E25 a CaLoSiL®IP25 have a good penetration ability into coarse porous carbonate materials. Repeated consolidation led to a significant increase of tested materials' solidity, what was proved by mechanical properties examinations, drilling resistance trials and also by ultrasound measuring. Both mechanical properties trials and ultrasound transmission point out that the consolidation effect is higher in the case of CaLoSiL®E25.

The visible and scalable colour change towards the lighter hues was observed when more cycles (10) were applied.

Surface protection of 4 marble types from the "Marblepalace" of Potsdam – Comparison of selected water repellents in 10 years outdoor exposure

Gottfried Hauff
University of Applied Sciences Potsdam

The Marble Palace of Potsdam, an outstanding early classicist summer residence of one of the Prussian kings is decorated mainly with 4 types of marble, three of them Silesian (*Grosskunzendorf, Kauffung and Prieborn marble)* and *Carrara marble* from Italy.

The objective of this research study, which started in 1997 was to compare a choice of common water repellents in their effect and durability by an outdoor exposure test. The results should then be the basis of a protection concept for the building after the restoration.
The project was designed to be a low budget long term project using as simple methods as possible and having student teams as researchers under the direction of the teaching staff and external consulting scientists.

In a selection process six water repellents were chosen. Three protectives on the base of an acrylic resin in combination with a silicone resin make reference to the apparently good experiences with the "Bologna-Cocktail" and it's Austrian, respectively German equivalent. Three more protectives on the base of waxes with comparatively high melting points were chosen because again there are good experiences reported in literature and restorer's experiences.

Six non destructive test methods were chosen and tested themselves on test samples and – very restrictedly – on building test areas, namely capillary water uptake by immersion, capillary water uptake by the water drop method, capillary water uptake according to Mirowski, gloss and colour measurements.

After 10 years exposure the whole project was evaluated. Some of the most important results – and these will be described and interpreted more in detail in a presentation - were:

- **research with students**, incorporated into the program of our study course was a valuable experience for all persons involved. One of the problems is the exactness of the measuring procedures with changing teams.

- of the 6 **test methods** the water drop measurement was the most meaningful. The others gave results, which were less meaningful or open for different interpretations.

- the **treatment** increased the **surface gloss** of the samples to some extent and caused a comparativelay stronger decrease of the **wateruptake**

- the **exposure** of the samples caused a strog drop of the **surface gloss** values within the first 2 years

- the **exposure** caused a drop of **water uptake** of the treated samples, differing according to both, marble type and repellent type.

- a **"real repellent effect"** (expressed by a lower water uptake than that of the initially untreated sample) is effectiv only 1 to 2 years.

- a **"relative protective effect"**, expressed by a lower water uptake than that of the also aging untreated sample is effective for ca. 3-4 years.

- the **difference** of the **repellents** does not seem very big.

- the "Bologna Cocktail" seems to be the **most effective and durable** of the tested repellants

In the end there are also other important criteria to be considered for the choice of a suitable repellent, like the depth of penetration, the influence on drying properties, retreatability and "extractability".

"Reinforcement of historical limestone objects by means of tetraethoxysilane-based treatments"

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Tetraethoxysilane and its oligomers are applied in conservation since 1925, mainly for the reinforcement of sandstone objects. Additionally, some attempts were focused on the reinforcement of other mineral materials like limestone, lime mortars, plasters and even gypsum sculptures. The effectiveness of the reinforcing impregnation is evaluated in very different manners. Many researches point out the harms resulting from the introduction of a hard artificial silicon gel into historical matter. On the contrary, other researchers consider the impregnation with tetraethoxysilane-based treatments as an inevitable conservation measure. The main positive feature of tetraethoxysilane-based impregnants consists in far going similarity between the introduced silicone gel and the silicates being present in the majority of natural rock including lime-based. The tetraehoxysilane-based impregnates are superior to other synthetic polymers (applied in conservation) because they preserve water wet ability of such treated stones. The following topics are described in the presentation in detail:

1. The influence of the structure and properties of tetraethoxysilane-based treatments on the effectiveness of limestone reinforcement as well as the reinforcement of lime and cement mortars.
2. The influence of chemical modification (plasticization) on the structure of silica gel in pores of stone.
3. Biological resistance of reinforced stones.
4. Erosion and corrosion of limestone sculptures of limestone sculptures reinforced by means of tetraethoxysilane-based.

POSSIBILITIES FOR USING CaLoSiL E-25 PREPARATION FOR CONSERVATION OF DISINTEGRATED LIME MORTARS EXEMPLIFIED WITH TWO RESTORATION PROJECTS: CONSERVATION OF THE CELLARS OF THE MIDDLE CASTLE IN MALBORK AND THE FAÇADE OF THE CHURCH OF THE VISITATION ORDER IN WARSAW. A CASE STUDY.

Małgorzata Musiela, RESTAURO, Toruń, Poland

In the years 2008 and 2009 Restauro's conservation team carried out complex conservation and restoration works in the medieval cellars of the Middle Castle in Malbork and on the facade of the Church of the Visitation Order in Warsaw. These two different projects were connected by a common conservation problem: the need to save valuable, original mortars which were preserved in the state of advanced destruction. In the case of Malbork, they formed the plasterwork of the vaults, in the case of the church, the figurative and ornamental stucco decoration of the façade.

In both cases the conservation works started with assessment of the state of preservation of the objects, thorough investigations and preparation of a conservation program. The conservation problems of the two monuments were very complex and concerned a wide range of building materials: brick walls, natural stone, metal and also mortars.

The analysis of the investigations' result as well as the assessment of the state of preservation and the required works persuaded the conservators to seek innovative technological solutions in order to consolidate the heavily disintegrated mortars. Application of organosilicon preparations might not have been effective enough. On the basis of initial consolidation tests the colloidal solution of nano-particle calcium hydroxide in ethanol – CaLoSiL E-25 – was chosen. The presentation explains the conservation process and the obtained results.

Case nr 1.

Cellars of the Middle Castle in Malbork The cellar under the Great Refectory.

The cellar complex of the Middle Castle in Malbork was constructed in the 13th century. It consists of a passage of ten rooms situated under the west wing of the castle, lying on the North-South axis. The cellars were built with fired bricks laid on lime mortar. The rooms are vaulted with barrel and stellar vaults. Some of the vaults were covered with plaster. Original plasterwork had been partly preserved in the Cellar under the Great Refectory. Its state of preservation was very bad despite conservation works undertaken in the recent past (1998). Advanced disintegration of the mortars and of the brick ground caused the materials to lose adherence and to weaken. Standard procedure of consolidating and stabilizing degraded materials using KSE proved to be ineffective in this case. Therefore an attempt was made to perform a consolidation procedure using CaLoSiL E-25 preparation. The preparation was introduced into the structure of the plaster and of the brick ground through already existing cracks and cavities, by means of injection. Spraying was also applied. The preparation was introduced until the complete saturation of the plasters. The impregnated plasters were seasoned under a foil cover and sprinkled

with water haze during the following week. Within short time a gluing procedure of the plasters by means of injection was begun, which utilized liquid injection masses based on dispergated lime. Gaps in the plasters were filled with salt-absorbing plaster due to the salinity of the brick ground. Works which were carried out according to the developed technological concept advanced smoothly. There were no loses of the original substance and the results of the consolidation were visible. Intensive powdering was stopped; the range of voids and "deaf" spots that indicated occurrence of exfoliation was reduced.

After the conservation, the object was continuously observed. As a result of construction works the microclimate in the interior of the cellars changed. First of all the humidity of the air decreased. After a few months partial powdering of the plasters was observed. It was then decided to repeat impregnation procedures, this time applying KSE 100.

The previously executed consolidation of the plasters and of the brick ground with CaLoSil E-25 preparation allowed further works and conservation procedures to be safe and effective. The condition of the plasters clearly improved; visible results of reinforcement and consolidation were obtained.

Case nr 2

Facade of the Church of the Visitation Order in Warsaw

It is a remarkable creation of baroque sacral architecture; the only monument of the Royal Route in Warsaw that survived the devastation of the World War II. Erected in the years 1727-33, it went trough several thorough refurbishments. It obtained a splendid stone and stucco decoration. The stucco decoration, consisting of full sculptures as well as floral and heraldic ornaments, was in a very bad condition. Conservators' numerous and often improper from the technological point of view interventions contributed to the serious damages and destruction of the original. The lime and gypsum-lime mortars which had been repaired with tight and stiff cement mortars were very disintegrated, cracked and exfoliated in their structure. Also in this case stabilization of the disintegrated ground was the key procedure, on which the possibility of carrying out any further conservation works would depend. The choice of nano-particle calcium hydroxide in colloidal solution allowed simultaneous work with a system of lime-based materials for reinforcing, consolidating and filling stuccowork.

The observation of the results during the works as well as the structural investigation of the mortars after the conservation proved the effectiveness of CaLoSil E-25 preparation in consolidating disintegrated mortars.

THE ROLE OF NANO- PARTICLES TO WATER REPELLENCY OF LIME-BASED MORTARS

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Keywords :lime mortars, Si nano- particles, hydrophobicity

EXTENDED ABSTRACT

It is well known that mortars due to their porous nature are the veneers through which solutions enter into the masonry mass. Different ways of protecting masonry from solutions entering into the structure of building materials have been developed though time. The application of renderings with specific technological characteristics concerning the thickness of successive layers and the compaction of each layer was an effort to create a skin of the structures. Masons were also familiar with the use of wax or oil in order to protect the materials from direct exposure to the sun or to the rain. The evolution of chemical industry in the 19th century introduced products such as resins which were widely used. Problems such as dis-colorization and biological alterations restricted their usage. During the last decades of the 20th century organic polymers and emulsions were used for surfacial protection of building materials. The emerging science of nanotechnology provides materials with new properties which efficiency in water repellency of lime-based mortars is tested in the frames of this experimental work. Nano-silica is used as additive in the structure of lime-based mortars and as a solution for surface protection in order to test its hydrophobic properties.

EXPERIMENTAL WORK

Mortars based on lime and natural pozzolan in 1:1 proportion by weight were manufactured in the laboratory using standard sand as aggregate. The binder: aggregate ratio was 1:3 by weight and the workability was measured according to EN1015-3:1999. Prisms of 4x4x16cm were produced. Some mortar samples were treated with solutions and some were left untreated for comparison reasons.

Silica nano-particles were used in two forms. In the first case, they were introduced into the mortar structure as additives in 1% by weight of binders. In order to achieve a homogeneous distribution of the nanoparticles, ultrasounds were applied for 10minutes.

In the second case, a series of mortars containing nano-particles was treated with solutions enriched with nano-particles of 0.3%by weight of the solution. The nano-particles were synthetic, hydrophobic amorphous silica produced via flame hydrolysis. Their density is 2.2g/cm^3 and BET surface is 170-230m^2/g.

The tests performed after 28 days of curing at 95%RH and 20±2^{o}C were the determination of mechanical and physical properties of the samples. Compression and flexural tests, porosity measurements and capillary absorption tests were performed in order to examine the behavior of the mortars.

RESULTS

The technique of adding nano-particles inside the mortar structure seem to restrict capillary action. The additional surfacial protection by water repellent layer also containing nano-particles acts supplementary, forming an integral protection system against water intrusion. The duration of the protection and their long-term behavior will be tested in future

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Effects of impregnation of mortars with nano-lime on their physical characteristics

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CaLoSiL is a suspension of nanoparticles of calcium hydroxide in ethanol. This research is part of the European Program Stonecore. As a preliminary study, the product was evaluated on laboratory samples before on-site testing. Several types of mortars were studied: pure lime mortars, cementitious mortars, clay mortars and gypsum mortars. Some of the results from the preliminary study are presented here for lime and cementitious mortars.

Pure lime mortars were produced with different aggregate/binder ratios, in order to obtain cured mortars with different pore-size distributions and different strengths. After full carbonation, these lime mortar samples were consolidated with CaLoSiL E-25: the product was applied by dropping on the top surface, until saturation. 5 series of lime mortars were studied: non-treated, 2 cycles, 4 cycles, 6 and 10 cycles of CaLoSiL E-25.
After treatment, we could observe that the colour of the specimens was strongly affected by the application of CaLoSiL E-25.
The consolidation effect of the treatment was measured by three techniques developed in our laboratory for determination of flexural, compressive and tensile strengths of small samples. The tests proved that the strength increased with the amount of product applied, and that the influence is stronger on weak materials.
The water uptake by capillarity was measured to evaluate changes due to the treatment. A decrease of the water uptake was observed after treatment with CaLoSiL E-25.

Another series of tests was carried out in ICVBC on artificial stone modeled by means of a cementitious mortar. This material was cured long time before the experiments and the surface was completely carbonated and hydrated. The samples were consolidated with CaLoSiL IP-25. Peeling test was performed to evaluate the surface consolidation due to the treatment.
The surface consolidation was quite high but there was probably low penetration into the bulk of these specimens.

Penentration, distribution and precipitation of CaLoSiL in laboratory tests on different sand fractions by means of optical microscopy and SEM

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Introduction

Laboratory tests with consolidants applied to substrates require well defined material parameters and product application procedures in order to yield significant results. On the other hand, the substrates to be tested should reflect characteristic states of structural decay. These prerequisites are difficult to meet with either natural stones and mortars or with synthesised solids. One solution is therefore to use loose mineral aggregates and consolidate them by capillary penetration. They represent components of defined mineralogical composition, grain size and grain shape in conditions close to a complete structural breakdown due to weathering.

Microscopic methods form the most valuable tools to assess the efficacy of such treatments. They allow detecting the in-depth distribution of the consolidant, and the way it is bound to grain surfaces and filling or bridging pores.

Aim of the study and methods

Aim of the present study performed within the EU-project STONECORE was to investigate the penetration and precipitation behaviour of selected nano-sol systems based on $Ca(OH)_2$, as a function of grain size and mineral composition of loose aggregates.

To that end, three types of mortar were crushed and sieve-fractioned, and one commercially available sand was applied. Two of the mortars were historic materials: a dolomitic mortar with quartz and a gypsum stucco without aggregates. The third one was a modern laboratory-produced mortar , again with quartz components and a dolomitic binder, and with an extremely low binder:aggregate ratio.

In addition to the mortars, the loose sand was composed of calcite.

All of the above materials were crushed and sieved to obtain separate fractions of the size 0.16 – 0.09 / 0.09 – 0.063 / 0.063 – 0.032 mm. The aggregates were filled into plastic cylinders without further compaction, and the products were applied from the top. The methods of analysis applied were polarising microscopy (PM) on thin-sections, and scanning electron microscopy (SEM) on polished sections. In addition, the amount of absorbed consolidant was determined gravimetrically.

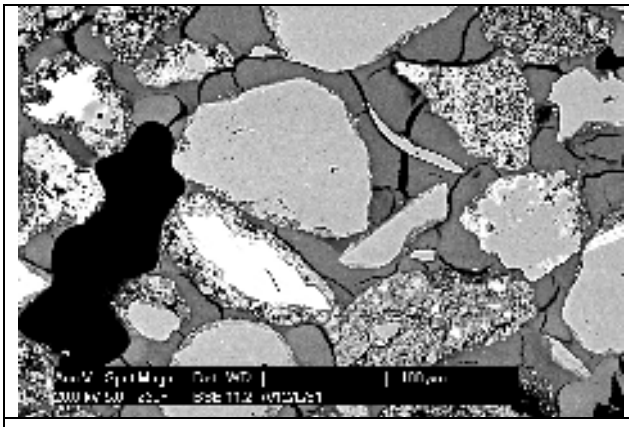
Results

Both methods – polarising microscopy and SEM – proved useful for the detection of the consolidant in the pore space of the samples. Thus, important parameters such as the in-depth distribution of the sol, possible gradients of its precipitation, and the formation of surface layers could be evaluated.

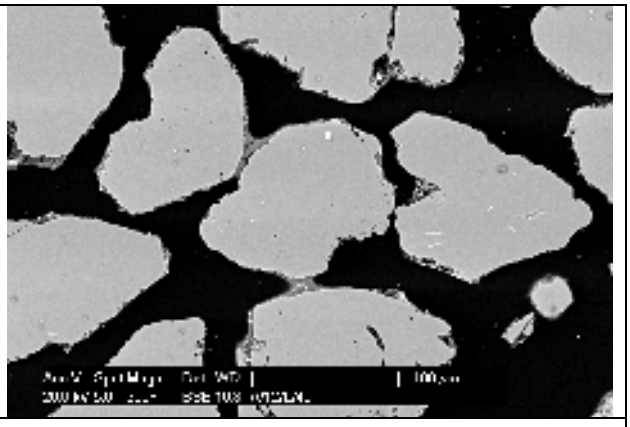
The main impacts on the above properties are due to the different sand materials rather than to the grain fractions or the applied products.

Consolidant could be detected down to the bottom face of all samples. Thus, it was proved that no significant filtering effect had occurred.

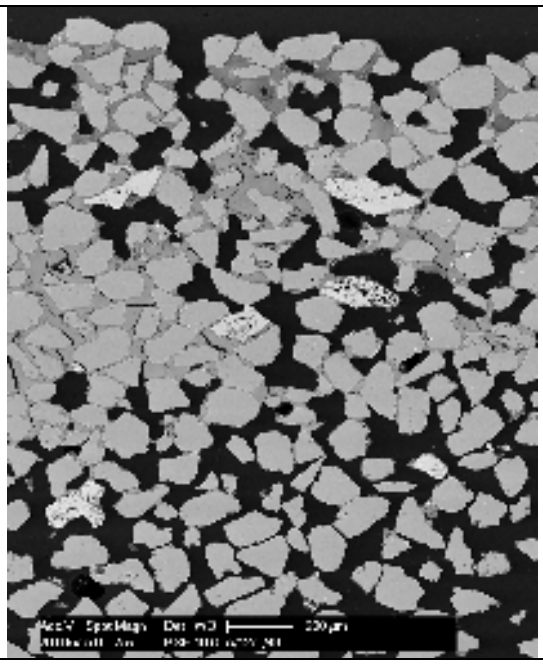
However, particularly the quartz-containing materials had most of the consolidant deposited in thin zones at the bottom and especially at the top, with almost no detectable consolidant in the central parts. For the marble sand, this observation was much less pronounced, while the gypsum sample showed a largely homogeneous distribution of the consolidant.

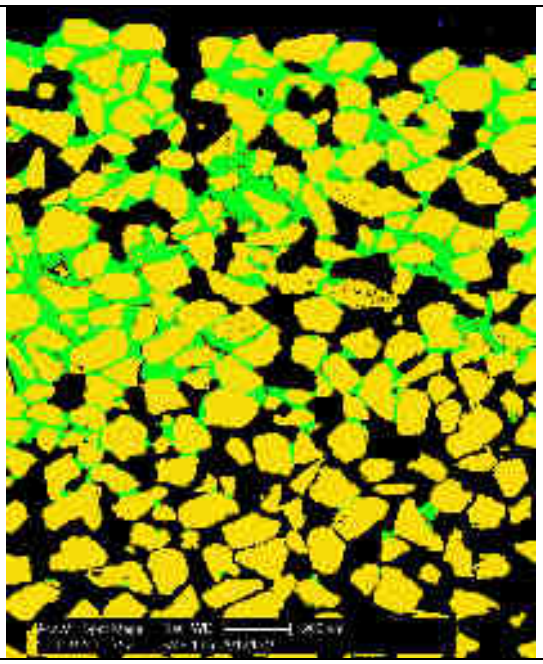


Example of high degree of pore filling by consolidant (sample: aggregates of historic dolomitic mortar with quartz); SEM/BSE



Example of narrow bridges of consolidant linking quartz grains (sample: aggregates of laboratory-made dolomitic mortar with quartz); SEM/BSE





Example of inadequate precipitation of consolidant, with formation of a compact surface layer (sample: aggregates of laboratory-made dolomitic mortar with quartz); SEM/BSE before and after pseudocolour editing (grains: yellow, consolidant: green). A rate of pore filling of 45 % was calculated for the upper part of the image, and just 7 % for the lower part

NON-DESTRUCTIVE ANALYSIS AND IDENTIFICATION OFANCIENT EGYPTIAN PIGMENTS

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Wall paintings and polychromatic decoration of tombs, temples in ancient Egypt had a long tradition over three millennia. Painting materials used in ancient Egypt can be classified into two categories: natural pigments which make up a palette of natural polychromatic minerals and synthetic pigments prepared from natural materials and metal scrap by firing the mixtures at high temperatures.

Ion beam analysis is one of currently used techniques providing data of the nature of pigments, their chronology, and the identification of domestic and imported materials. It has been demonstrated that the ancient color hues cannot be determined from present visual perceptions because many pigments have been subjected to severe chemical reactions which have entirely changed their original colors. Many misidentifications of materials were made by archaeologists before the introduction of accurate instrumental analytical techniques.

Optical microscopy, PIXE and μ PIXE used to analysis the pigments from archeological sites in Egypt like: tomb of Thutmos III located in Valley of Kings and tomb of vines Valley of Noble men.

Keywords: Egyptian wall painting; PIXE; μ PIXE; Optical Microscopy; Egyptian Blue; Orpiment; Egypt.

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Consolidation of wall paintings with nanolime: in situ and laboratory evaluation

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The wall paintings in the baroque Church of the Assumption of Our Lady (Ljubljana, Slovenia) have deteriorated extensively, resulting in the powdering and flaking of the surface, the formation of blisters, and the decohesion of mortar layers. Due to their good physical and chemical compatibility with the original mortar and the technique used to create the wall paintings, and their good penetration depth, the decision was made to consolidate the surface of the wall paintings with nanosized calcium hydroxide particles (nanolime). A suspension of these particles in a non-aqueus solvent (2-propanol) was applied with a brush over Japanese paper to the painted surface. The effectiveness of the consolidation product was then evaluated by means of USV measurements, focussing on the ability of the restoration of the original mechanical behaviour, i.e. on an improvement in mechanical resistance. The measurements were carried out before treatment, and then a consolidation period of five months. The mineral and petrographical composition of the bedding mortars of the wall paintings from the Church was determined. The effectiveness of the nanolime consolidation was also evaluated on prepared samples of lime mortars. Using different techniques, samples with various applied mixtures of nanolime were investigated.

Consolidation of mortars and stucco with calcium hydroxide and calcium sulfate nanosols – results and questions

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Based on the two objects in Germany which are foreseen as reference objects in the STONECORE project, the following materials were investigated: dolomitic lime plaster and stucco (Leuben castle) and gypsum plaster and stucco (Dahlen castle). Laboratory specimens (prisms, 10 x 2 x 2 cm^3) were made from weak dolomitic lime and from weak gypsum mortar. Subsequently, they were impregnated (3 times) with different calcium hydroxide nanosols (CaLoSiL) and calcium sulphate nanosol. Secondly, original material from the objects was treated in the same way. Three-point bending strength of the laboratory made mortars was significantly increased to multiple by treatment with both types of nanosols. The original samples, initially of a much higher strength as the laboratory specimens, did not show a clear consolidation effect after treatment with a selection of different nanosols. Water accessible porosity was measured by capillary water uptake. The original materials showed no or minor decrease of porosity after treatment with CaLoSiL in the case of lime mortars and calcium sulphate nanosols in the case of gypsum mortars. In contrary, the laboratory made lime mortar showed decrease of porosity of about one third after treatment with different calcium hydroxide nanosols, although a minor proportion of the available pore space was estimated to be filled by the consolidant.

NON-DESTRUCTIVE CHARACTERIZATION OF NATURAL AND ARTIFICIAL MATERIALS USING ELECTROMAGNETIC METHODS

CLAUDIO PATRIARCA AND EVERT SLOB
Stonecore Public Workshop, Litomysl, Czech Republic

Abstract. High-frequency, ultra-wideband penetrating radar can be used as a non-invasive inspection technique for buildings, providing information about the dielectric properties of materials. These information, combined with imaging techniques, are fundamental to support refurbishment works and to assess repair work success. We conducted numerical and laboratory experiments using a proximal, stepped-frequency continuous-wave (SFCW) radar system operating over the 3-8 GHz frequency range. The reconstruction of the materialelectrical properties is achieved by resorting to full-waveform inverse modeling. Numerical experiments showed that for typical electric permittivity and electrical conductivity values of concrete and plaster, it is possible to retrieve the physical properties of the material and to detect millimetric fractures. We investigated the effect of measurement related uncertainties on the amount of realistic retrievable information. In particular, we considered the limitation imposed by inaccurate transfer function systems and limited resolution receiver on data accuracy. A set of simulations were performed to examine the capability of the field system to detect thin layering in building materials. Laboratory experiments were conducted on non-reinforced concrete and plaster test slabs lying in different configurations. For all these cases, the frequency dependence of the electrical properties had to be considered. The results showed the good potential of the proposed method to (1) accurately localize imperfections in buildings and artworks; (2) non-invasively characterize the samples in terms of electrical properties. Systematic errors characterization is partially possible, limiting uncertainty in the data. A relation between the data precision and the possibility of information extrapolation for different settings is established.

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Ground Penetrating Radar: A non-invasive geophysical method for the quantitative evaluation of stone damage

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Abstract
Cracks, as a common stone damage, affect the strength and the appearance of all architectural parts of the Hellenic Ancient Monuments. To improve the capabilities of the surface crack detection on Monuments restoration works, ground penetrating radar (GPR) was employed. The principle and method of GPR for cracks investigation are described. As an application example, field measurements aiming to detect the depth and geometry of cracks of one floor element from the Ancient Theater of Megalopolis (Southern Greece) are presented. A 2.3 GHz GPR antenna was used, which allows the acquisition of multi-component data. The data were processed to produce vertical profiles and a 3D cube for each component and survey. The interpretation of the profiles is in good correlation visual surface characteristics of the stone. The cracks of limestone structure were located, and the geometrical distribution and depth of cracks are inspected and estimated by GPR. The results of investigation show GPR is accurate and efficient to detect cracks in stone's interior and can contribute in a significant way in pre and post-treatment control of major Monuments restoration works.

Key words: GPR, cracks, non invasive, Monuments.

ELECTROMAGNETIC CHARACTERIZATION OF MATERIALS DESTINED TO REFURBISHMENT ACTIVITIES

CLAUDIO PATRIARCA AND EVERT SLOB
Stonecore Public Workshop, Litomysl, Czech Republic

Abstract. The objective of this study is to find how materials that are commonly used in refurbishment react to applied EM fields. For this purpose,a coaxial transmission line is set up with a vector network analyzer to measure the reflection and transmission of samples using transmission line theory.
Both the objects of the refurbishment and the materials used in the treatment are studied. We concentrate only on the complex permittivity characterization because it implicitly contains the electrical conductivity, and because the materials of interest are non-magnetic. The frequency dependance of the permittivity is also studied in the 0.3 - 3 GHz range for all materials. A pure ethanol based solution is analyzed first to determine its electric permittivity.
A common sandstone sample in dry conditions is analyzed then. Time laps measurements are taken when the solution is injected in the porous sample, i.e. when the saturation is distributed differently due to changes in the porosity.
Changes in the electric permittivity can be observed during the precipitation process. Final measurements are performed when the solution has precipitated and no further flushing is possible due to the porosity reduction. These very simple experiments can be used to test the relevance of a single parameter change during the fracture healing and material consolidation process. The importance of this research does not only lie in the merely physical characterization of a fundamental process, but it reflects in other electromagnetic methods support.

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Nondestructive Ultrasonic Testing – NDT

Rolf Krompholz, Geotron Elektonik, Germany

Ultrasonic tests as a nondestructive testing methods yield the material parameter of acoustic velocity of the longitudinal wave (Vp / pressure wave).
The wave velocity is affected by material properties such as porosity, density, humidity, or microstructure. Acoustic velocity is therefore directly linked with material strength.
The object state before and after repairs can thus be described with the help of acoustic velocity measurements.

Due to the typical heterogeneous structure and the acoustic damping of natural stone, ultrasonic measurements are not carried out with reflection methods as it is done in medical applications, for example, but with transmission methods from two accessible sides, also called through-transmission.

The measurement can be performed on freely accessible objects with any geometry and a size up to several meters.
The results are spatially assigned to the measurement points as numerical values.

Results of evenly shaped objects are displayed by assigning the individual acoustic velocities to the measurement points and mapping to a chart or diagram. Previously, the measured data was imported to Excel and displayed with a chart function.

Our project activities also include the expansion of functions of the measuring software. An assistant is here responsible for the selected geometry and the measurement points together with the respective associated results. After the measurement, simple tomography is used to visualize results. This reduces the analysis time significantly.

A special through-transmission variation consists of measuring the surface. Ultrasonic transmitter and receiver are here linked on one side, namely the material surface. The measuring distance should be between approx. 5 and 50 cm. By selecting the test frequency and the associated wavelength, it is possible to affect the penetration depth of the ultrasonic wave. The wavelength is calculated from the acoustic velocity and test frequency.

$\Lambda = Vp/f$ $\Lambda = 4000\ m/s / 40000\ Hz = 0.1\ m$

The following disadvantages must be taken into consideration when using this method:

1. If the superficial layer (e.g. crusting) has a higher acoustic velocity than the lower layers, these cannot be measured.
2. If the top surface layer (e.g. scaling) is not connected with lower layers, these cannot be measured.

This means additional measurement methods must be used for corresponding repairs and restoration tasks such as determining the penetration depth of preservative solutions.

The small destructive process of a drilling resistance measurement makes it possible to generate a profile of the layers close to the surface up to a depth of approx. 5 cm. This measurement requires the use of drills with a diameter of 3 to 5 mm. The drilling resistance is calculated from the reciprocal value of the penetration speed in seconds per millimeter.

Another method consists of ultrasonic measurements in bore holes. Here, the measurement is a through-transmission between two bore holes or one bore hole and the parallel exterior side with a distance of 20 to 60 mm. This means the bore holes can be used for other measuring tasks after removing the cores, for example.

The device developed for this application is designed for ultrasonic measurements in bore holes with a diameter of 20 to 25 mm. Ultrasonic transmitter and receiver can be repositioned for measurements at different depths.

The new device thus circumvents the disadvantage of the surface measurement of not yielding any detailed depth information. A special feature worth mentioning is the "dry" linking of the sensors without the usually required coupling means.

This means it is possible to generate an acoustic velocity profile across the depth before and after treating with preservation solution.

The performance of an induced calcium oxalate surface on Globigerina Limestone

Tabitha Mifsud & JoAnn Cassar
Department of the Built Heritage, Faculty for the Built Environment, University of Malta, Malta
(formerly Institute for Masonry and Construction Research, University of Malta, Malta)

ABSTRACT:

This paper focuses on the second set of results obtained from a research programme that consisted of an induced surface conversion of weathered Globigerina Limestone from calcium carbonate to calcium oxalate and the prospects of it being used for the conservation of this stone. The first part of the research concerned the verification of the surface conversion, and the determination of its extent, using X-Ray Diffraction. The results obtained confirmed the formation of calcium oxalate in all cases, and its extent, relative to the stone's surface texture and its salt content. [1]

The second phase of the research was aimed at evaluating the performance of the induced calcium oxalate surface. The range of samples included fresh quarry samples, artificially (sodium sulphate) weathered quarry samples and naturally weathered samples. From each group of stones, both desalinated and non-desalinated samples were studied. The samples were subjected to a 5% ammonium oxalate poultice for 5 hours, after which testing took place on both treated and untreated samples. The effectiveness of the treatment was evaluated by means of Scanning Electron Microscopy (SEM) observations, Mohs hardness test, Tape test, acid resistance tests, depth of calcium oxalate formation investigations, sodium sulphate weathering tests, water absorption tests (by capillarity) and visual analyses. These studies showed that the production of calcium oxalate resulted in the formation of a more compact, more cohesive, and harder surface when compared to untreated samples. No colour differences were seen between treated and untreated samples, as verified by comparisons to Munsell Colour Charts. Also, the hydrophilic and wetting properties of the stone were retained in the treated samples. The formation of calcium oxalate on the treated samples was also found to have increased the stone's resistance to acid attack (acetic and hydrochloric acid), and to salt weathering (using sodium sulphate).

These promising results present ammonium oxalate treatment of "franka" Globigerina Limestone in a positive light. In practical terms, it can in fact be concluded that this treatment can potentially be used in the field of conservation as a protective treatment, possibly with some consolidating properties, on historic buildings and monuments built with this stone in the Maltese Islands. Further investigations to this effect are presently being planned.

[1] Mifsud, T. & Cassar, J 2006. The treatment of weathered Globigerina Limestone: the surface conversion of calcium carbonate to calcium oxalate. In: *Heritage, Weathering & Conservation – Fort, Alavrez de Buergo, Gomez-Heras & Vazquez-Calvo (eds).* Taylor & Francis Group, London.

CONDITION ASSESMENT, SAMPLING AND TRIAL TREATMENT PRELIMINARY REPORT

Dr. Demosthenes Giraud, Hellenic Ministry of Culture, Greece

During the first 18 months of *STONECORE PROJECT* most of the tasks included in the Work Description Annex have been carried through. The work of documentation, sampling and characterization of the objects foreseen for trial testing and demonstration has been completed, while extensive measurements have been also carried out at the ancient theater of Megalopolis. Furthermore trial treatment has been initiated on real objects at the same archaeological site in order to establish and evaluate the developed materials and methods. After the trial tests conducted in July 2009 and for a period of approximately six months the consolidation effect was being thoroughly observed. According to the final reports established at the end of December 2009 there have been some visible effects and results of the application, according to which penetration and consolidation has been accomplished at least in the cases of small cracks. Nevertheless the actual and accurate results for the effectiveness of the application will be established by geophysical and ultrasonic measurements of the treated area and only after an adequate period of time has passed by. Similar treatments will take place at the ancient fortress of Lilaia.

CONSOLIDAS

KUNST & KULTURGUT

„The concept of full consolidation of stone- from acrylic resin to functional Silanes“

Often the conservator or heritage preserver is confronted with the decision of removing a heavy deteriorated stone sculpture or monument from its natural surrounding into a more stable climatic situation and replace it with a replica or copy.
Besides doubling the inventory, the original artwork and the former ensemble suffer a tremendous loss of authenticity.
With the concept of full consolidation the possibility is provided to keep the irreplaceable
original artwork in its original setting.
Developed more than 35 years ago, full consolidation with acrylic resin provides invisible stability for heavily deteriorated and damaged sand- and limestone.
In this method, a certain amount acrylic resin is polymerised inside the stone from MMA to PMMA, known as acrylic glass. Afterwards, the stone is completely water repellent. The applied measures are not visible on the surface.

In the recent development, several mineral consolidants, mainly functional Silanes, had been tested for the method of full consolidation. The new found compound consists of a mixture of several Organoalcoxysilanes. The now patented consolidant supplies a polysiloxane structure in the pores of the deteriorated stone with the advantage of increasing the bending tensile and burst strength with only a little increase of the modulus of elasticity. In a research project (finished August 2009) the parameters of the consolidant as tool for structural and full consolidation had been evaluated.

Speech: about 20-30 minutes. Language: English.
Speaker: Dipl. Restaurator- Univ. Martin Pracher

CONSOLIDAS

KUNST & KULTURGUT

Company Information

In the beginning of 2009 the ConsolidaS Arts & Cultural Relics GmbH was founded with the support of the Technical University Munich, Chair of conservation, and the Foundation of Prussian Castles and Gardens, Sanssouci Palace. The company is located at the "Old Brickyard" in Scheßlitz, near Bamberg, Bavaria, Gemany.
Besides the development of sustainable solutions for the structural consolidation of organic and inorganic art- and construction material, we have focused on the active and passive decontamination of biocide- contaminated artworks and historic construction materials. Our multidisciplinary team consists of conservators, sculptors, lab technicians and academic conservators.

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NON-DESTRUCTIVE ANALYSIS AND IDENTIFICATION OFANCIENT EGYPTIAN PIGMENTS

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[5]National Research Center ,Cairo ,Egipt.

Wall paintings and polychromatic decoration of tombs, temples in ancient Egypt had a long tradition over three millennia. Painting materials used in ancient Egypt can be classified into two categories: natural pigments which make up a palette of natural polychromatic minerals and synthetic pigments prepared from natural materials and metal scrap by firing the mixtures at high temperatures.

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Optical microscopy, PIXE and μ PIXE used to analysis the pigments from archeological sites in Egypt like: tomb of Thutmos III located in Valley of Kings and tomb of vines Valley of Noble men.

Keywords: Egyptian wall painting; PIXE; μ PIXE; Optical Microscopy; Egyptian Blue; Orpiment; Egypt.

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Fungal and Algal Growth on Stone and its Remediation

Pete Askew: IMSL, United Kingdom

The ubiquity of microorganisms within all ecosystems on the planet is well recognised whether they be in deep ocean trenches, around undersea volcanic vents, in soil systems, on ice floes, in freshwater and marine systems, within other organisms or on the surface of and within organic and inorganic matrices. Either singly or as consortia, they are largely responsible for all of the major biogeochemical cycles such as the sulphur and nitrogen cycles as well as being the principle mediator of carbon cycling. Their activity is vital to the maintenance of the global environmental processes that maintain ecological balance and they perform the primary processes for both the maintenance of fertility within soil and freshwater environments as well as introducing fresh organic and inorganic material into such environments. In the latter context, microorganisms are always the primary colonisers of new environments and thus, stone is a natural habitat for a wide range of microbial species and so structures made from stone such as monuments and buildings are equally susceptible to microbiological colonisation.

One of the potential functions of nano-particulate lime dispersions is the removal of biological growth from the surface of both natural and man-made surfaces. Similarly, it is considered possible that remediated surfaces may be less susceptible to microbial colonization. This presentation describes the work to date within project Stonecore to survey the biological growth present on a number of trial objects / monuments / sites. Work so far has been concentrated on the generation of a library of isolates that can be used in laboratory tests on the effects of the experimental nanosols on both the removal of growth and the prevention of colonisation of stone and mortar. Special attention is being paid to the growth within fissures and voids within natural stones and models of such growth are being created.

“The effectiveness of natural stone reinforcement by means of CaliSil impregnates”

Jadwiga W. Łukaszewicz
Uniwersytet Mikołaja Kopernika, Toruń, Poland

The research program consisted in the reinforcement of light “Pińczów” limestone and wide-pore sandstone “Żerkowice”. The latter served as a reference material. Two types of impregnates were applied: CaLosil E25 and CaLoSil IP25. The impregnates were obtained as free demonstration samples and were applied strictly following the manufacturer recommendations. The effectiveness of stone consolidation has been estimated by:

1. Determination of time of capillary rise.
2. Determination of consolidate absorption of stones.
3. Determination of the amount of introduced reinforcing treatment and its distribution inside the pores of stone.
4. Determination of the consolidating substance structure in the pores of stone.
5. Determination of the mechanical strength of reinforced stones.

The combination of nano-lime and silicic acid esters as new possibility for the structural consolidation of scaling and peeling surfaces

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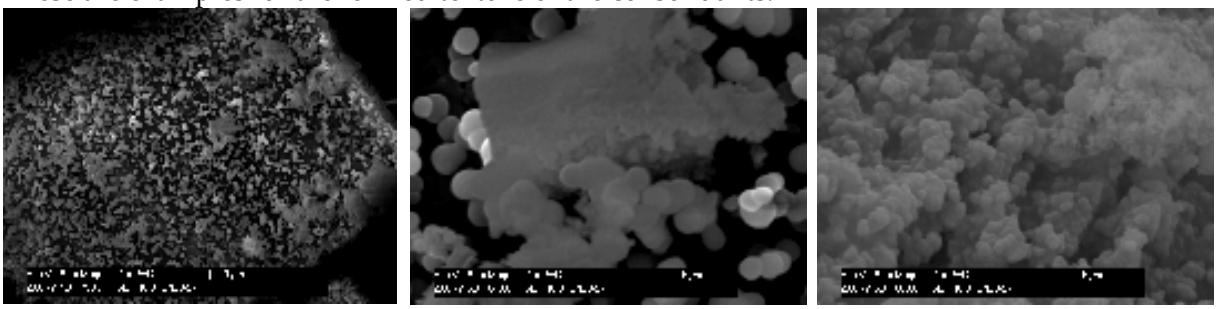
During the last months Strotman + Partner tried to find a possibility of combining CaLoSiL and silicic acid ester. Based on former experiments which revealed that a consolidation with only one of these components does not give a satisfactory result because of serious disadvantages dummy samples of different types of stone were made to find out in how far the consolidation can be improved by using both consolidants.

The following types of CaLoSiL were tested and combined with either silicic acid ester Funcosil 100 or Funcosil 300:
1. CaLoSiL 25 g/l in ethanol with acetone
2. CaLoSiL 25 g/l in ethanol with heptane
3. CaLoSiL 25 g/l in IPA with heptane
4. CaLoSiL 25 g/l in IPA with acetone
5. CaLoSiL 25 g/l in ethanol
6. CaLoSiL 25 g/l in IPA
7. CaLoSiL 25 g/l in n-propanol
8. CaLoSiL 25 g/l in ethanol grey

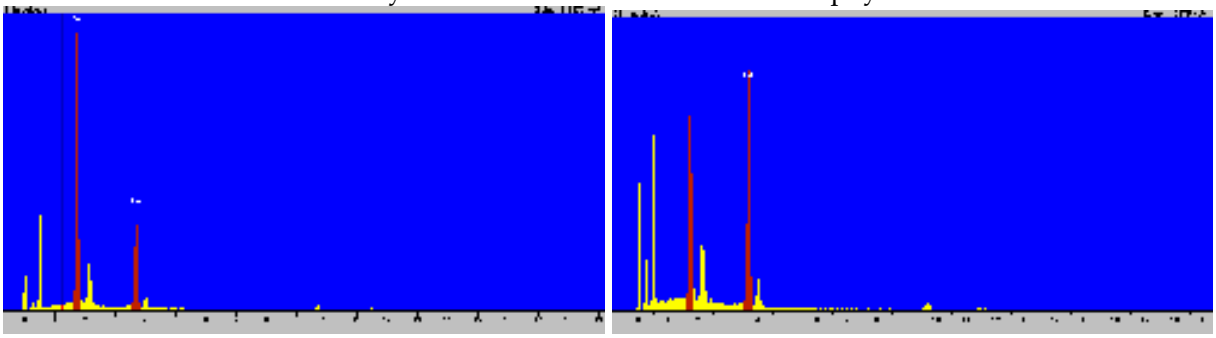
Different consolidation methods were tested to find out if this influences the behaviour of the consolidant and so the result. CaLoSiL was always applied first and after 1-8 days silicic acid ester. Also the consolidation of wet stone was tested.
Before and after the consolidation the stones were examined for their physical characteristics, for example tensile bending strength, compressive strength, water absorption, porosity or their reaction to freeze-thaw-change.
Furthermore SEM and EDAX- analyses were made to learn more about the structure of the gel which is formed inside the stone. The formed structures can be very different depending on which type of stone, Funcosil or CaLoSiL-compound is used.

These are examples for the formed texture of the consolidants:

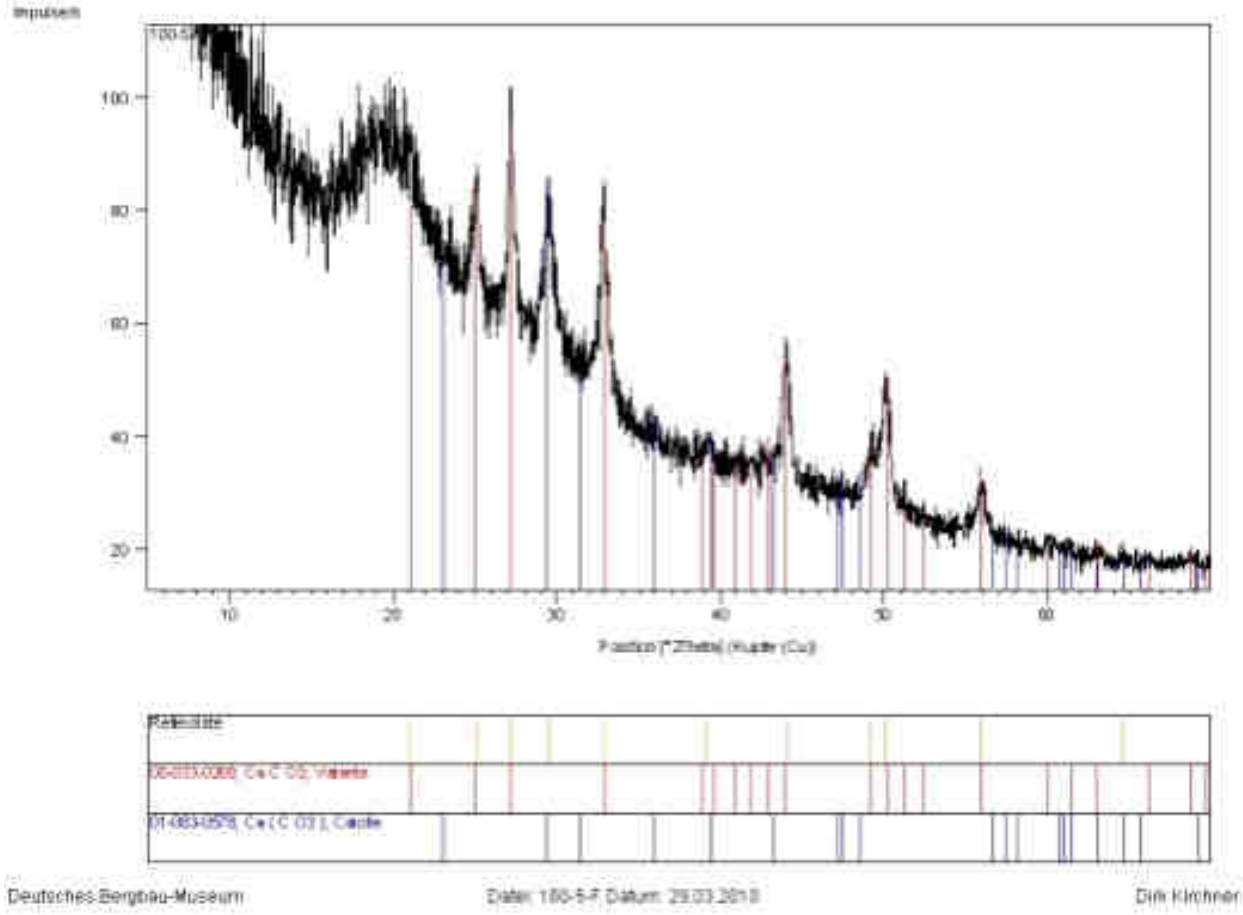


First application of CaLoSiL in ethanol 25 g/l, afterwards addition of Funcosil 100

In every sample the EDAX-analysis shows the formation of amorphous structures by connections of silicon and calcium. It is not yet obvious if these are chemical or physical connections.



Also X-ray analyses of the different consolidated materials were made.
Here is an example of an analysis of CaLoSiL in ethanol 25g/l and Funcosil 100 that was applied one day later:



The analysis shows the formation of vaterites and calcites.

The most important results:

- A combination of CaLoSiL and silicic acid ester meets the requirements to a consolidant for stone conservation.
- The best result can be obtained with a six-time consolidation with CaLoSiL 25g/l in ethanol and one day later with silicic acid ester Funcosil 100 or 300.
- The follow-up-consolidation with silicic acid ester should be carried out 1 to 8 days afterwards.
- This consolidation method is also suitable for wet stone.

- The white haze formation can be reduced with silicic acid ester Funcosil 300.
- After the consolidation the samples remain capillary and hydrophilic and their mechanical properties increase.
- Compositions form calcites, calcium hydroxide and vaterites. The compositions also contain a big amount of amorphous structures which cannot be identified by X-ray examinations. SEM and EDAX proved a big amount of amorphous structures (chemical or physical) which consist of silicon and calcium.
- The structure of the formed film depends on the type of silicic acid ester, CaLoSiL, stone and the application method.
- The application of water one day before the consolidation influences the mechanical properties positively.
- Samples consolidated with CaLoSiL in ethanol show resistance to salts and frost.