



**SINT**  
**Technology**

# DRMS PUBLICATIONS AND SCIENTIFIC MATERIAL

*Alessio Benincasa, SINT Technology*



LAB N° 0910

Laboratory authorized by the Italian Ministry of Innovation, of University and Research (D.M. n° 593 / 2000, art.14). Accredited Test Laboratory (ISO / IEC 17025) - DNV Quality Certification n° 02678-98-AQ-FLR-SINCERT



# IN SITU MECHANICAL CHARACTERIZATION OF THE MORTAR IN MASONRY BUILDINGS WITH DRMS

SACoMaTiS 2008 Varenna (LC) – Italy

○ Authors:

- *Emanuele Del Monte, Andrea Vignoli (DICEA, University of Florence, Italy)*

This paper presents the first results of a research project carried on to validate a Non Destructive Test (**NDT**) to determine the in situ mechanical **characteristics of mortar** in **historical masonry buildings**. On the basis of the compositions of historical mortars, the most common used in Tuscan buildings, there were produced 15 types of mortar with different mechanical characteristics, obtained with 5 classes of lime (hydraulic and aerial) and river sand with 3 grading curves.

For each class of mortar there were obtained 15 samples, 6 of which were subjected to flexural and compression tests and the other 6 to the Drilling Resistance Measurement System (DRMS).

The mechanical characteristics of mortar in masonry buildings affect the load bearing capacity of the structure under gravitational loads and horizontal actions, such as seismic action.

About the **DRMS** tests, there were executed **10 holes for each sample** with a hole depth of **30 mm**. The holes were made so as alternating to not cause noise between tests. During the tests the acquisition of data was of 20 records every 1 mm, then for each hole there are 601 records, for each sample  $601 \times 10 = 6010$  and therefore for each mortar there are  $6010 \times 6 = 36060$  values.

The wearing state of the drill bit was assessed at short regular interval to obtain homogenous and comparable results. So, all drill bits were previously tested and then their state of function was checked at regular intervals of 10 holes.

### Statistical treatment of data

In order to validate the DRMS diagnostic method was necessary to perform a careful **statistical analysis** of data. The goal is the validation of a correlation curve between the drilling resistance and the compressive strength for the drilling parameters considered.

The mortars tested with these parameters were those with hydraulic binder (NHL2 and NHL3.5) and larger aggregate, and were used to observe the response of the system in presence of mortar of good quality and aggregate of more size. The regression curves were obtained for each mortar comparing the average value of compressive strength tests and the average value of the test with DRMS.

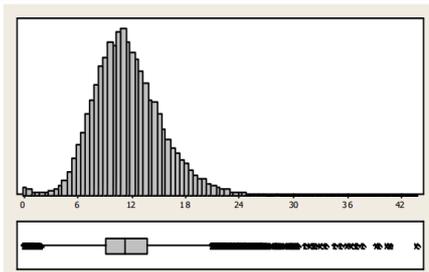


Figure 5 : data mortar N35/01

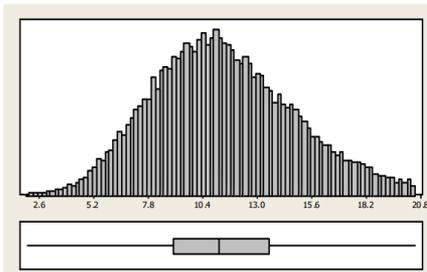


Figure 6 : data mortar N35/01 without outliers

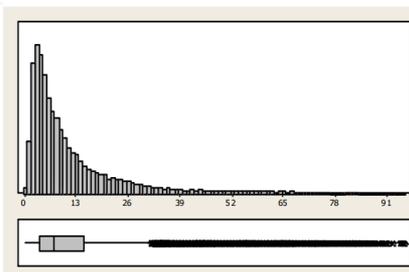


Figure 9 : data mortar N35/08

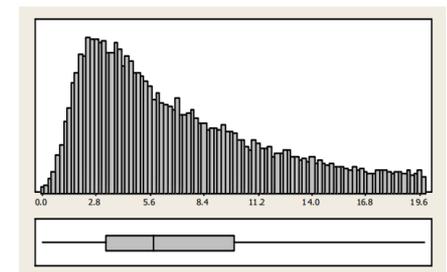


Figure 10 : data mortar N35/08 without outliers

Once performed all statistical elaboration it was possible to find the range x1-x2 to calculate the characteristic average to carry out the **regression analysis**.

The regression curves validated are:

$$R_c = - 1.107 + 0.483 DR \quad (1)$$

$$R_c = - 0.580 + 0.864 DR \quad (2)$$

where  $R_c$  is the **compressive strength** estimated by DR (drilling resistance). The Eq. (1) refers to drilling parameters  $v = 40$  mm/min e  $\omega = 100$  rpm, while the Eq. (2) is valid for  $v = 40$  mm/min e  $\omega = 300$  rpm.

The results of the regression analysis and the graphs  $R_c$  vs DR (Figure 17) and  $R_c(DR)$  vs  $R_c$  (Figure 18) show that the curves validated fit well to data and the errors on forecasting results about compressive strength are modest.

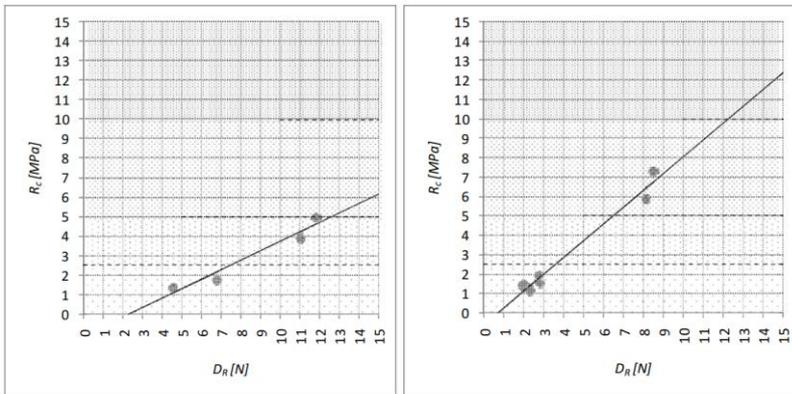


Figure 17 : curve of regression

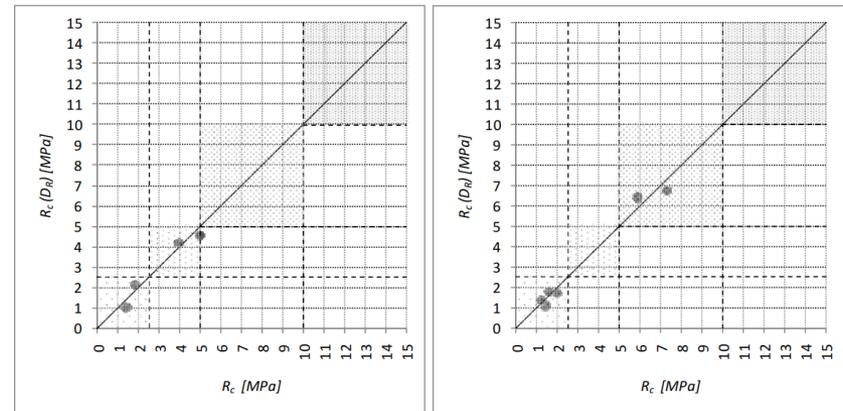


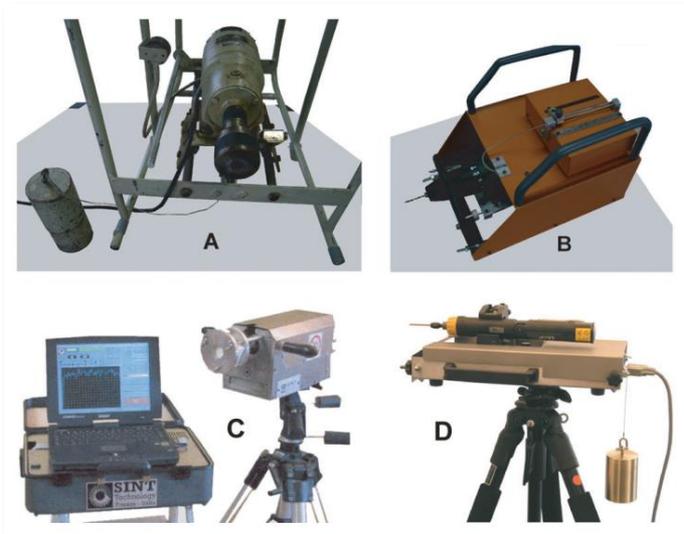
Figure 18 : compressive strength estimated  $R_c$  ( $D_R$ ) vs compressive strength observed  $R_c$

# DRILLING RESISTANCE: OVERVIEW AND OUTLOOK

*Z. dt. Ges. Geowiss., 158/3, p. 665–676, Stuttgart, September 2007*

● Authors:

- *Marisa Pamplona, Luís Aires Barros (Laboratório de Mineralogia e Petrologia do Instituto Superior Técnico, Lisboa, (Portugal))*
- *Mathias Kocher, Rolf Snethlage (Bavarian State Department of Historical Monuments, Munich (Germany))*

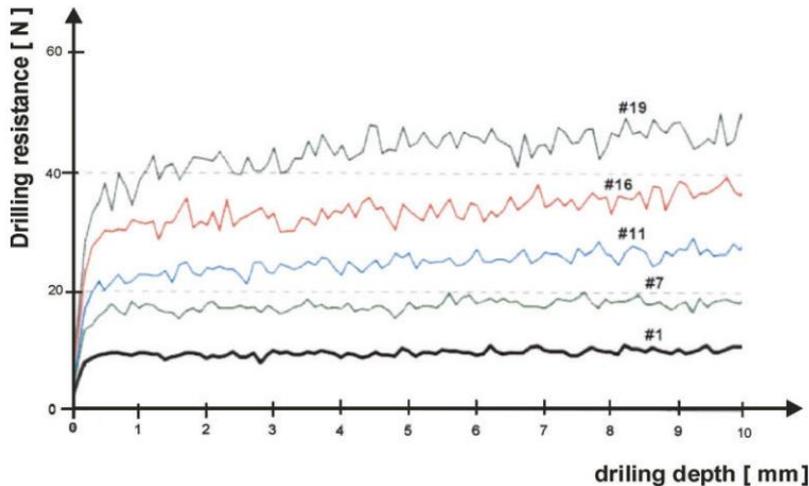


Drilling machines from the 60s to nowadays (photos A, B and D taken from Geotron-Elektronik, photo C taken from Sint Technology).

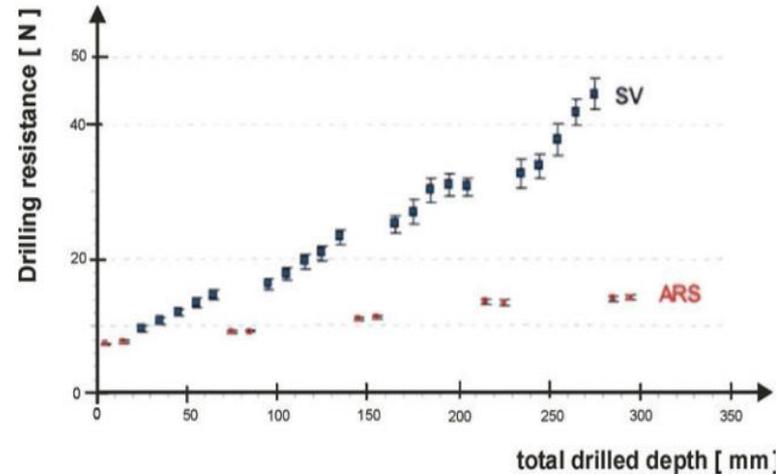
Drilling resistance technique is a valuable tool to be used in the field of conservation and restoration of our cultural heritage. Strength and strength profile measurements allow scientists to detect forms of deterioration and address adequate conservation actions. In this paper the drilling technique is described regarding its development during the last century and its function principle. Some advantages and some limits are highlighted. A correspondence between drilling force and drill bit diameter is established, so results obtained with bits of different diameter can be directly compared. Using results from different sources a linear **relation** between **drilling resistance** (DR) with uniaxial compressive strength (**UCS**) is derived. Furthermore, the drilling resistance and UCS values were related to the well-known Mohs **hardness** scale. A wider spreading of the drilling resistance method can be achieved by direct comparison with other strength parameters if the correlation expressed in this paper is further tested.

## Wear effect on the cutting tool

The wear effect is a major problem concerning abrasive stones measurements, because the drilling resistance increases during the tool working life even when drilling a more or less homogeneous material. Attempts to correct this effect are based on the assumption that the wear is constant and so an abrasion rate can be estimated. Calibration materials are used at the beginning and at regular intervals during the tool working life. Correction formulas are then applied to the measured drilling forces, based on the drilled length and resistance value of the first hole.



Increasing wear effect by drilling successive holes in an abrasive material (Sander sandstone), using a drill bit  $\phi$  5 mm at 600 rpm and 10 mm/min advancing rate



Differential abrasion increase of one drill bit  $\phi$  5 mm, measured on two materials with identical initial drilling resistance value. Total of 20 holes made on Sander sandstone (SV) and 10 holes made on artificial reference sample (ARS) under the same working conditions

## Dust accumulation in the drill hole

Recently, studies were carried out in order to reduce the packing effect due to dust accumulation and the chisel edge contribution on indentation. Indentation is the compression caused by an indenter over a surface. The chisel edge is the edge of the drill tip where the cutting lips are connected. In drilling resistance measurements, the indentation corresponds to the initial part of the graph in which the force grows rapidly and where drilling is mainly characterized by hammering, so cutting is not yet taking part of the process. The pilot hole method is also used in mechanical engineering to reduce the chisel edge contribution on the thrust force.

For special cases a combination of **pilot hole** and **pressured air** may be used to improve the reproducibility of results (Mimoso & Costa 2006), although such procedure turns the method less handy especially for in situ measurements.

## Drilling resistance versus drill bit diameter

In drilling resistance measurements a 5 mm diameter drill bit is normally used. Measuring very hard materials might be not possible due to the DRMS machine limited force of 100 N so, in this case it might be useful to use a 3 mm diameter to reduce the drilling force. In an opposite way, it might be useful to use a 7 or 10 mm drill bit diameter in very soft materials in order to increase the sensitivity of the measurement. So, by using **different diameters** an optimization of the DRMS response can be achieved. On the other hand, data collected with different drill bit diameters should be comparable. The tested working conditions were: 10 mm depth, 10 mm/min penetration rate, 600 rpm and low resolution. Results were calculated by using equation (5) in which the length of the cutting tip is equal to the drill bit diameter.

$$DR_i = \frac{DR_m}{l}$$

# CONSOLIDATION OF WEATHERED LIMESTONE USING NANOLIME

*Ice (Istitute of Civil Engineers) 2012*

- Authors:
  - *G.L. Pesce, D. Morgan, M. Allen, R. J. Ball (University of Bath (UK))*
  - *A. Henry (English Heritage (Swindon, UK))*
  - *D. Odgers (Odgers Conservation Consultants (UK))*
- Material under test
  - *Chilmark stone of Salisbury Cathedral (UK)*
  - *Bath stone of Bath Abbey (UK)*



Many of the United Kingdom's **historic masonry buildings** suffer damage and decay of their external surfaces caused by processes such as natural weathering, pollution and human contact.

DRMS, and other other techniques, was used in order to analyze the **response of the stones after the consolidation** of Chilmark and Bath stone with nanolime.

**Nanolime** is as a suspension of colloidal nano-sized particles of calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) in an alcohol such as ethanol, isopropanol or n-propanol.

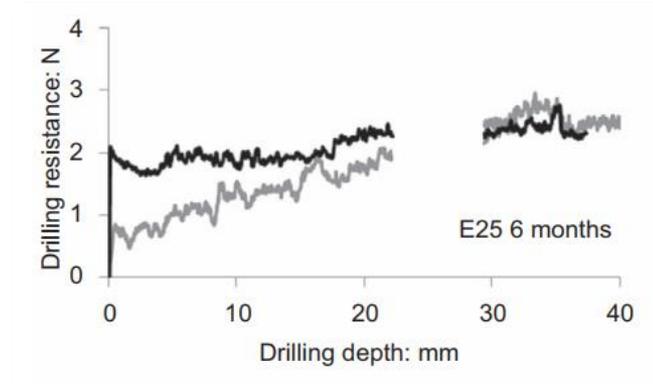
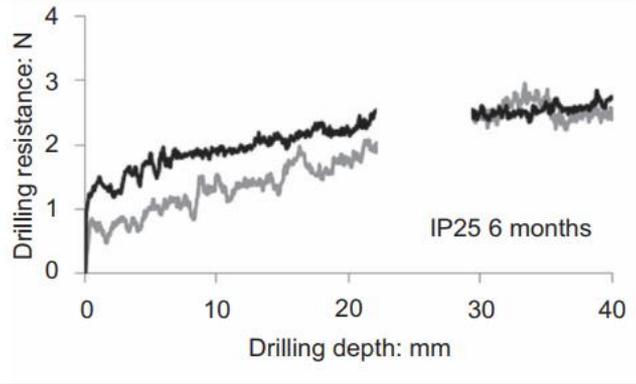
The consolidation effects were studied on weathered and non-weathered surfaces.

In the study was used CaLoSiL E25 and IP25 produced by IBZ-Freiberg

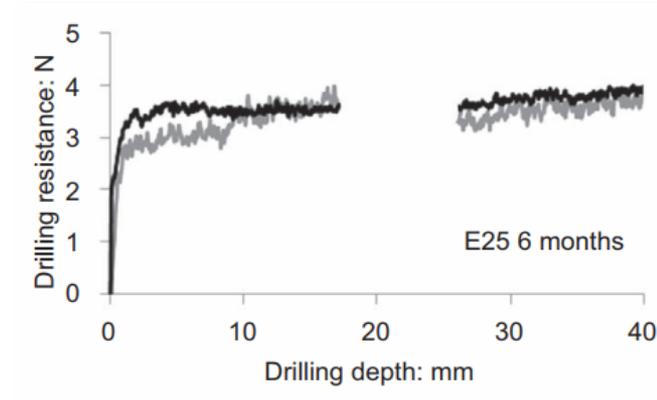
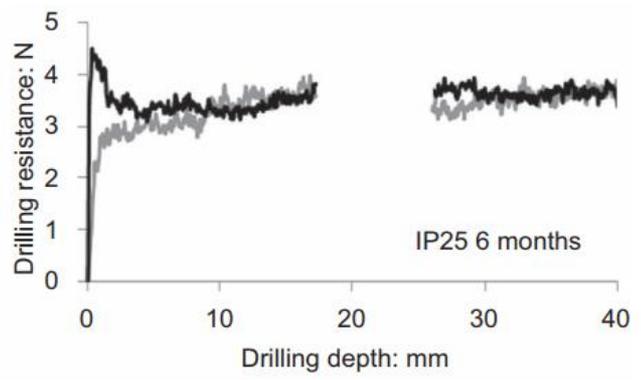
## DRMS Test result

Grey line is untreated response

### Chilmark stone weathered



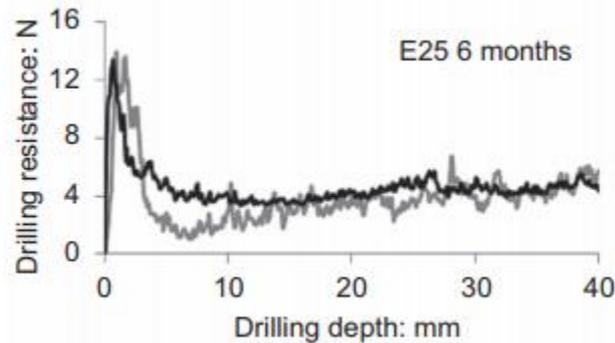
### Chilmark stone non-weathered



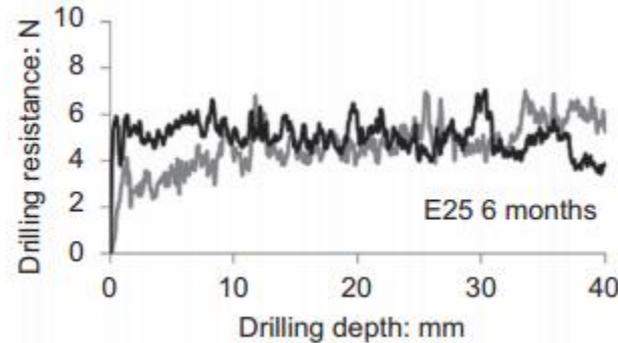
## DRMS Test result

Grey line is untreated response

### Bath stone weathered



### Bath stone non-weathered



## Conclusion

The drilling resistance measurement system successfully measured **changes in mechanical properties** with depth into the stone surface. Variations in the penetration force were attributed to both the effect of weathering, most notably by the hard crust on the Bath stone surface.

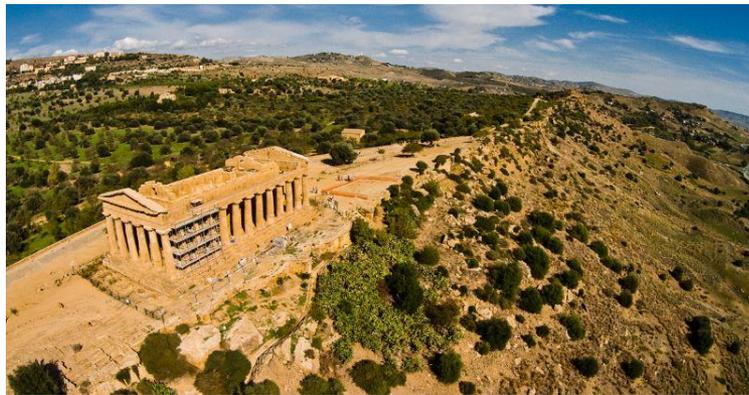
Changes in drilling resistance were also attributed to the nanolime treatments applied.

# THE BIOCALCARENITE STONE OF AGRIGENTO (ITALY): PRELIMINARY INVESTIGATIONS OF COMPATIBLE NANOLIME TREATMENTS

*Journal of Cultural Heritage Volume 30, March–April 2018, Pages 92-99*

## ○ Authors:

- G. Taglieri, V. Daniele, G. Gioia, L. Macera (Department of Industrial and Information Engineering and Economics, University of L'Aquila (Italy))
- J.Otero, V. Stranieri (Materials and Engineering Research Institute, Sheffield Hallam University (UK))
- A.E. Charola (Museum Conservation Institute, Smithsonian Institution, Washington, DC (USA))



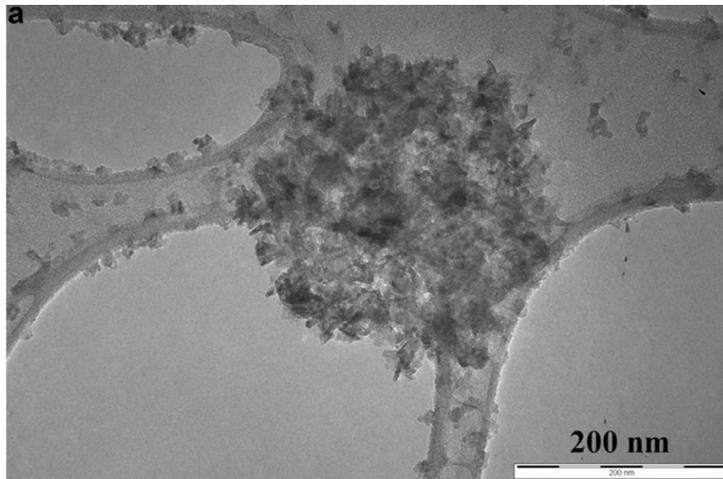
**Nanolime** is a promising **consolidant** for the conservation of most historic structures thanks to its high compatibility with carbonate-based substrates.

Nanolime can recover the superficial cohesion of deteriorated surfaces thanks to its potential to complete the carbonation process, recreating a thin network of new cementing calcium carbonate.

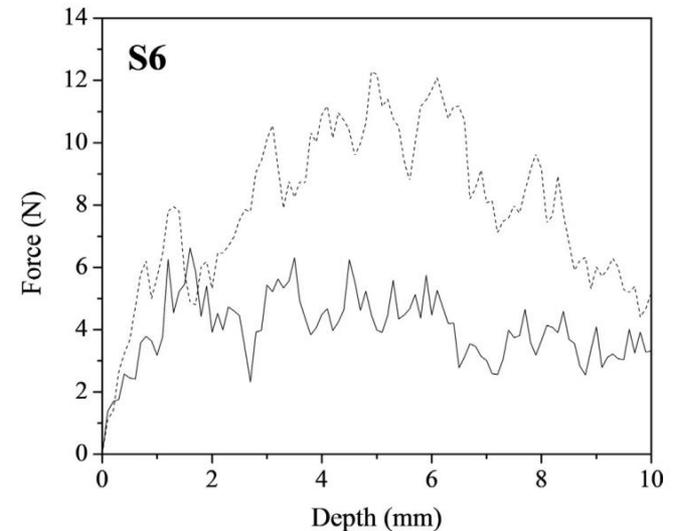
In this case, the nanolime was produced by an innovative, time and energy saving and scalable method, and its efficacy was tested preliminary on **biocalcarene stones from Agrigento**.

The stones characterization as well as the treatment effectiveness, in terms of protection against water and superficial consolidation, was investigated by several techniques such as X-ray fluorescence, X-ray diffraction, scotch tape test, water absorption by capillarity, mercury intrusion porosimetry, drilling resistance measurement system and colorimeter.

Nanolimes were applied by brushing until stone saturation was achieved, by using about 100 mg of calcium hydroxide for each stone surface. Brushing treatment was applied to the side with largest and mostly flat surface. Straight after treatment, samples were wiped with a wet cloth to remove the excess of consolidant and to mitigate surface whitening. Stones were stored for 2 days at  $RH = (75 \pm 5)\%$ . Then, the samples were oven-dried at  $60^\circ\text{C}$  until constant mass was reached, and then stored in a desiccator.



TEM micrograph of the nanolime sample



Drilling resistance profiles of untreated and treated (dotted line) biocalcarenite

# A NEW METHOD FOR DATA CORRECTION IN DRILL RESISTANCE TESTS FOR THE EFFECT OF DRILL BIT WEAR

*Journal of Cultural Heritage Volume 30, March–April 2018, Pages 92-99*

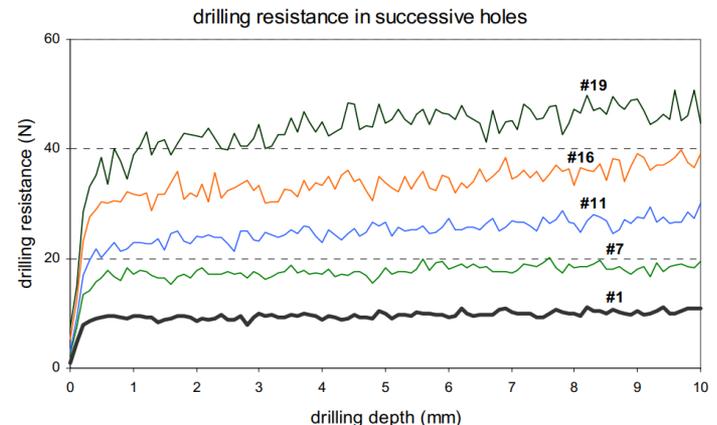
## ● Authors:

- J. Delgado Rodrigues and D. Costa (National Laboratory of Civil Engineering, Lisbon (Portugal))

The determination of the drilling resistance of abrasive materials is strongly affected by the continuous and progressive **wear** suffered by the drill bit tip.

The paper presents a new methodology to carry out the **corrections** due to the drill tip **abrasion**.

This method avoids the obtention of negative results and is able to deal with regular and irregular wear behavior, such as when the wear rate increases with the drilled length.



**The following methodology is proposed:**

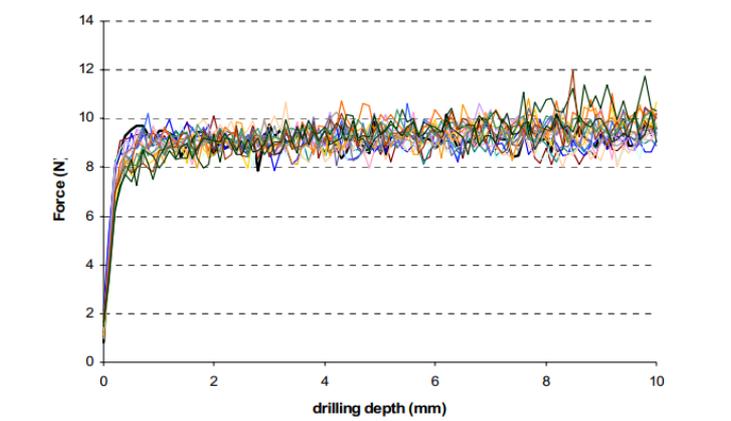
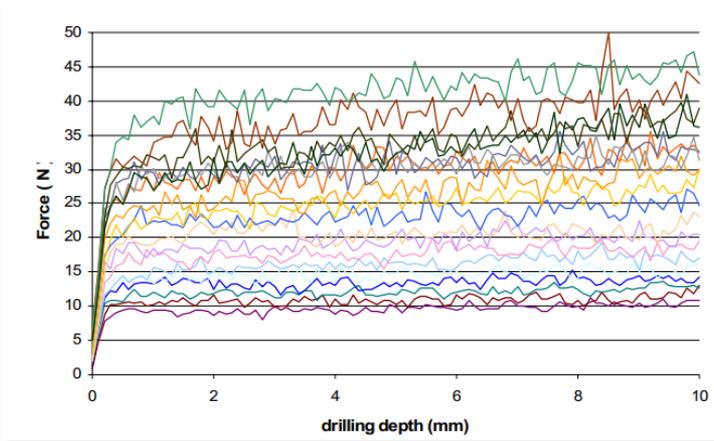
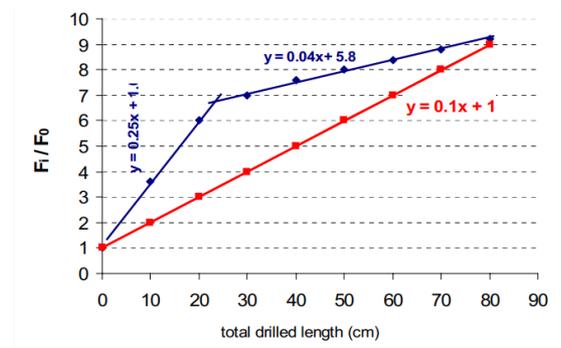
1. Select a specimen of “stone A” on which the correction holes are to be made. This will remain as the “correction specimen”.
2. Make the first hole with the new drill bit in the “correction specimen” (1cm depth). The average resistance of this hole shall be called  $F_0$ . In order to avoid the influence of the first 1-2 mm, where the drill bit tip form has a large influence, the interval between 2 and 8 mm is suggested.
3. Drill the required test holes in the specimens under testing,
4. At regular intervals of the drilling life of the drill bit, drill a new 1cm hole in the “correction specimen” (these resistances are called  $F_i$ )
5. The interval between two correction holes has to be adapted according to the abrasivity of the stone. It shall be shorter for more abrasive stones and longer for the less abrasive ones
6. For the case of Sander sandstone, for a diamond drill bit, the correction holes may be carried out at every 50 mm of drilling length until five reference holes are available. From then onwards, one new reference hole should be made at every 100 mm.

**The implementation of the proposed correction methodology can be made through the following protocol:**

1. The drilling resistance obtained at every correction hole shall be averaged between 2 and 8 mm
2. Successive  $F_i$  resistances shall be divided by  $F_0$
3. The average resistance of the hole is assigned to the starting point of that specific hole, that is to say, for the first point,  $F_1/F_0$  shall be plotted at  $x=0\text{mm}$ ; for the hole drilled between 80 and 90 mm, the result  $F_i/F_0$  shall be plotted at  $x=80\text{ mm}$
4. For all the available correction holes, plot the drilling resistance in function of the total length drilled until that specific hole
5. In ordinates plot  $F_i/F_0$ . In abscissa plot the total drilled length until point  $i$ ,
6. Adjust a regression line to the correction data. Use one or more straight lines according to the type of data.
7. VII The first regression line shall, in principle, cross at point  $XY (0;1)$  or very close to it. Large divergences from this assumption shall be analyzed carefully and explained before proceeding with the correction procedure,
8. Determine the regression equation for each regression line.

$$F_{C_i} = \frac{F m_i}{a + b x_i}$$

$F_{C_i}$  = corrected resistance at point  $i$   
 $F m_i$  = measured resistance at point  $i$   
 $x_i$  = total length drilled with the concerned drill bit until point  $i$   
 $a$  = ordinate at the origin.  
 $b$  = angular coefficient of the regression line



The proposed methodology is suitable to correct drilling data affected by progressive wear of the drill bit tip in abrasive stones. The methodology is simple to use but it requires a systematic method in order to have reliable data in all the time span of the drill bit service life. As far as the present knowledge permits, the **correction function** has to be determined in the **same material**, since wear is not equal in materials with different compositions

# CONSOLIDATION OF LIME MORTARS WITH ETHYL SILICATE, NANOLIME AND BARIUM HYDROXIDE. EFFECTIVENESS ASSESSMENT WITH MICRODRILLING DATA

*Journal of Cultural Heritage 29 (2018) 43–53*

## ● Authors:

- José Delgado Rodrigues (National Laboratory of Civil Engineering, Instituto Superior Técnico, Universidade de Lisboa, Lisbon (Portugal))
- Ana Paula Ferreira Pinto, Rita Nogueira, Augusto Gomes (CEris, ICIST, Instituto Superior Técnico, Universidade de Lisboa, Avenue Rovisco Pais, 1, 1049-001 Lisbon, Portugal )

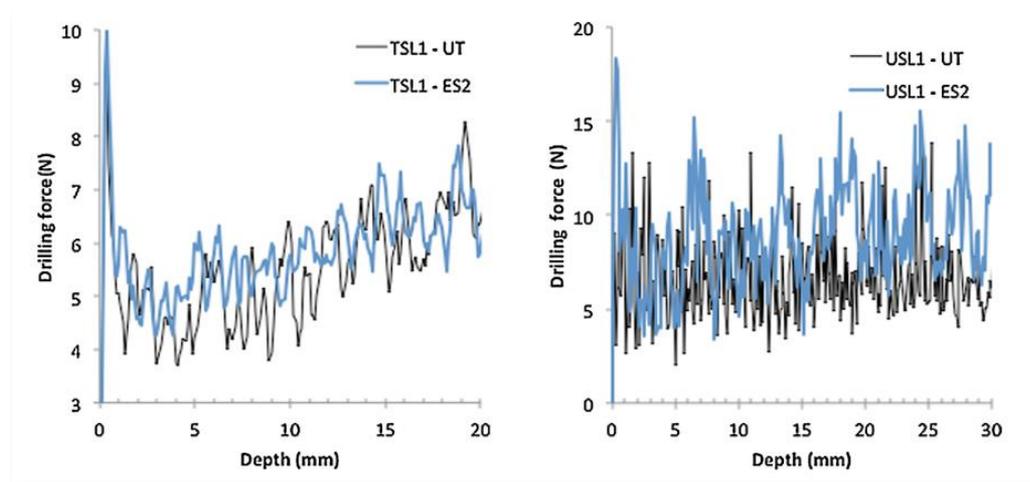
Two lime mortars were treated under laboratory conditions to assess the potential effectiveness of three consolidation treatments performed with: an ethyl silicate, a nanolime and a solution of barium hydroxide. The consolidation products were applied by direct contact capillarity. The duration and number of applications were adapted to the specific requirements of each product. Compressive and bending strength and drilling resistance were used to assess the potential effectiveness of the three treatments

The ethyl silicate was able to consolidate about 16 mm in thickness, while for nanolime this value only reached a maximum of 5 mm. The treatment with barium hydroxide showed a very distinct behavior in both mortars reaching a larger consolidated thickness in the coarser mortar, while keeping the resistance increment ratio in a moderate value.

The drilling data before and after treatment were interpreted in two ways:

- with all the tests drilled in a same condition averaged and compared
- after proceeding with a segmentation methodology addressed to identify the binding matrix and to detect the consolidation directly on it.

The two methods proved to be complementary ways to characterize lime mortars and to study their consolidation



Finish-coat (TSL1) (left) and base-coat (USL1) (right) mortars treated with ES2.

# IN-SITU STUDY OF THE CONSOLIDATION OF WALL PAINTINGS USING COMMERCIAL AND NEWLY DEVELOPED CONSOLIDANTS

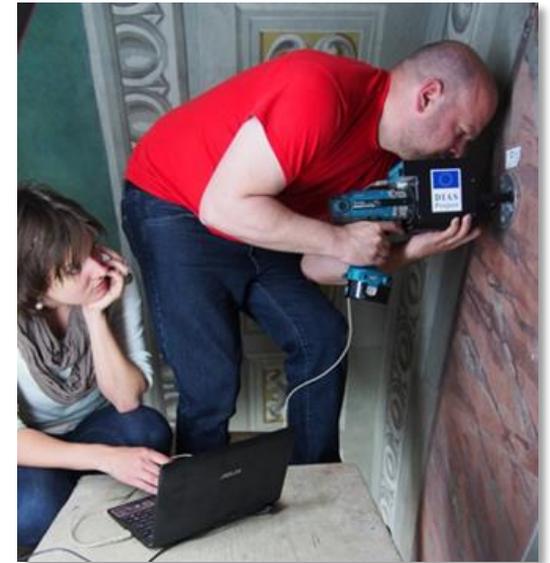
*Journal of Cultural Heritage (2017)*

## ○ Authors:

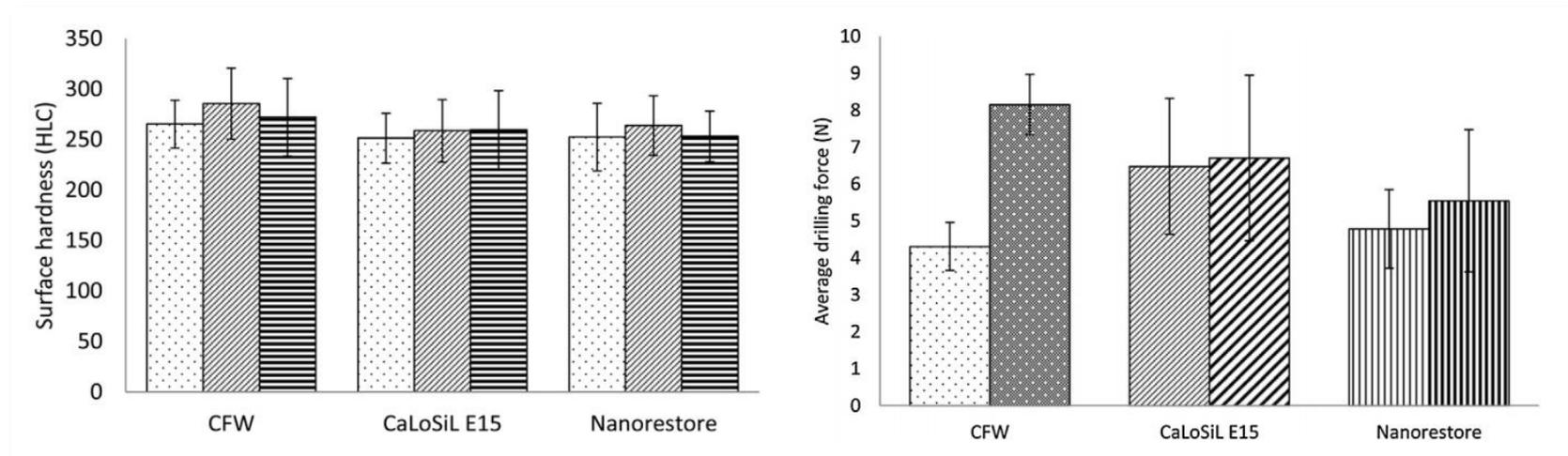
- Slovenian National Building and Civil Engineering Institute, Ljubljana (Slovenia)
- Faculty of Chemistry and Chemical Technology, University of Ljubljana, Ljubljana (Slovenia)
- Conservation Centre, Institute for the Protection of the Cultural Heritage of Slovenia, Ljubljana (Slovenia)

This paper presents a **comparison of consolidant effectiveness** for a newly developed consolidant based on soluble calcium compound calcium acetoacetate and two nano-lime-based consolidants available on the market, i.e., CaLoSiL® E15 and Nanorestore®.

Impressionist wall paintings made using the fresco technique in the Franciscan Church in Ljubljana were selected for in-situ studies. In order to monitor the colour differences and consolidation effectiveness before, and a few months after, the application of the consolidants, different non-destructive and micro-destructive methods were used.



Research showed the best recovery in mechanical properties with an increase in ultrasound velocity, drilling resistance and surface hardness, with no significant effect on the colours, after the treatment with the newly developed CFW consolidant. The results obtained in this study showed less effective consolidation for the other two selected nano-lime-based consolidants CaLoSiL E15® and Nanorestore®, since they showed no obvious increase in the drilling resistance, and moreover a clear effect on the colours after the treatment due to the formation of a white haze.



Hardness values measured before (dotted columns), 1 month (italic dashed columns) and 3 months (horizontally dashed columns) after the treatment with different consolidants

Average drilling-resistance force up to 8 mm in depth measured before (left column) and 3 months after consolidation (right column) with: CFW, CaLoSiL® E15 and Nanorestore®.

# ONSITE TESTING OF AMMONIUM OXALATE TREATMENT APPLIED TO HISTORICAL SALT-INFESTED LIMESTONE

*Journal of Civil Engineering and Architecture 11 (2017) 175-183*

## ○ Authors:

- Tabitha Dreyfuss and JoAnn Cassar (Department of Conservation and Built Heritage, University of Malta, Malta)

## ○ Material under test

- Maltese Globigerina Limestone (St. Sebastian Bastion, Marsamxett Harbour, Valletta, Malta)



The Maltese Islands in the central Mediterranean, measuring 316 km<sup>2</sup> and located 93 km south of Sicily and 288 km north of Africa are home to an immense wealth of architectural heritage which dates back to prehistoric times.

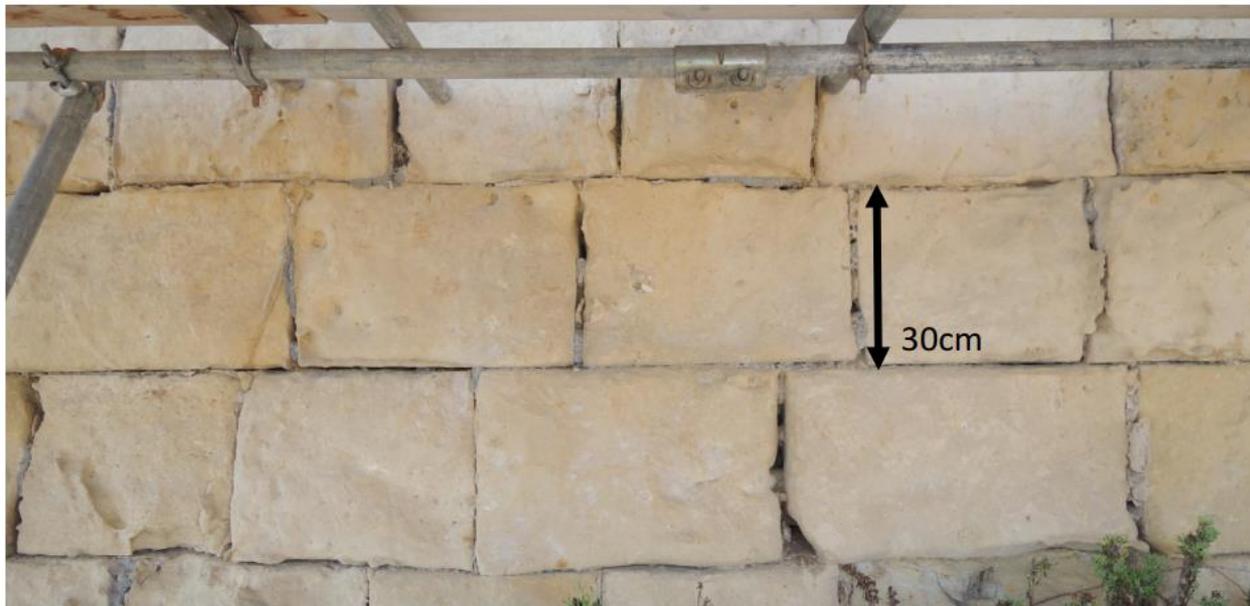
These buildings and monuments were built using **Maltese Globigerina Limestone**, a highly porous calcareous stone.

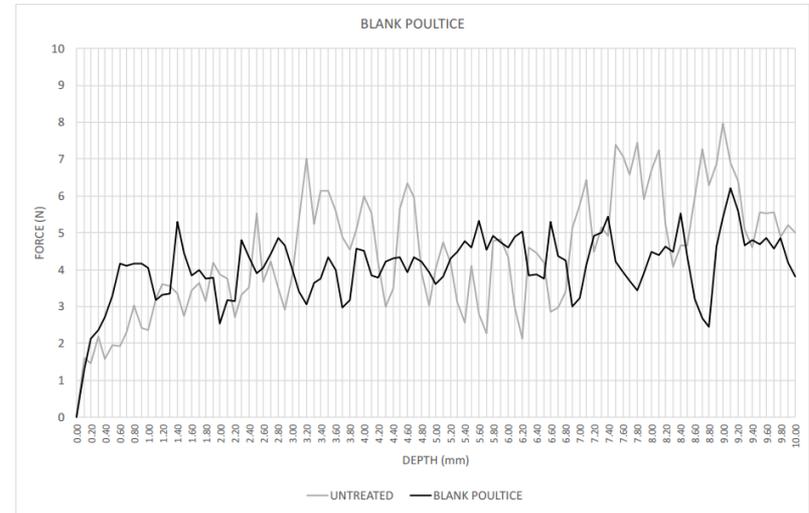
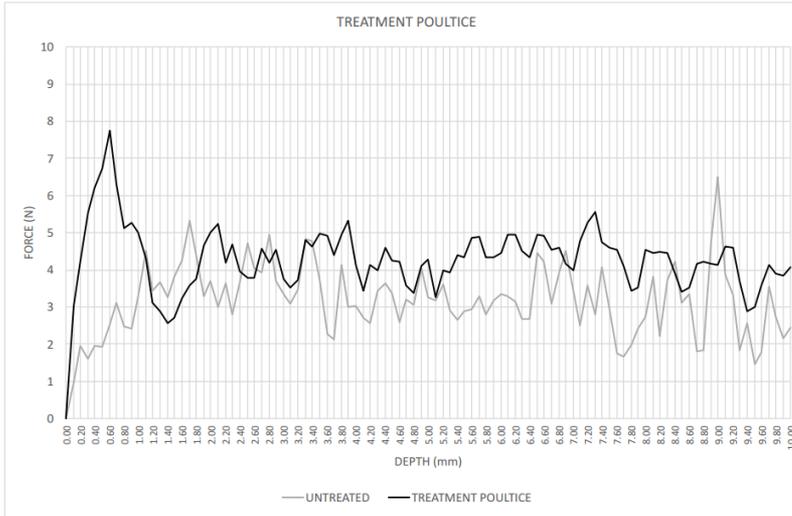
This paper presents the results obtained from onsite testing that aimed at evaluating the treatment in terms of its aesthetic performance, the depth of treatment, the mechanical properties of the consolidated stone and the influence on water transport.

To this end, the testing program included colorimetry, DRMS (drilling resistance measurement system) and water absorption through the contact sponge method.



Two poultice types were then applied to the selected stones, one as a blank (water only) poultice and the other with ammonium oxalate. The blank poultice was used to be able to identify any effects of poulticing on the stone with water only, independently of the ammonium oxalate. Both poultices included a cellulose pulp (300  $\mu\text{m}$ ). The blank poultice consisted of deionised water and the treatment poultice included a 5% ammonium oxalate monohydrate solution.





Some important conclusions may be drawn from this study, which are specific to onsite testing. From a diagnostic point of view, the tests used to characterize the stone colorimetry and DRMS show that **relevant information** can be obtained **by non-destructive and micro-destructive tests**, which may serve to characterize the stone as well as to establish baseline data prior to intervention.

This study also highlights that some of the changes to treated stone, may be due, at least in part, to the application method as well as to the treatment itself, as was proved by the blank poultice where changes in color and drilling resistance were recorded even though no ammonium oxalate was used.

# THE DRILLING RESISTANCE MEASUREMENT ON THE CHARACTERISATION OF LOW-STRENGTH MORTARS

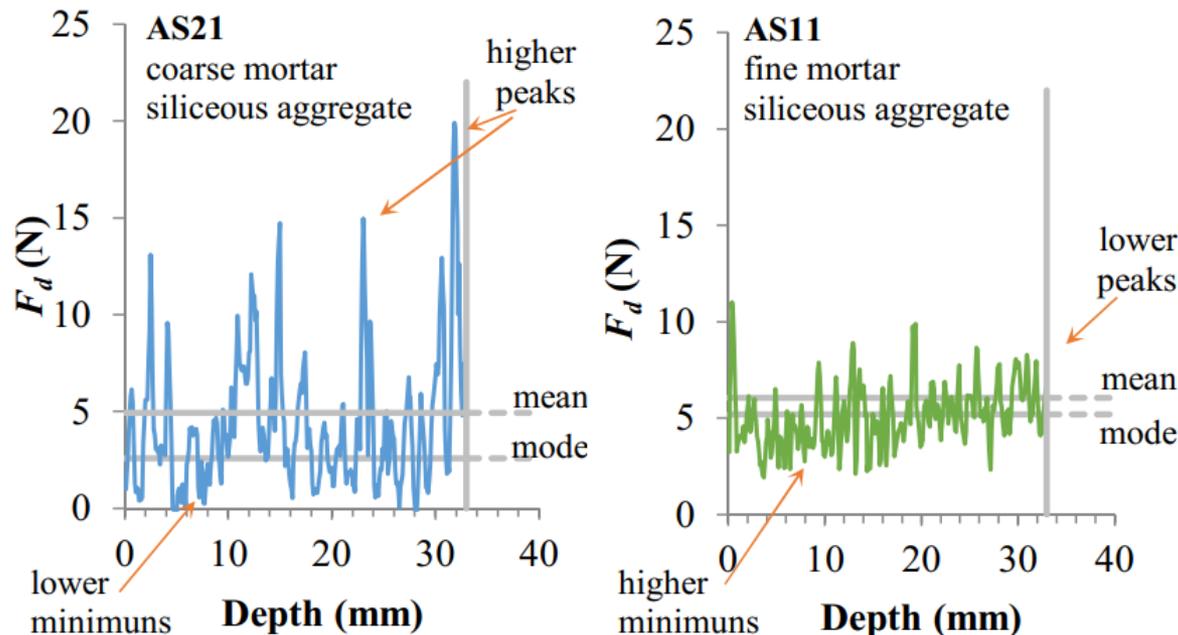
*3rd International Conference on PROTECTION OF HISTORICAL CONSTRUCTIONS (Lisbon, Portugal,2017)*

## ○ Authors:

- *Rita Nogueira, Ana Paula Ferreira Pinto and Augusto Gomes (CEris, ICIST, Instituto Superior Técnico, Universidade de Lisboa)*

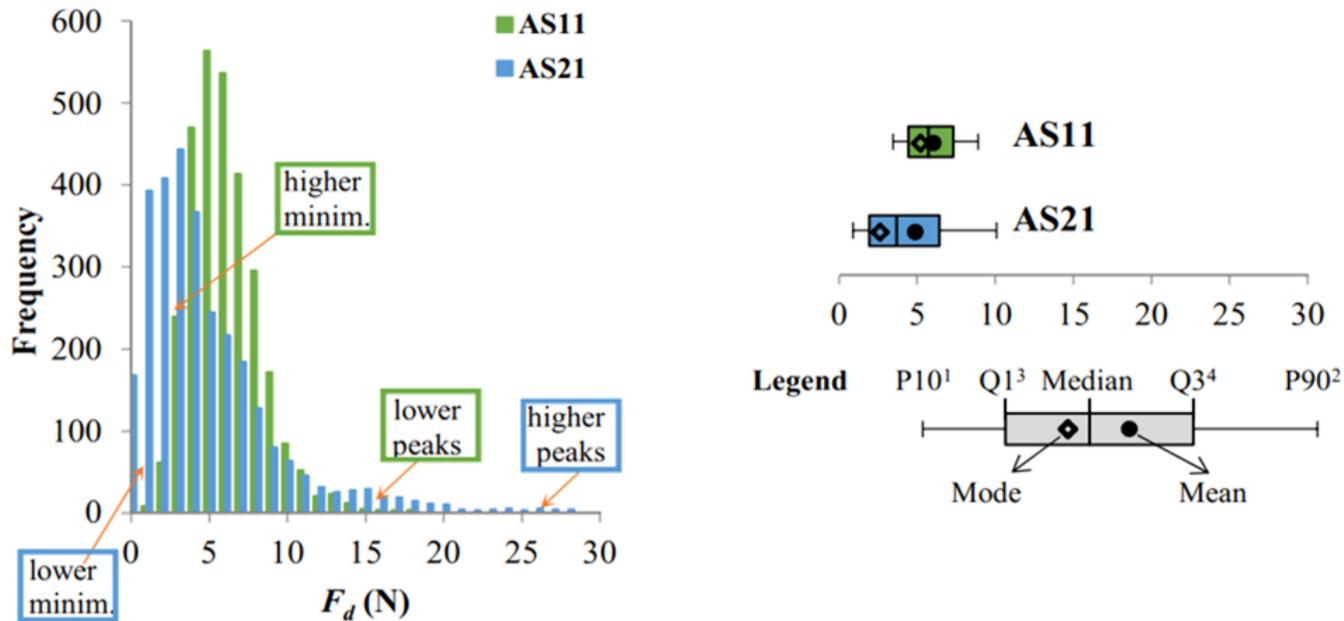
The drilling resistance is very useful for the in situ characterization of renders as it provides the strength profile in depth with very little intrusion. Its use is widespread in soft homogeneous materials, like carbonate stones. However, in **heterogeneous materials**, it yields very irregular profiles, difficult to interpret. **Low-strength mortars**, such as the ancient lime-based ones, are very heterogeneous due to the diverse characteristics of matrix (soft and weak) and the hard aggregate grains (frequently of siliceous nature). In this work, fifteen diverse lime mortars with compressive strength below 5 MPa were tested. The diverse mortars were attained varying the mixture design and adopting carbonate and siliceous aggregates of different size. The results showed that the distribution of the drilling measurements is very **sensitive to the strength and homogeneity of the mortars**.

The drilling resistance of quasi-brittle materials is strongly affected by the mechanical resistance, stiffness and heterogeneity. In more resistant, stiff and homogeneous materials the crack propagation caused by the drill action is restricted to a limited zone around the bit. There is fine fragmentation and these fragments inevitably accumulate in a narrow damaged zone. As a result, the internal friction between the fragments (milling) becomes crucial and  $F_d$  is forced to rise as it becomes more difficult to drive the drill bit, keeping the drilling parameters constant. The drilling profile shows an increasing trend, when it should theoretically show, on average, a nil slope. This  $F_d$  in-depth increasing trend corresponds to the so-called package effect.



The results showed that the drilling resistance test has potential to be used in the characterization of low-strength heterogeneous mortars, despite the irregularity of the drilling profiles. This characterization is based on the **analysis of the drilling force distribution** and in the determination of some **relevant statistical parameters**.

The **skewness** (which quantifies the symmetry of the histogram) proved to be a very good indicator of the compressive strength, better than the mean value of the distribution. These results point out that this method may be very useful in the characterization of low-strength mortars.



# NEW CONSOLIDANT-HYDROPHOBIC TREATMENT BY COMBINING $\text{SiO}_2$ COMPOSITE AND FLUORINATED ALKOXYSILANE: APPLICATION ON DECAYED BIOCALCAREOUS STONE FROM AN 18TH CENTURY CATHEDRAL

*Coatings 2018, 8, 170*

○ **Authors:**

- *Dario S. Facio, Jose A. Ordoñez, M. L. Almoraima Gil, Luis A. M. Carrascosa and Maria J. Mosquera (Department of Physical-Chemistry, Faculty of Sciences, University of Cadiz, Puerto Real, Spain)*

○ **Material under test:**

- *San Cristobal biocalcarenite of Cathedral of Jerez de la Frontera*



An effective procedure has been developed to consolidate and hydrophobize decayed monumental stones by a simple sol-gel process. The sol contains silica oligomer, silica nanoparticles and a surfactant, preventing gel cracking. The effectiveness of the process on biocalcareous stone samples from an 18th century cathedral has been evaluated, and it was found that the gel creates effective linking bridges between mineral grains of the stone. **Silica nanoparticles** produced a significant **increase in the mechanical resistance and cohesion** of the stone. The application of an additional fluorinated oligomer onto the consolidated stone gave rise to a surface with lasting hydrophobicity, preventing water absorption.



- (a) View of the main facade of the Cathedral of Jerez de la Frontera
- (b) Stone altarpiece of the Cathedral from which samples were obtained
- (c) Detail of the stone altarpiece affected by efflorescence, superficial detachments and sand disaggregation

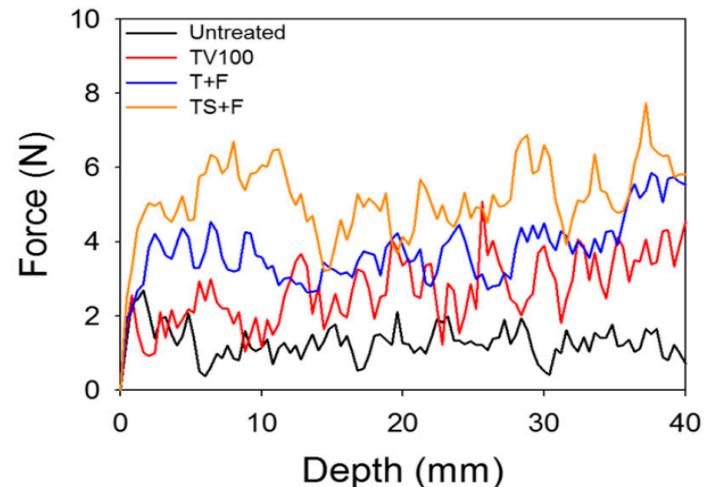
The following treatments were selected for testing:

- TV100 (commercial consolidant, Tegovakon V100)
- TS Starting sols were prepared by mixing a silica oligomer, n-octylamine and silica nanoparticles. Wacker TES40 is a mixture of monomeric and oligomeric ethoxysilanes, with an average chain length of approximately five Si–O units. Aerosil OX50 (OX50, Evonik Industries, Essen, Germany) is hydrophilic fumed silica with an average particle diameter of 40 nm.
- +F Treatments with fluorinated layer
- T Same of TS but without the silica colloidal particles

Improvement in mechanical properties of the treated stones was evaluated by using a drilling resistance measurement system (**DRMS**).

Drill bits with 4.8 mm diameter were employed with a rotation speed of **600 rpm** and a penetration rate of **10 mm/min**. The test was repeated on two samples for each treatment, and **five holes** were drilled in each specimen.

Since the stonework of the Cathedral of Jerez de la Frontera is affected by salt decay, with water being the main vehicle for the decay agents, the choice of product with consolidant and hydrophobic performances is highly required. From the results of the tests performed on the biocalcarene samples extracted from the quarry, we concluded that the UCA-TS + F treatment is the best product to be applied on the Cathedral stonework, as it showed the highest consolidant performance due to the SiO<sub>2</sub> nanoparticles added to the starting sol.



# CONSOLIDATION OF ARCHAEOLOGICAL GYPSUM PLASTER BY BACTERIAL BIOMINERALIZATION OF CALCIUM CARBONATE

*Acta Biomaterialia 10 (2014) 3844–3854*

## ● Authors:

- *Fadwa Jroundi, Maria Teresa Gonzalez-Muñoz (Department of Microbiology, Faculty of Sciences, University of Granada, Granada, Spain)*
- *Ana Garcia-Bueno (Department of Painting, Faculty of Fine Arts, University of Granada, Granada, Spain)*
- *Carlos Rodriguez-Navarro (Department of Mineralogy and Petrology, Faculty of Sciences, University of Granada, Granada, Spain )*

## ● Material under test:

- *Remains of historical gypsum plasters collected from the archeological site “Alcázar de Guadalajara”*



Gypsum plasterworks and decorative surfaces are easily degraded, especially when exposed to humidity, and thus they require protection and/or consolidation. However, the conservation of historical gypsum-based structural and decorative materials by conventional organic and inorganic consolidants shows limited efficacy. Here, a new method based on the bioconsolidation capacity of carbonatogenic bacteria inhabiting the material was assayed on historical gypsum plasters and compared with conventional consolidation treatments (ethyl silicate; methylacrylate–ethylmethacrylate copolymer and polyvinyl butyral).

### **Treatment with conventional consolidants**

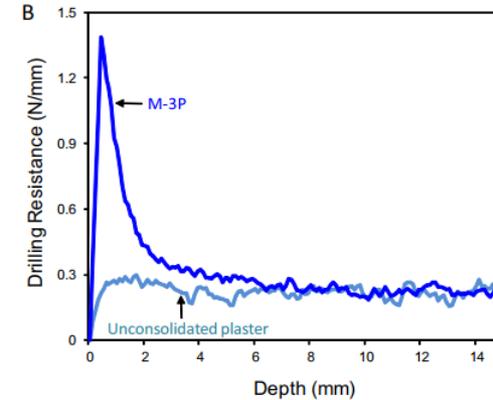
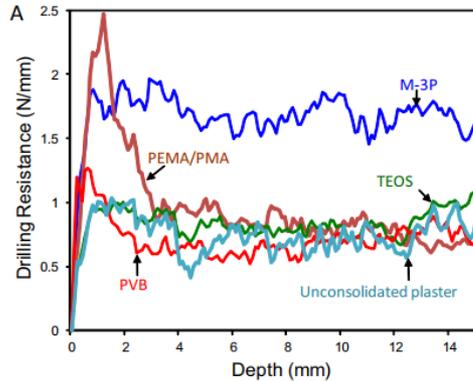
Three sets of gypsum plaster pieces (a total of 9 samples) were treated with the following conventional consolidants: commercially available tetraethoxysilane (TEOS), a copolymer of ethylmethacrylate and methylacrylate monomers (PEMA/PMA), a copolymer of ethylmethacrylate and methylacrylate monomers (PEMA/PMA). Because the material was highly weathered, as shown by its powdering and tendency to disintegrate (even under careful handling), the treatment involved seven successive impregnations.

### **Bioconsolidation methodology**

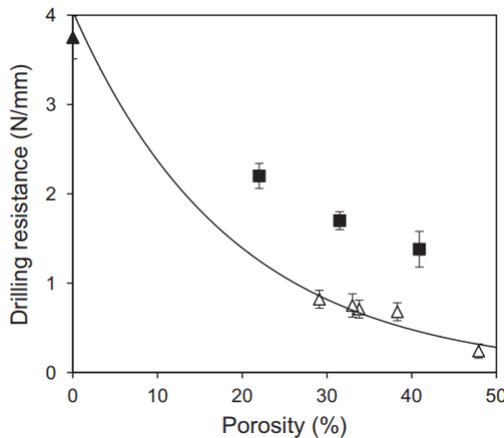
Briefly, pieces of gypsum plaster (4 samples) were treated with sterile M-3P nutritional solution (1 wt.% Bacto Casitone (a pancreatic digest of casein), 1 wt.%  $\text{Ca}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ , 0.2 wt.%  $\text{K}_2\text{CO}_3 \cdot \frac{1}{2}\text{H}_2\text{O}$  in a 10 mM phosphate buffer, pH 8), according to a patent by Gonzalez-Muñoz et al. The solution was sprayed on the upper surface of the samples until saturation, twice a day, 6 days in succession. Control samples (two blocks) were treated with sterile distilled water.

## Results

DR curves of gypsum plasters before and after treatments application are shown in figure.



DR of gypsum plasters. (A) DR measured on gypsum plaster pieces with initial porosity of 29–38% treated with three conventional consolidants and M-3P culture medium. (B) DR of untreated and bacterially treated gypsum plaster with an initial porosity of 48%. Values are average of three to five drill holes.



Variation of gypsum plasters DR vs. porosity.

The solid line shows the best fit to equation below for non-porous gypsum crystals (▲) and unconsolidated gypsum plasters (◇). DR values corresponding to bacterially consolidated gypsum plasters are also shown (■). Values represent the average of three to five drilling tests.

$$S = S_0 e^{-fP}$$

where S and S<sub>0</sub> are the strengths at porosity P and zero, respectively, and f is an empirical constant.

## Conclusion

Although consolidants have been extensively used for over a century in cultural heritage conservation, their selection is still largely based on empirical considerations without proper determination of the consolidant effectiveness and compatibility with the material it is applied to. In the case of the studied archaeological gypsum plasters, conventional consolidation treatments using an alkoxysilane (TEOS) and organic polymers and copolymers (PEMA/PMA and PVB) at best consolidate only a few millimeters near the surface. Better results are obtained using a bioconsolidation treatment based on the application of M-3P culture medium to activate the carbonatogenic microbiota present in the plasters.

These bacteria produce **newly formed calcium carbonate biocement**. This biocement is formed by vaterite, which in turn is made up of oriented inorganic nanounits embedded in an organic matrix. The brick and mortar mesostructure of bacterial vaterite cement provides a high mechanical resistance. On the other hand, the depth of consolidation achieved by this biotreatment, which does not alter the appearance of the treated material, is noteworthy (up to 2 cm). Our results show that the bacterial biotreatment would be applicable for in situ consolidation of archeological gypsum plasters. Finally, we would like to point out that such a bacterial bioconsolidation treatment may help strengthen and reduce the resorption rate of gypsum biomaterials.



**SINT**  
**Technology**

# DRMS PUBLICATIONS AND SCIENTIFIC MATERIAL

*Alessio Benincasa, SINT Technology*



LAB N° 0910

Laboratory authorized by the Italian Ministry of Innovation, of University and Research (D.M. n° 593 / 2000, art.14). Accredited Test Laboratory (ISO / IEC 17025) - DNV Quality Certification n° 02678-98-AQ-FLR-SINCERT

