Science and Art: A Future for Stone

Hughes, John; Howind, Torsten

Published: 01/01/2016

SC16

SCIENCE and ART: A Future for Stone

Proceedings of the 13th International Congress on the Deterioration and Conservation of Stone – Volume I

Edited by
John Hughes & Torsten Howind

SCIENCE AND ART: A FUTURE FOR STONE

PROCEEDINGS OF THE 13TH INTERNATIONAL CONGRESS ON THE DETERIORATION AND CONSERVATION OF STONE

6th to 10th September 2016, Paisley, Scotland

VOLUME I

Edited by
John J. Hughes and Torsten Howind

ISBN: 978-1-903978-57-3
ISBN: 978-1-903978-54-2 (eBook)

ISBN: 978-1-903978-59-7 (Set: Volume 1&2)
ISBN: 978-1-903978-56-6 (eBook-Set: Volume 1&2)

Hughes J.J. and Howind T. (Editors), "Science and Art: A Future for Stone. Proceedings of the 13th International Congress on the Deterioration and Conservation of Stone", University of the West of Scotland, Paisley, September 6th to 10th, 2016.

Cover image: The front door of the Paisley Technical College building, now University of the West of Scotland. T.G. Abercrombie, architect 1898. Photograph and cover design by T. Howind.

IN SITU ASSESSMENT OF THE STONE CONSERVATION STATE BY ITS WATER ABSORBING BEHAVIOUR: A HANDS-ON METHODOLOGY

D. Vandevoorde[1,2*], T. De Kock[2] and V. Cnudde[2]

Abstract

This paper describes the use of the water absorbing behaviour (WAB) for assessment of the conservation state of stone *in situ*. A test methodology, consisting of a combination of techniques for *in situ* measurement of the WAB, was applied in a case study on Lede stone, a sandy limestone, used in a medieval facade in Ghent, Belgium. The methods used were the contact sponge method (CSM), the Karsten tube (KT) and the droplet method (DM). Additionally, the residual hardness of the stone was measured by Schmidt hammer (SH). After careful selection of representative measuring points, a qualitative analysis of the stone's condition could be made, based on its WAB, residual hardness, reference data from laboratory experiments and thorough visual observations. From this analysis it could be concluded that CSM, KT and SH generated coherent and compatible results; that DM could indicate superficial alterations which were not necessarily representative for the sub-superficial WAB and that this methodology could give an insight on the conservation state, beyond visual observations, when combined with reference data.

Keywords: porous stone materials, water absorbing behaviour, contact sponge method, Karsten tube, droplet method, capillary uptake

1. Introduction

Water absorption of a natural stone is of significant importance for its conservation, as water is a key element in most degradation processes. The water absorbing behaviour (WAB) of a stone material is governed by its pore structure and is constituted of the open porosity, pore size distribution, pore geometry and interconnectivity and chemico-physical properties of the internal pore surface (Hall and Hoff 2002). Consequently alterations in this pore structure will induce differences in the WAB (Kourkoulis 2006). These differences can be caused by variations in the natural state of the stone, such as different stone qualities and weathering degrees, presence of salts, bio colonization, crusts, etc. Additionally, due to treatments such as (old) water repellent treatments or the lack thereof, recent consolidation treatments, anti-graffiti etc. the WAB can be altered. When assessing a stones conservation state *in situ*, the WAB will be governed by a complex combination of these factors.

[1] D. Vandevoorde*
Ghent, Belgium and Rephine Stoneworks, Belgium

[2] D. Vandevoorde*, T. De Kock and V. Cnudde
Pore-scale PROcesses in Geomaterials RESearch Group (PProGRess), Ghent University, Belgium
d.vandevoorde@ugent.be

*corresponding author

In this paper a methodology, consisting of a combination of *in situ* applicable WAB measuring techniques, namely the droplet method (DM), contact sponge (CSM) and Karsten tube (KT) is presented as diagnostic tool or the assessment of a stones conservation state. As these methods are quick, cheap and do provide directly applicable and easily understandable information, this methodology meets the requirements of the practical field and the restrictions of large scale *in situ* investigations. The methodology was tested in a case study regarding the pre-investigation of the condition of Lede stone, used in the medieval facade. Previous to this case study, the WAB methods were extensively tested under laboratory conditions on non-weathered stone samples (Vandevoorde *et al.*, 2009 and 2013) and Lede stone was elaborately studied by De Kock *et al.* (2015). Consequently this case study was the ideal opportunity to test knowledge acquired in laboratory under *in situ* conditions, where weathering, previous treatments and environmental factors could influence the measurements. The aim of the study was twofold: (1) Investigation of the practical applicability and compatibility of the methods under *in situ* conditions and (2) investigation of the sensibility of the methods for indication of structural variations of the stone, degree of weathering and presence of past treatments within the facade.

The case study on WAB was part of a more elaborate study of the condition the medieval facade of the Free Boatmen's Guild House (1531) in the centre of Ghent, Belgium (Fig. 1a). The Lede stone was aged and weathered and was subjected to several restoration campaigns in the past. In order to define an adequate restoration strategy, which would ensure an optimal conservation of the facade, a pre-investigation of the conservation state and study of consolidation treatments was prescribed. The examination of the conservation state consisted of a historical study of the facade, a thorough visual analysis, determination of deterioration patterns, analysis of types of Lede stone, laboratory investigation of samples, measurement of stone strength with a Schmidt hammer, measurement of the WAB, and IR-thermography. This paper will focus on the measurement of the WAB.

2. Materials and methods

2.1. Lithotype: Lede stone

The Lede stone is a sandy limestone with a high historical importance in north eastern Belgium and adjacent parts of the Netherlands. Due to the natural variety within the stones components and fabric, porosities can range from below 5 vol-% to above 10 vol-%. This is mostly related to the amount of macrofossils and concomitant moldic pores. Microscopically, the stone is a packstone to grainstone with a large siliciclastic fraction, mainly quartz and some glauconite. When exposed outside an iron rich patina will form at the surface. Taking into account the geological variability, the stones in the facade were macroscopically divided in a fine grained and a coarse grained type (Fig. 1b and Fig. 1c). The fine grained type was defined by a compact, low porous and homogeneous texture with a low macrofossil content. The coarse grained type is defined by the presence of foraminifera *(Nummulites variolarius)* and serpulids (*Ditrupa strangulate),* which results in a rougher surface due to differential erosion.

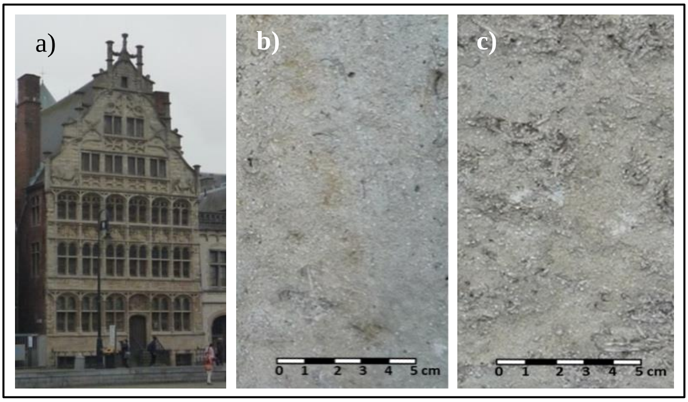

Fig. 1: Facade of the Free Boatsmen's Guild House in the historic centre of Ghent, Belgium (a) and details of the fine grained (b) and coarse grained (c) Lede stone.

2.2. Test methodology

2.2.1. Registration of measuring points and environmental conditions

For an accurate interpretation of the obtained data, one should register all information which could have influenced the measurements. A datasheet was completed for each stone, containing the encoding and location of measuring point, time of measurement, weather conditions and environmental temperature and relative humidity. All measurements were performed in the same period under similar weather conditions to minimize the possible influence of variable environmental conditions. As a liquid, water could be present in the stone due to rain, fog, rising damp, hygroscopic salts, etc. To help indicate the presence of liquid water, the moisture content and temperature of the surface were recorded by using a moisture meter and IR-thermometer. Photographs were taken before and after measurement to register visual anomalies which occurred during the measurements such as absorption in microcracks, hindrance by soiling, leaks, etc.

2.2.2. Measuring techniques

The WAB measuring techniques used in this study were the droplet method (DM), the contact sponge method (CSM) and the Karsten tube (KT). The residual hardness of the surface was tested using a Schmidt hammer (SH). A combination of all data was used for evaluation of the stone quality, together with visual observations. In this paper the methods will be described only briefly. For further information the authors refer to previous publications (Vandevoorde *et al.*, 2009 and 2013).

The DM (Fig. 2a and Fig. 2b) is based on the classical contact-angle method (ASTM D7334-08 2008). *In situ* applicable variations of this method are frequently used in the field by conservators and restorers for e.g. assessment of the residual efficiency of water repellent treatments (Blaüer *et al.*, 2012, Ferreira-Pinto and Delgado-Rodrigues 2000, *pers. comm.* Andrew Thorn and Olivier Rolland). In this case study a calibrated droplet of 100 µl demineralized water was dropped on a vertical surface using an Eppendorf-micropipette. The width and the length of the path of absorption were measured immediately after the droplet had completely been absorbed. The width was measured twice, at the start and end of the droplets path and the average width was calculated. The degree of hydrophobicity of the surface was calculated by dividing the average width of the path by its length and multiplying the result by 100 for a better readability of the results. Consequently a low value indicated a water repellent surface, and a high value a water absorbing surface.

The CSM (UNI 11432-2011) consists of pressing a wet sponge against the stone surface (Fig. 2c) and measuring the water absorption from the sponge by the difference in mass before and after contact. The WAB is calculated by dividing the amount of water absorbed by the contact surface and the time of contact. In this case 5g of water was added to the sponge and the contact time was 90s.

The KT (RILEM 25-PEM 1980, NEN EN 16302 2013) consists of an open tube with a larger cylindrical body at the end which is attached to the surface of the stone by plastiline and is then filled with water. The amount of water absorbed over a certain period of time can be recorded by the reduction of the water in the graded tube (Fig. 2d). The WAB was calculated by dividing the amount of water absorbed between the 5th and the 15th minute by the contact surface and the time of contact (600 sec). The contact area is variable for each measurement due to the inwards expansion of the plastiline joint when pressing the pipe against the stone. Therefore, the contact area was calculated based on the average diameter, measured from the plastiline after removal of the pipe for each measurement.

The SH (ASTM D5873-14 2014, NEN EN 12504-2 2012) was used to measure the residual hardness of the stones. The test consists of hitting the stone surface with a controlled impact by a spring loaded steel hammer ('Classic Schmidt hammer, Type 'N' with impact of 2.207 Nm, Fig. 2e). The distance of rebound of the piston is measured and gives an indirect indication of the stones hardness, which can be used only for qualitative comparison. This measurement provides information on possible structural disintegration of the stone just below the surface (Bostenaru *et al.,* 2009). For each stone, the average value of ten consecutive measurements divided over de surface was calculated.

For interpretation of the results obtained *in situ*, reference measurements for the different methods were made in laboratory on sound fine and coarse grained dry Lede stones. The reference values are the average values with standard deviation of ten consecutive measurements for each method.

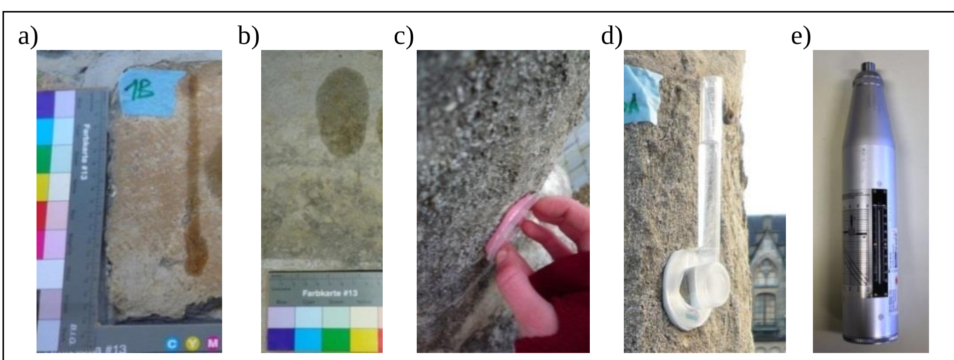

Fig. 2: Illustration of DM for a hydrophobic (a) and hydrophilic (b) surface, CSM (c), KT (d) and SH (e).

Tab. 1: Overview of field of application of WAB methods

Characteristic	**DM**	**CSM**	**KT**
Measured area	surface	subsurface to bulk	subsurface to bulk
Capillary uptake of stone	all	low uptake	elevated uptake
Sensitivity	-	0.01g	0.1g
Contact time	-	30 s up to120 s	5 min up to 1 h or more

3. Results and discussion of the test methodology

The registration of the measured stones included the description of the lithotype (fine/coarse), the observed weathering (using the ICOMOS-ISCS Illustrated glossary on stone deterioration patterns by Vergès-Belmin 2008) and the known treatments. Based on this description the measured stones were classified in 9 categories (Tab. 2).

Tab. 2: Classification of measured stones based on their conservation state

Cat.	**Stone type**	**Condition**
1	Fine	Granular disintegration and delamination. Stones used for consolidation tests.
2	Fine	Sound stone with iron rich patina at the surface.
3	Fine	Spalling of iron rich patina. Measurements made on patina, not on spalled parts.
4	Fine	Sound stone, without iron rich patina. Surface is bleached by exposure to rainfall.
5	Fine	Spalling of the iron rich patina and granular disintegration.
6	Fine	Heterogeneous stones with laminated structure. Hair cracks.
7	Fine	No iron rich patina. Surface is bleached by exposure to rainfall. Slight soiling and biological colonization
8	Coarse	Spalling, soiling and biological colonization.
9	Coarse	Differential erosion and roughening of the surface Soiling and biological colonization.

As mentioned above it has to be taken into account that variable factors (such as environmental factors or moist) can influence the measurements *in situ*. Besides, in larger monuments, only a selection of measuring points of the overall surface can be analysed. Therefore, quantitative and statistically solid results will be hard to obtain. Hence, qualitative analysis of the results, in combination with visual observations and results of other diagnoses will be more apt to assess the conservation state of larger structures such as monuments. In this study a qualitative comparison was made. Therefore the results of each method were visualized in scalebars (Fig. 3). Each scalebar contains the reference values for sound fine (Rf) and coarse (Rc) Ledestone, obtained from measurements in laboratory on dry and non-weathered stones. Below the scalebars the *in situ* obtained values for the nine categories were plotted. For evaluation of the stones condition the *in situ* obtained values were compared to the reference values, and interpreted in combination with the visual observations.

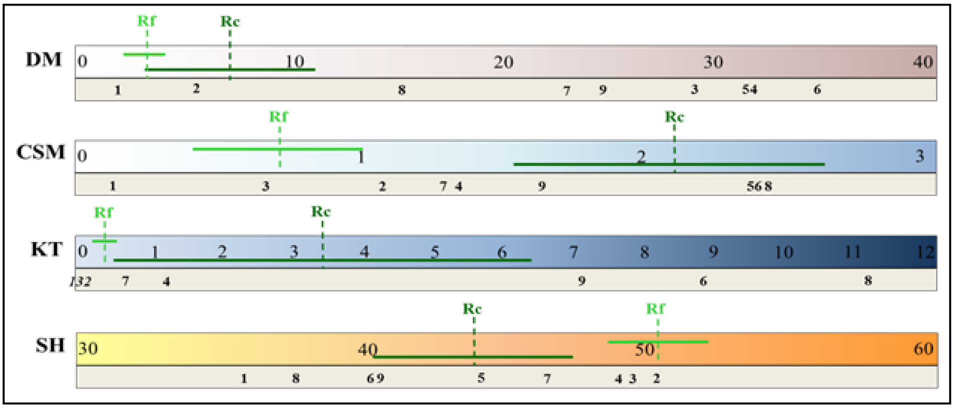

Fig. 3: Scalebars showing the results for DM (mm/mm), CSM (g/m²s), KT (g/m²s) and SH (Schmidt Hammer Rebound Value).Rf and Rc are the average reference values for fine and coarse Ledestone. Their respective standard deviations are indicated with the green lines in the scalebars. Below the scalebars the average results for each of the nine categories (1 to 9) is plotted.

For DM most categories clearly exceed the reference values, meaning that the surface absorbs more water than sound stones. From the visual observations it could be noticed that the surface is weathered and superficially rough compared to non-weathered stones. Therefore it can be assumed that the weathering of the surface causes the higher absorption. Only Cat. 1 and 2 display a remarkably lower absorption compared to the rest of the facade. Cat. 1 showed clear repellence of the droplet, which can be ascribed to the consolidation tests. As ethylorthosilicate consolidant was freshly applied, the solvent was still present in the pores, making the stone temporarily water repellent until evaporation of the solvent. The lower absorption for Cat. 2 could be related to its healthy condition, but remained lower than Cat. 3, which had a similar superficial conservation state. Consequently an additional alteration of the surface was presumed. The conjecture was made of an anti-graffiti treatment, from the fact that this hydrophobicity was only present in the lower part of the facade and that traces of washed off graffiti were present on certain stones.

CSM indicated an acceptable WAB compared to the reference values for Cat. 2, 3, 9. This indicates that these stones show no sub-superficial irregularities in their pore structure. Cat. 4 and 7 show a slightly higher, but still acceptable WAB, probably caused by the absence of the iron rich patina after natural weathering. The higher WAB of Cat. 5, 6, 8 is in accordance with their poor visual condition. Cat. 1 was clearly hydrophobic, also in depth, due to the presence of the solvent of the recent consolidant.

KT could only be used in case of higher WAB. Therefore, no results could be obtained for Cat. 1, 2, 3. The tests also failed for Cat. 5, as excessive leaking occurred, caused by the granular disintegration. For Cat. 4, 6, 7, 8, 9 measurements with KT confirm the visual observations as well as the results of CSM. This illustrates the coherence of CSM and KT for the analysis of Lede stone. Also their limitations were highlighted. KT was not able to measure the less absorbing stones of Cat. 1, 2, 3, while CSM reached its limit for testing the more absorbing stones of Cat. 6, 8, 9. It has to be mentioned that practical hindrances of KT noticed in laboratory, were clearly more predominant *in situ*, causing more tests to fail. Moreover, the standard deviation for KT was remarkably higher than for CSM.

SH confirms the visual observations and WAB measurements made by CSM and KT for all categories. Cat. 1, 5, 6, 7, 8 have a lower residual hardness and Cat. 2, 3 and 4 an acceptable hardness compared to the reference values. The inferior structural hardness corresponds to a higher WAB due to the more open structure of the weathered stones.

From the combination of these measurements and visual observations following considerations concerning the different categories could be made:

- *Cat. 1:* Weakened stone with a very low superficial and sub-superficial absorption measured with DM, CSM and KT. Freshly consolidated weakened stone.
- *Cat. 2:* Lower superficial absorption shown by DM, but normal sub-superficial absorption according to CSM. Conjecture of anti-graffiti on sound stone.
- *Cat. 2 and 3:* Stones in a good condition according to CSM and SH.
- *Cat. 4, 7 and 9:* Higher WAB and lower residual hardness compared to sound stones, but generally sound condition without precarious deterioration patterns.
- *Cat. 5, 6 and 8:* Higher superficial and sub-superficial absorption by DM, CSM and KT and low hardness by SH. Stones in a poor conservation state.

Concerning the performance of the methods, the results of CSM, KT and SH turn out to be coherent for all categories, indicating the compatibility of the methods. DM analyses the superficial absorption and demonstrates that this is not always related to the sub-superficial condition of the stone.

4. Conclusions

The WAB measuring techniques DM, CSM, KT and the hardness meter SH showed to be valuable and compatible tools for *in situ* analysis of the stones conservation state, ranging from surface to sub-surface and bulk. As expected, DM illustrated to be capable of indicating superficial alterations, while CSM, KT and SH gave an insight in the sub-superficial condition. Considering the variable factors present *in situ* and the large scale investigation, results could only be assessed qualitatively in relation to reference values of sound stones and in combination with a thorough visual analysis of the stones conservation state.

References

ASTM D5873-14, 2014, Standard Test Method for Determination of Rock Hardness by Rebound Hammer Method, West Conshohocken, PA, USA.

ASTM D7334-08, 2008, Standard Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement, 3.

Bläuer, C., Franzen, C., Vergès-Belmin, V., 2012, Simple field tests in stone conservation, in proceedings of the 12th International Congress on Deterioration and Conservation of Stone, Colombia University, New York, USA. *In press*.

Bostenaru, M.D., Prikryl, R., Török, A., 2009, Materials, Technologies and Practice in Historic Heritage Structures, Springer Science & Business Media, ISBN 9048126843, 371.

De Kock, T., Boone, M., Dewanckele, J., De Ceukelaire, M., Cnudde, V., 2015, Lede stone: a potential "Global Heritage Stone Resource" from Belgium, EPISODES, 38(2), 91–96.

Ferreira-Pinto, A.P., Delgado-Rodrigues, J., 2000, Assessment of durability of water repellents by means of exposure tests, in proceedings of the 9th International Congress on Deterioration and Conservation of Stone, Venice, Italy, 273-285.

Hall, C., Hoff, W.D., 2002, Water transport in brick, stone and concrete, Spon Press, ISBN 9780415564670, 374 .

Kourkoulis, S.K., 2006, Fracture and Failure of Natural Building Stones, Applications in the Restoration of Ancient Monuments, Springer Netherlands, ISBN 9781402050770, 592.

NEN EN 12504-2, 2012, Testing concrete in structures - Part 2: Non-destructive testing - Determination of rebound number, 8.

NEN EN 16302, 2013, Conservation of cultural heritage – Test methods – Measurement of water absorption by pipe method, 15.

RILEM 25-PEM, 1980, Recommended tests to measure the deterioration of stone and to assess the effectiveness of treatment methods, Materials and structures, 75, Paris, France, 175-253.

UNI 11432-2011, 2011, Beni culturali, Natural and artificial stone – Determination of the water absorption by contact sponge, Milano, Italy.

Vandevoorde, D., Cnudde, V., Dewanckele, J., Brabant, L., de Bouw, M., Meynen, V., Verhaeven, E., 2013, Validation of in situ applicable measuring techniques for analysis of the water adsorption by stone, in proceedings of Yococu 2012, Antwerp, Belgium, Procedia Chemistry, online paper, 317-327.

Vandevoorde, D., Pamplona, M., Schalm, O., Vanhellemont, Y., Cnudde, V., Verhaeven, E., 2009, Contact sponge method: Performance of a promising tool for measuring the initial water absorption, Journal of Cultural Heritage, 10(1), 41-47.

Vergès-Belmin, V., 2008, Illustrated glossary on stone deterioration patterns, ISBN 9782918068000, 78.