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Paper 4: Mechanical Properties of Bricks of the Coudenberg Archaeological Site in View of a Proper Conservation Strategy

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Archaeological sites are essentially submitted to decay phenomena inherent on their history as well as the environment to which they are exposed after excavation. The history of such archaeological sites is particularly related to their long-term underground conservation conditions during which they were subject to salt migration into the architectural remains. This paper describes the results of drill resistance measurements using a drill resistance measurement (DRMS) device to evaluate the mechanical damage resulting from salt crystallization pressures. Further tetraethoxysilane (TEOS) was applied on powdered bricks of often moist archaeological remains. It was noticed that TEOS serves as potential nutrient for present moulds. The mechanical consolidation effect was evaluated by comparing the hardness profiles after treatment with the reference ones. Bricks used for the construction of the Aula Magna, situated underneath the Royal Square, are characterized by severe patterns of fissures for which the damage diagnosis was built up.

KEYWORDS archaeological remains, salt damage, mechanical properties, DRMS

Introduction

Although the construction of the Coudenberg site goes back to the twelfth century, its history is governed by the dramatic fire in 1731 that destroyed half of the palace (see Paper 1, this issue). The ruins of the palace, left almost completely abandoned for forty years, as well as numerous surrounding buildings were levelled to the ground to make way for the creation of a new square: the Royal Square. The square was to be bordered with neo-classical buildings that can still be seen today. The remains of the former palace of Brussels, the Hoogstraeten House, and the former Isabella Street all constitute the excavated archaeological site of Coudenberg. They extend partly

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under the 'Royal Square' and the 'Royal Street', both being included in a tramway passage causing substantial vibrations to the underground site.

Since its opening in 2000, several individual climate interventions aiming to render the environment of the site comfortable for visitors were considered necessary. However, severe damage to the building materials occurred, mainly in the form of powdering, whereas the bricks of the Aula Magna, situated underneath the Royal Square, are characterized by severe patterns of fissures.

As the process of fissure formation continues and damage accumulates even more than a decennia after the excavation, probable causes are investigated, of which the hygric swelling and shrinkage of low fired clay minerals, the mechanical stress due to vibrations induced by the traffic, especially heavy lorries and trams, crossing the Royal Square above the Aula Magna and the salt contamination (see Paper 2, this issue).

Salt-induced decay is widely considered as one of the main phenomena which can seriously compromise the conservation of porous building materials (Coussy, 2006: 1517–47; Rodriguez-Navarro, et al., 2000: 1527–34; Steiger, 2005a: 455–69; Steiger, 2005b: 470–81). Salts may originate from different sources, which can often act simultaneously. In particular salts originating from contaminated soils are inherent in the case of archaeological sites. Once salts have penetrated the capillary pores of building materials, they are subject to crystallization exerting pressures on the pore's surfaces. Due to different environmental conditions and to their typical solubility, salts can either tend to accumulate near the surface or be deposited in the bulk of building materials. Moreover, variations in thermal and hygrometric conditions of the surrounding environment may determine cycles of crystallization and dissolution within the porous structure (Lubelli, 2006; Van & Al-Mukhtar, 2008: 317–24).

A systematic investigation on the moisture and salt load of the building materials of the Coudenberg site and a monitoring of the environment showed an important, but non-systematic, salt contamination of the building materials. The climate was characterized by extreme fluctuations of temperature and relative humidity resulting in frequent cycles of crystallization/deliquescence of salts present in the superficial layer (see Paper 3, this issue), explaining the visual deterioration phenomena of the authentic building materials for which an appropriate conservation strategy was required.

Of the many difficulties present during the restoration of historical buildings, consolidation is certainly one of the most challenging: it involves in depth irreversible treatments, whose positive outcome is not readily visible, and whose efficiency is rarely monitored on a long-term basis. Many different products have been used over the past century, but the consolidant that best seems to be standing the test of time in terms of efficiency, durability, and absence of side effects on damaged historical masonry, is ethyl silicate (tetraethoxysilane or TEOS) (Grissom, 1981). Its vast current use is also due to its easy application and versatility, for it has proven to be efficient on limestone, sandstone, and brick masonry (De Witte, et al., 1977: 190–96; De Witte, et al., 1985: 709–18; Delgado Rodrigues, et al., 1998: 653–66). TEOS hydrolyses in the presence of atmospheric moisture to form silanols, which further polymerize in a condensation reaction yielding a silicon polymer. Consolidation effects are usually evaluated in relation to the type of the dry substrate and the type of product. The often missing link between practitioners (restorers) in search of answers on the performance of a consolidant applied in humid conditions and how to apply it, and researchers who wish to give sound advice (Jovanovic, et al., 2009: 171–86; Vicentini, et al., 2012a: 61–70; Vicentini, et al., 2012b: 71–80), lies in the necessity of systematically introducing the use of convenient in situ methods of control and evaluation of consolidation treatments. For this reason this study has included drill resistance measurements using a DRMS portable drilling device (Tiano, et al., 2000: 133–50). The DRMS technique was developed as a movable analytical tool especially for direct use on buildings. It has proven to be practical and valuable for on site evaluation, for it provides a hardness profile of the stone, running up to max. 3.5 cm deep, hence allowing an evaluation of the weathering degree and depth before, and the effect and degree of penetration of the product after treatment.

Experimental part

A visual inspection of the whole Coudenberg archaeological site identified ten test areas — distributed over the whole site — for the investigation of the mechanical properties of the brickwork. The mechanical resistance properties of the outer layer of the bricks were investigated by means of the portable Drilling Resistance Measurement System (Sint Technology DRMS Cordless). After evaluation of the DRMS results, *in situ* treatments with TEOS (Artisil SVS 100, 100% solution, dry weight 51%) were carried out. The applications were done in two- or threefold. Each application was carried out wet-in-wet as to obtain a run-off of at least 10 cm. The time between successive treatments was at least 24 h. One month after the last application, the consolidation effect was evaluated by means of repeated DRMS measurements. Consequently, the hardness profile of the treated stone could be compared to that of the untreated one serving as reference. On each test area, at least three measurements before and after treatment were carried out. In this paper, the results of the most relevant zones are presented.

The hygric swelling and shrinkage of clay minerals present in the bricks of the Aula Magna were approached by measuring the weight changes of a brick sample induced by variations of RH. The brick sample was placed on a precision balance (type Sartorius ED423S-DS, maximum weight 420 g, accuracy 0.001 g) within a climatic chamber (type Firlabo SP 90-BVEHF). To enable the climate changes within the closed glass chamber of the precision balance, the glass side lids of the balance were left open over a distance of approximately 1 to 2 cm while the top lid remained closed. With this set up, the influence of the airflow within the climatic chamber on the balance's measurements is limited. The temperature and the RH were controlled in the climatic chamber as well as in the semi-closed measuring cell of the balance using climatic dataloggers (Madgetech RHTEMP 1000SS). Both measurements correlated with each other.

A severely fractured brick sample (reference weight of 373.57 g at 20° C and 40% RH) was submitted to cycles of RH while the temperature in the climatic chamber maintained constant at 20.43 ± 0.05° C. Two different tests were carried out: i) conditioning at 80% RH until constant weight followed by a decrease of the RH from 80 to 40% until constant weight is reached and ii) RH cycles between 40 and 80%.

The last programme consists of 6 hours at 40% RH, an increase to 80% RH over 7 hours, 3 hours at 80% RH, and a decrease to 40% RH over 8 hours. These RH cycles approach more or less the daily changes of RH measured in the archaeological site. The actual values of RH recorded in the climatic chamber were 42.6 ± 0.4% and 79.0 ± 0.5% RH. The weight of the sample was registered automatically by recording the average weight every minute based on an average of 30 readings.

Results and discussion

Mechanical surface properties of bricks

Figure 1 presents the areas submitted to the investigation of the mechanical properties and which are subject of the discussion in this paper. The first area is a part of the wall in brickwork masonry of the **Isabella Street** (Figures 2 and 3: zone a in Figure 1). This wall is quite dry and shows local damage in the form of powdering

FIGURE 1 Floor plan of the archaeological site of Coudenberg with identification of the zones submitted to the investigation of the mechanical properties (1. main building; 2. Rue Isabelle (Isabella Street); 3. Aula Magna; 4. Chapel; 5. Hôtel Hoogstraeten (Hoogstraeten House)).

FIGURE 2 Upper end of Isabella Street.

FIGURE 3 Detail of brickwork along Isabella Street.

of the brick. The salt contamination (see Paper 3 this issue), comprises mainly gypsum, sodium chloride, and sulphate. The evaluation of the crystallization behaviour was determined by means of the environmental control of salts (ECOS) program. The prediction of the crystallization sequence is based on the principle of the thermodynamic behaviour of salt mixtures. The ECOS output of the experimentally determined ion mixture, revealed that the salts crystallize within a broad range of relative humidity (RH), between 40 and 95%. This range of RH corresponds to the climatic variations recorded in the Isabella Street (see Paper 2 this issue). Hence, the conditions of the surrounding environment cause recurrent cycles of crystallization and dissolution within the porous structure of the bricks resulting in their powdering (Figure 3). The hardness profile obtained by means of the DRMS-device illustrates that the brick as such is very soft, having a resistance up to 3 N (Figure 4). The outer 16 mm shows an even lower cohesion that gradually increases with depth. This pattern is quite typical for building materials contaminated with salts situated in an environment of which the hygrometric conditions favour cycles of crystallization/deliquescence. In conditions of RH above the transition point of the salts, the salts absorb moisture from the surrounding environment and dissolve (deliquescence). A descending of the RH across the transition point will result in a re-crystallization of the salts. The crystallization of salts consists of nucleation and crystal growth that might exert crystallization pressures on confined pores resulting in mechanical damage, as confirmed through hardness profiles.

The building materials of zones b (Figure 5) and c, situated further down the descending **Isabella Street** (Figure 1), are again quite dry but contain especially sodium salts, partly linked to carbonates in the form of trona ($Na_3H(CO_3)_2).2H_2O$. The salt crystallization sequence of the remaining ion mixture of the first centimetre of the mortar revealed that crystallization occurs within quite a narrow range of RH, i.e. between 60 and 66%, out of which sodium nitrate is the major salt. A high nitrate contamination is also detected in the surface layer of the brick. In conditions of descending RH, especially potassium nitrate and darapskite ($Na_3NO_3SO_4.H_2O$) will crystallize between 70 and 63% RH. At around 45% RH, darapskite re-dissolves and

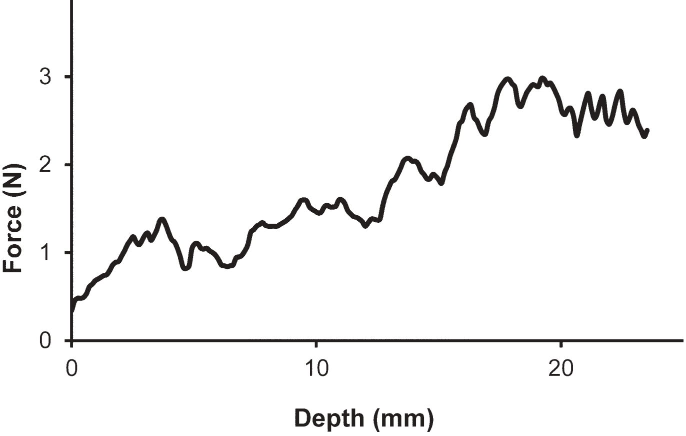

FIGURE 4 Hardness profile of a brick from zone a (Isabella Street).

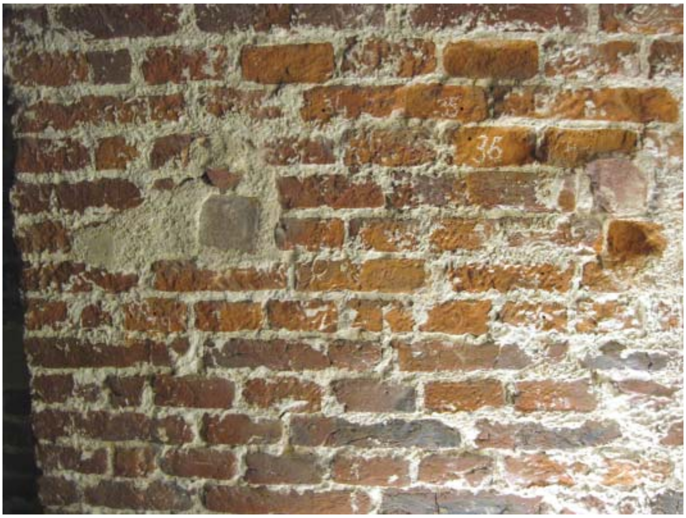

FIGURE 5 Detail of the brickwork on the lower part of Isabella Street.

thenardite (Na_2SO_4) crystallizes. According to the actual climate conditions, sequences involving the darapskite-thenardite transformation occur in spring (April–May) and autumn (October) as the RH fluctuates regularly between 40 and 50%. Sequences of crystallization of sodium and potassium nitrate and darapskite will preferentially occur during the summer (from June till mid-October). Hence, the current climate conditions stimulate cycles of crystallization/deliquescence resulting in powdering of the brick (Figure 5). The loss of cohesion resulting from the exerted crystallization pressures is confirmed by the hardness profiles, illustrated in Figure 6. The weathering depth varies from 11 mm (zone c, Figure 6a) to 22 mm (zone b, Figure 6b). Again, it concerns bricks which are originally characterized by low mechanical properties, probably due to a low firing temperature which is quite typical for the Middle Ages.

The mortar of zone d, situated in the basement level of the **Chapel** that was built in the sixteenth century (Figures 1 and 7), is wet having a moisture content up to 22 w% partially due to the hygroscopic properties for the hygroscopic moisture content reaches values up to 52 w%. The salt load of the building materials is characterized by an excess of sodium indicative for the presence of sodium carbonates, such as trona, which was also detected in the efflorescence. The crystallization sequence, as predicted by means of the ECOS model, of the remaining ion mixture is such that in conditions of descending RH salts crystallize from a RH of 60%. The environment of this zone, situated close to the inlet of outdoor air, is characterized by strong RH fluctuations (see Paper 2 this issue). In winter, cold air is blown inwards and heated up to 20° C resulting in a dramatic decrease of the RH in the Chapel down to 18%. In these dry conditions, all detected salts are continuously crystallized. From June until mid-November, frequent fluctuations of RH between 40 and 80% RH are recorded. Again, frequent transitions of deliquescence/crystallization of the present salts will result in salt damage, which was experimentally determined by means of DRMS-measurements. This soft brick shows an overall hardness of about 3 N and a weathered surface layer of 10 mm (Figure 8).

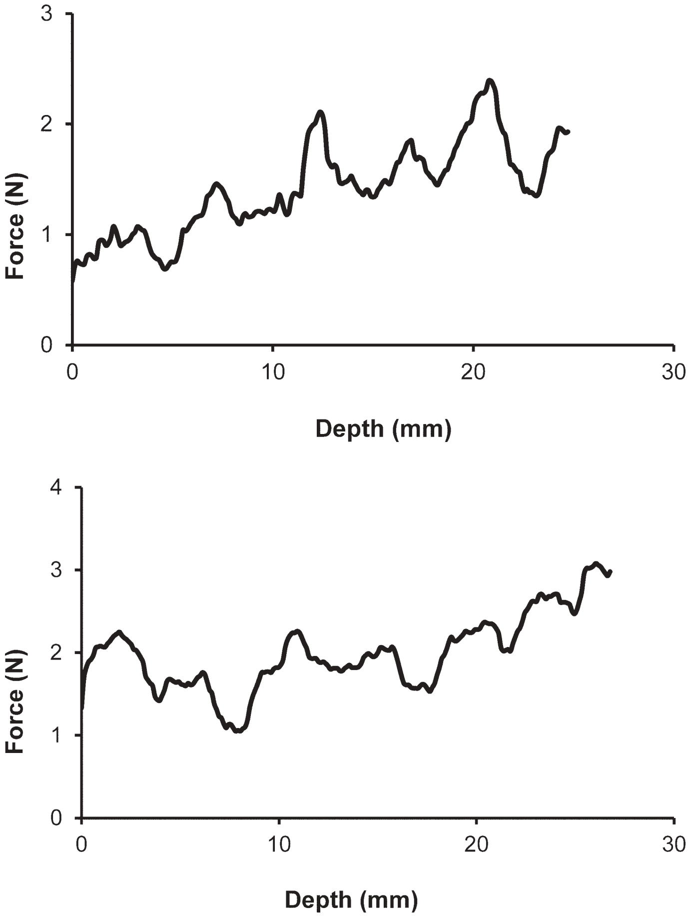

FIGURE 6 Hardness profile of bricks from zone c (top) and zone b (bottom).

Process of crack formation of the bricks of the Aula Magna remains

What remains of the **Aula Magna** (Figure 9), the previous palace's banqueting hall, are cellars located directly underneath the Royal Square. This level housed kitchens and storage rooms of the fifteenth-century palace. The bricks of the Aula Magna (zone e) are generally characterized by a complex fissure pattern (Figure 10). The presence of several fissures leads to an irregular hardness profile (Figure 11). Fissures are detected at a depth of at least 2 cm.

From a detailed visual monitoring on site, it was noticed that the process of fissure formation was still active. The microscopic investigation of thin sections of the brick revealed a general heterogeneous composition and that the clay was not fully mixed prior to the firing of the brick (see Paper 6, this issue). Hence a low quality of the brick used for the construction of the Aula Magna, and this right from the beginning, is concluded. Despite the low quality of the brick being identified, the phenomena activating the process of fissuration remained unknown. Within this investigation, following possible mechanisms were examined:

- Frost damage
- Salt damage
- Hygrothermic damage
- Damage induced by vibrations.

FIGURE 7 The Chapel.

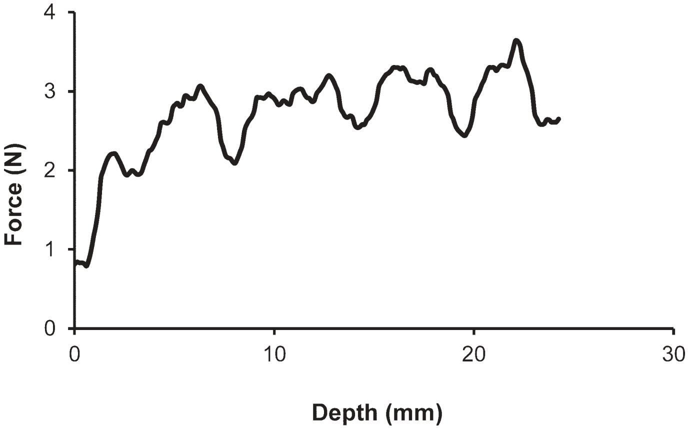

FIGURE 8 Hardness profile of the brick from zone d (Chapel).

FIGURE 9 Isabelle Street, upper part, showing the steps at the entrance to the Aula Magna.

Frost damage is related to wet building materials that are frost sensitive and submitted to freeze-thaw cycles. During freezing, liquid water is transformed into ice. This process is accomplished by an increase in volume of about 9%. Present in the pore structure, the ice formed can exert a crystallization pressure to the walls of pores resulting in frost damage. The moisture content of the brick masonry of the Aula Magna during the measuring campaign was quite low. Moreover, the monitoring of the climate showed that in winter the temperature does not cross the freezing point. Hence, the process of fissuration is not a consequence of freeze-thaw cycles.

FIGURE 10 The severely fissured bricks of the Aula Magna.

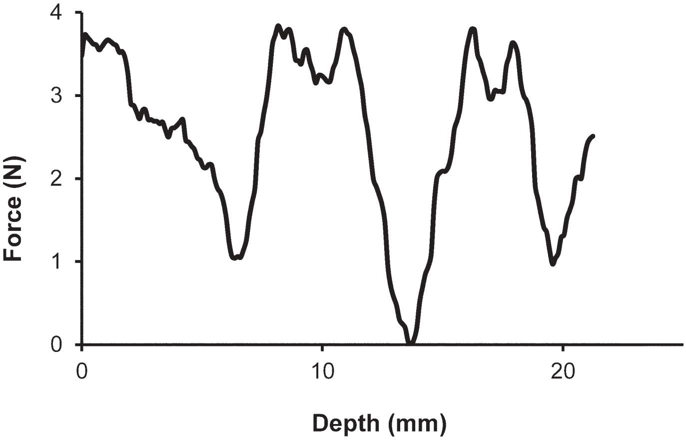

FIGURE 11 Hardness profile of a brick from the Aula Magna (zone e).

A non uniform salt contamination of the brick and the mortar was observed: some measuring points situated in cracked bricks were characterized by a low salt content, generally consisting of some gypsum, while on others a rather high salt contamination was detected. There was no relation between the salt contamination and the conservation state with respect to the presence of fissures. From this unsystematic salt load, it was concluded that salt crystallization pressure could not explain the process of the crack formation.

The examination of a damage process induced by variations of RH was justified since from the microscopic investigation of the thin sections of the bricks, a quite heterogeneous structure was noticed due to the inadequate milling and mixing of the clay used for the brick production. Hence insufficient temperature circulation and a low firing temperature might have resulted in an incomplete firing of the original clays. Simultaneous thermal analysis (Netzsch STA 449 F3 Jupiter) of a severely damaged brick sample indicated a firing temperature of 900° C at maximum. Moreover, still active compounds present in the brick were identified inducing transformations at higher temperatures.

It is known that clays, characterized by a lamellar structure, are very sensitive to variations of RH. In conditions of high RH, some clays absorb ambient moisture in their lamellar structure resulting in swelling phenomena while in conditions of low RH moisture will desorb from their lamellar structure. Frequent cycles of swelling-shrinkage might cause damage to clay containing building materials (Scherer & Jimenez Gonzalez, 2005: 51–61; Jimenez, et al., 2002: 21–27). Therefore, a severally damage brick lifted from the masonry of the Aula Magna was submitted to variations of RH. At first, the brick, pre-conditioned at 80% RH, was conditioned at 40% during which its weight was recorded continuously. After 4 days, equilibrium was reached resulting in a weight decrease of 0.8 w%. Further, the brick was submitted to cycles of RH of alternately 40 and 80% during which its weight was followed up. Figure 12 illustrates that the pattern of the weight evolution is quite similar to the one of the RH, with a delay of 6 hours. The maximum difference in weight was up to 0.3 w%,

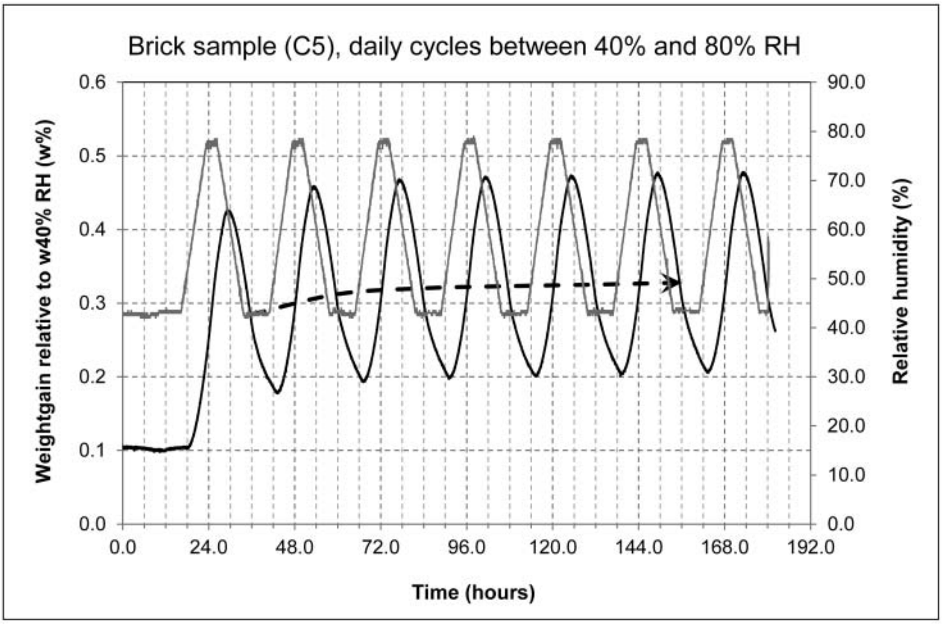

FIGURE 12 Weight evolution of the brick of the Aula Magna submitted to 24 hours cycles of RH of alternately 40 and 80% (black, solid line). The curve presenting the evolution of the RH is presented by a grey solid line. The black dotted curve is the trend of the weight increase of the brick.

which is less than half the value obtained in case of conditioning during 4 days. The weight variations resulting from the moisture absorption-desorption process provoked by cycles of changing RH were considered too low to explain the procedure of fissure formation.

Finally, the problems related to the vibrations caused by the passage of the tram and heavy vehicles on Royal Square, above the Aula Magna, remain the only plausible explanation for the damage process of the bricks. The vibrations — felt on-site — could not be quantified at present. A methodology was set up to consolidate the fissured bricks by means of a grouting mortar. The results are discussed in (see Paper 6 this issue).

Effect of strengthening treatments

Several zones revealing surface weathering due to salt damage were treated with TEOS. One month after the application, the strengthening effect was evaluated by means of DRMS-measurements. Figure 13 illustrates the hardness profiles before and after two applications with TEOS. The consumption corresponds to about 4 l.m^{-2}. An over-consolidation was generally noticed, i.e. the hardness of the weathered zone is after treatment higher than the underlying brick material. It was concluded that one application with TEOS is sufficient to increase the cohesion of the weathered zone to an acceptable level.

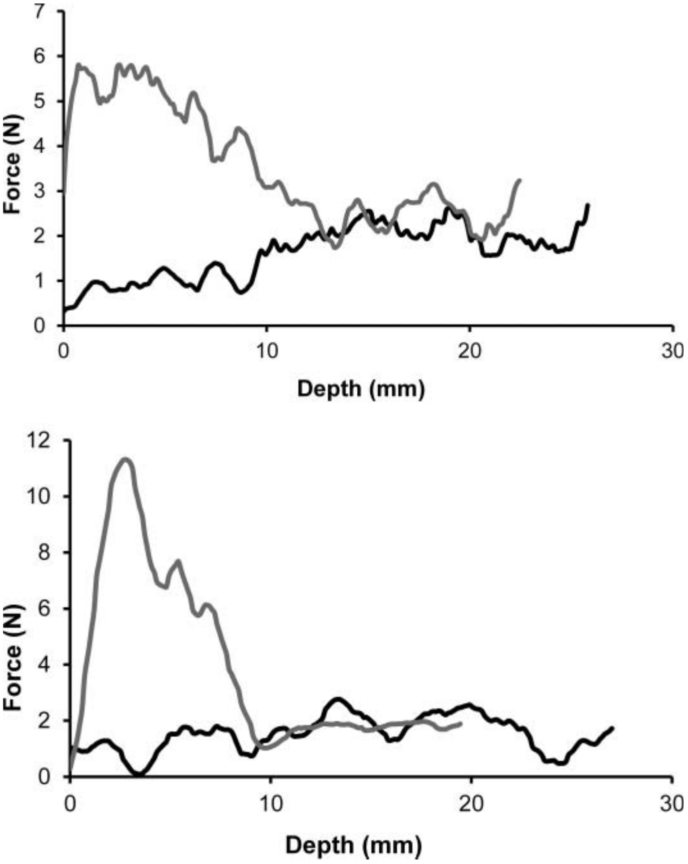

FIGURE 13 Hardness profile before (black solid line) and after (grey solid line) 2 applications with TEOS of brick of zone a (upper part Isabella Street) (top) and b (lower part Isabella street) (bottom). The consumption is about 4 l.m^{-2}.

Consequences of treatments in archaeological sites

A general problem related to archaeological sites is related to humid conservation conditions and the presence of moulds. Moulds present in building materials, as such considered as uncomfortable for visitors, are activated through the application of water based restoration products as well as organic products. Figure 14 shows a detail of the zone a of the Isabella Street, four weeks after the treatment with TEOS. Severe mould growth is noticed being incompatible with health regulations. A further treatment with a biocide based on quaternary ammonium salts was capable of reducing the mould appearance significantly.

Conclusion

The Coudenberg archaeological site is characterized by decay phenomena inherent on its history as well as the environment to which it is exposed after excavation. During the long term underground conservation salts could migrate into the building materials of the architectural remains. In this research, the mechanical destructive effect of salt crystallization was investigated through hardness profiles obtained using a portable drilling device.

The mechanical effect of a consolidation with tetraethoxysilane (TEOS) was evaluated by comparing the hardness profiles after treatment with the reference ones. It is concluded that such a treatment was capable to consolidate powdered bricks, despite the often high moisture content and salt contamination. These results confirm the ones obtained in previous laboratory investigations carried out on samples having a different salt and moisture content (Jovanovic, et al., 2009: 171–86, Vicentini, et al., 2012a: 61–70; Vicentini, et al., 2012b: 71–80). However, when applied on powdered bricks of moist archaeological remains, it was noticed that TEOS serves as potential nutrient for present moulds.

Bricks used for the construction of the Aula Magna, situated underneath the Royal Square, are characterized by severe patterns of fissures. It is concluded that the

FIGURE 14 Detail of the zone a of Isabella Street, 4 weeks after the treatment with TEOS. A severe mould growth can be seen.

fire, and consequently collapse of walls, in 1731 initiated the process of crack formation. Today, microcracks formed further develop as a consequence of the vibrations induced by the passage of the tram and heavy vehicles on the Royal Square above the Aula Magna.

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