Final project report

Surface properties of monumental stone and restoration mortar; monitoring the changes

Project no. K-116532

Summary

The surface properties of monumental stone and restoration mortars were assessed in a complex way in this research programme. The changes at the laboratory scale and on-site in the monuments were evaluated using various techniques, including lithological and mineralogical description, colour analyses, density and water absorption, ultrasonic pulse velocity testing and strength tests such as uniaxial compressive strength and tensile strength. The durability was also assessed using freeze-thaw cycles and salt-crystallisation tests. With the frame of the project, not only cylindrical specimens but also aggregates were tested. The latter ones are used as potential filling materials for mortars. Not only freshly quarried stones but weathered monumental stones were also analysed, representing various lithologies. These included porous limestone monuments (Budapest), volcanic tuffs (Sirok Castle), as well as various types of limestone and granite (Székesfehérvár ruin garden). Samples of weathered surfaces were collected from various monuments of Europe before Covid and were tested from mineralogical and morphological points of view, helping in the clarification of pollutant sources. Repair mortars and their aggregates were also tested. Curing conditions and the role of various aggregates in mortar properties were also analysed. Samples representing a dual system: porous limestone substrate, and repair mortar were also made. The binding ability of mortar to the stone surface and the importance of pull-off tests were also assessed. Special emphasis was placed on new research directions focusing on aggregates that can be sued as part of repair materials. New monitoring techniques such as TLS and drones were used to evaluate the weathering-caused instability of cliff faces. The durability of aggregates and long-term behaviour were also assessed in order to move the emphasis from on-site tests (that were not possible due to Covid restrictions) to laboratory testing of repair materials. Stress factors such as freeze-thaw or salts and the behaviour of consolidants that can be used to repair materials were also evaluated. In summary, the Covid pandemia hampered our research activities and caused a delay, but with the prolongation of the project and finding slightly new directions, the research aims were fulfilled. Within the research programme, ***21 journal papers were published***, of which ***17 are listed in WoS***. In addition to this, before Covid several international conference presentations form part of the dissemination of results.

Research results

The research results are arranged according to research periods since Covid pandemia hampered our activities and required the adjustment of some of our research plans.

At the beginning, an overview of the sustainable use of stone materials and their changing use in human history, as well as the stone decay, have been outlined (Prykril et al. 2006), providing a general context of this current research. The first part of the research – prior to Covid - focused on the selection of stone and mortar and laboratory tests. Stone blocks were obtained from Sóskút quarry (porous Miocene limestone), Süttő (Pleistocene travertine) and compact red limestone of Tardos (Jurassic). For comparison rhyolite tuff from Eger and Sirok area was also collected. These stones represent the most common lithologies in Hungarian monuments. Test specimens were prepared under laboratory conditions, and parameters such as petrological description, spectral analyses (colour), ultrasonic pulse velocity, water absorption and conductivity, surface roughness and slip resistance, uniaxial compressive strength and tensile strength were recorded. Additional tests were made on stone consolidants testing the effect on strength and pore-size distribution of porous limestone. Besides stone, repair mortars used for monumental stone repair were also tested. Test specimens representing different binder/aggregate ratio were cast and physical parameters and composition were measured. Special emphasis was put on the stone/mortar interface testing. Samples representing a dual system: porous limestone substrate and repair mortar were made. Monuments were selected and lithologies and decay forms were mapped. The selected buildings and structures include limestone monuments in Budapest (Citadella, Central Building of Budapest Technical University and Economics, a retaining wall in the 2nd district) and structures made of rhyolite tuff in NE Hungary, Eger Castle and Sirok Castle. In the laboratory scale the comparative colour measurements of various fresh (non-weathered) lithologies have been elaborated and the applicability of this method was tested on various lithotypes (Antal et al. 2017), i.e. colour is a parameter that can be used to asses stone decay and can be used as a monitoring parameter of surface processes. Our laboratory tests have shown that the consolidation significantly modifies the pore-size distribution of porous limestone (Pápay and Török 2016). These results have proved that the moisture content and temperature monitoring of consolidated stone surfaces require a slightly different approach than the weathered stone surfaces. The interpretation of such results needs further studies. The properties of repair mortars and especially strength and water absorption, were measured (Szemerey-Kiss and Török 2016). The failure mechanism of stone/mortar interface was also assessed by using porous limestone and four types of repair mortars. Besides pure mortars, additional test specimens were also made by adding 50wt% of porous limestone aggregate. The most important outcome of the tests is that 4 different failure mechanisms were identified. The bond between the mortar and stone is strongly influenced by surface pre-treatment (bond-rich layer) and the added limestone sand aggregate (50%) increased the surface roughness and water but reduced the pull-off (Szemerey-Kiss and Török 2017).

In the next phase of research, it was still possible to perform on-site tests of selected buildings with the application of new techniques. The laboratory tests were made on porous limestone (Miocene), travertine and rhyolite tuff as well as on commercially available mortars and laboratory made repair mortars. Lithological and mineralogical descriptions, water absorption, freeze-thaw tests and salt weathering simulations were made. Density, ultrasonic pulse velocity and strength parameters were also measured with colour analyses. Pull-off strength of repair mortar stone interface was also tested.

On site tests were carried out to describe weathering forms and their distribution. One study site where various lithotypes occur was the ruin garden of Székesfehérvár. From that site field and laboratory data were evaluated in collaboration with a Greek colleague, it was possible to compare the behaviour of various limestone types that are exposed to the same environmental stresses. The weathering forms and properties of fine-grained and medium-grained porous oolitic limestone and travertine showed significant differences. It was possible to perform micro-drilling resistance tests. The porous limestone had a larger drilling resistance at the upper surficial zone (upper 1-2 mm) than below, indicating that a crust was formed at the surface. The drilling resistance of porous limestone under shelter had different resistance pattern, the upper zone has lower resistance which is followed by increasing drilling resistance at depth. The shelly limestone, the sandy calcarenite and red compact limestone also showed an increase in drilling resistance at the topmost app. 2 mm zone. It was possible to demonstrate that non-invasive and micro-destructive techniques helped in the identification of endangered zones at monuments and these test methods help in the condition assessment of stones at heritage sites, where sampling is limited (Theodoridou and Török 2019). (Theodoridou and Török 2018). Moisture content and surface strength were measured on lithologies that are sensitive to weathering. Volcanic tuff structures were studied (Germinario and Török 2019). Samples of weathering forms were also collected for further textural, mineralogical and elemental analyses. A new technique was also used. UAV (drone) and terrestrial laser scanner (TLS) were applied at Sirok Castle to detect morphological changes of cliff faces and stone structures (Török et al. 2018). Besides Hungarian monuments, international sites with limestone monument were also analysed. Limestone samples were obtained from Assisi (Italy) and local structures were also studied with the help of Italian colleagues. Additionally dust samples and host carbonate rocks from various monuments of Europe (Belgium, Czech Republic, France, Germany, Italy, Poland) and also from Hungary were collected to obtain a broad overview on how limestone substrates behave under different pollution regimes (Farkas et al. 2018). This data set can be used to design the exposure trial tests of exposed and non-exposed sites.

The next phase of the project focused on on-site testing of monuments and detecting long-term changes on stone and mortar surfaces. Parallel to this, laboratory tests continued to assess the compatibility of stone and mortar and to test physical changes caused by environmental stresses, such as water, salts, freeze-thaw and elevated temperature. Additionally, the analyses of settling dust samples collected from monuments also continued to assess the behaviour of limestone under different climatic conditions and pollution fluxes. The first numerical calculations were also made using computer codes that simulate pollution fluxes.

Large amount of data on surface properties of stones were recorded during the project. On site measurements were focused on selected buildings and sites. The porous limestone and travertine of the central building of Budapest University of Technology and Economics were studied in details since the pandemia hampered our travel. More than 22,000 collected data were obtained from this site. The sensitivity of porous limestone to environmental stresses was also documented (Pápay and Török 2018); therefore, consolidation trials were also made to improve the durability of these stones. The consolidated porous limestone samples were exposed to heat and salt. Within the frame of the project with researchers from ETH Zürich we compared the thermal behaviour of five compact limestones. An interesting finding of this experiment was the role of stylolites in the durability of these low porosity limestone (Nevin et al. 2018). The composition of settling dust was also measured from many countries and the results were published this year. The data suggests that not only the climate influences the decay of limestone, but also pollution fluxes, thus there is a strong link between the deterioration of limestone and background pollution. Therefore we started some experiments with pollution fluxes under laboratory conditions (stone tablets were exposed to diesel engine exhaust gas). The physical changes such as weight loss, ultrasonic pulse velocity and porosity differences were detected. The results of clean samples and diesel soot covered samples were compared (Farkas and Török 2019a). Computer simulations of pollution fluxes (dust) and stone surface settling were also made (Farkas and Török 2019b). Stone decay tests also continued. The two sites where these techniques were tested represent the two most sensitive rock types: porous limestone (Budapest) and rhyolite tuff (Sirok). The results from Sirok and Eger Castle were published (Török et al. 2018, Germinario and Török 2019). The rhyolite tuff of Sirok Castle and Eger Castle was also analysed under laboratory conditions, too. Collected samples were tested not only for physical parameters but also for mineralogical changes and geochemical changes (Germinario and Török 2020). The compatibility of stone mortar is also a key research area of this project. The experiments focused on the freeze-thaw durability of mortars and porous limestone. To measure the frost resistance of adhesions the individual and bonded limestone and repair mortar specimens were subjected to freeze-thaw cycles (test method is given in EN 12371). The weight loss was continuously monitored. The adhesion bond between the stone and the mortar was also observed during the cycles. Composition of samples and petrography were tested in thin sections. The petrographic and textural changes were recorded before and after the freeze-thaw test. Mercury intrusion porosimetry (MIP) was used to test the changes in the pore size distribution of mortars and stones. The main decay features observed at laboratory conditions were the rounding of the edges of cubic test specimens, delamination, blistering, powdering and granular disintegration. Some samples were broken during the frost tests; stone/mortar interface was dismounted and typical failure patterns were described. The tests demonstrate that the small pores (around 0.1 micrometres, measured by MIP), control the weathering behaviour of tested porous materials (Török and Szemerey-Kiss 2019).

Besides the main line of the research programme due to the Covid we had to implement new research directions that are feasible in restricted travel conditions. This second part of the research activity is linked to stone materials that can be used as aggregates in binders. For the latter, one of the most common volcanic stones of Hungary, andesite, was considered. In this context, strength parameters and durability in terms of abrasion was compared. First the long-term durability of this aggregate was tested (Czinder et al. 2020). Cylindrical test specimens were tested, and aggregates from the same material were crushed and analysed using abrasive tests (Czinder and Török 2021a). Additionally, the role of salt crystallisation was also assessed using it as a tool in the durability analysis of aggregates (Czinder and Török 2021b). A new focus area was the consolidation and testing of stone durability when consolidants are applied in the repair of monumental stones. For these tests, Miocene porous limestone cylindrical samples were consolidated with four different ethyl silica-based consolidants. After consolidation, the samples were exposed to freeze-thaw cycles and salt crystallisation tests. Parameters such as water saturation under atmospherically pressure, capillary water absorption indirect tensile strength tests will be measured on treated and untreated samples to estimate the effect of consolidation. In addition, mass loss due to salt crystallisation and freeze-thaw effects will be tested (Pápay et al. 2021). The thermal behaviour (heat exposure) of consolidated stones was also analysed (Pápay and Török 2018).