



Methodology for the characterization of Cultural Heritage materials and their degradation state, using ex-situ laboratory analysis and microscopies: the HERACLES project approach

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The in-depth knowledge of material properties and their weathering behavior is an important basis for developing and planning effective and cost-effective preservation actions, considering the aim of the HERACLES project (HERitage Resilience Against CLimate Events on Site, GA 700395). The well-accepted systematic approach to a sustainable CH asset material preservation can be organized into three phases: anamnesis, diagnosis, and therapy. The anamnesis is followed by the diagnosis. Here, the interdisciplinary research of geologists, chemists, architects, engineers, etc. is required. The overall aim of diagnosis is analysis, quantifications, interpretation, and assessment of material deterioration and damage rate, by considering weathering factors, processes and weathering characteristics as well as the material type, the monument/asset characteristics and the time factor. At the same time, optimization of diagnosis procedures is a crucial step. An accurate diagnosis, together with a correct anamnesis, is the basis of preventive preservation measurements, such as cleaning, desalination, surface protection, materials repair, and replacement, etc.

The diagnosis methodological approach includes in-situ materials investigation and ex-situ laboratory analysis and it is focused on the different scale of deterioration. Particularly, the in-situ analyses are applied to obtain information on visible deterioration at mesoscale and at the microscale, while the laboratory (ex-situ) analysis provide information at the microscale and at the nanoscale (non-visible deterioration).

Sampling campaigns are necessary for the detailed laboratory analysis. The samples are collected from deteriorated materials (stone, mortars, concrete etc.), located in problematic areas, such as those in close contact with the ground or those exposed to weathering factors. Fresh rock samples are also collected from the original building rocks for comparative analysis.

The laboratory analyses of the samples can reveal detailed and accurate information about the not visible degradation. It can also confirm or contradict the suppositions and theories from the in-situ investigations (macroscopic investigations).

The laboratory (ex-situ) materials characterization is performed by means of different analytical procedures and allows to assess different properties such as composition, micro-texture, porosity properties, etc. Therefore, it is crucial to select the appropriate method, or combination of methods, which ensure the most effective approach to document the materials degradation issues.

The overall approach towards a systematic analytical strategy for material characterization and their degradation state within HERACLES project can be outlined as follows:

- Definition of material composition through X-ray Diffraction (also micro) (XRD), X-ray Fluorescence (also micro) (XRF), Fourier Transform Infrared Spectroscopy (FTIR), micro Raman, spectrophotometry (UV-Vis-NIR), X-ray photoelectron spectroscopy (XPS), Laser Induced Breakdown (LIBS)
- Definition of material structure and texture through Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), micro Raman, Non-Linear microscopy (NL), 4D Surface Volume Topography
- Definition of physical properties through Thermogravimetry, Differential Thermal Analysis and Differential Scanning Calorimetry (TG-DTA and DSC) and Ellipsometry.

The methodology developed in the HERACLES project aimed at addressing not only the needs of the four HERACLES test-sites (Knossos Palace and Koules Venetian Fortress in Crete, Greece and Consoli Palace and Town Walls, in Gubbio, Italy), but also for showing that it could be of more general application.