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Study and testing of the "Porta Férrea" stone materials, University of Coimbra, Portugal

Lidia Gil Catarino (1), Francisco P.S.G. Gil (2), and Mário Quinta-Ferreira (1)

(1) Geosciences Center, Dep. Earth Sciences, University of Coimbra, Portugal (lidiagil@dct.uc.pt; mqf@dct.uc.pt), (2) CFisUC Center, Dep. Physics, University of Coimbra, Portugal (fgil@fis.uc.pt)

For the conservation and restoration of Porta Férrea (Iron Gate), the main entrance to the heritage buildings of the University of Coimbra, several sampling and testing, as well as specialized monitoring during the work, were done:
1. Whitewash and burnish (on stone or on plaster):
- identification of pigments using X-ray diffraction (XRD) and/or X-ray fluorescence (XRF) and/or Raman spectroscopy; - determining the number and thickness of layers (under the stereographic microscope); - presence of organic substances to identify the binder (using Raman spectroscopy).
2. Limestone weathering:
- identification of salts and oxides (XRD and/or XRF); - measurement of porosity and water absorption to assess the possible treatment penetration zone.
Samples were collected in stone flakes detaching from columns and dome and in mortar detaching from the dome; in surface weathering powder from sculpture E4 and yellow whitewash of sculpture E3, summing 20 samples. Additional 4 samples were collected to study the yellowish coloring in the East and West faces of the portal and to identify the pigments and binders by Raman spectroscopy.
A few main considerations can be pointed out.
- The East Portal is less exposed to temperature variations in winter, promoting the retention of both rainwater and air moisture, that combined with the high porosity and high water absorption induce degradation by dissolution. Halite and weddelite salts were identified by XRD. The gypsum would be the result of sulfation processes associated with cycles of wetting/drying.
- The exploration hole performed in the E4 sculpture allowed detecting salts until 3 cm deep.
- Lead was identified particularly in the East side, associated with sulfur and possibly zinc, due to atmospheric contamination.
- The yellow pigments match ocher (α-FeO (OH)), which is also consistent with the results obtained by XRF.
- No organic substances could be identified by the Raman spectroscopy.
- The material collected in the alveoli of the West Portal columns seems to correspond to anthropogenic fills of preexisting cavities.
- The water absorption and porosity of the limestone are high, promoting the exchange between the exterior environment and the sample interior, favoring degradation processes.
- The temperature and relative moisture amplitudes in the west face is the higher, promoting successive cycles of wetting-drying favored by sun exposure and incidence of NW winds. In the east side the type and intensity of degradation is dependent on the constant presence of water, boosted by the atmospheric contamination.
The results allowed preparing a wised intervention in order to preserve a monument that is part of the UNESCO Word Heritage.