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2D high lateral resolution synchrotron µXRF and µXRPD Mapping for Enhanced Characterization of Inorganic Treatments for Stone Consolidation

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Stone consolidation is a crucial practice in the conservation of building and stone artifacts of Cultural Heritage. To prevent the irreversible loss of stone material, with significant cultural value, various consolidants have been studied over the last decades. However, the diverse properties of stone materials (limestone, biocalcarenite, and sandstone), the state of conservation of the artifacts, and the use of diversified consolidants in the field have led to unresolved challenges in the consolidation practice. A comprehensive understanding of the consolidation process remains a significant analytical challenge, particularly regarding the effects induced by inorganic treatments (i.e., nano limes, ammonium oxalate, diammonium hydrogen phosphate) and the composition of major and minor crystalline phases formed within the treated stone materials (Sena da Fonseca, 2023).

This research is based on p an innovative approach using SR-µXRF and SR-µXRPD mapping carried out at the ID13 beamline of the European Synchrotron Radiation Facility (ESRF, Grenoble, France) to investigate the complex mixture of crystalline phases formed with ammonium oxalate solution (AmOx - (NH4)2C2O4) and diammonium hydrogen phosphate (DAP - (NH4)2HPO4)) on carbonatic porous stone substrates, whose phase identification is an analytical challenge (Osticioli, 2017). Here, the combined µXRF and µXRPD mapping approach has been used to study transversal cross-sections of AmOx and DAP-treated samples as well as double-treated ones (AmOx followed by DAP, DAP followed by AmOx). Noto limestone, a highly porous carbonatic stone widely used in the historical buildings of the Noto Valley (UNESCO's World Heritage List), was selected as a carbonate substrate. This synchrotron-based methodology thanks to its sub-micrometric resolution and high sensitivity (Cotte, 2022), resulted in an effective tool for studying the crystalline phases formed during consolidation improving our knowledge of the chemical and mineralogical transformations occurring within the stone matrix (Conti, 2023).

The PyMca ROI imaging software was employed as a tool to create average XRPD patterns over a selection of pixels and perform principal component analyses as well as batch-fit the XRF data (Cotte, 2016). The qualitative analysis of XRPD patterns of different regions of interest (ROI) was carried out. The analysis of distribution maps of specific crystalline phases with maps of marker elements via an RGB correlation enabled the identification of the crystal-chemical composition of the newly formed phases as well as the exploration of their spatial distribution at the microscale level, shedding light on the existence of stratigraphy of phases within the stone structure. The new calcium phases have been identified and localized in the calcite matrix; novel oxalate and phosphate phases have been observed in a mixture with phases detected in previous studies. Moreover, this detailed analysis offered crucial insights into the phases' penetration depth. Additionally, it was possible to investigate the distribution of the new phases below the treated surface down to the inner portions of the stone matrix: this diffusion is essential to consolidate decayed portions and reconnect them with the sound original matrix. Furthermore, this methodology examines the crystallinity of these phases, and whether the newly formed phases exhibit preferential crystal growth. Understanding these aspects is crucial for optimizing consolidation treatments and promoting a homogeneous diffusion of treatments.

The experimental findings indicate that SR-µXRF and SR-µXRPD mapping is an efficient methodology to study the effects induced by inorganic-mineral conservation treatments for stone consolidation and gain a deeper understanding of consolidation mechanisms.

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