

MS43-1-2 2D high lateral resolution XRPD mapping and micro-Raman analyses for the in-depth characterization of red stains on heritage marbles

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A. Suzuki ¹, E. Cantisani ¹, M. Ricci ², S. Vettori ¹

¹Institute of Heritage Science-National Research Council ISPC-CNR - Sesto fiorentino (FI) (Italy), ²Department of Chemistry “Ugo Schiff”, University of Florence - Sesto fiorentino (FI) (Italy)

Abstract

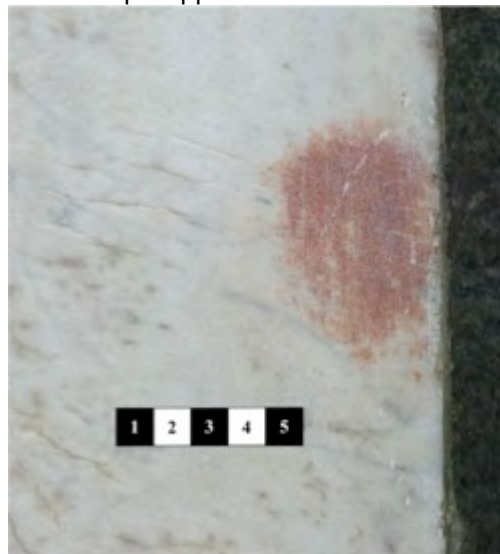
Red stains are a common alteration phenomenon on heritage marbles all over the world, very difficult to remove and affecting the aesthetical appearance and cultural and economic values of manufacts. The causes of this phenomenon have been debated for decades and several possible origins were identified (such as microorganisms, previous restoration treatments etc.). In the context of the main Florentine monuments, previous multi-analytical studies demonstrated that the red staining in these cases is due to the presence of minium (lead tetraoxide) concentrated prevalently in the calcite crystal boundaries, but also the presence of other Pb crystalline phases was suggested by preliminary micro-Raman analyses. Possible sources of Pb, the role of microorganism, the environmental conditions and physical characteristics of marbles were also evaluated [1].

In order to identify the presence of other Pb compounds and their distribution in the red stained areas, samples were collected from the marble cladding facades of the Baptistery of San Giovanni and Santa Maria del Fiore Cathedral in Florence (Italy). Thin sections of these samples were analysed with 2D high lateral resolution (few microns) X-Ray Powder Diffraction (XRPD) mapping (map size of 400 x 400 μm^2) available at ID13 (ESRF synchrotron facility) [2] and the same areas were analysed with micro-Raman spectroscopy. The mapping at the micrometre scale of the crystalline phases with both techniques has been successful in shedding light in the in-depth distribution of the several lead oxide phases present in the marble red stains.

References

- [1] Cantisani E., et al., *Analyst*, 2019, 144 (7), 2375 – 2386
[2] Cotte M., et al., *Molecules*, 2022, 27, 1997.

Macroscopic appearance of a marble red stain.



Cross section of a red stain sample.



250 um