

Synchrotron micro-XRD and XRD-CT reveal newly formed lead-sulfur compounds in Old Master paintings – Supporting information

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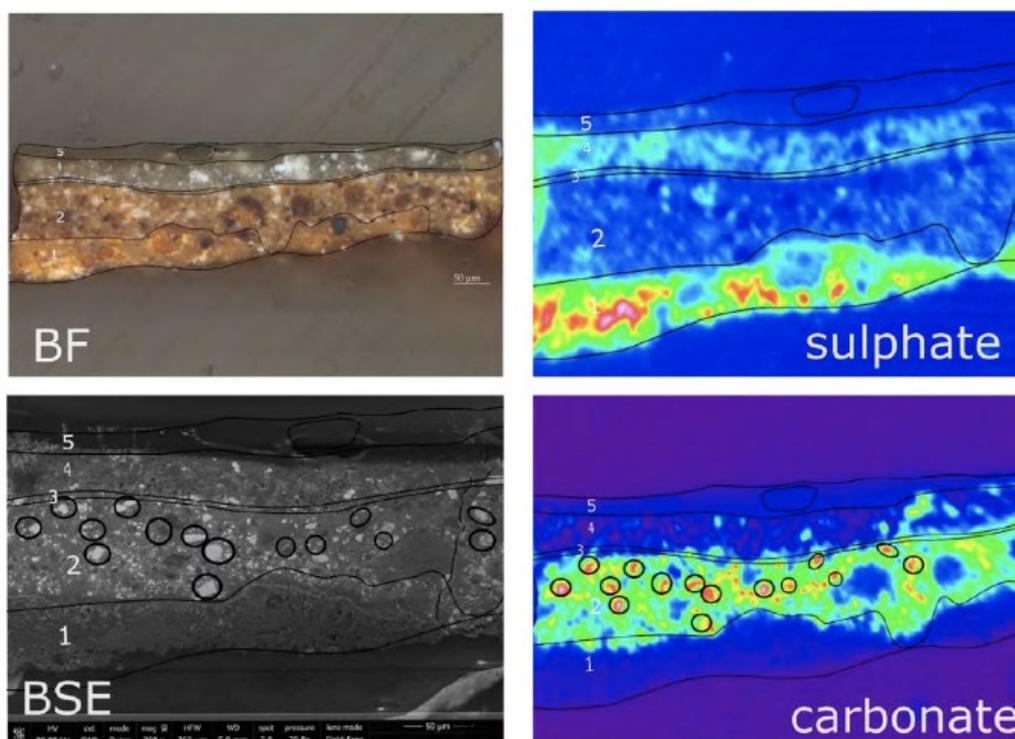
SEM-EDX and ATR-FTIR measurements

Scanning electron microscopy (SEM) images were recorded on each cross-section using a Thermo Fisher Nova Nano SEM 450 electron microscope. Backscattered electron images were taken at a 20 kV accelerating voltage, and eucentric working distances between 5 and 6 mm, low vacuum (50-70 Pa) using a Gaseous Analytical solid-state, back-scattered electron detector (GAD). For the purpose of elemental identification, energy dispersed X-ray spectrometry was performed using the coupled Thermo Fisher EDX system.

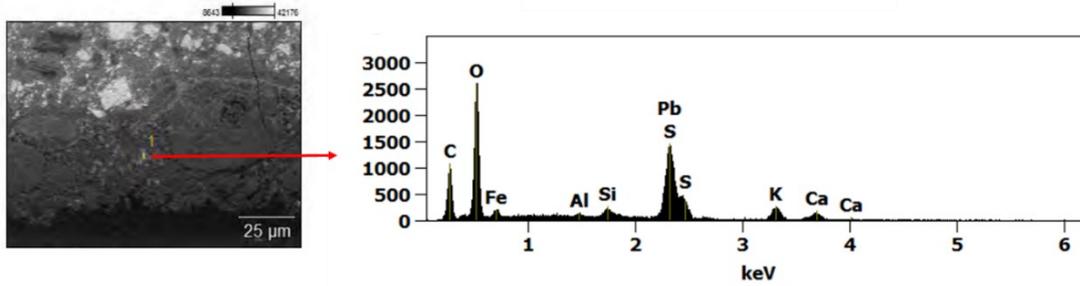
Attenuated total reflectance FTIR micro-spectroscopy (μ -ATR-FTIR) analysis was performed using a Perkin Elmer Spectrum 100 FTIR spectrometer coupled to a Perkin Elmer Spectrum Spotlight 400 FTIR microscope equipped with a germanium ATR imaging accessory, and a 16x1 pixel linear mercury cadmium telluride (MCT) array detector.

Weenix

1) FTIR mapping

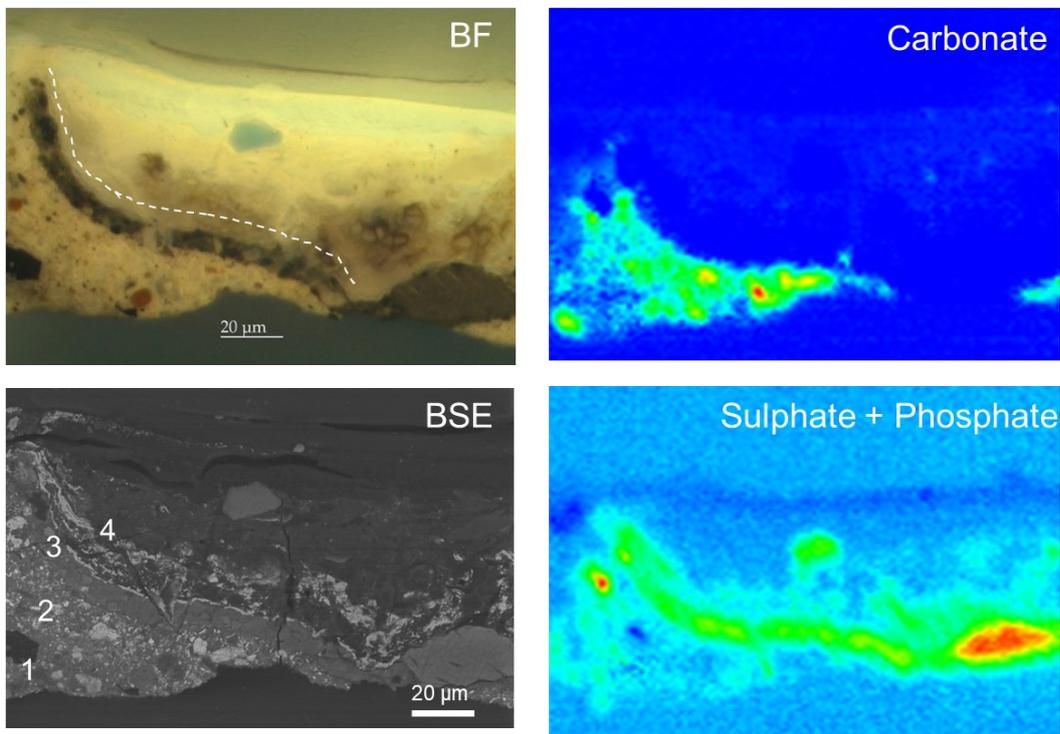


2) SEM-EDX



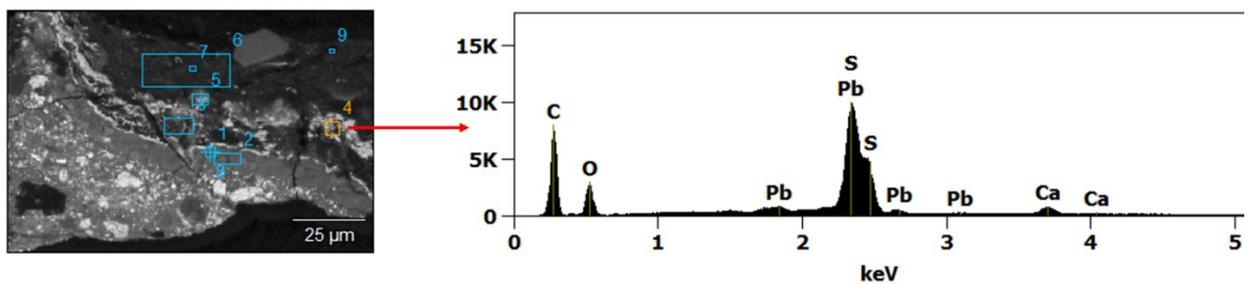
Rembrandt

1) FTIR mapping



For this sample, the FTIR signal of sulphates and phosphates was difficult to discriminate, the presented map is a sum of those two signals.

2) SEM-EDX



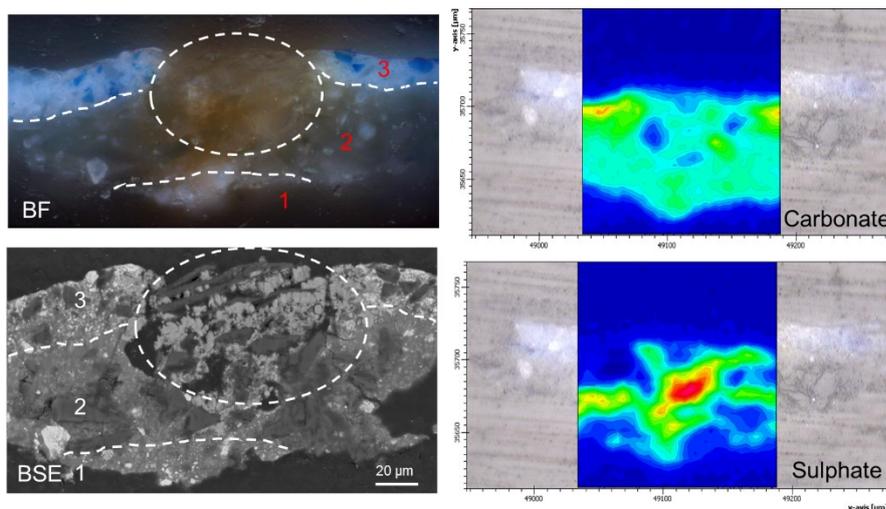
Wouwerman

For this sample, the experimental conditions were different :

For SEM, the sample was analyzed using an XL30 SFE high vacuum electron microscope (FEI, Eindhoven, The Netherlands) equipped with an EDX system with spot analysis and elemental mapping facilities (EDAX, Tilburg, The Netherlands). Backscattered-electron images were recorded at 20 kV accelerating voltage, at a 5 mm eucentric working distance and a spot size of 3, which corresponds to a beam diameter of 2.2 nm with current density of approximately 130 pA. EDX was done at a spot size of 4, which corresponds to a beam diameter of 2.5 nm and current density of 550 pA, to obtain a higher count rate. Mapping settings were 256 x 200 matrix, 200 ms dwell time, and 17 ms amplitude time.

ATR-FTIR measurements were undertaken at the IPANEMA Lab, on a Bruker HYPERION 3000 FTIR microscope using a focal plane array (FPA) detector, coupled to a VERTEX 70 FTIR spectrometer. An ATR hemisphere sample accessory was used equipped with a base germanium hemisphere crystal with a 1 mm diameter flat top that is brought in contact with the sample. The spectra were collected with 4 cm^{-1} spectral resolution in the range $4000\text{-}900\text{ cm}^{-1}$ averaging 64 scans. Data were processed using the OPUS 7.2 software.

1) FTIR mapping



2) SEM-EDX

