Energy-Filtered Secondary Electron Imaging for the Study of Degradation Processes in Paintings

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Energy-Filtered Secondary Electron Imaging (EFSE) is complementary to scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS), two techniques that are well-established for cultural heritage analysis and routinely used. EFSE shows the sample only in a very narrow secondary electron energy window at a time and the variations in signal can reveal minute differences in chemical composition [1]. Compared to EDS it is about a hundred times faster and better spatially resolved. We have developed a miniaturized EFSE device that can be inserted inside a SEM chamber onto the sample holder [2]. It has a 0.5 mm entrance slit, 0.02% energy resolution for input angles ±6°, good S/N ratio.

EFSE was used in a study of the degradation of arsenic-based pigments in wall paintings with the aim to explain the origin of mimetite Pb₅(AsO₄)₃Cl as the corrosion product resulting from originally used arsenic- and lead-based pigments. Unusual mimetite was found on valuable Gothic wall paintings uncovered in the St. Gallus church in Kuřívody, Northern Bohemia [3]. A series of model samples imitating the possible composition of original paint layers was exposed to various corrosive agents (rising moisture, salt solutions, oxalic acid or their combinations). Samples in which the presence of mimetite was detected by XRD were then submitted for a detailed study by SEM, EDS and EFSE. Fig.1 shows an SEM image of a one such microsample, in which EDS analysis verified the prevalent presence of As in the smooth area and Pb in the rough area.

Fig.1 - On the left, SEM image of a sample (area 70x50 μm) containing mimetite. On the right, EFSE analysis of the area at 2 keV primary energy shows a marked difference between the peaks of the most probable secondary electron energies of the two respective areas, by as much as 1.5 eV.


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