EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Luminescence properties of preserved and altered Cadmium Yellow oil paints	**Experiment number**: HG194
Beamline: ID16B	**Date of experiment**: from: 03/11/2022 to: 06/11/2022	**Date of report**: 12/12/2022
Shifts: 9	**Local contact(s)**: Valentina Bonino	*Received at ESRF:*

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Context and motivation of the research:

The proposal is related to the characterization of cadmium yellow (CdY) oil paints through XEOL measurements combined with XANES at Cd K-edge. CdY paints are based on the compound cadmium sulfide (CdS), a well-known luminescent semiconductor. The NIR luminescence of CdS has been widely used for its non-invasive identification in paintings [1, 2], although its origin and its eventual modification with paint degradation have not been conclusively established. In order to effectively exploit the luminescent emission as a diagnostic conservation method and as a possible marker for CdY deterioration process [3], it is essential to establish its origin and whether there is a spatial correlation between the optical emission and the chemical composition.

The experiment conducted during this beamtime was aimed at characterizing the optical emission of different unaged and artificially aged CdY model paints through XEOL point measurements and at correlating the X-ray induced luminescence with the chemical composition obtained at the same point through XANES at Cd K-edge. The modification of the emission with the variation of the excitation energy (before, at and above the Cd K-edge) was also evaluated by acquiring three XRF and XEOL maps at the three different energies (26.635 keV, 26.695keV and 26.755keV).

Analysed samples:

The model paint samples were prepared by mixing cadmium yellow pigments (synthesized from two different historical recipes and commercially available) with linseed oil. The paints were artificially aged for 45 days

under light and at high relative humidity conditions (RH ~80%). For each artificially aged sample, a corresponding sample was prepared and kept in the dark at ambient conditions as a control. Thin sections were prepared to map the distribution of the chemical species through the cross-section. Pigment powders were also measured by dispersing them on tape.

Status of the data processing:
Data are still under analysis.

Main results obtained:
Measurements performed on powder samples did not detect any XEOL signal. Therefore, it was not possible to verify the behaviour of optical emission as a function of excitation energy and to compare the emission of pigments synthesized with different methods.

The paint samples show very weak optical emission, contrary to expectations. This can be ascribed to the combination of the high heterogeneity of the samples, the low concentration and high dispersion of CdS and the small beam size (62 × 60 nm, h × v). Due to the low luminescence signal, it was not possible to assess its variation as a function of energy. Acquisitions with a good signal-to-noise ratios generally require integration times in the order of 5-50 s, resulting in sample damage. Future experiments conducted with a de-focalised beam or a larger beam could help to solve these problems and to improve the acquisition of the luminescence signal.

References:

1. Thoury M, Delaney JK, Rie ER de la, et al (2011) Near-Infrared Luminescence of Cadmium Pigments: In Situ Identification and Mapping in Paintings. Appl Spectrosc 65:939–951. https://doi.org/10.1366/11-06230
2. Monico L, Cartechini L, Rosi F, et al (2020) Probing the chemistry of CdS paints in The Scream by in situ noninvasive spectroscopies and synchrotron radiation x-ray techniques. Sci Adv 6:. https://doi.org/10.1126/sciadv.aay3514
3. Comelli D, MacLennan D, Ghirardello M, et al (2019) Degradation of Cadmium Yellow Paint: New Evidence from Photoluminescence Studies of Trap States in Picasso's Femme (Époque des "Demoiselles d'Avignon"). Anal Chem 91:3421–3428. https://doi.org/10.1021/acs.analchem.8b04914