The combination of FTIR and X-ray microscopes for the analysis of artistic materials at the ID21 beamline, European Synchrotron Radiation Facility

Emeline Pouyet[1], Marine Cotte [1,2], Murielle Salome [1], David Bugnazet [1], Barbara Fayard [3], Marie Radepont [2,4], Francesco Sette [1]

1. European Synchrotron Radiation Facility, 6, rue Jules Horowitz, F-38000 Grenoble, France
2. LAMS (Laboratoire d'Archéologie Moléculaire et Structurale) UMR-8220, 3 rue Galilée 94200 Ivry-sur-Seine, France
3. Laboratoire de Physique des Solides, UMR 8602 CNRS, Paris, France
4. University of Antwerp, Department of Chemistry, Universiteitsplein, 1, B2610 Antwerp, Belgium

email.pouyet@esrf.fr
Keyword: Synchrotron, Painting, Chemical mapping, Micro-spectroscopy

The complexity and heterogeneity of artistic materials make their chemical characterization uneasy. Micro-imaging techniques are commonly applied to attain full 2D descriptions of fragments cross-sections.

At the European Synchrotron Radiation Facility (ESRF), Grenoble, France, the ID21 beamline is primarily designed for scanning X-ray microscopy. Since 2004, it also hosts a Fourier Transform Infrared (FTIR) end-station. The combined access (fig. 1) on a single beamline to both X-ray and FTIR micro spectroscopies has become a main asset in the characterization of very complex heterogeneous samples at the micron scale in several research fields, such as Cultural Heritage Sciences with a particular focus on paintings.

At the ID21 beamline, the X-ray microscope is devoted to micro X-ray fluorescence and micro X-ray Absorption Near-Edge Spectroscopy (XANES) in the tender X-ray domain (2-9 keV). Therefore, it is very well suited to reveal the chemical state of pigments with a very high lateral resolution (~0.3×0.7µm^2) and very good detection limits (ppm). Moreover, the FTIR spectro-microscope, composed of a Thermo Nicolet Nexus infrared bench associated with an infrared Thermo Continuum microscope, gives essential molecular information, in particular regarding the organic binders (chemical characterization, 2D mapping).

Other classical microscopic techniques are also available such as Environmental Scanning Electron Microscopy and optical and UV microscopes.

The current increase of experiments related to painting analysis at ID21 is certainly due to these multi-modal and non invasive 2D chemical mapping capabilities.

The user community is pushing towards an improved and easier combination of X-ray and infrared microscopies [1]. In this context, we are making efforts in these two directions:

i) the improvement of painting sample preparation for transmission FTIR and XANES micro analyses. The most common preparations [2] will be discussed and new strategies will be presented.

ii) the development of new micro-spectroscopy tools improving and diversifying the capabilities of ID21.

In particular, the new XANES full-field imaging station (fig. 2) developed and installed on the beamline will be introduced [3] and illustrated thanks to the first tests realized on painting samples. Thanks to the simultaneous acquisition of up to 4.106 XANES spectra over large sample areas at the sub-micron scale, this technique is very well suited in the study of archeological material, and in particular of paintings. By comparison to the scanning techniques currently available at ID21, the full-field approach presents the major advantage of decreasing the acquisition time, reducing consequently possible effects of radiation damage and improving statistical data significance.

References

[1] M. Cotte *et al*., Applications of synchrotron-based micro-imaging techniques to the chemical analysis of ancient paintings, Journal of Analytical Atomic Spectrometry, **23** (2008), p. 820-828
[2] M. Spring, Evaluation report on protocols for representative sampling, D2.1-1 – ver.0.4, CHARISMA, (2010), p. 1-39
[3] V. De Andrade *et al.,* Submicrometer hyperspectral X-ray imaging of heterogeneous rocks and geomaterials: applications at the Fe k-edge., Analytical Chemistry, (2011), p. 4220-4227

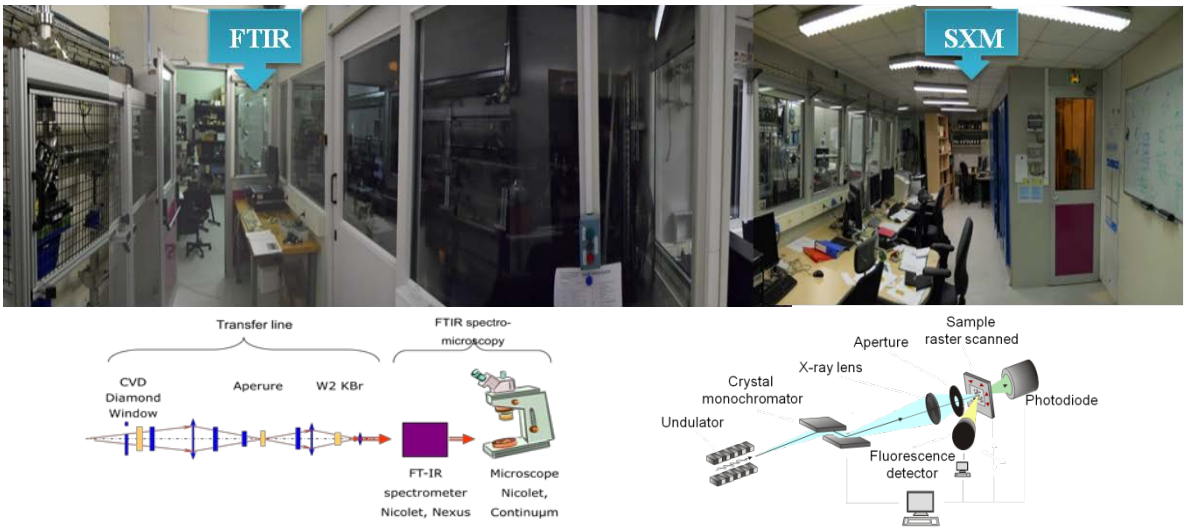

Figure 1. *The ID21 X-ray microscopy beamline at the ESRF houses two microscopes: a Scanning X-ray Microscope (SXM) and an infrared microscope (SR-FTIR microscope)*

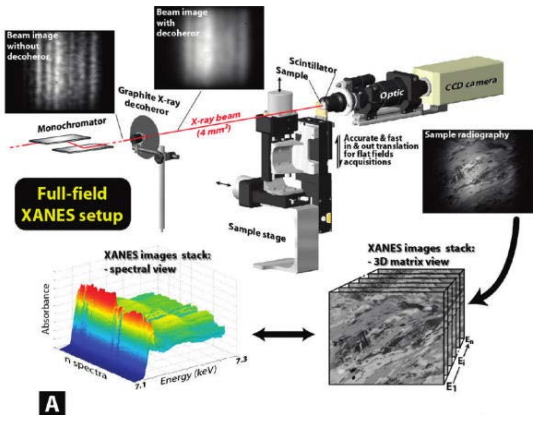

Figure 2. *Full Field set up available at the ID21 beamline (V. De Andrade et al., Submicrometer hyperspectral X-ray imaging of heterogeneous rocks and geomaterials: applications at the Fe k-edge., Analytical Chemistry, 4221, 2011)*