Elettra Sincrotrone Trieste

IAEA
International Atomic Energy Agency

Examples of application of XAS and XRF techniques to real case studies

By Simone Pollastri: simone.pollastri@unimore.it

ISTITUTO OFFICINA DEI MATERIALI

UNIVERSITÀ DEGLI STUDI
DI MODENA E REGGIO EMILIA

BACKGROUND

Field work

Geology/Mineralogy

- **29/06/2012: Master's degree in Geological Sciences at the UniMoRe.**

Thesis title: "Crystal chemistry of Italian asbestos cement and characterization of their thermal transformation product".

- **1/2013 - 4/2016: Ph.D. school "M2SCS" at the UniMoRe.**

Main activities: Characterization of mineral fibres aimed at understanding their toxicity potential. Didactic and disseminating activity; mentoring of graduating students.

- **4/2016 - 6/2017: Post-doc at the Geology Department of UniMoRe.**

Main activities: Characterization of ACM from Netherlands in view of their recycling through thermal treatment. Disseminating activity; mentoring of graduating students.

- **7/2017 - 6/2020: CERIC post-doc position at the XAFS beamline of Elettra.**

Main activities: Contribution to the design and development of a fuel cell for operando XAS and SAXS experiments for the study of innovative Pt/ceria catalyst systems.

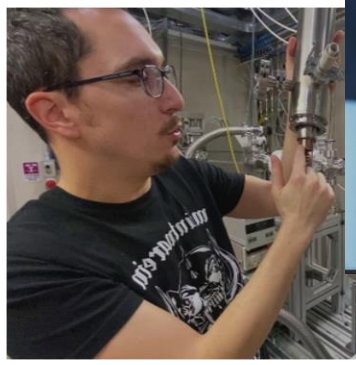
- **7/2020 - 2/2023: Post-Doc Researcher at the XRF beamline.**

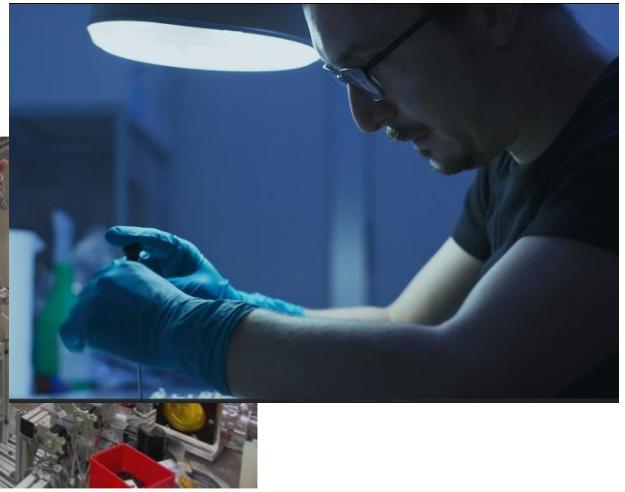
Main activities: LC of scheduled exps., involvement of new research groups in order to start new projects/collaborations, didactic and disseminating activity.

- **Current position: RTDa, Physics dep. UniMoRe in collaboration with CNR and Elettra**

Main activities: Design and development of an electrochemical cell for operando XAS experiments for the study of innovative MOFs chiral catalysts. Didactic activity.

Lab work





Catalysis/Environmental sciences

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ESRF
The European Synchrotron

LISA
Linea Italiana di Spettroscopia di Assorbimento x

The chemical environment of iron in mineral fibers. A combined X-ray absorption and Mössbauer spectroscopic study

Simone Pollastri[a] and Alessandro F. Gualtieri[a]

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diamond

Environmental pollution monitoring

Mineral fibres: An overview

The general term “mineral fibres” refers to a group of minerals ubiquitous on the Earth crust. Among them, the most relevant and certainly the most feared ones are asbestos minerals and fibrous zeolites such as erionite (Mossman *et al.* 1990; Baumann *et al.* 2013)

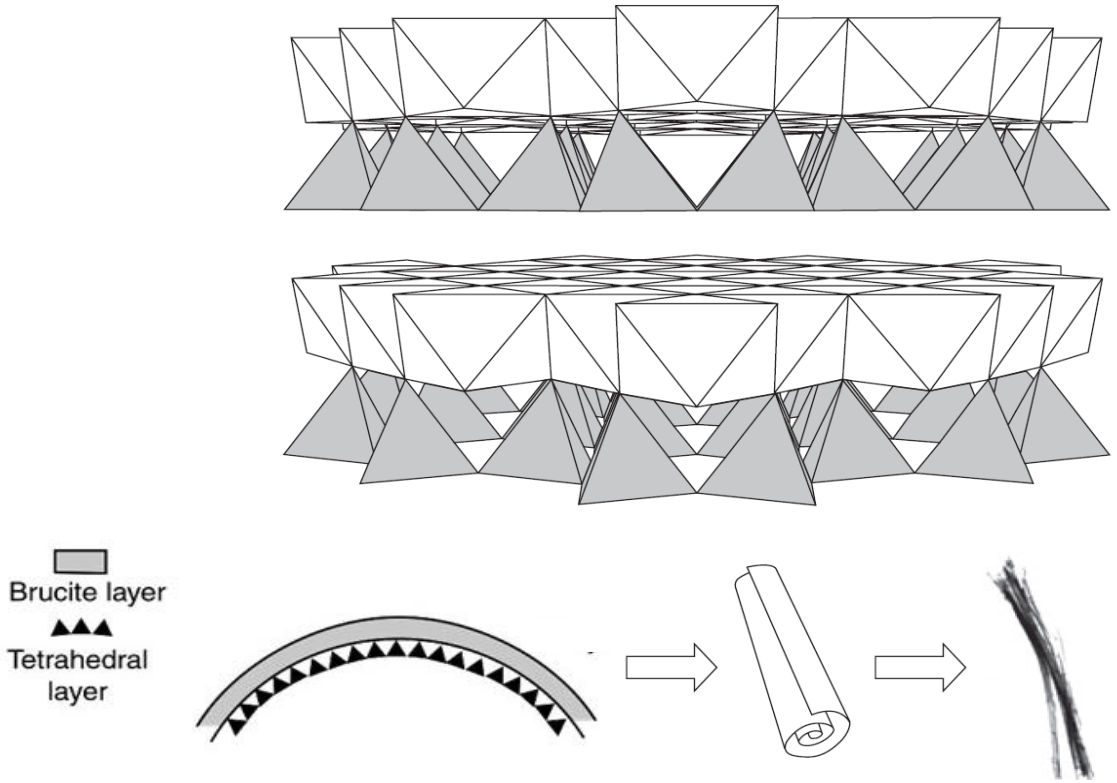
Asbestos minerals are further subdivided into two major groups:

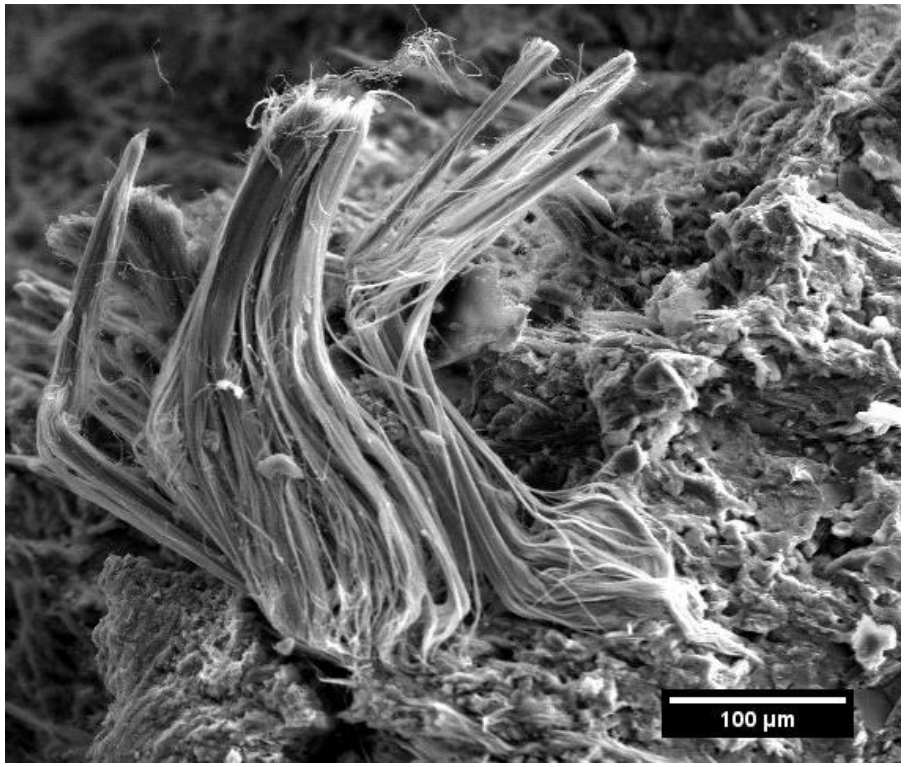
Serpentine asbestos, whose fibrous-asbestiform variety is called chrysotile.

Amphibole asbestos, whose fibrous-asbestiform varieties are called amosite, actinolite, anthophyllite, crocidolite and tremolite.

Chrysotile asbestos

Chrysotile is a layer silicate composed of Si-centred tetrahedral (T) sheets in a pseudo-hexagonal network joined to Mg-centred octahedral (O) sheets in units with a 1:1 (T:O) ratio. The general ideal chemical formula of chrysotile is $Mg_3Si_2O_5(OH)_4$.



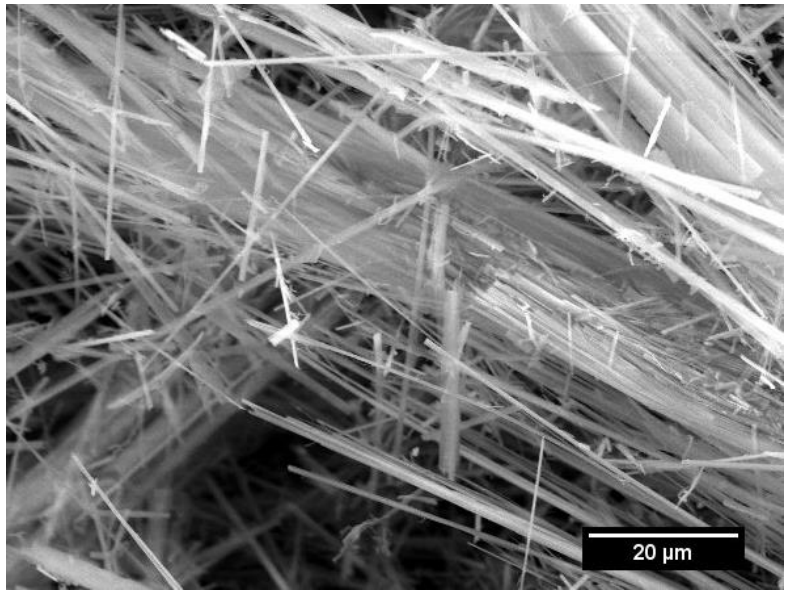


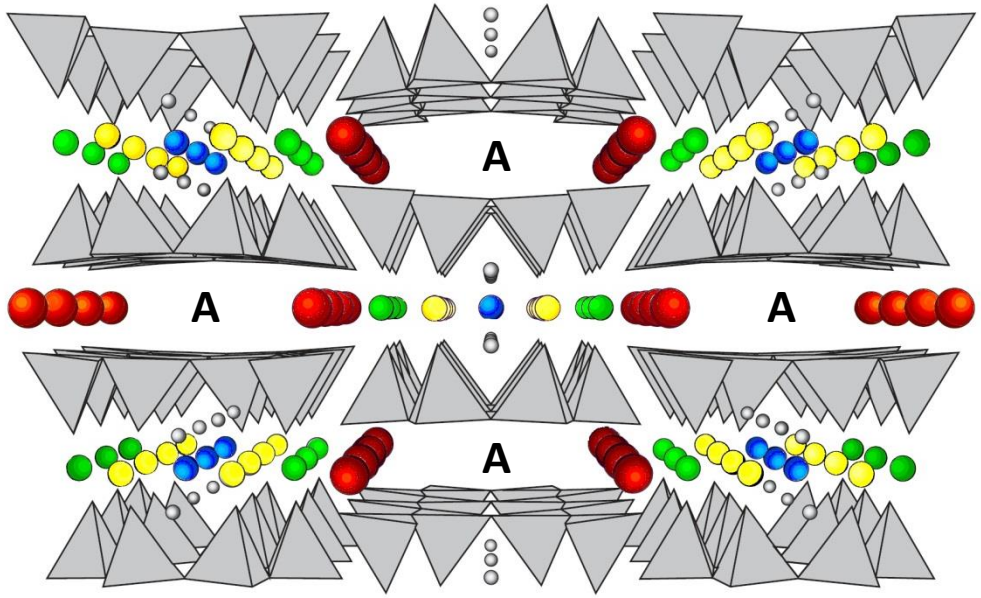
As a result of the polarity of the TO unit and the misfit between the T and O sheet, a differential strain occurs between the two sides of the layer. The strain is released by rolling the TO layer around the fibril axis.


5 µm

Amphibole asbestos

Amphiboles are double-chain silicates with a Si(Al):O ratio of 4:11 and the oxygen atoms of the chains coordinated not only to Si(Al) but to a variety of other cation sites, yielding the following simplified general formula (Veblen, 1981): $A_{0-1}B_2C_5T_8O_{22}(OH)_2$



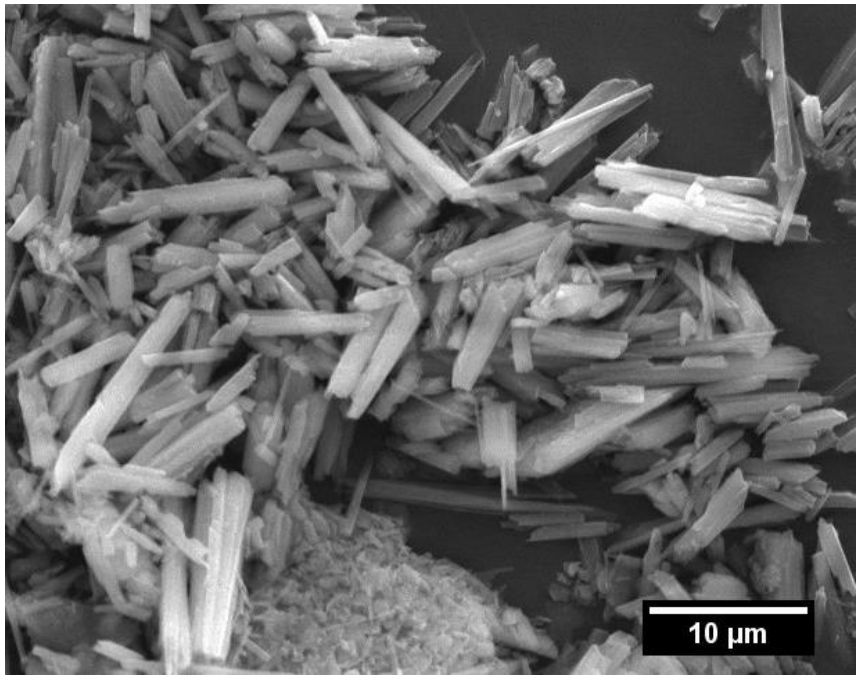


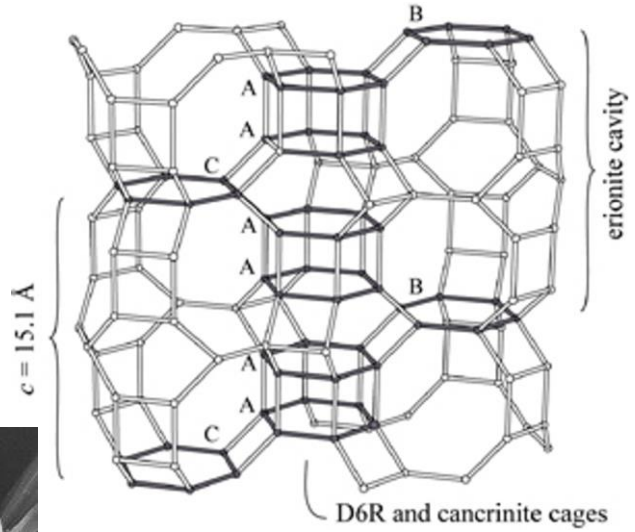
Main amphiboles fibres	Ideal chemical formula	Crystal System
fibrous actinolite (byssolite)	$Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$	Monoclinic
amosite (fibrous variety of grunerite)	$(Fe^{2+},Mg)_7Si_8O_{22}(OH)_2$	Monoclinic
fibrous anthophyllite	$(Mg,Fe^{2+})_7Si_8O_{22}(OH)_2$	Orthorombic
crocidolite (fibrous variety of riebeckite)	$Na_2(Fe^{2+},Mg)_3Fe_2^{3+}Si_8O_{22}(OH)_2$	Monoclinic
fibrous tremolite	$Ca_2Mg_5Si_8O_{22}(OH)_2$	Monoclinic

Fibous zeolite

Erionite is a tectosilicates, widespread natural zeolite-group mineral; its fibrous form has usually sedimentary origin and often occurs as altered product of volcanic tuffs (Virta, 2002; Ballirano *et al.* 2009). Its framework consists of columns of cancrinite cages connected along the *z* direction by double six-membered rings of tetrahedra, forming hexagonal prisms.

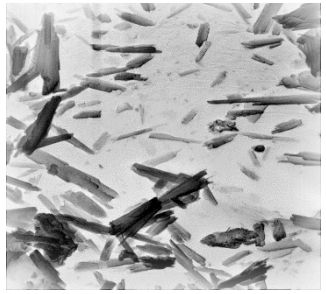
Homes in Karain, Turkey

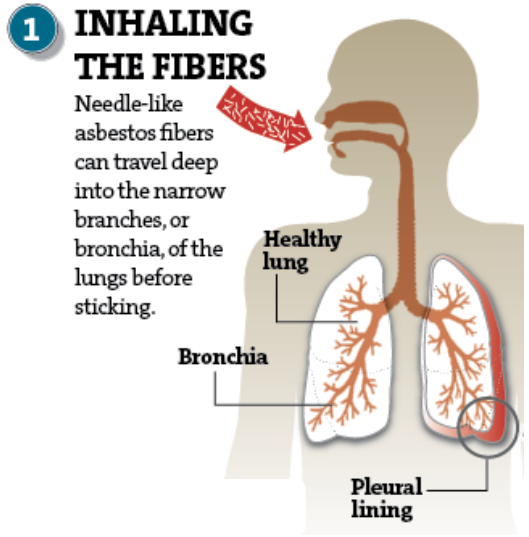




Why to study mineral fibers?

In the last two decades, these mineral fibres have been the subject of intensive multidisciplinary investigations as the mechanisms by which they induce cyto- and geno-toxic damage remain poorly understood. In general, the cause-effect relationship between exposure to the fibres and the onset of mesothelioma and other lung diseases remains ambiguous.



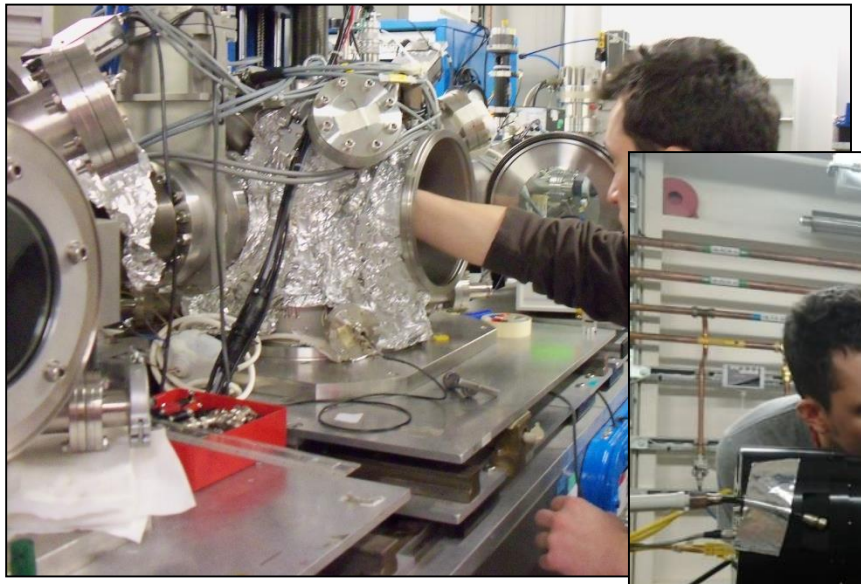


Questions:

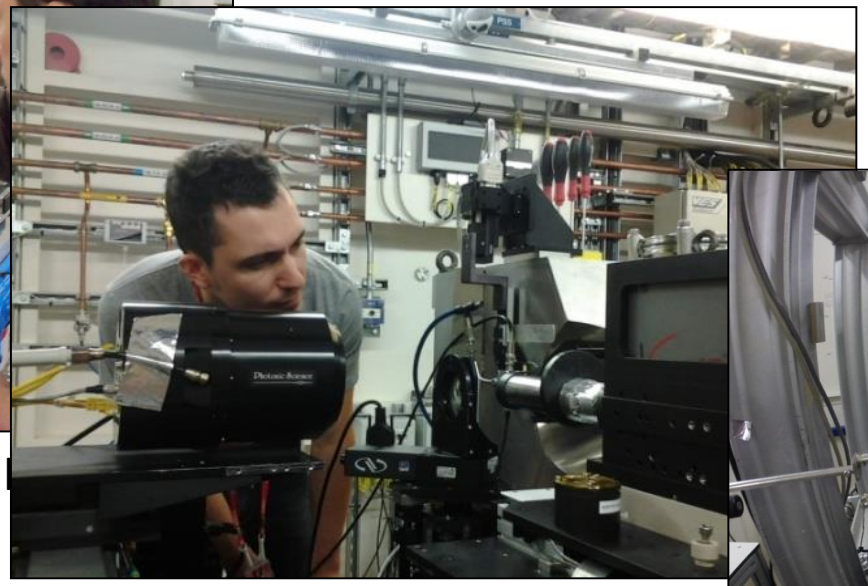
Which is the Fe chemical environment among different fibers?
If different, is it playing a role in the toxicity?

Methods:

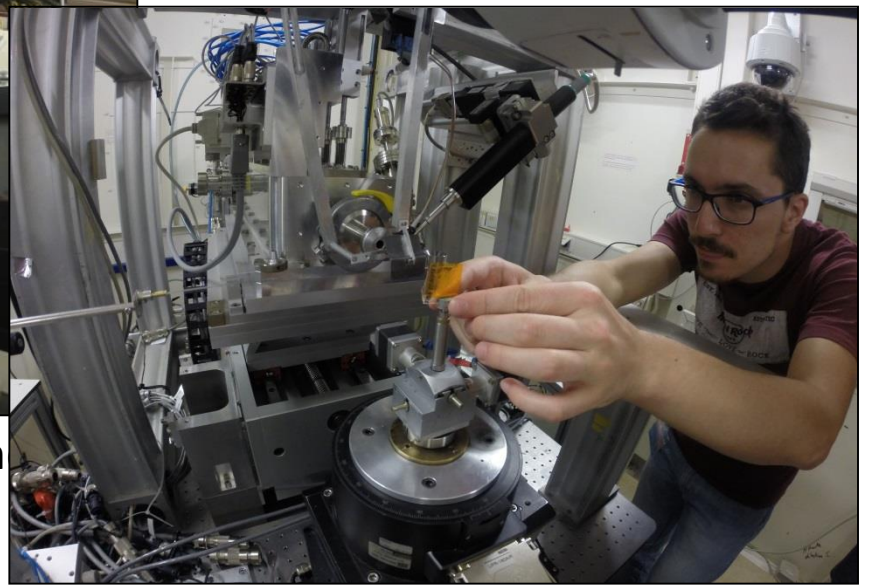
Such samples have been investigated using conventional and non-conventional sources, and many experiments were conducted at synchrotron radiation facilities:



ESRF – BM08 (LISA) for XAS anal



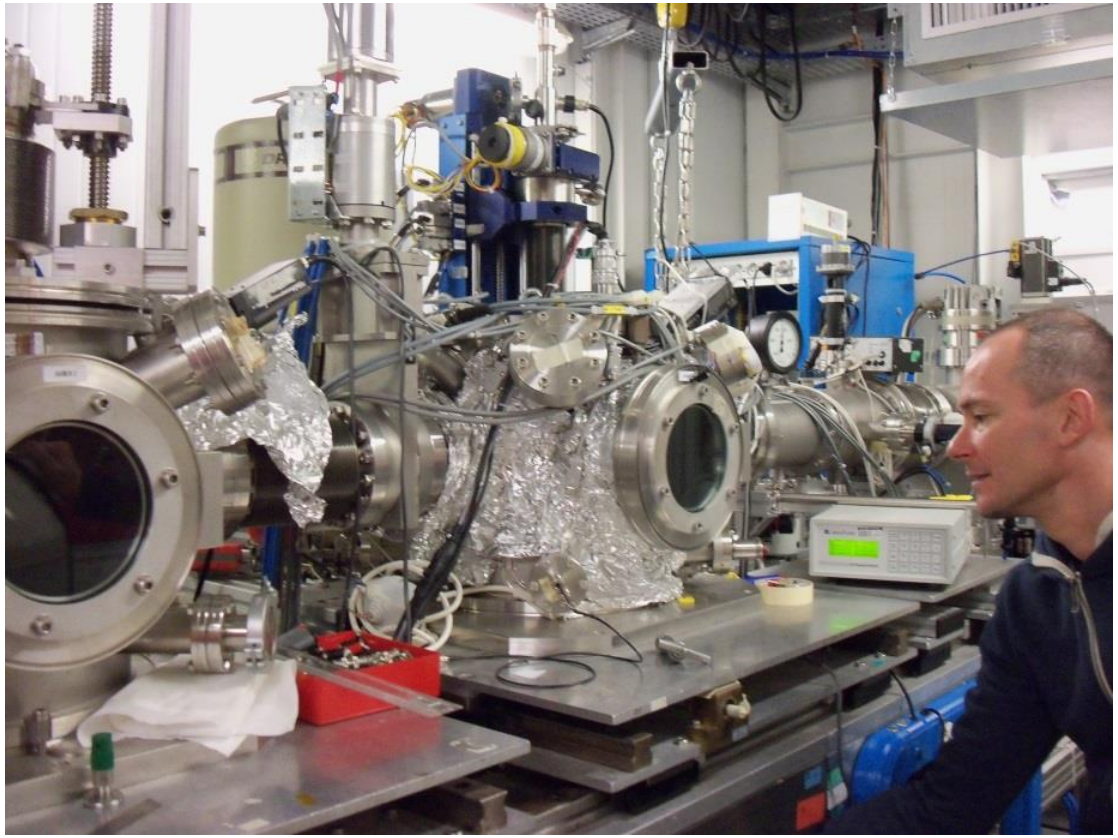
DLS – I18 For *in situ* XANES, XRD an iron mapping.



ESRF – ID13 for XRD analysis.

Experimental setup:

Fe K-edge XAS spectra were collected on **all raw samples** at the LISA-CRG beamline (ESRF, Grenoble, France) using a dynamically and sagittally focusing Si (311) double XX monochromator.



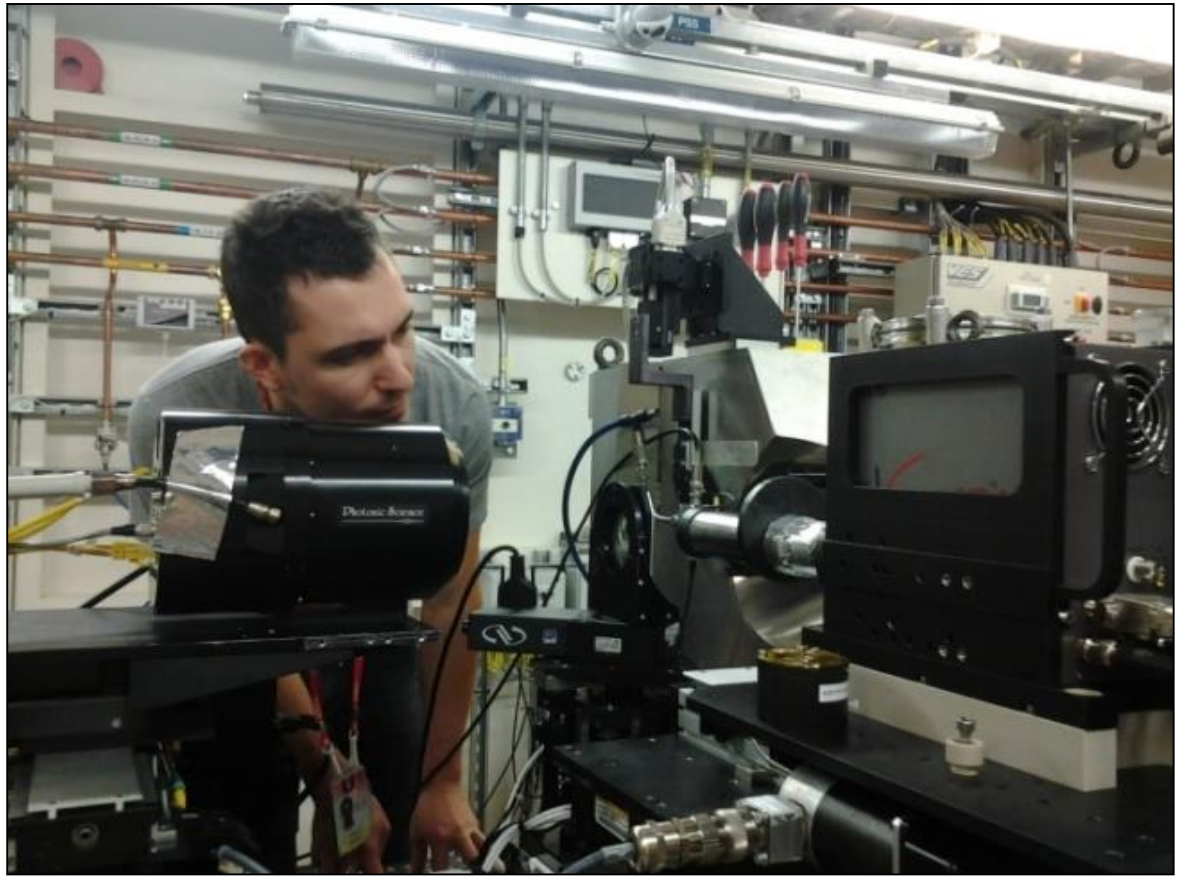
ESRF
The European Synchrotron

LISA
Linea Italiana di Spettroscopia di Assorbimento x

All measurements were conducted at RT, both in transmission and in fluorescence mode. For all the experiments, energy calibration was achieved using iron foil as reference. XANES data reduction with **Origin 8.0** and analysis with **PeakFit 4.12**. EXAFS spectra analyzed with the **IFEFFIT suite** (v. 1.2.9: Newville, 2006).

Experimental setup:

Moreover, **3 selected representative samples** were put in contact with human cell cultures for different contact times. After exposure, samples were washed, filtered and sealed between two kapton sheets, for *in situ* µXANES investigations.



Experiments were conducted at the I18 beamline (DLS, Oxford, UK). The beamline uses a cryogenically cooled Si (111) monochromator; the beam size on the samples was 2x2 µm.

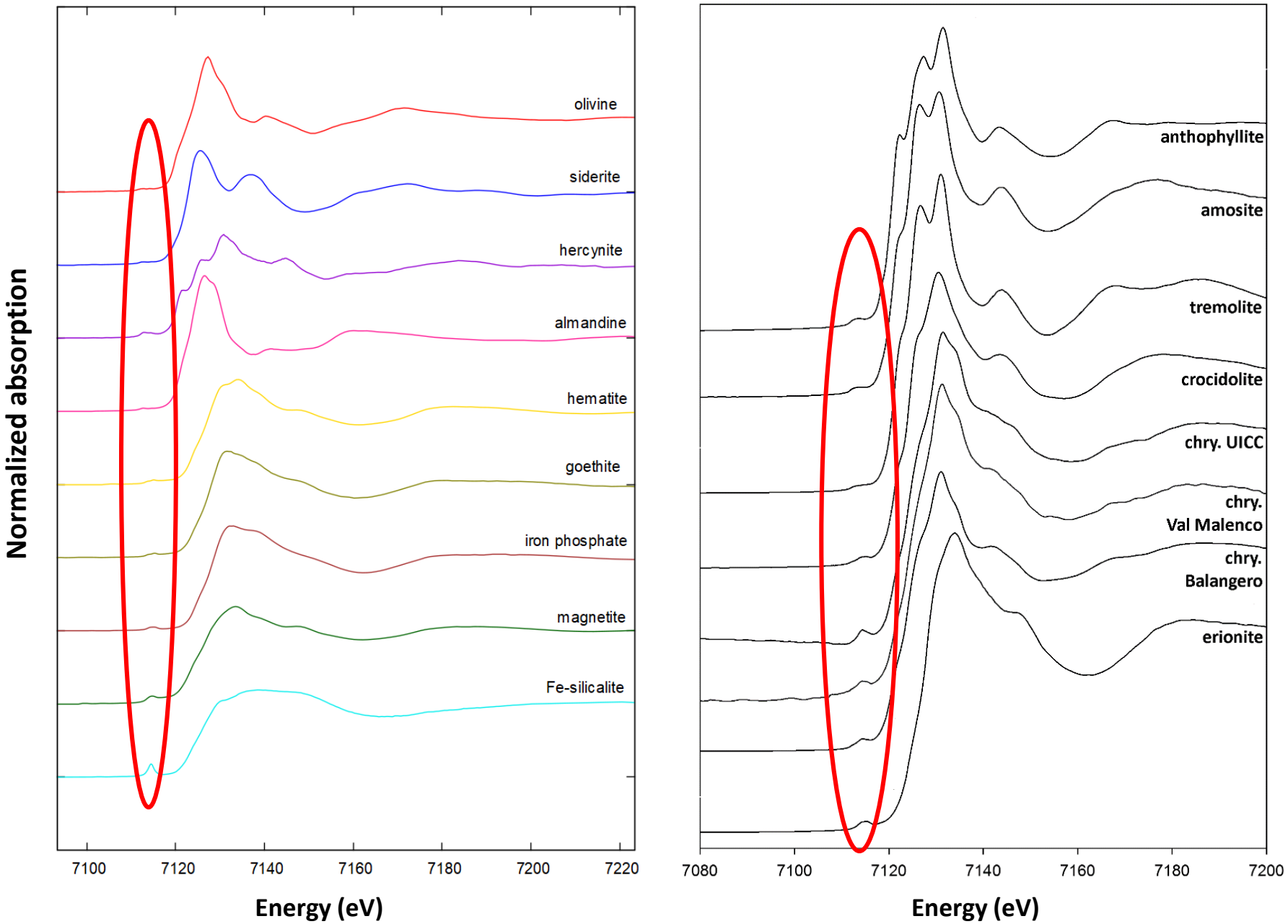
All measurements were conducted at RT, both in transmission and in fluorescence mode. For all the experiments, energy calibration was achieved using iron foil as reference. XANES data reduction with **Origin 8.0** and analysis with **PeakFit 4.12**. EXAFS spectra analyzed with the **IFEFFIT suite** (v. 1.2.9: Newville, 2006).

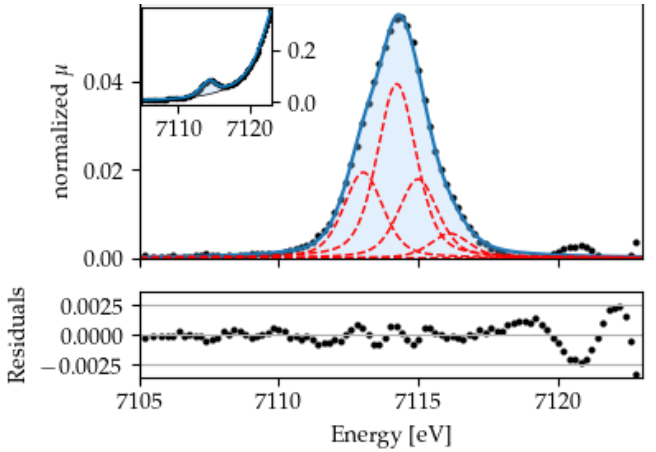
Investigated samples:

Sample	Calculated chemical formula (from TG/DTA and EMPA data)
chrysotile UICC	$(Mg_{5.93}Fe^{2+}_{0.11}Al_{0.02}Fe^{3+}_{0.01})_{6.07}Si_{4.03}O_{10}(OH)_{7.66}$
chrysotile Balangero	$(Mg_{5.81}Fe^{2+}_{0.21}Al_{0.27}Fe^{3+}_{0.03}Cr_{0.01})_{6.33}Si_{3.97}O_{10}(OH)_{7.11}$
chrysotile Val Malenco	$(Mg_{5.85}Fe^{2+}_{0.11}Al_{0.02}Ni_{0.01})_{5.99}Si_{4.01}O_{10}(OH)_{7.86}$
amosite	$(Ca_{0.02}Na_{0.01})(Fe^{2+}_{5.36}Mg_{1.48}Fe^{3+}_{0.11}Mn_{0.06})_{7.01}(Si_{7.93}Al_{0.01})_{7.94}O_{21.94}(OH)_{2.06}$
anthophyllite	$Ca_{0.04}(Mg_{5.81}Fe^{2+}_{0.92}Fe^{3+}_{0.21}Mn_{0.04})_{6.98}(Si_{7.83}Al_{0.02})_{7.85}O_{21.63}(OH)_{2.37}$
crocidolite	$(Na_{1.96}Ca_{0.03}K_{0.01})_2(Fe^{2+}_{2.34}Fe^{3+}_{2.05}Mg_{0.52})_{4.91}(Si_{7.84}Al_{0.02})_{7.86}O_{21.36}(OH)_{2.64}$
tremolite	$(Ca_{1.91}Na_{0.06}K_{0.01})_{1.98}(Mg_{4.71}Fe^{2+}_{0.22}Fe^{3+}_{0.08}Mn_{0.02})_{5.03}(Si_{8.01}Al_{0.02})_{8.03}O_{22.14}(OH)_{1.86}$
erionite	$(Na_{5.31}K_{2.18}Ca_{0.15}Mg_{0.11}Fe^{3+}_{0.29})_{8.04}(Si_{27.84}Al_{7.85})_{35.69}O_{72}\cdot 20.3H_2O$

Ex-situ results: XANES

Normalized Fe K-edge spectra of samples (right) and reference compound (left).



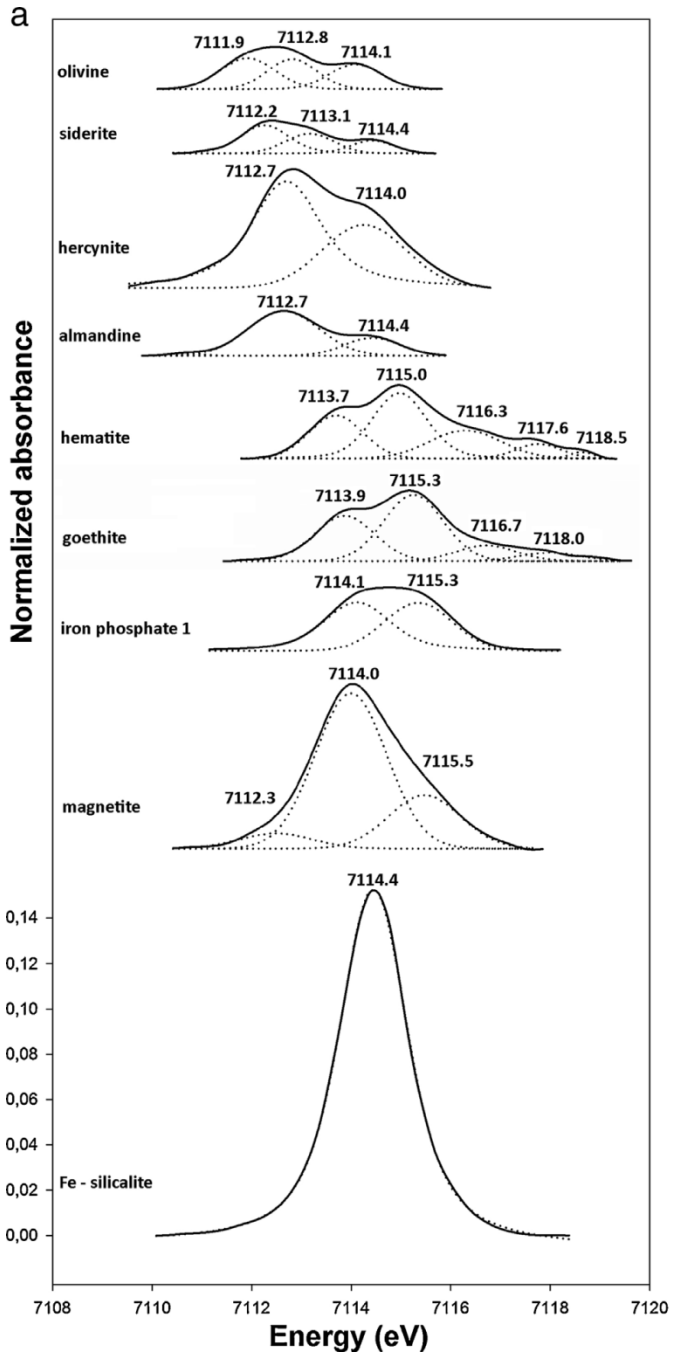


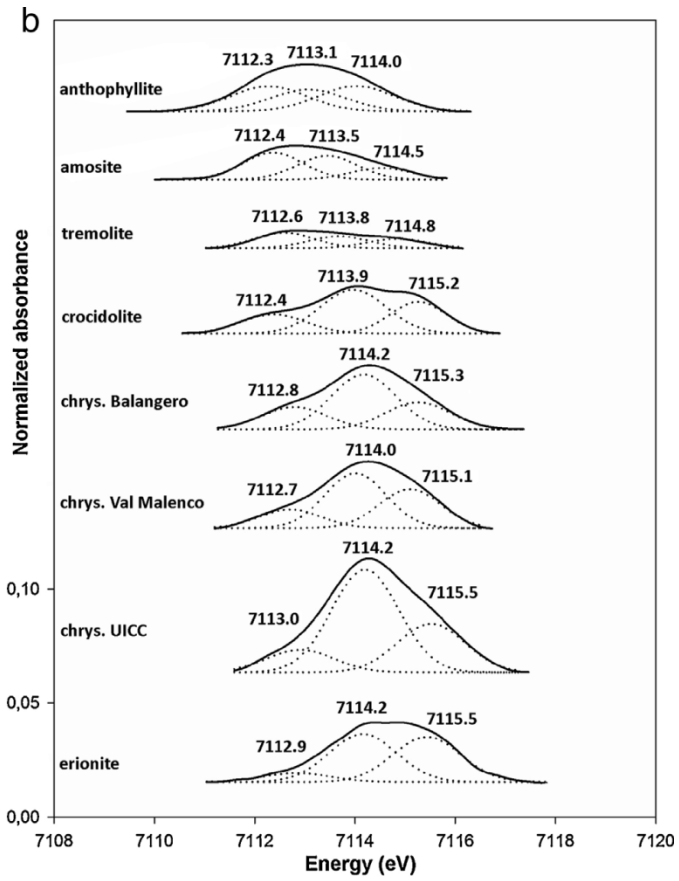
Pre–edge structures are influenced by electronic transitions and hence by local geometry of the photoabsorber.

Ex-situ results: XANES

XANES pre-edge parameters of investigated samples.

Sample	Component position (eV)	Component area	Total area	r^2	Centroid (eV)
Anthophyllite	7112,44	0.026	0.054 (5)	0.9992	7113.12 (3)
	7113,77	0.028			
Amosite	7112,57	0,025	0.045 (5)	0.9992	7113.23 (5)
	7113,92	0,018			
	7115,50	0,002			
Tremolite	7112,44	0.008	0.032 (8)	0.9993	7113.56 (9)
	7113,48	0.016			
	7114,87	0.008			
Crocidolite	7112,38	0.011	0.055 (7)	0.9997	7114,05 (5)
	7113,96	0.026			
	7115,26	0.017			
Chrysotile	7112,69	0.016	0.074 (7)	0.9993	7114,04 (9)
Balangero	7114,32	0.055			
	7115,61	0.004			
Chrysotile	7112,50	0,009	0.072 (5)	0.9993	7114,13 (5)
Val Malenco	7114,21	0,055			
	7115,46	0,008			
Chrysotile	7112,75	0,011	0.129 (8)	0.9998	7114.42 (5)
UICC	7114,17	0,081			
	7115,52	0,037			
Erionite	7112,35	0,001	0.076 (2)	0.9991	7114.65 (3)
	7114,31	0,051			
	7115,51	0,024			

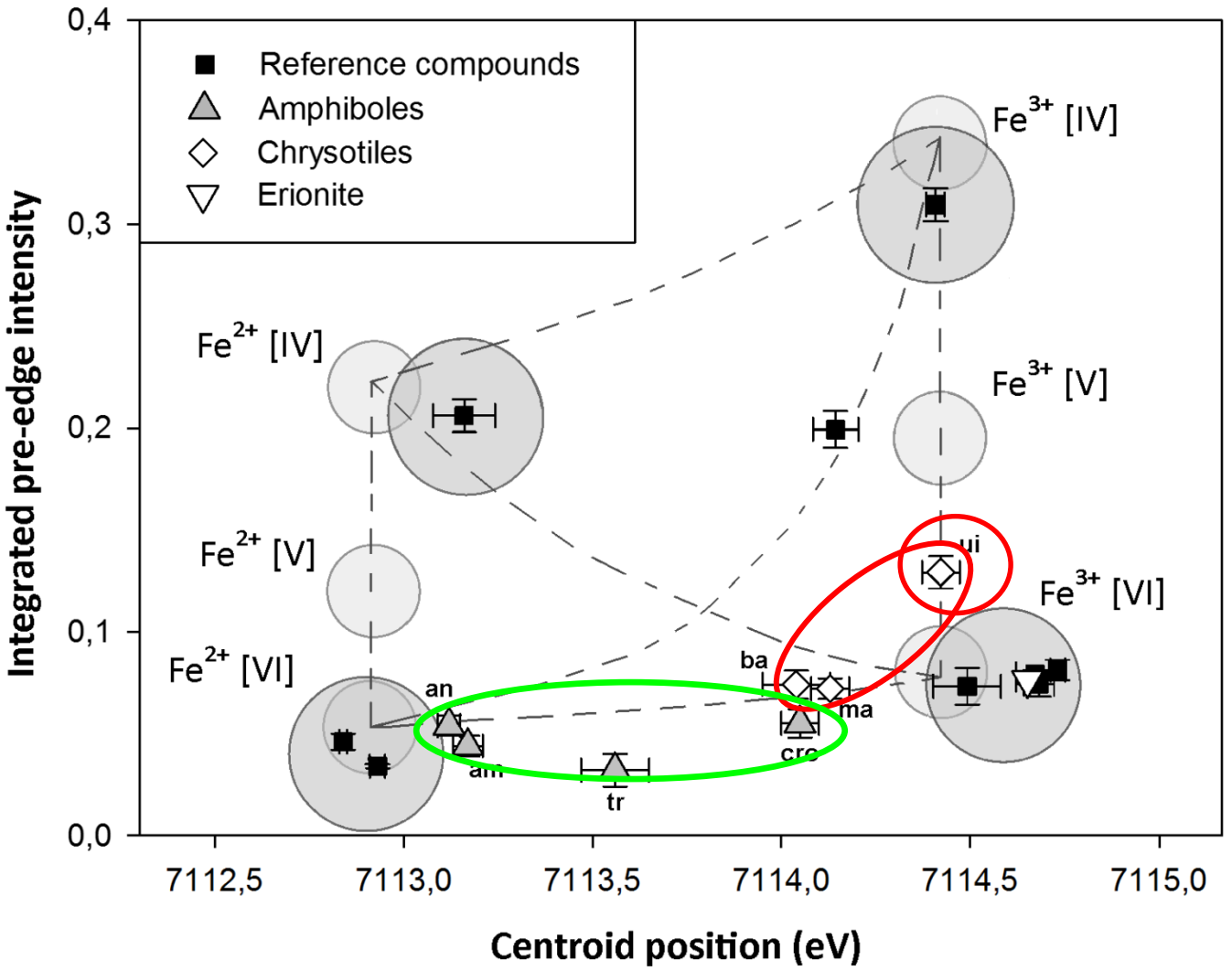




Result of the detailed study of the pre-edge peaks of reference compounds (a) and samples (b).

Ex-situ results: XANES

Final output of the obtained XANES pre-edge parameters of raw samples.



Chrysotile samples contain more Fe^{3+} with respect to amphiboles.

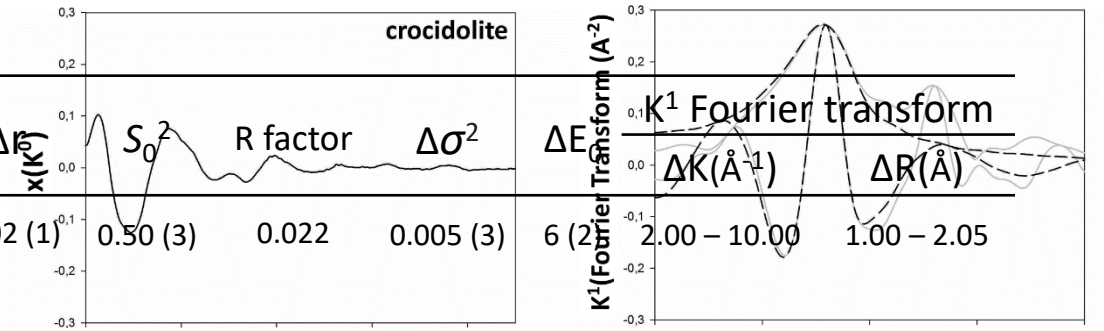
UICC chrysotile sample contaminated by magnetite.

Ex-situ results: EXAFS

Crystallographic data, forward/inverse Fourier transform ranges and structural parameters as obtained from the *R*-space fit by using the theoretical references.

Representative experimental data $\chi(K^0)$, Fourier transformed experimental (gray solid line) and fitted (black medium dash) data of investigated samples, both supplied with the [illegible] part

Shells	%	N.	R(Å)	Δr	S_0^2	R factor	$\Delta\sigma^2$	ΔE
anthophyllite				-0.02 (1)	0.50 (3)	0.022	0.005 (3)	6 (2)
Fe-O$_1$		2	1.93					
Fe-O$_2$		2	2.037					



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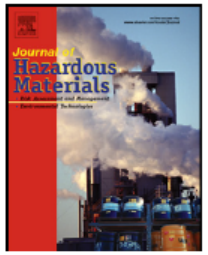


The chemical environment of iron in mineral fibres. A combined X-ray absorption and Mössbauer spectroscopic study

Simone Pollastri[a,*], Francesco D'Acapito[b], Angela Trapananti[b], Ivan Colantoni[c], Giovanni B. Andreozzi[d], Alessandro F. Gualtieri[a]

In-situ experiment: selected representative samples

Chemical composition and physical parameters

Results (from Pollastri *et al.* 2015) of the Fe K-edge XAS experiment conducted at the LISA (ex GILDA) beamline at ESRF: a) Fit of the XANES pre-edge peaks; b) Results of the fit of the EXAFS spectra.

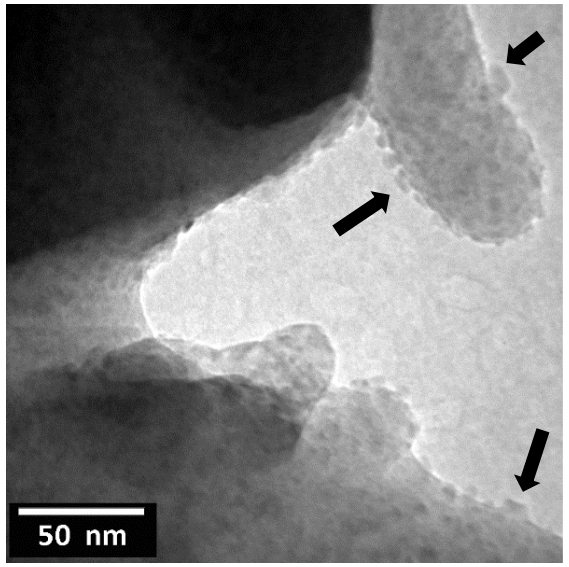
Sample	Calculated chemical formula and detected impurities (from TG/DTA, EMPA and XRD analysis).	Short fibers sample		Long fibers sample	
		Surface area (m^2/g)	Fiber length (µm)	Surface area (m^2/g)	Fiber length (µm)
chrysotile UICC	$(Mg_{5.93}Fe^{2+}_{0.04}Al_{0.02}Fe^{3+}_{0.08})_{6.07}Si_{4.03}O_{10}(OH)_{7.66}$ Impurities: brucite, calcite, clinochlore, dolomite, magnetite, microcline, pyroaurite and talc.	42(1)	5(2)	29(1)	99(5)
crocidolite UICC	$(Na_{1.96}Ca_{0.03}K_{0.01})_2(Fe^{2+}_{2.34}Fe^{3+}_{2.05}Mg_{0.52})_{4.91}(Si_{7.84}Al_{0.02})_{7.86}O_{21.36}(OH)_{2.64}$ Minor impurities: hematite, magnetite and quartz.	16.1(6)	6(1)	11.5(4)	30(3)
erionite	**1** **2** $(Na_{5.35}K_{2.19}Ca_{0.15}Mg_{0.11}Ti_{0.05})_{7.85}(Si_{28.01}Al_{7.90})_{35.91}O_{72}\cdot 28.13H_2O$ Impurities: clinoptilolite.	28(1)	9(1)	12.7(5)	16(1)

From the convergence of XANES, EXAFS and Mössbauer analysis

Where is the iron?!

In-situ experiment: selected representative samples

Chemical composition and physical parameters



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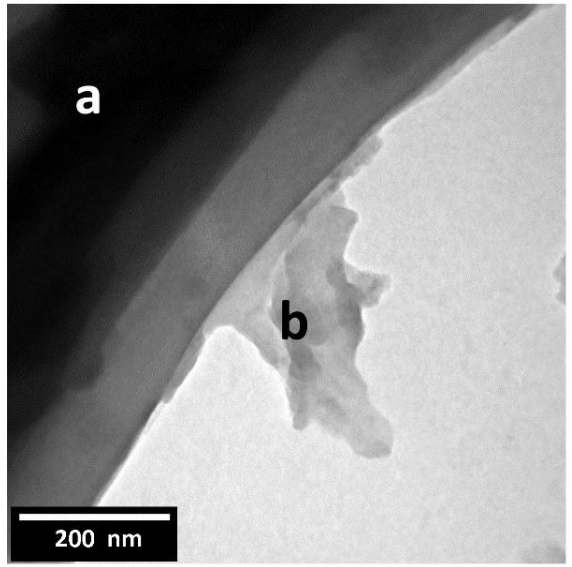

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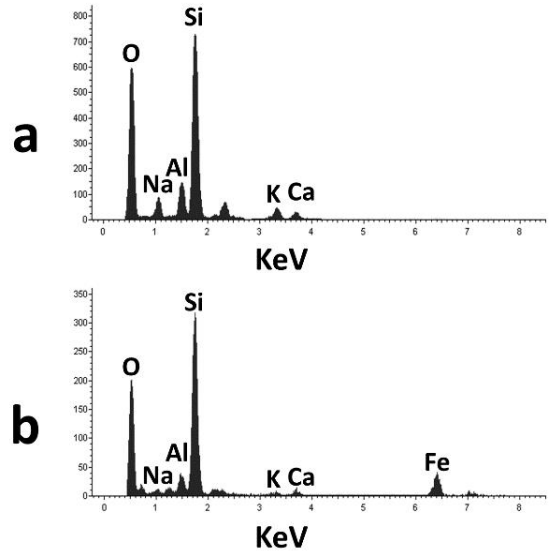
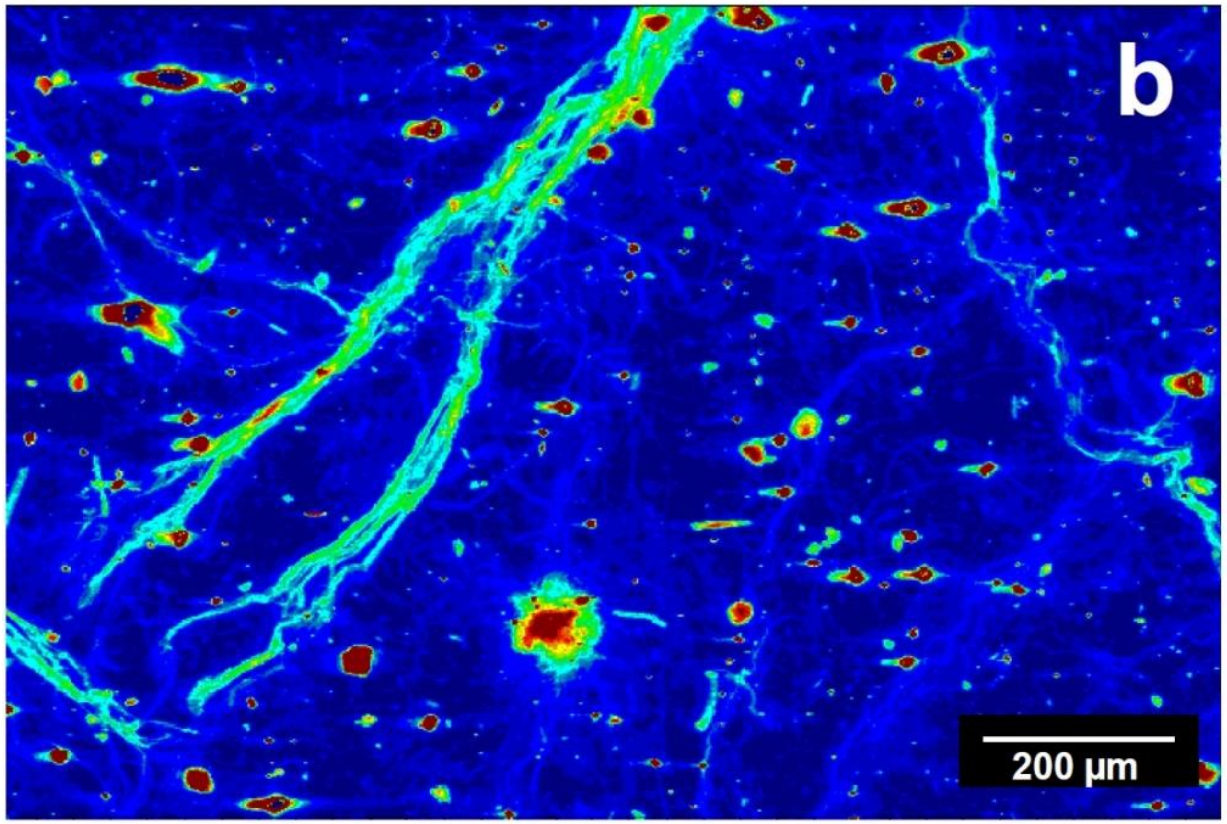


High-resolution TEM images of erionite sample: 1) fibre with spherical nanoparticles on the surface (indicated by arrows); 2) clustering of particles with EDS spectra.

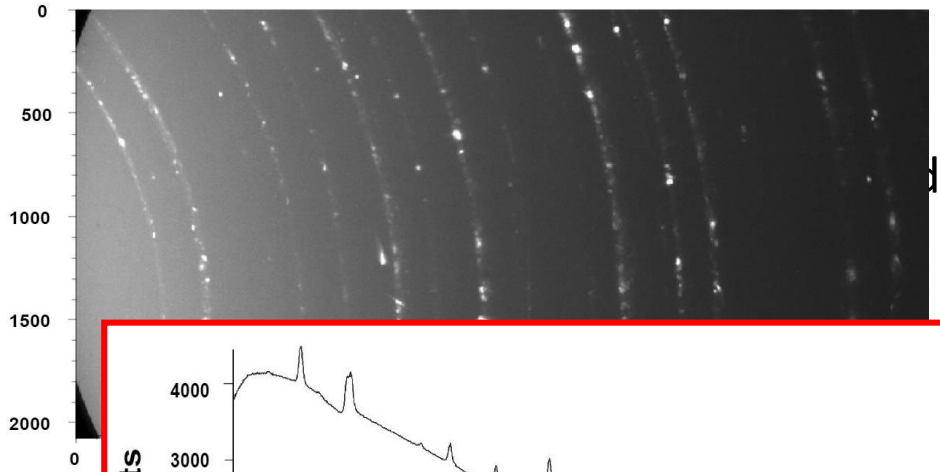
In-situ experiment: Experimental procedure

First, sample were observed in optical mycroscopy in order to identify suitable areas for µXRF iron mapping (based on the asbestos-like morphologies).

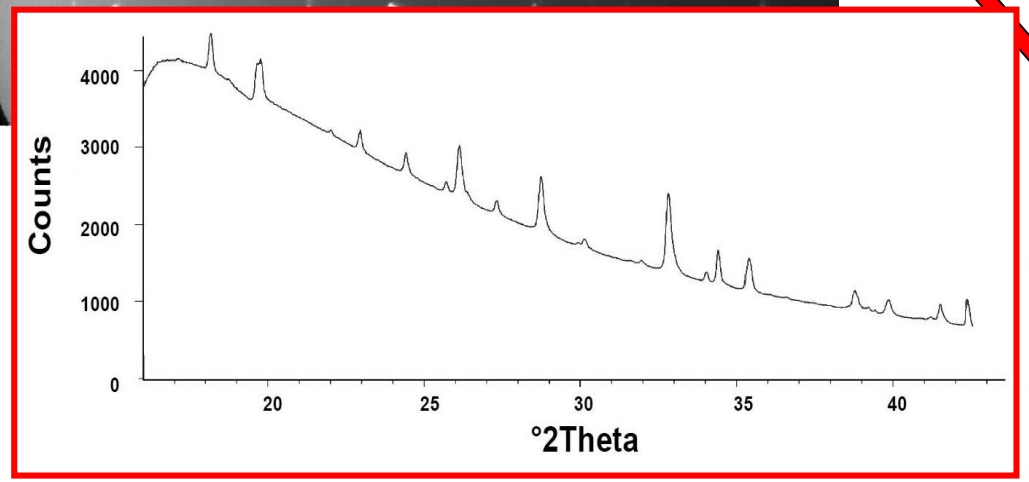


Visible light image of chrysotile fibres after 96h of incubation in MetHA cell culture. The corresponding Fe XRF map, showing the distribution of iron.

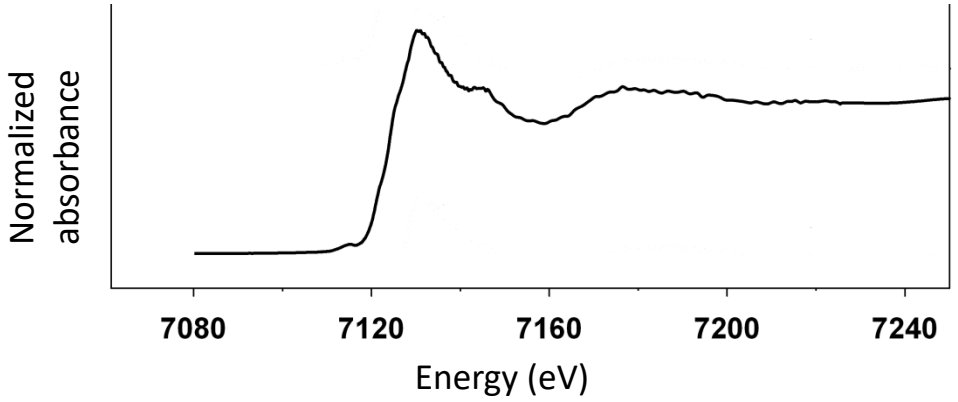
In-situ experiment: Experimental procedure

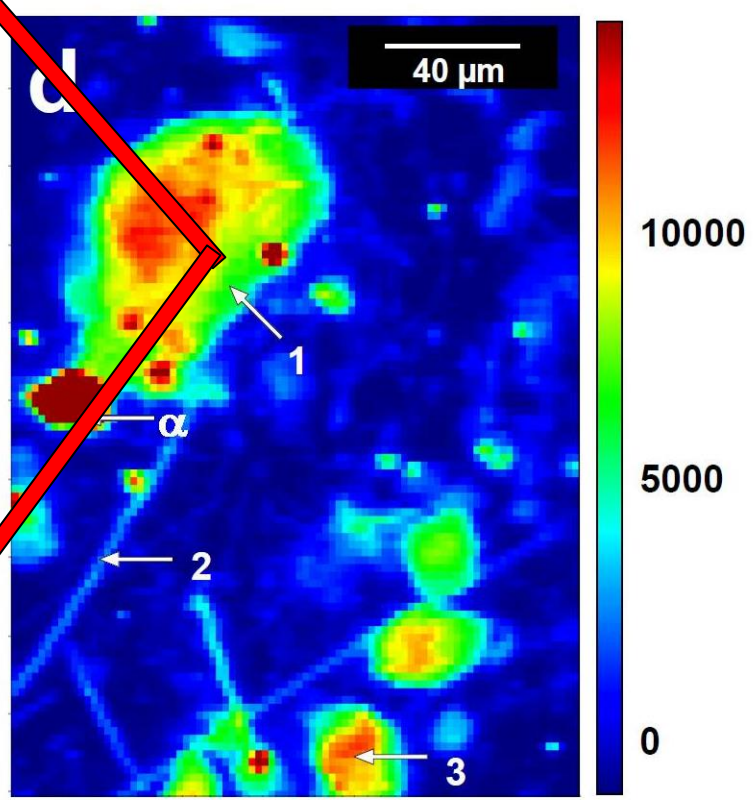


The same pattern after integration



of iron from the XRF maps (Fe K edge





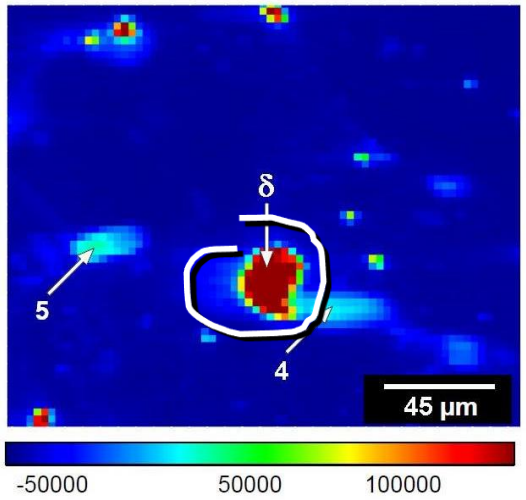
Crocidolite treated for 96h *ct* in Beas2b cell culture.

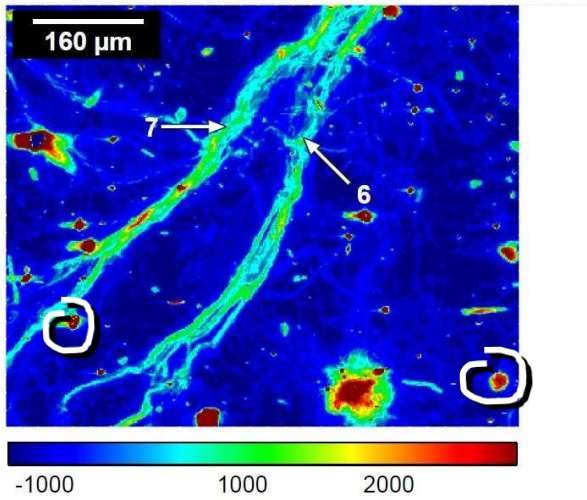
In-situ experiment: Results

About 20 XRF maps were collected; for each one, iron intensity were rescaled.
Every map posses arrows indicating the point at which XRD and XANES spectra were collected.

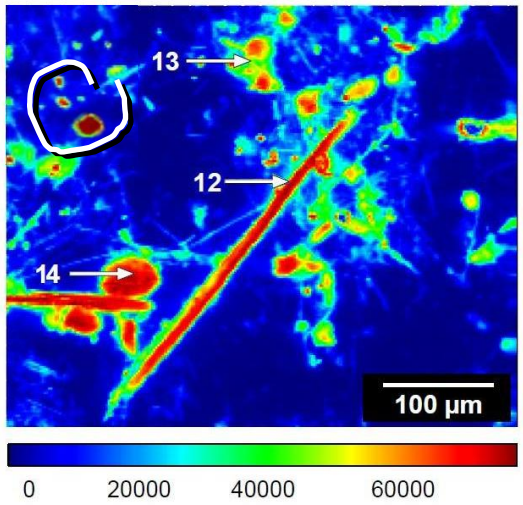
24h ct | **96h ct**

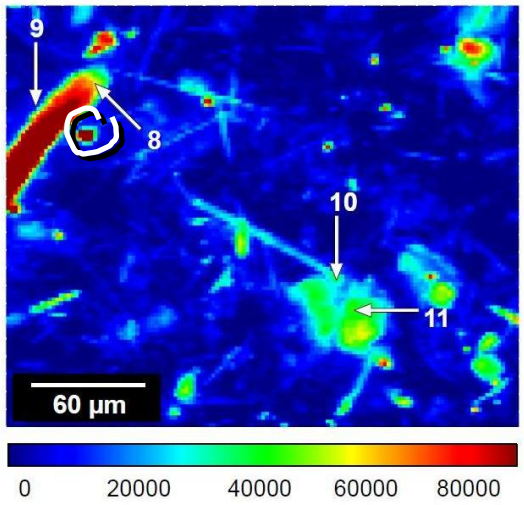
chrysotile





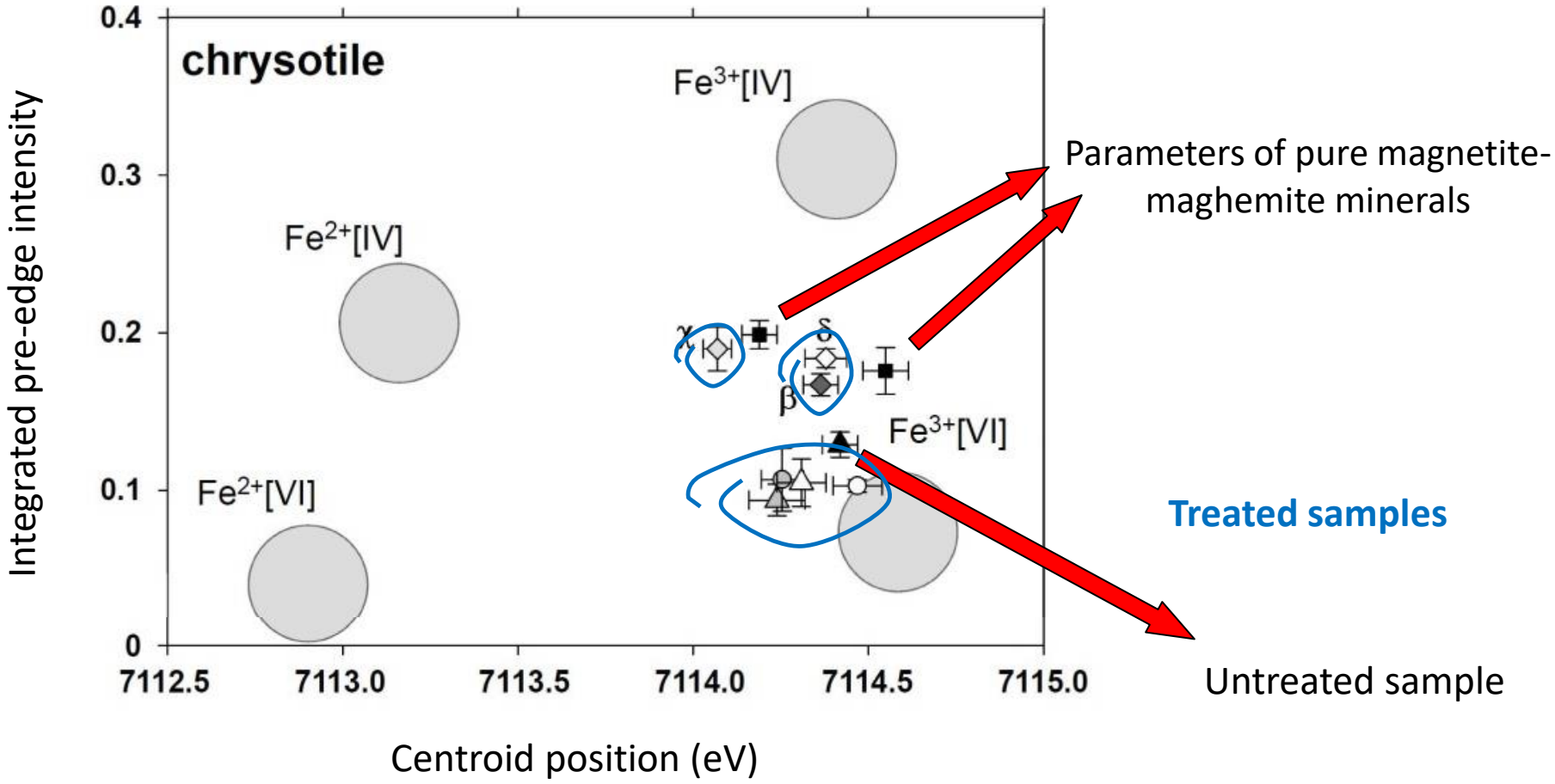
crocidolite





In-situ experiment: Results

For all collected XAS spectra, XANES pre-edge peaks analysis have been performed. Results obtained (total area and centroid position) were compared with those of untreated samples (from the previous XAS study).



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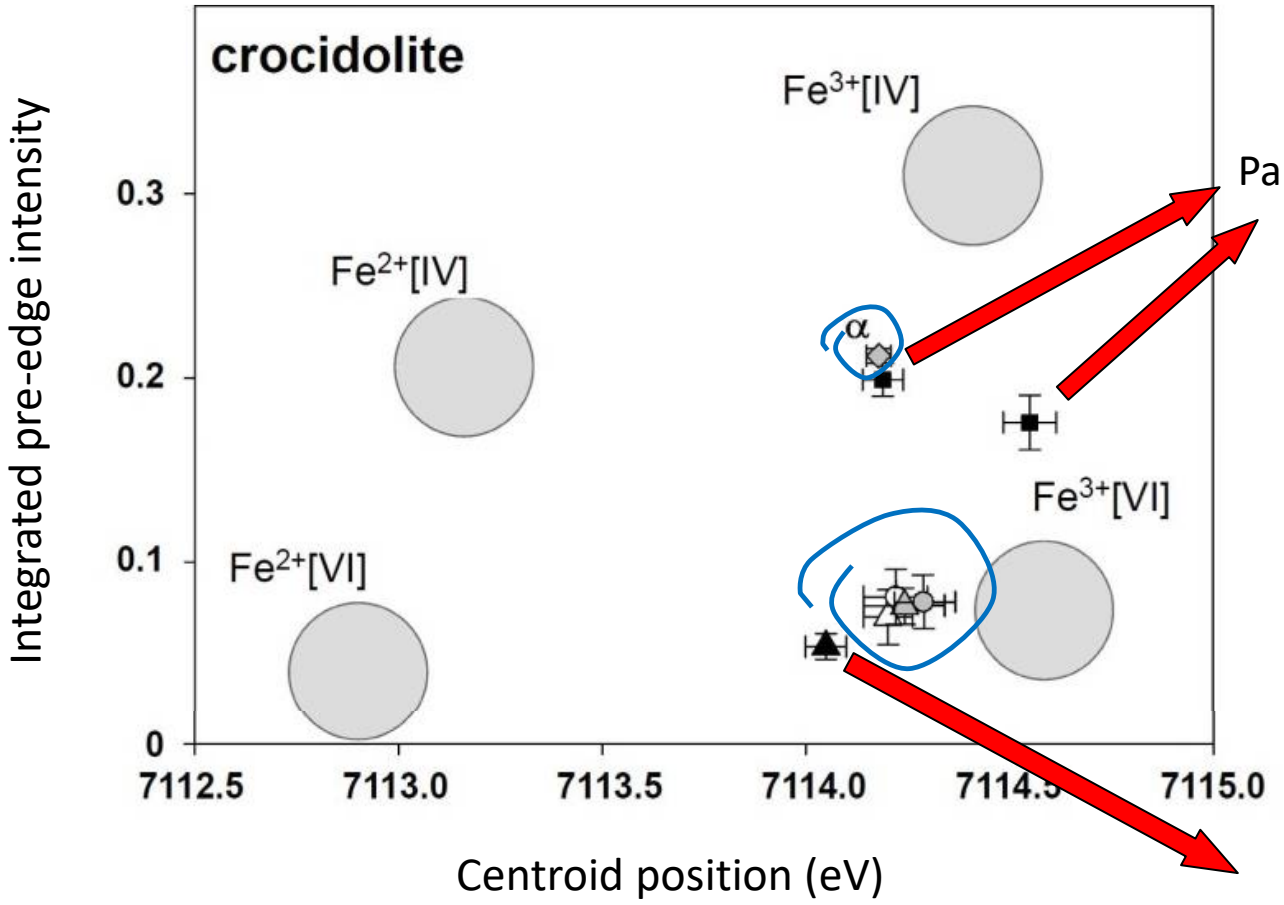


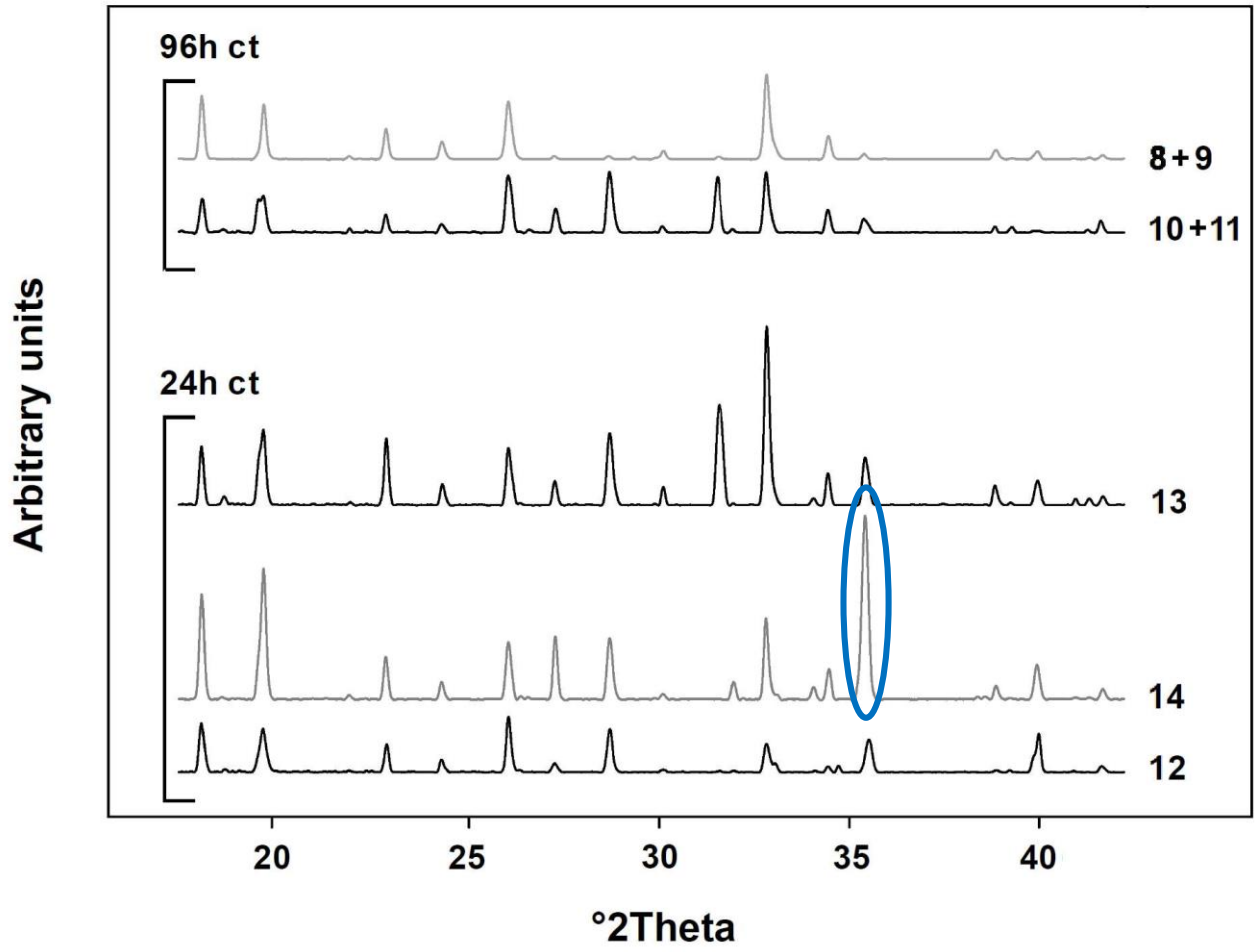
Parameters of pure magnetite-maghemite minerals

Treated samples

Untreated sample

In-situ experiment: Results

For all collected XRD patterns, peak profiles have been modeled.



Pattern of chrysotile crocidolite treated with Met5A.

Pattern of chrysotile crocidolite treated with Met5A.

Contribution due to magnetite-maghemite particles (impurities)

In-situ experiment: Results

The present data, together with μXRD and further TEM investigations, were published in the Chemosphere journal

Pattern of erionite treated with Met5A.

Chemosphere 164 (2016) 547–557

Contents lists available at ScienceDirect

Chemosphere

journal homepage: www.elsevier.com/locate/chemosphere

Stability of mineral fibres in contact with human cell cultures. An *in situ* μXANES, μXRD and XRF iron mapping study

Simone Pollastri [a, *], Alessandro F. Gualtieri [a], Ruggero Vigliaturo [b], Konstantin Ignatyev [c], Elisabetta Strafella [d], Armanda Pugnaloni [d], Alessandro Croce [e]

Elettra Sincrotrone Trieste

XAFS

Clean energy research

Characterization of innovative Pt-ceria catalysts for PEMFC by means of ex-situ and operando X-Ray Absorption Spectroscopy

Simone Pollastri[a,d], Marco Bogar[a,b], Roman Fiala[a,c,f], Heinz Amenitsch[b], Yurii Yakovlev[c], Alessandro Lavacchi[e], Giuliana Aquilanti[d] and Vladimir Matolin[c]

[a]CERIC-ERIC, Strada Statale 14 34149 Basovizza, Trieste, Italy

[b]Graz University of Technology, Institute for Inorganic Chemistry, Stremayrgasse 9, 8010 Graz, Austria

[c]Faculty of Mathematics and Physics, Charles University, Prague, Czech Republic

[d]Elettra - Sincrotrone Trieste S.C.p.A., SS 14 - km 163,5, 34149, Basovizza, Trieste, Italy

[e](CNR-ICCOM), Via Madonna del Piano 10, 50019, Sesto Fiorentino, Italy

[f]RadBee Technology s.r.o., Kokorov 8, 33501 Zinkovy, Czech Republic

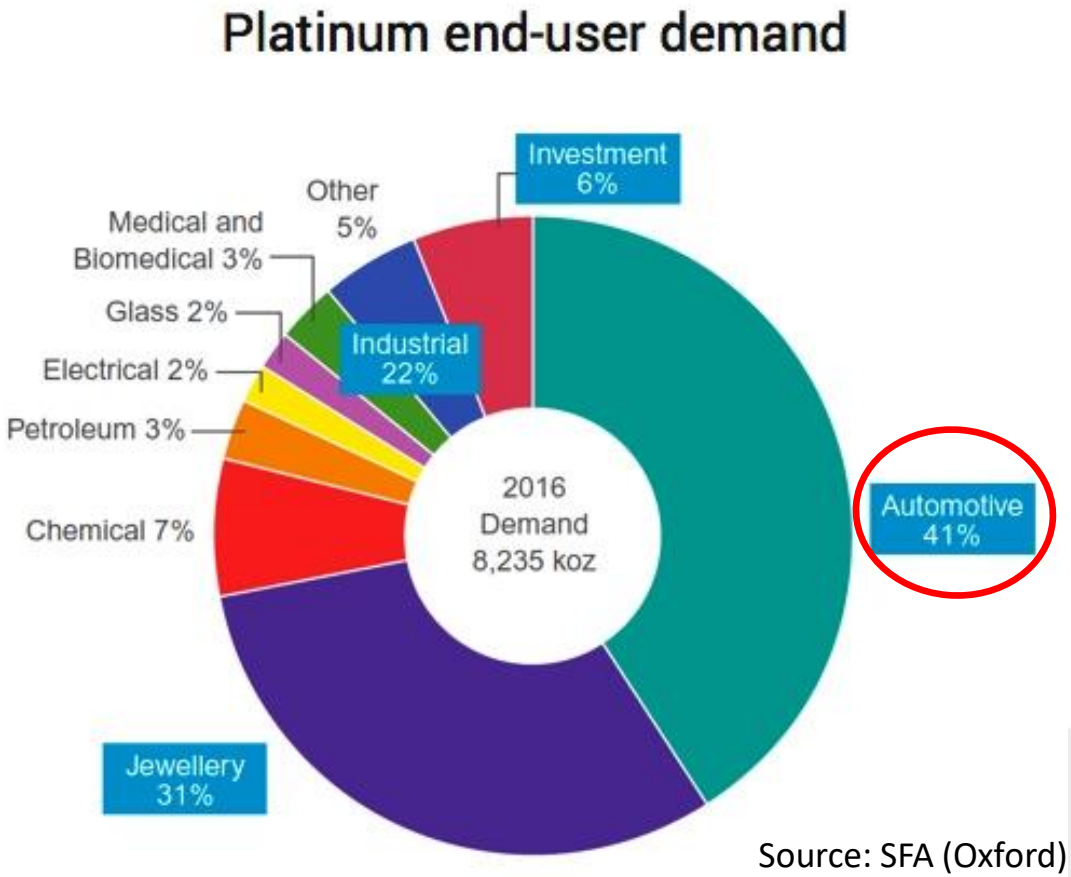
CERIC Central European Research Infrastructure Consortium

Case study: The CEROP project

CEROP is one of the CERIC grant research project, aimed at deciphering single-atom catalysis in Pt/ceria systems (Trovarelli & Fornasiero, 2013; Mullins, 2015). These systems are receiving great attention being a valid alternative to the expensive commercial Pt-based catalysts.

Indeed, the high cost of Pt and the fact that approximately 40% of the worldwide produced Pt is used for automobile catalytic converters (Gandhi et al., 2003) led the research to substitute it with Pt alloys.

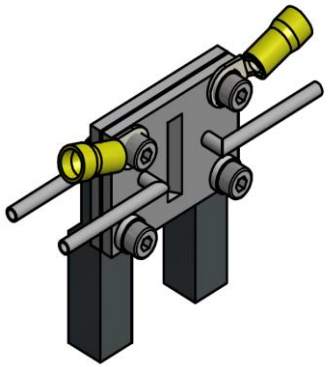
However (unfortunately) those kind of devices display a reduced stability and premature aging, mainly due to coarsening and degradation phenomena at the electrodes (Fiala et al., 2016).



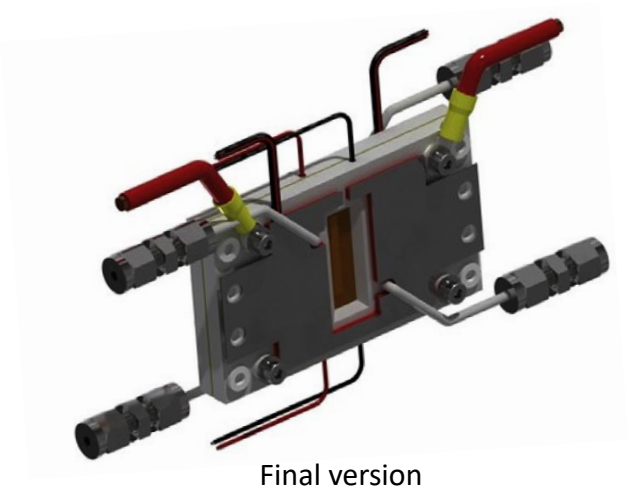
Source: SFA (Oxford)

Case study: The CEROP project

The CEROP project

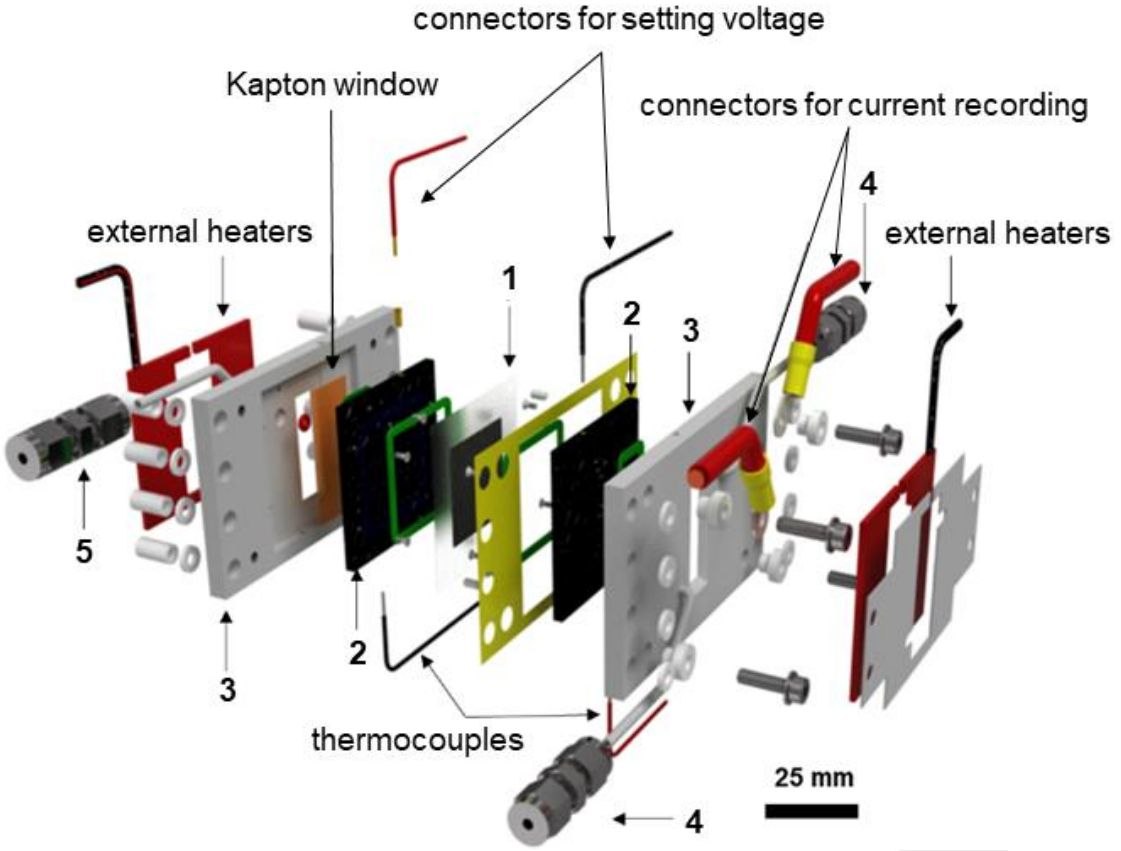


Very first draft of the cell



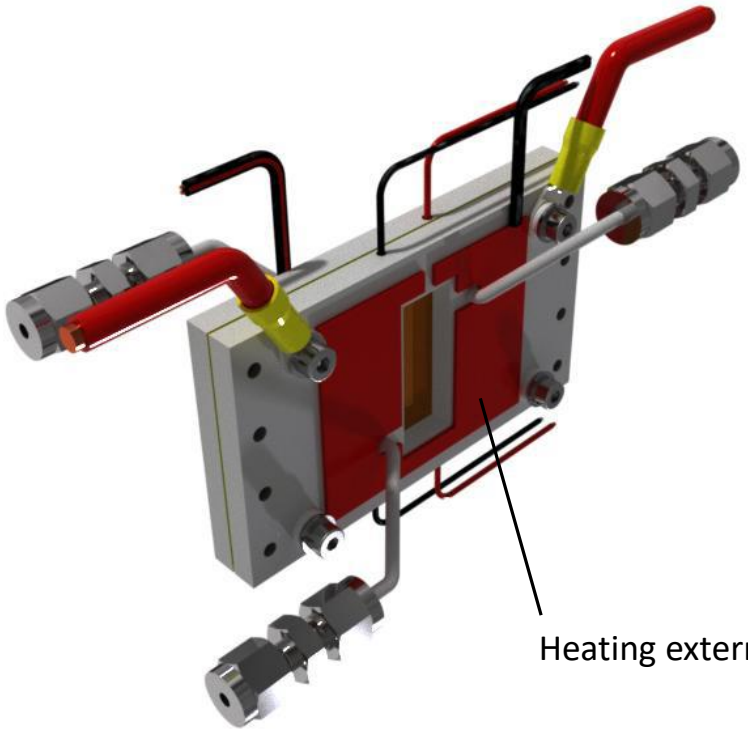
Final version

The role of batteries and fuel cells within the energetic transition is undeniable, but fuel cell systems are still currently not employed worldwide mainly because of their cost, which is due to the large amount of Pt used in catalyst layers.
The CEROP project aimed at investigating the stability of innovative Pt-CeOx anode catalyst deposited on different supports and characterized by means of *operando* X-ray Absorption Spectroscopy (XAS) and Small Angle X-ray Scattering (SAXS).

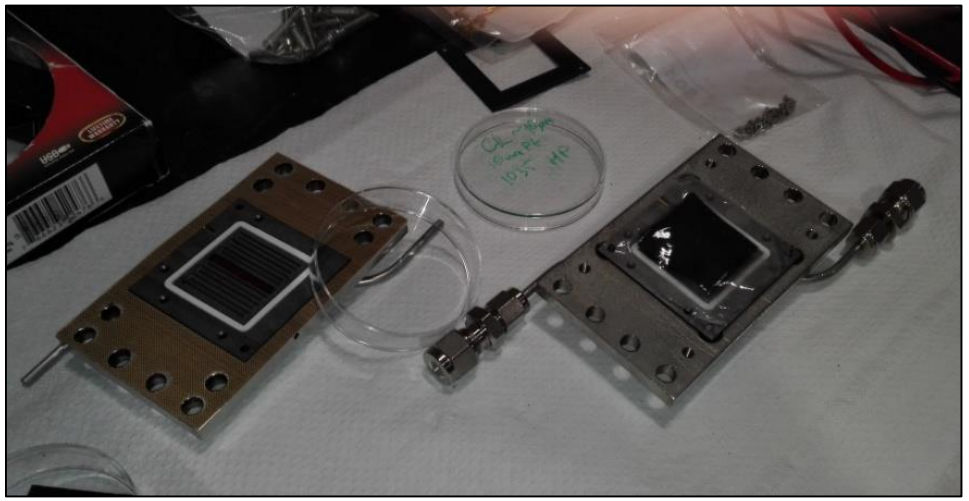


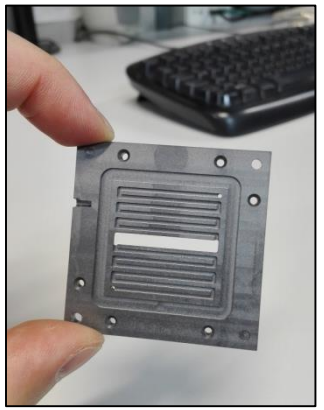
Case study: The CEROP project

A special PEMFC has been designed for the characterization of the anode catalyst *in operando* conditions at the Elettra synchrotron facility.



The assembled PEMFC



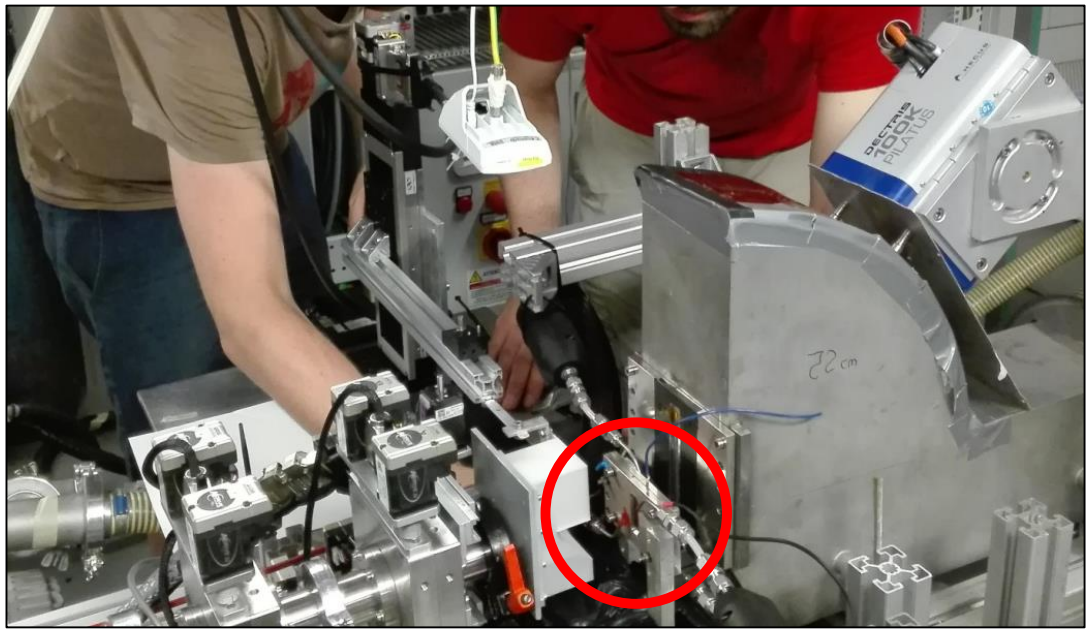


Assembling the various components of the cell (on the left, one of the graphite gas distributors).

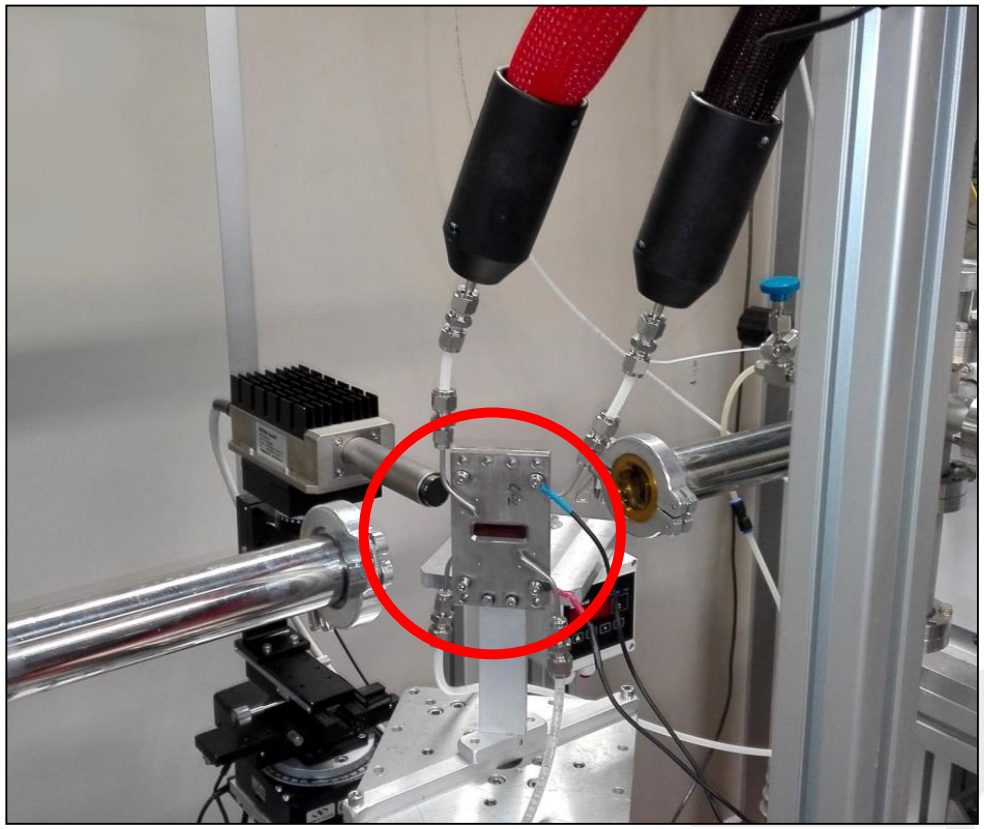
Image courtesy of Roman Fiala.

Case study: The CEROP project

The working PEMFC has been analyzed *in operando* conditions first at the Small Angle X-ray Scattering (SAXS) beamline and then at the XAFS beamline. The great advantage is represented by the possibility to combine the morphological information from SAXS (nanoscale structure, particle sizes, shapes, distribution and porosity) with the chemical information (both at the Pt and Ce L_3-edges, 5.72 and 11.56 keV, respectively) from XAFS (oxidation state and coordination geometry).



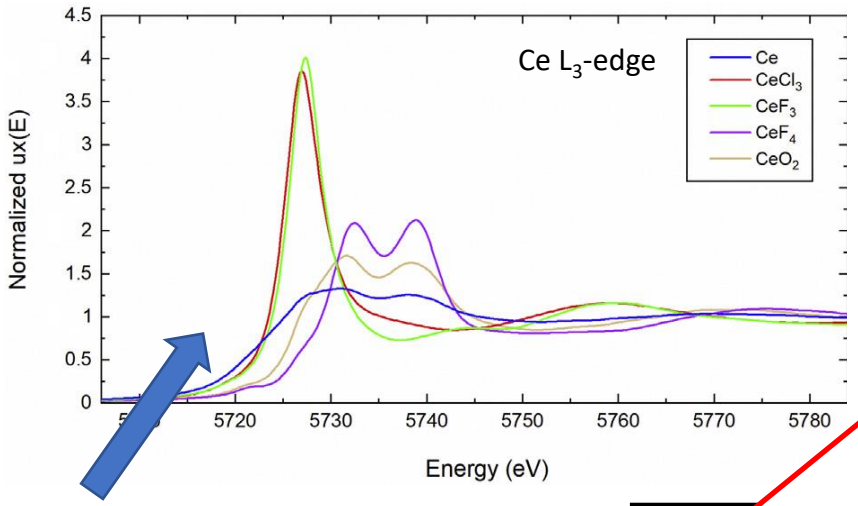
PEMFC mounted on the SAXS sample station.



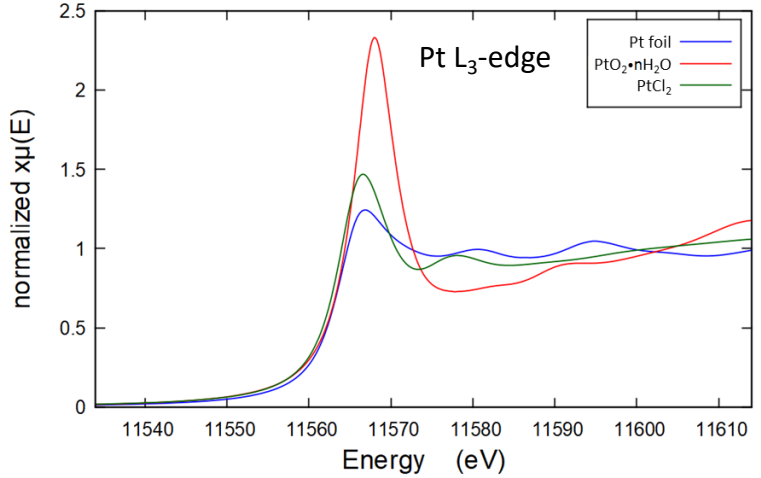
The same PEMFC mounted on the XAFS sample station.

Case study: The CEROP project

Firstly, we finely characterized *ex situ* samples and collected spectra from both Pt (EXAFS) and Ce (XANES) reference compounds.



Ce L_3-edge: Good XANES but no EXAFS.



XAFS bm (in air)

Pt L_3-edge: Good EXAFS but "bad" XANES.

XRF bm (in vacuum)

Q: Why no EXAFS for Ce L3-edge?

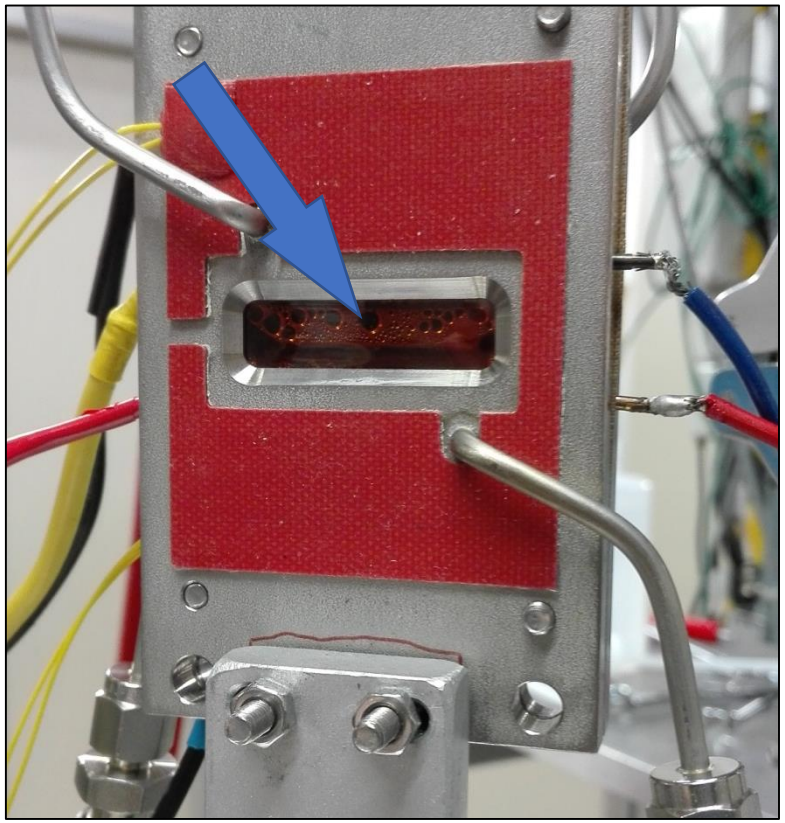
Table 1 ...phic data and structural parameters as obtained from the R-space fit by using the theoretical refer...

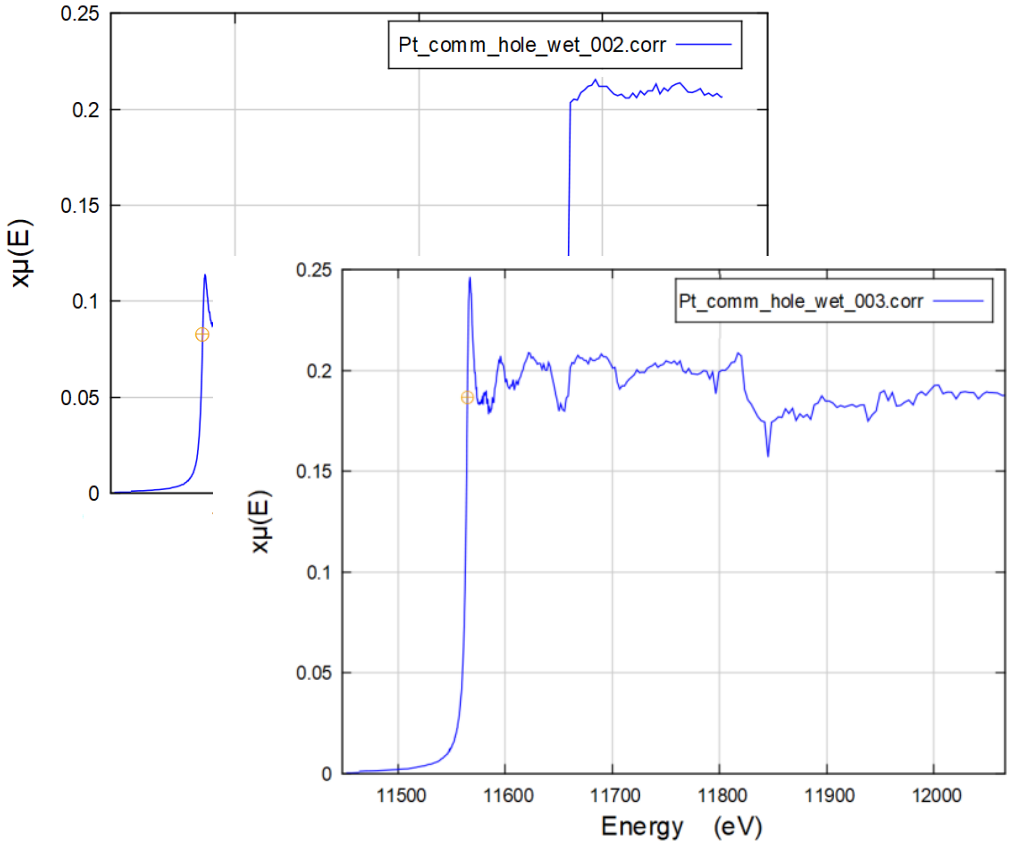
S...	%	N	Atom	R(Å)	S_0^2	R-factor	σ^2 (Å2)	ΔE_0 (eV)
		12[a]	Pt	2.761 (13)	0.76 (5)	0.009	0.0042 (5)	7.4 (4)
	0.90 (7)	12[a]	Pt	2.750 (24)	0.63 (7)	0.009	0.006 (1)	6.9 (9)
	0.10 (7)	4[b]	O	1.958 (17)			0.002 (8)	
...1μg	0.76 (7)	12[a]	Pt	2.730 (44)	0.68 (7)	0.008	0.007 (1)	5 (1)
	0.24 (7)	4[b]	O	1.963 (19)			0.009 (1)	
$Pt10\mu gCeO_x$	0.70 (5)	12[a]	Pt	2.729 (45)	0.69 (8)	0.007	0.006 (1)	4 (1)
	0.30 (5)	4[b]	O	1.941 (34)			0.006 (1)	
$Pt1\mu gCeO_x$	0.51 (4)	12[a]	Pt	2.733 (41)	0.77 (11)	0.010	0.008 (2)	6 (2)
	0.49 (4)	4[b]	O	1.983 (10)			0.007 (2)	

[a] Shell calculated from the crystallographic data of metallic Pt of Wyckoff (1963) [56];
[b] Shell calculated from the crystallographic data of Pt_3O_4 of Muller and Roy (1968) [57].

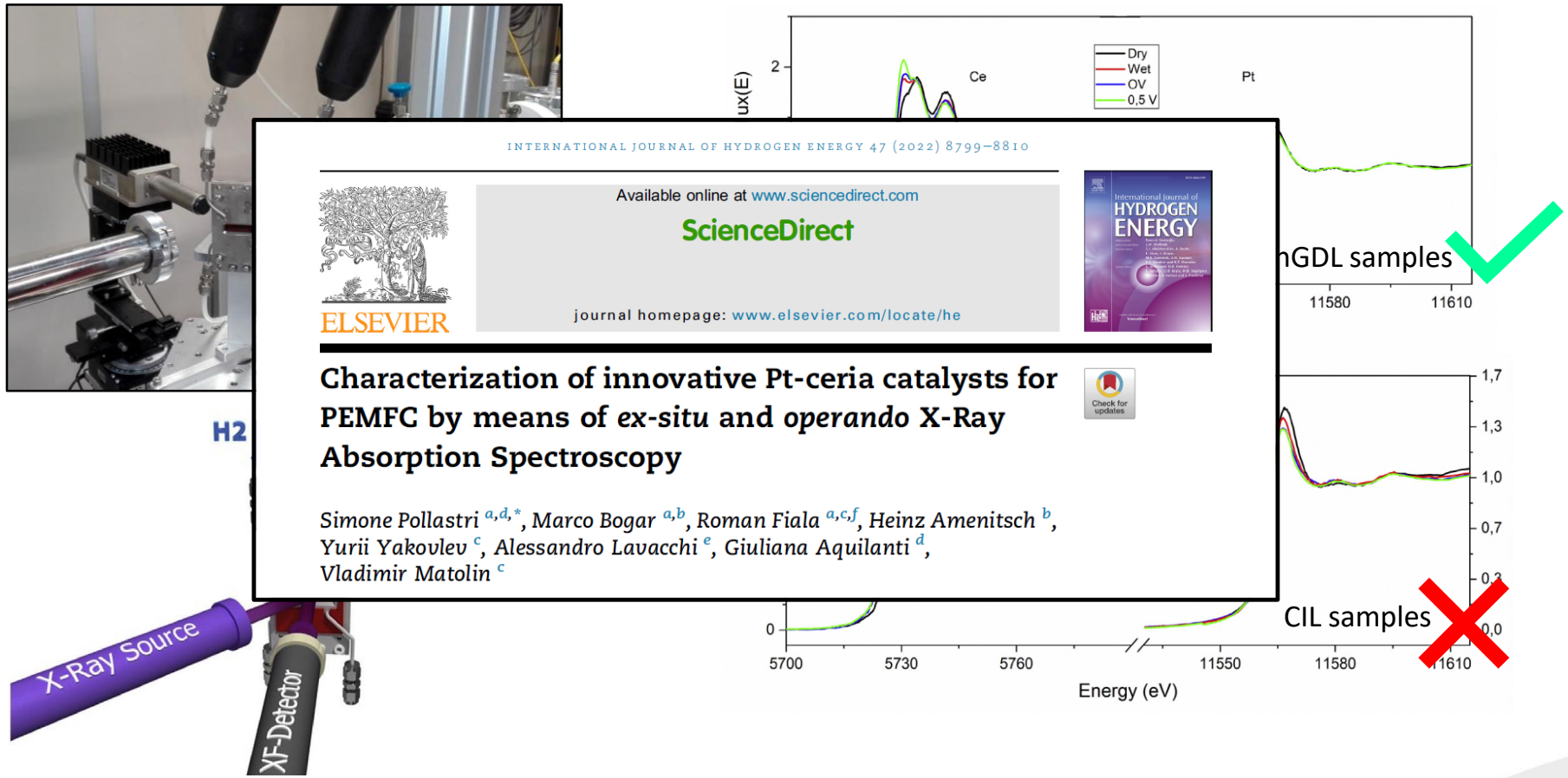
Case study: The CEROP project

However, during *operando* measurements is not rare to encounter many kind of problems.
In this case, the H_2O vapors carried with flowing of H_2 and O_2 gases lead to the formation of water drops that strongly affected the data, as during the collection of one single spectra they moved, passing in front of the beam.





Case study: The CEROP project



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Characterization of innovative Pt-ceria catalysts for PEMFC by means of *ex-situ* and *operando* X-Ray Absorption Spectroscopy

Simone Pollastri [a,d,*], *Marco Bogar* [a,b], *Roman Fiala* [a,c,f], *Heinz Amenitsch* [b], *Yurii Yakovlev* [c], *Alessandro Lavacchi* [e], *Giuliana Aquilanti* [d], *Vladimir Matolin* [c]

Elettra Sincrotrone Trieste

Sustainability and circular economy

A combined XRF and XANES study on bottom ashes from municipal solid Waste-to-Energy plant

Simone Pollastri[1,2], Chiara De Matteis[3], Luciana Mantovani[3] and Mario Tribaudino[4]

[1]Elettra - Sincrotrone Trieste S.C.p.A.

[2]University of Modena and Reggio Emilia, Earth sciences department, Modena, Italy

[3]Università di Parma, Dipartimento di Scienze Chimiche, della Vita e Sostenibilità Ambientale

[4]Università di Torino, Dipartimento di Scienze della Terra

NOT YET PUBLISHED data, contact simone.pollastri@unimore.it for more info

Elettra Sincrotrone Trieste

Detection of bioaccumulated Cd in non-crushed oyster and queen scallop shells using XRF mapping and XANES spectroscopy.

Pollastri S., Martucci A., Pasti L., Chenet T., Baldi A.

NOT YET PUBLISHED data, contact simone.pollastri@unimore.it for more info

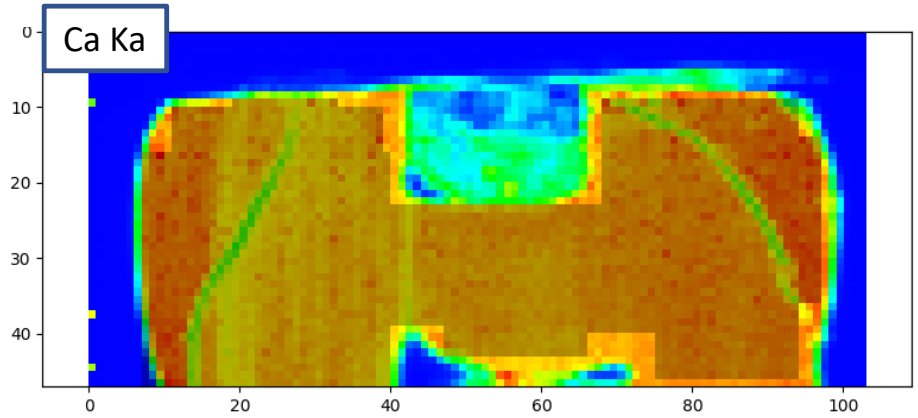
Paleontology

Elettra Sincrotrone Trieste

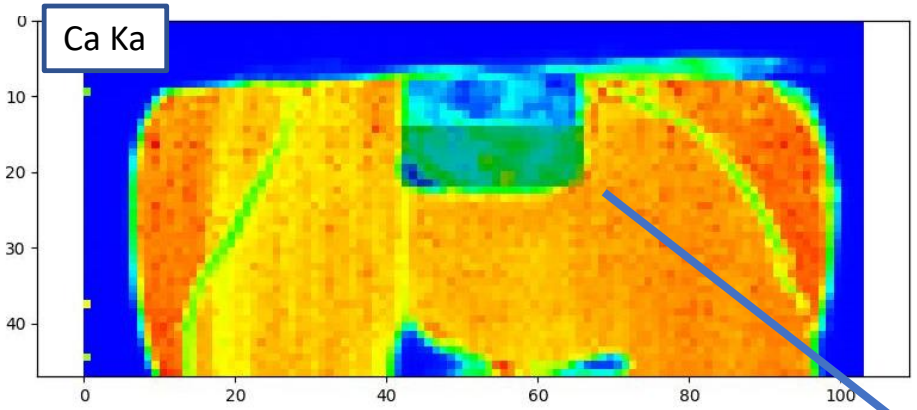
Insights on the morphological Structure of Remineralized Dentin Obtained by Synchrotron Radiation and micro-PIXE

Seyedeh Zahra Karrari, Hossein Afarideh, Hamid Kermanshah, Giuliana Aquilanti, Davoud Agha Aligol, Zahra Shahidi, Simone Pollastri, Danilo Oliveira de Souza

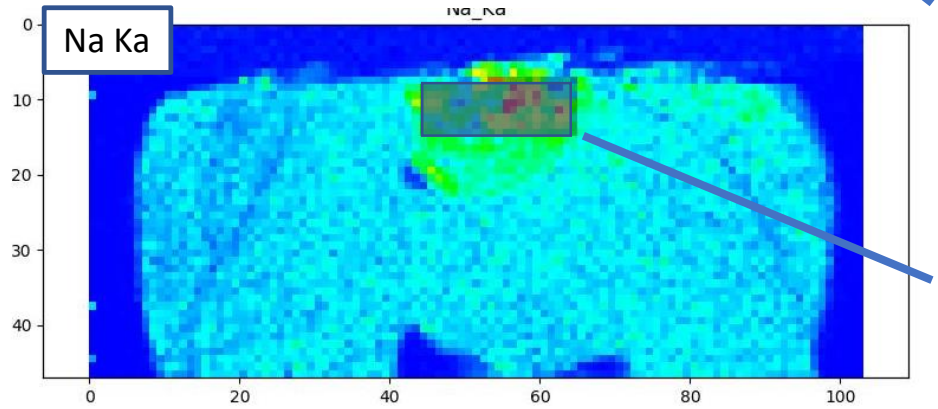
Medicine



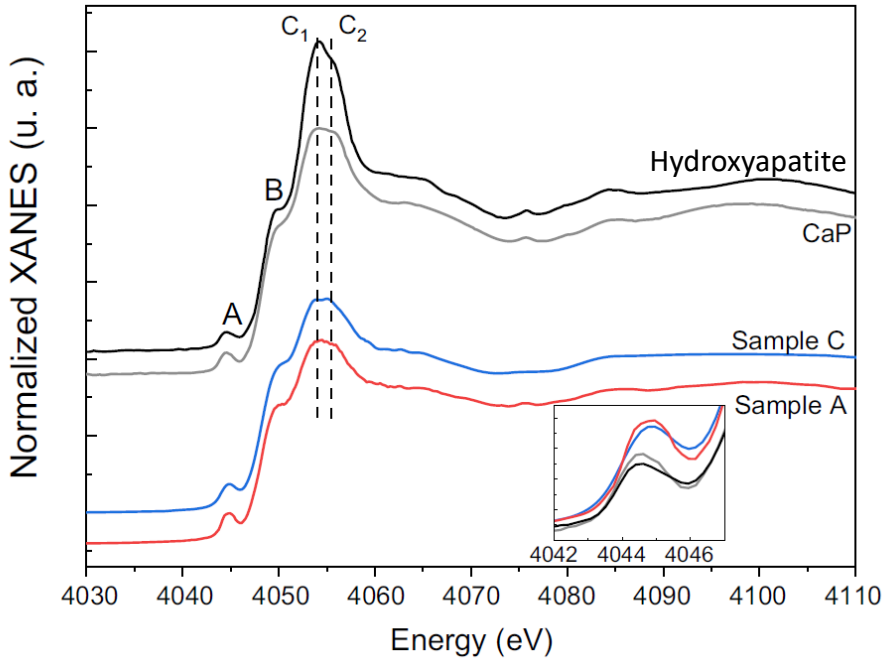
Areas integrated to get the average healthy tooth spectrum

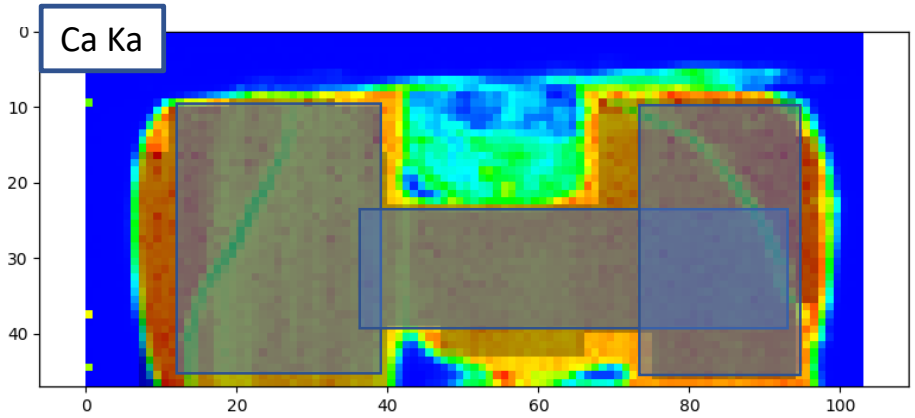


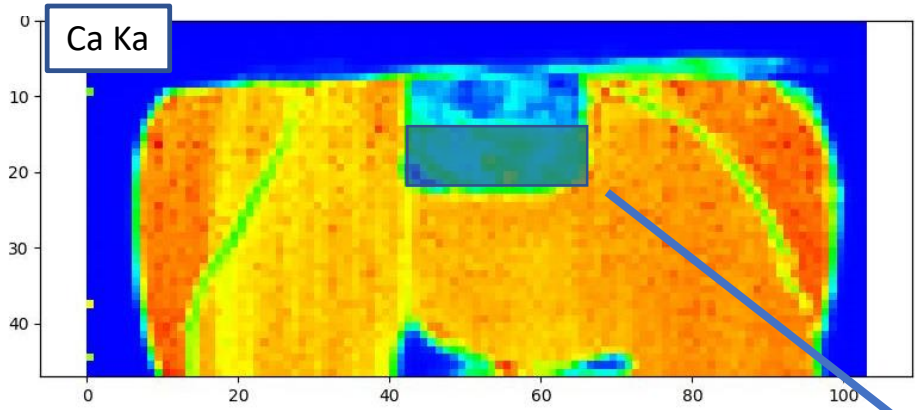
Area integrated to get the remineralized representative spectrum

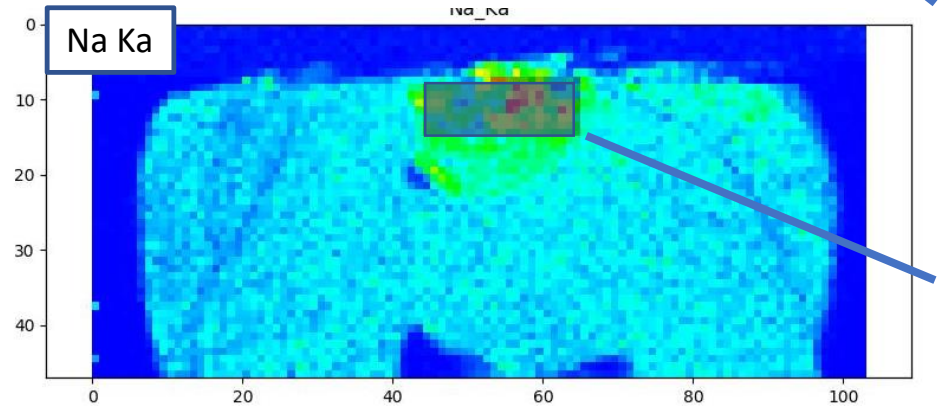


Area integrated to get the Bioactive representative spectrum

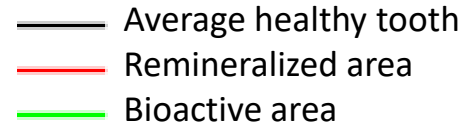


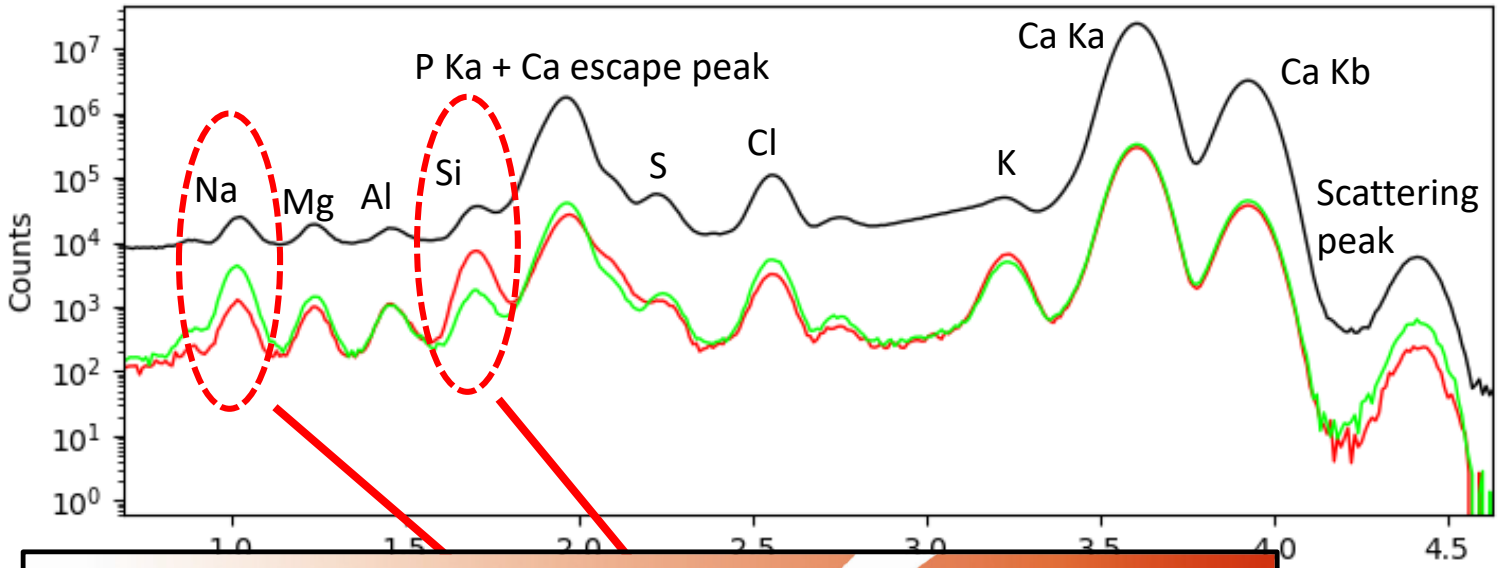






Areas integrated to get the average healthy tooth spectrum





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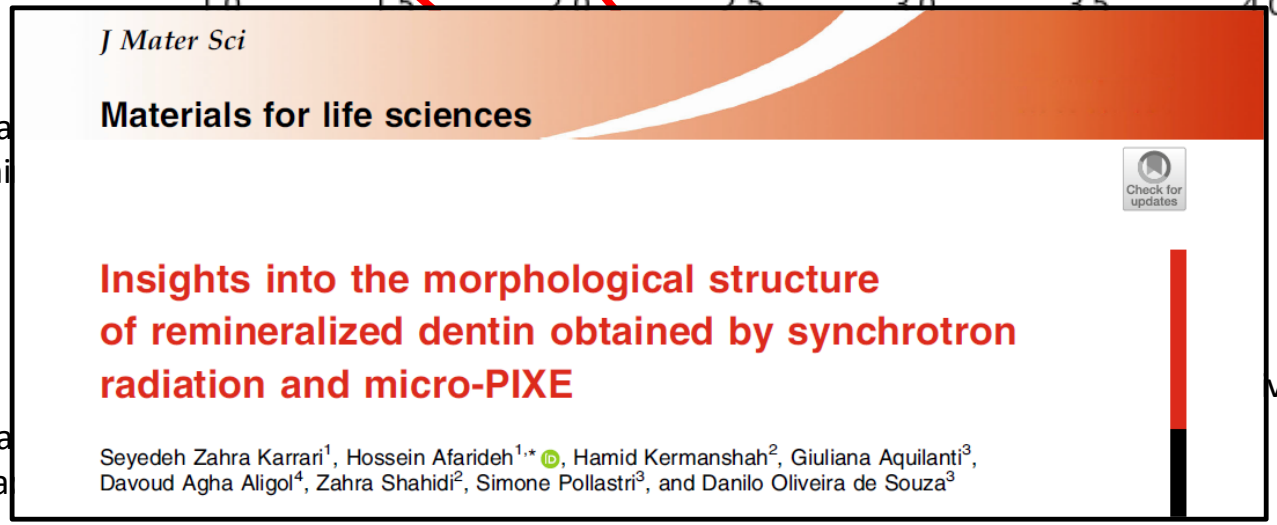


J Mater Sci

Materials for life sciences

Check for updates

Insights into the morphological structure of remineralized dentin obtained by synchrotron radiation and micro-PIXE

Seyedeh Zahra Karrari[1], Hossein Afarideh[1,*], Hamid Kermanshah[2], Giuliana Aquilanti[3], Davoud Agha Aligol[4], Zahra Shahidi[2], Simone Pollastri[3], and Danilo Oliveira de Souza[3]

Elettra Sincrotrone Trieste

A multidisciplinary study unveils the nature of a Roman ink of the I century AD

Mirta Sibilia, Chiaramaria Stani, Lara Gigli, Simone Pollastri, Alessandro Migliori, Francesco D'Amico, Chiara Schmid, Sabina Licen, Matteo Crosera, Gianpiero Adami, Pierluigi Barbieri, Jasper R. Plaisier, Giuliana Aquilanti, Lisa Vaccari, Stefano Buson & Federica Gonzato

scientific reports

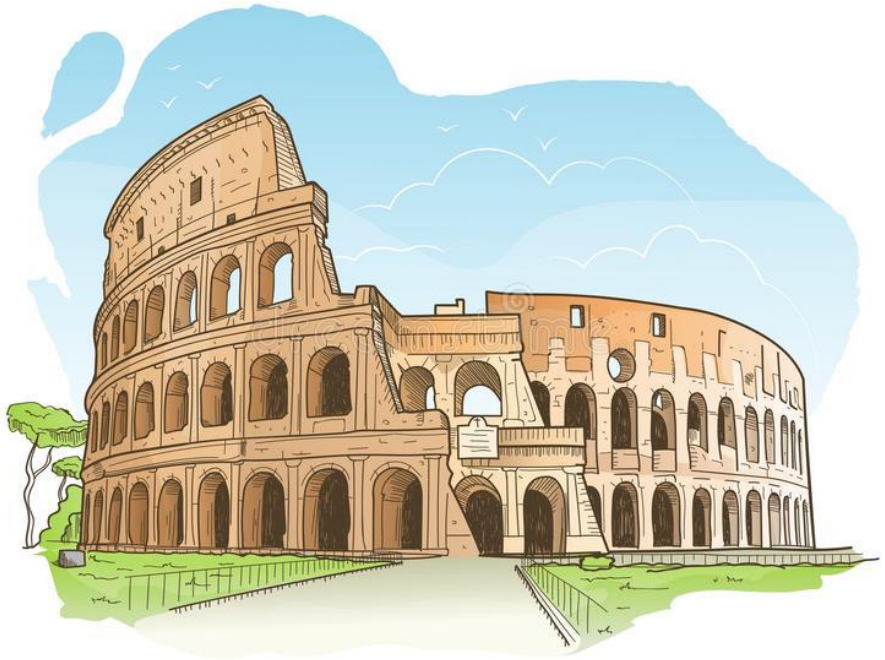
Check for updates

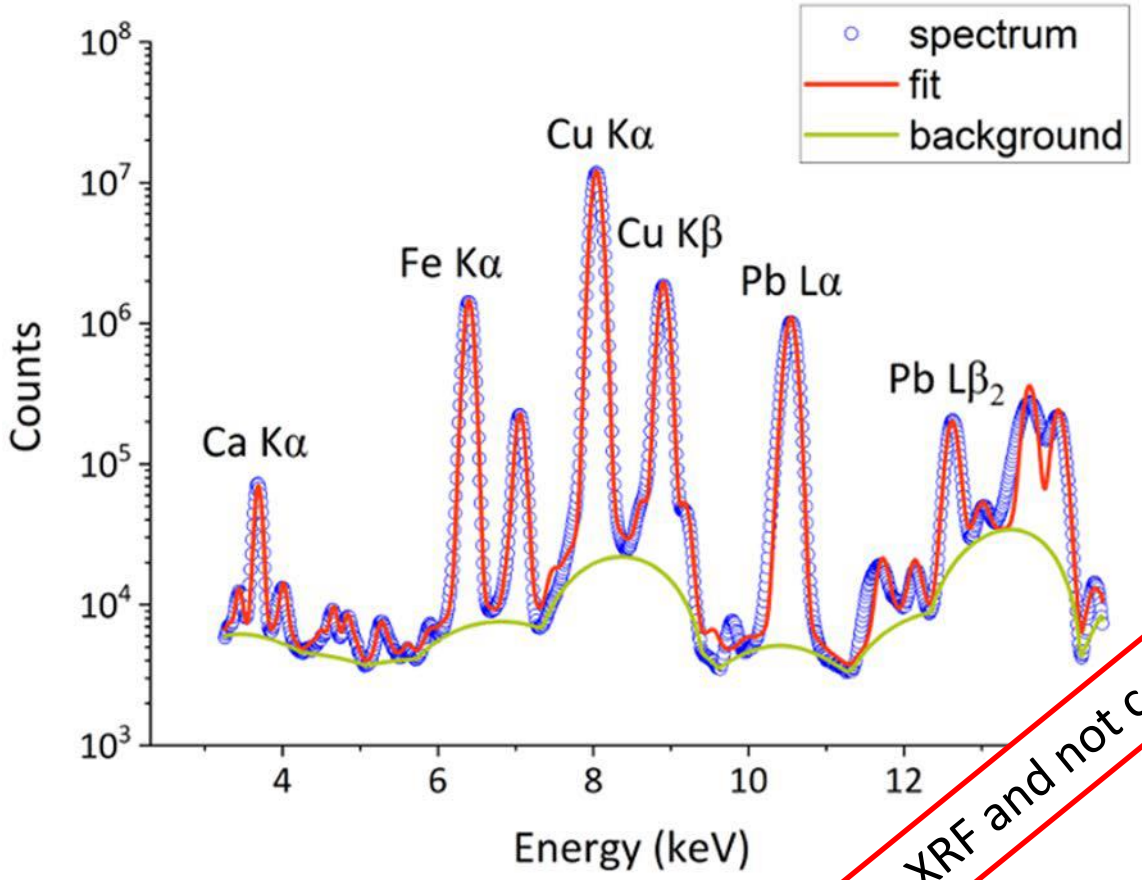
OPEN

A multidisciplinary study unveils the nature of a Roman ink of the I century AD

Mirta Sibilia[1], Chiaramaria Stani[2✉], Lara Gigli[3], Simone Pollastri[3], Alessandro Migliori[1], Francesco D'Amico[3], Chiara Schmid[4], Sabina Licen[5], Matteo Crosera[5], Gianpiero Adami[5], Pierluigi Barbieri[5], Jasper R. Plaisier[3], Giuliana Aquilanti[3], Lisa Vaccari[3], Stefano Buson[6] & Federica Gonzato[6✉]

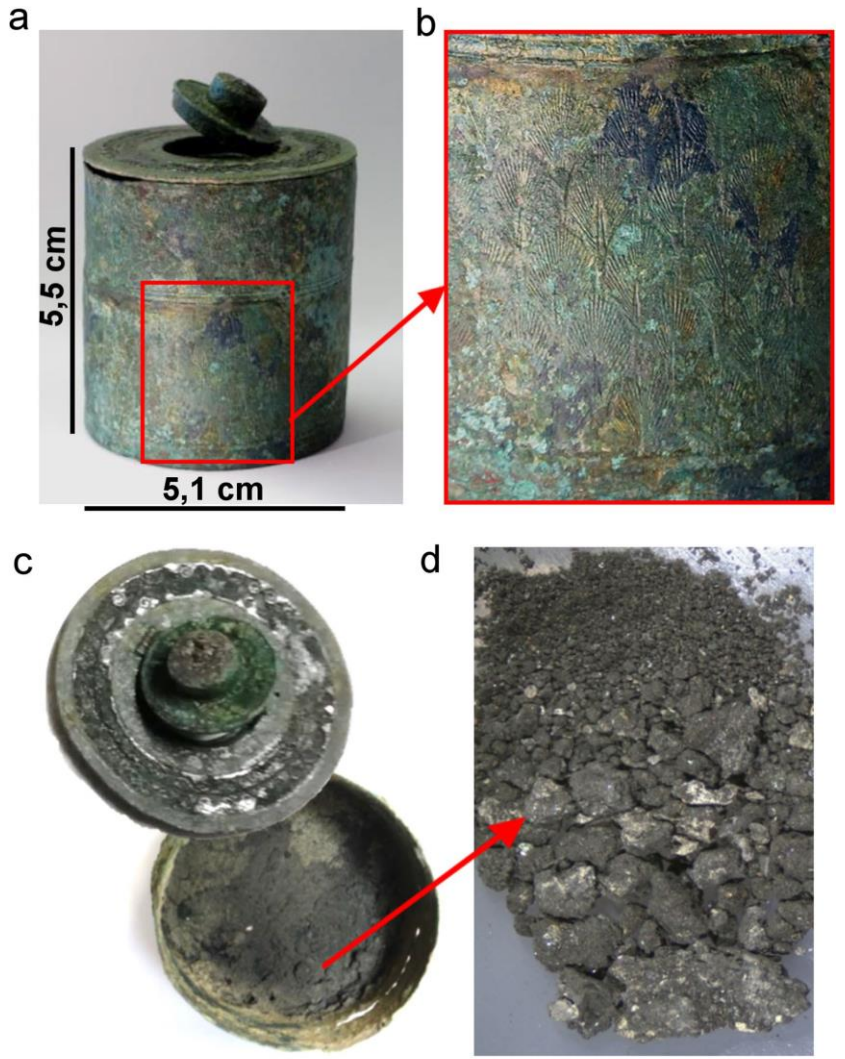
Cultural heritage



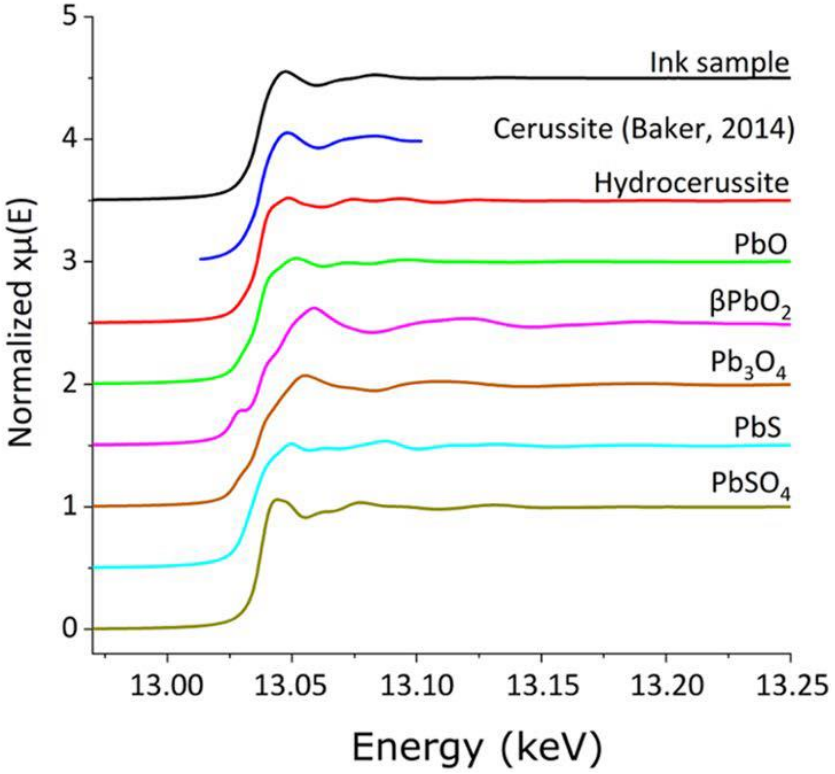


(**a**) SR-XRF average spectrum of th sample (experimental: empty blue circle) a t obtained with PyMca (red curve);

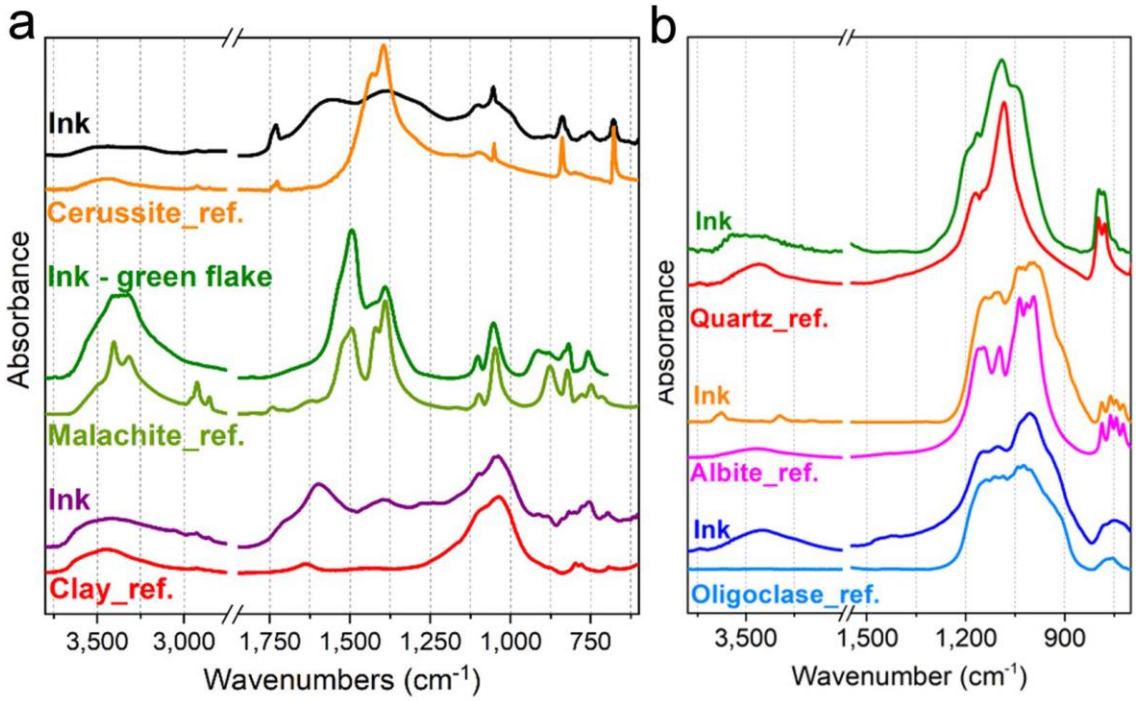
Q: Why SR XRF and not conventional lab?

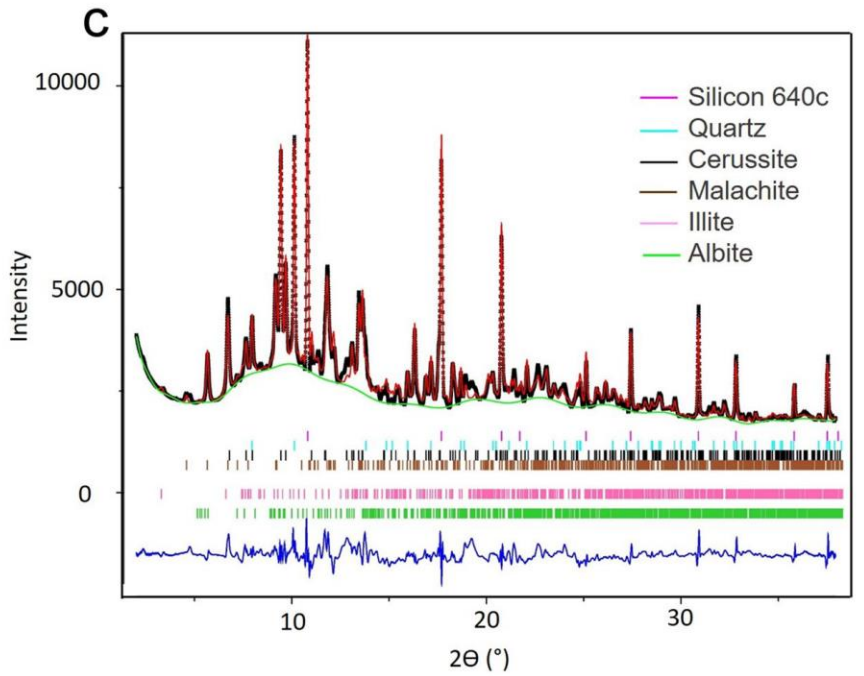


(**a**) the inkpot; (**b**) the inkwell bronze decoration; (**c**) an internal view of the inkwell with the black powder on the bottom; and a top view of the lid: traces of silver agemina are clearly visible; (**d**) the black powder with colored particles.



Normalized spectra of the ink sample and all the collected XAS data of reference compounds. The cerussite (PbCO3) spectrum is taken from Baker et al.





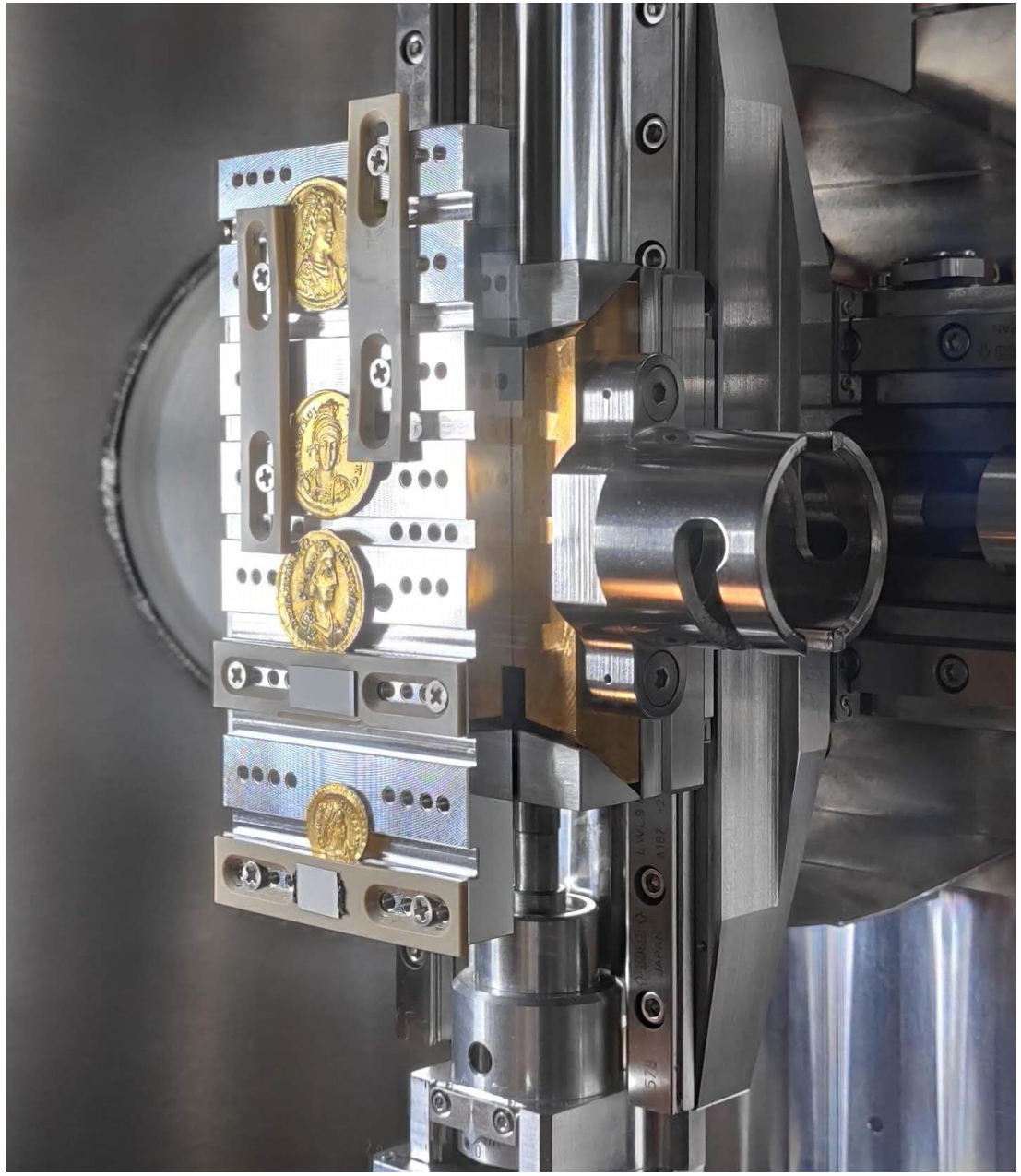
(**a,b**) Infrared spectra of several ink samples and possible reference spectra from the database (**c**) XRPD Rietveld refinement profile fit of the ink sample: black crosses are the experimental data, in red the calculated pattern. The residuals are displayed on the bottom in blue and the reflection ticks of each phase with the colours reported in the legend.

Another example for cultural heritage application:

Investigation on a set of Roman gold coins of the III - IV century

Collaboration project with the University of Trieste, to study a set of gold coins dating back to the III - IV century
Thanks to the high sensitivity offered by the X-ray beam, the gold purity could be evaluated with high precision, providing fine details to describe the economic inflation during the Roman Empire.

Not only gold, but also trace elements were inspected: elements like Hg and Pb shed light on the processes that Romans used to obtain coins of extraordinary purity; while Pd and Pt are valuable elements to determine the mine location. Besides the precious metals, some attention was paid also to the incrustations found on the coins surface: thanks to the analysis of the elements found in such areas, the fate of the coins across the centuries will be unravelled.



For more info, ask Ilaria ilaria.carlomagno@elettra.eu

Elettra Sincrotrone Trieste

Not only k edges and hard x rays...

Clean energy research

In-operando investigation for cheaper and efficient Electrochemical Energy Converter (EEC) devices using a novel (precious metals-free) chiral catalyst.

Simone Pollastri[a], Roberto Biagi[a], Elena Magnano[b], Silvia Nappini[b], Ilargi Napal Azcona[b], Martina Campi[a]

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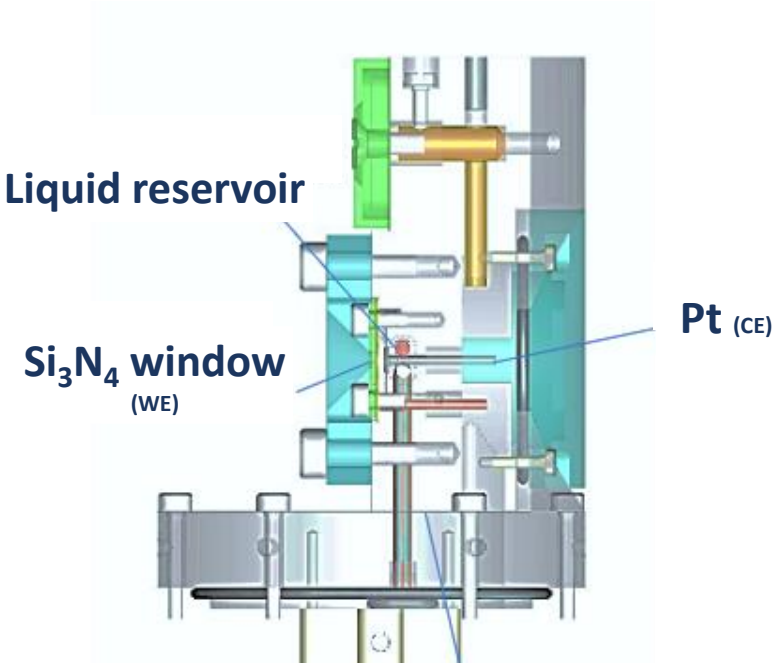
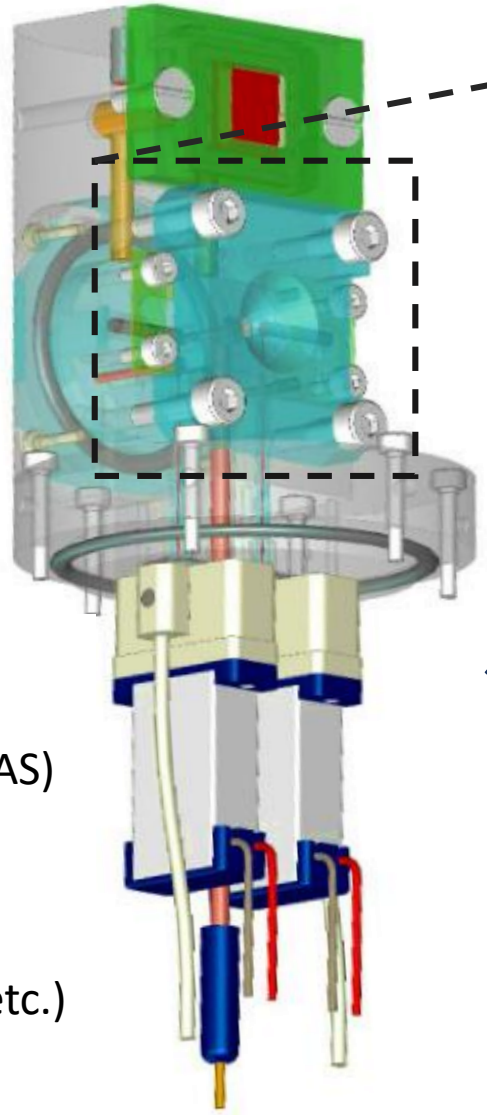
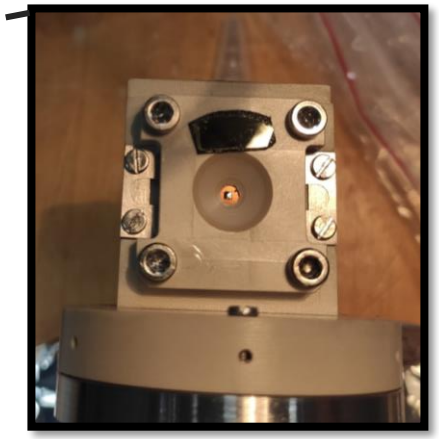
ISTITUTO OFFICINA DEI MATERIALI

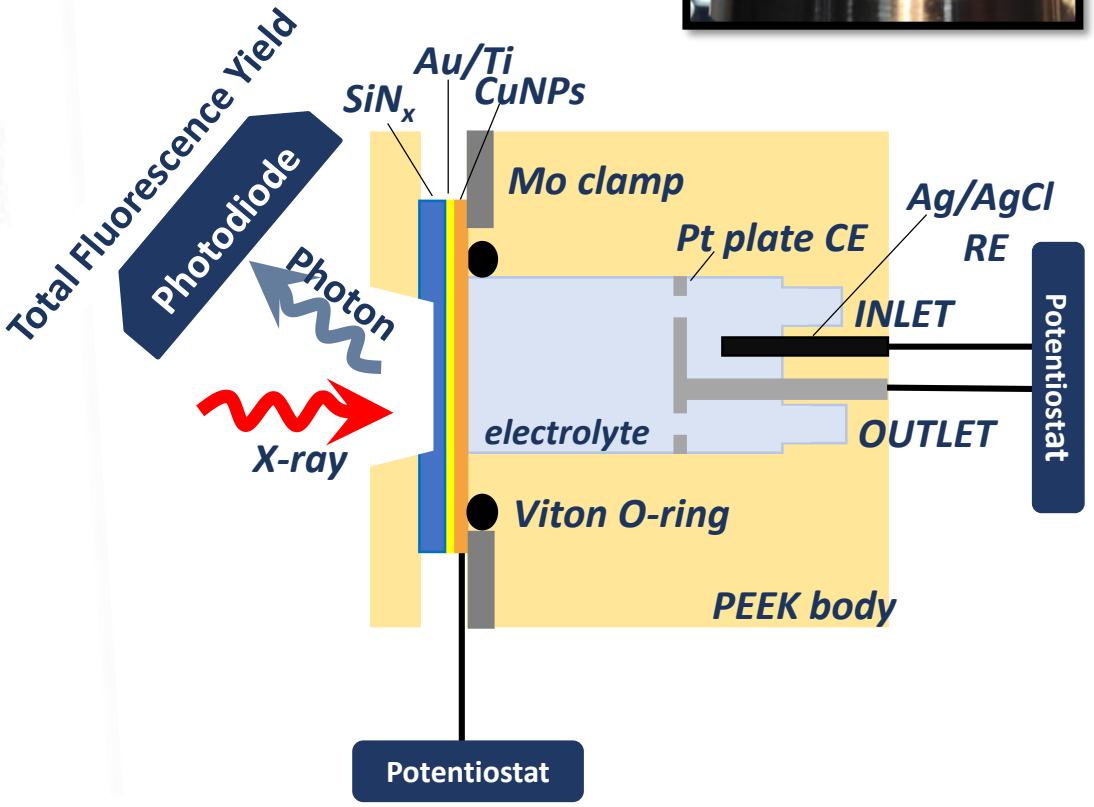
UNIVERSITÀ DEGLI STUDI DI MODENA E REGGIO EMILIA

Preliminary XAS data collected in operando using a microfluidic electrochemical cell developed at BACH beamline.









Main soft X-ray limitations:

- Forced to work in ultra high vaccum (10^{-9})
- Limited probing depth (no transmission XAS)

Main soft X-ray advantages:

- Possible to probe light elements (O, C, N etc.)
- Less damage to sensitive samples
- Higher efficiency for TEY measurements

NOT YET PUBLISHED data, contact simone.pollastri@unimore.it for more info