EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Revealing modifications of asbestos after prolonged stay in the lungs	**Experiment number**: LS3009
Beamline:	**Date of experiment**: from: 05/04/2022 to: 08/04/2022	**Date of report**: 09/09/2022
Shifts:	**Local contact(s)**: Francesco D'Acapito	*Received at ESRF:*

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Report:

The aim of the experiment was to investigate the local structure and oxidation state of Fe sites in synthetic samples that can mimic variety of Fe states that can be encountered in Fe-bearing mineral fibers. To this aim, synthetic glasses with different structure, Fe content, and Fe2+/Fe3+ ratio were synthetized (e.g. diopside, egirine, hedenbergite, and jadeite). Reference Fe3+ and Fe2+ compounds, such as magnetite, hematite, and olivine were measured to build a calibration curve to perform pre-edge peak analysis at the Fe K-edge. With this analysis we were able to calculate the Fe2+/Fe3+ and the geometry of the Fe sites (tetrahedric, octahedric or and admixture of the two (Figure 1). Structural refinements performed on the EXAFS part of the absorption spectra revealed the local structure of the Fe sites for each samples, and the results were compatible with the pre-edge analysis (Table 1).

X-ray absorption spectra of asbestos bodies recovered from lung tissue, and of ferrihydrite and goethite reference compounds, were also acquired to confirm the results obtained by XRD measurements performed on ID11 (see experimental report LS3009). The results show that the spectrum of an asbestos body is very similar to both that of ferrihydrite and of goethite.

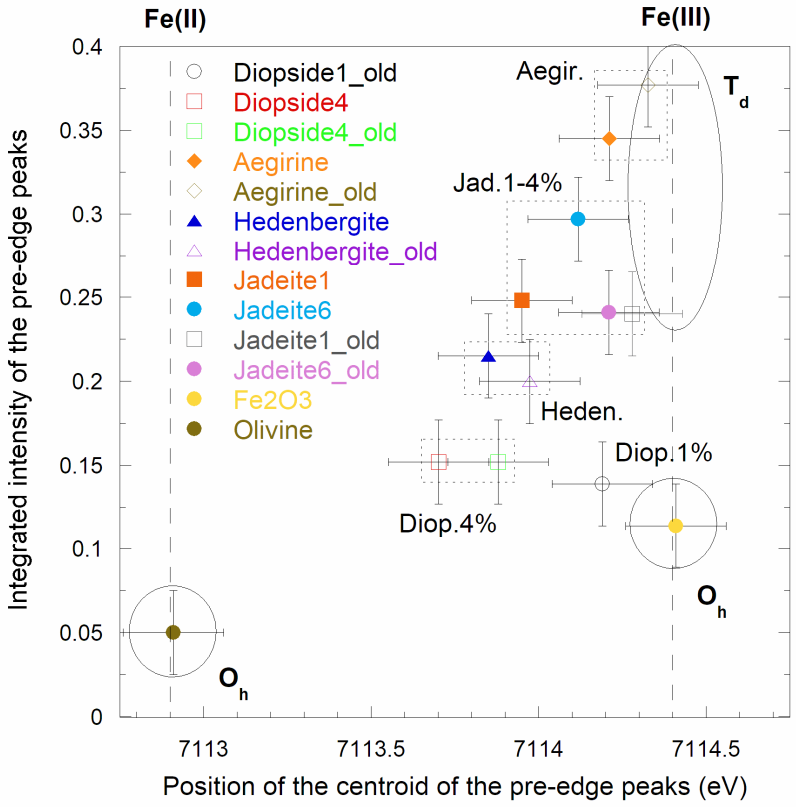

Figure 1. Position of the centroid vs. the integrated intensity of the pre-edge peaks. The analysis is based on the work of Wilke et al., 2001 (*American Mineralogist, Volume 86, pages 714–730, 2001*).

Table 1. Structural parameters of the Fe-bearing glasses and Fe reference compounds (hematite, olivine, Magnetite, and OIph1-a). The coordination numbers (CN) were kept fixed. The error on the refined parameters is ±0.05 Å on the bond distances (R), ±0.005 Å^2 on the Debye-Waller factors (σ^2), and up to 25% on the weight factor *x*. The reduced (χ_ν^2) value is also reported. The error on the Fe(III)/Fe$_{tot}$ ratio (from XANES analysis) is ±0.1.

		EXAFS					XANES		Moss.
	Fe %		Fe-O$_1$	Fe-O$_2$	O_h/T_d	χ_ν^2	Fe(III)/Fe$_{tot}$	Fe(III)/Fe$_{tot}$	Fe(III)/Fe$_{tot}$
							Old	New	
Diopside	1	CN R(Å) σ^2(Å^2)	4 1.93 0.001	2 2.10		1.2	0.86	-	0.28
Diopside	4	CN R(Å) σ^2(Å^2)	4 1.89 0.001	6 2.03	0.34	15	0.65	0.53	0.51
Jadeite	1	CN R(Å) σ^2(Å^2)	4 1.87 0.003			18	0.92	0.70	0.80
Jadeite	6	CN R(Å) σ^2(Å^2)	4 1.87 0.003			29	0.87	0.81	0.73
Hedenbergite	23	CN R(Å) σ^2(Å^2)	4 1.88 0.001	6 2.02	0.36	7.8	0.72	0.63	0.75

Aegirine	36	CN R(Å) σ^2(Å^2)	4 1.87 0.003			9.2	0.95	0.87	0.90
Olivine		CN R(Å) σ^2(Å^2)	6 2.09 0.06			30			
Hematite		CN R(Å) σ^2(Å^2)	4 1.95 0.004	2 2.10		8.1			
Magnetite		CN R(Å) σ^2(Å^2)	4 1.91 0.008	6 2.04	0.34	33			
[1]OlPh-1a		CN R(Å) σ^2(Å^2)	4 1.85 0.003						

[1]phonolite synthetic glass containing tetrahedral Fe(III) (Giuli et al., 2011).