ESRF	**Experiment title:** XAS analysis for the determination of the iron chemical enviroment of mineral fibers of economic and industrial importance.	**Experiment number:** 08-01-954
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Names and affiliations of applicants (* indicates experimentalists):

S. Pollastri*, A. F. Gualtieri* - Department of chemical and geological sciences, Largo Sant'Eufemia 19, 41121, Modena, Italy.

Report:

In order to determine the structural environment (oxidation state and coordination) of iron in mineral fibers of social and economic importance, XAFS spectra at the iron K-edge of seven asbestos fibers and a zeolite species were collected and quantitatively analysed.

Depending on the iron content, we have calculate the amount of sample in order to have an edge jump ($\mu_t d$) of 1.5 and an absorbance step between 0.2 – 1.0 for transmission mode measurements; samples with a smaller absorbance were analyzed in fluorescence mode. Powdered samples were mixed with 80-100 mg of cellulose with the exception of chrysotiles, for which boron nitride was used to increase the homogenization degree of the mixture. Mixtures were manually grounded and homogenized in an agate mortar and then pressed at 7 t to obtain pellets with diameter of 1.3 cm and area of 1.327 cm^2. All pellets were sealed between two kapton sheets in order to increase their resistance and minimize the fibers release risk into the environment.

For all experiments, energy calibration was achieved using iron foil as reference and the position of the first inflection point was taken at 7112 eV. The following reference compounds were used: almandine (Fe^{2+} [VIII]), hercynite (Fe^{2+} [IV]), siderite (Fe^{2+} [VI]), olivine (Fe^{2+} [VI]), biotite (Fe^{2+} [VI]), hematite (Fe^{3+}[VI]), goethite (Fe^{3+}[VI]), magnetite (Fe^{3+} [IV–VI]) and iron phosphate (Fe^{3+} [IV]).

All spectra were collected with a variable step energy as a function of energy as follows: 5 eV from 6900 eV to 7080 eV; 1 eV from 7080 eV to 7107 eV; 0.2 eV from 7107 eV to 7120 eV; 0.3 eV from 7120 eV to 7145 eV; 1 eV from 7145 eV to 7211 eV; 2 eV from 7211 eV to 7510 eV; 4 eV from 7510 eV to 8100 eV.

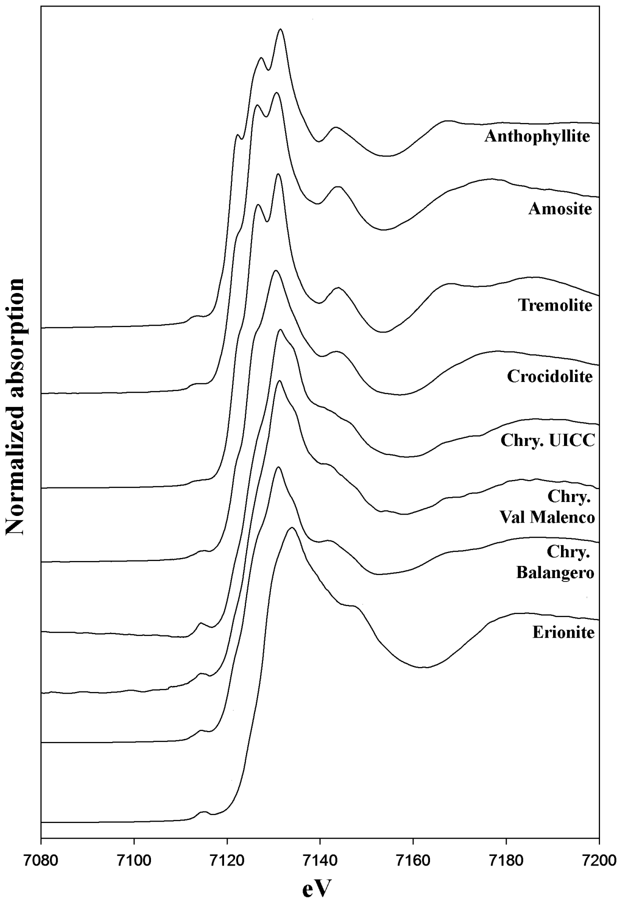
EXAFS spectra were collected up to 950 eV after the edge ($k=16$ Å^{-1}, k being the photo electron wave vector in atomic units), spectra of all samples were collected at room temperature.

Obtained results, both regarding XANES and EXAFS regions, are in general agreement with literature data [1-10].

Although we found some differences compared to the results obtained from electron microprobe analysis, the discrepancies are attributed to iron-rich impurity phases.

Regarding EXAFS analysis, besides the problems with the impurities, data should be considered only in qualitative terms, especially for chrysotiles because it was not possible to homogeneously distribute samples within pellets; in addition the majority of collected spectra presents an interference in the area around 7600 eV (about 11 K).

In Fig. 1, the Fe K-edge XANES spectra of investigated samples (used to carry out detailed studies of the pre-edge peaks) are reported. Preliminary results of the pre edge peaks' centroid positions of samples with respect to reference compounds seggest that there is no predominant oxidation state of iron within investigated samples.



Regarding Fe coordination, total areas of pre-edge peaks suggest that in the majority of samples, iron should be in a more centrosymmetric environment with respect to a tetrahedral coordination geometry.

Informations that we have obtained about the oxidation state of iron as well as its state of coordination within the crystal structure of the different species of mineral fibers will help us to draw a general model on the role of iron to the cyto- and geno-toxicity of the fibers.

This experiment is part of a larger project for the mineralogical-microstructural characterization of mineral fibers; the good outcome of this investigations should have a global impact of both social and economic value as a conclusive comparison of the toxicity of chrysotile and amphibole asbestos (crocidolite) will be done.

Figure 1 - Fe K-edge XANES spectra of investigated samples.

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