EXPERIMENT REPORT

The role of Fe ions in the formation of foxing spots in archival documents
ME-1294: 9-15 November 2005

Aim of this experiment was to investigate by X-ray Spectroscopy the role of the iron ions in the formation of foxing and how they are linked to the paper. We prepared three series of reference samples on Umbria paper (cotton 34%, cotton linters 66%, glued with starch):

a) samples treated with Fe(II) sulphates aqueous solution
b) samples treated with Fe(III) sulphates aqueous solution
c) samples treated with an equi-molar Fe(II) + Fe(III) sulphates aqueous solution

Each series of samples was artificially aged in two different ways, trying to understand if a different ageing could vary the foxing formation mechanism. One ageing process was carried out at 80°C and 65% RH, the other one at 23°C, 50% RH and under UV irradiation.

Our goal was to investigate the Fe K-edge and pre-edge to obtain information on the Fe oxidation state and local coordination. However, we encountered enormous problems due to radiation damage and we spend a considerable amount of time investigating them. We thus decided to record conventional absorption spectra on a large series of samples trying to find out the best experimental conditions for this experiment and precious information to plan a new experiment.

We observed that also using a fast shutter, the second spectrum collected from the same spot shows big evidences of radiation damage as we can see in fig.1 where the edge changes drastically during irradiation. However, it was interesting to note that there is no evidence of degradation looking at the pre-edge features, and in fact this is used now for our analysis (fig.2). Moreover we observed that the first spectra collected from different point in the same foxing spot and from different foxing spots generated by the same solution are completely reproducible. In this way, we could collect one spectrum from each sample and then make an average to increase the statistics that was not so good in this experiment.

In fig.1 we report the Fe K-edge XANES spectra collected from not aged Umbria paper treated with Fe(II) sulphates aqueous solution, but the same features were observed in each sample investigated.

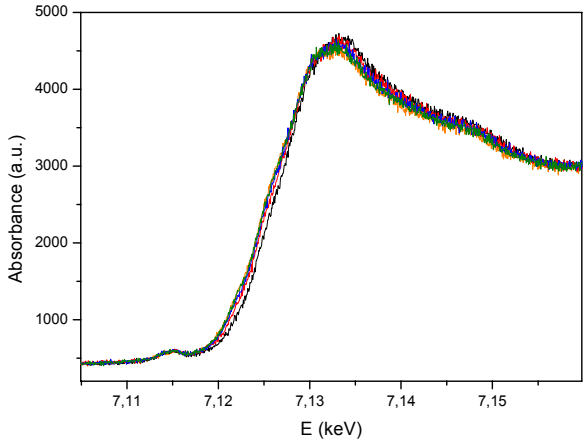

Fig.1

The investigation of the pre-edge is giving us some important information about the different kind of foxing spots that could be used in the next experiment to identify similar structures in historical samples. As an example, in fig.2 we report the Fe K pre-edge spectra collected from foxing spots artificially aged (at 23°C, 50% RH and under UV irradiation for 28 days) obtained by the injection of Fe(II) sulphates aqueous solution (red line) and of an equi-molar Fe(II) + Fe(III) sulphates aqueous solution (black line). In order to investigate the pre-edge features, we are using the method published by Westre et al and Wilke et al.

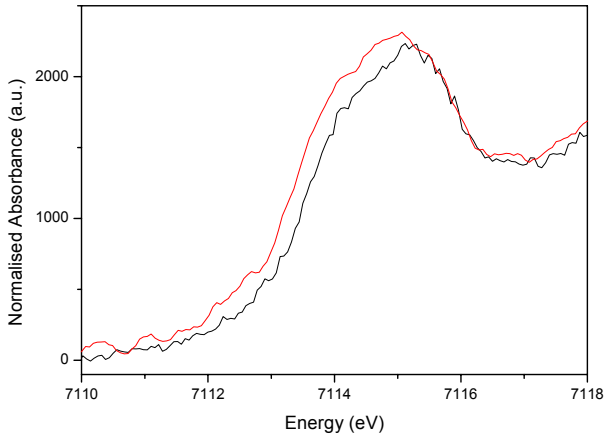

Fig.2

We furthermore recorded Kβ satellite spectra on one sample as a feasibility study to assess the countrates that can be expected.

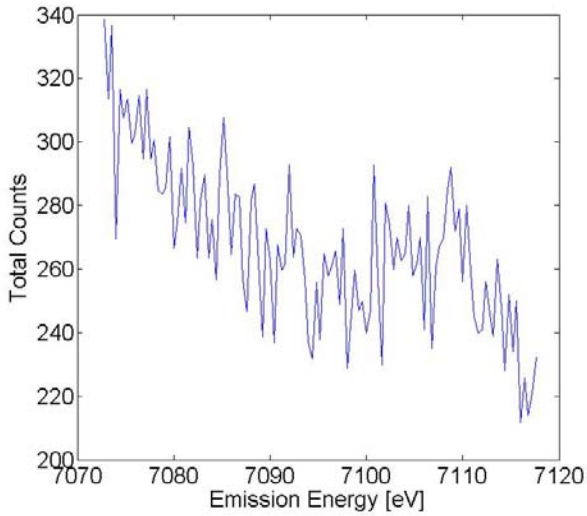

Fig.3

The figure shows the Kbeta satellite emission of the Fe contained in the paper. The total couting time is 64 seconds per data point. The planned new spectrometer on ID26 will gain a factor of 20 by capturing a larger solid angle. It will thus be possible to record the emission lines with reasonable statistics.