

X-ray Fluorescence Spectroscopy of Aqueous Samples

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Synchrotron-based X-ray fluorescence spectroscopy of wet samples has recently shown to have a large variety of applications in chemical, biochemical and life sciences. With the use of thin window compartments containing water or solutions of interest, fluorescence and electron yield spectra are recorded as the energy of incident x-rays is varied. In this presentation, we report some preliminary results acquired from several aqueous samples, i.e. sodium chloride, iron (III) chloride, iron (II)-ethylenediaminetetraacetic acid (EDTA) solutions and laundry blue suspension. The wet sample spectra were also compared to those from their dry forms.

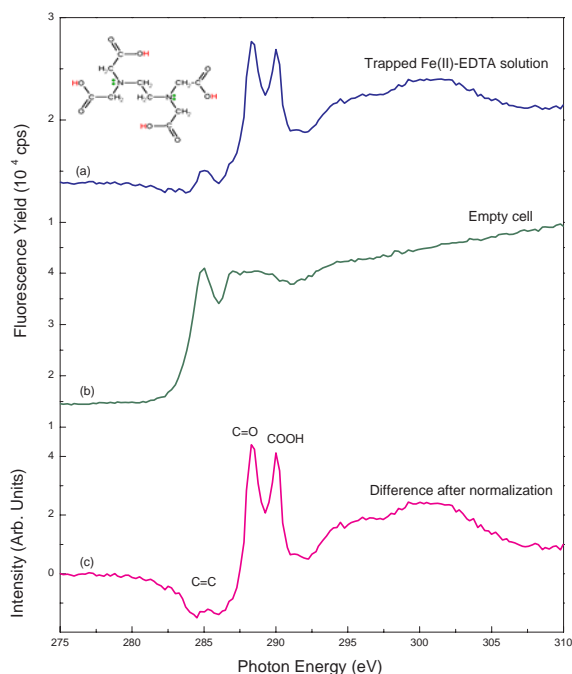


Figure 1: Normalization of fluorescence yield spectra of 1 mol Fe(II)-EDTA solution trapped in a double-sided silicon nitride cell. (a) Spectrum of the trapped solution sample, (b) Spectrum of the empty cell, and (c) Normalized fluorescence intensity.

background from the O in the water. The new cell design and configuration are currently addressed to improve the detection.

References:

- [1] J. Stohr, NEXAFS Spectroscopy, Springer, Berlin, Germany (2003).
- [2] V. Pérez-Dieste et al., Appl. Phys. Lett., **83**, 5053 (2003).

Figure 1 shows one example in which fluorescence spectra were recorded from both 1M aqueous Fe(II)-EDTA solution trapped in a double-sided silicon nitride cell (a) and an empty cell (b). The inset is the molecular structure of EDTA. The wet sample spectrum displays the C K-edge at 285.0 eV and two more much higher peaks at 288.3 eV and 290.0 eV. The empty cell, however, illustrates a larger C K-edge. The bottom curve (c) shows the difference between these two after normalization and scaling before the edge jump^[1]. It is observed that on the normalized curve the C K-edge almost disappears, indicating that C is largely from contamination other than from the solution, as shown with the absence of C=C bonds in the inset. The two peaks at 288.3 eV and 290.0 eV are correlated to the existence of C=O and COOH bonds^[2], although the chelated Fe(II)-EDTA complex causes small binding energy shifts. This is in a good agreement with the bond structure present in the inset.

Under the experimental settings, we were not able to examine the fluorescence spectra at the Fe edge presumably due to the high fluorescent

background from the O in the water.

The new cell design and configuration are currently