Polycapillary Optics for Micro-XRF and Spatially Resolved XAS

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Introduction

In numerous environmental applications of synchrotron-based methods, it is necessary to focus the beam down to a small spot size to probe the heterogeneity of the sample and acquire spatially resolved spectroscopic data. Here we show how a Kumakhov polycapillary lens can be used on a bending magnet source to produce a relatively small spot size (full width at half maximum [FWHM] of ≈100 μm), map the chemical composition of environmental samples, and obtain spot-size x-ray absorption spectra. The primary advantages of this method is that it can be readily deployed at any beamline, that the optic does not need to be realigned after changing the energy of the incoming x-rays, and that good x-ray absorption spectroscopy (XAS) data quality can be achieved. We present x-ray fluoresence (XRF) and XAS data from an arsenic (As) hyperaccumulating plant to illustrate the use of this focusing optic.

Polycapillary collimating optics were first developed in 1986 by M. Kumakhov [1-3]. They can be used to efficiently collect x-rays produced from a sample or to focus an incoming x-ray beam to a small spot. In the latter case, they allow an increase in beam power with a decrease in beam size.

Given the large source collection area of the second- and third-generation x-ray sources, polycapillary optics provide an interesting, easy-to-use approach for focusing the beam, and they also provide a wide energy bandwidth (1-30 keV). The position and size of the focal spot weakly depends on the position and size of the x-ray source. Therefore, the optics can be aligned at one specific energy yet be used to perform multiple experiments at various energies without realigning the beam. During a typical XAS experiment, the beam position does not exceed 2 μm, an insignificant change when compared with the beam spot size. Finally, the compact size of the lens allows it to be easily positioned in the beam, right after the first ionization chamber, for example.

We tested the use of a polycapillary optic lens for performing elemental mapping of a plant that hyperaccumulates As: the Chinese brake fern [4]. The primary objectives were to study the spatial distribution of As within a leaf and to determine changes in the As oxidation state within the sample by x-ray absorption near-edge structure (XANES).

Methods and Materials

Experiments were performed at the DND-CAT (sector 5) bending magnet beamline at the APS. A Si(1,1,1) piezo-driven double-crystal monochromator was used to set the energy of the beam. The sample was mounted on an X-Y stage (Newport) that was controlled through spec software. A polycapillary lens (X-ray Optical Systems, Inc.) was positioned after an Oxford ion chamber used to monitor the incident beam energy (Fig. 1). The sample was positioned on the stage at a location 5 cm from the end of the polycapillary optic. A four-element solid-state detector (Canberra) was used to monitor the As fluorescence from the sample.

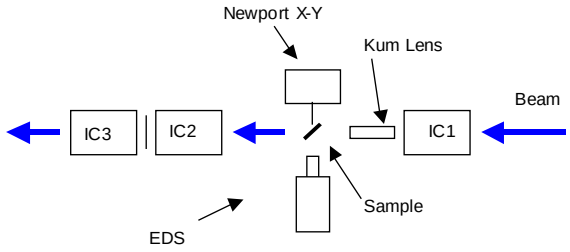

FIG. 1. Schematic representation of the instrumental arrangement. IC = ion chamber, EDS = solid-state detector, Kum Lens = polycapillary optic lens.

Maps were produced by raster scanning the sample on a regular grid mesh. In order to determine concentration maps, the fluorescence signal was calibrated by following the procedure given by Pickering et al. [5]. After the sample was mapped, several spectra were collected at various locations within the specimen to study the oxidation state of As.

A leaf of the Chinese brake fern, *Pteris vittata*, has the ability to tolerate high concentrations of As in soil (up to 1,500 mg/kg). The As concentration in the fronds can reach up to 20 g/kg, whereas the As concentration in the roots is generally low (<300 mg/kg). A recent XAS study [6] showed that As is primarily present as As(III) and that it is likely stored in vacuoles. When As is at high concentrations in the plant, one can detect a significant ligation of As by sulfur.

Results and Discussion

The XRF mapping results are presented in Fig. 2, and scans in the XANES region are shown in Fig. 3. The mapping of the fern shows that, after normalization due to thickness effects, the distribution of As within the plant is pretty uniform at the spatial resolution permitted by the lens.

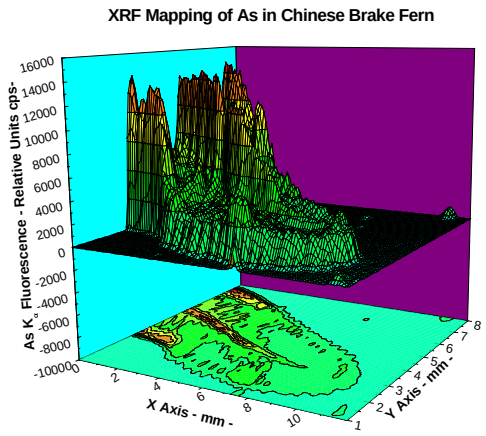

FIG. 2. The distribution of As within a frond of the Chinese brake fern obtained from the raw XRF fluorescence data. The higher concentration shown in the frond central nervure is related to thickness effects.

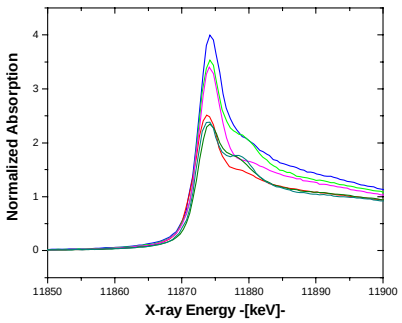

FIG. 3. XANES spectra collected at various locations within the frond show that As is present in different coordinations and valence states.

The analyses of XANES spectra that were collected at various locations within the frond are consistent with the bulk XAFS analyses performed previously [6]. The As is primarily present as As(III) in the leaf tissue, but along the frond nervure a significant fraction of As(V) is present, as exemplified by a shift in the As edge energy.

Acknowledgments

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