



	Experiment title: Spatial distribution of Cr(VI) across soil grains in remediated and unremediated Cr contaminated soils.	Experiment number: ME-52
Beamline: Id21	Date of experiment: from: 3/5/00 to: 8/5/00	Date of report: 10/5/00
Shifts: 12 (4 days)	Local contact(s): Ray Barrett	<i>Received at ESRF:</i>

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Report:

This experiment followed from the pilot study (September 1998). There have been many improvements to the beamline since, notably quicker scanning times. This has resulted in a vast improvement in the quality and quantity of data collected. The possibility of photo-alteration of Cr(VI) to Cr(III) and vice versa in the focussed beam was investigated by repeatedly analysing the same spot in Cr(III) and Cr(VI) standards (Cr_2O_3 and $\text{K}_2\text{Cr}_2\text{O}_7$, 5 wt. % in LiCO_3 , respectively). Consecutive spectra for each standard were identical and we therefore conclude that there is no photo-alteration in these type of compounds.

The Cr oxidation state across grains in four samples of chromium ore processing residue were analysed. Samples had been previously characterised by electron microprobe analysis (EMPA) and x-ray diffraction analysis (XRDA). Specific areas of interest were located using scanning x-ray microscopy images in both transmission and fluorescence modes.

Three major types of grains were found:

- 1) **Hydrogarnet and hydrocalumite.** EMPA/XRD had implied these Cr(VI) phases were present as small (approx. 5-10 μm diameter) grains. We were able to confirm these grains

as Cr(VI)-rich.

- 2) **Chromite ore.** We were able to confirm that grains indicated by EMPA/XRD to be the original chromite ore were Cr(III)-rich. These grains were often found with small Cr(VI)-rich grains surrounding them, presumably as a result of the ore processing.
- 3) **Mixed Cr(VI)/Cr(III) grains.** The presence of grains containing mixed Cr oxidation states had not been previously indicated by EMPA/XRD. A large mixed grain was found and spectra taken every 4 μm down the grain (Fig. 1). This type of grain is of great interest. The relatively thick coating of Cr(VI) (20-30 μm) may have implications for the efficiency of the remediation techniques which are designed to reduce Cr(VI) to Cr(III).

In conclusion, scanning x-ray microscopy has been shown to be an extremely useful tool for examining Cr oxidation states across soil grains and, in conjunction with EMPA and XRD, has provided unique information on the speciation of Cr in these soils. Because of time constraints, it was not possible to analyse remediated soils on this occasion but we now hope to analyse these in the near future. In addition, through bulk XAS analyses of mixed Cr(VI)/Cr(III) phases, we intend to quantify the relative proportion of Cr(VI) present in these grains.

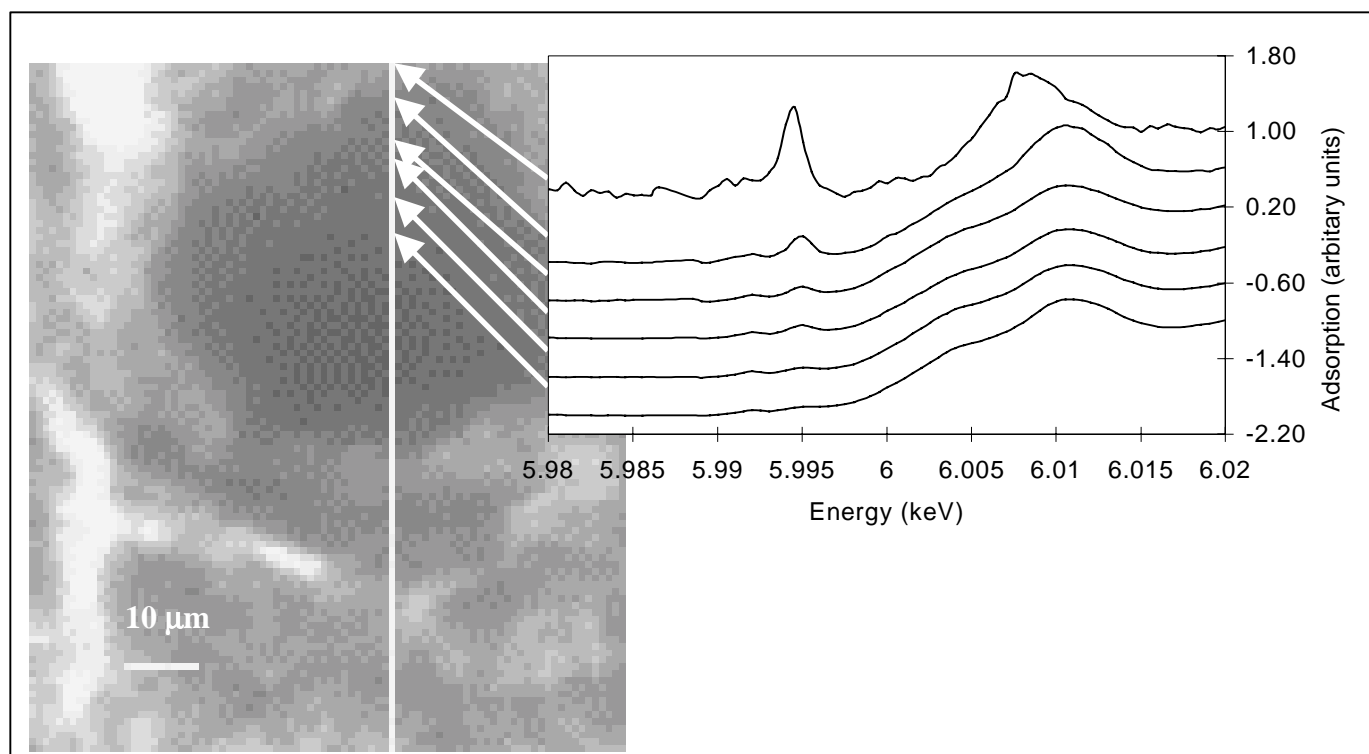


Fig. 1 Mixed Cr(VI)/Cr(III) grain from the chromium ore processing residue. Picture is a transmission x-ray adsorption pixel map showing the Cr-rich grain adsorbing strongly. Selected spectra from the linescan (indicated as a white line) show a zone of Cr(VI) surrounding the inner core of Cr(III). The presence of Cr(VI) is indicated by a strong pre-edge peak at 5.995 keV.