



Mercury speciation in polluted soils by combined spectroscopic and microspectroscopic techniques employing synchrotron generated X-rays.

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Soil samples collected inside and outside an industrial polluted area of national environmental interest located in “Val Basento” (Basilicata, Italy) have been studied by a combination of different advanced analytical techniques exploiting high intensity synchrotron generated X-rays. Mercury (Hg) concentration in the soil ranged from 1 up to 250 $\mu\text{g/g}$.

Bulk EXAFS (Extended X-ray Absorption Fine Structure spectroscopy) and XANES (X-ray Absorption Near Edge Structure spectroscopy) were performed on soil samples sieved at 2 mm and on the clay fraction ($<2 \mu\text{m}$), where the highest amount of Hg was concentrated.

In addition to bulk XAS investigations, microanalyses on soil thin sections by combined $\mu\text{-XRF}/\mu\text{-XRD}$ (micro X-ray fluorescence/micro X-ray diffraction) and $\mu\text{-XANES}$, with a resolution from 10 to 20 μm , were also performed. $\mu\text{-XRF}$ maps were collected in order to localise microscopic Hg containing particles in areas of ca. 1 mm^2 . Simultaneous to $\mu\text{-XRF}$ spectra, microdiffraction patterns were collected in each point of the maps, in order to identify any correlation between Hg and its mineralogical forms. Once points of interest were localised, $\mu\text{-XANES}$ spectra were collected and compared with the spectra of known Hg standard compounds.

The combination of bulk XAS and microbeam measurements not only provide a detailed Hg speciation at the microscale level, enables better interpretation of the higher length bulk scale XAS spectra in the light of the more easily manageable information obtained from microanalyses.

From this combined synchrotron X-ray spectroscopic investigation, we find that Hg was mainly speciated as HgS (cinnabar) and Hg(0) in the samples collected inside the industrial site and as Hg₂Cl₂, HgCl₂ and Hg bound to organic matter in the samples collected in the areas surrounding the industrial site. Quantification of the percentage of the various Hg forms in the soil samples was also obtained.

This type of pollution is typical of industrial areas where chlor-alkali plants have been operating in the past. The detailed Hg speciation will allow for a better assessment of the environmental and human risk connected to Hg contamination of these soils, as well as to develop more effective remediation strategies for an environmental restoration of the area.