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Trace elements in contaminated soils and sediments: microscopic chemical and mineralogical characterization of particles containing trace metals

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The toxicity and mobility of inorganic pollutants heavily depend on their chemical forms and binding states: the recognition of the phases in which trace elements occur in contaminated soils and sediments can be useful to evaluate their hazardousness.

Sequential extraction procedures are commonly used to determine the solid-phase forms of metals in soils and sediments. These methods have been always extensively criticised for various reasons: lack of selectivity of reagents, partial dissolution of some phases, pH changes, reabsorption and/or redistribution of some elements, influence of the operating conditions (for example, extraction times, solid/liquid ratios, type of shacking procedure, methods of solid/liquid separation, washing, amount of sample analysed, etc.). In particular micro-particles seem to be susceptible to random results and further analyses without any chemical treatment of the sample appear helpful.

Combined analyses using EDX semi-quantitative chemical microanalysis and X-ray micro-diffraction have been carried out in this work to determine microscopic mineralogical phases containing trace elements.

A soil sample collected at depth of 40-50 cm in an industrial area located in the South of Italy was studied. This sample contains significant amounts of Cd, Cr, Cu, Hg, Ni, V, Zn. The fraction <2 mm was separated by sieving and settled on an aluminium stub. Also soil thin sections were prepared. Microscopic particles of special relevance containing trace elements were observed and analysed by using Field Emission Scanning Electron Microscopy and EDX semi-quantitative chemical microanalysis; Feature software was used to facilitate and automate the localisation of particles containing high amounts of high Z elements (by selecting an appropriate intensity for the backscattered electrons images). The collection of images at different magnifications has allowed to find back these particles at the optical microscope mounted on a X-ray micro-diffractometer; X-ray collections were carried out at different time ranges and positions of the sample, and using collimators with different beam diameter (10-50 μm).

Therefore semi-quantitative chemical analyses and X-ray data collections on the same microscopic particles was successfully carried out by using laboratory instruments and allowed to identify in soil the mineral phases bearing the highest amounts of trace elements.