

# **METALS PARTITIONING IN SOILS BY SCANNING ELECTRON MICROSCOPY (SEM-EDX) MAPPING OF THE ELEMENTS DISTRIBUTION: APPLICATIONS FOR GEOCHEMICAL EXPLORATION AND ENVIRONMENTAL SCIENCES**

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## **Introduction**

Metals dispersion mechanisms in soils are the subject of abundant researches, both in environmental sciences and mineral exploration. From the environmental standpoint, the leaching or binding of metals from anthropogenic sources in contaminated soil is studied under specific, narrow and well documented conditions. Conversely, the mineral exploration industry is interested in the natural dispersion of these metals on a large scale in order to identify potential mineral deposits, typically occurring under poorly documented conditions. Soils in northern climates are composed of various components that may play a role in metals binding: humic substances, clays, ferruginous or calcium crusts, charcoal, residual minerals, atmospheric dust and meteoritic spherules, anthropogenic contamination, etc.

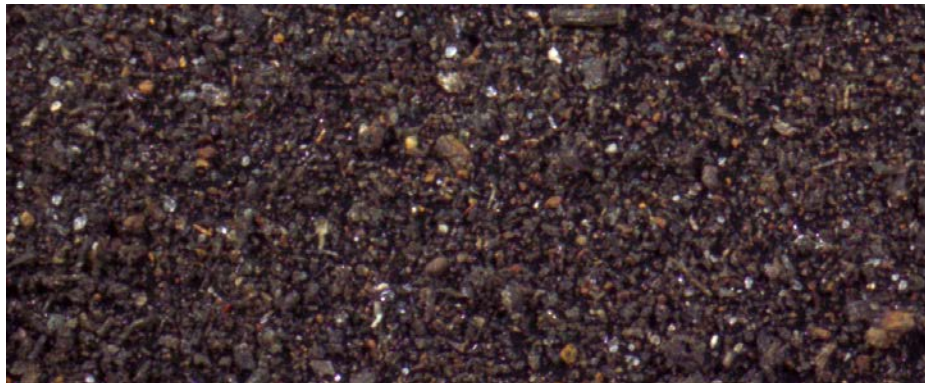
For mineral exploration purposes, single extractions, or partial leaching, are commonly used to find out the content of an element bounded to a specific soil component (exchangeable, carbonate, oxides, organic matter, residual, etc.). But the chemical reagents used are rarely completely specific. There is a potential of the redistribution of the elements (Belzile *et al.*, 1989). Incomplete extraction and formation of artifacts can also happen (Baize, 2000, LaSalle & de Kimpe, 1985 and Romagera *et al.*, 2008). The soil heterogeneity causes variability between similar samples. Sampling campaign results show high variability even between two samples collected very closed in the same horizon and type of soil (Gaudreault & Fournier, 2013). In practice, the information collected on the partitioning of metals, or binding sites, are not always conclusive.

## Methodology

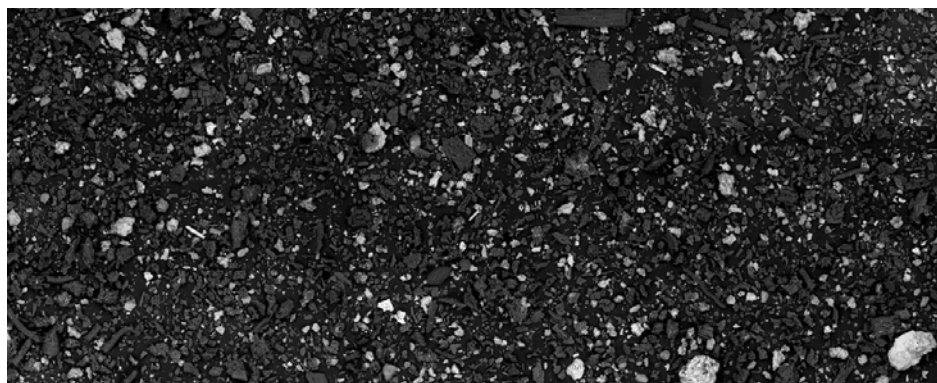
The current objective was to determine the metal's binding sites in anomalous soils at the microscopic scale using non destructive analytical methods.

The experiment was carried out on a collection of humic soils from sites known to show various natural metal enrichments, after a partial leaching analysis (Na-Pyro-ICP-MS). Most physicochemical parameters (pH, Eh, CEC, TDS, *etc.*) were available. Among the chosen samples, ten were significantly anomalous in manganese, three in copper and three in zinc. Other metals were also anomalous in certain soil, but were not directly studied. Samples with anthropogenic contamination were also studied.

In the first place, soils were observed and photographed using a binocular stereomicroscope Leica M205C and a camera Pixelink PL-B686CU. This has facilitated the identification of constituents in soils. Each sample was then subjected to scanning electron microscope analysis (Zeiss EVO MA15) with a backscattered electron detector (BSE) and energy dispersive spectrometers (EDX-SDD). The figure 1 and 2 show an example of the comparison between the binocular and the BSE image.

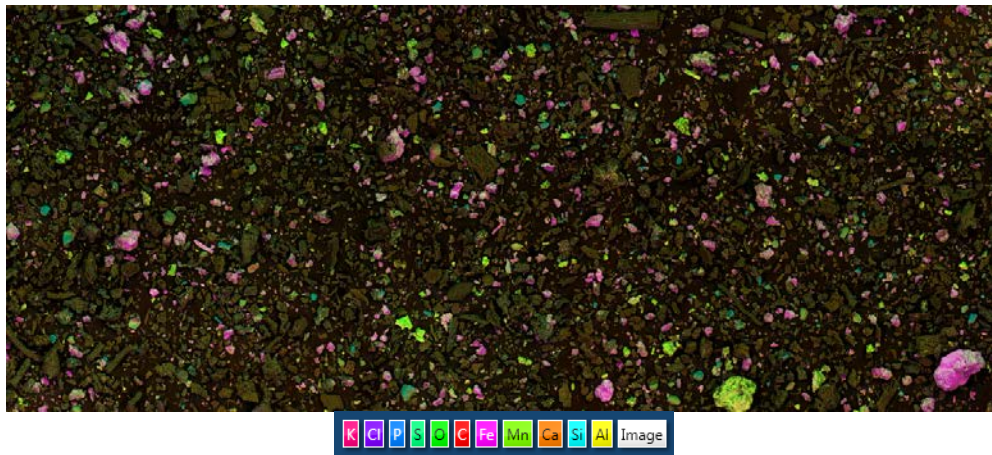


**Figure 1. Binocular image of a soil sample mounted on a carbon tape.**



**Figure 2. Backscattered electrons image of a soil sample.**

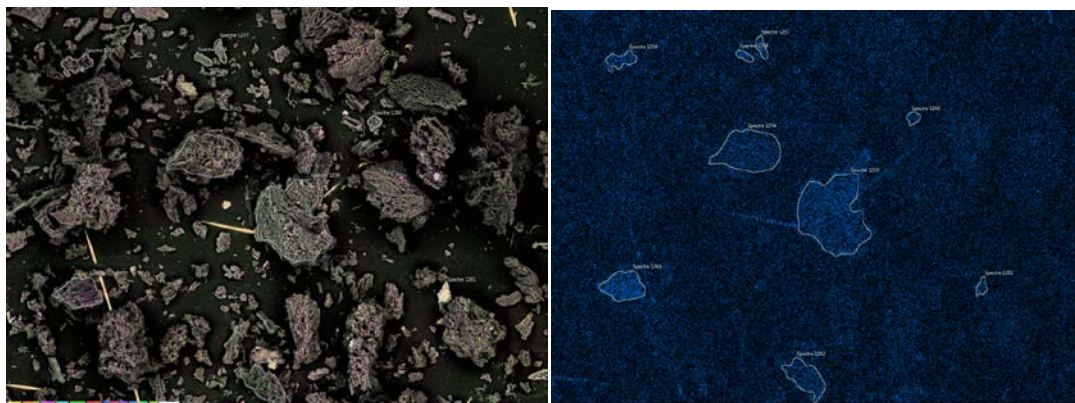
The Aztec Large Area Mapping software was used to map the chemical elements distribution covering the surface of each sample. The acquisition was configured to divide the analysis area into several fields of view, in which a chemical spectrum measuring every element (excepted H, He and Li) was acquired for each pixel. All elementary maps were then superimposed on the backscattered electrons image, allowing to quickly locate the different metals, even if they were in trace amounts, with a resolution of a few microns. High magnification imaging and microanalyses were performed to characterize the nature of the chemical signal (organic matter, oxides, clays, residual minerals, etc.).



**Figure 3. EDX-SDD superposition image of a soil sample.**

## Results

Results show, for example, that copper in humic soil dominantly precipitates on organic matter up to a saturation point, where it start precipitation as distinct authigenic sulfides, and in a lesser extent to accumulates in iron and manganese oxides. Other soil samples showed manganese accumulation as oxides, sometime associated with iron oxides. Each metal has its own distinctive behavior.



**Figure 4. EDX-SDD superposition image and elemental map of copper showing accumulation sites.**

Contaminated soils showed a lot of variability in their chemical composition. For example, a sample of soil from a shooting range showed accumulation of copper, lead, antimony and zinc. Those metals, which come from bullet fragments, were mainly fixed to small particles, 15  $\mu\text{m}$  or less, like droplets on the surface of sand grains. From the environmental point of view, those chemical interactions between metals and mineral grains will probably need a different decontamination process than if they were bounded to organic matter.

## Conclusions

For the mineral exploration industry, knowledge of the metals partitioning would determine their dispersion mode and improve the deconvolution of the signal in order to locate their source. This soil characterization technique may help to discriminate true from false anomaly. In regard of environmental sciences, a better comprehension of the metals binding sites will help to anticipate the risk associated with the contamination (mobility, toxicity, bioavailability, etc.).

## References

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