

**Title**

Microscopic and Spectroscopic Speciation of Ni in Soils in the Vicinity of a Ni Refinery.

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**abstract**

Accurately predicting the fate and bioavailability of metals in smelter-contaminated soils is dependent on a careful determination of metal speciation. Traditional speciation approaches have relied on less-direct methods including sequential extraction, a technique which may potentially introduce artifacts. In this study, Ni speciation in soils in the vicinity of a former Ni refining plant was directly determined using X-ray absorption fine structure (XAFS) spectroscopy and micro-focused synchrotron-based X-ray fluorescence (micro-SXRF) spectroscopy. After bulk phase characterization on soils having a range of pH values (5.3 – 8.1), organic matter contents (5 – 35%), and Ni concentrations (600 – 11,000 mg/kg), elemental maps were collected using micro-SXRF. Results indicated Ni mostly occurred in concentrated hot spots with little correlation to other elements. When Ni was more diffusely distributed in the samples, it was generally associated with Fe. These findings were used in fitting the bulk XAFS data. XAFS revealed Ni was mostly present as Ni oxide and Ni bound to Fe, with some minor Ni phases present as well. Quantification of Ni species in these soils will be presented using a principle component analysis approach.

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