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Research article

Leaching potential of geogenic nickel in serpentine soils from Taiwan and Austria

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ABSTRACT

Serpentine soils may be natural sources of metal leaching and pollution. In this study, two contrasting serpentine soils from Taiwan and Austria were selected to evaluate the leaching potential of geogenic nickel (Ni). We applied selective sequential extractions and dissolution kinetics with three inorganic acids (HCl, HNO₃, and H₂SO₄) and three organic acids (citric, acetic, and oxalic acids) in concentrations ranging from 0.05 to 10 mM to determine the release rate of Ni in the soils with respect to pH and acid types. Chlorite and serpentine were the major Ni-bearing minerals in the studied soils. Ni was dominantly bound in unavailable forms within these silicate minerals, but smaller fractions of acid-soluble, Fe–Mn oxide-bound, and organically bound Ni represented more labile Ni sources in the soils. The release rate of Ni from the soils increased with decreasing pH in all acids. However, the organic acids caused stronger pH dependences than the inorganic acids, likely because of ligand-promoted dissolution. The maximum total rate of Ni dissolution occurred with citric acid in both soils. The dissolution of Ni strongly increased when the ionic strength in the background solutions increased. We observed marked differences in dissolution rates and ligand effects between the Austrian and Taiwanese soils, which reflect differences in labile Ni pools, especially in the organically bound fraction. Our results demonstrate that labile fractions control the leaching potential of Ni in serpentine soils and that Ni associated with soil organic matter may contribute to leaching at moderately acidic pH levels.

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1. Introduction

Soil contamination by heavy metals is of great public concern, because of their long persistence in the environment and their toxicity for humans and other organisms (Kabata-Pendias, 2011). Soils can become contaminated with heavy metals from various anthropogenic sources including mining, smelting, wastewater discharge, pesticides, fertilizers, sewage sludge application and others (Adriano, 1986). In addition, geogenic sources, such as serpentinites, have been reported to cause high concentrations of certain heavy metals in soils (Becquer et al., 2006; Cheng et al., 2011; Hseu and Iizuka, 2013). Serpentine soils can be formed from ultramafic rocks alone and also from hydrothermally altered ultramafic minerals in the presence of serpentine minerals

(McGahan et al., 2008). Serpentine terrains are abundant worldwide in ophiolite belts along tectonic plate margins. Moreover, ophiolites are sections of the oceanic crust and the subjacent upper mantle that have been uplifted or emplaced and exposed within continental crustal rocks (O'Hanley, 1996).

In comparison to other rocks, ultramafics are richer in Ni with concentrations up to 3600 mg kg⁻¹ (Kabata-Pendias, 2011). In serpentine soils, Ni concentrations of 10,000 mg kg⁻¹ have been reported (Oze et al., 2004), while the global average concentration of Ni in soils is only about 34 mg kg⁻¹ (McGrath, 1995). Ni is substituted for Mg²⁺ in the silicate structures of minerals in serpentine soils (Becquer et al., 2006), and as chemical weathering progresses, these minerals destabilize and release Ni. This results in more bioavailable and leachable Ni fractions and provides a source of non-anthropogenic metal contamination in serpentine landscapes (Hseu et al., 2015a).

Several studies on the mobility of Ni are available for serpentine soils using single or sequential extractions. Cheng et al. (2011)

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demonstrated by sequential extraction that the largest amount of Ni was partitioned in the silicate framework, but Ni was gradually transformed into labile phases in serpentine soils. Ho et al. (2013) determined the validity of using the diethylenetriamine pentaacetate (DTPA)-extractable concentration as a phytoavailability index of Ni that can be used to estimate Ni uptake by plants growing in serpentine soils. In serpentine soil profiles, the concentrations of Ni extracted using DTPA and 0.1 N HCl increased toward the soil surface, indicating that Ni availability is higher in more weathered surface soils than in less weathered subsoils (Hseu, 2006; Hseu et al., 2015b). Pedogenic Fe and Mn oxides are regarded as crucial Ni sinks in serpentine soils (Becquer et al., 2006; Hseu, 2006; Cheng et al., 2011), and, thus, the roles of Fe and Mn oxides in Ni retention have been assessed by conducting selective extractions (Hseu et al., 2015b). Also, the anaerobic conditions of flooded soils derived from serpentines cause the reductive dissolution of Fe and Mn oxides and release of adsorbed Ni, causing Ni to be mobilized in the soil pore water (Antić-Mladenović et al., 2011; Rinklebe et al., 2016).

The weathering of serpentine minerals results in the release of Ni from serpentine soils into the environment and is therefore crucial for ecological functioning, agricultural production, and human health in a serpentine ecosystem. The dissolution of heavy metals from rocks, minerals, and soils through chemical weathering is regulated by factors including solution pH, ionic strength, and the type and concentration of inorganic and organic acids (Tack et al., 1999). In the dissolution kinetics of minerals, two of the most important chemical weathering reactions in soil environments are proton-promoted (pH effect) and ligand-promoted dissolution reactions (Hamer et al., 2003). The amount and composition of inorganic and organic acids in soil solutions can vary over a wide range, depending on variables of climate, vegetation, and land use. In the course of soil development, different processes lead to input/formation of protons and ligands from acid precipitation and low-molecular-weight organic acids from humus and root exudates (Ramos et al., 2014). Using inorganic and organic acids to study weathering reactions under laboratory conditions, Rajapaksha et al. (2012) demonstrated that Ni release from serpentine soil was accelerated by complex-forming ligands and that both protons and ligands corroborated to accelerate the release of Ni from the soil.

An improved understanding of the factors and processes that control Ni solubility in serpentine soils is needed, particularly in view of appropriate risk assessment and management strategies to reduce Ni mobilization, bioavailability, and transfer to the surroundings. By coupling sequential extractions with chemical kinetic interpretations, it is possible to gain greater insight with respect to where and how Ni is bound in the soil. The objectives of this study were therefore: (1) to partition Ni fractions in contrasting serpentine soils developed under different pedoclimatic conditions in Taiwan and Austria and (2) to determine the release rate and reaction kinetics of Ni dissolution in the soils with respect to pH and acid types with and without organic ligands.

2. Materials and methods

2.1. Soil collection and characterization

Surface soil samples (0–10 cm) were collected from two contrasting serpentine sites in Taiwan (TW) and Austria (AT). The TW site (23°02'48"N, 121°12'24"E) is located in Eastern Taiwan at an altitude of approximately 410 m, with a mean annual rainfall of 1800 mm and a mean annual air temperature of 22.5 °C. The parent materials of the TW site are exotic blocks of ophiolite from the Philippine Sea plate. The vegetation is dominated by grass species (especially *Miscanthus sinensis*), with few tropical trees. The AT site (47°04'14"N, 12°42'04"E) is located in the Grossglockner area in the

Austrian Alps at an elevation of approximately 2200 m. The annual precipitation in the area lies between 1500 and 2000 mm and the mean annual temperature is <1.5 °C. The AT site is close to the tree line with vegetation dominated by larch trees (*Larix decidua*), mosses and few grasses. The two soils were classified as Regosols in the WRB classification system (IUSS Working Group WRB, 2006) and as Lithic Udorthents in the US classification system (Soil Survey Staff, 2014). The soil samples were air-dried, ground, and passed through a 2-mm sieve for subsequent laboratory analyses.

Soil particle size distribution was determined via the pipette method (Gee and Bauder, 1986). The specific surface area of the soils was measured with a SA 3100 Analyzer (Beckman Coulter, USA) based on nitrogen adsorption at 77 K. Soil pH was measured in a mixture of soil and deionized water (1:1, w/v) using a glass electrode (McLean, 1982). Total organic carbon (OC) content was determined using the Walkley-Black wet oxidation method (Nelson and Sommers, 1982). Cation exchange capacity (CEC) was determined via the ammonium acetate method (pH 7.0) (Rhoades, 1982). Additionally, analysis of major and trace elements in the soil samples was conducted after digestion of soil with concentrated HNO₃ and HCl (1:3, v/v) (*aqua regia*). The filtered suspension was then measured with a flame atomic absorption spectrophotometer (FAAS) (Hitachi Z-2300, Tokyo, Japan), following the procedure recommended by the International Organization for Standardization (ISO, 1995). To identify the mineral composition of the studied soils, X-ray diffraction (XRD) patterns of whole fine-earth fractions were obtained from 0° to 50° 2 θ , at a rate of 0.2° 2 θ min⁻¹ (Rigaku D/max-2200/PC diffractometer; Ni-filtered Cu K α radiation generated at 30 kV and 10 mA).

2.2. Sequential Ni extraction

Sequential selective extraction (SSE) techniques are commonly utilized to evaluate metal fractionation in soil solid phases. A typical SSE procedure employs progressively stronger solvents to sequentially solubilize various metal fractions from water or weak-acid soluble form to mineral structure form. The three-step SSE procedure used in this study is described in the Standards, Measurements and Testing Program (formerly, European Community Bureau of Reference; therefore, the procedure is known as the BCR method). A detailed description of the BCR-protocol is given in Quevauviller et al. (1994). This protocol specifies the sequential use of dilute acetic acid, followed by a reducing agent and an oxidizing agent. Here, an additional step was included using *aqua regia* to dissolve Ni from the residue remaining after the three extraction steps. Hence, during the four steps of the employed SSE procedure, Ni was divided into the following fractions: (1) acid extractable (F1), (2) reducible (F2), (3) oxidizable (F3), and (4) residual (F4). Except for the residual fraction, the other fractions (F1–F3) are recognized as potentially labile pools of heavy metals in soils (Cheng et al., 2011). Each of the chemical fractions is operationally defined as follows:

- (1) F1: 1 g soil (dry wt.) was extracted using 20 mL of 0.11 M HOAc (acetic acid) in Teflon centrifuge tubes with a 16 h shaking period at 25 °C.
- (2) F2: Residue from F1 was extracted with 20 mL of pH 1.5, 0.5 M NH₂OH·HCl (hydroxylamine hydrochloride) with a 16 h shaking period at 25 °C.
- (3) F3: Residue from F2 was digested with 5 mL of pH 2, 8.8 M H₂O₂ (hydrogen peroxide) for 1 h at 25 °C (vessel covered) and then extracted with H₂O₂ for 1 h at 85 °C with occasional shaking to reduce the volume to < 3 mL (uncovered vessel); an additional 5 mL of H₂O₂ for 1 h at 85 °C with occasional agitation to reduce the volume to < 1 mL.

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