

Effect of wetting and drying processes on ultramafic and mafic tailing minerals amended with topsoil

Lewis Fausak^{A,*} , Anne Joseph^A , Ana C. Reinesch^A , Skylar Kylstra^A , Fernanda Diaz Osorio^A , Autumn Watkinson^A  and Les Lavkulich^A 

Environmental context. Mine tailings are a mixture of fine materials obtained after crushing, processing and extracting the valuable minerals from ore. Ultramafic and mafic mine tailings have the potential to mineralise carbon, offering a solution to offset greenhouse gas emissions from the mining sector. The study revealed that the effects of wetting and drying ultramafic and mafic mine tailings under atmospheric conditions have the potential for carbon sequestration and acid mine drainage.

For full list of author affiliations and declarations see end of paper

***Correspondence to:**

Lewis Fausak
Faculty of Land and Food Systems,
University of British Columbia, Office 112A,
H.R. MacMillan Building, 2357 Main Mall,
Vancouver, BC, V6T 1Z4, Canada
Email: lewis.fausak@ubc.ca

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ABSTRACT

Rationale. As the result of their carbon mineralisation potential, there is an increasing interest in using ultramafic and mafic (U+M) mine tailings as a feedstock for carbon (C) sequestration. However, little is known about the relative chemical stability of U+M minerals, and it is unclear whether acid mine drainage may be generated during weathering. **Methodology.** This study determined the relative stability of the constituent minerals of several U+M tailings from mines in British Columbia, Canada, and Australia using selective chemical extractions and an 18-week laboratory experiment of cycles of wetting and drying to simulate conditions that may be experienced under field conditions. Tailings were mixed with topsoil to investigate the potential use as a soil amendment. **Results.** Initially, the tailing sample's pH was 8.4–9.7 and decreased to 7.7–9.1 over the 18 weeks. Soil additions lowered the initial pH and converged with the tailing's pH over 18 weeks. Sequentially weathered minerals determined by X-ray diffraction were consistent with the empirical Goldich weathering sequence. Metal concentrations from inductively coupled plasma–mass spectrometry supported X-ray diffraction results, confirming the stability of the U+M tailings using different methods and the need for future studies on potential metal contamination. **Discussion.** Minor concentrations of sulfur seemed to have resulted in larger fluctuations in pH when low amounts of carbonates were present. However, reactive oxides and basic cations in the U+M tailings maintained pH above 7. Therefore, C sequestration was likely supported, although there was a slight reduction in total C content for almost all samples. This study suggests future research is required under field conditions to confirm C sequestration and to investigate the use of U+M tailings for restoration applications.

Keywords: carbon sequestration, CO₂ mineralisation, hydroxide minerals, mafic tailings, magnesium silicates, soil amendments, ultramafic tailings, weathering.

Introduction

Approximately 419×10^6 Mg of ultramafic and mafic (U + M) mine tailings are produced yearly from the global production of metals and minerals (Power *et al.* 2013). Mine tailings are the mixture of fine materials obtained after crushing, processing and extracting an ore's valuable minerals (Power *et al.* 2020). U + M tailings can support climate change mitigation with a long-term and stable form of carbon (C) sequestration obtained through a process called carbon mineralisation, due to their high magnesium and iron content, whereby carbon dioxide (CO₂) reacts with the divalent metal-bearing oxides groups in U + M tailings (magnesium silicates and hydroxide minerals) to produce carbonate minerals (Power *et al.* 2020). However, little is known about the relative chemical stability of the U + M minerals, especially in relation to acid mine drainage (AMD), over the long term (Baumeister 2012; Power *et al.* 2020; Lu *et al.* 2022).

Silicate minerals found in U + M tailings include olivine (forsterite-fayalite solid solution, $(\text{Mg, Fe})_2\text{SiO}_4$), pyroxenes including clinopyroxene and orthopyroxene (diopside, $\text{CaMgSi}_2\text{O}_6$; and enstatite, $(\text{Mg, Fe}^{2+})(\text{SiO}_3)_2$), serpentine ($\text{Mg}_3(\text{Si}_2\text{O}_5)(\text{OH})_4$), chromite (FeCr_2O_4), amphiboles (such as anthophyllite, $(\text{Mg, Fe}^{2+})_7(\text{Si}_8\text{O}_{22})(\text{OH, F})_2$; and tremolite, $\text{Ca}_2(\text{Mg, Fe}^{2+})_5(\text{Si}_8\text{O}_{22})(\text{OH, F})_2$), garnet including pyrope ($\text{Mg}_3\text{Al}_2\text{Si}_3\text{O}_{12}$) and grossular ($\text{Ca}_3\text{Al}_2\text{Si}_3\text{O}_{12}$), and spinel (MgAl_2O_4). Chakravarthy *et al.* (2020) and Power *et al.* (2020) have documented that these minerals and their chemical composition influence the environment in which they are found, and their importance for climate change mitigation. U + M minerals were formed under conditions very different to those at the Earth's surface, where they encounter oxidation and hydrolysis reactions. Thus, they are chemically unstable when exposed to a surficial oxidising environment and are subject to chemical weathering. Although U + M tailings are potential feedstock to sequester C, the weathering process may lead to AMD, once the basic cations become exhausted to neutralise the acid-generating minerals. AMD can lead to metal leaching causing adverse environmental impacts including contamination of groundwater, surface water and soil (Saria *et al.* 2006).

The classical weathering sequence proposed by Goldich (1938) describes the relative stability of the dominant mafic minerals from least to more stable as olivine, pyroxene, hornblende and biotite. As weathering of minerals occurs, U + M tailing minerals contribute to metal leaching and release of heavy metals which are present in accessory minerals or included as trace metals within silicate minerals, including iron (Fe), cadmium (Cd), arsenic (As), chromium (Cr), thallium (Th) and lead (Pb), as well as other potentially harmful elements including aluminium (Al), cobalt (Co), nickel (Ni) and vanadium (V) (Saria *et al.* 2006; Evans 2013; Malli *et al.* 2015).

Weathering of sulfide containing minerals, notably pyrite, is the primary source of acid generation responsible for AMD (Hayes *et al.* 2014). Although acid-producing minerals such as pyrite are not abundant in U + M ore bodies, they can occur in small amounts and potentially contribute to acid generation in the long term (Keays 1987). AMD results from the oxidation of sulfides in the presence of water and oxygen and is accelerated by microorganisms to release sulfate (SO_4^{2-}), metals (Fe^{2+}) and protons (H^+) into solution (Schippers *et al.* 2010). Additionally, U + M minerals, when subjected to prolonged saturation with water, can become acidic and dissolve iron (Fe), resulting in Fe sulfide oxidation (Center for Science in Public Participation 2014; Kefeni *et al.* 2017). These low pH and metal enriched waters can negatively affect the quality of the ecosystem and aquatic life, largely affecting nearby rivers, lakes, estuaries and coastal waters (Lottermoser 2010). Once initiated, AMD and metal leaching from tailings can continue long after the cessation of mining activities, posing a long-term liability to mining operations (Malli *et al.* 2015). However, simulations of rate reaction calculations show that silicate minerals have

some level of neutralising capacity, which can aid with AMD (Eary and Williamson 2006).

Acid neutralisation also occurs if basic cations are released during AMD and may mitigate the acidification process. However, the rate at which acid or basic cations are released may not be congruent and commonly results in an acidic condition (Cruz-Hernández *et al.* 2019). These processes have been studied by sequential dissolution techniques as relative measures of the release of basic elements, such as calcium (Ca) and sodium (Na), that promote acid neutralisation (Davidson *et al.* 2004; Saria *et al.* 2006; Kefeni *et al.* 2017). Additionally, soils can be added as an amendment to mine tailings to dilute metal concentrations, inoculate tailings with microbes, or alter pH (Dvořáčková *et al.* 2022). However, this amendment has not been thoroughly investigated for use in mitigating AMD of U + M tailings. Soils with high clay or organic matter content could be particularly useful in stabilising the mine tailing's physical, chemical and biological characteristics, including changes in pH, electrical conductivity, metal contents, soil C and soil organisms (Feng *et al.* 2019).

To investigate the effects of weathering on U + M tailings, an 18-week laboratory experiment of wetting and drying cycles was conducted under atmospheric conditions to simulate chemical weathering of U + M tailings with the overall goal of assessing U + M tailing's potential for C sequestration and AMD.

The objectives of this study were to:

1. To determine the relative stability of the constituent minerals of U + M tailings by selective chemical extractions with an increasing degree of strength of extractability – using mineral acid (HCl), complexing extractant (ammonium oxalate acid, AAO) and 'total' (aqua regia).
2. To quantify changes in the pH, chemical and mineralogical composition of U + M tailings (with and without topsoil amendments) subjected to repeated wetting and drying cycles.

Experimental

Sample selection and preparations

Five U + M mine tailings with different mineralogical compositions were selected from Western Australia and western Canada (Table 1). Mining operations did not remove sulfides during processing of ore. The topsoil used was collected from the 0–30 cm of cultivated surface of an agricultural field on Westham Island, Delta, BC. The soil texture was silt loam – silty clay loam with total soil C content of 0.97%, total sulfur (S) of 0.12%, a pH of 4.1–4.7 and soil mineralogical composition was dominated by smectite clay (Table 1).

The soil was air-dried, sieved through a 10-mesh (2-mm) sieve and stored at room temperature ($\sim 20^\circ\text{C}$) until used in

Table 1. Location and mineralogical composition of ultramafic and mafic (U+M) tailing samples and topsoil.

Samples	Location	Mineralogical composition
Serpentine (MKKNi)	Mount Keith Nickel Mine, Australia	Serpentine minerals, antigorite, lizardite and minor chrysotile ($Mg_3(Si_2O_5)(OH)_4$), with hydroxalcalite-group minerals including iowaite ($Mg_6Fe_2(OH)_{16}Cl_2 \cdot 4H_2O$) and woodallite ($Mg_6Cr_2(OH)_{16}Cl_2 \cdot 4H_2O$) with occasional pyroaurite ($Mg_6Fe_2(OH)_{16}CO_3 \cdot 4H_2O$), stichtite ($Mg_6Cr_2(OH)_{16}CO_3 \cdot 4H_2O$) and uncommon mountkeithite ($(Mg, Ni)_{11}(Fe, Cr)_3(OH)_{24}(SO_4, CO_3)_{3.5} \cdot 11H_2O$) (Wilson <i>et al.</i> 2014).
Fine processed kimberlite (FPK)	Gahcho Kue Diamond Mine, Northwest Territories, Canada	Calcite ($CaCO_3$), chrysotile ($Mg_3(Si_2O_5)(OH)_4$), forsterite (Mg_2SiO_4), microcline ($KAlSi_3O_8$), montmorillonite ($(Na, Ca)_{0.3}(Al, Mg)_2Si_4O_{10}(OH)_2 \cdot nH_2O$), phlogopite ($KMg_3(AlSi_3O_{10})(OH)_2$), quartz (SiO_2) and talc ($Mg_6(Si_8O_{20})(OH)_4$) (Johnson and Pilotto 2018; Cutts <i>et al.</i> 2020).
Nickel smelter granulated slag (SS)	Kalgoorlie, Western Australia	Major phases include: pentlandite ($(Fe, Ni)_9S_8$), heazlewoodite (Ni_3S_2) and awaruite (Ni_3Fe), magnetite ($Fe \cdot Fe_2O_4$), bornite (Cu_5FeS_4), chromite ($FeCr_2O_4$) and fayalite ($Fe_2(SiO_4)$) constitute the minor and trace phases (Page 1982).
Gabbro deposit (GT)	Tulameen, British Columbia, Canada	Dunite (dominantly olivine, $(Mg, Fe)_2SiO_4$), olivine clinopyroxenite (Mg_2SiO_4), hornblende clinopyroxenite ($Ca_2(Mg, Fe, Al)_5(Al, Si)_8O_{22}(OH)_2$), peridotite, clinopyroxenite, hornblende-olivine clinopyroxenite, hornblendite and pegmatite (dominantly quartz, SiO_2 ; muscovite, $K_2Al_4(Si_6Al_2O_{20})(OH, F)_4$; and feldspar, $(K, Na)(AlSi_3O_8)$) (Findlay 1969; Nixon and Rublee 1988).
Serpentinised intrusive (GINi)	Turnagain Nickel Cobalt Mine, British Columbia, Canada	Forsterite (Mg_2SiO_4), lizardite ($Mg_3(Si_2O_5)(OH)_4$), magnetite ($Fe \cdot Fe_2O_4$) and maghemite ($\gamma\text{-}Fe_2O_3$), diopside ($CaMgSi_2O_6$) and minor minerals (quartz, SiO_2 ; brucite, $Mg(OH)_2$; clinocllore, $(Mg_5Al)(AlSi_3O_{10})(OH)_8$; troilite, FeS ; and sjoegrenite, $MgAl_2O_4(OH)_4 \cdot 6H_2O$) (Cutts <i>et al.</i> 2020).
Delta Soil (Topsoil)	Westham Island, Delta, British Columbia, Canada	Smectite ($1/2(Ca, Na)_{0.7}(Al, Mg, Fe)_4((Si, Al)_8O_{20})(OH)_6 \cdot nH_2O$), vermiculite ($(Mg, Ca)_{0.7}(Mg, Fe^{2+}, Al)_6((Al, Si)_8O_{20})(OH)_4 \cdot 8(H_2O)$), mica (illite ($K_{1-1.5}Al_4(Si_{7-6.5}Al_{1-1.5}O_{20})(OH)_4$), chlorite ($Mg, Al, Fe)_{12}((Si, Al)_8O_{20})(OH)_{16}$), plagioclase ($Na(AlSi_3O_8)\text{-}Ca(Al_2Si_2O_8)$) and quartz (SiO_2) (Luttmerding 1981).

These data were gathered from several different sources.

the simulated chemical weathering experiment (SCW). Prior to chemical analysis, mine tailings were ground using a ceramic mortar and pestle and passed through a 120-mesh (125- μ m) sieve.

Selective chemical extraction and digestion

HCl and acid ammonium oxalate (AAO) extractions, and aqua regia (AR) digestion, were used to assess the relative mineral stability and elemental release from the tailing samples (Fig. 1) (Fanfani *et al.* 1997; Moncur and Smith 2012). Based on the manual from the Canadian Council of Ministers of the Environment (2016), samples were extracted using: 0.1 M HCl for an estimate of the readily soluble elements and used to measure mobile or plant available elements; AAO, a complexing agent, to estimate the poorly crystalline material, and amorphous inorganic and organically complexed forms of iron, aluminium and manganese; and AR to provide an estimate of total elemental concentration, except for elements held within aluminosilicate compounds (McKeague and Day 1966; Chen and Ma 2001; Evans 2013; Weaver *et al.* 2018). Extractions and digestion were conducted in duplicate.

Hydrochloric acid (HCl)

A 5.00-g sample (± 0.05 g) of each tailing was measured into a 100-mL Falcon tube with 50 mL of 0.1 M HCl and

placed onto a shaker with 5 cm, 240 oscillations per minute for 1 h. The suspension was filtered (Whatman number 42, pore size ~ 2.5 μ m), placed into clean 100 mL Falcon tubes and stored at 4°C until analysed by inductively coupled plasma–mass spectrometry (ICP-MS) (Chen and Ma 2001). The residue in the filter paper was air-dried and stored for X-ray diffraction (XRD) and C and S analysis.

Acid ammonium oxalate (AAO)

A 1.00-g sample (± 0.05 g) of each tailing was weighed into 100-mL Falcon tubes and mixed with 40 mL of AAO solution (0.2 M ammonium oxalate and 0.2 M oxalic acid). The tubes were stoppered and placed on a shaker with 240 oscillations per minute for 4 h. The suspension was filtered (Whatman number 42, pore size ~ 2.5 μ m) and stored at 4°C until analysed by ICP-MS, as above. The residue on the filter paper was treated as above.

Aqua regia (AR)

A 0.5-g sample (± 0.05 g) of each tailing was weighed into a 500-mL Erlenmeyer flask and 12 mL of AR was added (3:1 ratio of HCl/HNO₃ (v/v)). The suspension was heated on a hot plate to 110°C for 3 h or until dry. In total, 5 mL of AR was added to the mixture and heated until dry. Finally, 20 mL of 4% HNO₃ solution was added to the dried samples, the samples were then filtered (Whatman number 42, pore

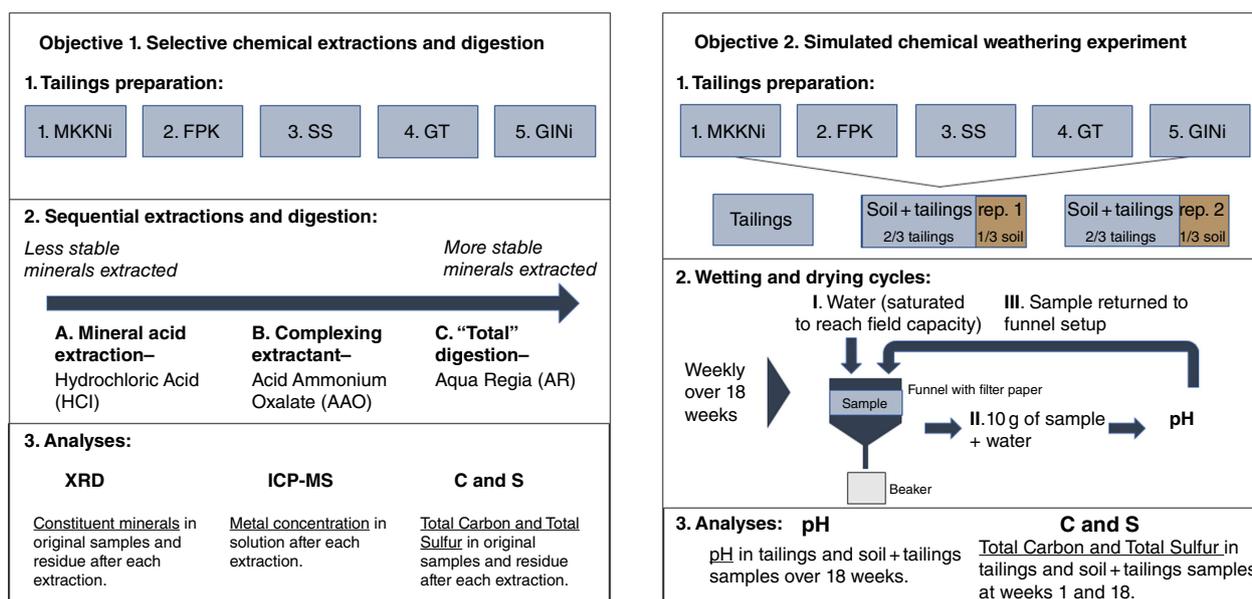


Fig. 1. Experiment set up and respective analyses related to the two study objectives – the selective chemical extraction and digestion and the simulated chemical weathering experiment (SCW).

size $\sim 2.5 \mu\text{m}$), placed into a 100-mL volumetric flask and made to volume with 4% HNO_3 . The filtrate was stored at 4°C until analysed by ICP-MS analysis. The residue on the filter paper was treated as above.

Mineralogical identification

X-Ray diffraction (XRD)

The tailings and filter residues from the three extractions were milled and analysed using a Bruker D8 Focus diffractometer equipped with a Lynx Eye detector and fine-focus Co X-ray tube operating at 35 kV and 40 mA (Fig. 1). Data were collected over a range of $3\text{--}80^\circ 2\theta$ with a step size of $0.03^\circ 2\theta$ and a counting time of 0.07 s per step. Identification of minerals was performed using DIFFRAC.EVA V.5 (Bruker AXS) and referencing patterns from the ICDD PDF-4+ database using the Rietveld (2014) method.

Inductively coupled plasma–mass spectrometry (ICP-MS)

Metal analysis was conducted using ICP-MS to detect Al, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, Si, V and Zn in leachate samples and extracts (Fig. 1). Ten standards of varying concentrations and one blank were prepared in 2% HNO_3 and the samples were analysed using a Varian 725ES Optical Emission Spectrometer (Varian, Santa Clara, CA, USA).

Simulated chemical weathering experiment (SCW)

The SCW was set up to assess how tailings vary in pH after repeated wetting and drying cycles that simulated normal atmospheric conditions at room temperature (21°C) and to determine how topsoil amendments may

affect changes in pH and mineral composition (Fig. 1). As an exploratory process-focused study, only one tailing sample of 300 g and two replicates for each soil + tailing sample were used. For the soil + tailing samples, 200 g of each mine tailing were homogenised with 100 g of topsoil. To simulate redox conditions encountered during weathering, we created conditions under which oxidation and reduction were likely to occur by saturating samples with deionised water and air drying on a weekly cycle. Büchner funnels lined with filters (Whatman number 42, pore size $\sim 2.5 \mu\text{m}$) were used, to which the samples were added and placed on a rack with Erlenmeyer flasks below (Fig. 1). Once per week (for 18 weeks), the samples were saturated with deionised water and mixed to ensure saturation (reducing conditions). Water was drained to allow samples to reach field capacity. To measure pH, 10 g of the sample at field capacity was added to 20 mL of deionised water and shaken at 240 oscillations per minute for 1 min. The sample was allowed to settle for 20 min, and the pH was measured using an Oakton PC700 series benchtop meter. The samples were returned to the funnels, mixed thoroughly and left to dry at room temperature ($22\text{--}25^\circ\text{C}$) for 1 week between pH measurements to create oxidising conditions. The deionised water used was made fresh daily and had a pH range between 6 and 7.

Carbon (C) and sulfur (S) analysis

The tailings, sample residues from the three extractions and samples after the 18-week SCW were ground with a mortar and pestle, sieved to $< 150 \mu\text{m}$ (100-mesh sieve) and analysed for C and S using a Heraeus Micro Analyser (The Elementar Vario MICRO cube) (Fig. 1). As AAO contains C, these extracts were not analysed for carbon. The C content

of the soil + tailing samples were estimated using the ratio of soil to tailing (1:2) based on the C content of the Delta soil and tailings samples.

Data presentation and statistical analysis

As the objectives of the study were by design exploratory, the experimental design was not amenable to the application of parametric statistics, thus non-parametric methods were used to calculate the standard error (s.e.) using the formula:

$$\text{s.e.} = \text{s.d.} \div \sqrt{n}$$

where s.d. is the standard deviation and n is the number of replicates.

Where concentrations were below the detection limit for ICP-MS and the C and S analysis, non-detected (n.d.) was assigned. Concentrations of Cd and Pb were below the detection limit for all samples and values were not used in further analysis. Metals with total concentrations from AR extraction below 1000 ppm were classified as trace metals and total concentrations above 1000 ppm as macro metals. The total metal concentrations were compared to [Canadian Council of Ministers of the Environment \(2022\)](#) guidelines for the Protection of Environmental and Human Health for Agricultural and Residential–Parkland purposes to identify any metals that may be released at unsafe amounts.

Results

X-Ray diffraction mineral identification

Most tailings showed no noticeable dissolution of minerals with AAO and HCl extraction, except for brucite, hydrotalcite and hydromagnesite for MKKNi and GINi (Table 2). The AR dissolved most minerals in the majority of tailings, and transformed the mineral into amorphous–precipitate materials (Scott *et al.* 2021). However, quartz, feldspar, mica and serpentine minerals remained after all three extractions.

Calcite, brucite, hydrotalcite and hydromagnesite were more easily weathered than other secondary minerals. Other minerals with relatively high weathering potentials in the mine tailings included calcite, chlorite, olivine, clays, magnesite and magnetite. The primary minerals amphibole, feldspar, mica and quartz in some of the tailings are more stable and exhibited little dissolution (Table 2). The main Mg sources were brucite, hydrotalcite, hydromagnesite, magnesite, olivine and magnetite (Table 2). Main Fe sources were magnetite, serpentine and olivine. The main source of Ca was calcite and the Al main sources were hydrotalcite, clay and serpentine (Table 2).

Metal analysis

For the bioavailable extraction (HCl), the elemental concentrations of Cr, Cu, V and Zn were the lowest, whereas the

Table 2. Minerals in tailing samples detected by X-ray diffraction after selective chemical extraction by hydrochloric acid (HCl) and acid ammonium oxalate (AAO) and aqua regia (AR) digestion.

Sample	Tailing minerals	Minerals dissolved relative to starting material		
		HCl	AAO	AR
Serpentinite (MKKNi)	Brucite		✓	✓
	Hydromagnesite	✓	✓	✓
	Hydrotalcite		✓	✓
	Magnesite			✓
	Magnetite			✓
	Serpentine			✓ ^A
	Talc			✓ ^A
Fine processed kimberlite (FPK)	Calcite			✓
	Chlorite			✓
	Pyrophyllite			✓
	Dolomite			✓
	Feldspar			
	Mica			✓ ^A
	Quartz			
	Serpentine			✓
	Talc			✓
	Nickel smelter granulated slag (SS)	Magnetite		
Olivine				✓
Amorphous material				✓
Gabbro deposit (GT)	Amphibole			
	Calcite			✓
	Chlorite			✓
	Dolomite			
	Feldspar			
	Mica			
	Quartz			
Serpentinised intrusive (GINi)	Brucite	✓	✓	✓
	Chlorite			✓
	Hydrotalcite	✓	✓	✓
	Magnetite			✓
	Mica			✓
	Olivine			✓
	Serpentine			✓ ^A

^AMinerals that exhibited partial dissolution.

highest were Fe, Mg and Ca (Table 3). For the AAO extraction (Table 4), Ca and Mn had concentrations below the detection limit for most samples. Fe had the highest

concentrations, followed by Mg and Si. The results of the AR digestion (Table 5) were used to determine whether a metal was present in macro ($>1000 \text{ mg kg}^{-1}$) or trace ($<1000 \text{ mg kg}^{-1}$) concentrations. For AR digestion (Table 5), most samples had low concentrations of Si, all samples had high concentrations of Mg and Fe and most had a high concentration of Al.

Changes in pH

At the beginning of the SCW, the pH of the tailings was between 8.4 and 9.7 (Fig. 2). Most pH values remained within this range until week 12, fluctuated and then decreased slightly (Fig. 2). The soil + tailing samples had

lower initial pH values than the tailing samples alone (Fig. 2). However, the pH for most of the tailing and soil + tailing samples converged by week 18.

Initial C content was low (0.03–1.37%) and decreased by the end of the SCW in all tailing and soil + tailing samples (0.01–0.68%), except for GINi tailings, which increased by 0.07% (Table 6). The highest C content was found in MKKNi tailings, whereas the lowest was detected in GINi and SS. The soil amendment seemed to increase C content in the soil + tailing samples. The total S content was also low (0.13–0.41%) and seemed to decrease (0.05–0.29%) after the SCW in tailing and soil + tailing samples (Table 6). Total S content was generally higher in the tailing samples compared to the soil + tailing samples. The highest

Table 3. Mean concentration of metals extracted by hydrochloric acid extraction from tailings.

Sample	Macro metals (CCME) (mg kg^{-1})								Trace metals (CCME) (mg kg^{-1})						
	Al	Fe	K	Ca	Mg	Na	Ni (45)	P	Co (40)	Cr (64)	Si	Cu (63)	Mn	V (130)	Zn (250)
MKKNi	n.d.	23.0	21.0	33.0	2160	462	11.0	n.d.	1.59	0.89	n.d.	0.85	6.62	0.50	0.40
FPK	35.0	157	172	1460	536	190	10.0	60	1.69	0.15	280	1.59	8.17	1.74	1.05
SS	178	2550	2.20	347	212	21.0	69.0	n.d.	9.58	1.80	1040	3.04	0.70	1.10	10.2
GT	113	419	52.0	1040	84.0	22.0	1.00	264	1.44	0.89	82.0	6.26	7.94	1.54	0.65
GINi	2.80	289	26.0	138	1260	8.00	18.0	n.d.	1.99	0.00	129	4.39	10.5	0.75	0.90

Serpentinised intrusive (GINi), serpentinite (MKKNi), fine processed kimberlite (FPK), gabbro deposit (GT) and nickel smelter granulated slag (SS). n.d. indicates the concentration was below detection limits and CCME indicates Soil quality guidelines for protection of environmental and human health: agricultural, residential–parkland, and commercial–industrial (Canadian Council of Ministers of the Environment 2022).

Table 4. Mean concentration of metals extracted by acid ammonium oxalate extraction from tailings.

Sample	Macro metals (CCME) (mg kg^{-1})								Trace metals (CCME) (mg kg^{-1})						
	Al	Fe	K	Ca	Mg	Na	Ni (45)	P	Co (40)	Cr (64)	Si	Cu (63)	Mn	V (130)	Zn (250)
MKKNi	8.00	2170	n.d.	n.d.	2350	423	47.0	n.d.	5.58	64.9	145	2.90	n.d.	2.50	1.34
FPK	97.0	709	50.0	n.d.	1230	189	23.0	37.0	5.57	1.19	287	2.98	0.60	3.98	1.00
SS	284	3190	n.d.	n.d.	293	115	68.0	n.d.	13.7	7.12	3350	0.00	n.d.	3.56	2.57
GT	103	1440	n.d.	n.d.	61.0	73.0	1.00	145	5.57	3.58	7.00	3.18	n.d.	6.96	2.98
GINi	n.d.	1220	n.d.	n.d.	1350	n.d.	20.0	n.d.	5.44	1.55	658	0.19	n.d.	2.52	0.00

Serpentinised intrusive (GINi), serpentinite (MKKNi), fine processed kimberlite (FPK), gabbro deposit (GT) and nickel smelter granulated slag (SS). n.d. indicates the concentration was below detection limits and CCME indicates Soil quality guidelines for protection of environmental and human health: agricultural, residential–parkland, commercial, industrial (Canadian Council of Ministers of the Environment 2022).

Table 5. Mean concentration of metals from aqua regia digestion from tailings.

Sample	Macro metals (CCME) (mg kg^{-1})								Trace metals (CCME) (mg kg^{-1})						
	Al	Fe	K	Ca	Mg	Na	Ni (45)	P	Co (40)	Cr (64)	Si	Cu (63)	Mn	V (130)	Zn (250)
MKKNi	1200	35 400	25 500	1280	102 000	3600	1510	n.d.	64.7	456	206	62.8	386	22.6	26.5
FPK	16 300	27 600	39 000	11 500	75 400	3100	783	870	59.0	130	n.d.	72.0	369	82.0	58.0
SS	10 600	233 000	501	15 300	29 500	1260	4760	n.d.	1160	268	24.0	392	204	71.3	423
GT	14 400	27 100	1000	11 200	11 800	1770	46.2	1820	34.3	65.8	n.d.	94.1	258	145	39.1
GINi	1830	47 200	227	1060	128 000	432	2470	n.d.	120	110	n.d.	157	604	35.0	51.5

Serpentinised intrusive (GINi), serpentinite (MKKNi), fine processed kimberlite (FPK), gabbro deposit (GT) and nickel smelter granulated slag (SS). n.d. indicates the concentration was below detection limits and CCME indicates Soil quality guidelines for protection of environmental and human health: agricultural, residential–parkland, commercial, industrial (Canadian Council of Ministers of the Environment 2022).

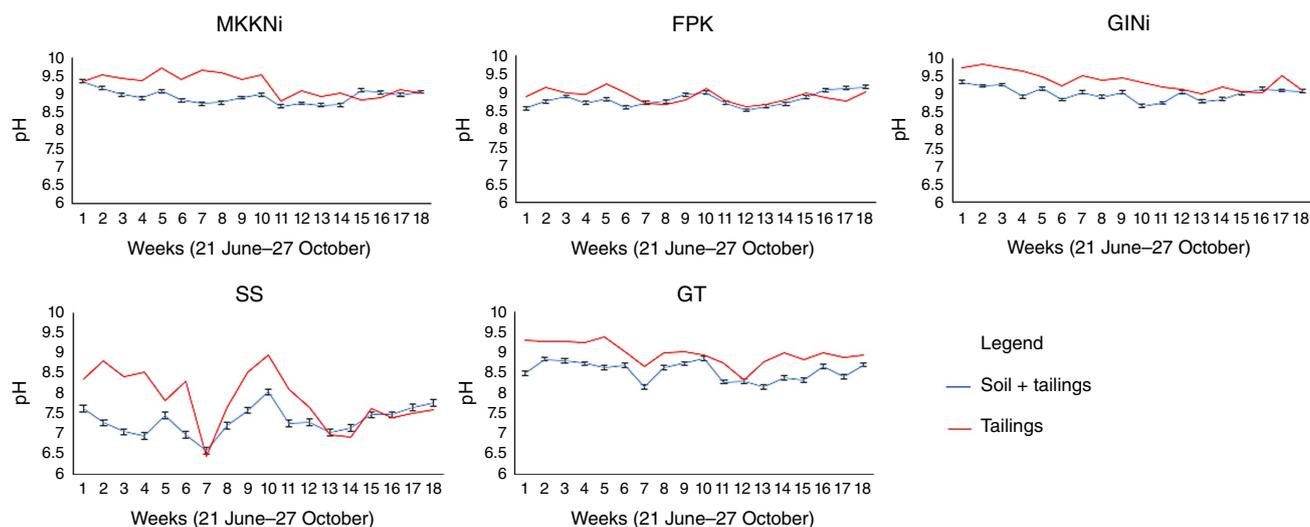


Fig. 2. Changes in pH of tailing and soil + tailing samples. Serpentinised intrusive (GINi), serpentinite (MKKNi), fine processed kimberlite (FPK), gabbro deposit (GT) and nickel smelter granulated slag (SS) over the 18-week experiment. Error bars represent standard error.

Table 6. Total carbon (C) and sulfur (S) content of the five different tailing samples under tailing (T) and soil + tailing (T + S) treatments at 1 and 18 weeks of the SCW.

Tailing sample	Treatment	C (%)			S (%)		
		Week 1	Week 18	Change	Week 1	Week 18	Change
MKKNi	T	1.37	0.68	-0.69	0.18	0.09	-0.09
	S + T	1.23	0.59	-0.64	0.16	0.08	-0.08
FPK	T	0.33	0.17	-0.17	0.14	0.06	-0.07
	S + T	0.63	0.33	-0.3	0.14	0.06	-0.09
SS	T	0.03	0.01	-0.02	0.36	0.17	-0.19
	S + T	0.34	0.24	-0.10	0.28	0.10	-0.17
GT	T	0.14	0.07	-0.08	0.13	0.05	-0.08
	S + T	0.42	0.24	-0.18	0.13	0.06	-0.07
GINi	T	0.05	0.12	+0.07	0.41	0.29	-0.12
	S + T	0.36	0.26	-0.1	0.31	0.21	-0.10

Tailing samples were serpentinite (MKKNi), fine processed kimberlite (FPK), nickel smelter granulated slag (SS), gabbro deposit (GT) and serpentinised intrusive (GINi).

percentage of S was found in GINi and SS tailings, whereas the other tailings had similar amounts.

The HCl extraction reduced total C and S in all tailings and AAO extractions also reduced S in all tailings (Table 7). HCl extracted a larger proportion of C in MKKNi and FPK (around 90%), and larger proportions of S in MKKNi, FPK and SS (around 60%). Comparing both extractions for total S content, AAO dissolved S more than HCl in general (Table 7).

Discussion

Relative chemical stability

Generally, mine tailing minerals gradually break down from primary to secondary minerals according to their relative

stability and environmental factors such as temperature, pressure, oxidising conditions and microbial activity (Jackson and Sherman 1953; Hayes *et al.* 2014). Our results were consistent with Stokreef *et al.* (2022) who also reported calcite, brucite, hydrotalcite and hydromagnesite being more easily weathered than other secondary minerals in ultramafic silicate minerals.

The metal concentrations from each extraction were consistent with the mineral composition found by the XRD analysis, confirming the stability of the U+M tailings using different methods. Mg carbonates and Mg hydroxides were less stable, with Mg showing high concentrations after the HCl extraction. By contrast, aluminosilicates are relatively stable and were only extracted after AAO extraction and AR digestion. The AAO dissolved more Fe and Al

Table 7. Total carbon (C) and sulfur (S) content (%) of the five tailing samples before and after extraction with hydrochloric acid (HCl) and acid ammonium oxalate (AAO).

Tailing sample	C (%)		S (%)		
	Before extraction	HCl extraction	Before extraction	HCl extraction	AAO extraction
MKKNi	1.37	0.04	0.18	0.05	0.07
FPK	0.33	0.03	0.14	0.06	0.04
SS	0.03	0.02	0.36	0.15	0.15
GT	0.14	n.d.	0.13	0.11	0.05
GINi	0.05	0.03	0.41	0.32	0.26

Total C was not analysed after AAO extraction. Tailing samples were serpentinite (MKKNi), fine processed kimberlite (FPK), nickel smelter granulated slag (SS), gabbro deposit (GT) and serpentinised intrusive (GINi).

compounds, with higher silica concentrations than the other extractions, and AR extracted almost all minerals, except ‘silicates’ as expected (Evans 2013).

Soil quality guidelines for protection of environmental and human health: agricultural, residential–parkland, commercial, industrial were used to assess any potential harm from the use of tailings (Canadian Council of Ministers of the Environment 2022). The AR extraction found that Ni, Cr and Cu were present in levels above the Canadian Council of Ministers of the Environment guidelines for all tailings, Co was above for most samples except GT, and V and Zn were above guidelines levels for GT and SS respectively (Table 5). The AAO extraction found that Ni was above guideline levels for MKKNi and SS samples, and Cr was above guidelines for MKKNi (Table 4). The HCl extraction found that only Ni was above Canadian Council of Ministers of the Environment guidelines for the SS sample (Table 3). These heavy metals in high concentrations are potential metal leaching sources and could be potential sources of contamination of soil, surface water or groundwater (Concas *et al.* 2006; Huang *et al.* 2013). Therefore, using these tailings with soil as an amendment would need further studies to confirm potential risk of contamination.

Effects of wetting and drying (simulated redox) on pH

Not unexpectedly, the chemical composition of the minerals had a major influence on the pH trends of the tailings during the 18-week SCW. The reasonably high initial pH of the tailings suggests that samples contained a high amount of basic cation containing minerals. Kandji *et al.* (2017) found similar results with pH values ranging between 9 and 10 in a column test for mafic tailings from Dumont Nickel Project (RNC Minerals, Québec, QC, Canada). According to the selective extractions, as predicted, the least stable minerals were calcite, brucite, hydrotalcite and hydromagnesite. These minerals contain both Ca and Mg carbonates and hydroxides, contributing to the relatively higher pH of MKKNi and GINi. Stokreef *et al.* (2022) also found that

these minerals were less stable in comparison to other common mafic minerals and found that other carbonates, such as magnesite and dolomite, are less reactive. Calcite content may also have contributed to the FPK and GT tailings’ high pH during the SCW. A study by García-Arreola *et al.* (2018) on gold and silver mine tailings also reported higher neutralisation capacity in tailings with higher concentrations of calcite.

According to the empirical Goldich sequence, basic minerals are consumed and weathered faster, while acid-generating silicate minerals continue to react more slowly over a long time period (Goldich 1938; Franke and Teschner-Steinhardt 1994). Sulfur is the major contributor to pH changes in mine tailings (Price and Errington 1998). Sulfur containing minerals were not detected in XRD results, but other studies have found that GINi and SS tailings contained minor amounts of sulfide minerals (Page 1982; Cutts *et al.* 2020). SS contained relatively lower content of carbonates and two S containing minerals, pentlandite and bornite. As a result, SS tailings exhibited the largest fluctuation and difference in pH between the tailing and soil + tailing samples among the samples studied.

The variability of the minerals in each sample was consistent with the XRD results. GINi and MKKNi had the two highest pH values throughout the SCW, which is consistent with the mineral composition. These tailings were similar among all extractions and were associated with higher levels of Mg and lower Al concentrations. By contrast, SS had the lowest pH average throughout the SCW (Fig. 2), with higher total Fe and Ni concentrations and relatively low total Mg concentrations compared to GINi and MKKNi (Tables 3–5). For the SS sample, Fe and Al were higher in the HCl (Table 3) and AAO (Table 4) extractions than in other tailings, suggesting that the Fe and Al were present in more available forms and could contribute to changes in pH over time. Higher Fe and Al contents could be potential acid-generating components in the tailings, such as in minerals containing Fe (magnetite, serpentine and olivine) and Al (serpentine and clay), by the oxidation of sulfides with Fe and the precipitation of hydroxides and oxides of Al and Fe (Chadwick and Chorover 2001; Hayes *et al.* 2009).

The use of soil amendment seemed to slightly decrease the pH of the soil + tailing samples for the first 12 weeks of the SCW. However, the pH for most of the tailings and soil + tailings samples converged and became more stable toward week 18. Since the soil had a lower pH, the mix of soil and tailing lowered the initial pH. The soil contained smectite, vermiculite and mica which may have buffered any changes in pH due to their cation exchange capacity. However, this was likely exhausted starting by week 7 in some tailings due to the high basic cation content of the U + M tailings and the reasonably low amount of acid-producing minerals (Table 2). As the U + M tailings weather, the basic cation containing minerals should increase the pH. However, all tailing and soil + tailing samples had a pH above 7 during the 18-week experiment, suggesting there were sufficient basic cations to neutralise acid-generating minerals and that the tailings likely continue to sequester C.

Kelemen *et al.* (2020) found that U + M tailings were a sink for CO₂ when exposed to weathering under atmospheric conditions; however, our results showed a slight reduction in total C and S content for almost all samples except GINi (Table 6). There are several reasons that could help explain the decrease in total C. It may be that an 18-week incubation experiment under normal atmospheric conditions is not enough time for C to become sequestered. Further research is needed to determine the contribution of organic and inorganic C that could account for C sequestration over longer periods of time. However, this study did not differentiate between organic and inorganic C and did not analyse microbial populations, which requires elucidation. Furthermore, it could be that a passivating layer of crystalline nesquehonite was formed on brucite or a silica layer on olivine, which would have impeded C sequestration (Béarat *et al.* 2006). Using optimal conditions of partial pressure, temperature, CO₂ concentration, water availability and grain size have been shown to increase the C sequestration potential of mafic tailings (Stokreef *et al.* 2022).

Conclusions

Contamination from mine tailings and CO₂ emissions from operations are critical environmental issues facing the mining industry. This research provides information about the use of U + M mine tailings as a feedstock for C sequestration and the potential of mineral soil as an amendment in U + M tailing materials. This study assessed the relative chemical stability of minerals among five U + M mine tailings and evaluated the potential environmental impacts, specifically metal release and potential for acid-generating substances, using a SCW in the laboratory.

Sequential extractions and digestion, XRD and ICP-MS results revealed that the elemental variations and the minerals identified were consistent with the Goldich stability series. The total concentration of the heavy metals, including

Ni, Cr and Cu, were above the Soil quality guidelines for protection of environmental and human health: agricultural, residential–parkland, commercial, industrial (2022). These U + M tailings are therefore potential sources for metal leaching and require further research to use in soil amendment applications.

In general, the C analysis showed a slight reduction in C content over time, suggesting that 18 weeks may not be a sufficient time period to have C sequestration. Total S was detected in minor concentrations and was associated with increased variability in pH. However, the pH of the five tailings analysed, with and without topsoil, remained above 7 during the SCW. Therefore, the presence of reactive oxides and basic cations in the U + M tailings was neutralising any acid-generating components and likely still supporting C sequestration. Further research is needed to quantify the C sequestration potential with and without varying amounts and types of topsoil, differentiating between organic and inorganic carbon and the assessment of microbial populations, to better address the use of U + M tailings for restoration applications. Future studies should also include XRD analyses of minerals at more regular intervals throughout the experiment or examine leachate from samples to understand how mineralogy affects the pH and C sequestration.

Supplementary material

Supplemental material contains five figures with XRD data. Supplementary material is available [online](#).

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Data availability. Data will be provided upon request.

Conflicts of interest. The authors declare that they have no conflicts of interest.

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Author affiliation

^AFaculty of Land and Food Systems, University of British Columbia, Office 112A, H.R. MacMillan Building, 2357 Main Mall, Vancouver, BC, V6T 1Z4, Canada.