Geochemistry and Mineralogy of Solid Mine Waste: Essential Knowledge for Predicting Environmental Impact

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Weathered Pb–Zn tailings from the Calumet Island mine, Québec

Large volumes of waste rock and mine tailings are stored at mine sites. Predicting the environmental impact of these wastes requires an understanding of mineral–water interaction and the characterization of the solid materials at the microscopic scale. The tendency of mine wastes to produce acid or neutral drainage containing potentially toxic metals generally reflects the ratio of primary sulfide to carbonate minerals and the trace element concentrations inherited from the ore deposit, as well as any ore processing that may have created new compounds. Whether potentially toxic elements are released to surface water, groundwater, or bodily fluids (in the case of ingestion or inhalation) depends on the host mineral and the possibility of sequestration by secondary minerals.

KEYWORDS: mine tailings, secondary minerals, synchrotron microanalysis, acid–base accounting, bioaccessibility

INTRODUCTION

Senior executives of mining companies have been heard to say that their business is as much concerned with moving and managing solid waste as it is with producing metals. The sheer volume of tailings and waste rock produced is astounding: on a per capita basis, North Americans produce more than 10 times as much solid mine waste as household garbage. Despite opportunities to recycle and reuse this material (Lottermoser 2011 this issue), most of it is simply stored. Aside from the physical issues associated with enormous piles of waste rock and voluminous tailings in impoundments or filling lakes and streams, the chemical reactivity of some mine wastes, specifically the release of potentially toxic and bioavailable elements to natural waters and, if ingested, bodily fluids, causes the greatest environmental damage. It is the geochemical and mineralogical character of solid mine waste that determines the nature of hazardous drainage and harmful dust, the amount of contamination and, ultimately, the risk to human and ecosystem health.

This article will show how the prediction of the risk associated with mine wastes, particularly whether they will produce acid, sulfate- and metal-rich drainage or, alternatively, pH-neutral or even alkaline drainage with potentially toxic elements, is rooted in their geochemical and mineralogical composition. Examples of exciting new microanalytical techniques will illustrate how detailed mineralogical analysis can provide key insights into the transformation of mine wastes through weathering and the implication of these reactions for environmental damage.

GEOCHEMISTRY OF MINE WASTE

Since more than 90% of the material handled during most metal mining is discarded, mine wastes are, essentially, geochemically equivalent to the rock mined. Exposure of these wastes to the atmosphere, the hydrosphere and the action of microorganisms generates drainage that may be acid and rich in dissolved metals and sulfate. As explained by Nordstrom (2011 this issue), the chemistry of the drainage is mainly the result of the oxidation of iron sulfide minerals, such as pyrite and pyrrhotite. This reaction generates acidity and sulfate and releases elements such as Cu, Zn, Cd, and Pb from sulfide minerals that are soluble in low-pH water. If sufficiently high amounts of carbonate minerals are present, the drainage may be neutralized, but some potentially toxic elements, such as arsenic and selenium, may still be present in relatively high concentration in these higher-pH waters.

The first important attribute inherited from the deposit that governs the chemical reactivity of mine wastes from metal mines is the balance between reactive, acid-generating sulfide minerals (notably pyrite and pyrrhotite) and acid-neutralizing minerals (primarily calcite and other carbonate minerals such as dolomite). This balance is evaluated using acid–base accounting methods (Jambor 2003; Price 2009). These static tests have two parts: (1) determination of the acid-producing potential (AP) based on sulfide content (assumed to be pyrite), and (2) evaluation of the neutralization potential (NP) by titrating the sample with acid, usually sulfuric. Those materials with NP/AP < 1 are considered potentially acid-generating. In modern mining, hundreds of samples are tested, and the results are used to assess the risk that acid mine drainage might be produced from the wastes. Kinetic tests provide additional information on the rate of acid generation and neutralizing processes; using "humidity cells", crushed rock is exposed to alternating dry and humid air over periods of weeks or months and then rinsed, and the drainage produced is analyzed for pH, sulfate, and other solutes.

The second important attribute of mine wastes inherited from the deposit is the trace element association, particularly the potentially toxic elements that are hosted in ore minerals or accompanying sulfides. The potential for the leaching of such elements is evaluated using various types of leaching experiments, including simple field tests—where samples are mixed with water and shaken for several minutes and the solution is then decanted and analyzed—and multiyear field bin or column studies designed to simulate mine wastes exposed to weathering conditions (Price 2009).

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Both attributes can be predicted, to some degree, by the type of ore deposit mined or prospected. For example, volcanogenic massive sulfide base-metal deposits are likely to produce acid mine drainage since the ratio of neutralizing carbonates to reactive sulfide minerals is low, and the potentially toxic elements that could be released include Cu, Zn, Pb, and Cd. On the other hand, orogenic gold deposits are less likely to produce acid mine drainage since they have lower concentrations of sulfide minerals and often contain carbonate minerals (Seal and Hammarstrom 2003). However, wastes from orogenic gold deposits may liberate As from arsenopyrite during weathering, and this metalloid can be mobile at neutral pH.

MINERALOGY OF MINE WASTE

The most serious environmental problems associated with solid mine wastes are their tendency to chemically react with air and water to produce contaminated water (Nordstrom 2011) or to be ingested or inhaled by humans and react with bodily fluids (Plumlee and Morman 2011 this issue). Airborne dust may also transport metal to soils and surface water. The fundamental processes that control whether potentially toxic elements are released or sequestered involve *mineral–water interaction*. Therefore, to understand the chemical reactions involved, it is important to characterize, in detail, the reactive minerals present in fresh and weathered mine wastes. For example, during production at the Giant mine in the Canadian Northwest Territories from 1949 to 1999, ore was roasted to release gold, transforming arsenopyrite into Fe oxides (maghemite and hematite) (FIG. 1). Since the most potentially toxic elements, As and Sb, are now hosted in oxide rather than sulfide phases in the tailings, optimal remediation of this site in the future needs to ensure that these materials are not placed in an oxygen-deficient, reducing environment where they may destabilize and undergo reductive dissolution, possibly releasing As and Sb to solution (Fawcett and Jamieson 2011). This knowledge will help to improve both risk assessment and remediation design. In another study, Grosbois et al. (2011) used a combination of mineralogical techniques to identify the As-hosting minerals in suspended particulate matter collected from streams draining a former gold-mining district in France. They show how the transport of arsenic depends on whether aggregates of clay minerals or Fe and Mn oxyhydroxide particles are the important carriers, and this, in turn, depends on hydro-
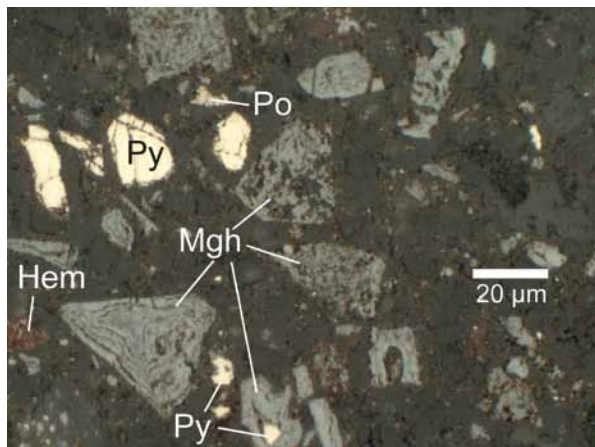
logical factors, such as high-flow conditions. During floods, mechanical erosion introduces larger particles in suspension, and these have lower surface area and less adsorbed As than the smaller particles that dominate the sediment load during low-flow conditions.

Types of Minerals in Mine Wastes

Minerals and other solid compounds in the mine wastes discussed in this article fall into four groups: (1) primary sulfide minerals, (2) primary non-sulfide minerals, (3) compounds produced by ore processing, and (4) secondary minerals formed by weathering. TABLES 1 AND 2 show examples of the first and last groups; Alpers et al. (1994) and Lottermoser (2010) have compiled more comprehensive lists.

Primary Sulfide Minerals

The sulfide minerals comprising or accompanying ore affect the degree and rate of sulfide oxidation and the nature of the potentially toxic elements released. As an example, FIGURE 2A depicts the primary ore assemblage at the Geco Cu–Zn deposit in northwestern Ontario, Canada, and FIGURE 2B shows tailings from the same deposit that have been exposed to the atmosphere for three years. Pyrrhotite, a rapidly oxidizing sulfide (Jambor and Blowes

TABLE 1	SELECTED PRIMARY SULFIDE MINERALS FOUND IN MINE WASTES
Mineral	**Formula**
Pyrite	FeS_2
Marcasite	FeS_2
Pyrrhotite	$Fe_{(1-x)}S$
Chalcopyrite	$CuFeS_2$
Bornite	Cu_5FeS_4
Sphalerite	$(Zn,Fe)S$
Pentlandite	$(Fe,Ni)_9S_8$
Enargite	Cu_3AsS_4
Galena	PbS
Molybdenite	MoS_2
Tetrahedrite–Tennantite	$(Cu,Fe)_{12}Sb_4S_{13}–(Cu,Fe)_{12}As_4S_{13}$
Cinnabar	HgS
Cobaltite	$CoAsS$
Stibnite	Sb_2S_3
Realgar	AsS
Orpiment	As_2S_3

TABLE 2	SELECTED SECONDARY MINERALS FOUND IN MINE WASTES
Mineral	Formula
Iron oxyhydroxides	
Goethite	α-FeO(OH)
Lepidocrocite	γ-FeO(OH)
Ferrihydrite	$Fe(OH)_3$
Aluminum oxyhydroxides	
Gibbsite	$Al(OH)_3$
Böhmite	$AlO(OH)$
Sulfate minerals	
Gypsum	$CaSO_4 \cdot 2H_2O$
Jarosite group minerals	$(K,Na,H_3O)Fe_3(SO_4)_2(OH)_6$
Schwertmannite	$Fe_{16}O_{16}(SO_4)_2 \cdot nH_2O$
Melanterite	$FeSO_4 \cdot 7H_2O$
Copiapite	$Fe^{II}Fe_4^{III}(SO_4)_6(OH)_2 \cdot 20H_2O$
Epsomite	$MgSO_4 \cdot 7H_2O$
Anglesite	$PbSO_4$

FIGURE 1 Reflected-light photomicrograph of calcine residue (roasted-sulfide concentrate) from the Giant mine, Yellowknife, Canada. Grey grains exhibiting spongy and concentric textures are As-bearing roaster Fe oxides. They are mostly maghemite (Mgh), with some hematite (Hem) exhibiting red internal reflections. Bright grains are relict sulfides, including yellowish white pyrite (Py) and brownish white pyrrhotite (Po). FROM WALKER ET AL. (2005), REPRINTED WITH PERMISSION FROM THE MINERALOGICAL ASSOCIATION OF CANADA
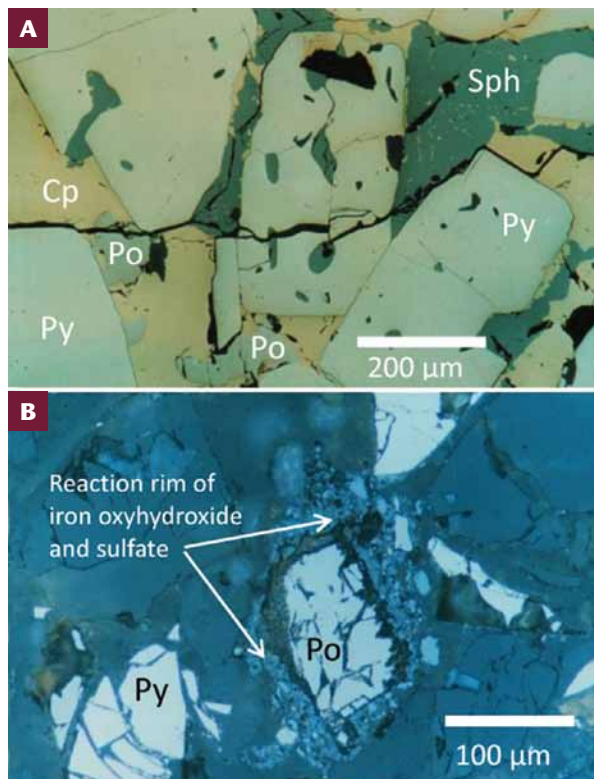
FIGURE 2 Reflected-light photomicrographs of (**A**) unaltered Cu–Zn ore from the Geco mine, Ontario, and (**B**) tailings from Geco exposed to the atmosphere for 3 years. Pyrrhotite has partially oxidized, while pyrite remains unreacted. Py = pyrite, Po = pyrrhotite, Sph = sphalerite, Cp = chalcopyrite. SOURCE: JAMIESON ET AL. (1995)

acid-generating potential of the wastes (FIG. 1). Another interesting example is described by Jambor et al. (2009), who discovered cyanide-bearing compounds formed in gold-mine tailings mixed with organic-rich lake sediments; these compounds included $Fe^{III}_4[Fe^{II}(CN)_6]_3$, commonly known as Prussian Blue. These authigenic cyanide precipitates contained significant amounts of Ni, Cu, and Zn.

Secondary Minerals

Secondary minerals formed from the weathering of primary sulfide minerals comprise a large group of mine waste minerals. Selected examples of oxyhydroxides and sulfates are given in TABLE 2, but secondary carbonates, sulfides, silicates and oxides may also precipitate (Alpers et al. 1994; Lottermoser 2010). Many of these minerals are fine grained and have a high capacity for adsorption of potentially toxic metals. These precipitates can therefore limit the aqueous concentration of such elements, although not necessarily to concentrations that are low enough to meet water-quality guidelines. Identification of these secondary minerals provides information on the aqueous conditions under which they form (pH, redox, metal and sulfate concentrations). For example, under acid conditions galena may oxidize to anglesite ($PbSO_4$), whereas in a pH-neutral environment the secondary mineral will be cerussite ($PbCO_3$). Spectacular examples of secondary minerals are the Fe sulfate precipitates that occur underground at the Richmond mine at Iron Mountain in northern California (Nordstrom et al. 2000; Nordstrom 2011) and downstream from the Rio Tinto mine workings in Spain (Hudson-Edwards et al. 1999, 2011 this issue). In many other cases, secondary minerals occur as weathering rims on primary minerals (FIG. 2B) or as poorly crystalline crusts and cements. Even when present in minor amounts, these secondary minerals are very important in mineral–water interaction, and characterizing them requires a combination of careful and novel analytical techniques.

Methods of Mineral Characterization

Identification of the minerals associated with mine wastes is key to understanding mineral–water reactions. Particle size, porosity, surface area, degree of crystallinity, and trace element concentration are also important since these characteristics may control the long-term stability of the minerals and their role in releasing potentially toxic elements to water and organisms. Selected examples of the techniques used to characterize mine waste minerals are given in TABLE 3. If significant quantities of a monomineralic sample can be collected, as in the case of the Richmond mine Fe sulfate minerals, then "bulk" techniques, including X-ray diffraction (XRD) or the partial or complete dissolution of the mineral samples followed by chemical analysis of the resulting solution, can be used (e.g. Peterson 2003). More often, however, samples are mixtures. Conventional techniques, particularly petrography (reflected and transmitted light), electron microprobe analysis, and scanning electron microscope imaging and analysis, can be very helpful in identifying minerals and characterizing textures (Jambor and Blowes 1998; Hudson-Edwards et al. 1999). FIGURE 3 shows how element mapping and electron imaging using an electron microprobe can characterize a primary sulfide grain rimmed by a secondary mineral. Researchers have recognized that many mineral–water interactions occur in microenvironments (FIG. 4) and require nanoscale characterization (Hochella et al. 2005; Petrunic et al. 2009).

The availability of powerful X-ray beams generated at synchrotron facilities has produced a new family of techniques that are very useful for characterizing mine waste minerals. Synchrotron-based X-ray absorption spectroscopy (XAS) can be used to identify the minerals hosting potentially toxic elements such as Pb (Cotter-Howells et

1998), exhibits textural evidence of dissolution and a reaction rim of Fe oxyhydroxide and sulfate. The adjacent pyrite grain has less-evident oxidation, suggesting a slower rate of reaction. The element of most concern in drainage from the Geco deposit is Zn released from sphalerite, which also appears to dissolve readily in tailings (Jamieson et al. 1995).

Primary Non-Sulfide Minerals

Carbonate minerals in ore deposits or their host rocks have an important role in neutralizing acid drainage. In mine-tailings impoundments, it has been observed that calcite is the most soluble carbonate and dissolves rapidly. Dolomite and ankerite dissolve more slowly (Jambor and Blowes 1998) but can still provide neutralizing capacity. However, if Fe and Mn carbonate minerals dissolve under oxidizing conditions, Fe^{2+} and Mn^{2+} can hydrolyze and produce acid.

Silicate minerals may dissolve slowly in mine drainage and are not usually effective in controlling pH (Jambor et al. 2002). In some cases, however, soluble silicates may significantly influence the composition of drainage. At the Ekati diamond mine, Northwest Territories, Canada, crushing and washing of kimberlite during the diamond-removal process produces alkaline, high-Mg discharge water due to the rapid dissolution of serpentine and olivine (Rollo and Jamieson 2006). This also involves the trapping of atmospheric CO_2 and production of secondary carbonates, a process that can balance the greenhouse gas emissions of mining operations (Wilson et al. 2009; Lottermoser 2011).

Compounds Produced by Ore Processing

On-site processing (such as ore roasting, heap leaching and pressure oxidation) may produce new solid compounds that are part of mine wastes. In some cases, this involves converting sulfide minerals to oxides, thus reducing the

Analytical technique	Application to mine waste mineralogy	Example studies cited in this article
Scanning electron microscopy (SEM)	Imaging, element mapping and qualitative spot chemical analysis	Hudson-Edwards et al. (1999); Walker et al. (2005)
Electron microprobe analysis (EMPA)	Element mapping, quantitative spot chemical analysis	Walker et al. (2009); Corriveau et al. (2011)
Transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM)	High-resolution imaging; may include chemical information and electron diffraction	Petrunic et al. (2009)
X-ray diffraction (XRD), including Rietveld analysis	Mineral identification based on crystal structure; Rietveld analysis provides relative amounts of crystalline phases in mixtures	Wilson et al. (2009)
Micro-X-ray diffraction using conventional or synchrotron sources (micro-XRD)	Grain-scale mineral identification based on crystal structure; application to poorly crystalline materials	Walker et al. (2009); DeSisto et al. (2011)
Synchrotron-based X-ray absorption spectroscopy (XAS)	Mineral characterization based on oxidation state and short-range structure	Foster et al. (1998)
Synchrotron-based X-ray fluorescence (micro-XRF)	Element mapping and semi-quantitative spot analysis	Hayes et al. (2009); Walker et al. (2009)

al. 1994) and As (Foster et al. 1998; Hudson-Edwards et al. 2005). These techniques are called X-ray absorption near edge spectroscopy (XANES) or extended X-ray absorption fine structure (EXAFS), depending on which part of the absorption spectrum is used. If a microfocused synchrotron beam is used, near-simultaneous analysis using micro-XAS, micro-XRF (which uses the X-ray fluorescence signal to provide information on elemental composition) and micro-XRD (X-ray diffraction) can be accomplished (Walker et al. 2005, 2009). FIGURE 5 shows an example of synchrotron-based micro-XRD, which is similar to conventional XRD in that the unknown pattern can be compared with thousands of reference mineral patterns; however, micro-XRD has the special advantage that a microfocused synchrotron beam can collect information from individual grains or alteration rims of minerals that are present in low abundance and would not be apparent in a bulk XRD pattern. Moreover, the powerful synchrotron beam tends to produce diffraction from microcrystalline and nearly amorphous materials, typical of secondary minerals in mine wastes. In fact, micro-XRD tends not to work well for coarser

mineral crystals formed by high-temperature geological processes since the large individual crystals are unlikely to diffract using the microfocused monochromatic X-ray beam. Thus the method eliminates patterns from quartz, feldspar, and other abundant minerals that tend to dominant mine waste, enhancing the ability to discern metal-bearing minerals of interest.

Rarely is one method adequate to thoroughly characterize the minerals in mine waste, especially as multiple hosts are to be expected, and most researchers use a combination of techniques to characterize mine waste minerals and coexisting water (e.g. Hudson-Edwards et al. 2005; Fawcett and Jamieson 2011). Identification of contaminant-hosting grains is not possible with conventional tools. This last point is an important consideration for risk assessors in situations where the potential hazard associated with mine waste depends on the mineral host, as explained in the following example.

GEOCHEMICAL AND MINERALOGICAL CONTROLS ON THE ENVIRONMENTAL AND HEALTH IMPACT OF MINE WASTE: AN EXAMPLE

Abandoned gold-mine tailings from several sites in Nova Scotia, Canada, contain high concentrations of As, a result of mining and processing arsenopyrite-rich ore from 1860 to 1945. Many of these tailings are now located close to residential areas, and they are publicly accessible and used
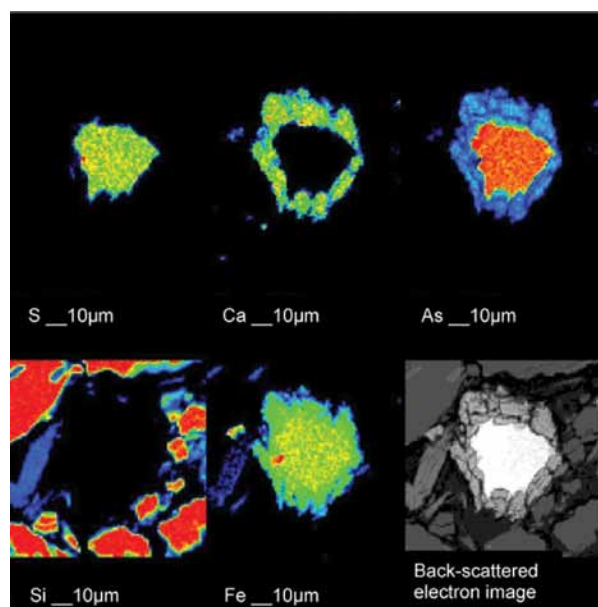

FIGURE 3   Element maps for S, Ca, As, Si, and Fe and a backscattered-electron image of a grain of arsenopyrite found in gold mine tailings; the grain has partially oxidized and is surrounded by a rim of secondary Ca–Fe arsenate. SOURCE: CORRIVEAU ET AL. (2011)
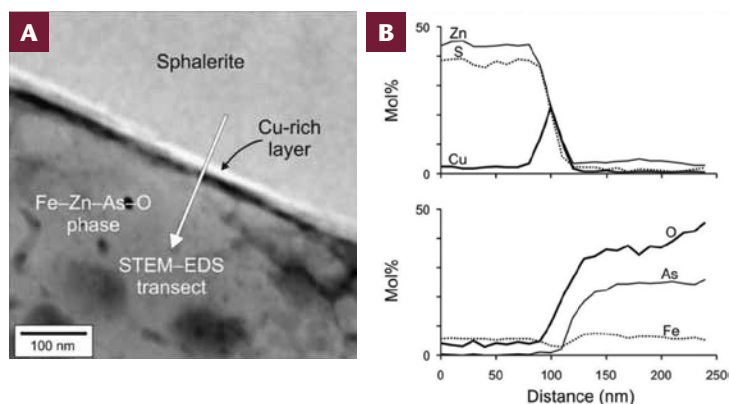

FIGURE 4   (A) TEM image of sphalerite with a bright Cu-rich zone visible along the outer margin. (B) STEM–EDS data collected along the path shown in (A) using a nominal probe size of 5 nm and showing concentrations in mol%. SOURCE: PETRUNIC ET AL. 2009, REPRINTED WITH PERMISSION
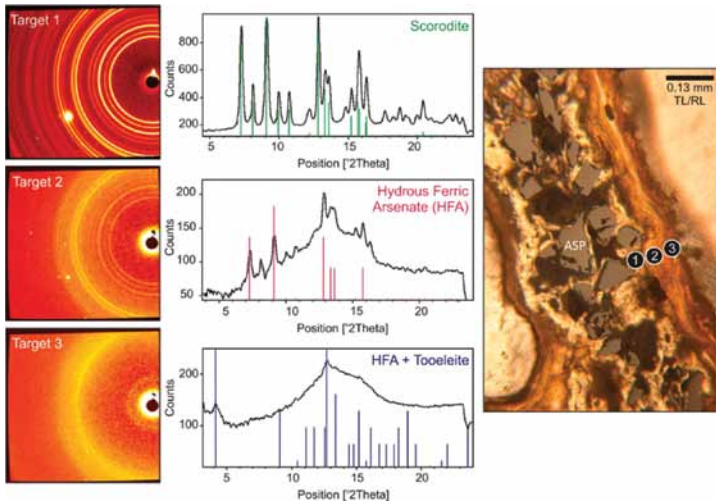
FIGURE 5 Micro-XRD results from a transect across cemented layers in an As-rich hardpan in gold-mine tailings. The left and center panels of the figure show diffraction data for the three points indicated in the thin section photo (transmitted + reflected light; ASP = arsenopyrite) on the right. These data can be compared with those from reference minerals in order to identify the phases present. The stick patterns shown in the central panel correspond to scorodite ($FeAsO_4 \cdot 2H_2O$, spot 1, top), a mixture of HFA (= amorphous hydrous ferric arsenate) and scorodite (spot 2, middle), and HFA with traces of tooeleite [$Fe_6(AsO_3)_4(SO_4)(OH)_4 \cdot 4H_2O$, spot 3, bottom]. In the middle and bottom stick figures, the HFA component does not have sticks due to its amorphous nature but is identified by the broad hump in these patterns. Scorodite, which is stable at low pH, is found in close proximity to oxidizing arsenopyrite where pore water is acid. Moving away from the arsenopyrite, grain-scale pH changes result in the differing cement composition, which influences the stability and long-term As sequestration of the hardpan. SOURCE: DESISTO ET AL. (2011)

for recreational purposes, such as dirt biking and trail walking (FIG. 6A). The total As concentration (in the <150 µm fraction) measured in a suite of 14 near-surface materials from sites throughout Nova Scotia ranges from 0.07 wt% As to over 30 wt% As. Arsenic in the tailings was originally present mainly as arsenopyrite (FeAsS), but decades of exposure to the atmosphere has transformed the sulfide into various arsenate and, more rarely, arsenite minerals (Walker et al. 2009). Typically, a near-surface tailings sample contains at least four types of As-hosting phases.

In the Montague gold district, one of the abandoned and unremediated sites within a few kilometers of the city of Dartmouth, Nova Scotia, several geochemical types of mine wastes have been delimited, based on element ratios and the balance between acid-generating sulfide and neutralizing carbonate minerals. The typical tailings exposed at surface and their unoxidized counterparts submerged in the nearby wetland have a ratio of acid potential to neutralization potential slightly less than one, indicating that these materials may generate acid (FIG. 6B). Past processing practices resulted in the deposition of sulfide concentrates, some of which have weathered to form hardpans consisting of residual arsenopyrite cemented by Fe arsenate minerals (DeSisto et al 2011). These materials are clearly acid-generating (FIG. 6B). In terms of their influence on surface water and groundwater compositions, the typical tailings, unoxidized tailings and hardpan would conventionally be considered the riskiest mine waste and warrant the most attention for remediation. The high-Ca/As tailings found in some areas of the site provide neutral drainage and are not acid-generating (FIG. 6B) and, on these grounds, would be considered less risky.

However, a different interpretation of the risk associated with this site arises when we consider the possible oral ingestion of fine dust. Based on near-surface tailings

samples from several abandoned gold mines in Nova Scotia, Meunier et al. (2010) showed that As bioaccessibility (that is, the fraction of arsenic dissolved in simulated gastric and intestinal fluids) depends on As mineralogy. As explained by Plumlee and Morman (2011), bioaccessibility testing provides a measure of how easily potentially toxic elements are released to bodily fluids. In particular, samples rich in scorodite ($FeAsO_4 \cdot 2H_2O$) and arsenopyrite exhibited relatively low As bioaccessibility, whereas samples containing Ca–Fe arsenate minerals, such as yukonite [$Ca_7Fe_{12}(AsO_4)_{10}(OH)_{20} \cdot 15H_2O$], have relatively high bioaccessibility. This is consistent with the known character and geochemical behavior of these minerals. It is not surprising that the coarse-grained primary arsenopyrite does not release much As (0.62% of total As present in the sample which is mostly arsenopyrite) during the 1-hour gastric (pH = 1.5) and 4-hour intestinal (pH = 7) bioaccessibility tests. The scorodite-rich sample released only 0.13% of its total As in the gastric step and 0.32% in the intestinal step, reflecting the pH dependence of scorodite solubility. The significantly higher As bioaccessibility (49%, 29%) associated with samples where Ca–Fe arsenate is the major As host is probably related to the fact that these tailings contain residual carbonate and that the As-hosting Ca-bearing minerals dissolve more readily in the gastric fluid. From the point of view of the oral ingestion of dust, the high Ca/As tailings might therefore be considered the riskiest material (Meunier et al. 2010).
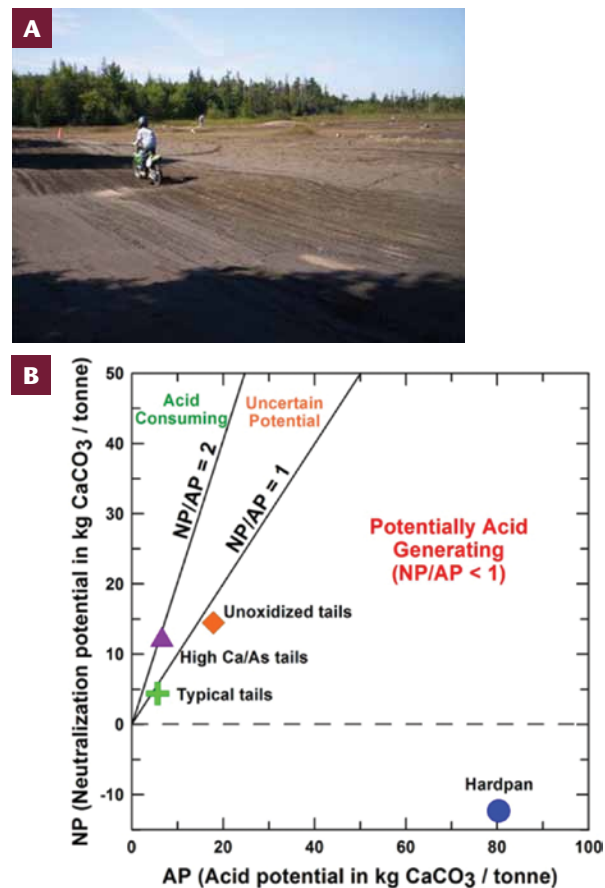

FIGURE 6 (**A**) Children riding dirt bikes on abandoned tailings at Montague Gold Mines, Nova Scotia, Canada, which contain very high concentrations of arsenic. SOURCE: S. DESISTO. (**B**) Net acid and net neutralization potentials of different types of tailings from Montague. The standard unit for expressing AP (acid potential) and NP (neutralization potential) is the equivalent kg $CaCO_3$ per tonne. Criteria are from Price (2009). See text for further explanation. COURTESY OF J. KAVALENCH, QUEEN'S UNIVERSITY, AND M. B. PARSONS, GEOLOGICAL SURVEY OF CANADA (ATLANTIC)

OUTLOOK

Reducing the environmental and health impact of mine wastes requires expertise and skills from several disciplines. Central to these are geochemistry and mineralogy. In particular, understanding mineral–water interaction requires characterizing phases at the micrometer scale or less, which is usually accomplished by a combination of conventional and newly developed analytical techniques. Very few of those who make multimillion-dollar decisions to grant permits for mines to operate, approve clean-up plans for mine closure, or cope with disastrous events such as tailings-dam failures or community health impacts related to mining are able to tackle or interpret mine waste mineralogy. Our professional community can make important contributions in this area by providing expertise and access to novel analytical techniques and by training students.

ACKNOWLEDGMENTS

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