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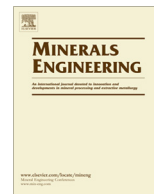
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The process mineralogy of mine wastes

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ABSTRACT

The discipline of process mineralogy developed through the recognition that metallurgical flowsheets could be optimised by thorough characterisation of the precursor ore mineralogy, mineral associations, grain size and textures. In a procedure analogous to process mineralogy it is shown here that effective characterisation of mine wastes for Acid Rock Drainage and Metal(loid) Leaching (ARDML) potential must follow a similar set of robust practices which include: (i) representative sampling; (ii) static/screening level geochemical tests and qualitative mineralogical assessment; (iii) longer-term kinetic geochemical tests and quantitative mineralogical assessment; and (iv) quantitative numerical modelling to assess source term chemistry associated with the mine facilities and thereby determine potential impacts to receptors. This process is dependent on a sufficiently robust drill core database and a detailed mine plan through which an assessment of mine wastes is possible. Such detailed characterisation may be limited by insufficient budgets, however omission of a thorough mineralogical investigation may lead to a lack of understanding of the primary geochemical controls on mine waste behaviour. In turn, this can lead to over- or under-engineering of mine facilities, which can have financial and/or environmental implications. Several case studies are presented to illustrate how mineralogy can be applied to solve problems in ARDML prediction and mitigation, specifically within waste rock assessment.

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1. Introduction

Process mineralogy is an integrated discipline which combines quantitative (and qualitative) mineralogical techniques with metallurgical testwork (Lotter et al., 2011). The aim for the process mineralogist is to provide information on specific aspects of the ore mineralogy and mill products and, in so doing, to help the chief metallurgist optimise metallurgical flow-sheets (Henley, 1983). As such, process mineralogy is usually applied to the optimisation of grades and recoveries within a working mine and has been applied to numerous deposit types including gold (Zhou and Gu, 2008), platinum group elements (PGEs) (Cabri et al., 2005) phosphates (Sant'Agostino et al., 2001), uranium (Bowell et al., 2011) and silicon (Grammatikopoulos and Clark, 2001). In brief, the principal features of modern process mineralogical techniques can be divided into four phases; (i) representative sampling; (ii) ore characterisation; (iii) mineralogical assessment and (iv) metallurgical testing. The benefit of applying this robust technique to Acid Rock Drainage and Metal Leaching (ARDML) assessments is twofold. Firstly, process mineralogy has provided real and measurable improvements in flowsheet optimisation and grade-recovery problem shooting (e.g. Charland et al., 2006; Lotter et al., 2003).

Secondly the offshoot of getting an ARDML assessment wrong has the potential for major effects (including contaminated fishery sources, contaminated water, habitat destruction and livelihood disruption where the potential is underpredicted and conversely excessive and unnecessary engineering if over prediction occurs). By using the same flowsheet it is anticipated that similar improvements in mine waste assessment, handling and eventual effective disposal and management can be achieved, particularly if mineralogy investigations are fully integrated with kinetic (long-term) geochemical testwork.

2. Process mineralogy for ARDML

ARDML refers to the generation of acidic, metal and/or sulfate-rich waters that result from the weathering of sulfide minerals (particularly pyrite/marcasite – FeS₂) under oxidising conditions (Evangelou and Zhang, 1995; Nordstrom, 1982; Nordstrom and Alpers, 1999). The process may occur naturally in sulfide-bearing rock strata, but is commonly accelerated by mining activities, which increases the likelihood of exposure of sulfide minerals to air and water, effectively accelerating natural weathering processes.

In the context of ARDML the minerals that are considered deleterious are sulfide minerals and, to a lesser extent, minerals such as elemental sulfur and hydroxyl-sulfates (e.g. jarosite, alunite). By far the most common sulfide mineral within many mineral

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deposits is pyrite (FeS_2), which due to its relative abundance is usually the greatest contributor to ARDML (Nordstrom and Alpers, 1999). Other sulfides that may be commonly observed and which may contribute to ARDML include, pyrrhotite (Fe_{1-x}S), bornite (Cu_5FeS_4), arsenopyrite (FeAsS), realgar (As_4S_4), orpiment (As_2S_3), stibnite (Sb_2S_3), sphalerite (ZnS), galena (PbS) and chalcopyrite (CuFeS_2) (Plumlee, 1999).

The composition and distribution of sulfide minerals will vary depending on the nature of the host rocks, with some deposits largely barren of sulfides (e.g. some iron–magnetite skarns) whilst others contain substantial amounts (e.g. volcanogenic massive sulfide (VMS) deposits). Indeed within a deposit the amount of sulfides may vary in terms of abundance, type and texture as the host rock varies or cross-cutting features such as dykes and veins bring in or disrupt sulfide mineralisation (Plumlee, 1999). In particular it is important to consider the texture of sulfide mineralisation and its textural relationships with other minerals (e.g. silicates, carbonates), as this is likely to have implications for the long-term weathering behaviour of the waste (Parbhakar-Fox et al., 2011). It is also necessary to consider that the nature of the proposed waste rock may vary over the course of the life of mine as mining methods change (e.g. open pit to underground mining) or as new resources are developed. It is best if such site-specific requirements are considered before mine development and re-considered during changes in operation and through to mine closure (e.g. (INAP, 2010)).

An additional component of any mineralogical assessment is the assessment of the textures and abundances of any neutralising minerals which will counteract the negative products of sulfide oxidation (namely; acidity, sulfur species, total dissolved solids and metal(loid)s). The reactivity of the neutralising minerals determines their effectiveness at which any acidity can be buffered. This reactivity varies widely, with most carbonate minerals being very effective acid consumers but with other minerals such as the “pyroboles” (Ca–Mg amphiboles, pyroxenes and micas) providing more long-term neutralising potential (e.g. Jambor, 2003; Nesbitt and Jambor, 1998; Table 1). In general, effective neutralisation potential requires the abundant presence of carbonate minerals (typically a threefold excess compared to sulfide minerals) (INAP, 2009).

If waste-rock is liable to produce acid then pH tends to decrease along a series of plateaus, where each plateau is controlled by buffering to a series of mineral assemblages (Fig. 1). Since this figure was produced it has become apparent that neutralisation can occur at higher pHs through ultramafic mineral assemblages, providing they are present in sufficient abundance (Nesbitt and Jambor, 1998). It is important to note that the weathering of acid-buffering carbonate and silicate minerals in this way may result in a lag time before acid-generating conditions are produced (see Fig. 1), even in material that will eventually be highly acid-generating. Caution must therefore be practised when interpreting the early results of static geochemical characterisation testing, and it is here that adequate mineralogical characterisation may play an important role in interpreting analytical results.

If left unmitigated, ARDML can be environmentally and socially destructive, potentially causing both short-term and long-term impacts, which may be subsequently very expensive to clear-up (Price, 2003, 2009). As such, the correct characterisation of potential waste rock and tailings is necessary during the early stages (pre-feasibility and feasibility) of mine planning. This will allow the implementation of any necessary mitigation measures to minimise potential impacts to the environment as well as result in potential long-term cost savings. The use of mineralogy within ARDML assessments and waste rock characterisation has developed rapidly over the last two decades with numerous case studies (e.g. Blowes and Jambor, 1990; Downing and Madeisky, 1997;

Table 1

Typical neutralisation potential (NP) values and pH buffering ranges for some common minerals (Blowes et al., 2003; INAP, 2010; Jambor, 2003). Me = monovalent, divalent or trivalent cation.

Group	Formula	Buffer pH	Neutralisation potential range (kg)
Carbonates			500–1350
Calcite/aragonite	CaCO_3	5.5–6.9	
Siderite	FeCO_3	5.1–6.0	
Malachite	$\text{Cu}_2\text{CO}_3(\text{OH})_2$	5.1–6.0	
Oxides			
Gibbsite	$\text{Al}(\text{OH})_3$	3.7–4.3	
Goethite	FeOOH	3.0–3.7	
Ferrihydrite	$\text{Fe}(\text{OH})_3$	2.8–3.0	
Jarosite	$\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$	1.7–2.0	
Aluminosilicates		0.5–1.5	
Feldspar Group			
K-Feldspar	$(\text{K},\text{Na})\text{AlSi}_3\text{O}_8$		0.5–1.4
(Ab100–Ab50)	$\text{NaAlSi}_3\text{O}_8$		0.5–2.6
Albite			
(An51–An100)	$\text{CaAl}_2\text{Si}_2\text{O}_8$		5.3–12.5
Anorthite			
Pyroxene group	$(\text{Me})(\text{Si},\text{Al})_2\text{O}_6$		0.5–9.5
Amphibole group	$(\text{Me})_{7-8}(\text{Si},\text{Al})_4\text{O}_{11}(\text{OH})_2$		0.2–8.1
Mica group			
Muscovite	$\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$		0.3
Biotite	$\text{K}(\text{Mg},\text{Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$		2.7–8.8
Chlorite	$(\text{Mg},\text{Fe},\text{Al})_6(\text{AlSi}_4\text{O}_{10})(\text{OH})_8$		0.8–21.6
Clay group	$(\text{Me})(\text{Si},\text{Al})_4\text{O}_{10}(\text{OH})_2$		2.7–29.0
Garnet group	$(\text{Ca}, \text{Mg}, \text{Fe}, \text{Mn})_3(\text{Al}, \text{Fe}, \text{Cr})_2(\text{SiO}_4)_3$		1.3–6.3
Apatite group	$\text{Ca}_5(\text{PO}_4)_3(\text{F},\text{Cl},\text{OH})$		2.7–11.3
Miscellaneous			
Talc	$\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$		1.7
Serpentine	$\text{Mg}_6\text{Si}_4\text{O}_{10}(\text{OH})_8$		15.1–87.6
Epidote	$\text{Ca}_2(\text{Al},\text{Fe})_3\text{Si}_3\text{O}_{12}(\text{OH})$		1.0–3.0
Wollastonite	CaSiO_3		440

Goodall, 2008; Lindsay et al., 2009; Paktunc, 1999; Parbhakar-Fox et al., 2011) illustrating the integral nature of mineralogy within a standardised ARDML characterisation and prediction study (Verburg et al., 2009).

Despite these advances, the full systematic integration of mineralogical techniques within ARDML assessments still requires some development that takes into account factors such as representative sampling (e.g. (Lotter et al., 2011)), and a thorough mineralogical textural analysis (e.g. Parbhakar-Fox et al., 2011). It is argued that assessment of ARDML potential is best carried out using a procedural flowsheet analogous to a typical process mineralogical flowsheet. This has been split into four phases, namely (i) representative sampling, (ii) static/screening level geochemical characterisation tests with qualitative mineralogical assessment, (iii) longer-term kinetic geochemical tests with quantitative mineralogical assessment; and (iv) quantitative numerical modelling with mitigation recommendations (Fig. 2). In particular it is argued that the current use of mineralogy within mine waste assessments is not fully integrated with Phase 3 of this flowsheet. Mineralogy plays a key role in the first three phases and these will each be considered in turn. Several case studies have been presented which highlight the requirement for mineralogical characterisation within ARDML assessments.

2.1. Phase 1 – representative sampling

Sampling of representative waste rock can be best performed using available drill core from the exploration program (given the availability of a robust drill-core database). As with metallurgical

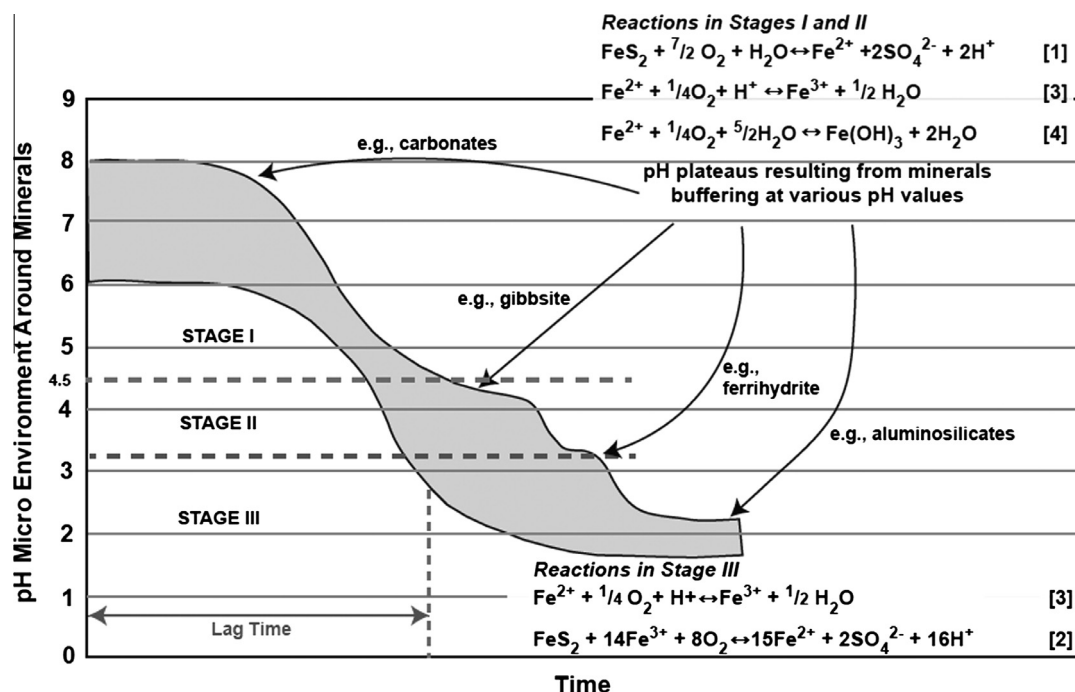


Fig. 1. Stages in the Formation of ARD (Broughton and Robertson, 1992).

process mineralogy, it is essential that sampling of waste rock is both spatially and lithologically representative (Lotter et al., 2011), as failure to do so may omit potentially important materials in terms of metal leaching or acid generation. If the mine is already operational then existing waste rock can be sampled and this can prove very valuable in calibrating results from the predictive test-work. It is generally not advisable to generate a waste rock blend/composite as this may not contain sufficient material to adequately characterise potentially problematic features and identify potentially problematic lithological units. A useful format on which to select samples is through the use of three dimensional geological visualisation tools such as Leapfrog™ that can be used to assess spatial distributions of specific lithological units, alteration types and oxidation. Such software may also be used to generate useful information through a 'merged interval' tool which catalogues the percentage length of each logged lithology present within the waste rock.

Although lithological classification of waste rock units can be a useful first tool in delineating the likely waste rock produced through mining it is more beneficial to split the waste-rock lithologies into 'geo-waste' units based on their likely behaviour during weathering. This is analogous to the use of geometallurgical units within modern process mineralogy applications (e.g. Fragomeni et al., 2005; Lotter et al., 2003). 'Geo-waste' units take account of the primary lithology, the percentage of acid generating sulfide minerals, the percentage of acid neutralising carbonate minerals and the degree of alteration and oxidation (Fig. 3). Dependent on the nature of the waste rock it may also be useful to consider other factors including: (i) the percentage content of amphiboles, pyroxene and mafic micas which provide some long-term (albeit limited) silicate buffering potential (Nesbitt and Jambor, 1998); and (ii) the variation in grain size and texture, particularly of sulfides and/or carbonate minerals, which may affect the weathering behaviour of the waste rock. Once the geo-waste units or material types are defined it is possible to select a proportional representation thereof (e.g. using the 'merged intervals' tool in Leapfrog™) to ensure collected samples are spatially representative (Fig. 4). Frequently there is not sufficient information within the drill core

log to accurately assign the waste rock into geo-waste units during the early stages of the ARDML assessment so the characterisation becomes an iterative process with an initial geochemical and mineralogical assessment followed by re-assessment after the results of laboratory testwork are received (Fig. 2).

Any samples collected from the metallurgical testwork program must also be representative of tailings that will ultimately be produced during mine life, with at least one sample assessed for each metallurgical process attempted. Tailings samples produced from a metallurgical testwork program are usually fine-grained and sub-sampling would ideally be undertaken using a spinning split riffler to produce representative sub-samples.

2.2. Phase 2 – static geochemical assessment and qualitative mineralogical characterisation

The purpose of the initial assessment phase (Phase 1) is to identify geo-waste units and tailings samples that will be representative of any material produced as waste during mining operations. Stage 2 focuses on the overall potential of the identified samples to generate acid and/or leach metals into the wider environment through a series of short-term static tests. As part of the Phase 2 assessment, effective mineralogical characterisation can prove very useful in developing an understanding of the likely geochemical behaviour of waste rock following disposal and can be integrated with the results of the static (short-term) laboratory testwork to provide a comprehensive understanding of likely geochemical behaviour (e.g. Parbhakar-Fox et al., 2011).

Static tests provide a rapid assessment of the bulk acid-generating and metal leaching potential of any given material without considering any temporal effects that may occur in leachate chemistry as weathering progresses. Static testwork methods that are typically employed as part of an ARDML assessment include Acid Base Accounting (ABA), Net Acid Generation (NAG) testing, a multi element assay and deionised water leach testing to assess potential elemental mobility. A summary of the main static tests and mineralogical techniques are provided below.

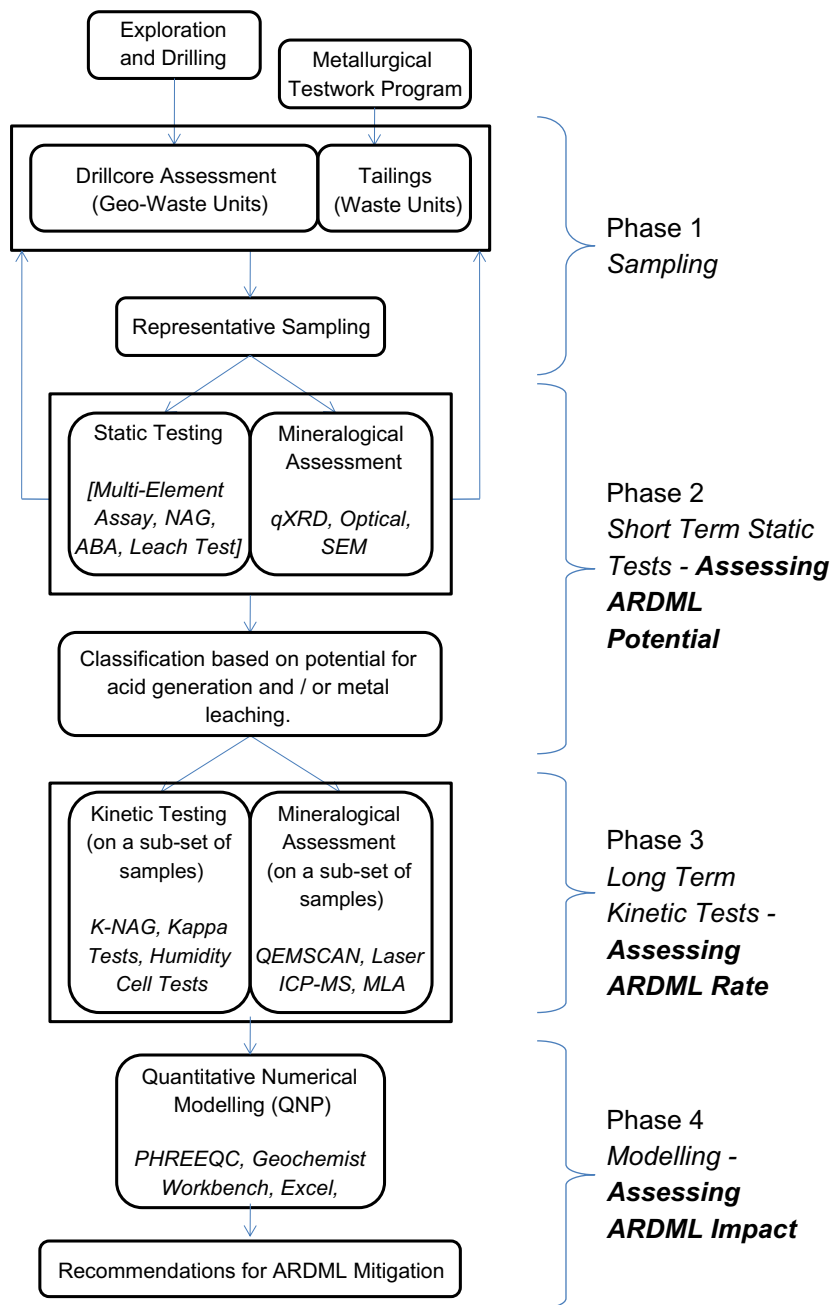


Fig. 2. Flowsheet for the assessment of waste rock and tailings samples.

2.2.1. Acid base accounting

Acid Base Accounting (ABA) indicates the theoretic potential for a given material to produce net acid or net neutral conditions. The most common ABA method applied during mine waste characterisation studies is the modified Sobek method (Sobek et al., 1978), which includes both laboratory analysis and empirical calculations to assess the balance of acid generating sulfide minerals and acid neutralisation carbonate minerals in a given sample. The results of ABA testwork are used to determine the acid generating potential (AP) and neutralising potential (NP) of each sample based on sulfide sulfur and inorganic carbon content, respectively, with results being expressed as kg CaCO₃ equivalents per tonne. The balance between the acid generating mineral phases and acid neutralising mineral phases is referred to as the net neutralisation potential (NNP), which is equal to the difference between NP and

AP. The NNP allows classification of the samples as potentially acid consuming or acid producing. A positive value of NNP indicates the sample neutralises more acid than is produced during oxidation. A negative NNP value indicates there are more acid producing constituents than acid neutralising constituents. Material that would be considered to have a high potential for acid neutralisation produce a net neutralising potential of greater than 20 kg CaCO₃ eq/ton. Acid Base Accounting data is also described using the neutralisation potential ratio, which is calculated by dividing the NP by the AP (i.e., NP:AP, also referred to as NPR).

2.2.2. Static Net Acid Generation (NAG) testing

Net Acid Generation (NAG) tests (c.f. Miller et al., 1997) are based on the principle that hydrogen peroxide accelerates the oxidation of sulfide minerals. The acid produced consequently dissolves

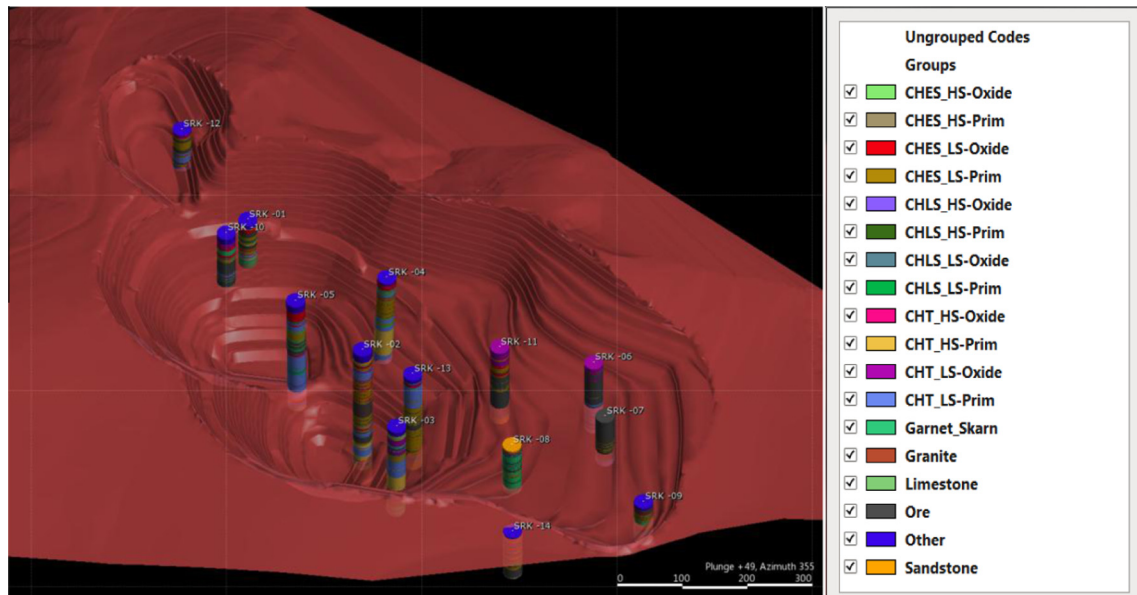


Fig. 3. Screenshot of drill-hole data where different colours represent different geo-waste units. The key on the right shows the different geo-waste units that were generated (e.g. CHES_HS-Oxide refers to all units with CHES as a primary lithology with high sulfide (HS) contents and greater than moderate degrees of alteration (oxide). The red shell represents the original estimation of the pit-shell location. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

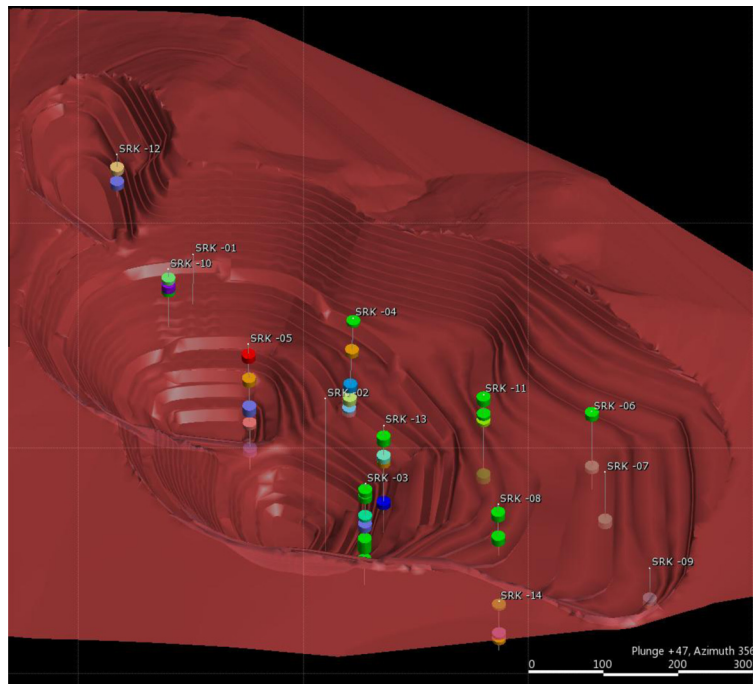


Fig. 4. Leapfrog™ screenshot showing final sample selection for waste-rock characterisation within the pit shell. Over 40 samples were selected for geochemical characterisation, but budget was only available for 10 of these to be assessed mineralogically. These constraints may cause problems and delays during later stages of mine planning and mine life.

neutralising minerals present, with the net result that both acid production and neutralisation can be measured directly. The static NAG test differs from the ABA test in that it provides a direct empirical estimate of the overall sample reactivity, including any acid generated by semi-soluble sulfate minerals (e.g. alunite) as well as other potentially acid-generating sulfate and sulfide minerals. As such, the NAG test often provides a better estimate of field acid generation than the more widely-used ABA method, which defines acid potential based solely on sulfide content.

2.2.3. Multi element assay

A multi element assay is typically completed as part of an ARDML assessment to indicate the geochemical composition of a given sample and provide an absolute upper limit of available metals for leaching. The analysis involves a strong multi-acid digestion followed by ICP-OES/ICP-MS analysis. This includes determination of major elements (e.g. Al, Ca, Mg, Na, K, Fe and S) and trace elements (e.g. Zn, Cu, Cd and Pb). The actual total release of these elements is likely to be lower, as those held within refractory silicates and oxides are

considered unavailable for environmental leaching. Multi element assay data is typically analysed using the Geochemical Abundance Index (GAI) (INAP, 2002), which compares the concentration of an element in a given sample to its average crustal abundance (Faure, 1998). According to the INAP (2002) protocol, a GAI value greater than three (equating to 12 times average crustal concentrations) indicates significant enrichment. Using mineralogy can confirm interpretation of the calculated GAI values and provide clarification of host phases and likelihood of leaching under different geochemical conditions.

2.2.4. Deionised water leach test

Leach testing using deionised water can be carried out to give an indication of short-term metal mobility and to identify constituents that are immediately available for release from a given material type. A number of standard leach test methods may be used, including the Nevada Meteoric Mobility Procedure (MWMP) test (ASTM, 2012), the US EPA Toxicity Characteristic Leaching Procedure (TCLP) test (US EPA, 1992) or the BS EN 12457 method (British Standard, 2002). Mineralogy of a waste rock can identify the main phases that would be soluble in such testwork. Examination of residues from these tests can confirm these predictions and where secondary precipitates occur document the geochemical mechanisms that occur in the weathering of a waste rock material.

2.2.5. Qualitative mineralogy

A mineralogical examination is typically undertaken on a selected subset of surface grab samples and/or core sample material, usually 1 per identified 'geo-waste' unit (Parbhakar-Fox et al., 2011). Selected samples are subjected to three principal types of investigation which include optical microscopy, scanning electron microscopy (SEM) and X-ray Diffraction (XRD). The aim of any investigation is to identify the principal minerals and provide a semi-quantitative analysis of mineral abundance. This is followed by an assessment of mineral textures, particularly focusing on acid-generating and acid-consuming minerals.

The principal method of mineralogical analysis used for most studies is optical microscopy, which can be undertaken on polished thin sections of material (e.g. Blowes and Jambor, 1990; Lindsay et al., 2009; Parbhakar-Fox et al., 2011). A Meiji MX9000 microscope fitted with a mounted Canon EOS 300D digital camera was used in the case studies provided in this paper.

Scanning electron microscopy with INCA wave- and energy-dispersive X-ray spectroscopy can be utilised for further detailed qualitative analysis of minerals present within polished thin sections. This method allows micro-chemical data to be collected that reports the chemical composition of the surface of the mineral phase in the polished section. The electron beam utilised to gather the information required is approximately 1–5 µm in diameter, so even very small phases can be quantified.

X-ray Diffraction analysis may be carried out to assess the bulk mineralogical composition of waste rock samples (e.g. Dold and Fontbote, 2001; Hammarstrom et al., 2003; Jamieson, 2011; Marescotti et al., 2008; Moricz et al., 2009; Parbhakar-Fox et al., 2011). For the case studies provided in this paper, XRD analysis was carried out using a Philips PW1710 Powder Diffractometer at the Department of Earth Science, Cardiff University, UK. Scans were run using Cu K α radiation at 35 kV and 40 mA, between 2 and 70° 2 θ at a scan speed of 0.04° 2 θ /s. From the scans, phases are identified and from the peak areas, semi quantitative analysis is performed and a percentage of each phase present calculated.

2.3. Phase 3 – kinetic geochemical assessment and quantitative or high resolution qualitative mineralogical characterisation

The requirement for kinetic testwork and more detailed mineralogical assessment is where static testwork results identify a

sub-set of material types that exhibit any uncertain or highly variable geochemical behaviour requiring further characterisation. This may be through either longer-term kinetic leach tests or through quantitative mineralogy in order to better understand the rates and mineralogical controls of solute release. The results of these more detailed geochemical and mineralogical tests are ultimately used in quantitative numeric predictions to assess likely leachate chemistry associated with the mine facilities. Therefore a thorough understanding of long-term controls on weathering is essential. Humidity cell testing (HCT) (ASTM, 2001) is the most widely recognised method of kinetic testing, however other methods are also in use, including kinetic NAG tests (KNAG) (Miller et al., 1997; Sapsford et al., 2008), Kappa tests (NEN, 1995) and column leach tests.

Humidity cell testing is designed to simulate the long-term weathering of material under accelerated laboratory conditions. This allows prediction of sulfide mineral oxidation rates in addition to the long-term potential for acid generation and metal mobility. HCT testing is typically carried out in accordance with the ASTM D5744-96 (ASTM, 2001) protocol, which is the industry standard kinetic test method for accelerated leaching of mine waste. The method involves exposing the samples to a weekly cycle of humid and dry air prior to a weekly leach and subsequent leachate analysis. Humidity cells are by nature a long term test, and thus mineralogy of the feed and residue materials is essential to characterising the mechanisms that occur within the weathered cells.

2.3.1. Quantitative or increased resolution qualitative mineralogical techniques

Using quantitative mineralogy in conjunction with these static and kinetic testwork procedures can bring clarification to the results and aid the interpretation of the ARDML potential of different geo-waste units. Quantitative mineralogical techniques that may be used during this phase of an ARDML assessment include the quantitative evaluation of minerals by scanning electron microscope (QEMSCAN) and Laser Ablation ICP-MS (LA-ICP-MS) (Ohlander et al., 2007). These latter techniques can be extremely powerful tools in mineralogical assessments and quantifying trace element concentrations within mineral phases. Unfortunately both techniques are rarely used in ARDML assessments as a result of the frequently high disbursement cost involved. This lack of use at this stage may lead to the adoption of conservative assumptions. There is therefore the potential to over-engineer mine waste impoundments due to the uncertainty over the behaviour of critical minerals with time within the particular textural development of the mine waste in question.

It is proposed that mineralogy techniques need to be more fully integrated with this phase taking account of the changes with time of the mineralogy of the mine waste. All kinetic testwork consists of monitoring the change in release rate of key constituents from mine waste samples over time. Integration of mineralogical analysis into kinetic testwork will involve the assessment of pre-test and post-test samples which should include:

- i. Quantitative or semi-quantitative XRD.
- ii. Optical microscopy, with some form of textural analyser.
- iii. Automated mineral analyser such as MLA or QEMSCAN.

The purpose is to assess the change in particle size, mineral distribution and mineral texture which will impact on changes in mine waste behaviour with time. The use of MLA in conjunction with humidity cell testing was recently trialled by Barazzoul et al. (2012) who used MLA to assess the length of free sulfide surface available for reaction. The MLA proved particularly useful at identifying and quantifying the trace sulfide mineralogy, and suggested secondary controls on sulfate release such as variable pyrite grain size and the proportion of exposed sulfide edges. However

the results were just indicative at this stage and more work is required to effectively integrate quantitative mineralogy into kinetic testwork assessments.

For current mineralogical techniques already in use the primary drive is to improve accuracy and resolution, to reduce turnaround times and cost, thereby allowing techniques such as automated mineralogy to be more freely and effectively used within ore and mine waste assessment. Further down the line the next-generation of laboratory mineralogical assessment tools will be 3D imaging equipment using high resolution X-ray micro tomography (HRXMT) (Miller and Lin, 2012), possibly with the lens based optics and detector technology of synchrotron based X-ray microscopes (Lau et al., 2012) or a combination of TEM and FIB–SEM (Thyse et al., 2012). Finally, as ore characterisation moves toward the routine use of down-hole probes and cross-belt assessment, and mine-sites routinely use on-site assessment tools and portable quantitative mineralogy, so too will the assessment of mine wastes evolve.

3. Case studies

Several case studies are presented which look at the importance of integrating geochemical testwork with qualitative and semi-quantitative mineralogy in ARDML assessments. The case studies generally cover the first three phases of a typical ARDML assessment flowsheet.

3.1. Case study 1: Pellivuoma – low acid generation and silicate buffering

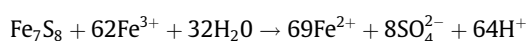
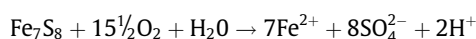
The iron ore deposit of Pellivuoma is located in the Norrbotten region of Northern Sweden, approximately 18 km west of the small rural community of Kaunisvaara and approximately 950 km north of Stockholm and close to the Finnish border. The deposit is located in the large Precambrian Fennoscandian Shield, the largest of its type in Europe. The bedrock in this area consists of Archean granitoid-gneiss unconformably overlain by Paleoproterozoic supracrustal sequences. In the Pellivuoma area these consist of a Karelian (2.5–2.0 Ga) sequence of komatiitic to tholeiitic metavolcanic rocks and terrestrial to shallow marine metasedimentary units. The Pellivuoma deposit is located just west of a significant flexure along the westernmost shear zone of the Pajala Shear Zone (PSZ) initially formed during collision of the Norrbotten and Karelian cratons (approximately 1.89–1.86 Ga) (Aker Solutions EandC Ltd., 2009; Lindholm, 2009).

The PSZ hosts approximately 30 iron ore deposits some of which contain economic concentrations of copper and gold. The Pellivuoma iron ore deposit is classed as a magnetite-dominated, calcium–magnesium and magnesium silicate skarn-hosted deposit. These occur throughout Northern Sweden and are generally referred to as “Skarn Iron Ores”. However, they also share many similarities with the Iron Ore Copper Gold (IOCG) deposits (Aker Solutions EandC Ltd., 2009; Lindholm, 2009).

Geochemical characterisation testing was carried out on the Pellivuoma materials, which included a Leco analysis to determine the acid generating potential and neutralising potential of each sample based on sulfide sulfur and inorganic carbon content, respectively. The results of the static laboratory testwork program for the Pellivuoma project demonstrated that one of the waste rock samples (a serpentine skarn) could be classed as potentially acid forming (PAF) based on a high sulfide mineral content (1.9 wt%) and a significant excess of acid generating sulfide minerals compared to acid neutralising carbonate minerals. Despite this PAF classification based on static testwork results, humidity cell testing on this sample revealed no acid-forming leachate, even after 60 weeks of testing. In order to ascertain the potential reason for

this discrepancy in static and kinetic testwork results, a detailed mineralogical study was undertaken.

Mineralogical analysis of the sample included semi-quantitative XRD, optical microscopy and SEM. Work was undertaken on the original sample as well as on the humidity cell residue after 60 weeks of analysis. XRD analysis revealed that the bulk sample initially consisted of lizardite, phlogopite, forsterite, tremolite and pyrrhotite (Fig. 5). The observation of pyrrhotite provided an early indication as to a possible limitation on acid-generation in the humidity cell tests if compared to standard methods of acid-generation potential based on pyrite oxidation. The theory is that the higher Fe to S ratio in pyrrhotite results in incomplete oxidation, producing native sulfur and sulfoxy intermediates. These intermediates are acid consuming and partially offset the acid production inherent in the theoretical or congruent oxidation of pyrrhotite (Belzile et al., 2004). However due to crystallinity and the chemistry of pyrrhotite it is generally more reactive than pyrite and oxidation by oxygen or ferric iron should still produce acidic conditions;



This is further demonstrated in experimental studies by Thornber (1975) who showed that pyrrhotite oxidation is net acidic and this is corroborated by theoretical evaluation presented by Jambor (2003). Therefore the lack of observed net acid generation in the samples cannot be interpreted as solely dependent on the presence of pyrrhotite rather than pyrite as the principal sulfide.

XRD analysis of the humidity cell residue revealed that the changes in mineralogy during the course of the testwork had been almost exclusively to the silicate mineralogy, with the breakdown of forsterite and tremolite to lizardite. This was confirmed in the SEM analysis of the HCT residue sample and reflects the relatively rapid breakdown of these silicate phases. In contrast, the coarser sulfides present within the HCT residue were largely unweathered (though the finer-grained ones had weathered away) indicating that some of the coarser-grained sulfides were resistant to weathering due to stability of the larger crystals and the higher chemical energy required to oxidise coarser stable sulfide minerals. The rapid weathering of the silicate minerals forsterite and tremolite provided the required additional buffering capacity against the

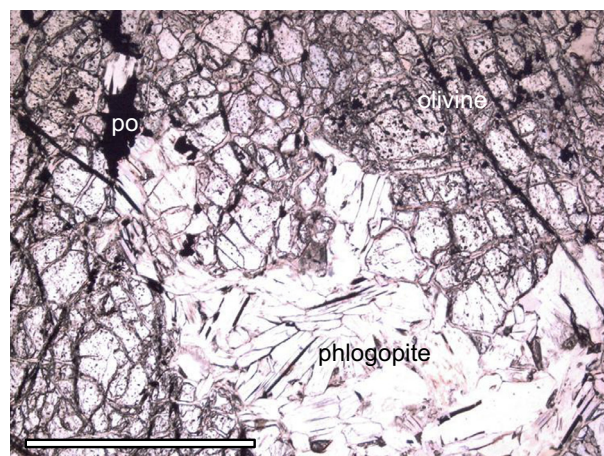


Fig. 5. Plane polarised image. X2.5 field of view: 4.64 mm. High relief olivine grains showing partial alteration to lizardite and minor magnetite. Low relief phlogopite fills the centre of the FOV. The opaque mineral to the top left of the field of view is pyrrhotite (po). The scale bar represents approximately 2 mm.

Table 2

XRD analysis of a fresh and residue sub-sample from SRK0824. There are overlapping peaks for phlogopite and lizardite so their relative proportions are uncertain. Based on optical and SEM work it would appear that lizardite is more prevalent in the residue sample and that phlogopite is more prevalent in the fresh sample.

Mineral	XRD analysis (%)	
	Fresh sample	Residue sample
Phlogopite + lizardite	77	85
Forsterite	15	10
Tremolite	5	2
Pyrrhotite	3	3

acid-producing weathering of the pyrrhotite. As approximately two-thirds of the original forsterite was found to remain within the residue after 60 weeks of testing, then this buffering capacity is expected to continue, though without continuation of the humidity cells it is not clear for how long (Table 2).

ABA analysis of the sample in question both pre-leach and post-leach revealed a third phenomenon which further aided the acid-neutralising capacity of the sample. Re-testing of the HCT residue sample still suggested that it would be potentially acid-generating but to a far lesser extent. This is because carbonate minerals were beginning to form in the residue through the sequestration of CO₂ from the atmosphere. The rapid breakdown of forsterite and tremolite released Mg which reacted with the CO₂ in the atmosphere to produce magnesite within the leach residues (e.g. Giammar et al., 2005; Sverdrup, 1990; Sverdrup and Warfvinge, 1995). The effect was an increase in carbonate percent from <0.01% (below detection limit) to 0.24%, and a subsequent reduction in NNP and NPR values (Table 3). As forsterite was not consumed by the end of the humidity cell testing (still approximately two thirds remaining) then the formation of magnesite is expected to continue. The ABA test result of the residue sample still suggests the sample should be acid-generating, but to a lesser degree. However, the factors that explained the lack of acid-generation within the first 60 weeks are still applicable and therefore no acid-generation is expected in the short to medium term.

In summary, the results of the static geochemical characterisation testwork unambiguously suggested that this particular sample from the Pellivouma deposit would be acid generating. However, humidity cell testwork run over 60 weeks showed no evidence for acid generation. Further investigation of the sample mineralogy showed that the lack of acid-generation was down to three main factors; (1) the presence of pyrrhotite as the dominant sulfide rather than pyrite; (2) the rapid weathering of silicate minerals which provided increased buffering capacity; and (3) the formation of carbonate within the residue sample due to the sequestration of CO₂ from the atmosphere with the Mg released from silicate weathering. This shows the importance of a thorough mineralogical investigation in interpreting the results of geochemical characterisation testwork.

3.2. Case study 2: San Manuel Tailings – low acid generation and sulfide encapsulation

The San Manuel porphyry copper ore deposit is located 64 km northeast of Tucson, Arizona, USA. Approximately 700 million tons

of ore was mined and milled over a period of 45 years, to produce a copper concentrate. The resulting tailings were deposited adjacent to the San Pedro River, near the town of San Manuel. The impoundments were constructed using the upstream method, with cyclones used to separate the coarser sands for embankment construction. From 1994 to the end of operations in 1999, tailings were deposited on the impoundments by the spigot method.

ABA testwork results indicate there is a moderate potential for the coarse-grained tailings to produce net acid conditions and generate leached constituents. The fine-grained tailings do not appear to generate acid immediately. Relative to known total concentrations, less than 3% of the total sulfur in the San Manuel Tailings is removed in deionised water tests. Mass balance calculations showed low short-term leaching for all metals and metalloids (less than 2% of total). These results generally suggest a low rate of reactivity, which was also observed in the NAG tests. The older tailings show a higher magnitude of elemental leaching than younger tailings, which implies a potential for increased metal(loid) leaching with age or exposure.

Kinetic testing, using humidity cell tests conducted over a time period of 40 weeks established rates of constituent leaching from the tailings. Within the humidity cells, the coarse sand component of the tailings showed initial high rates of elemental leaching. The initial high rates resulted from rinsing of secondary salts that had formed as a consequence of the higher sulfide contents and active weathering on the tailings embankments. After six leach cycles, the coarse sands showed reduced metal/metalloid leaching behaviour, indicating that the secondary salts were mostly removed after 6 weeks of HCT testing. By contrast, the fine-grained slimes showed minimal reactivity. The high initial levels of carbonate cement and alkaline porewater precipitates maintained a high alkalinity in the effluent throughout the 40-week test. Although some sulfide oxidation (up to 20%) occurred over the test period, the rate of sulfate release was significantly lower than the rate of release of carbonate.

The mixed tailings that comprised of blend of coarser sand and fine grained slimes were highly variable in reactivity. Some humidity cells were similar to those of the coarse sands; these cells generally contained similar concentrations of initial sulfides and showed acid generation. Other humidity cells showed minimal reactive material; these cells generated alkaline effluent throughout the test.

The presence of carbonate and sulfate salts in the tailings, as well as sulfide oxidation in the presence of comparatively greater volumes of carbonate-buffering minerals, resulted in a cyclic pattern of pH. Despite this, the release of chemical constituents remained low after initial flushing of the cells. By week two for most tailings samples and by week six for the most reactive coarse sands, most of the chemical constituents had been released and the rate of release was very low thereafter.

Mineralogical investigations revealed that a substantial portion of the sulfides in the tailings were encapsulated within silicate gangue minerals, principally quartz and feldspar. Chalcopyrite, pyrite, molybdenite, bornite, and chalcocite are the most abundant encapsulated sulfides (Fig. 6). Rare examples of pyrite were observed as coarsely crystalline, free grains in the tailings matrix (Fig. 6). It also showed that carbonate minerals are present and

Table 3

ABA testwork results for the original and testwork residue samples. Italicised values indicate the sample is likely to be acid generating. See text for details.

Sample number	Sulfide S	Total inorganic carbon (calc)	Neutralisation potential (calc)	Acid-generation potential (calc)	Net-neutralisation potential (NNP)	Neutralisation potential ratio (NPR)
	%	%	kg CaCO ₃ eq/t			
Original sample (pre HCT leach)	1.90	0.01	0.83	59.38	–58.54	0.01
Residue sample (post HCT leach)	1.80	0.24	19.99	56.25	–36.26	0.36

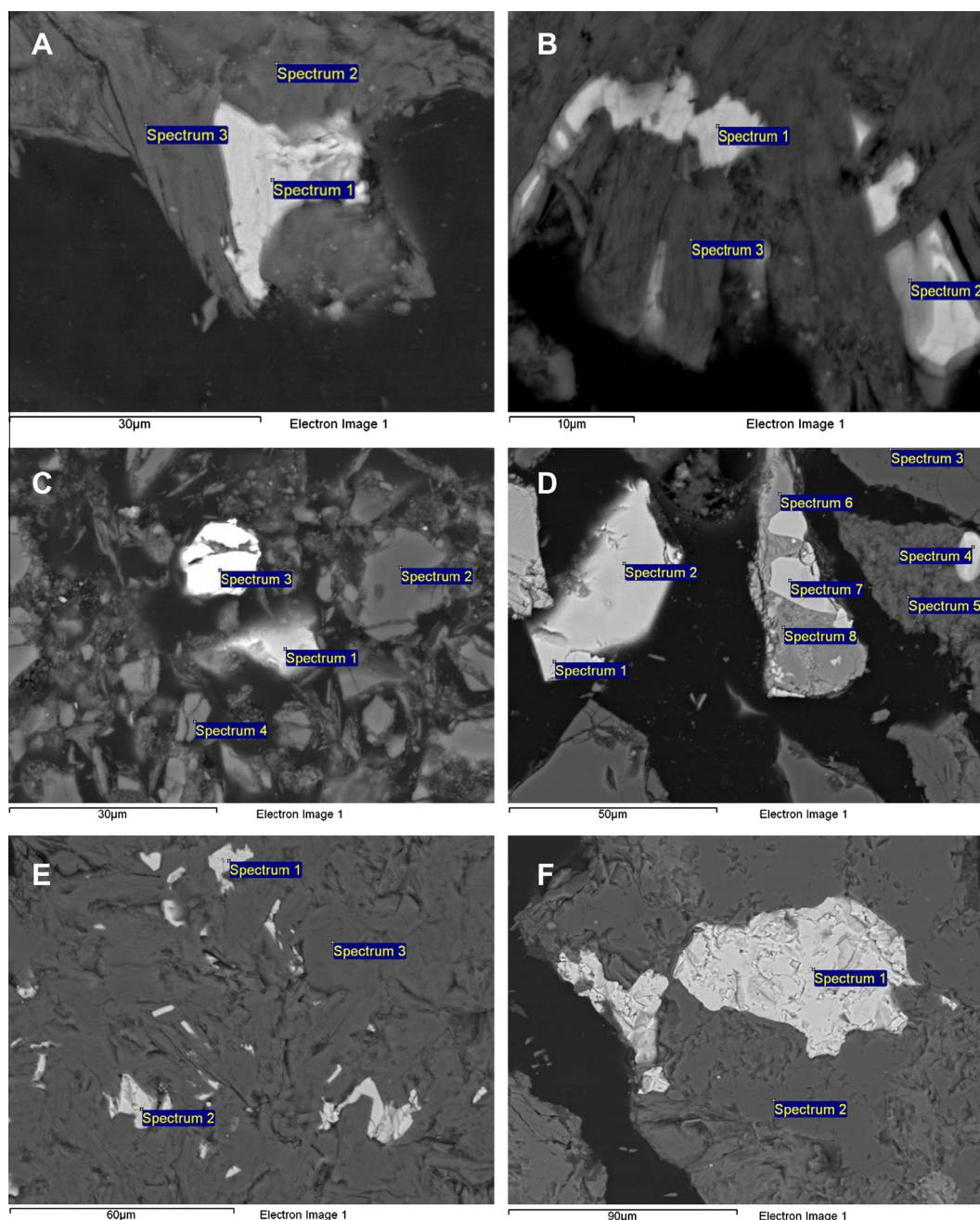


Fig. 6. Examples of sulfides within the San Manuel deposit. (A): Chalcopyrite grain predominantly encapsulated within microcline; (B): variably altered chalcopyrite (spectra 1 and 2) predominantly encapsulated within microcline; (C): liberated grains of pyrite (spectrum 1), quartz (spectrum 2), monazite (spectrum 3) and microcline (spectrum 4); (D): liberated grain of pyrite (spectra 1 & 2) associated with albite (spectrum 3), a predominantly encapsulated grain of pyrite (spectrum 4) within microcline (spectrum 5), and hematite (spectrum 7) within amphibole (spectra 6 and 8); (E): small grains of chalcopyrite (spectra 1 and 2) encapsulated within microcline (spectrum 3); and (F): chalcopyrite (spectrum 1) encapsulated within quartz (spectrum 2). All instances of microcline show alteration to illite.

comprise up to 17% of the tailings. The composition of the carbonates is complex and commonly includes coarsely crystalline grains of calcite, ankerite, and siderite. Carbonates are also present in the tailings as carbonate cement that is poorly crystalline and fine grained (i.e. highly reactive).

In summary, based on mineralogical evidence, the amount of sulfide available to react is less than the total amount of sulfide present, and this contrasts with freely available and highly reactive carbonate minerals. Thus, the available sulfide may be depleted before all available alkalinity is removed, explaining the lower than expected reactivity observed in the testwork. In addition the high degree of sulfide encapsulation explains the lower than expected release rates for the metal/metalloid constituents.

3.3. Case study 3: summer camp pit, Getchell Nevada – controls of wall-rock mineralogy on pit lake chemistry (Bowell and Parshley, 2005)

The Getchell mine is located on the eastern flank of the Osgood mountains in northwestern Nevada, roughly 72 km northeast of Winnemucca. Recent operations have been active at this site since 1983 with mining primarily focussed in open-pits until 1996. Summer camp pit was one such open-pit, mined in 1990–1991 and continuously de-watered during the operations. At closure the pit was left to fill with groundwater (Bowell and Parshley, 2005).

Exploration boreholes prior to 1990 from the summer camp pit had shown the water to be alkaline and to contain arsenic. During

the early stages of pit development water quality remained constant with As concentrations of up to 0.082 mg/L, Se concentrations of 0.009 mg/L and Zn concentrations of 0.12 mg/L. The pH of bore-hole water around the summer camp pit remained circum neutral (pH 7.7–8.3) which strongly indicated an excess of neutralisation potential.

By May 1991 the effects of pyrite oxidation and/or secondary mineral dissolution had resulted in acidic water within the sump used for de-watering the pit. This was counteracted at the time by the pumping of greater volumes of groundwater into the sump which raised the pH of the sump water. However, shortly after closure of the mining operations in December 1991 the quality of the sump water quickly returned to being acidic (pH ~ 3.1).

Mineralogical analysis of the pit walls revealed a host of secondary minerals, some of which were common to all lithologies and some of which were highly dependent on their host lithology. Common to all rock types was the presence of gypsum, hydrous ferric oxide, goethite, kaolinite–illite, baryte and pyrolusite. On the western edge of the pit an outcrop of pyrite-andalusite and pyrite–carbon hornfels contained jarosite and other Fe-sulfates (including pickeringite–halotrichite, copiapite, melanterite, langite and bukovskyite as well as arsenates from the mansfieldite–scorodite series. On the eastern and southern edges of the pit are marble and andalusite hornfels host rocks which are dominated by secondary gypsum and calcite (Bowell and Parshley, 2005).

The dissolution of reactive secondary minerals on the pit wall acted as a seasonal source of acidity, alkalinity and metals (particularly arsenic) during the spring snowmelt (e.g. for similar examples see Nordstrom, 2009). The acidification of the pit lake following closure of the summer camp pit indicated that the dissolution of the secondary Fe-sulfates was far more significant than the fine-grained carbonate resulting in lower pit lake pH. This was remedied by increasing the groundwater inflow. This increased the lake volume and therefore reduced the relative importance of the pit wall leachate.

3.4. Conclusion

With the general deterioration in ore grade may come a concomitant increase in waste rock complexity, volume and deleterious mineral content as increasingly marginal deposits are exploited (e.g. Lottermoser, 2010). With the process mineralogy of ore the advances in mineralogical laboratory technology, in particular the use of automated mineral analysers and quantitative XRD have revolutionised ore characterisation (Baum, 2012). Within environmental mineralogy the assessment of mine wastes and particularly waste rock will be undergoing a similar revolution with the increased routine use of high resolution qualitative and quantitative mineralogy within the third phase highlighted above (Fig. 2). It is also expected that the development of affordable next-generation mineralogy techniques (e.g. portable infra-red microanalyzers – PIMA) will also begin to be applied to mine waste characterisation.

The three case studies illustrate that without thorough mineralogical investigation the interpretation of key testwork results, or unexpected mine waste behaviour, becomes much more difficult. For Pellivuoma the identification of pyrrhotite over pyrite and the buffering potential of the highly reactive silicates explained the lack of acid-generation and may reduce the need to over-engineer any waste rock storage facilities. Similarly, in the case of the San Manuel Tailings the identification of sulfide encapsulation meant that waste leachate was better than expected and thus reduced the likelihood of over-engineering the waste storage facilities. From the summer camp pit the identification of reactive pit wall surfaces had follow-on implications for other open-pit closures, with the relatively simple solution of backfilling reactive

surfaces with inert waste material and raising the pit-lake water level to control water quality. This paper has shown that the application of process mineralogy techniques to the characterisation of mine wastes can be used to understand the behaviour of key portions of the mine waste and thus aid in the optimisation of mine waste storage.

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