Mineralogically-Based Determinations of Neutralization and Acid Potential Using Automated Mineralogy

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ABSTRACT

The determination of the neutralization potential (NP) and acid potential (AP) of environmentally reactive material is a crucial step in the characterization and prediction of acid rock drainage (ARD) and metal leaching (ML) in mine waste for environmental planning. The calculations performed from laboratory tests (static testing) make assumptions regarding acidification and neutralization potential without mineralogical information. The role of mineralogy in ML/ARD is significant and mineralogical characterization can be used to check assumptions or enhance the prediction of water quality.

Mineral Liberation Analyzer (MLA), a scanning electron microscopy-based software program, can provide highly detailed information regarding the abundance, variability, chemistry, and physical characteristics of various acid producing and acid neutralizing minerals. Mineralogical information was determined for several mining projects in Canada using MLA and AP and NP were calculated from modal mineralogy. These results were compared to the results of static testing. Automated mineral analysis is suitable for mining projects with unusual circumstances, such as cases where detailed characterization of acid-neutralizing and acid-generating phases is called for or where concentrations of key mineral phases are below detection limits of other mineralogical characterization techniques such as XRD. Notably, the energy dispersive X-ray spectroscopy associated with MLA is ideal for the identification and quantification of iron and manganese bearing carbonates such as siderite and ankerite, which are measured as carbonates in static tests of NP but can contribute acidity from iron and manganese when dissolved. In addition, there are many other factors that control ML/ARD that can be analysed using automated mineral analysis, including mineral associations, exposure of minerals, and grain size.

Keywords: automated mineralogy; acid base accounting

INTRODUCTION

Automated quantitative mineralogy is a powerful tool that allows us to answer questions regarding mineralogical controls on the behavior of mining-related waste in the environment. Mineral Liberation Analyzer (MLA), an SEM-based mineralogical software program, and its sister software QEMSCAN have traditionally been used by the metallurgical industry for characterizing ore and mill feed. Tailings, waste rock, polished rock thin sections, mining impacted soils, and other sediments can be analyzed using MLA to determine the relative abundance of minerals in mine waste to better understand the processes that formed them and their fate. MLA has been applied to whole rock thin sections of waste rock to develop a mineralogical ARD index by Parbhakar-Fox et al. (2011), to assess the levels of base metals in mining-impacted estuarine sediments in Cornwall by Pirrie et al. (2003), and to identify mineralogical products important to weathering processes from waste rock at the Antamina Mine in Chile (Blaskovich, 2013).

In our research program, we have applied SEM-MLA to the following mine waste problems: (1) determination of acid potential and neutralization potential of mine tailings, with results that compare favorably with static testing, (2) comparison of the modal mineralogy of mine-impacted stream sediments based on SEM-MLA, bulk-XRD, and X-ray absorption spectroscopy, (3) prediction the fate of Hg, As, Cd and other elements in tailings from a complex, oxidized orebody (4) calculation of the relative proportions of anthropogenic and natural As-bearing grains in lake sediments and (5) explanation of variations in Pb bioaccessibility in tailings samples. This paper focuses on the first application.

Laboratory static tests for AP and NP are not mineralogically based and do not take into account the variability in chemical content of various acid-producing and acid-neutralizing minerals. Mineralogy plays a significant role in the neutralization of ARD and mineralogical characterization is recommended as part of every drainage chemistry prediction program to check assumptions made by laboratory tests (Price, 2009). The most important acid neutralizing minerals are in the carbonate group, which react readily and freely. Some silicate minerals such as olivine and wollastonite provide neutralization (Jambor et al., 2002) but with very slow reaction rates. It is expected that the onset of ARD precedes the reaction rates of neutralizing silicates (Jambor et al., 2002) and most researchers recommend the use of neutralization potential derived from carbonates such as calcite and dolomite, excluding iron and manganese carbonates (Jambor, 2000; Price, 2009).

The presence of iron and manganese carbonates such as siderite [$FeCO_3$], rhodochrosite ([nCO_3] and ferroan dolomite [$Ca(Fe,Mg)(CO_3)_2$, Fe>Mg] complicates neutralization potential, because the alkalinity produced by the dissolution of the carbonates is offset by the acidity contributed by iron and manganese oxidation and hydrolysis (Frostad, Price & Bent, 2003; Price, 2009). At present, the industry standard is static laboratory tests to determine sulfur species and neutralization potential, and kinetic laboratory tests where material is leached over a period of time to analyze reaction rates and metal loads. These tests utilize crushed samples and do not take mineralogical factors explicitly into account.

Automated mineralogy provides a way to look at the mineralogical and physical factors that control metal leaching and acid rock drainage. For these studies, modal mineralogy was obtained from MLA, an automated, quantitative SEM-EDS based mineralogical software program. MLA works on particle thin sections and determines modal mineralogy while collecting sample images and allowing for the analysis of sample texture and particle size. These results were expressed in terms of AP and NP, and then compared with conventional laboratory determinations of these parameters. This approach was applied to tailings from three unique sites: a massive sulfide ore that does not produce acid drainage, a syenite-hosted rare earth element (REE) deposit with very low sulfide content, and a massive sulfide ore that is known to produce ARD.

Investigated Sites

New Calumet

The former New Calumet Mine is located on Ile du Grand Calumet in Quebec, Canada, approximately 90 km northwest of Ottawa, Ontario. The New Calumet deposit consists of massive sulfide lenses and disseminated sulfides associated with cordierite-anthophyllite-bearing gneisses that have been metamorphosed from hydrothermally altered rocks (Corriveau et al. 2007). The ore consists mainly of sphalerite and galena with smaller amounts of pyrrhotite, silver, gold, chalcopyrite, and tetrahedrite. Gangue minerals are quartz, calcite, amphibole, pyroxene, biotite, orthoclase, plagioclase (anorthite and albite), titanite, goethite, and spinel (Williams, 1992).

From 1943 to 1968, 3.8 million tonnes of Pb-Zn ore grading 5.8% Zn, 1.6% Pb, 65 g/tonne Ag, and 0.4 g/tonne Au were extracted using flotation processing (Bishop, 1987). Approximately 2.5 million tonnes of tailings remain exposed on site in multiple locations: the Gobi Desert (GD), Mount Sinai (MS), and the Beaver Pond (BV). To date, remediation has been limited to re-vegetation in some areas.

The pH of drainage from the three New Calumet tailings deposits remains neutral to alkaline based on testing from the 1960s to present day (Doonkervort, 2007; Praharaj & Fortin, 2008; Dongas, 2013). However, Jaggard (2012) noted the occurrence of anglesite [$PbSO_4$], a mineral stable under acidic conditions, in the fine (<20 μm) fraction. This suggests the presence of an acidic environment, even if only on a small scale or in certain environments within the tailings.

Nechalacho

The unmined Nechalacho rare earth element deposit is situated at Thor Lake, approximately 100 km east of Yellowknife, Northwest Territories, Canada. The deposit is hosted within a hydrothermally altered layered nepheline-sodalite syenite in the peralkaline Blatchford Lake complex. The main REE ore minerals are zircon, fergusonite, allanite, monazite, bastnäsite, and synchysite/parisite. Gangue minerals are largely K-feldspar, albite, biotite, quartz, and magnetite (Sheard et al., 2012). Sulfides are uncommon in the deposit, representing < 0.01 % of the mineralogy (Purdy, 2014), most commonly occurring as pyrite, although sphalerite, galena, molybdenite, and chalcopyrite have also been reported in trace quantities (Pinckston & Smith, 1995; Purdy, 2014; Sheard et al., 2012). Carbonate minerals are also not abundant in the deposit, typically comprising about one to two percent of the rock, and include ankerite, siderite-magnesite, calcite, dolomite, bastnäsite, and synchysite/parisite (Purdy, 2014). It remains unclear how much neutralization potential is contributed by bastnäsite and synchysite/parisite.

Geco

Geco is a former Cu-Zn-Ag-Au massive sulfide producer located near Manitouwadge, roughly 250 km east of Thunder Bay, Ontario. The deposit is situated within the Manitouwadge greenstone belt, locally hosted in quartz-feldspar-biotite ± hornblende gneisses and quartz-sillimanite ± muscovite schists (Petersen, 1986). The ore mineralogy comprises chiefly of pyrite, pyrrhotite, sphalerite, and chalcopyrite, with minor galena, and marcasite. Gangue minerals are largely quartz, plagioclase, K-feldspar, muscovite and amphibole (Jamieson, Shaw & Clark, 1995), although other minerals have been documented in the deposit (Petersen, 1986). Carbonate minerals occur only in trace amounts and acid drainage from tailings has been documented (Jamieson, Shaw & Clark, 1995).

METHODS

Tailings samples from New Calumet Mine were prepared as thin sections for MLA by Vancouver Petrographics in Langley, BC, Canada. Samples were impregnated with set epoxy, mounted to glass slides, and ground to 30-50 µm thickness. Thin sections are doubly-polished and liftable, and mounted onto glass using Krazy Glue™. Water and heat were not used during preparation of thin sections, to avoid any chemical alteration of the samples. Kerosene was used when fluids were necessary.

Tailings from the Nechalacho deposit and Geco Mine were prepared at Queen's University, Kingston, Ontario, Canada. The Geco samples were sieved into three size fractions (< 125 µm, 63-125 µm, and < 63 µm), and the Nechalacho samples were left unsieved due to their fine grind size (80 % passing at 38 µm). All samples were mixed 2:1 with graphite to decrease particle density and particle agglomeration before being mixed with epoxy in a plastic vial lightly coated with petroleum jelly. The mixture was stirred thoroughly to ensure the entire sample was suspended in the epoxy and then placed in an ultrasonic bath for 10 minutes to reduce particle agglomeration. Samples from the Geco Mine were left as epoxy mounts and polished, whereas the samples from the Nechalacho deposit were cut vertically and mounted as thin sections before being polished. Water was used in the sample preparation of both the Nechalacho and Geco samples as dissolution of secondary phases was not identified as a concern.

Samples were carbon coated and analyzed under high vacuum at Queen's University on a Quanta 650 FEG-E-SEM with FEI's MLA software.

MLA Methods

MLA requires optimization of several project specific parameters, such as operating voltage, magnification, spot size, brightness and contrast. FEI recommends calibrating brightness and contrast on the brightest (highest atomic number) mineralogical phase in the sample to achieve the best range of gray scale coloring in back scatter electron view for particle distinguishing.

An important part of using Mineral Liberation Analyzer is the creation of an accurate and complete Mineral Reference Library that is used to classify the minerals in a sample or sample suite (Fandrich et al., 2007). The Mineral Reference Library is made up of mineral names or chemical composition groupings with associated EDS spectra. Each EDS spectra collected in an MLA run is compared to the Mineral Reference Library and assigned a classification (mineral name or chemical composition grouping) based on the EDS spectra it matches most closely. An accurate mineral reference library is critical, as multiple mineral types contribute to acid generation and acid neutralization.

Static Testing – Acid Base Accounting

All of the samples collected from New Calumet and Geco, and one mixed sample of mixed tailings from Nechalacho were subjected to static tests at a commercial laboratory. The analyses included paste pH, total inorganic carbon, sulfur speciation, determination of AP and NP, method modified after Sobek and CO_3-NP(Price, 2009).

RESULTS AND DISCUSSION

Determination of Neutralization Potential

Table 1 compares the NP calculated by SEM-MLA based on the modal amounts of carbonate minerals with the NP measured using chemical tests (total inorganic carbon or CO_3-NP, and the modified Sobek test). Calculating the neutralization potential of material from modal mineralogy via MLA allows for the consideration of Fe and Mn carbonates (Frostad, Price & Brent, 2003; Paktunc, 1999). For the Calumet samples, the ferroan dolomite grains contain an average iron mole % of 3.9%, and for the Nechalacho samples, the ankerite grains contain an average iron mole % of 16.24% (Buckwalter-Davis, 2013; Purdy, 2014). The total NP was calculated according to Frostad, Price & Bent (2003) from two components. $CaCO_3$-NP takes into account the modal mineralogy of calcite and dolomite and subtracts the contribution from iron (Equation 1) and $FeCO_3$-NP takes into account ferroan dolomite/ankerite and siderite and removes the contribution from iron (Equation 2).

$$\mathrm{CaCO_3 - NP}\left(\frac{\mathrm{kg\ CaCO_3}}{\mathrm{t}}\right) = \left\{\text{calcite wt \%} + \text{dolomite wt \%} + \left(\text{ferroan dolomite wt. \%} \times \left(1 - \left(\frac{\text{Fe mole\%}}{100}\right)\right)\right)\right\} \times 10 \tag{1}$$

$$\mathbf{FeCO_3 - NP}\left(\frac{\mathbf{kg\ CaCO_3}}{\mathbf{t}}\right) = \frac{\left((\textbf{ferroan dolomite wt.\%} \times \textbf{Fe mole\%}) + \textbf{siderite wt.\%}\right)}{\mathbf{115.86^{*} \times 100.09^{\#} \times 10}}$$

*115.86 = molecular weight of $FeCO_3$

#100.09 = molecular weight of $CaCO_3$

(2)

The NP values calculated from MLA are broadly similar to those measured by CO_3-NP (static test) for all three mine sites. The CO_3-NP value, based on total inorganic carbonate, is generally considered to be a more relevant static test than the modified Sobek, which can overestimate NP by including non-carbonate neutralizing minerals that are unlikely to actually react in the field (Price, 2009). Studies to measure the neutralization potential of common rock-forming minerals and common rock types and compare it to NP calculated from modal mineralogy have been done by Jambor et al. (2002); Jambor, Dutrizac & Raudsepp (2006); and Jambor, Dutrizac & Raudsepp (2007). Non-carbonate minerals (with estimated NPs) that may contribute to NP in a minor way in a modified Sobek test include plagioclase, chlorite, enstatite, anthophyllite, muscovite, and orthoclase (Jambor et al., 2002; 2006). The Nechalacho ore minerals bastnäsite and synchysite-parisite are fluorocarbonates with possible neutralization potential; however they were not included in the calculation as their contribution to NP remains uncertain.

Table 1 Neutralization Potential calculated from MLA vs. determined by static testing

Sample	**$CaCO_3$-NP (MLA)** (kg $CaCO_3$/t)	**$FeCO_3$-NP (MLA)** (kg $CaCO_3$/t)	**Total NP (MLA)** (kg $CaCO_3$/t)	**CO_3-NP (static test)** (kg $CaCO_3$/t)	**Modified Sobek NP (static test)** (kg $CaCO_3$/t)
New Calumet					
GD-VEG1	83	1.7	85	93.33	104.0
GD-VEG2	77	1.3	78	75.00	85.0
GD-non VEG	71	0.7	71	69.17	77.5
BV-VEG	65	1.7	67	66.67	74.6
BV-non VEG	45	1.3	47	44.17	54.7
MS-non VEG	10	1.7	12	8.33	14.4
Nechalacho					
UZLG	6.8	2.9	10	13.4	22.4
UZAG	10	3.4	14	13.4	22.4
BZMP	15	3.0	18	13.4	22.4
BZAG	11	2.2	14	13.4	22.4
Geco					
Main Tailings Beach	0.05	0.02	0.07	0.1	4.7
Glory Hole	0.45	0.00	0.45	0.1	0.4
Red Pond	0.46	0.10	0.56	0.1	4.3
E3 Dam	0.19	0.02	0.21	0.2	1.9

Acid Potential

Acid potential can be calculated from modal mineralogy by calculating the sulfur content of acid-generating minerals. Static testing uses a determination of the amount of sulfur in the sample to calculate acid potential. The total sulfur results from static testing compare well to the total sulfur assay determined by MLA (Table 2). Sulfide sulfur from static testing was calculated by subtracting the sulfate sulfur content from the total sulfur measurement. This is a recommended indirect method for estimating sulfide sulfur where organic sulfur is not present (Price, 2009).

Table 2 Comparison of measured total sulfur between MLA and static testing

Sample	**MLA** %S (total)	**Static Testing** %S (total)
New Calumet		
GD-VEG1	0.44	0.43
GD-VEG2	2.66	2.28
GD-non VEG	2.40	2.32
BV-VEG1	3.89	2.92

BV-non VEG	3.11	2.25
MS-non VEG	2.04	2.46
Nechalacho		
UZLG	0.003	0.039
UZAG	0.010	0.014
BZMP	0.001	0.008
BZAG	0.005	0.006
Geco		
Main Tailings Beach	4.32	7.04
Glory Hole	1.44	1.17
Red Pond	12.64	9.84
E3 Dam	15.21	15.1

AP from modal mineralogy was calculated in two ways: with pyrite and pyrrhotite and arsenopyrite only (Py+Po+As) and with all the sulfides that have the potential to oxidize (sulf). Table 3 shows that MLA-calculated AP is generally similar to AP determined by laboratory-measured %S.

Table 3 Comparison of measured and calculated acid potential (AP)

	%S(S_2) (py+po+as) MLA	**%S(S_2) (sulf) MLA**	**%S(S_2) %S(total)-%S(SO_4) Static**	**AP (py+po+as) MLA**	**AP (sulf) MLA**	**AP Static**
New Calumet						
GD-VEG1	0.32	0.45	0.40	9.85	14.11	12.50
GD-VEG2	2.07	2.74	2.23	64.75	85.72	69.69
GD-non VEG	1.57	2.37	2.04	48.93	74.08	63.75
BV-VEG1	2.85	4.01	2.87	89.13	125.2	89.69
BV-non VEG	2.24	3.20	2.21	69.97	99.87	69.06
MS-non VEG	1.59	2.06	1.95	49.78	64.46	60.94
Nechalacho						
UZLG	0.005	0.005	< 0.01	0.17	0.17	0.31
UZAG	0.010	0.010	< 0.01	0.33	0.33	0.31
BZMP	0.001	0.001	< 0.01	0.02	0.02	0.31
BZAG	0.005	0.005	< 0.01	0.17	0.17	0.31
Geco						
Main Tailings Beach	4.29	4.35	6.79	134	136	212
Glory Hole	0.36	0.36	1.15	11.3	11.3	35.9
Red Pond	12.40	12.65	8.04	388	395	251
E3 Dam	14.78	15.11	13.6	462	472	425

Neutralization Potential Ratio

Acid base accounting involves taking the results of static testing, including paste pH, sulfur species, and neutralization determination, to make estimations as to the potential for metal leaching and acid rock drainage. The neutralization potential ratio (NPR) evaluates NP/AP. For this analysis, two NPR calculations are compared in Table 4. NPR (static testing) is calculated from AP(static testing) and the NP calculated from modal mineralogy, and NPR(MLA) is calculated from the AP and NP calculated from modal mineralogy. Samples are considered potentially net acid generating (PAG) if the NPR < 1, non-PAG if NPR > 2 and uncertain if NP/AP is between 1 and 2 (Price, 2009). Three of the samples from New Calumet are potentially acid-generating based on these criteria, one non-acid-generating, and two uncertain. Subsequent MLA analysis on five additional samples from Mt. Sinai (MS) confirmed NPRs near zero for all samples (Dongas 2013). The Nechalacho tailings all have high NPR and are thus non-PAG, whereas the Geco tailings have very low NPR and are classified as PAG.

Table 4 Neutralization Potential Ratios calculated from values obtained by MLA and static testing

Sample	NPR (MLA NP/ MLA AP)	NPR (MLA NP/ static AP)	NPR (static CaNP /static AP)
New Calumet			
GD-VEG1	8.63	6.8	7.47
GD-VEG2	1.2	1.12	1.08
GD-non VEG	1.45	1.11	1.08
BV-VEG	0.75	0.75	0.74
BV-nonVEG	0.67	0.68	0.64
MS-non VEG	0.24	0.2	0.14
Nechalacho			
UZLG	58	31	33.3
UZAG	41	44	33.3
BZMP	1103	59	33.3
BZAG	81	44	33.3
Geco			
Main Tailings Beach	0.001	0.000	0.02
Glory Hole	0.039	0.012	0.01
Red Pond	0.001	0.002	0.02
E3 Dam	0.000	0.000	0.00

CONCLUSIONS

Our results have shown that the NP and AP calculated from SEM-MLA results compare well with the results of static tests for three different mine sites, and the calculated NPR places the samples in the same category with respect to potential acid generation as the static testing.

NP and AP calculated from modal mineralogy provide several advantages over laboratory tests, notably the explicit consideration of Fe and Mn carbonates, and the identification and quantification of important acid-generating or acid neutralizing minerals that may be found in smaller amounts and with greater chemical accuracy than could be detected by other quantitative techniques such as XRD.

Particle size and liberation were not analyzed here, as they are not comparable between thin section analysis using MLA and static testing. MLA works on intact samples and preserves sample texture and particle size. Static testing utilizes subsamples crushed to <74 micrometers or <120 micrometers (depending on laboratory) and the crushing and grinding creates new particles and surfaces (Price, 2009). However, preliminary assessment of the degree of liberation of sulfide minerals in the New Calumet samples suggest that after 50 years of exposure, most of the remaining sulfide grains are very small (<50 micrometers) and locked within silicate grains, and likely unavailable to contribute to AP even in samples crushed for static testing. This would explain the persistently pH-neutral drainage at this site despite the NPR value between 0 and 1 for most samples.

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