Assessment of Acid Neutralization Rates from Site Rock for AMD Control

Roger Smart[1], Joseph Ciccarelli[1], Shengjia Zeng[1], Rong Fan[1], Jun Li[1], Nobuyuki Kawashima[1], Andrea Gerson[1] and Russell Schumann[2]

1. *Minerals and Materials Science and Technology, Mawson Institute, University of South Australia*
2. *Levay & Co Environmental Services, University of South Australia*

ABSTRACT

In principle, a sustainable approach to acid mine drainage (AMD) management and final closure should be to reduce the acid generation rate (AGR) sufficiently that the acid neutralization rate (ANR) can match the AGR from rock and tailings disposal. This approach has not yet been fully designed or achieved although some parts of the overall strategy, including surface passivation of sulfide minerals to reduce AGR, have been implemented. Most mine sites have non-value minerals capable of providing some neutralization of AMD. They are often not considered or surveyed in primary site ore assessment. Standard site assessment methods define total potential acidity (AP) or alkalinity (NP) of these materials, *e.g.* NP/AP ratio, but do not consider the **rates** at which acid generating and neutralising reactions may take place. It is these relative rates in disposal of rock and tailings wastes that determine whether acid and metalliferous drainage occurs. Methods are now available to assess the rates at which neutralization can be supplied from reactive silicate minerals (additional to carbonates) in on-site waste rock types. Knowledge of both the amount and the rates of acid generation and neutralization can be used to assess future acid rock drainage liabilities but, more importantly, to plan greenfield or operating disposal to make maximum use of these on-site materials. The complete definition of geochemistry and mineralogy of site materials can provide more effective and reduced-cost management of these mining wastes. The application of this approach is particularly pertinent for use with emerging waste emplacement construction techniques. Definition of ANR in non-acid forming wastes used for encapsulating acid generating wastes during controlled placement storage can potentially be used to define oxygen flux and moisture targets to achieve matching AGR. Examples where neutralising waste rocks have been identified and assessed at three sites in the AMIRA P933A project are discussed.

Keywords: acid neutralization; kinetics; silicates; site mineralogy

INTRODUCTION

A sustainable approach to acid mine drainage (AMD) management in pre-planning or in operation should be to use, where possible, geochemical resources available at the mine site to reduce the acid generation rate (AGR) sufficiently that the acid neutralization rate (ANR) can match the AGR from rock and tailings disposal before final rehabilitation. This matching of acid generation and neutralization rates in kinetically controlled processes is, in principle, the only sustainable option for long-term closure. This has not yet been fully designed or achieved, but this paper will describe this approach with case studies illustrating some parts of the overall strategy that have been implemented to date. There are real opportunities for improved practice using full geochemical and mineralogical assessment of site materials.

In principle, AMD management involves strategies, at any scale from molecular to site storage, to minimise the interaction of reactive sulfide surfaces with air and/or water. At the site engineering scale, minimisation and control strategies selected are influenced by a range of factors including climate, topography, hydrology, mine geology, geochemistry and mineralogy of waste rock, tailings and available neutralising materials. Our focus is on the geochemical and mineralogical part of this control. Implementation of this geochemical strategy requires, firstly, treatments to reduce the AGR sufficiently that the ANR can match the AGR (Gerson et al., 2014). Reduction of AGR (50-90%) by formation of silicate-stabilised iron hydroxide layers on pyrite has been addressed in other publications (*e.g.* Miller et al. 2009; Schumann et al. 2009; Zeng et al., 2013; Gerson et al., 2014). The addition of carbonates in covers and layers, where available as limestone or dolomite on site, is recognised as the primary geochemical method of AMD control in acid rock dumps (GARD Guide). Studies, both laboratory (Schumann et al. 2009; Huminicki and Rimstidt 2009; Nicholson et al. 1990) and field (Li et al., 2012; Li et al., 2011; Miller et al., 2009), have shown that under neutral pH conditions maintained by added carbonates, pyrite oxidation rates, *i.e.* AGR, can be reduced by more than one order of magnitude. The mechanism for the reduction in sulfide oxidation rate is found to be through formation of continuous, coherent iron oxyhydroxide coatings which develop on the surface of the reacting sulfide and reduce oxygen diffusion rates to the surface (Huminicki and Rimstidt 2009; Nicholson et al. 1990). The nature and stability of these surface passivating layers has been extensively investigated in our research (Smart et al. 2010; Schumann et al. 2009). Under conditions of pH>6, multi-layered coatings of amorphous iron oxyhydroxide develop notably as stable, thin, continuous and conformal coatings often overlaid with a thicker (1-2 μm) coating of (semi)-crystalline goethite-like material. In field samples, the amorphous oxyhydroxide pyrite-passivating layer normally contains significant (5-10 mol%) silicate content. Where clay minerals are present in the waste, strongly adhering particles with micaceous texture can form a further external armouring layer (Miller et al., 2009).

The roles of pH and dissolved silicate in stabilising these passivating layers are critical. Pyrite oxidation rates are reduced when the pH is maintained above 6 and when the surrounding solution contains dissolved silicate (10–20 mg/L as Si) (Zeng et al. 2013). In the presence of dissolved silicate, the iron oxyhydroxide layer formed during pyrite oxidation retains its amorphous, conformal structure, while in the absence of silicate, conversion of this layer to the more-crystalline and less-passivating, separate goethite-like crystals occurs. These differences are illustrated in Figure 1. If initial alkaline amendments are made so that these passivating layers are formed and maintained then, for wastes with low to moderate AGR containing some reactive silicates, AGR may be sufficiently reduced (more than 90%) to enable matching by silicate ANR in the long-term. This sulfide passivation, with the use of reactive silicates from site rocks, forms the first part of the geochemical strategy for sustainable AMD management.

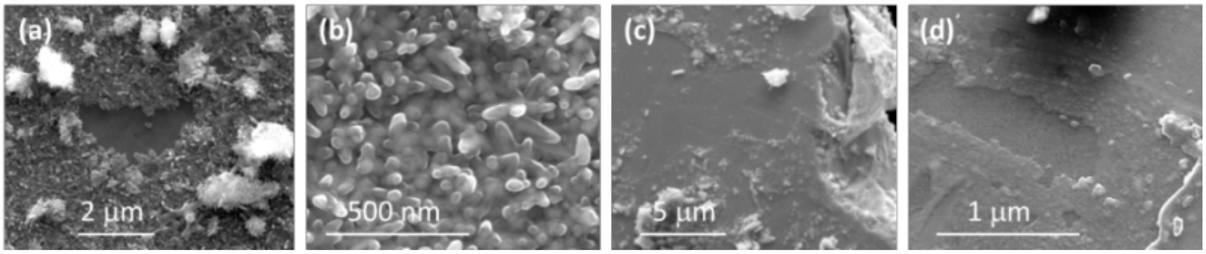

Figure 1 Evidence of the effect of the presence of silicate on pyritic surface layers at neutral pH. (a) and (b) pyrite surfaces immersed in calcite saturated water for 160 days showing clear crystalline overgrowths; (c) and (d) pyrite immersed under the same conditions with added silicate (20 mg/L Si) showing clear difference in surface layer morphology and significantly reduced pyrite oxidation (Zeng et al., 2013).

A specific example of the use of on-site alkaline materials for amelioration of AMD from previous rock dump fills has been provided by Cook et al. (2008). Re-mining of the Buffalo Creek coal seam site (Virginia, USA) to obtain coal from lower in the geologic column exposed new overburden, including an alkaline sandstone, that was used to add covers to the existing valley-fill AMD rock dumps. Drainage from the fills had resulted in high acidity/high metal (pH 3.5–4.5 with 100–200 mg/L $CaCO_3$ acidity) in the sub-watersheds. Addition of the sandstone covers improved water in the main stream of each drainage area from the fills. The water quality in the tributaries of Laurel Creek dramatically improved to pH 6.2 and acidities of <1 mg/L. Placement of alkaline material, recognised in the new mineralogy, into the extended valley fills eliminated the need for chemical treatment. With more than 10 years of data, it is apparent that there is high probability for the long-term success of the amendments.

A second example is the successful implementation and testing of on-site amendments to AMD from the historic B-Dump in the Savage River Rehabilitation Program (SRRP), a cooperative arrangement between the Tasmanian Government and current operators Grange Resources. Studies at this site have already been fully documented in previous ICARD meetings (Hughes et al., 2009, Hutchison et al. 2009, Li et al., 2011, Li et al., 2012). This used a water-shedding compacted top cover (from graded material) with over-dumped side covers and a base-dumped flow-through barrier comprised of chlorite-calcite schist waste material from the site. The covers have effectively reduced the overall AGR within the dump by about 43% over the past five years. Alkalinity from the covers has been migrating down into the acid-forming waste and forming passivating layers of silicate-stabilised iron oxy-hydroxide verified on pyrite grains (Li et al. 2011). Sulfate, Al and Cu flux in Main Creek have decreased by 50% after B-dump was capped. The discharge from Main Creek to the river has neutral pH, low sulfate (218 mg/L) and metal concentrations, *e.g.* Al 200 μg/L and Cu 30 μg/L, that are now below the SRRP fish target (Hughes et al. 2009).

In the context of requirements for implementation of the geochemical approach, a critical second requirement remains, however, to recognise, **measure** and control relative rates of acid neutralization from different waste mineralogies available on site. One of the significant inputs employed for the SRRP remediation is a comprehensive report (Thornett, 1999) on the rock types and mineralogy of the site prepared for the previous owners Australian Bulk Minerals. This identified a large variety of potentially neutralising mineralogy including the abundant chlorite-calcite schists with some dolomite, magnesite, talc-carbonate schist, dolerites and reactive silicates in mafic rocks, amphibole-chlorite-albite schists, serpentinites and possibly tonalite. This was recognised by site personnel and Rumble (2005) in planning of the amendments and provided the basis for the successful remediation.

Hence, there are two requirements that can potentially improve current practice in AMD control. The first is the need for detailed mineralogy, additional to standard AMD classification testing, of all site lithology, not just value lithology, preferably in greenfield assessment but also in operating and legacy sites. This enables a first-level estimate of both AGR and ANR for different lithologies based on the mineralogy alone (Ciccarelli et al., 2009, Miller et al. 2010). The AGR from the waste rock in passivated form at pH 6 can be estimated from well-established oxygen consumption and sulfate release methods (*e.g.* Hollings et al., 2001; Sracek et al., 2006). Associated with this should be

ANR carbonate and ANR non-carbonate (ANRnc; mainly reactive silicates) testing of potentially neutralising rock types using the dissolution cell and mineralogical methods to be described. These estimates can provide a reasonable basis to assess whether the ANR can match the reduced AGR at source. The second requirement is to plan to use these materials in layer dumping with AMD wastes and in cover design together with the engineering considerations. Short-term costs of this approach are likely to be greater than current practice but with the very high costs of closure and on-going management and liability in retained capital (*e.g.* several hundred million dollars at Newmont, Dowd, 2005), it may ultimately be more cost-effective. This comparison has not yet been fully evaluated but is clearly worthy of further development for sustainable AMD control.

METHODOLOGY

Current AMD test methods use static acid base accounting (ABA) assessment of potential acidity and neutralization together with long-term kinetic leach columns (KLC) usually requiring years to give reliable data (AMIRA/EGi ARD Test Handbook, 2002; MEND, 2009). The methodology required to use the our kinetic approach is a combination of AGR and ANR measurements (both in total mg H_2SO_4/kg/week or g H_2SO_4/tonne/week) on a time scale that allows snap-shot assessment of the ARD dump or tailings material prior to and during evolution of the AMD profile. The well-established oxygen consumption and sulfate release methods (*e.g.* Hollings et al., 2001; Sracek et al., 2006) on representative samples can provide reliable cross-checks and estimates of AGR at any point in this evolution. The requirement for short-term cross-checks and estimates of ANR can be met by a combination of dissolution rates from the sample mineralogy with dissolution cell measurement of released cations at the measured sample pH.

The mineralogical ANR estimates combine mineralogical assessment (Rietveld XRD, QEMSCAN or MLA) with dissolution rate data of individual minerals. The non-iron carbonate content of the mineralogy can be assumed to provide ANR for the equivalent AGR, *i.e.* at the same mg H_2SO_4/kg/week since their reaction rates exceed pyrite oxidation rates (Blodau, 2006). It is the additional neutralization from reactive silicate minerals that requires assessment. Eary and Williamson (2006) combined mineral dissolution rate data from Palandri and Kharaka (2004) with solution speciation modelling to examine dissolution of a number of theoretical rocks, with silicate mineralogy ranging from silicic through to mafic and containing 0–3 wt.% pyrite. Their results suggested that anorthite feldspar and some mafic minerals such as forsterite, augite and hornblende may dissolve quickly enough to neutralise acidity from pyrite oxidation. For instance, anorthite gives measurable short-term neutralization in the ABA Sobek test with estimated ANC of 10 kg H_2SO_4/t (AMIRA/EGi, 2002; MEND, 2009). Abundant chlorite minerals have also been shown to mediate acidity in both the Waite Amulet (Jambor et al., 1999) and Savage River (Li et al. 2012) examples. A similar mineralogical approach was used by Miller et al. (2010), wherein silicate ANR derived from mineral dissolution rates were compared with rates calculated from leachate analysis of solutions obtained from long-term kinetic leach column tests from which any carbonates had been previously exhausted. They found good agreement between the rates derived from mineralogy and those obtained from leach column tests when the pH was below 3. However at pH>4, ANR values calculated from mineralogy generally exceeded those derived from leach column testing (over 2-10 years) based on assays for metal cations (Na, K, Ca, Mg and Al) released in neutralization reactions as in the method of Paktunc (1999).

The amounts (mmol) of each cation in the 4-weekly leachate are determined from the assayed concentrations and the leachate volume. Thus (static) non-carbonate acid neutralising capacity ANCnc (mg H_2SO_4/kg) was determined from equation (1) with *Na, K, Mg, Ca* and Al in mmol; the factor 49 converts mmol H^+ ions to mg H_2SO_4; *m(kg)* is the mass of the sample in the column. *e.g.*

$$ANCnc = [(Na + K) + ((Mg + Ca) \times 2) + (Al \times 3)] \times 49 / m(kg) \qquad (1)$$

For solution assays collected over specific time periods, usually 4 weeks, this can be represented as a rate, *i.e.* ANRnc in mg H_2SO_4/kg/week (or kg H_2SO_4/t/yr) for comparison with AGR measurement from sulfate release. The reason for the lower ANRnc measured by this method at pH>4 is likely to be due to the removal of some of these ions by secondary mineral formation particularly gypsum.

To improve the comparison with mineralogy, a short-term kinetic dissolution cell test to measure the ANRnc was developed using an initial pH-controlled flow-through method from which assays for metal cations (Na, K, Ca, Mg and Al) can be obtained after a steady-state dissolution rate is attained (usually 2 weeks) and ANRnc calculated as in Eq 1. This experimental procedure has been previously described in Ciccarelli et al. (2009). A summary is given here. The sample is first reacted with pH 2 HCl to remove all carbonates (verified with XRD and carbonate assay). The reaction cell is connected to an input reservoir at the pH of the original site sample and this solution is pumped through the reaction cell by a peristaltic pump with the sample in a holder with 0.45 μm filter papers placed above and below the sample to restrict sample loss. Leachate discharged from the exit port near the top of the reaction chamber is collected in output reservoirs for analysis of dissolved metals. Each of the collected effluent solutions is daily transferred into a sealed bottle pre-purged with Ar gas. The continuous flow-through tests are run under standard temperature (25°C) and atmospheric pressure conditions at an effective flow-rate of 40-50 mL per day, calibrated daily.

An example of this procedure applied to a fresh Savage River chlorite-calcite schist after carbonate removal is shown in Figure 2. This delivered an ANRnc of 480 mg H_2SO_4/kg/week at influent pH 4 and 90 mg H_2SO_4/kg/week at pH 5 both with effluent pH 7 at steady state. In the dump, the effective pH (as free proton concentration) close to the pyrite surfaces is likely to be below that in the bulk solution generating faster dissolution of reactive silicates adjacent to the pyrite. This can be compared with sulfate AGR from the B dump, to which this schist was added, of 10 mg H_2SO_4/kg/week in the seep at the toe of the dump (Li et al., 2012). The initial fast dissolution of Mg and later K may be due to ion exchange at high energy surface sites in silicate fines, *i.e.* chlorite and minor anorthite (Brantley and Chen, 1995) remaining after the pH 2 pre-treatment to remove carbonates. This initial greater reactivity can roughly double the effective initial ANRnc. More detail and other examples can be found in Ciccarelli (2012).

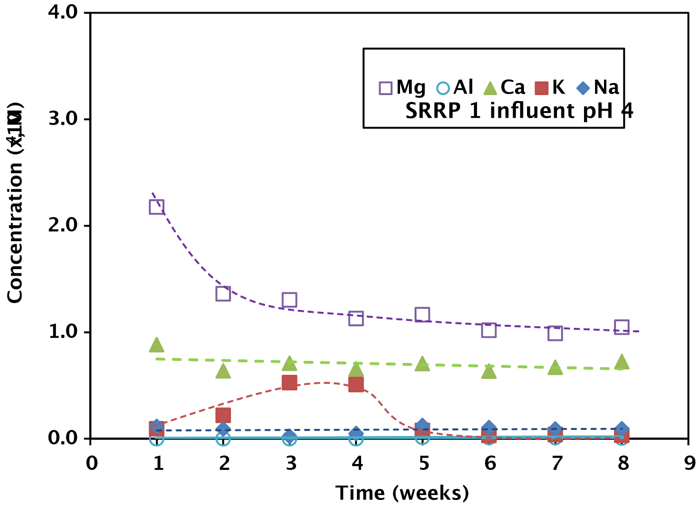

Figure 2 Major cation concentrations from the kinetic dissolution cell test of a Savage River chlorite schist waste sample at a flow rate of 4 L/kg/day and an influent solution pH of 4.

RESULTS AND DISCUSSION

Two other examples of the recognition and measurement of ANRnc in waste rocks potentially offering site treatment options not previously envisaged are discussed here.

BHP Billiton Iron Ore (BHPBIO)

A BHPBIO case study investigated the strategy of pyrite passivation and AMD reduction through the use of on-site ANR-generating wastes to minimise AGR in overburden storages at Mt.

Whaleback, Pilbara, Western Australia (Gerson et al., 2014). A Jeerinah shale and the highly reactive Mt. McRae shales were used as AGR-generating materials in kinetic leach columns (KLC). For ANR generation, a Jeerinah dolerite gave promising results. The mineralogy (determined from Rietveld XRD, QEMSCAN, bulk assay and carbon analysis) contains low carbonates (calcite 2-3 wt.%, ankerite 0.5-1 wt.%) with abundant reactive silicates including chlorite >30 wt.% (≈20 wt.% poorly crystalline not seen in QEMSCAN but not in XRD), anorthite 5 wt.%, tremolite 9 wt.% with a large amorphous fraction, 54 wt.%, in XRD probably consisting of chlorite-like material (from bulk assay reconciliation). The ANC of the carbonates from assay is around 30 kg $CaCO_3$/t but the measured ANC (modified Sobek test, AMIRA/EGi, 2002) of the dolerite is 47 (pH 2.4) to 84 (pH 1.2) kg $CaCO_3$/t depending on the final pH after the acid aliquot choice. Calcium concentrations were independent of initial pH suggesting that calcite and ankerite were quantitatively dissolved in each case with the remaining silicates (principally clinochlore chlorite) present in the dolerite contributing between 17 and 54 kg $CaCO_3$/t, depending on the *in situ* reaction pH. This initial characterisation is valuable for short-term neutralization but, given the abundance of silicates in most mine waste materials, it is the **rate of dissolution** rather than total concentration which determines the ability of silicates to buffer acidity resulting from sulfide oxidation. The ANRnc estimate from the mineralogy at pH 4 is 395 mg H_2SO_4/kg/week and at pH 5 is 81 mg H_2SO_4/kg/week suggesting significant neutralization as well as potential AGR reduction via pyrite passivation.

Blending 10 wt.% dolerite into the Jeerinah shale resulted in leachates characterized by neutral pH and a slight excess of alkalinity with low dissolved metal concentrations (not detectable in most cases). These results indicate that co-disposal of Jeerinah shale with Jeerinah dolerite, will substantially reduce the risk of drainage containing elevated levels of metals emanating from waste emplacements in which these materials are stored. Initially, dolerite was evaluated as a cover material for Mt. McRae shale. Results demonstrated that Jeerinah dolerite failed to supply sufficient alkalinity to keep the pH high enough to bring about pyrite passivation. However, leachates from the column tests in which Jeerinah dolerite was added to Mt. McRae shale as a base layer or as a vertical interlayer were characterized by: pH>7 effluent; no acidity (slight excess of alkalinity); low salinity (conductivity ≈100-200 μS/cm); low dissolved metal concentrations (non-detectable in most cases) and unexpectedly low sulfate, Ca and Mg concentrations. It appears that, under these test conditions, dolerite provides a preferential flow path for solution in the presence of Mt. McRae shale and could potentially be used in waste rock emplacements to channel acidic water flowing from ARD waste and to treat the seepage by removing both acidity and salinity.

Using the analyses for AGR and ANR, the evolution of W39 Terrace Dump at Mt. Whaleback has been characterized using water monitoring data collected at the AMD dam. The water samples had an average pH of 4.2 with high Mg (273 - 1210 mg/L), Al (up to 98 mg/L) and Si (up to 96 mg/L) concentrations suggesting that dissolution of (Mg, Al, Si)-containing chlinochlore has been the major buffering mechanism within the dump. The calculated AGR/ANRnc (ANR non-carbonate) data estimated from each of the AMD dam water samples fall within the values measured for long-term KLC tests, suggesting that the estimates made for the dump are quite reasonable. The average ratio of AGR/ANR of 1.02 indicates that the AGR was fairly close to the ANR provided by silicate minerals within the dump in 2008-2009 and that further reduction in AGR from the dump should give increased water pH in the AMD dam.

Hidden Valley Joint Venture (PNG)

A review of all previous reports on Hidden Valley (HV) waste rocks including waste classifications, site and consultants' KLC tests was completed (Gerson et al., 2014). The lithologies of the main rock types at the site are granodiorite (GD) and metasediment (MS) (the latter hosting most of the ore). The metasediment lithologies are all AMD-generating. Both lithologies can contain significant pyrite, but the HV granodiorite (HVGD) mineralogy contains calcite (11 wt.%) with the majority silicates as reactive plagioclase (anorthite with albite), chlorite, muscovite, orthoclase and quartz as well as significant pyrite content (4 wt.%). The overall ANC of the HVGD is high, *i.e.* 133 kg H_2SO_4/tonne and the ANRnc estimate from this mineralogy at pH 4 is 128 mg H_2SO_4/kg/week and at pH 5 is 30 mg H_2SO_4/kg/week delivering significant neutralization.

To test whether passivation of pyrite and AGR-ANR matching can be achieved with HVGD, KLC tests of a composite metasediment sample (2 wt% pyrite) with HVGD surface layers or in recycle from flow-through HVGD sub-columns were set up. The ANR (from effluent analysis) of the HVGD surface-layer column is initially about 50% of the AGR but the ANR matched the rapidly decreasing AGR at around 70 weeks, as the fine pyrite was depleted, but with pH still near 2.5. In the water recycle system, the effluent subsequent to both the MS composite column and the sub-column containing HVGD maintained neutral drainage and provided alkalinity up to 56 weeks. In recycle of this effluent to the MS composite column, the pH of the main recycle MS composite column in this system was only slightly increased but the acidity of the effluent was significantly lower (≈50 %) than for the MS composite control column after 16 weeks. Although recycle from the HVGD sub-column was not able to provide sufficient ANC to match the AGR of the MS composite main column, the results showed that the HVGD sub-column neutralised the acidity from the MS composite effluent in low–flow conditions. This suggested that HVGD may be useful as a flow-through reactive barrier (similar to that used at the Savage River Mine) for low-flow seeps to reduce current lime additions. To test this proposition, the effluent from a 2 kg batch of MS composite was used to flush a 1 kg HVGD sub-column in standard KLC flush conditions. The pH of the effluents from the HVGD sub-column maintained neutral to slightly alkaline pH across 60 weeks. Most of the toxic metal ions were precipitated and were below detection in the HVGD effluent except Mn. The ANR (derived from effluent concentrations) of the HVGD sub-column was slightly greater than the AGR of the MS composite up to 16 weeks but became slightly less than the MS AGR beyond 16 weeks. However, HVGD ANR is underestimated due to precipitation of gypsum (*i.e.* loss of Ca) in the HVGD sub-column, so that it is likely that the actual ANR of the sub-column exceeds the AGR of the MS column across more than 80 weeks. SEM analysis confirmed that the pyrite in the HVGD sub-column was passivated under these conditions.

This sample of HVGD had relatively high pyrite content (≈4 wt.%), twice as much as the MS composite material. However, the neutralization rate of the calcite/anorthite/chlorite in the HVGD was sufficient to enable formation of passivated pyrite surfaces within the HVGD reducing the pyrite oxidation rate. These results suggest that testing to locate HVGD lithology with lower pyrite content outside the MS areas may provide greater ANR for potentially matching AGR from MS wastes in co-disposal or layer dumping.

CONCLUSION

Most mine sites have non-value minerals capable of providing some neutralization of acid mine (rock or tailings) drainage often not considered or surveyed in primary site ore assessment or AMD dump construction. Standard assessment methods define only total acidity potential (AP) or neutralization potential (NP) of these materials, *e.g.* NP/AP ratio. This does not consider the **rates** at which acid generating and neutralising reactions may take place, although it is these relative rates in disposal of rock and tailings wastes that determine whether acid and metalliferous drainage actually occurs. Methods are now available to assess both AGR and ANR from both carbonates and reactive silicate minerals in on-site waste rock lithologies. The ratio of AGR/ANR can be followed using these assessments. These short-term, snap-shot methods can be applied at any point in the evolution of the dump or tailings from greenfield planning to operating dumping to assess current and future acid rock drainage liabilities and to make maximum use of these on-site materials. The complete definition of geochemistry and mineralogy of site materials can provide more effective and reduced-cost management of these mining wastes. Examples where neutralising waste rocks have been identified and assessed at Savage River (Tasmania), BHP Billiton Mt. Whaleback and Hidden Valley (PNG) have illustrated the value of this approach.

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