WHY VARIABLE OXIDATION RATES ARE NEEDED FOR THE PREDICTION OF AMD FROM DYNAMIC WASTE ROCK DUMPS

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ABSTRACT

Much effort, time and capital, is expended at mine sites on predicting the potential and quality of acid and metalliferous drainage (AMD) originating from waste rock dumps (WRDs). For many sites, WRDs represent the largest potential source of AMD and subsequently more often than not, reflect the highest risk associated with AMD management and successful mine closure. Compounding this is the dynamic nature of a WRD, which increases the complexity of AMD predictions when incorporating the shifting fluxes of water, gas, and temperature.

Typical AMD predictions for a given WRD will involve combining static and kinetic testing data with net percolation calculations to estimate potential AMD loads, which can also include geochemical modelling of the data. Kinetic data which is traditionally collected from industry standard humidity cell, free draining leach column, or oxygen consumption type tests, are applied to estimated quantities of sulphide-bearing waste rock to predict the quantity of acidity and/or contaminants that are produced with time. However, the oxidation rates applied to these predictions are often fixed, and do not allow fluctuation as water and air availability change within the WRD in response to climatic conditions.

A new kinetic method has been developed that allows dynamic oxidation rates to be measured in parallel with fluctuating moisture, temperature, and airflow. Oxidation rates have been measured for black shale waste rock samples, collected from WRDs within the Pilbara, over a 23 month period in conjunction with replicate industry standard free draining leach columns. Rates were generally significantly faster than their free draining leach column replicates and were found to fluctuate by more than an order of magnitude as the system responds to wetting events, which were applied to achieve liquid to solid ratios similar to those expected within the Pilbara. This paper presents the findings of the completed program.

1.0 INTRODUCTION

Acid and metalliferous drainage (AMD) is recognised internationally and within Australia as one of the most significant and difficult environmental issues facing mining operators and regulators (Egiebor and Oni 2007; Watkins 2007). A key reason for its importance is that AMD has the ability to cause significant ongoing pollution of the surrounding environment that could potentially persist for hundreds of years. As such, AMD has been highlighted in mine closure guidelines by Western Australian regulators as one of the key environmental issues relevant to mine closure (Department of Mines and Petroleum 2015).

For sites requiring disturbance of reactive waste rock, predicting AMD contaminant loads from existing and future waste rock dumps (WRDs) is therefore a key step to understanding a site's

closure liability with respect to AMD. However, predicting contaminant loads from WRDs, as well as tailings storage facilities (TSFs) and pit voids (although not a focus of this paper), is very complex when considering the dynamic forces that play a significant part in the formation, mobilisation, and release of AMD. Contaminant load predictions are then further complicated through the incorporation of parameters determined from standard kinetic testing methods that largely do not give consideration to the dynamic forces within a WRD.

1.1 Dynamic Waste Rock Dumps

There are several factors influencing AMD risk such as waste geochemical and physical properties, WRD geometry and construction method, and climate (Pearce et al. 2016). More often than not, the focus of AMD risk and characterisation assessments focus on the geochemical and climatic factors with very little consideration to the WRD internal structure (Pearce et al. 2016).

How a WRD is constructed, as well as the physical properties of the waste, will determine how accessible reactive minerals are to water and oxygen for participation in chemical reactions. Depending on climatic conditions (e.g. wet versus dry season), the supply of water and oxygen to these reactive surfaces will vary throughout a given year. Therefore, if reactant supply is variable, the generation of products will also be variable. And like the variable generation of contaminant products, the mobilisation and release of these products will be primarily influenced by the dynamic conditions within the WRD, largely set by the relationship between its internal structure and climatic conditions.

1.2 Typical AMD Prediction Methods

Sulfide (or pyrite) oxidation rates for acid generating waste materials can be used as a proxy for chemical weathering (Sapsford et al. 2009). Oxidation rates can then be used to estimate the rate of acidity/contamination generation resulting from chemical weathering. The rate of acid generation is a key parameter that mining operator's measure to assist the development of effective waste management plans such as WRD construction method, appropriate cover systems design, and contingency water treatment strategies. The two main methods of estimating sulfide oxidation are the sulfate release method and the oxygen consumption method (Elberling 1993; Elberling et al. 1994; Hollings et al. 2001; Kempton et al. 2010):

- Sulfate release method Utilises the relationship between measured sulfate production and stoichiometric sulfide (usually pyrite) consumption to estimate the oxidation rate (Elberling et al. 1994).
- Oxygen consumption method Estimates the sulfide oxidation rate by measuring the decrease in oxygen concentration over time (Hollings et al. 2001).

The sulfate release method is the most common method utilised by industry (e.g. humidity cell and free draining leach columns) and utilises analytically determined sulfate concentrations from column leachates (AMIRA 2002 and ASTM 2012). Without mineralogical data specifying what sulfide minerals are present in a sample, pyrite is often assumed to be the dominant sulphide, as it is the most common (Sapsford et al. 2009). The mass of sulfate released over time can then be related to the quantity of pyrite oxidised over time by the stoichiometric relationship in Eqn. [1] (Sapsford et al. 2009).

[1]
$$FeS_{2(s)} + 3.5O_{2(g)} + H_2O_{(l)} \rightarrow Fe^{2+}_{(aq)} + 2SO_4^{2-}_{(aq)} + 2H^+_{(aq)}$$

An important concept to understand when applying the sulfate release rate is the distinction between the release and production rates with the latter (not the former) representing the oxidation of pyrite (Sapsford et al. 2009). If not all generated sulfate is released then the oxidation rate determined from the sulfate release rate will underestimate the actual rate. To ensure production equals release, high flush volumes unrepresentative of field liquid to solid ratios are applied in the laboratory with steady-state release rates being typically used in AMD load predictions (Maest and Nordstrom 2017). This over-flushing (volume and regularity) then negates any possibility for the stored contaminant dominant systems observed in semi-arid regions to be replicated in the laboratory (Price 2009; Maest and Nordstrom 2017). The small size of the columns also discourage leachate concentrating effects resulting from increased pore water residence time.

1.3 Alternative Kinetic Testing Method

A new method using advanced customisable leach columns (ACLCs) was developed to address limitations in existing industry standard kinetic testing methods (Pearce and Pearce 2016). Limitations addressed include:

- Flexibility to incorporate variable air supply (WRD gas flux)
- High liquid to solid ratios and short leachate residence times (WRD water flux)
- Low or fluctuating experimental temperatures, and
- Low sample volumes and small particle size.

In addition to typical variables collected from standard kinetic testing, the developed ACLCs allow airflow, liquid to solid ratios, and system temperatures to be calibrated to site conditions. Predicted or measured internal airflows within WRDs for various construction methods can be replicated using continuous air supply and flow regulators. Wetting regimes, from a liquid to solid ratio perspective, can be customised to better reflect field net percolation rates. The ACLC enclosure is a temperature controlled room with the flexibility to be operated between 6–50°C (Fig. 1). The larger capacity columns (1 m height; 150 mm diameter), which can take up to 25 kg material, accommodate larger particle sizes (up to 100 mm) in comparison to standard kinetic methods, facilitate longer leachate residence times that promotes contaminant concentrating effects observed in the field. Dynamic system parameters temperature, pressure, humidity, airflow, oxygen concentration, and carbon dioxide concentration are continuously logged so that dynamic oxidation rates dependent on both airflow and water availability can be measured.



Fig. 1 ACLC enclosure with 16 operating columns.

2.0 COLUMN CONFIGURATION AND OPERATION

This paper discusses the findings from four replicate ACLCs constructed from one composite sample. The four replicate columns investigated the effects of variable air and water over a period of 12 months. To allow comparison to industry standard, free-draining leach columns, duplicate columns using the AMIRA 2002 method were constructed from the same composite material. However, as the free-draining leach columns do not facilitate variable airflow rates, a duplicate column and a high temperature column were substituted against the ACLC low and high air columns. The four replicates were named as per the following:

•	Replicate 1:	ACLC – Control Column	AMIRA – Control Column
•	Replicate 2:	ACLC – Low Air Column	AMIRA – Control Column Duplicate
•	Replicate 3:	ACLC – Low Water Column	AMIRA – Low Water Column
•	Replicate 4:	ACLC – High Air Column	AMIRA – High Temperature Column

The 100 kg composite sample was prepared from 12 waste rock primary samples collected from a WRD sonic drilling program. The WRD investigation included drilling of more than ten boreholes into four WRDs at an iron ore mine in Western Australia's Pilbara region. The primary samples were majority black shale with the composite sample having the chemical and physical characteristics listed in Table 1.

Table 1. Chemical and physical properties of the composite sample on a weighted average basis.

Parameter	Units	Value
Total Sulfur	wt%	2.89
Sulfate Sulfur	wt%	1.44
ANC	kg H₂SO₄/t	10.3
Total Carbon	wt%	2.8
^A PSD <2.36 mm	wt%	32.0
^A PSD +2.36 mm	wt%	14.8
^A PSD +4.75 mm	wt%	11.2
^A PSD +6.70 mm	wt%	23.4
^A PSD +13.7 mm	wt%	18.6

^APSD not applicable for free draining leach column duplicate samples as method requires crushing of samples to <4 mm.

2.1 ACLC Air Flow Regime

Air flows were adjusted within the first four months due to initial flow regulators not providing sufficiently low air flow control. Air flows for the ACLC Control, Low Air, and High Air Columns were set using airflows estimated from field oxygen sensor data coupled with particle size data collected from the sonic drilling program. Air flow for the Low Air Flow Column was set at 0.008

L/min and represents lift heights of approximately 10 m. The Control Column was set at 0.06 L/min which reflected lift heights of 20-30 m. The High Airflow Column was set at 0.2 L/min which reflects an unconstrained WRD with respect to oxygen supply. Note that the airflow used for industry standard humidity cell tests is 2.5 times greater than this unconstrained airflow (ASTM, 2012).

2.2 ACLC Wetting Regime

Distilled water was added to each of the columns at the time of column loading. Water was added so that the resulting moisture content was within the range observed in the field (8-12 wt%) with the exception of the low water replicate which was loaded with two thirds the volume of the Control Column. To avoid excessive addition of water to the columns as to achieve liquid to solid ratios as close as practicable to moisture conditions observed in the field, matric potential was monitored in each of the columns. This examination of internal drying rates and comparison with field matric potential resulted in the first application of water after 10 months of operation. A second flushing event was applied to the ACLCs at the completion of the program to determine potential pore water concentration of key contaminants. This wetting regime closely reflected the typical Pilbara pro-longed drying cycle before a large cyclonic wetting event.

2.3 Free Draining Leach Column Wetting Regime

The intention of the weekly wetting applications is to wet the sample with minimal leachate loss during these non-flushing weeks. Initially 0.2 L of distilled water was added during the wetting weeks with no leachate observed. This wetting volume was increased to 0.3 L which generated a very small leachate volume in some columns (0.15 L for the Low Water free draining leach column). The volume applied for the monthly flushing event was 0.8 L for all free-draining leach columns accept the Low Water replicate which had 0.4 L applied.

2.4 Temperature Regime

The ACLC enclosure is a temperature controlled room and was operated at 35–36°C at all times. This temperature range is the recorded average annual internal temperature of instrumented WRDs at the site investigated. This regime is significantly different to that applied to the free-draining leach columns. The drying phase for the free-draining leach columns is induced by heat lamps with 150W bulbs automatically operated for eight hours per day for five consecutive days each week. The heat lamps are used to simulate air temperature, during the eight hours of heat lamp operation, of between 30 and 35°C.

3.0 RESULTS

The following section presents selected results for the four replicate ACLCs over a 12 month period as well as key comparison points to the free draining leach columns.

3.1 Oxidation Rates and Airflow

The intrinsic oxidation rate (IOR) can be considered the overall rate of oxygen consumption reactions within the column. That is, the IOR incorporates oxygen consumed from both pyrite and organic carbon oxidation reactions. Parallel carbon dioxide measurements allowed the conservative calculation of oxygen consumption by organic carbon, that is, all measured carbon dioxide was assumed to be a product of Eq. [2]. Carbon dioxide release into the column air exhaust through the dissolution of carbonate was considered negligible with respect to this specific assessment of replicate columns. For reference, the measured organic carbon oxidation rate (COR) was generally greater than an order of magnitude slower than the pyrite oxidation rate

(POR) when calculating POR from Eq. [3]. Therefore, for the purpose of this assessment, IOR was assumed to be a reasonable proxy for POR.

[2]
$$CH_2O + O_2 \rightarrow CO_2 + H_2O$$

$$[3] POR = IOR - COR$$

Figure 2 presents the differences observed between IORs when varying water and air supply in comparison to the Control Column. The IORs for the Low Air Flow Column (7.7E-8 to 9.3E-7 kg $O_2/m^3/sec$) are approximately an order of magnitude lower that the control column over the same period (2.3–4.1E-6 kg $O_2/m^3/sec$). This indicates that airflow rates are a key limiting factor for oxidation rates and therefore acidity production. Initially, the Low Water Column did not have a meaningful lower IOR relative to the Control Column indicating that controlling moisture content may not likely influence oxidation rates. However, as the Low Water Column approached suction values of 900–1,000 kPa (June-July 2015), the IOR dropped considerably and remained low until the next wetting event. The positive correlation between suction and IOR up to the 900–1,000 kPa range indicates that loss of moisture can accelerate the IOR until the point at which moisture becomes the limiting factor in the oxidation reaction. The High Airflow Column had a significantly faster IOR than the Control Column demonstrating the positive influence of air supply on oxidation rates. The maximum IORs for the High Air Flow Column were approximately 2.0E-5 kg $O_2/m^3/sec$ which is almost five times the measured IOR for the Control Column.

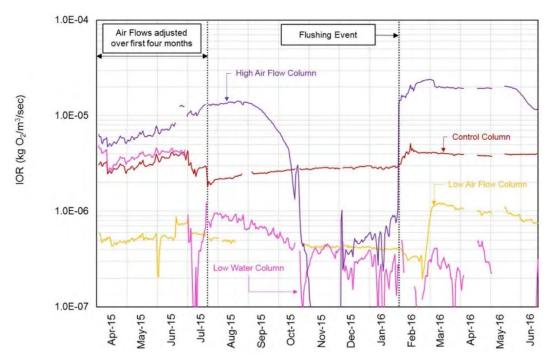


Fig. 2 IORs for each replicate column.

Figure 3 shows the predicted normalised pyrite consumption using the dynamic oxidation rates on a monthly basis for the Control, Low Air, and Low Water Columns plotted against field data collected from WRD installations from iron ore mines within the Pilbara. Using the monthly IORs

measured allows incorporation of dynamic oxidation rates that are changing as the WRD internal conditions respond to climatic conditions (e.g. drying, wetting, change in airflow).

The field data plotted on Fig. 3 represents two construction alternatives (10 m vs 20-30 m lifts) that subsequently result in two different air permeability's. The estimated air permeability's from these two different placement methods were used to set air flows for the ACLC's, specifically the Control and Low Airflow Column. The figure demonstrates one of the most significant findings of the ACLCs in that they were able to closely replicate oxidation rates observed at the field scale when applying the measured dynamic oxidation rates for long-term AMD predictions.

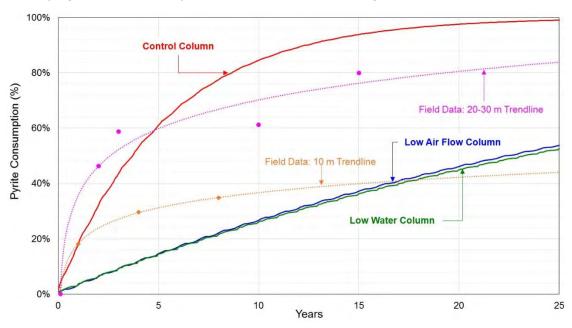


Fig. 3. Predicted pyrite consumption using monthly IORs measured within each of the replicate ACLCs and material geochemical data.

3.2 Oxidation Rates and Drying

Suction data for the replicate columns are presented in Fig. 4. An increase in suction can be inferred to represent drying of the materials in the columns. That is, as moisture is removed from the system suction pressure will increase. Moisture is typically removed from larger pore spaces initially which is indicated by the first inflection change in Fig. 4. (e.g. April to July for the Control Column). Once moisture removal from smaller pore spaces begins, a second inflection in the drying curve can be seen (e.g. September for the High Air Flow Column). Conversely, suction decreases rapidly in all columns after the flushing event. Because all the materials in each column have a unique PSD then the drying curves will be different and as such suction can be expected to occur at both a different rate and to a different final value in each of the columns.



Fig. 4. ACLC suction pressures as measured by installed MPS2 sensors for the four replicate columns. Vertical dotted line represents the 10 month wetting event. Data values (kPa) presented refer to negative pressure.

Figure 5 presents suction and IOR data for the High Air Flow Column and highlights a key finding from the ACLC columns not able to be observed with conventional kinetic testing procedures. The High Air Flow Column produced an initial IOR of 4.8E-6 kg O₂/m³/sec, this rate increased steadily to a maximum of 1.4E-5 kg O₂/m³/sec after four months which was correlated by a simultaneous rise in suction values as the material in the column experienced drying as a result of internal advective drying. Following reaching the maximum IOR and at a suction of 900 kPa, the rate decreased rapidly by almost an order of magnitude to 3.7E-7 kg O₂/m³/sec, before rebounding sharply to just below 2.0E-5 kg O₂/m³/sec, where it remained constant for the following four months. When water was added to the column in January 2016, the IOR increased rapidly corresponding with a drop in suction to around 10 kPa. Suction increased progressively as a result of advective drying until approximately 1,000 kPa was reached in late May and again, the rate dropped rapidly. Given that the IOR was seen to record a rapid drop coinciding with suction reaching approximately 900–1,000 kPa, this likely reflects the point at which water becomes the rate limiting factor in the pyrite oxidation reaction.

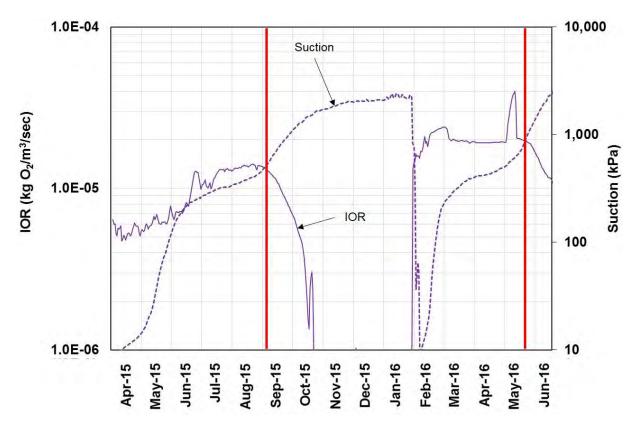


Fig. 5. IOR plotted against suction to illustrate correlation between decreased IOR and second inflection point on suction graph representing point at which pyrite oxidation becomes water limiting.

Once moving beyond the second inflection point on the matric potential figure (approximately 900–1,000 kPa), representing the loss of tightly held water, oxygen consumption ceases. Theoretically, if net percolation can be reduced significantly through sustained (e.g. multiple consecutive) dry seasons, and advective drying continues to the point at which suction can approach this second inflection point in the field, then the IORs within the WRD may be managed in the field. What is evident from Fig. 5 is the ability for the ACLCs to produce a dynamic IOR that is influenced by both air supply and changing moisture content, as would be expected in the field.

The High Air Flow Column demonstrates that a high availability of air, such as that from advective forcing of air, will have significant effects on IOR. During the same testing period, it also highlights the influence that water will have when it becomes limiting in the oxidation reaction as seen after 7–10 months of operation, that is, the IOR reduces sharply but then rebounds fast as water is added such that is experienced in the Pilbara during the wet season.

Figure 6 compares monthly average IORs calculated from oxygen consumption (ACLC) and steady-state sulfate release (free draining leach columns) for the first 12 months of operation for the four replicate columns. Key findings from this comparison is the significantly faster IOR (order of magnitude) of the High Air Flow Column relative to its free draining leach column duplicate, and the significantly lower IORs (up to 5-10 times slower), when airflow and water are controlled (Low Air and Water Columns).

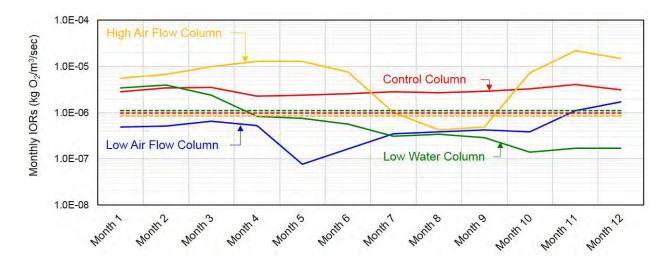


Fig. 6. Comparison of oxygen consumption (ACLC) and sulfate release (AMIRA) calculated monthly average IORs for the first 12 months of operation for the four replicate columns. Free draining leach column duplicates represented by dashed coloured lines of associated ACLC replicate. IORs for free draining leach columns are calculated from the average sulfate values for the final five months representing steady-state conditions as is typically employed for this method (Maest and Nordstrom 2017).

Table 2 presents published IORs for black shale samples, specifically Mt McRae Shale, using the three most common kinetic testing methods for determining oxidation rates. Four of the five IORs are generally lower than the Low Air and Low Water ACLC Columns ,and the highest value is consistent with the IORs determined using the free draining leach column method in this study.

Table 2. Measured oxidation rates in kg $O_2/m^3/sec$ for several black shale samples associated with Western Australian iron ore deposits utilising industry standard kinetic methods. All total sulfur values are in wt%. Table adapted from Pearce (2015).

Oxidation Rate Measurement Method	Total Sulfur	IOR Range
Free draining leach column (EGi, 2006)	3.0	1.5E-6
Free draining leach column (EGi, 2006)	1.5	4.2E-8
Free draining leach method (Linklater, 2015)	1.8	1.7E-7 ^A
Humidity cell method (Linklater, 2015)	5.1	1.3E-7 ^A
Oxygen consumption method (Earth Systems, 2012)	3.0	6.2E-7 ^B

AReported results were converted from average sulfate release rates (Linklater, 2015) BReported results were converted from normalised PORs (Earth Systems, 2012).

What is evident from Fig. 6 and Table 2 is the ability of the ACLCs and the inability of standard methods to produce a dynamic IOR that is influenced by both air and water availability, as would be expected in the field. The High Air Flow Column data for example shows the effects that a high availability of air, and the suction state of the material will have on an IOR within a WRD that may be influenced by dynamic water and gas flux. The clear influence that water content will have when it becomes the limiting factor in the oxidation reaction is shown in months 7–10. As the figure shows, the IOR reduces sharply but then rebounds fast as water is added in month 10. This variability in suction state is analogous to the semi-arid climate of the Pilbara which experiences cyclonic rainfall events within the wet season.

3.3 Leachate Quality and Liquid to Solid Ratio

Due to the larger sample size and infrequent wetting of the ACLCs to better reflect climatic conditions of the Pilbara, the liquid to solid ratios are far different to those of the free draining leach columns, as presented in Table 3. After the final flushes for each column, the liquid to solid ratios for the ACLCs are over 30 times lower and are far more representative of those observed in the field.

Table 3. Liquid to solid ratios for ACLCs and free draining leach columns following first and final flushes.

Column	ACLC First	Free Draining Leach Final	ACLC Final	Free Draining Leach Final
Control Column	0.15	0.4	0.25	14
Low Air Column	0.15	0.4	0.23	14
Low Water Column	0.10	0.2	0.16	7
High Air Column	0.14	0.4	0.23	14

The restricted application of water and larger column length (increased pore water residence time) allowed the ACLCs to develop a saturated system with respect to secondary oxidation products. Following the final flush, concentrations of key contaminants were generally orders of magnitude greater than those produced by the free draining leach columns during steady-state conditions, despite similar pH values (2.3-2.6). Figure 7 presents sulfate and zinc data for each of the ACLC replicates and their free draining leach column duplicates within first and final flush leachates. First flush concentrations are fairly consistent, with the higher concentrations shown by the freedraining leach columns likely due to the crushing of samples pre-experiment. The key difference is observed in the final flush leachates where zinc is up to two orders of magnitude greater within ACLC leachates and sulfate is generally greater than order of magnitude. Note that, the leachate for the Control Column was collected five months after the replicate columns, as this column was continued (along with its free draining leach column duplicate) as part of a greater study. Therefore, the Control Column (ACLC and free draining leach columns) had an additional five months for the storage of oxidation products which emphasised the importance of allowing the system to saturate, as you would expect within a WRD within the Pilbara. If using steady-state leachate concentrations (release rates) from free draining leach columns to predict pore water chemistry for AMD loading calculations, contaminant concentration within pore water could be underestimated by orders of magnitude.

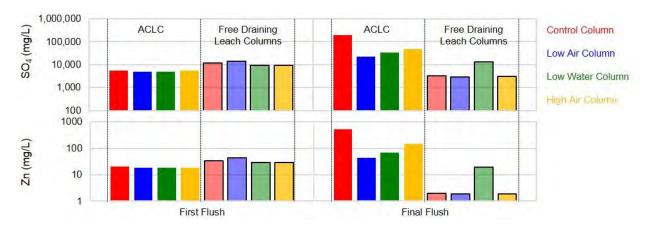


Fig. 7. Comparison of sulfate and Zn concentrations between ACLC and AMIRA first and final flush leachates for the operating period. Free-draining leach column duplicates represented by shaded colour of associated ACLC replicate.

3.4 System Temperatures

Internal temperature was recorded within a free draining leach column to allow comparison with temperatures recorded within the ACLCs containing the same material. The internal temperature fluctuation observed in Fig. 6 for the free draining leach column is a result of the method prescribed drying regime which cycles heat lamps on and off.

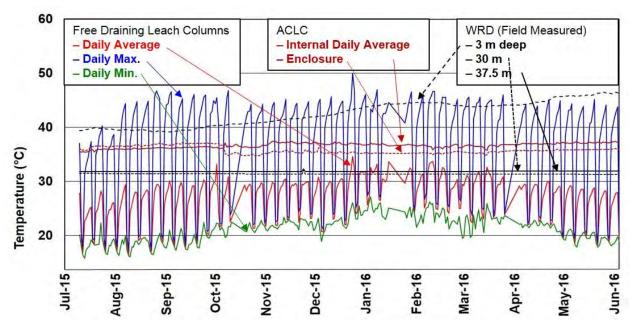


Fig. 8. Daily average, minimum and maximum internal temperatures for a freedraining leach column plotted against measured the internal temperature of an ACLC testing the same material, the ACLC enclosure temperature, and internal WRD temperatures from three instrumented depths.

The internal temperatures for the free-draining leach column were highly variable with minimum and maximum temperatures of 16°C and 50°C respectively. Relevant points regarding the trends noted in the temperature data include:

- The temperature fluctuations in the free-draining leach column appears to be more comparable to those experienced in the outer (<3m) layer of a WRD, where climatic forces have a more pronounced influence, such as a cover system, than the interior of a WRD.
- The internal ACLC and the ACLC enclosure data lie between the temperatures measured in the field at depths of 3-37.5m in one of the boreholes from which the samples were collected.

It should be further noted that temperatures greater than 40°C and 60°C respectively are noted in other WRDs monitored at the same site. When considering that the average daily temperatures of the free-draining leach columns are lower than the average internal ACLC temperatures, it can be concluded that temperature conditions of the free-draining leach columns are not as representative of the internal conditions of the WRDs being monitored. This is an important distinction as temperature is a key factor for oxidation reaction rates.

4.0 CONCLUSIONS

Dynamic oxidation rates were determined through continuous measurement of oxygen and suction using a new kinetic testing method that incorporates the dependency of IORs on air flow and moisture. The method allows variable air flows to be set based on specific WRD construction methods that replicate air supply to reactive minerals. The method also incorporates site calibrated wetting regimes so that liquid to solid ratios observed in the field can be applied and over flushing, typical of standard kinetic testing methods, can be avoided. This allows the build-up of saturated systems (with respect to secondary oxidation products), as is common to the Pilbara, and the production of more realistic leachates reflective of concentrated pore waters. As the method incorporates dynamic WRD variables such as gas and water flux, the combination of the produced dynamic oxidation rates with saturated system leachate data facilitates more site specific AMD contaminant load predictions.

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