

# NET NEUTRALISATION POTENTIAL (NNP) IN KIMBERLEY DIAMOND TAILINGS AND SLIMES WASTE MATERIALS

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## ABSTRACT

Recent studies suggest that sulphide minerals are insignificant in the tailings and slimes of diamond mines. Other studies indicate that sulphide minerals are in fact present. An overabundance of neutralisation potential is also reported, suggesting that long term environmental risks associated with acidity and leaching of heavy metals is insignificant. The knowledge of acid generation potential and metal mobility in the slimes and tailings conditions is therefore necessary.

The objective of this study was to evaluate the potential for acid generation and to assess if conventional pyrite oxidation occurs in the slimes and tailings materials. This was assessed using standard geochemical testing techniques such as Acid Base Accounting (ABA) and accelerated weathering testing by means of humidity cell experiments. A total of 131 samples, representing 35 tailings and 5 slimes facilities were sampled, tested and analysed using static tests. Six samples (three tailings and three slimes) were selected based on Neutralization potential (NP): Acid Potential (AP) ratios and subjected to humidity cell testing.

ABA results show that paste pH ranges from 7.5 to 10. Neutralisation Potential Ratio (NPR) was found to be greater than 4 for all tailings and slime samples, indicating that the materials are non acid generating. The deionised water leachate metal concentrations in alkaline conditions did not indicate elevated concentrations relative to the NAG leachate concentrations especially for metals like Zn that could be soluble in neutral pH. However, sulphate and sodium occurred at concentrations indicating that they may require further consideration with respect to the impact on site water quality.

Long term leaching using column leaching cells is recommended to quantify the long term sulphide oxidation and neutralization depletion rates. This will lend insight into metal leaching rates under ambient conditions.

**Keywords:** *Tailings and slimes, ABA, Kinetic testing, Diamond mining, Kimberley*

## 1. INTRODUCTION

The general classification of diamond mines as category B or C (Zhao and Pulles, 2005) suggests that sulphide minerals are insignificant in the tailings and slimes. Recent studies suggest that sulphide minerals are, in fact, present in the tailings residue and slimes, and that conventional pyrite oxidation processes occur (Golder, 2004 and Golder, 2009). Significant amounts of thenardite ( $\text{Na}_2\text{SO}_4$ ) are also reported which could release sulphate through simple dissolution processes.

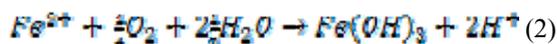
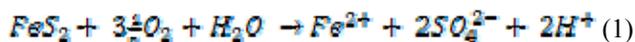
An over abundance of neutralisation potential is also reported, suggesting that long term environmental risks associated with acidity and leaching of heavy metals is insignificant. However, long term risks may exist with regard to sulphates, sodium and neutral/alkaline soluble metals in the matrix of the Kimberley mines geology. While the solubility of aluminium, iron and copper is greatly lowered in neutral pH drainage, elements such as antimony, arsenic, cadmium, molybdenum, selenium and zinc remain relatively soluble and can occur in significant concentrations. Therefore, knowledge of long term acid generation potential and metal mobility in the presence of excess neutralization potential is necessary.

The objective of this paper is to evaluate the NNP in Kimberley diamond slimes and tailings waste material and to assess the likelihood of metal mobility in the presence of excess neutralisation potential environment. In order to understand NNP, an understanding of the geochemistry of pyrite oxidation and the geochemistry of neutralisation is necessary.

### Pyrite Geochemistry

A large number of different sulphide minerals can produce acidic oxidation products. The most common and notable one is pyrite ( $\text{FeS}_2$ ). Kleinman et al. (1981) explained the pyrite oxidation process in three stages:

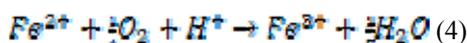
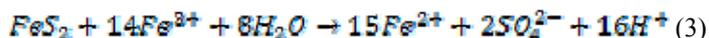
**Stage 1** occurs at relatively high pH  $\geq 4.5$  with the two reactions in equations 1 and 2 below occurring.



These reactions proceed abiotically and the oxidation of ferrous iron is rapid at pH  $\geq 5.0$ . The net result is a leachate containing high sulphate concentrations, lower pH and low iron concentrations.

**Stage 2** occurs as the pH continues to drop below 4.5, typically in the range  $2.5 \leq \text{pH} \leq 4.5$ . The same reaction as in stage 1 dominates, but bacterial activity now catalyses the reactions, especially the oxidation of ferrous iron. The leachate contains elevated sulphate concentrations and acidity. Dissolved iron concentrations also increase due to the increased solubility of ferric iron at pH  $< 3.5$ .

**Stage 3** is initiated as the pH continues to drop to pH  $\leq 2.5$ . Ferric iron activity is increased by the further increased solubility and the decreased precipitation of Fe(OH)<sub>3</sub>. The following two reactions dominate:



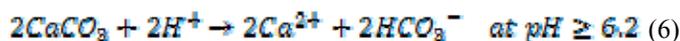
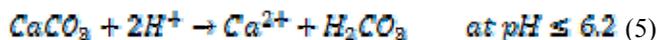
Ferric iron then becomes the primary oxidant of pyrite and the availability of the ferric iron is dictated by the oxidation of ferrous iron. The leachate contains high concentrations of sulphate, iron and acidity. The ferric iron : ferrous iron ratio is largely determined by the redox potential in the tailings environment.

### Geochemistry of Neutralization

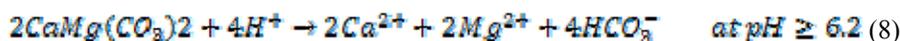
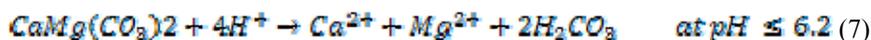
Common minerals which typically contribute to the neutralisation of acidity include calcite [CaCO<sub>3</sub>], magnesite [MgCO<sub>3</sub>], dolomite [CaMg(CO<sub>3</sub>)<sub>2</sub>] and ankerite [CaFe(CO<sub>3</sub>)<sub>2</sub>].

Neutralization of the pyrite oxidation products (specifically acidity) is mainly achieved by calcite and dolomite. Aluminosilicate minerals, such as clays and feldspars, may also contribute to the process. The conventional neutralisation reactions include reactions in equations 5 to 8 in acidic and alkaline conditions.

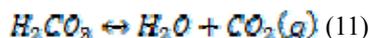
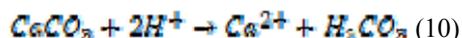
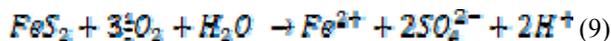
Calcite:



Dolomite:

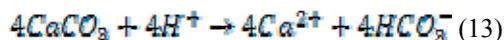
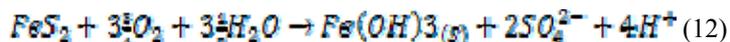


From the pyrite oxidation and neutralization reactions, the net oxidation/neutralisation reactions in an acidic environment can be summarised as outlined in the reactions presented in equations 9 to 11.



The molar ratio of calcium to sulphate from the net oxidation/neutralization reactions in acidic environment (pH  $\leq 3$  typically) is then [Ca]/[SO<sub>4</sub>] = 0.5.

In a mildly acidic to alkaline (pH  $\geq 6.2$ ) environment, the net oxidation/neutralisation reactions can be summarised as outlined in the reactions in equations 12 and 13.



The molar ratio of calcium to sulphate is then [Ca]/[SO<sub>4</sub>] = 2.0 in a mildly acidic to alkaline environment.

Morin and Hunt (1994) found that feldspars are slow reacting, but can also contribute to the neutralisation of acidity. The stoichiometry of these neutralisation reactions can be complex and tends to be site specific. The rate and nature of feldspar neutralisation is dependent on several factors including type of feldspar mineral, environmental conditions (specifically pH) and acid generation. Feldspar neutralisation is not included in the scope of this paper.

## 2. METHODOLOGY

### Sample Collection

Test pits were excavated mechanically through the Kimberlite tailings and slimes waste facilities using a Tractor Loader Backhoe (TLB) and composite samples were collected to a depth of 3 m. In cases where different layers were identified, samples representing the different layers were collected. A total of 131 samples were collected consisting of 110 samples and 21 duplicates.

Saturated slimes samples were collected from the slimes dam (CTP) at the point of deposition into the slimes facility. Other samples were collected at the point of deposition of tailings materials into the wastes facility from the conveyor belt (CTR29). Collection of tailings/slimes materials for reprocessing was underway at some of the tailings facilities (Reservoir Tailings Resource (RTR), Colville TR2, TR12, TR8C, TR22, TR6 and TR19) at the time of sampling and composite samples were collected from heaps of excavated material at the point of loading into the transportation trucks.

All samples were placed in clean PVC bags, labelled, excess air removed and tightly sealed. The samples were kept cool in a cool store and delivered to the laboratory for geochemical analysis.

### Analytical Methods

The following geochemical analyses were conducted on the tailings and slime samples as outlined:

- Mineralogical analysis including XRF (total elemental composition) and quantitative X-Ray Diffraction (XRD). (15 samples)
- Acid-Base Accounting (ABA) by the modified Sobek method, including paste pH, sulphur species (including total sulphur, sulphate sulphur and sulphide sulphur), acid generation potential (AP) (calculated using sulphide sulphur) and acid neutralization potential (NP). (118 samples)
- Net Acid Generation (NAG)(15 samples).
- De-ionised (DI) water leach test (4:1 liquid to solid ratio), conducted over a period of 24 hours (118 samples).
- Leachates from de-ionised water and NAG leach tests were analyzed for pH, conductivity, alkalinity, sulphate and dissolved trace metals.

## 3. RESULTS

### Mineralogical Analysis

Quantitative XRD was performed on representative slimes(3 samples) and tailings samples (11 samples) to establish the mineralogical identity of dominant phases in the materials.

The slimes and tailings consisted of mainly silicates with the dominant one being smectite (10% to 84%). Smectite is a group of swelling clay minerals made up of 2:1 unit layers, each layer consisting of two silicon-oxygen tetrahedral sheets enclosing one aluminium-oxygen (or hydroxyl) octahedral sheet (Vietti, 2003). Water and other polar molecules can enter between the unit layers causing the lattice to expand in the vertical direction (Van der Watt and van Rooyen, 1995) resulting into deep cracks observed in the slimes and tailings facilities.

Other silicate minerals found in the slimes and tailings included quartz (6% to 24%), plagioclase (3% to 20%), mica (3% to 18%), clinopyroxene (6% to 17%) and kaolinite/chlorite/serpentine (4% to 18%).

Sulphide mineral (pyrite) was found to be present in both slimes (1 to 2%) and tailings (2 to 4%) samples, indicating a potential for acid generation. Calcite, a fast neutralising carbonate mineral, was found in both slimes (4 to 7%) and tailings (2 to 10%). Siderite was also found in the tailings (1 to 5%) samples.

### Acid Base Accounting (ABA)

The purpose of ABA was to assess the acid generation potential of the slimes and tailings materials.

#### Acid generation potential

The potential for acid generation was evaluated by using the screening criterion set by Price (1997). These classifications are based on the Modified Sobek method (1978) and also on the Net Potential Ratio (NPR) given by the Neutralization Potential (NP)/Acid Potential (AP) ratio as follows:

$NP/AP < 1$  = Likely acid generating, unless sulphide minerals are non-reactive.

$1 < NP/AP < 2$  = Possibly acid generating if NP is insufficiently reactive or is depleted at a rate faster than sulphides.

$2 < NP/AP < 4$  = Low potential for acid generation unless there is significant preferential exposure of sulphides.

NP/AP > 4 = Non acid generating.

Sulphide sulphur concentrations are often used as an indicator for acid-generation potential. Under the Price (1997) guidelines, materials with a paste pH greater than 5.5 and a sulphide-sulphur concentration of less than 0.3% are considered unlikely to generate acid in most instances, due to the small amount of acidity that could be produced during sulphide oxidation. Low sulphide content materials that have low abundance of acid neutralizing minerals generally require additional geochemical characterisation in order to quantify acid generation potential.

Figure 1 shows a plot of ABA paste pH against Neutralization Potential Ratio that summarizes the results obtained for the tailings and slimes samples. Results obtained by Pulles, Howard and De Lange Incorporated (PHD) in 2005 are also included in the Figure for comparison purposes.

Total sulphur in the samples was found to be less than the 0.3% generally accepted as potentially insignificant with respect to acid generation potential. The paste pH range was obtained as  $7.5 \leq \text{paste pH} \leq 10$  and indicates near neutral to alkaline pH. NPR was greater than 4 ( $\text{NPR} > 4.0$ ) for all the tailings and slime samples indicating non-acid generating potential. This compares well with the PHD results of 2005 that concluded that tailings and slimes contain an abundance of neutralising minerals, hence the seepage is alkaline and will not become acidic in future.

#### Neutralisation potential

Bulk NP is measured by the dissolution of all acid neutralisation minerals in a sample during the reaction of the sample with sulphuric acid under ambient conditions (Price,1997). Bulk NP is a measure of the total NP in a sample, which is contributed by readily dissolved carbonate minerals and slower reacting silicate minerals. The bulk NP measured in the tailings and the slimes ranged from 21 to 148 kg/t  $\text{CaCO}_3$ .

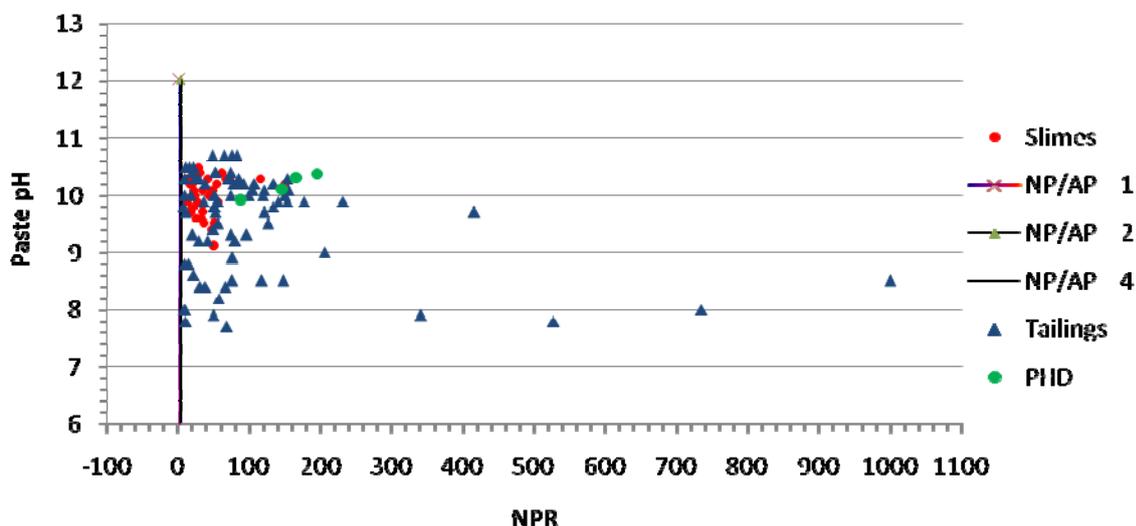


Figure 1. ABA paste pH versus NPR

#### Net Acid Generation (NAG)

NAG tests, including comprehensive analysis of NAG supernatants, was conducted to evaluate the composition of supernatant after the complete oxidation of all reactive minerals and dissolution of soluble phases. NAG testing was performed on fourteen samples and a summary of the results is presented in Table 1.

For the slimes samples, the NAG supernatant is neutral to slightly alkaline (7.2 to 8.8 pH units) and the solutions have trace metal and sulphate concentrations greater than those measured in deionised water leachates. NAG supernatant for the tailings reported neutral to slightly alkaline pH values ranging from 7.1 to 8.2 pH units. Alkalinity ranged from 2 400 to 2 700 mg/l in slimes and from 1 000 to 3 400 mg/l in tailings. The slimes sulphate concentrations ranged from 2 400 to 2 700 mg/l and the tailings sulphate concentrations ranged from 150 to 2 650 mg/l.

Table 1. Summary of NAG results for slimes and tailings (all units in mg/l)

Sample ID	pH	Alkalinity (CaCO <sub>3</sub> )	SO <sub>4</sub>	Al	Ca	Cr	Cu	Fe	Mg	Na	Zn
SLIMES											
CTP7	8.8	2600	2475	206	600	2.55	1.60	243	825	3650	1.93
NTP1B	7.6	2700	475	250	1200	3.48	1.45	214	1100	2100	1.73
NTP2C	7.2	2400	325	139	700	4.20	0.925	170	875	1175	0.950

TAILINGS											
CTR29B	7.7	2600	500	164	850	2.30	1.25	180	575	1850	1.03
TR3LT2	7.4	2700	425	235	750	2.75	1.75	275	750	2300	1.55
STR1A	7.5	2900	800	194	850	6.23	1.40	275	1075	1800	2.03
CTR2B	7.7	3400	475	325	725	3.85	1.70	375	1475	2850	1.80
ED4B	7.5	2600	1000	225	800	3.25	1.30	241	1200	2525	1.68
TR8C2	7.7	1900	975	34.5	625	7.05	0.325	65	425	900	0.005
TR28	7.1	1500	150	120	575	2.10	0.575	121	825	400	0.400
RTR3	7.8	1000	2650	79.8	975	9.55	1.95	237	725	125	0.525
TR5B	7.8	1500	1875	15.9	850	3.23	0.005	22	600	150	0.005
TR19A	7.2	2400	450	100	600	5.20	0.775	141	900	1100	0.525
TR10A	8.2	1500	550	162	725	4.03	1.03	178	975	900	0.850

### Molar Ratios

Molar ratios of calcium to sulphate, alkalinity to sulphate and alkalinity to calcium were calculated from the NAG results. Analysis of the molar ratio of calcium to sulphate in the samples gives an indication of the local environmental conditions under which pyrite oxidation takes place. The molar ratio of alkalinity to calcium gives an indication of the nett neutralization reactions involved. The molar ratio results are presented in Table 2.

The molar ratio of calcium to sulphate ranged from 1 to 6 in the slimes and from 1 to 9 in the tailings samples. The molar ratio of alkalinity to calcium ranged from 1 to 2 in the slimes and from 0.4 to 2 in the tailings. The 0.4 alkalinity to calcium ratio of sample RTR3 could be attributed to the low calcite (2%) relative to the pyrite content (4%) in this sample. However, sample RTR3 still has a relatively high NAG pH (7.8) that could be attributed to the presence other source of alkalinity in the sample like siderite (2%).

Table 2. Molar ratios of calcium to sulphate and alkalinity to calcium

Samples	Mineralogy (%)			pH	SO <sub>4</sub>	Ca	Alk(CaCO <sub>3</sub> )	Molar ratio		
	Pyrite	Calcite	Calcite:Pyrite					moles/l		Ca:SO <sub>4</sub>
SLIMES										
CTP7	2	4	2	8.8	0.026	0.015	0.026	1	1	2
NTP1B	2	7	4	7.6	0.005	0.030	0.027	6	5	1
NTP2C	1	5	5	7.2	0.003	0.017	0.024	5	7	1
TAILINGS										
CTR29B	2	6	3	7.7	0.005	0.021	0.026	4	5	1
TR3LT2	2	6	3	7.4	0.004	0.019	0.027	4	6	1
STR1A	3	5	2	7.5	0.008	0.021	0.029	3	3	1
CTR2B	2	4	2	7.7	0.005	0.018	0.034	4	7	2
ED4B	2	4	2	7.5	0.010	0.020	0.026	2	2	1
TR8C2	2	6	3	7.7	0.010	0.016	0.019	2	2	1
TR28	-	5		7.1	0.002	0.014	0.015	9	10	1
RTR3	4	2	1	7.8	0.028	0.024	0.010	1	0.4	0.4
TR5B	-	10		7.8	0.020	0.021	0.015	1	1	1
TR19A	-	7		7.2	0.005	0.015	0.024	3	5	2
TR10A	2	4	2	8.2	0.006	0.018	0.015	3	3	1

### Deionised (DI) Water Leach Tests

Leaching slimes samples yielded alkaline pH leachate (ranging from 9.2 to 10 pH units). The pH of the leachate for the tailings ranged from neutral to alkaline (7.4 to 10.6 pH units). Alkalinity ranged from 100 to 274 mg/l in the slimes and from 44 to 704 mg/l in the tailings.

Distilled water leachate consisted of high concentrations of sulphate. The highest concentrations of sulphates were found in CTP slimes (907 mg/l). The tailings sulphate concentrations ranged from 52 to 1267 mg/l. The DI leachate metal concentrations were lower than the NAG metal concentrations by more than an order of magnitude.

Table 3. Summary of DI water leachate results for slimes and tailings

Sample ID	pH	Alkalinity (CaCO <sub>3</sub> )	SO <sub>4</sub>	Al	Ca	Cr	Cu	Fe	Mg	Na	Zn
SLIMES											
CTP7	9.6	100	907	1.22	69	0.005	0.016	2.02	12	762	0.011
NTP1B	10	274	6	3.44	19	0.024	0.073	6.78	22	145	0.060
NTP2C	9.8	264	14	3.64	59	0.026	0.071	5.94	31	117	0.092
TAILINGS											
CTR29B	10.3	208	89	5.83	12	0.052	0.031	8.09	26	129	0.026
TR3LT2	10.4	293	73	20	20	0.129	0.165	33	80	166	0.160
STR1A	10	384	128	16	64	0.240	0.158	25	122	184	0.210
CTR2B	9.9	704	323	0.005	60	0.005	0.005	0.142	14	395	0.444
ED4B	9.6	424	403	1.58	70	0.021	0.062	5.48	36	301	0.147
TR8C2	9.2	80	510	0.005	265	0.005	0.005	0.084	35	280	0.005
TR28	9.1	132	52	1.94	39	0.015	0.040	2.88	25	42	0.005
RTR3	7.7	44	342	0.859	62	0.022	0.019	2.73	19	7	0.005
TR5B	7.4	24	1267	0.005	463	0.005	0.005	0.252	125	33	0.005
TR19A	9.6	252	210	2.13	50	0.043	0.069	5.07	40	108	0.045
TR10A	9.1	120	314	0.005	202	0.005	0.005	0.224	24	144	0.005

#### 4. DISCUSSION AND CONCLUSIONS

The potential for acid generation evaluated by ABA established that NPR is greater than 4 for all the tailings and slime samples. This indicates that the materials are non-acid generating and the paste pH is near neutral to alkaline. NAG results also indicate neutral to alkaline pH for the slimes and tailings materials.

Sulphide mineral, pyrite, is present in the slimes and tailings materials and elevated sulphate concentrations are recorded in the NAG results. However, conventional pyrite oxidation is inhibited by the neutral to alkaline conditions (pH range of 7.2 to 10) of the slimes and tailings materials.

Conventionally, Stage 1 of pyrite oxidation occurs at relatively high (pH  $\geq$  4.5). This process proceeds abiotically and the oxidation of ferrous iron is rapid at pH  $\geq$  5.0 (Kleinman et al (1981). The slimes and tailings may undergo Stage 1 of pyrite oxidation but the alkaline conditions of the materials inhibits the process from proceeding to Stage 2 where bacterial catalysis of ferrous would be expected to take effect at acidic pH (range of 2.5 to 4.5).

The conventional net oxidation/neutralisation reactions in alkaline (pH  $\geq$  6.2) environment (Equation 12 and 13) indicate that the molar ratio of calcium to sulphate is two ([Ca]/[SO<sub>4</sub>] = 2.0). The calcium to sulphate molar ratios from the NAG results of the slimes and tailings were calculated to be more than two ([Ca]/[SO<sub>4</sub>]  $\geq$  2.0) except for one slimes and two tailings samples (Table 2). This could be attributed to high levels of pyrite (4%) in some samples (RTR3) relative to the calcite (2%) levels. It could also be due to acidic micro environments within the samples caused by acidic spots in the samples.

The excess Ca demonstrated by the Ca:SO<sub>4</sub> molar ratios of more than 2 could be attributed to the excess calcite found in most of the samples as shown by the calcite:pyrite ratio (Table 2). It may also result from the weathering of kimberlitic minerals that include smectite [(<sup>1</sup>/<sub>2</sub>Ca,Na)<sub>0.7</sub>(Al,Mg,Fe)<sub>4</sub>(Si,Al)<sub>8</sub>O<sub>20</sub>(OH)<sub>47</sub>nH<sub>2</sub>O], plagioclase [Ca,Na(Al<sub>2</sub>Si<sub>2</sub>O<sub>8</sub>)] and clinopyroxene [(Ca,Mg,Fe,Al)<sub>2</sub>(Si,Al)<sub>2</sub>O<sub>6</sub>]. An abundance of neutralisation potential was demonstrated.

Neutralisation of the pyrite oxidation products (specifically acidity) is mainly from calcite found in the slimes and tailings material. Siderite, that is also present in the slimes and tailings materials, does not contribute to the process but could act as a buffering agent in a low pH environment. The likely neutralization reaction in the slimes and tailings is, therefore, the calcite – acid reaction at a pH  $\geq$  6.2 represented by Equation 13.

The molar ratio of alkalinity to calcium gives an indication of the net neutralisation reaction involving calcite. This was established as equal to 1, [Alk]/[Ca] = 1 (Table 2) and corresponds to the calcium to alkalinity ratio in the conventional calcite neutralization equation (Equation 4).

The dilution of slimes and slurry waste with water containing dissolved carbonate species, such as bicarbonate from the addition of lime (CaO) before release into the slimes or tailings facilities, also contributes to achieve neutralisation. The addition of CaO produces calcium hydroxide (Ca(OH)<sub>2</sub>) initially that may absorb carbon dioxide (CO<sub>2</sub>) to produce bicarbonate (HCO<sub>3</sub><sup>-</sup>).

The DI water leachates are more alkaline (pH = 7.4 to 10) compared to the NAG supernatant (pH = 7.2 to 8.8). This could be attributed to the preferential dissolution of neutralising minerals. Van Niekerk (1997) noted that leaching is sensitive to the dissolution of calcite and that percolating water contains as much as 50 to 100 mg Ca/l and 60 to 120 mg CaCO<sub>3</sub>/l alkalinity (in a closed system at a pH 7.0 to 7.5) due to dissolution of calcite.

The DI leachate metal concentrations in alkaline conditions did not indicate elevated concentrations relative to the NAG supernatant concentrations especially for metals like Zn that could be soluble in neutral or alkaline pH. However, sulphate and sodium occurred at concentrations indicating that they may require further consideration with respect to the impact on site water quality.

A more comprehensive study of long term leaching with rain water is recommended to quantify the sulphide oxidation and neutralization depletion rates. This could be achieved through the use of column leaching cells that simulate the profile of the slimes and tailings and account for the impermeable layers. Leachates would then be collected from each layer as well as from the bottom of the profile. This will lend insight into metal leaching rates under ambient conditions. This could also be compared to water quality data from leachates collected in the field at longer term storage facilities for these materials and with groundwater data.

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