

# Potential of contaminated neutral drainage generation from waste rock at Raglan



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## ABSTRACT

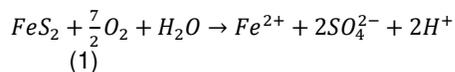
Fresh samples representing seven waste rock types from the Raglan Mine, property of Xstrata Nickel (Quebec, Canada), were tested to investigate their geochemical behaviour. A multidisciplinary characterization and column tests were performed on each rock type. Results from column tests show that all leachate samples remained at near-neutral pH for the entire duration of the tests and that they were not considered to be acid-generating using the oxidation-neutralization curves. Column tests showed that nickel was released at elevated concentrations by at least one rock type. Batch sorption tests showed that sorption phenomena could have a influence on drainage water quality from the rock.

## RÉSUMÉ

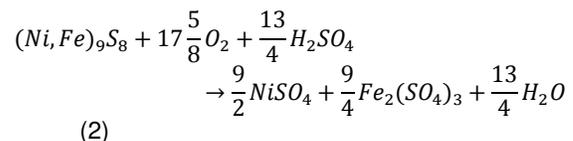
Une étude du comportement géochimique a été effectuée sur différents échantillons frais représentatifs de sept types de stériles de la Mine Raglan, propriété d'Xstrata Nickel. Une caractérisation pluridisciplinaire et des essais en colonnes ont été effectués sur chaque type de roche. Les résultats des essais en colonne montrent que les pH des lixiviats ont été neutres pour toute la durée du test, et une interprétation utilisant les courbes d'oxydation et de neutralisation a montré qu'ils ne sont pas potentiellement générateurs de drainage minier acide. Les essais en colonnes ont montré que du nickel est lixivié par au moins un type de roche sur sept. Des essais de sorption montrent que ce phénomène pourrait avoir une importance sur l'évolution de la qualité des eaux de drainage provenant de ces roches.

## 1 INTRODUCTION

Tailings and waste rock are important solid by-products produced during mining operations. Their management can sometimes be problematic due to their physical and chemical stability. One of the main environmental issues related to mine waste management is the production of acid mine drainage (AMD), in which acidity and dissolved heavy metals contaminate mine drainage due to the oxidation of sulphide minerals contained in the host rocks. It takes place when the mine wastes have no, or insufficient, neutralizing potential. Equation 1 shows the oxidation of pyrite, a common sulphide, that produces acidity and dissolved iron (Nordstrom 1982).



Another possible phenomenon (less studied) is the production of contaminated neutral drainage (CND), which consists of the release of soluble contaminants in the water at circumneutral pH. For example, the oxidation of pentlandite releases soluble Ni without acidity, as seen in Equation 2 (Kodali et al. 2004).



Different elements can be found in CND such as cobalt, nickel, copper, and other metals (Stantec 2004). The presence of these elements in effluents at concentrations above environmental regulatory criteria may require management prior to release to the environment.

It has been recognized through laboratory and field investigations that some rock types from the Raglan Mine release nickel, even if they usually have low sulphide content and the pH of the contact water is neutral (Nicholson et al. 2003; Rinker et al. 2003).

This study was undertaken to evaluate more precisely the geochemical behaviour of seven representative waste rock types from the Raglan mine. The ultimate goal is to identify the rock types that have CND generation potential to manage them differently, and consequently more safely at the site to reduce the potential for environmental impact.

## 2 STUDY SITE AND PREVIOUS INVESTIGATIONS

Rock samples were obtained from the Raglan Mine, property of Xstrata Nickel, a nickel-copper-cobalt mine located in a Nordic climate (mean annual temperature of -10.3°C) in the north of the province of Quebec, Canada. The mine has been in operation since 1997.

The main geological composition of rock extracted from the different pits and underground stopes are olivine-pyroxenite, gabbro, peridotite, argillite, and volcanic (see Figure 1). They are temporarily stockpiled, and will be reused to backfill open pits and underground stopes.

Two studies were undertaken to determine the contamination potential of Raglan waste rock. An assessment study was first performed on nickel leaching potential at neutral pH using humidity cells (Rinker et al. 2003). The study showed that nickel leaching occurs from certain rock types that were considered to be non-acid generating. A mineralogical characterisation and static tests were also performed on different rock samples from the Raglan mine by URSTM in 2008 (Bussière et al. 2008). This study concluded that the AMD potential cannot be clearly defined by static tests for half of the rock types studied because of the uncertainties related to the test. It was also concluded that some samples may have AMD potential as a result of the metals content. Column tests, rather than humidity cells, were considered the more appropriate tools to evaluate AMD and AMD generation potential (Pepin 2009; Plante 2010). These tests were performed in this investigation and the main results are presented below.

## 3 MATERIALS AND METHODS

Column tests were used to evaluate AMD and AMD generation potential of seven rock samples from the Raglan mine site. All samples were characterized for their chemical, mineralogical, and physical properties, using the methods presented in the following section.

## 3.1 Sampling and sample preparation

Seven fresh rock samples were taken from three different underground mines at the Raglan site: peridotites from mines 3 and Kikialiq, olivine-pyroxenite from mines 2 and 3, gabbro from mine 2, argillite from mine 3 and volcanic from a nearby quarry. Peridotite and olivine-pyroxenite were taken from these different zones to evaluate the differences between them. The size of the sampled rocks was from a few centimetres to a few decimetres. Before testing, they were crushed with a jaw crusher (5 cm opening) and sieved to reject the particles smaller than 1.3 cm, except for the column C10 in which the fines were retained. The use of columns without fines was chosen to eliminate the effect of fines movement in the column that adds a level of complexity to results interpretation (Aubertin et al. 2008) and to have better control on specific surface area of the rock particles.

## 3.2 Analytical methods

A chemical, mineralogical, and physical characterization of the samples was performed. The chemical waste rocks compositions were determined by inductively coupled plasma (Perkin Elmer Optima 3100 RL ICP-AES) analyses after acid digestion. The mineralogical study was performed with the X-ray diffraction method (XRD) using a Bruker A.X.S. Advance D8 apparatus. The software packages Eva and Topas, with the Rietveld method, were used respectively for mineral phase identification and quantification with a detection limit and precision of 0.5% wt. The relative density of the grains ( $D_r$ ) was determined with a helium pycnometer (Micromeritics Accupyc 1330) with a precision of 0.01. Grain size distribution was evaluated with sieves and the ASTM E 11-87 standard.

Leachate samples from columns were acidified, and then analysed with the ICP-AES technique for elemental analysis. Acidity and alkalinity were measured by acid-base titration respectively to pH endpoints of 4.5 and 8.3 with a Metrohm Titrino Plus 870 titrator.

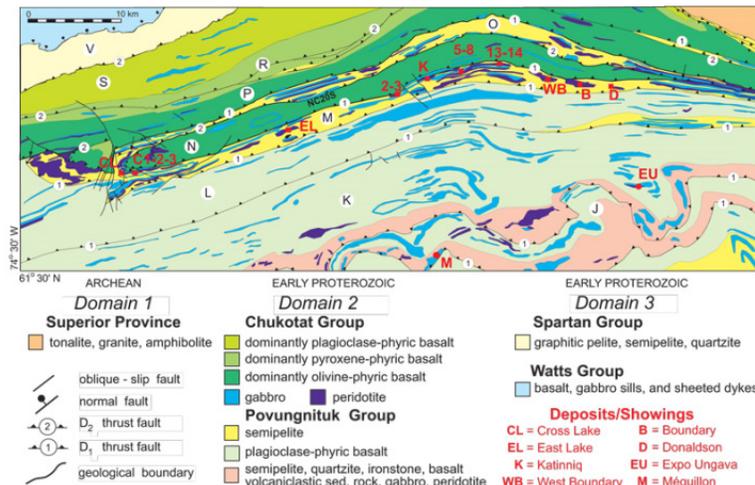


Figure 1 Map illustrating the geology at the Raglan mine site (Leshar 2007, modified from St-Onge et Lucas 1993)

The APHA norm (1995) has a relative error of 9% for an acidity value of 20 mgCaCO<sub>3</sub>/L and a 1 mgCaCO<sub>3</sub>/L precision for alkalinity values between 10 and 500 mgCaCO<sub>3</sub>/L. Sample pH was obtained with a pH electrode with temperature compensation Orion Triode model 9157 with a precision of 0.02. Redox potential (Eh) was determined with an Orion Pt/Ag/AgCl electrode combined with a Benchtop Meter Orion 920A. The Eh results were corrected to a standard hydrogen electrode (SHE). Conductivity was determined with an Oakton Acorn CON 6 conductivity meter.

### 3.3 Column tests

The experiments were performed in vertically mounted PVC columns with a diameter of 30 cm and a height of 80 cm, as shown in Figure 2. A geotextile and a perforated plate were placed at the bottom of each column. The total duration of the experiment was 1 year (12 cycles of 28-32 days), though only 8 months were used for the two duplicate columns. Each cycle began with the inflow of about 24 litres of distilled water from the base of the column. The contact time with the rocks was 4 hours. After this period, the water was drained and analysed for pH, Eh, conductivity, acidity, alkalinity, and elemental analysis. Columns were exposed to air for the remaining days of the cycle. The tests were still in progress at the date of submission of this paper.

Ten columns were set up: one for each waste rock type, two duplicates and one with a finer grain size distribution (without removing particles smaller than 1.3 cm) to investigate the effect of fines. A summary description of the columns is presented in Table 1.

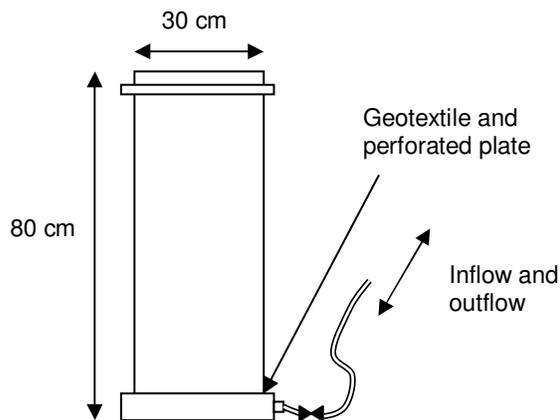


Figure 2. Column configuration

### 3.4 Sorption tests

Batch sorption tests were performed for the seven rock types (methodology inspired from Limousin *et al.* 2007 and Plante 2010). 800 grams of materials (grain diameter smaller than 4.75 millimeters) were placed in beakers with 350 milliliters of water containing 10 parts per million of nickel. The ratio of water:solids was similar to the one in column tests. The contact time was 48 hours and

samples were collected at different time intervals and submitted for chemical analysis.

Table 1. Description of the columns

#	Column content	Mass of rocks (kg)
C1	Peridotite mine 3	72,1
C2	Peridotite Kikialiq (East Lake)	69,6
C3	Olivine-pyroxenite mine 3	67,8
C4	Olivine-pyroxenite mine 2	75,0
C5	Gabbro mine 2	76,8
C6	Argillite mine 3	59,4
C7	Volcanic	79,3
C8	Peridotite mine 3 duplicate	69,0
C9	Olivine-pyroxenite mine 3 duplicate	68,3
C10	Olivine-pyroxenite mine 2 with fines	75,4

### 3.5 Physical characteristics

The grain size distribution and the specific gravity of the samples were analysed. Figure 3 shows the grain size distribution curve of the materials used in the columns. The curves show a good homogeneity between columns, except for C10 that contains fine particles. Also, the grain size distribution of column C6 (Argillite mine 3) is finer due to the type of the rock (flat particles, more friable than the others). The D<sub>10</sub> varies between 17 and 20 mm, except for C6 and C10 (6-6.5 mm). The values of D<sub>30</sub> are between 24-27 mm (13 mm for C6), and for D<sub>60</sub> between 32-35 mm (19 for C6). The coefficient of uniformity ( $C_u = D_{60}/D_{10}$ ) is between 1.7-2.1 for C1 to C9, and 3.6 for C10. The value of the curvature coefficient ( $C_c = D_{30}^2 / (D_{10} * D_{60})$ ) is 0.9 for C6, 2.0 for C10, and is between 1.1 and 1.3 for the other columns. The relative density of the solid grains (D<sub>r</sub>) of the rock types are shown in Table 2. They range between 2.70 and 2.85 for columns C1, C2, C3, C4, C6, C8, C9, and C10, and were at 2.98 and 3 for C5 and C7. These variations reflect the particular mineralogy of each sample.

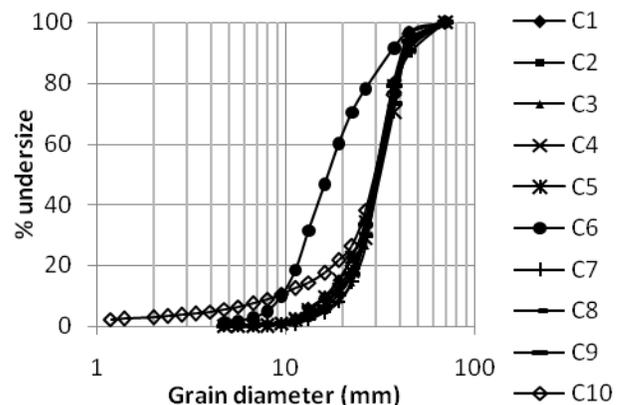


Figure 3. Grain size distributions of the waste rocks tested in the column tests

### 3.6 Chemical characterization

As shown in Table 2, the sulphur content of the peridotites and the olivine-pyroxenites is between 0.1 and 1.3%, and the nickel content between 0.2 and 0.4%. For the other rock types, Ni and S contents were lower than 0.05%. The copper content was lower than 0.08% for all type of rocks. In a previous study, it was observed that the sulphur content is almost entirely expressed as sulphide and the samples contained a negligible quantity of sulphate (Bussi re, 2008), showing a non significant weathering of rock samples.

The magnesium content was between 18-20% for the peridotites and the olivine-pyroxenites, and the calcium content was approximately 8% for the gabbro and the volcanic. Iron was present at 8-11%, except for argillite (approximately 4%). The quantity of aluminum was significant in gabbro, argillite and volcanic (5-10%).

### 3.7 Mineralogy of the Raglan rocks

The main minerals present in the rocks are presented in Table 2. The proportions of pyrite, pyrrhotite, pentlandite, and chalcopyrite are low in all rock types. Although they were not always detected with the XRD analysis, their presence was confirmed with an optical microscope. The total sulphide content is less than 5% in all rocks and is higher for peridotite from mine 3 and olivine-pyroxenite from mines 2 and 3. Pyrite and pentlandite can be present at values greater than 1%. The tested materials contain a large proportion of phyllosilicates (13 to 83%), as lizardite, talc, and chlorite. Pyroxenes (augite and diopside), amphiboles (actinolite and hornblende), and plagioclases (albite) are also present. Based on the geology of the mine, carbonates could also be present in the rocks but was not detected due to their low proportion.

Table 2. Characteristics of the studied samples

Waste rock type Location	Peridotite		Olivine-pyroxenite		Gabbro	Argillite	Volcanic
	Mine 3	Kikialiq	Mine 3	Mine 2	Mine 2	Mine 3	
Physical characteristics							
D, (-)	2.76	2.73	2.78	2.85	3.00	2.79	2.98
Main chemical components							
Ca (%)	1.65	1.10	0.669	3.21	7.62	0.576	7.95
Mg (%)	19.9	19.5	18.8	18.5	4.75	1.30	7.11
Mn (%)	0.085	0.099	0.095	0.111	0.106	0.029	0.122
S(%)	0.362	0.110	1.28	0.440	0.029	0.002	0.047
Ni (%)	0.194	0.205	0.335	0.301	0.019	0.032	0.041
Fe (%)	7.97	8.20	10.4	8.47	8.06	4.26	8.71
Cu (%)	0.017	0.010	0.077	0.055	0.026	0.000	0.006
Al (%)	2.02	2.07	1.87	2.37	8.02	9.08	5.85
Relative abundance of mineral phases							
Quartz	L	ND	ND	ND	L	A	Tr
Albite	ND	ND	ND	ND	A	M	M
Augite	L	L	L	A	ND	ND	ND
Actinolite	ND	ND	ND	ND	ND	L	ND
Lizardite	M	M	M	M	ND	ND	ND
Talc	ND	ND	VA	ND	ND	ND	ND
Chlorite	VA	VA	A	A	L	L	M
Magnetite	M	L	L	M	L	Tr	Tr
Hornblende	L	L	Tr	L	A	ND	VA
Pyrite	Tr	Tr	Tr	Tr	Tr	Tr	Tr
Pyrrhotite	Tr	Tr	Tr	ND	ND	ND	Tr
Chalcopyrite	Tr	Tr	Tr	Tr	Tr	Tr	Tr
Pentlandite	Tr	Tr	L	L	Tr	Tr	Tr

VA very abundant (40-60%), A abundant (25-40%), M medium (10-25%), L low (1-10%), Tr trace (<1%), ND not detected

## 4 COLUMN TESTS RESULTS

Results of water quality evolution during column testing are presented in this section. The variation of the main parameters and elements involved in the weathering processes (mainly oxidation and neutralisation reactions) are presented in Figure 4. Note that results from the original columns and their duplicates (C1-C8 and C3-C9) are similar, confirming the reproducibility of the tests; results from the duplicates will not be discussed further in the following (see Ethier 2010 for details).

### 4.1 Variations of pH, Eh and conductivity

The pH remained near-neutral for all columns and for the total duration of the test. Values are close to 7.5 for C2 and C6 and were close to 9 for the other columns. The Eh remained most of the time between 200 and 500 mV, with a value of approximately 250 mV for all columns at the last flushing cycles, attesting that the test was conducted under an oxidant media favourable to sulphides oxidation. The conductivity of all leachate samples, except for C10, decreased with time to reach a plateau at approximately 50  $\mu$ S/cm. These values are low and reflect the low reactivity or leaching rates of the rocks.

### 4.2 Acidity and alkalinity

Almost all samples produced no acidity; C2 and C6 produced effluents with measurable acidity values of 4 to 5 mg CaCO<sub>3</sub>/L (values close to the precision of the test). The alkalinity value of the leachates decreased with time to plateaus of 16-18 mg CaCO<sub>3</sub>/L for C3, C7, C9 and C10, 9-11 mg CaCO<sub>3</sub>/L for C4, C5 and C8, and 5 mg CaCO<sub>3</sub>/L for C2 and C6.

### 4.3 Main oxidation and neutralisation products

The main species related to the oxidation-neutralization phenomena (Ca, Mg, S and Ni) measured in the water collected from the columns are presented in Figure 4. Results are presented as milligrams of the measured species per kilogram of rock. For all columns, loadings decreased to reach plateaus after 100-150 days, except for species that were close to the detection limit value. One can also see that the amount of reaction products is higher for C10 than for C4. The presence of fine particles provided a higher specific surface area, which increases the reactivity.

### 4.3.1 Calcium and magnesium

The main neutralization products probably come from silicate minerals that dissolve when sulphides oxidize. The main silicates contained in the rocks (pyroxenes, amphiboles and phyllosilicates) have an intermediate neutralizing potential (Aubertin *et al.* 2002 from Sverdrup 1990). The impact of possible traces of carbonates and the higher calcium and magnesium loadings at steady state produced by C10 were discussed in more details in Ethier, 2010. Calcium can be leached by augite, actinolite, and hornblende.

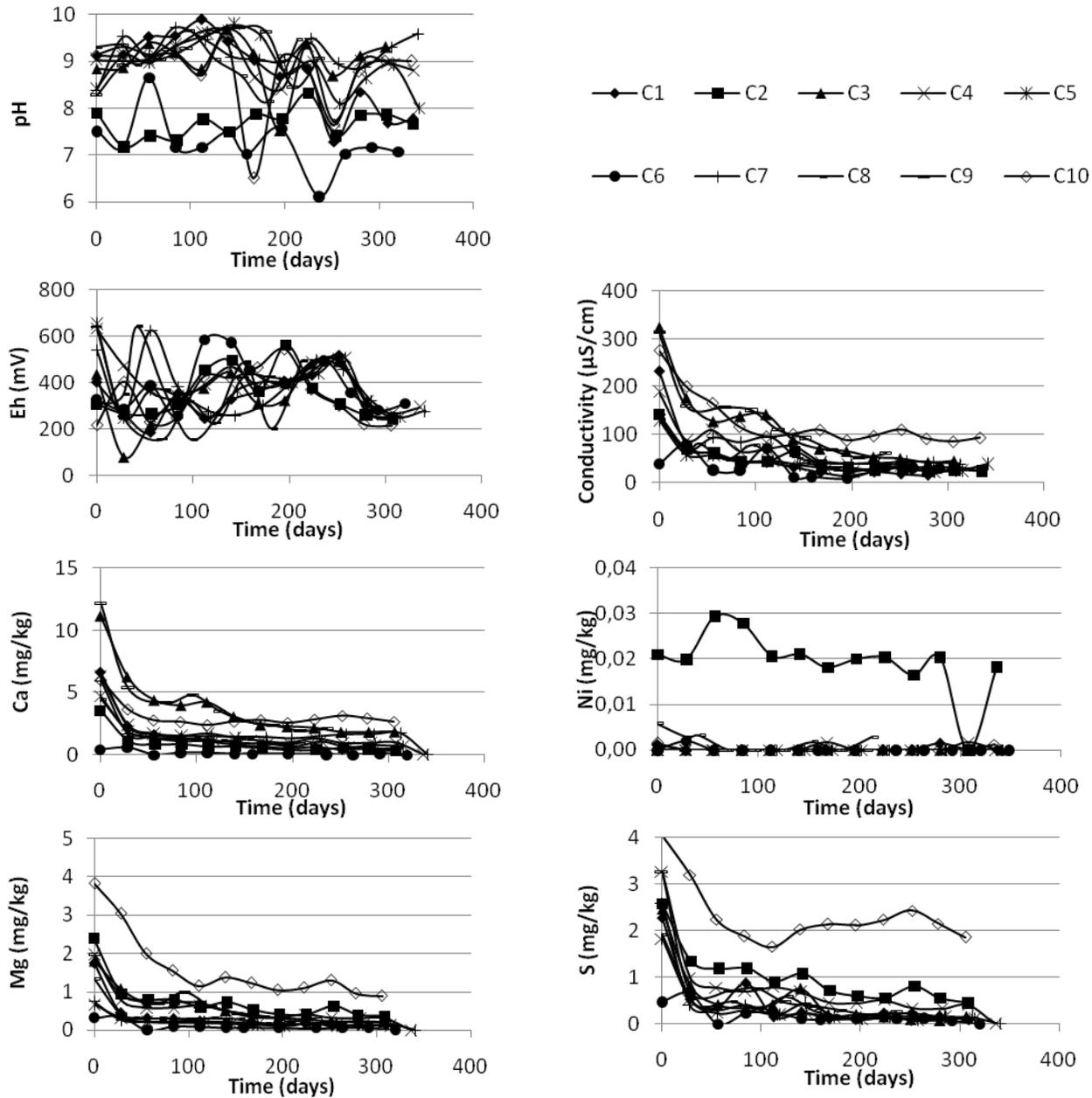


Figure 4. Water quality corresponding to the column tests on the Raglan waste rock samples

Figure 4 shows that calcium is leached mainly by C3 and C7; after 100 days, Ca loadings were between 0 and 5 mg/kg. The calcium loadings were almost nil for C2 and C6. Low loadings of magnesium were also measured with values less than 0.5 mg/kg, except for C2 that contains high proportion of lizardite and chlorite. The Mg loadings were usually between 1 and 2 mg/kg.

#### 4.3.2 Sulphur

The sulphur is assumed to be entirely in the sulphate form in the flushing water as shown in previous studies and as predicted by thermodynamic equilibrium modelling (Ethier 2010). The source of sulphur is from the oxidation of the main sulphide minerals pentlandite, chalcopyrite, pyrrhotite and pyrite (see Table 2). Figure 4 shows that C10 (which contains fine particles) produced the highest sulphur loadings (usually between 2 and 3 mg/kg). Column C2 produced the second highest sulphur loadings with values between 0.5 and 1 mg/kg, followed by C4 with values between 0.2 and 0.6. The other columns have sulphur values usually less than 0.5 mg/kg. The higher oxidation in C2 could lead to a greater acid production, and this fact combined with the low calcium production could explain the lower pH and higher acidity values, compared to those in the other columns. Despite a higher sulphur production in C4, the higher calcium production in that column would explain its high pH (between 8 and 9).

#### 4.3.3 Nickel and iron

Figure 4 shows that C2 is the only column that leached nickel at non negligible loadings of approximately 0.02 mg/kg Ni. Analyses of the iron concentration are close to or below the detection limit, and Fe is present in sulphides and in some silicates. It is possible that most of the iron produced precipitated as ferric hydroxide solids due to the neutral pH. Based on the chemistry and mineralogy, Peridotite from Kikialiq is probably not the only rock type to produce nickel, and other phenomena such as sorption could retain the nickel in the other rock types and impede its release in the flushing water. This hypothesis is discussed further in section 5.

## 5 DISCUSSION ON THE LONG-TERM GEOCHEMICAL BEHAVIOUR

Geochemical behaviour of the test results were further explored by considering the ratio of neutralization elements to oxidation elements (oxidation-neutralization curves) and by performing sorption tests.

### 5.1 Oxidation-neutralization curves

The long term acid generation potential is a concern for the rock types investigated because the static ABA prediction tests showed results in the uncertainty zone (Bussière et al. 2008). It was seen in the column tests that pH of the flushing water remained neutral for the duration of the test, suggesting that there is no acid generation in the short term. For the long term prediction,

the oxidation-neutralization curves were drawn in accordance with the criteria proposed by Benzazoua et al. (2001 and 2004). This approach assumed that the geochemical environment (represented by pH, Eh, temperature, the ratio between the neutralizing and oxidation products) stays constant (see Villeneuve et al. 2009 for more details). It is also important to keep in mind that the neutralization from the Raglan waste rock is provided mainly by the silicate minerals, whereas the method was developed for materials where the neutralization is provided by carbonate minerals.

This method consists of plotting the cumulative loadings of the neutralizing elements (Ca, Mg, and Mn in this case) against the cumulative sulphur loads. The linear relationships obtained are called "oxidation-neutralization curves". Oxidation-neutralization curves for the ten column tests are shown in Figure 5 (left graph). The slopes of the regressions (m) vary from 1.23 to 12.85 and the correlation coefficients ( $R^2$ ) are near 1, showing a linear behaviour. The correlation parameters are summarized in Table 3.

It is possible to predict the long term acid generation potential by plotting the data representing the initial total sulphur content and Ca, Mg, and Mn concentrations of rocks on the same graph. If the point representing the initial Ca+Mg+Mn/sulphur concentration is below the oxidation-neutralization projection line, the material is considered as acid generating because the neutralizing minerals will be depleted before the complete depletion of sulphide minerals. All samples have initial compositions that are located above their respective regression lines, so they would contain sufficient amounts of neutralizing elements to neutralize all acid generated by sulphide oxidation.

Table 3. Oxidation-neutralization regression slopes (m) and regression coefficients ( $R^2$ ) values

Column	m	$R^2$
C1	3,95	0,986
C2	1,36	1,000
C3	12,85	0,985
C4	3,06	0,999
C5	6,49	0,981
C6	1,23	0,990
C7	6,80	0,959
C8	4,06	0,991
C9	8,19	0,998
C10	1,87	0,996

### 5.2 Contaminated neutral drainage potential

As mentioned in the previous section, elevated nickel leaching occurs only from C2 – Peridotite from Kikialiq at a relatively constant and non negligible rate. However, this type of rock contains lower or similar total quantities of nickel, sulphur and sulphides than C1 – Peridotite from mine 3, C3 and C4 – Olivine-pyroxenites from mines 3 and 2. This distinct behaviour could be due to the lower pH in this column compared to the others, where the solubility of nickel and the oxidation rates of sulphides would be higher.

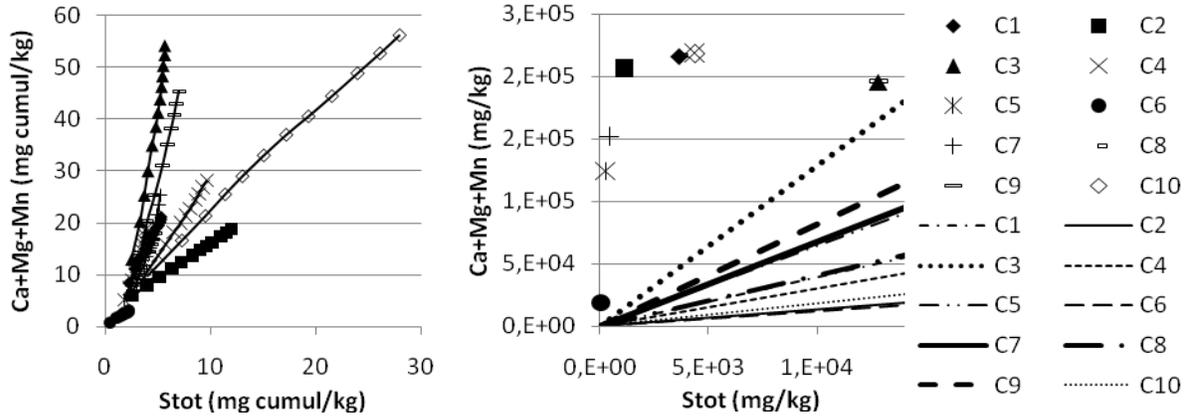


Figure 5. Relation between oxidation and neutralization for the long term AMD potential prediction for the 10 columns. The figure on the left shows the regressions of the Ca+Mg+Mn curves vs S while the right figure shows the extrapolated regressions (lines) and the initial chemical composition of the waste rocks (represented by a symbol)

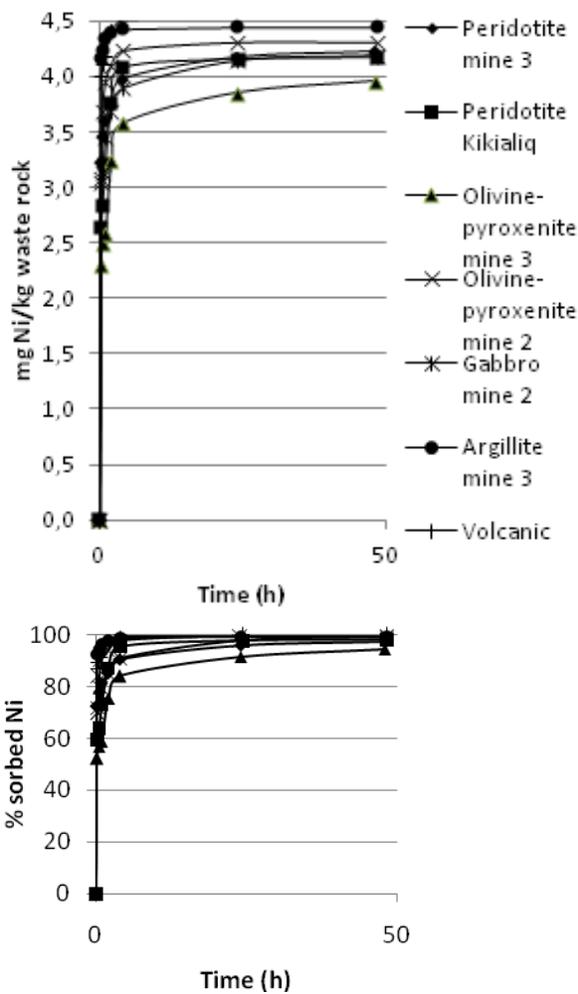


Figure 6. Static sorption tests results

Another hypothesis is that there is sorption of  $Ni^{2+}$  ions in the waste rocks, which reduces the Ni concentration at

the effluent as suggested by Rinker et al. (2003). Static batch sorption tests were performed on small quantities of each rock type of finer dimensions than the column samples to further investigate the impact of sorption on nickel leaching, as explained in section 3.3.

The main results are presented in Figure 6; the first graph shows the mass of Ni sorbed per mass of rock, while the second graph shows the percentage of the initial Ni contained in the water. The initial nickel concentration of 10 mg/L was rapidly sorbed for all rock types. The sorption was higher and faster for argillite and volcanic, and lower and slower for the olivine-pyroxenite. Many parameters can influence the different Ni sorption kinetic, including pH and oxidation rates. More information can be found in Ethier (2010). Therefore, recently extracted rock from the Raglan Mine could retain a significant quantity of nickel by sorption. When sorption sites are saturated and cannot accept more  $Ni^{2+}$ , nickel concentration in drainage water could increase. A lag time is consequently expected before reaching the maximum concentration in effluent or rock drainage. More work is necessary to better understand the mechanism of Ni retention and release in Raglan rock to be more representative of real site conditions (temperature, contact time between rocks and water, grain size distribution, etc.).

## 6 CONCLUSION

The series of column tests performed on seven different rock types from the Raglan Mine showed that they are not expected to be acid-generating suggested by the results of the oxidation-neutralization curve method. Results also showed that at least one type of rock, the peridotite from Kikialiq, can potentially generate neutral drainage with elevated nickel concentrations. Other rock types in this study (peridotite from mine 3 and olivine-pyroxenites from mines 2 and 3) also contained sulphide minerals and nickel concentrations similar to the peridotite from Kikialiq but had lower nickel loading rates. The low loadings of Ni from other rock types could be due to the sorption phenomenon that would temporarily retain the metal on

the rock surfaces. More tests with saturated sorption sites will be performed to assess the long term nickel generation risks of these rock types.

A more detailed mineralogical study with a scanning electron microscope equipped with a microanalysis system (energy dispersive spectroscopy) on sulphides concentrates obtained by sulphide flotation is planned in future studies to evaluate the source of nickel. As the Raglan mine is located in a Nordic region, mini-alteration cells (small humidity cell tests; see Hakkou et al. 2008 for details) will also be performed at different temperatures to investigate the effect of this parameter on mineral reaction rates in permafrost terrain.

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