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Analysis of shake flask experiments results conducted on residues from hydrometallurgical processes

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ABSTRACT: Hydrometallurgical facilities processing sulfide based ores produce waste residues in the form of sludges that contain concentrations of metals, as well as metal sulfides. As a part of the waste characterization and risk assessment process, a statistical design of experiment was used to assess the significant factors and interactions in the residue leaching process. Two shake flask experiments, a 2⁴ factorial design and 2³ central composite design were employed to evaluate the effect of mixing time, test pH, solid/liquid ratio and residue type on acidity, alkalinity, sulfate and metal concentration and pH of the resulting filtered leachate. The results indicate that the variable tested mixing time and solid/liquid ratio most strongly affect metal concentration in the filtrate from waste residue samples tested over a moderate test pH range. When tests were conducted over a longer test period and at lower test pH values, test pH and residue type were dominant factors contributing to residue filtrate metal concentration.

Key words: Process waste, leaching, characterization, batch tests, acid generation, factorial design

INTRODUCTION

The ore at the Voisey's Bay mine site in Newfoundland and Labrador exists mainly as nickel sulfide (pentlandite). Traditionally, when a smelter is employed to refine nickel sulfides, the deleterious minerals are removed from the matte in the form of a slag containing large quantities of iron and the sulfur is partitioned from the matte to the air in the form of SO₂ which is a major source of acid deposition. Vale Inco and Voisey's Bay Nickel Company (VBNC) is testing a novel hydrometallurgical process to refine its Ni, Co and Cu from the nickel sulfide concentrate. In this work, solid residue from this process is tested through shake flask experiments in order to provide relevant information related to the prediction of metal release to the environment. Initial testing of the Vale Inco pressure oxidative leach (POL) process was conducted at a 1:1000 scale plant (mini-plant) at Vale Inco's Sheridan Park facility in Mississauga, Ontario. A larger scale (1:100) demonstration plant was constructed in Argentia, Newfoundland and operated between October 2005 and June 2008 and the full scale facility is expected to be under construction by 2009 and operational by 2011. In the hydrometallurgical

process, a significant amount of the sulfur from the ore is dissolved, neutralized and then precipitated out largely in the form of CaSO₄.2H₂O (VBNC, 2006a). The process eliminates SO₂ emissions and transfers the sulfur into wastewater and residue. Residue and liquid wastes are easier to handle from a pollution control perspective but sulfur is not eliminated. In addition, as there has been limited experience in the use of hydrometallurgy to process nickel sulfide concentrate, the characterization information on process residue is limited. Although, the process residue will be neutralized before it is sent for disposal, it is important to perform both short and long term tests on the residue as it contains a significant percentage of sulfur and sulfur compounds. The tests will assist in determining its acid generating potential and metal leaching capacity with time and provide information to determine optimal treatment/mitigation/disposal options and the associated risks. These shake flask experiments are one of the established, kinetic tests used to predict the release of metals from mine waste to the environment (MEND, 1991).

The methodology for conduct both static and kinetic testing on mine waste is well documented (Price and Errington, 1998; MEND, 2000; Morin and Hutt, 2001b)

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and assists in predicting drainage chemistry. The kinetic tests assess the influence of time on the leachate characteristics of the mine materials and can include shake flask, humidity cell, column and lysimeter tests and large test cells. A comparison of different types of kinetic tests has been conducted by Bradham and Caruccio (1991) and different humidity cell methodologies were investigated by Frostad et al. (2000). Humidity cell experiments are often used to simulate weathering conditions experienced by subaerial disposal of waste rock and mine tailings and result acid rock drainage (Li and Bernier, 1999; Verburg et al., 2000; Morin and Hutt, 2000a; Lappako, 2003). Column tests are also widely used to assess mine leachate either through submerged tailings or after simulated rain events as reported by Li and Arnaud (1997), Doepker (1991) and Chapman et al. (2000). Lysimeter tests provide additional control of water flow through and across subaqueously deposited mine waste material and have been described by Dave and Paktunc (2003) and Tabouda et al. (1997). The shake flask experiment is simple and inexpensive to set up

typically a shorter duration test than the humidity cell, column or lysimeter. Variations in experimental methodologies used with examples have been described by Filipek *et al.* (1991) and Gleisner and Herbert (2002).

The shake flask experiment has been conducted to assess the leaching conditions of specific minerals, waste rock or tailings (Marchand and Silverstein, 2000; 2002; Harahuc *et al.*, 2000; Renman *et al.*, 2006). Frostad (2003) used shake flask experiments to aid the interpretation of kinetic test results. Johnson and Bridge (2002); Bilgin *et al.* (2004) and Darkwah *et al.* (2005) considered the effect of strains of bacteria on sulfide mineral oxidation while Widerland *et al.* (2005) used shake flask experiments to examine the effect of adding fresh water to an existing tailings impoundment. Results from this type of test on hydrometallurgical residues have not been widely reported in the literature.

Sampling site description

The hydrometallurgical plant concentrate feed is shipped from the mine site at Voisey's Bay located on



Fig. 1: Location of Argentia demonstration site and Voisey's Bay mine site

Labrador's North coast to the hydrometallurgical demonstration plant situated approximately 150 km west of St. John's, Newfoundland in Argentia (Fig. 1) in close proximity to the proposed location of the full scale facility.

Hydrometallurgical residue samples were taken from test campaigns conducted during the period of March through October 2006. The residues from the plant are derived through either precipitation processes or pressure leaching in the form of sludges.

Three main sources of sludges/residues are: 1. The solids remaining, neutralized leach residue (NLR), when the pulp from the pressure leaching is washed by counter current decantation (CCD);

2. The precipitate, neutralized gypsum residue (NGR), formed during the iron removal and neutralization stage; 3. A final source of sludge which is the precipitated metals impurities stripped from solution after the cobalt, copper and nickel have been removed. Under current strategy, this stream will not be combined with the residues. Each of these sludges has a solid and liquid portion. The solid waste from the hydrometallurgical process is approximately 60 % NLR, 40 % NGR and minor amounts of solids from water treatment processes. The current plan for the full scale facility is to combine the NLR and NGR as a neutralized combined residue (NCR) prior to disposal.

The NCR will be mixed with wastewater process effluent neutralization (PEN) to approximately 40 % solids and the slurry will be pumped to the residue disposal pond where the waste will remain under a water cover (Vale Inco, 2008).

The amount of residue predicted to be produced from the demonstration plant is 3500 ton/y while the full scale facility is approximately 375,000 ton/y (VBNC, 2002, VBNC, 2006b). For the full scale plant, Vale Inco has proposed the NCR-PEN slurry to be neutralized and then pumped into an existing natural pond for disposal (Vale Inco, 2008). Any discharge from the pond will be treated to meet regulatory guidelines.

The waste residue from the plant will contain a high percentage of sulfur and sulfur compounds, as well as a small percentage of non processed sulfide minerals. There is the potential that the present sulfur and sulfur compounds could be oxidized to form acid and subsequently cause leaching of metals from the residue or bedrock and acidification of disposal pond surface water. The oxidation of the iron sulfide mineral, pyrite, on exposure to dissolved oxygen and ferric iron has been described by Evangelou, 1998. Pyrite oxidation has been well documented in the literature and is well understood in comparison to that of pyrrhotite which is the main iron sulfide mineral present in the concentrate (Nicholson and Sharer, 1990; Belzile *et al.*, 2004). Nicholson and Sharer (1990) have reported on the oxidation pyrrhotite.

MATERIALS AND METHODS

Two shake flask experiments have been conducted on the demonstration plant hydrometallurigcal residues. The objective of these shake flask experiment is to assess how the chemical properties of water changes when exposed to differing concentrations of NGR and NLR over a relatively short term.

Residue composition

Scanning electron microscope (SEM), X-Ray diffraction (XRD) and elemental analysis (Steel *et al.*, 2006) indicate that NGR contains a high percentage of gypsum particles and small percentage of iron hydroxide particles with minor quantities of nickel hydroxides and other metal hydroxide compounds as well as metals adhered to iron hydroxides. NLR appears to consist primarily of Fe_2O_3 and amorphous iron sulfide and small quantities of unprocessed concentrate and sulfur.

The residues contain relatively high concentrations of Ni (0.2 %-1.1 %) Cu (0.05 %-0.6 %) and Pb (0.008 % -0.011 %) and the sulfur contents of NGR and NLR are in the order of 21 to 32 percent, respectively with the sulfate concentration of 54 percent and 6 percent, respectively. The metals appear to be, for the most part, associated with the iron hydroxides in NGR and iron oxides in NLR.

Experimental design

A factorial design of experiment was used in all shake flask experiments to optimize the required number of runs. The objective of the first experiment (experiment # 1), a 2⁴ factorial, was to explore the effect of various factors on the basic chemistry and metals concentration of the residue filtered leachate (filtrate) solution. The objective of the second experiment (experiment # 2), a 2³ centre composite design (CCD), was to explore the effect of longer mixing times and a broader pH range and to verify and improve relationships between the tested factors and the

Summary of experimental conditions for 2^4 experiment # 1							
Factor name	Units	Low level	Mid level	High level			
Test pH		3	4	5			
Mixing time	Days	2	8	14			
Solids/Liquid ratio		0.05	0.19	0.33			
Residue type		NLR		NGR			
Summary of experimental conditions for 2 ³ experiment # 2							
Factor name	Units	Low level	Mid level	High level			
Test pH		2	3.5	5			
Mixing time	Days	2	14.0	26			
Residue type		NLR		NGR			

Table 1: Summary of shake flask experimental conditions

Table 2:	Experimental	responses	on	filtrate	solution
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Response name Units		Normal instrument range	Method/equipment	
*		(Measurement resolution)	• •	
pH		1-14 (0.005)	Oakton pH1100/2100 combination pH electrode	
Electrical conductivity	mS/cm ²	$200-1999 \mu\text{S/cm}^2$ (5% full	Hach CO150 conductivity meter	
-		scale: $300 \mu\text{S/cm}^2$)	·	
Redox potential	mV	-2000 to +2000 (1 mV)	ExTech orp Electrode with Oakton pH1100/2100 meter	
Acidity	mg/L CaCO3	0-500 typical (5 mg/L CaCO ₃)	Titration with NaOH to 8.3 endpoint	
Alkalinity	mg/L CaCO3	0-500 typical (5 mg/L CaCO ₃)	Titration with HCl to 4.3 endpoint	
Sulfate concentration	mg/L	0-70 (3 mg/L without dilution)	Spectrophotometric: barium sulfate method	
Ferrous iron concentration	mg/L	0-3.00 (0.03 mg/L)	Spectrophotometric: 1,10 phenanthroline method	
Other metals	ppb	Variable 10-500 ppb (15 %)	Varian graphite furnace AA	

responses of the filtrate solution. The factors involved in two experiments are outlined in Table 1; they were pH of the test solution, mixing time, solids ratio (i.e. the mass of solids/mass of liquids) and residue type.

Twenty separate test runs were conducted for experiment # 1 and twenty four for experiment # 2 with the factor levels as shown in Table 1.

Experimental procedure

The glass and plastic labware used for the shake flask experiments was soaked in 2N HNO_3 for at least 24 h. After soaking in acid, all equipment was triple rinsed in nano pure distilled and deionized water. In this paper, the filtrate refers to liquid from the experiment that has been filtered using sterile disposable millipore 0.45 µm syringe type filters. One blank was run for every 10 samples for periodic checks on test procedures. The test procedure was as follows:

The samples of NLR and NGR were air dried. 10 g, 38 g or 50 g of solids were weighed into separate 250 mL erlenmeyer flasks to which 200 g, 200 g and 150 g, respectively of nano pure water was added to provide solids ratios of 0.05, 0.19 and 0.33. pH of the nano pure water was adjusted to the test pH with addition of hydrochloric acid as measured by the pH meter (Oakton, pH2100 series meter). The Next flasks were secured on a shaker table (VWR OS-500 shaker table) set at speed of 4.5 (relative 4.5/10) for the predetermined mixing period (2, 8, 14 or 26 days). The residue and water were fully mixed for the duration of the test period.

At the end of the mixing time, samples were allowed to settle and the supernatant was filtered through 0.45 µm filters into plastic containers. The types of responses measured on the filtrate are outlined in Table 2 along with the parameter normal range and the method of analysis. The measured metal concentrations included Fe, Ni, Cu, Co, Zn and Pb concentration. Sulfate and ferrous iron were measured by spectrophotometric methods with Hach DR/2000 spectrophotometer. Acidified samples were stored at approximately 4 °C until the individual metals analysis was conducted in duplicate by Varian Inc. atomic absorption graphite furnace. Approximately 20 % of the metals results were verified through Integrated catchment management plans (ICMP) and duplicate runs were made of 25 % of the samples. Also, Table 2 indicates the measurement resolution for each response.

RESULTS AND DISCUSSION

The data points from experiment # 1 were initially entered in stat-ease design expert for assessment of main effects, interactions, analysis of variance, determination of regression equations, evaluation of diagnostic plots and model graphs. As a first approach, the response relationships for experiment # 1 were assumed to be linear. Using this analysis, a description of the main trends and interactions of experiment # 1 were determined. Example plots of the results are shown in Figs. 2, 3 and 4. The data scatter evident in the plots can be attributed to the heterogenity of the samples, the difficulty in controlling the test water pH, especially at lower test pH values, the error associated with diluting samples (sulfate measurement) and potential

interference from other dissociated species (ferrous iron measurement).

The second experiment was conducted to assess the filtered leachate response over a wider test pH range (pH 2-pH 8) and long test times (2-26 days) while the solid to liquid ratio was held constant. In this experiment, intermediate data points were also added. Sample graphs of the results are provided in Figs. 5 and 6.

The duplicate sample results are not included in these plots due to the number of points, but the data showed the same trends. In general, the main factor



Fig. 2: Plots of mixing time versus filtrate pH, acidity and sulfate concentration at varying solids ratios from experiment # 1





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Fig. 5: Plots of mixing time versus filtrate pH, conductivity and alkalinity at varying test pH values from experiment # 2

affecting the responses was the test pH. In experiments # 1 and # 2, the effect of various factors on the chemistry of the filtered leachate solution from the hydrometallurgical plant residue was explored. Each of the experimental factors is now considered separately and discussed to assist in understanding the experimental responses. The geochemical modeling code (PHREEQC) (Parkhurst and Appelo, 1999) is used to consider the response of individual minerals in

solution, as well as the response of the minerals mixed in proportions similar to that found in each residue.

Experimental results

The solids ratio varied from 0.05 to 0.38 and had a strong effect on most parameters measured in experiment # 1 while being held constant in experiment # 2. It was the most significant factor in terms of pH and conductivity for NLR and sulfate concentration of



Fig. 6: Plots of mix time versus filtrate So⁻², Ni and Cu concentration at varying test pH values from experiment # 2

the filtrate for both residues. The interaction of the residue type and solids ratio was the most significant factor for filtrate acidity, alkalinity and cobalt concentration for both residues and nickel concentration for NGR. Mixing time and solids ratio was the main interaction factor affecting iron concentration. Increasing the solids ratio causes an increase in the conductivity and sulfate concentration for both residues through increased

ions in solution. NGR alkalinity decreases slightly with increasing solids ratio and the reverse is true for acidity. It was found that NGR acidity increases and NLR acidity decreases with increasing solids ratio. As NLR is highly neutralized with lime slurry (CaCO₃), a higher solids ratio translates to a higher lime content which in turn rapidly goes into solution to reduce acidity in the short term. For NLR, cobalt and ferrous iron concentration are increased with increasing solids ratio and to a lesser extent, nickel follows the same trend. For NGR residue, this trend is not as dramatic although the metal concentration is generally elevated with higher solids ratio.

Dissolution of calcite in NLR follows reaction (1), therefore, an increase in calcite concentration produces an increase in solution alkalinity and pH.

$$CaCO_{3} + H_{2}O \rightarrow Ca^{2+} + HCO_{3} + OH^{-}$$
(1)

The mixing time varied from 2 to 14 days for experiment # 1 and from 2 to 26 days for experiment # 2. Mix time was not the primary factor for any of the responses, however, it did impact the pH response, ferrous iron concentration, acidity and sulfate concentration (NLR only) for experiment # 1.

Increasing mixing time caused a slight increase in pH response. This is initially due to the pH of the filtrate being driven by the test pH then with longer test times the test solution was neutralized by the gypsum in the NGR and the lime in the NLR. Mixing time permitted more ferrous iron to go into solution.

As the NLR mixing time increased, the pH of the initially highly neutralized solution could drop due to the oxidation of sulfide minerals. With the wider experimental pH range (pH 2 to pH 8) of experiment # 2 mix time was significant only for filtrate sulfate concentration (decreasing slightly after the 14 day mix time). The effect of mix time has to be considered with the neutralizing capacity of the residues and the role of the sulfide minerals. The slight decrease in the residue filtrate sulfate concentration after 14 days could be in part due to sulfate being adsorbed on to the walls of the glass container or sulfate combining with other available ions in solution.

Further experimental work on mixed residue results from humidity cells tests will confirm the effect of extended mix times. In experiment # 2 when the test pH drops below 2.5, it is the most significant model term for filtrate pH, conductivity, sulfate, acidity and metals concentration. Extending the test pH from pH 5 to pH 8 had limited the effect on measured responses with the most noticeable being on sulfate concentration. Generally, the sulfate concentration was higher, the redox, the acidity and metals concentration was lower with higher test pH. These results indicate that an elevated pH as encountered when residue is disposed initially in a disposal pond may initially prevent metals from going into solution. As indicated previously, residue type is involved in the significant interaction effect for response pH, conductivity, acidity, alkalinity and metal concentration for experiment# 1 and # 2. The NLR metals may be less available than in the NGR for two reasons:

1. The NLR is strongly neutralized with lime slurry prior to disposal which will have a strong impact on responses at short mixing times.

2. In addition, the metals in the NLR may be more strongly bound by the micro structure of the iron oxide particles (Chen, 2006 and Steel, 2006) than in the metal hydroxide particles of the NGR. The NGR, on the other hand, is disposed without further treatment, therefore, is more strongly impacted by changes in the leach pH, the mix time and solids ratio.

Model results

The geochemical modeling code (PHREEQC) has been used to assess the impact of changes in the pH of water added to the main residue minerals on the concentration of dissolved species and final solution pH. Initially individual minerals were studied then minerals were combined in similar ratios as those estimated to be present in the actual residue and this modeled residue was evaluated. The minerals included goethite, gypsum, calcite, hematite, magnetite, pyrite, pyrrhotite, pentlandite, chalcopyrite, sulfur and FeS(ppt) (a freshly precipitated, less stable and more crystalline mackinawite). FeS (ppt) is used to represent the amorphous iron sulfide in the NLR. As both Fe₂O₂ modeled as hematite and iron sulfide modeled as FeS (ppt) appears to be present in the NLR in an amorphous form, the database equilibrium formulations may not accurately represent the compounds present. In addition, the PHREEQC batch simulations are equilibrium based which may not be achieved in the relatively short duration of the experimental shake flask experiments. However, this work reveals the long term trends of the minerals present.

PHREEQC results indicate when one mole of the individual minerals was added to 1 L of pure water, the minerals least affected by changes in test pH were hematite and goethite while gypsum, ferrihydrite, magnetite and FeS (ppt) gave final pH values slightly higher than that of the test pH. Pyrite, sulfur, pyrrhotite, pentlandite and chalcopyrite were not greatly affected by changes in test pH and resulted in final pH values between pH 2 and pH 5. Calcite was somewhat affected by test pH and with final solution pH values ranged from pH 8 to pH 10.

In the next stage, the NGR minerals gypsum and goethite were equilibrated with water at pH 2 through pH 8 at molar ratios similar to that found in the actual NGR. The final pH values were slightly higher than the test pH, ferrous iron concentration decreased with increasing test pH, sulfate ion concentration was fairly stable, hematite saturation index was above zero.

A non-neutralized NLR mineral composition was approximated with the minerals hematite, magnetite, sulfur, FeS (ppt) and a very minor amount of pyrrhotite, pentlandite and chalcopyrite to represent the portion of unprocessed concentrate. Again mineral molar ratios were similar to that found in the actual NLR. The high oxidizing strength of the sulfur and sulfide minerals dominated the composition of the resultant solution at all test pH values resulting in a final solution exhibiting a pH below 5. In general, the test pH only affected the concentration of ions in solution at lower test pH values. When 10 % calcite mineral was added to the NLR composition, the final solution pH was elevated above a pH of 5 at all test pH conditions. Gypsum, CO₂ and pyrite were supersaturated when calcite was added to the NLR. Only pyrite was supersaturated without the calcite mineral. The value in the PHREEQC simulations was to gain an understanding of the minerals having the greatest impact on final solution pH over a range of initial pH values and to identify compounds that precipitate out of solution.

The trace metals associated with the residues could exist in several different forms including: Sorbed to surfaces of the minerals, part of the unprocessed concentrate, precipitated hydroxides or oxides, or within the crystal structure of the minerals. It is expected that solution activity of trace metals will follow that of the minerals with which they are associated: Gypsum, iron hydroxides, iron oxyhydroxides, iron oxides, unprocessed concentrate and iron sulfide. To summarize the considered factors, including solids ratio, mix time, test pH and residue type, the test pH and residue type were the main factors that affected the majority of filtered leachate experimental responses. Residue type was a main factor in most of the responses over a range of test pH values; this reflects the importance of considering the very different nature of the two residues. Test pH was the most significant factor when it was lowered to pH 2. Results suggest that a significant drop in solution pH is required before a noticeable change in metal concentrations unless the solution solids ratio is elevated. In several cases, it was the interaction between two factors (such as residue type and pH) that constituted the main effect on the response. Solids ratio had a significant effect on the filtrate metal and sulfate concentration, conductivity and alkalinity. Mixing time was not a significant factor for most of the responses probably due to the relatively short test duration and the strong effect of the other factors on the responses of the fresh residues. Tests of this nature are valuable in the understanding of factors having short term affects on the chemistry of waters containing appreciable amounts of residue such as the surface water in a residue disposal pond. Further shake flask experimental work will be conducted on mixed residue (NGR and NLR) at proportions similar to that proposed for disposal at the future hydrometallurgical plant to elucidate the effect of longer mix times and the synergistic effects due to the mixed residue chemistry. Geochemical modeling is a useful tool to highlight the minerals most affected by pH variations and driving changes in solution pH.

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