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VALIDATION OF NICKEL LATERITE DIAGNOSTIC LEACHING PROTOCOL

By

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ABSTRACT

A typical nickel lateritic ore body contains several weathered zones. The limonitic and saprolitic zones exist closest to the surface and towards the bottom of the deposit, respectively, and there may also be an intermediate zone present, composed of clays. Nickel in these deposits is expected to be hosted mainly in goethite, clays (*e.g.* nontronite) and serpentines (*e.g.* lizardite). An understanding of ore mineralogy and elemental deportment is vital for the effective processing of nickel from an ore body. Common characterisation techniques, such as Quantitative X-Ray Diffraction (QXRD) and Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN) may be costly, time-consuming and not readily available at mine sites. Furthermore, these techniques may not be effective for nickel deportment analysis: QXRD can be used for major mineral prediction but cannot be used to identify where specifically the nickel is located, while nickel deportment analysis by QEMSCAN may be problematic if the nickel concentrations in minerals are low. A possible alternative to mineralogical analysis for determining nickel deportment is by the selective sequential leaching of nickel-containing minerals followed by the determination of nickel in solution after each leach. This approach could be a simple, accessible, cost-effective and more accurate alternative to mineralogical analysis. Previous literature research has presented sequential leach methodologies for determining nickel mineralogy, but none have targeted all zones of a lateritic ore body. A diagnostic leach for nickel laterite deportment in all three zones has been developed primarily for geometallurgical applications. However, predicted theoretical nickel recoveries by this method have not been compared to those determined from typical atmospheric or ambient leaching used in industry. Laboratory experiments have therefore been conducted to validate the theoretical model by assessing the correlation between the predicted and actual amounts of nickel leached.

INTRODUCTION

A typical lateritic ore body contains several different weathered zones (Figure 1); the limonitic zone closest to the surface and the saprolitic zone towards the bottom. The limonitic zone is predominantly made up of iron oxides, like goethite, while the saprolitic zone is least weathered as it is present in the deepest section of the deposit. It therefore contains mainly silicates such as serpentines. There may be an intermediate zone made up of clays such as nontronite which is usually only present in dry climates. Nickel in these deposits is expected to exist mainly in goethite, clays (*e.g.* nontronite) and serpentines (*e.g.* lizardite).

Figure 1: Typical laterite profile showing limonitic, nontronitic and saprolitic zones.

An understanding of ore mineralogy and elemental deportment is vital for the effective processing of nickel from an ore body. Common ore characterisation techniques, such as Quantitative X-Ray Diffraction (QXRD) and Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN) can be costly, time-consuming or not available at mine sites. QXRD combined with elemental analysis by Energy Dispersive X-Ray Spectroscopy (EDX) is commonly used to estimate nickel deportment. Although QXRD can be used for major mineral prediction it cannot be used to identify where the nickel is specifically located. Furthermore, techniques such as QEMSCAN may be ineffective for nickel deportment analysis; because the number of counts taken per sample is low, analysis using this technique may be problematic if the nickel concentrations in the target minerals are low.

A possible alternative to mineralogical analysis for determining nickel deportment is by the selective sequential leaching of nickel-containing minerals followed by the determination of nickel in solution after each leach. This approach, termed diagnostic leaching, could be a simple, accessible, costeffective and more accurate alternative to mineralogical analysis.

Traditionally, diagnostic leaching employs sequential leach stages to study the elemental speciation in an ore body. The extraction process consists of treating a sample with a series of successive chemical treatments, with each successive stage being more aggressive or differing in nature from the previous step (Hall et al. 1996). The stages often differ by the chemical properties of the extractants, extraction efficiency, experimental parameter effects, the sequence of extractions, matrix effects and the heterogeneity and physical associations in the sample (Kersten and Förstner 1989). By studying the metals removed at each stage, the different levels of mineral substitution can be determined within a sample.

Swamy et al. (2003), Sohlenius and Oborn (2004) and Dold (2003) have proposed sequential leach protocols for determining nickel deportment but none have targeted all zones of a lateritic ore body. Using these protocols as a basis, research was undertaken by Chapman et al. (2012) and a new five-step leach process for the diagnostic leaching of nickel laterite ores and determination of nickel deportment in all zones of the ore body was proposed. Each successive step in the proposed diagnostic leach targets specific mineral types. Analysis of nickel in solution after each leaching step allows for the prediction of the location of the nickel i.e. as water soluble, exchangeable, amorphous, adsorbed, structurally bound or "difficult to leach" nickel. A summary of the leach methodology is as follows:

Step 1: Dissolution of water soluble fraction to determine percentage of water soluble minerals or metals.

Step 2: Dissolution of exchangeable fraction where exchangeable cations are leached with ammonium acetate (1 M CH₃COONH₄, buffered to pH 4.5 using 1 M acetic acid):

$$
X^+ + CH_3COONH_4 \to NH_4^+ + CH_3COOX \tag{1}
$$

Step 3: Dissolution of amorphous fraction where amorphous materials are leached with ammonium oxalate (0.2 M $C_2O_4(NH_4)_2$, buffered to pH 3.0 using 0.2 M oxalic acid):

$$
XY + C_2O_4(NH_4)_2 \to 2NH_4^+ + Y^{2-} + C_2O_2X
$$
 (2)

Step 4: Dissolution of adsorbed fraction where adsorbed elements on mineral surfaces are leached with dilute sulfuric acid (0.05 M $H₂SO₄$):

$$
X^{2+} + H_2SO_4 \to 2H^+ + XSO_4(aq)
$$
\n(3)

Step 5: Dissolution of structurally bound fraction where all oxides and silicates leach using concentrated hydrochloric acid (32 % HCl):

$$
XY + 2HCl \rightarrow 2H^+ + Y^{2-} + XCl_2(aq)
$$
\n⁽⁴⁾

Using these diagnostic leaching steps, Chapman et al. (2012) have been able to characterise the origin of the nickel in selected ore samples. For example, previous tests on selected nickel laterite ores highlighted spinel minerals that were difficult to leach and quartz minerals present in these ores were found to be nickel-barren. Furthermore, a distinction could be made between nickel in the amorphous phases, namely manganese oxides and low-crystallinity hydrated iron oxides, and crystalline minerals such as goethite, nontronite, lizardite and chlorite-group minerals (*e.g.* clinochlore). It should be noted that the independent leaching of these main minerals was not possible using the diagnostic leaching technique and if more than one of these minerals is present, the amount of nickel associated with each mineral cannot be determined. However, the leaching methodology is rapid, uses simple solution analysis, and may be an affordable method to determine whether nickel is present in minerals that are difficult to leach (*e.g.* spinels), amorphous phases and/or leachable crystalline phases. Using these leaches, theoretical nickel leach recoveries can be determined, where the leachable nickel is a sum of all the nickel leached from the five step process and the nickel remaining in the residue after the diagnostic leach has been performed is termed "difficult to leach". Predicted theoretical nickel recoveries by this method have not yet been compared to those determined from typical atmospheric or ambient leaching processes used in industry. If this diagnostic leach protocol is found to be accurate, the use of diagnostic leaching for the characterisation of an ore body would allow for the determination of recoverable nickel and therefore, smarter mine planning (*e.g.* introduction of blending, selective processing of ore) to ensure maximum nickel output.

Typical nickel leaching processes include high pressure acid leaching (HPAL at high temperature and pressure), atmospheric leaching (high temperature, atmospheric pressure) and heap leaching (ambient temperature and pressure). There are a number of processes under development such as the Jaguar process, the sulfation atmospheric leach process and bioleaching (Kyle 2010), the Çaldağ heap leaching process (*e.g.* Oxley et al. 2006), the ARNi process (Pelser et al. 2009) and the Direct Nickel process (McCarthy and Brock 2011). HPAL is the most common process, and the only one of the above processes commercialised to date. It is said to be the most capital intensive, with high operating costs (Kyle 2010) and is normally used only for limonitic ores as magnesium

silicates (serpentines) are high acid consumers. While high capital costs may be acceptable for large project developments, smaller scale projects are investigating the use of atmospheric or ambient leaching which may be less capital intensive, but with higher acid and limestone consumptions (Kyle 2010).

According to Whittington and Muir (2000), sulfuric acid and hydrochloric acid are the most effective acids for use in atmospheric leaching, with sulfuric acid being the most widely used to date. They suggest using an acid with strength greater than 0.25 M, at temperatures of up to 95°C for 2 h. It is also suggested that pre-roasting and reducing agents are used to enhance the extraction of nickel from limonitic ores (Whittington and Muir 2000). Acid consumptions are relatively high for atmospheric leaching and range from 500–700 kg tonne⁻¹ of ore (Kyle 2010). Ambient leaches conducted via laboratory bottle roll tests are often used as an approximation to nickel heap leaching. According to literature, acid consumptions for bottle roll tests vary between 400–800 kg tonne⁻¹ ore (Horizonte Minerals 2012).

The aim of this project was to determine whether the theoretical nickel recoveries obtained from the diagnostic leaching steps developed by Chapman et al. (2012) are valid. The results from the diagnostic leach are not discussed in detail in this paper but are provided in the paper by Chapman et al. The diagnostic leach validation was done by assessing the correlation between predicted and actual nickel leached from six different samples in ambient (bottle roll) leaches. Bottle roll tests were conducted as an approximation to heap leaching at atmospheric pressure and ambient temperature to obtain nickel recoveries for comparison to the diagnostic leach predictions. Atmospheric leach tests at high temperatures were performed prior to the bottle roll leaches using literature-based experimental parameters to obtain a preliminary indication of the sample reactivity and acid consumption.

METHODOLOGY

Ore Preparation

Nickel laterite ores from three deposits (termed Sites 1 to 3) were used in this study. Two samples were used from each of the limonitic and saprolitic zones of the deposits. The ores were dried and milled (P_{80} < 50 µm) using a ring mill to ensure optimal mineral liberation. Mineralogical ore compositions were determined using a Panalytical Empyrean automated powder diffractometer fitted with cobalt long line focus X-ray tube (6.9 keV) between 2θ angles of 5.01-89.98° (at 45 kV, 40 mA). XRD analyses were performed using TOPAS (Version 3) software. Elemental concentrations in the head ore samples were confirmed by fusing the samples with borate flux and dissolving the resulting glass in weak mineral acid. Concentrations of Al, Ca, Cr, Co, Fe, Mg, Mn, Ni and Si in the resultant solutions were determined by inductively coupled plasma-optical emission spectrometry (ICP-OES) using a Varian Vista-Pro instrument.

Atmospheric Leaching

Approximately 150 g of the limonitic and saprolitic ore sample from each deposit were placed into 1 L HDPE plastic bottles, to which 105 g 98 % sulfuric acid and 435 mL de-ionised water were added (261 g L⁻¹ sulfuric acid, pulp density 25 % w/w). The bottles were placed on a shaker table for 6 h at atmospheric pressure and 70–80 °C (Thermoline Orbital shaker or New Brunswick Innova 4080 benchtop incubator shaker). Slurry samples (10 mL) were taken at 0, 0.5, 1, 2, 4 and 6 h intervals for all leaches. Each slurry sample was vacuum filtered, solution collected, solid repulped in de-ionised water, filtered, washed and dried at 40°C. Free acidities in the leach solutions were determined by the oxalate method: 0.1 mL of solution (0.2 mL solution for the 4 and 6 h samples) was placed in a 100 mL beaker and an appropriate amount of 50 % oxalate, adjusted to above pH 12, was added to the beaker. The solution was diluted to ~50 mL with de-ionised water and titrated with 0.05 M NaOH to an end point of pH 7.5 using a Metrohm Titrando 904 and sample processor with Tiamo software. Elemental concentrations in the resultant leach solutions were determined for all kinetic samples using ICP-OES on a Varian Vista-Pro instrument for an elemental suite of Al, Fe, Mg, Ni and Si for all solutions, with Ca, Cr, Co and Mn being analysed where present in the ore, based on original mineralogical analysis. Elemental and XRD analyses of the solid residue from the 6 h samples were determined as for the head ore samples.

Ambient (Bottle Roll) Leaching

Approximately 100 g of limonitic and saprolitic ore sample from each deposit were placed into 2 L HDPE plastic bottles, to which 170 g of 98 % sulfuric acid and 730 mL de-ionised water were added (pulp density 10 % w/w). The bottles were placed on a bottle roller (Labtechnics) for 80 days at atmospheric pressure and ambient temperature. Solution samples (5 mL) were taken daily for five weeks via filtration using a 0.45 µm acro-disc. Once ICP analysis showed extracted metal concentrations reaching a plateau, sampling was continued every 3 to 4 days for the remaining 6 weeks. Free acidities, elemental concentrations and XRD traces were obtained as for the atmospheric leach tests.

RESULTS AND DISCUSSION

Atmospheric Leaching

McDonald and Whittington (2008) discuss that nickel is more readily leached from clay-like silicates, such as smectites and saprolites than limonitic (goethitic) ores when leached with sulfuric acid. This behaviour was confirmed in the atmospheric leaching tests (where part of the aim was to obtain an indication of ore behaviour) as the limonites tended to leach more slowly than the saprolites (Figure 2).

Figure 2: Results from the atmospheric leaches performed on the limonite samples (on the left): Site (a) 1, (c) 2 and (e) 3 and the saprolite samples (on the right): Site (b) 1, (d) 2 and (f) 3 (150 g; 261 g L-1 sulfuric acid solution; 6 h; (a), (b) and (c) at 70 °C; (d), (e) and (f) at 80 °C; \triangle Al, **Fe**, \triangle Ni, \times Ca, \div Co and \times Cr).

A second aim of the atmospheric leach tests was to determine the acid consumption of the samples. It was found that literature guidelines on conventional acid concentrations were insufficient for leaching nickel in the ores and not all of those minerals expected to leach to completion did so.

Table 1 shows the initial and final acid concentrations and highlights the low final acid concentrations in most of the samples except for Site 1.

High recoveries of all measured elements from Site 1 were not achieved under the experimental conditions used, with the maxima being the nickel extractions of 60 and 80 % for the limonite and saprolite ores, respectively. Most of the nickel and iron were extracted from the Site 2 and 3 saprolite samples (Figure 2). The saprolite samples contain predominantly nontronite, serpentine (lizardite) and forsterite. The leaching of metals from the limonite samples from Sites 2 and 3 is incomplete, with recoverable element concentrations still increasing at the conclusion of the tests. This could be because of the high concentration of goethite in the limonite samples: Sites 2 and 3 contain 89 and 87 % goethite, respectively. Goethite is a high acid consumer and could limit the acid available for leaching the other minerals in the ore.

XRD results confirm the leach results in Figure 2. For example, a comparison of the XRD trace of the original Site 2 limonite sample with that for the residue after the atmospheric leach shows that not all the goethite (2θ = 24°) has been leached (Figure 3, main peak highlighted). This further confirms that there was insufficient acid present to leach the ores to completion.

Figure 3: Comparison of XRD traces for i) original sample and ii) residue after atmospheric leaching of Site 2 limonite ore.

Based on the incomplete leaching of minerals in the atmospheric leaching tests resulting from insufficient acid addition, it was decided that mineralogy would be a better predictor for theoretical acid consumption. Calculated acid consumption values were therefore used for the bottle roll tests.

Ambient (Bottle Roll) Leaching

From Figure 4, it can be seen that the nickel extractions for the saprolite ores were near complete within 11 days. Nickel leaching from the limonitic ores was slower, and in some cases incomplete: the Site 2 limonite showed complete nickel extraction after approximately 50 days of leaching, with those from Sites 1 and 3 still continuing to show slightly increasing nickel extractions at 80 days. The more rapid recovery of nickel from the saprolitic compared with the limonitic ores was expected from the atmospheric leach test results and from the review data of McDonald and Whittington (2008).

Figure 4: Results from the bottle roll leaches performed on the limonite samples (on the left): Site (a) 1, (c) 2 and (e) 3 and the saprolite samples (on the right): Site (b) 1, (d) 2 and (f) 3 (100 g sample, 188 g L¹ sulfuric acid solution, 80 days, ambient temperature and pressure; \triangle Al, \triangle Al, Fe, \triangle Ni, \times Ca, \times Cr and Mn).

The theoretical determination of acid required for the bottle roll tests was calculated using the elemental composition of the minerals plus an excess 10% of acid (Table 2). The highest theoretical acid consumption requirement of all six samples (Site 3 limonite) was chosen as the initial acid concentration for all six bottle roll tests.

Table 2: Theoretical determination of acid consumption in bottle roll tests (kg 98% sulfuric acid per tonne ore).

The theoretical determination of acid required for the bottle rolls tests was found to be sufficient for continued leaching of the samples over the 80 day period (see final acid concentrations in [Table 3](#page-9-0) compared with those in Table 1).

As opposed to the atmospheric leaching tests where insufficient acid was added, the XRD spectra from the bottle roll leaches show that the lizardite, nontronite and forsterite in the saprolite samples from all three sites leached to completion (Figure 5, main peaks highlighted, note that the lizardite peak in (i), indicated by the square, overlaps with (ii), but no lizardite is present in (ii)). It was assumed that the goethite would leach completely provided sufficient acid was added; however, goethite leaching was incomplete, for example, in the Site 1 and 3 limonite samples and the Site 2 limonite and saprolite samples. This is further confirmed by the lower percentage recovery of iron (~ 60–80 %) in the Site 1 limonite and saprolite and Site 3 limonite samples, suggesting that unleached goethite and other iron minerals are present in the samples. According to McDonald and Whittington (2008), one of the main factors for goethite leaching in sulfuric acid is a strong dependence on crystal structure. Goethite with nickel or cobalt substitutions will leach more readily than unsubstituted goethite or that substituted with aluminium or chromium. It is therefore possible to leach all the nickel present within the goethite mineral (*i.e.* obtain high nickel recoveries) without leaching the goethite as a whole. As an example, a comparison of the XRD traces of the original Site 2 limonite sample with that for the residue after the bottle roll leach shows that the goethite is still present after leaching, with the peaks appearing sharper in the residue, possibly because of increased crystallinity (Figure 6, main peaks highlighted).

Figure 5: Comparison of XRD traces for i) original sample and ii) residue after bottle roll leaching of Site 2 saprolite ore.

Figure 6: Comparison of XRD traces for i) original sample and ii) residue after bottle roll leaching of Site 2 limonite ore.

The XRD traces also show a broad peak appearing in the leached saprolite samples between $2\theta = 20-30^{\circ}$ (see for example, the Site 2 saprolite in Figure 7). This can be attributed to the leaching of minerals such as lizardite, nontronite and forsterite to form "amorphous" silica. Although the nontronite peak at $2\theta = 6^{\circ}$ is present in the XRD trace for the bottle roll leach residue, it is not apparent in the atmospheric leach residue (except for the Site 1 saprolite sample with less than 1 % nontronite in the residue). This confirms that sufficient acid was provided to leach the nontronite (and more of the other minerals) in the bottle roll leach tests compared with the atmospheric leach tests.

Figure 7: Magnified XRD traces for Site 2 saprolite i) original sample, ii) atmospheric leach residue and iii) bottle roll leach residue, showing amorphous peak at 2θ = 20–30° and disappearance of nontronite peak (2θ = 6°).

Comparison with Predicted Diagnostic Leaching Results

The nickel diagnostic leach as developed by Chapman et al. (2012) was conducted prior to this work on the same Site 1 to 3 samples (data not shown, refer to paper by Chapman et al.) to determine the nickel deportment in the ores. Table 4 provides the results from these tests conducted on the same samples as were used in this study where the nickel is classified to exist in either a water soluble, exchangeable, amorphous, adsorbed or structurally bound mineral or as a "difficult to leach" (residual) fraction. The total leachable nickel in the sample can be determined from this information (as a sum of the water soluble, exchangeable, amorphous, adsorbed and structurally bound minerals). Because the nickel is present in small amounts in some of these ore samples, the error associated with the analysis may be magnified, resulting in the total leachable amount being greater than 100 %. Most of the nickel in the limonitic ores is predicted to be structurally bound whereas the nickel in the saprolitic ores is said to be contained mostly in the adsorbed fraction followed by the structurally bound fraction. It is predicted that all nickel from the Site 2 saprolite and limonite samples and that from the Site 3 saprolite sample is leachable, with the remaining samples containing between 3 and 18 % unleachable nickel.

Table 4: Prediction of nickel present in ore samples, and possible nickel recoveries, determined by proposed diagnostic leach protocol.

	Predicted fractions of nickel present in ore samples (%)					Predicted nickel recovery (%)	
Sample	Water soluble	Exchangeable	Amorphous	Adsorbed	Structurally bound	Leachable	Residual
Site 1 Saprolite			15	40	32	97	
Site 1 Limonite				14	53	83	18
Site 2 Saprolite		16	13	51	24	108	
Site 2 Limonite				13	90	111	
Site 3 Saprolite		18	16	49	24	111	
Site 3 Limonite					78	97	

The predicted and actual total nickel recoveries from the bottle roll leach tests are given in Table 5 (with maximum predicted amount leached assumed to be 100 %). The predicted and actual nickel recoveries from the Site 1 and 3 limonites show the greatest absolute difference (9 and 14 %, respectively) with the remaining samples showing a difference in prediction of less than 3 %. It is not clear why the nickel recoveries from the Site 1 limonite ore are 9 % more than predicted and further investigation of the ore residues, for example, by scanning electron microscopy (SEM), is recommended. The fact that the nickel recovery from the Site 3 limonite was 14 % less than predicted may result from nickel entrapped in the goethite which was not leached in the time frame of the experiment. Further investigation of these solid residues would also be required to confirm this conclusion.

Table 5: Comparison between diagnostic leach prediction and actual nickel recoveries from bottle roll leaches.

CONCLUSIONS

Leaching results from the atmospheric leach tests confirmed that, as expected, limonitic ores tended to leach more slowly than the saprolitic ores. Furthermore, acid consumption is better predicted from mineralogical information than from standard literature recommendations.

Sufficient acid was added to the bottle roll leach tests to ensure the ores leached to completion where this was possible thermodynamically within the leach time employed. The nickel recovery from all three saprolite ores was complete within 11 days. Nickel leaching from the limonitic ore samples was complete after 50 days for the Site 2 limonite sample and appeared to be reaching completion for the remaining limonite samples after 80 days. XRD results confirmed that lizardite, nontronite and forsterite leached to completion in the bottle roll leach tests as confirmed by the appearance of an "amorphous peak" between 2θ = 20–30° for the saprolite ores. Goethite leached only partially in some cases, as confirmed by the incomplete percentage recovery of iron. Despite the incomplete goethite leaching, the nickel recovery in these samples was higher than expected. This could be because nickel-substituted goethite can leach more readily than unsubstituted goethite in sulfuric acid.

A comparison of the results for the predicted nickel recoveries from the diagnostic leach tests shows that the diagnostic leach predicts the bottle roll leach outcomes to within 3 % for the saprolite samples. The limonite samples prove harder to predict with an up to 14 % deviation in predictions. This could be because the goethite does not leach as well with sulfuric acid and/or under the conditions employed when compared to the diagnostic leaching step using 32% HCl. From the diagnostic leach, it was predicted that the nickel in the saprolite samples was dispersed across more of the fraction types, while that in the limonite is mainly structurally bound. SEM analysis of the residues from the bottle roll leaches may allow for the confirmation of the location of the nickel in the residues.

The results from the validation of the diagnostic leach by bottle roll leaching are preliminary in nature and there is scope for improvement and further work. However, we believe that initial results have been shown to be promising. Test work on additional laterite ores including mineralogical residue and possibly kinetic solid sample analysis is recommended to further confirm the diagnostic leach predictions.

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