

DEVELOPMENT AND PILOTING OF SKYE RESOURCES' SULPHATION ATMOSPHERIC LEACH (SAL) PROCESS AT SGS MINERALS

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CONTENTS

1. ABSTRACT	1
2. INTRODUCTION.....	1
3. PROJECT MANAGEMENT AND PROJECT SCHEDULE.....	3
4. SAL PROCESS.....	5
5. PILOT PLANT FLOWSHEET.....	6
6. PILOTING RESULTS OF SAL PROCESS.....	11
7. CONCLUSIONS.....	14
8. ACKNOWLEDGEMENTS	14
9. REFERENCES.....	14

1. ABSTRACT

SGS Minerals was contracted in April 2004 by Skye Resources Inc. to provide testing services for the development of a Sulphation Atmospheric Leach (SAL) process capable of treating limonite and saprolite ore from Skye's Fenix project in Guatemala. By means of a phased approach, a novel atmospheric leach process was developed, first at bench scale and subsequently at pilot scale. The overall testing program - starting with a scoping study and leading to the successful operation of a fully integrated pilot campaign - took less than 20 months. A total of 11 semi-integrated pilot plant campaigns and several continuous sulphation leaching campaigns were performed. This paper discusses the design of the overall test program and pilot plant and highlights the benefits of a carefully phased approach to the development of a novel hydrometallurgical process. The results of the final fully integrated pilot campaign that operated for 10 days in December 2005 are discussed.

2. INTRODUCTION

SGS Minerals Services is a global leader in the development and demonstration of bankable flowsheets and pilot plant programs. SGS Minerals has practical metallurgical experience with all metals, particularly gold, nickel and base metals and a wide range of industrial minerals. This ensures that clients can unlock the mineral wealth contained in orebodies around the world. With metallurgical, analytical, mineralogical and environmental technical expertise, clients are supported everywhere. The work carried out in this program was performed in the Lakefield, Canada facilities.

Skye Resources is a junior mining company focused on becoming a mid-tier nickel producer. Skye acquired the

rights to an established lateritic nickel project (the Fenix project) in Guatemala. Skye has completed a feasibility study for a 50 million pound per year ferro-nickel project at Fenix using conventional smelting technology and a preliminary assessment study of a 50 million pound expansion of this project. Skye contracted SGS Minerals in April 2004 to perform a process development program under the guidance of its Vice President Technology and Development, Mr. David Neudorf. From five initially suggested leach options, two promising processes

were developed at bench scale and one of these, the SAL Process, was further developed at pilot scale.

The SAL Process described in this paper is the subject of US Patent Application No. US 2006/0002835 A1 and PCT Patent Application No. WO 2006/000098 A1, both published on Jan. 5, 2006 (Neudorf 2006). The claimed priority date is June 28, 2004, the filing date of a related provisional US application. The patent application is based on testing carried out at SGS Minerals.

A simplified block process diagram is included in Figure 1. The leach slurry preparation (LSP) plant consisted of limonite sulphation and saprolite / sulphated limonite grinding. The "upstream" circuit consisted of atmospheric leaching (AL), primary iron removal (PIR) and counter current decantation (CCD). The "downstream" circuit consisted of secondary iron removal (SIR), mixed hydroxide precipitation (MHP), scavenger precipitation (SP) and manganese removal (MR).

The approach taken during this project has not been the standard approach of the average junior mining company that SGS has dealt with in the last several years. Skye and SGS ensured that sufficient time and effort was devoted at the bench scale to provide a solid base for process design and piloting. Our recent experience has been that clients / owners of properties requiring complex hydrometallurgical flowsheets have expressed the desire to move towards continuous piloting as soon as possible. As a result, only a minimum of bench tests was requested and the operation of one or several pre-pilot campaigns has often been neglected all together. While the reason for this is understandable, it is the view of SGS that this can be more expensive in the long run. Some typical arguments that lead clients to move to continuous piloting as quickly as possible are included below.

- An elaborate bench test program may require a minimum of several weeks to a few months to complete. Although the operation of such a bench program may represent only a fraction of the overall program costs, juniors often want or need to take advantage of market conditions and will elect to move ahead quickly. The 'loss' of several months may not be acceptable to them. To counter this, the cost of Skye's initial bench program was less than 10% of the overall program costs.
- Along similar lines, the operation of one or more pre-pilot campaigns may also take several weeks to complete even though this also only represents a fraction of the total costs. In this project, the operation of several small continuous campaigns of selected

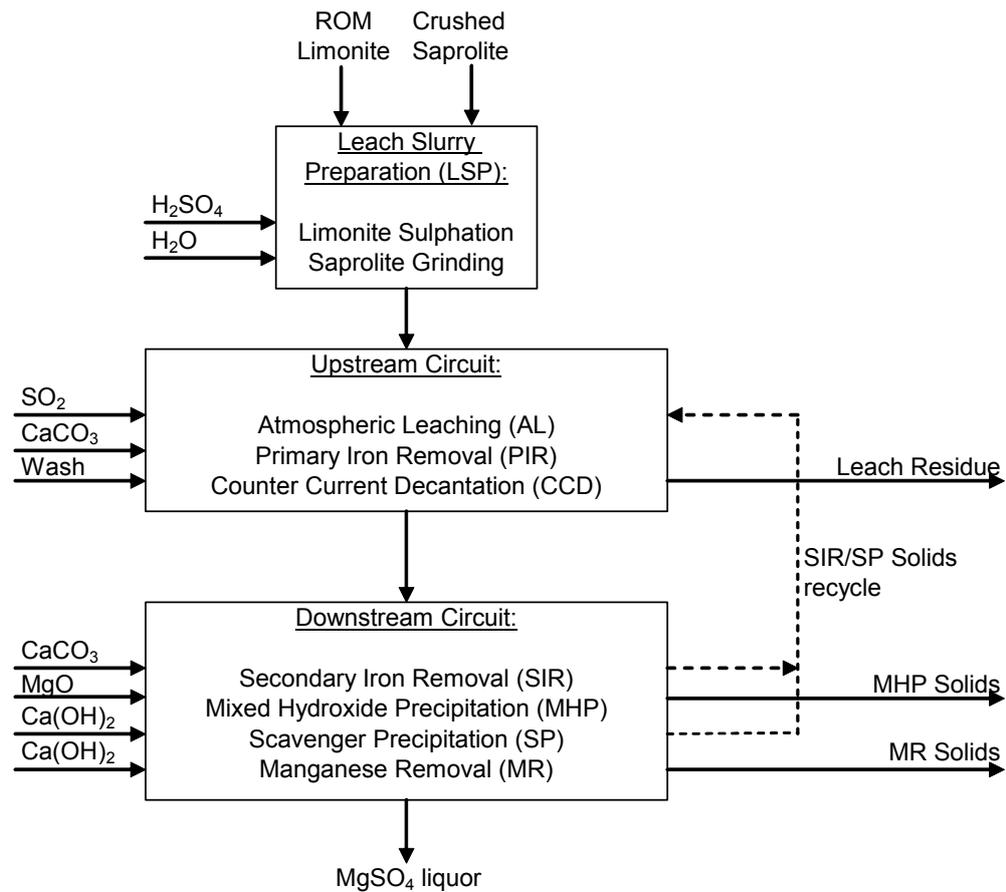


Figure 1 Simplified Block Process Diagram

- unit operations, most notably the continuous leach slurry preparation (LSP) operation, proved to provide invaluable information and it would have been considerably more expensive to find out the same information during a single fully integrated pilot plant stage.
- It is sometimes (mis)understood by our clients that the development of a 'new metallurgical process' takes place during piloting and required operating changes can be made 'on the fly'. Whereas it is true that small changes can be made to optimize circuit performance, it is much cheaper to test flowsheet changes at a bench scale level. Significant flowsheet changes should never take place during the "final campaign" when final design data is usually collected, vendors may be providing final testing, and/or potential investors may be conducting due diligence.

- Observe department of minor impurities;
- Produce product streams for equipment vendors to perform tests.

As stated above, this project at the behest of Skye Resource and with the whole hearted agreement of SGS, benefited from a different strategy, one in which a significant amount of time and effort was invested at the early stages to generate as much information as possible from bench scale leach and solid-liquid separation programs, as well as small scale pilot campaigns focusing on one or two unit operations only. This paper highlights the benefits of a phase wise approach by describing the development of the SAL process. By focusing on leaching, iron removal and solid liquid separation, key results from the final fully integrated pilot plant are presented.

- In general, pilot plants are operated with the following objectives:
- Confirm bench data and design criteria;
 - Obtain operating data under 'steady state' conditions;

2.1. FENIX PROJECT

Skye acquired the Fenix deposits in 2004 from Inco. The deposits are located in the Lake Izabal region of eastern Guatemala and were identified by Inco in the 1950's and 60's. The Guatemalan company, Exmibal (70% Inco, 30% Government of Guatemala), processed the saprolite portion of the ore body from 1977 to 1980 to produce a nickel sulphide matte in a single rotary kiln-electric furnace (RKEF) production (Sopko 1979). In 1980, the Exmibal plant was shut down and placed on care and maintenance due to a combination of high oil prices and low nickel prices. Upon acquiring the project (renamed Fenix) in 2004, Skye initiated a preliminary assessment of alternatives for restarting production, and, based upon this assessment, initiated a feasibility study for a ferronickel project in 2005 which was completed in September 2006. Skye has also completed a preliminary assessment of a subsequent expansion using hydrometallurgy.

3. PROJECT MANAGEMENT AND PROJECT SCHEDULE

Each SGS project is able to draw from a wealth of metallurgical pilot experience. Based on this experience, a wide range of supportive services have been developed such as elaborate metallurgical laboratories, pilot plant facilities, mineralogical and environmental experts. In addition, a 24/7 analytical laboratory provided quick turnaround where required in the bench and pilot plant studies.

This project was managed by a team of metallurgists supervised by a project/technical manager. In line with the flowsheet, separate sulphation, upstream and downstream metallurgists were selected, each responsible for operation and data collection of their respective unit operations during the piloting phases. In addition, shift supervisors were assigned the responsibility of achieving and maintaining operational targets. An organogram of the project team is presented in Figure 2. The organogram also shows the division of the program into 5 phases. The first three phases of the program were carried out with

a relatively limited team consisting of client, project manager and bench program technologists. The inclusion of the client in this organogram reflects SGS' experience that a close cooperation between client and SGS technical personnel is highly beneficial to the technical outcome of the project. In this project, Skye was an active and hands-on participant throughout the program.

3.1. PHASE WISE APPROACH

An aggressive time schedule was required by Skye to complete the process development, as shown in the Gantt chart included in Figure 3. The overall metallurgical project was divided into 5 stages, each phase having its own objectives. A description of each phase is included in Table 1. The scope of work increased during each phase, which ensured that during the early stages of the project, the costs were kept low by maintaining a small project team. Parallel to the metallurgical program, mineralogical and environmental studies were also undertaken by SGS, but these are beyond the scope of this paper.

3.1.1. PHASE 1 - BENCH LEACH SCOPING TESTS

An initial series of leach scoping tests was performed under the Phase 1 program. Several pressure and atmospheric leach flowsheet options were evaluated based on achieved recoveries and projected reagent consumption. The main focus and objective of this program was

to determine conditions resulting in optimum leach recovery. Issues such as solid liquid separation and downstream processing were ignored at this stage.

3.1.2. PHASE 2 DETAILED BENCH SCOPING PROGRAM

The two most promising leach options identified in Phase 1 were further evaluated in a more elaborate and detailed test program identified as the Phase 2 program. During this stage, the scope of testing was broadened to include solid liquid separation and downstream processing. This program delivered the bulk of design criteria for the pilot plant.

The inclusion of preliminary solid-liquid separation testing in the early stages of a process development program is critical to the successful completion of that project as discussed by Mezei et al. (2006) and Molnar (2006). If a material can not be settled in the lab, it is likely to behave in a similar manner at pilot scale. However, at pilot scale there may be as many as three or four shifts of 6-10 operators scheduled to work, an analytical lab prepared for the increased sample load and vendors scheduled to come in. And indeed, the (non) operation of a CCD circuit can prematurely stop a pilot plant.

Table 1 Project Stage Definition

PROJECT STAGE	TYPE OF PROGRAM	OBJECTIVE	LEACH PROCESS(ES)	SAMPLE SIZE
Phase 1	Bench scale program	Evaluate 6 different leach options	Various	100-200 kg
Phase 2	Bench scale program	Optimise 2 leach options. Scoping study of nickel recovery processes	SAL, other	As Phase 1
Phase 3	Continuous sulphation piloting	Develop SOP for continuous sulphation	SAL	2 - 5 tonnes
Phase 4	Separate up and down stream piloting	Establish operability of proposed process in split stages	SAL	15 - 25 tonnes
Phase 5	Integrated piloting	Prove operability of fully integrated flow sheet	SAL	15 - 25 tonnes

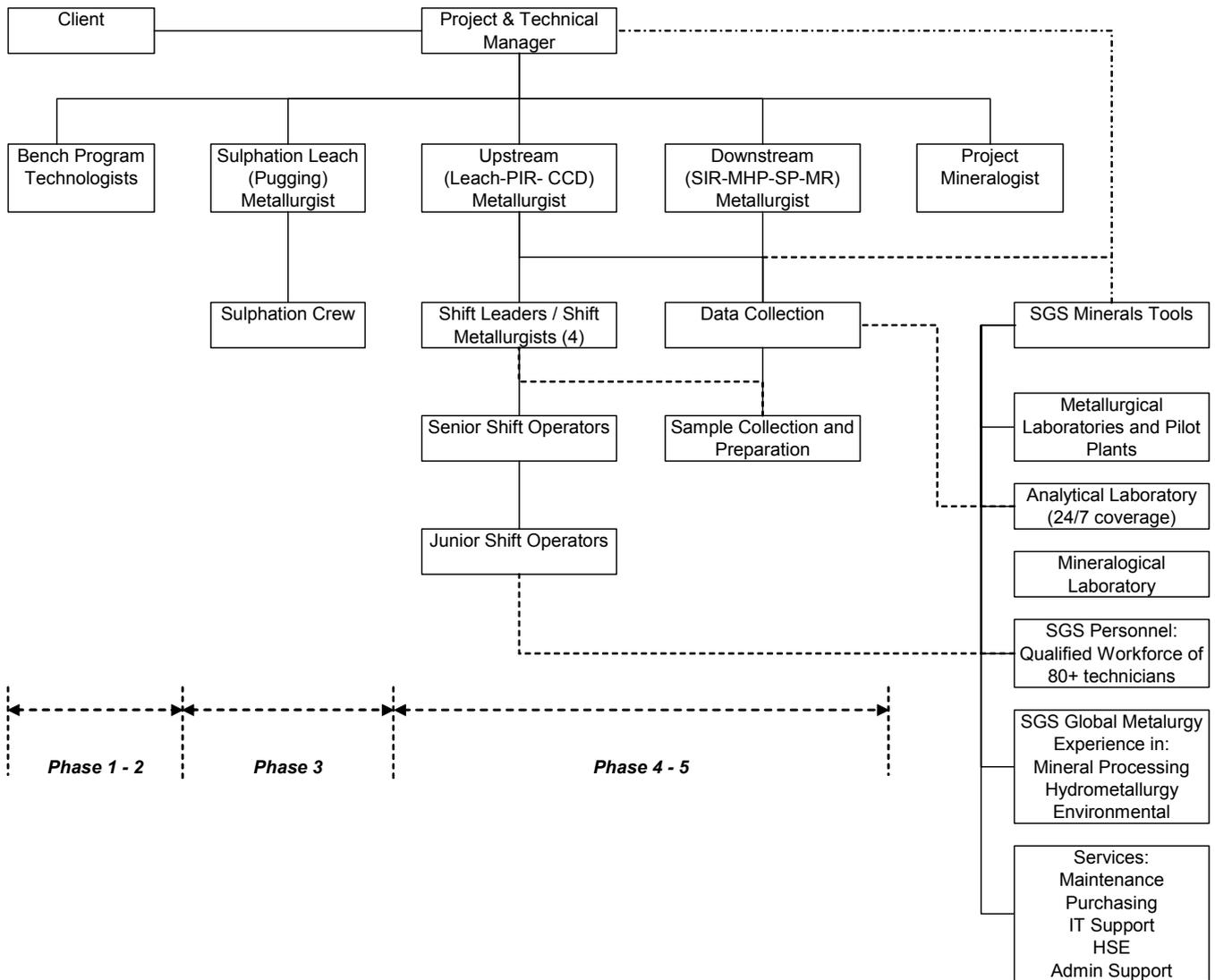


Figure 2 Project Organogram

The scope of such a solid liquid separation program begins with the preparation of a sample, representing the conceptual process as closely as possible. The preparation of such a sample is probably the most critical step in the process as any data derived from this testing reflect only the behaviour of that particular sample. Hence, the sample should be as fresh as possible to reduce pulp aging and temperature change issues. A 4L pulp sample at 25% solids was prepared in two stages. The first stage produced pulp for a flocculation scoping program during which a range of nonionic, cationic and anionic flocculants were evaluated at 100 mL cylinder scale by observing floc formation, initial settling rate and clarity. Based on the feed pulp % solids, a reagent dosage was determined. The second stage sample was produced half or full day later and was used for feed

for two series of four 2L raked cylinder tests. This way flocculation and settling tests were based on freshly prepared pulp samples. The first series of 4 tests determines optimum pulp density for settling and the second series of tests determined the optimum reagent dosage at optimum feed dilution. It is important to perform pulp dilution using liquor with an estimated composition of CCD2 o/f liquor. Dilution with water often results in false positive results due to artificially decreasing the ionic strength of the liquor.

Though the results of such S/L separation testing are preliminary in nature, predominantly because the settling work was performed using batch as opposed to continuously produced samples, it gives an initial estimate of required feed dilution and floc dosage. This information is then

inserted into a mass balance based Excel model developed by SGS which in turn calculates CCD1 o/f tenors based on a targeted wash efficiency or CCD recovery. In addition, all thickener flowrates are calculated allowing for suitable sizing of equipment. The CCD1 o/f tenors are used to design the downstream testing.

3.1.3. PHASE 3 - PILOTING OF STAND ALONE UNIT OPERATIONS

A distinctive feature of the phase wise approach taken in this project was the work carried out under the phase 3 program. This phase looked only into the development of continuous piloting of the limonite sulphation process. Given the novel nature of the process, this required some method development work. Various methods to carry out the limonite sulphation stage were evaluated at pilot scale. During this phase, the

Task Description	# Tests / Campaigns	% Cost	2004												2005												2006		
			04	05	06	07	08	09	10	11	12	01	02	03	04	05	06	07	08	09	10	11	12	01	02	03			
Phase 1 Leach Scoping Study (5 options)		3	[Gantt bars for Phase 1]																										
1.1 Leach Testing	30		[Gantt bars for 1.1]																										
1.2 Reporting			[Gantt bars for 1.2]																										
Phase 2 Detailed Leach Study (2 options)		5	[Gantt bars for Phase 2]																										
2.1 Leach Testing	~35		[Gantt bars for 2.1]																										
2.2 S/L Separation Testing	2		[Gantt bars for 2.2]																										
2.3 Downstream Testing	20		[Gantt bars for 2.3]																										
2.4 Reporting			[Gantt bars for 2.4]																										
Phase 3 Development of Continuous SAL Processing at Pilot Scale		5	[Gantt bars for Phase 3]																										
3.1 Limonite Sulphation (only) Testing	4		[Gantt bars for 3.1]																										
3.2 Combined Limonite Sulphation and Sapolite Grinding	5		[Gantt bars for 3.2]																										
3.3 Combined Limonite Sulphation and Sapolite Grinding - Combined Bench and Pilot Program			[Gantt bars for 3.3]																										
Phase 4 Separate Up and Down Stream Piloting		52	[Gantt bars for Phase 4]																										
4.1 Design, Purchasing, Construction Pilot Plants			[Gantt bars for 4.1]																										
4.2 Continuous SAL Campaigns	5		[Gantt bars for 4.2]																										
4.3 Upstream Piloting	6		[Gantt bars for 4.3]																										
4.4 Downstream Piloting	3		[Gantt bars for 4.4]																										
4.5 Supportive Bench	10		[Gantt bars for 4.5]																										
4.6 Supportive S/L Separation			[Gantt bars for 4.6]																										
4.7 Reporting			[Gantt bars for 4.7]																										
Phase 5 Integrated Piloting		34	[Gantt bars for Phase 5]																										
5.1 Piloting	1		[Gantt bars for 5.1]																										
5.2 Supportive Bench	10		[Gantt bars for 5.2]																										
5.3 Supportive S/L Separation			[Gantt bars for 5.3]																										
5.4 Vendor Testing - CCD6 u/f			[Gantt bars for 5.4]																										
5.5 Vendor Testing - MHP th u/f			[Gantt bars for 5.5]																										
5.6 Reporting			[Gantt bars for 5.6]																										

Figure 3 Project Gantt Chart

leach slurry preparation plant (LSP) plant was operated as a stand alone unit to develop standard operating protocols. Instead of evaluating its performance by going 'all the way' to atmospheric leaching, primary iron removal and CCD, it was evaluated by performing standard product leach bench tests that simulated the atmospheric leach process. Composite pulp samples from the continuous LSP tests were used for this. This mode of operation also allowed the evaluation of effects of cooling and aging of leach pulp feed at relatively low cost.

3.1.4. PHASE 4 – SEMI INTEGRATED PILOTING

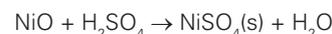
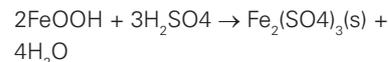
During the phase 4 program, a total of 5 upstream (LSP, AL, PIR, CCD) and 3 downstream (SIR, MHP, SP, MR) pilot campaigns of 5 days each were carried out. CCD1 o/f liquors produced in the upstream campaigns were used in the downstream campaigns, and recycle products (SIR and SP precipitates) produced in the downstream campaigns were used in later upstream campaigns. By not operating a fully integrated circuit, operating personnel were able to focus on fewer operational problems and the risk of partial circuit shutdown due to, for example, CCD malfunctioning was minimized.

3.1.5. PHASE 5 – INTEGRATED PILOTING

A fully integrated 10 day pilot campaign was carried out in the final phase of the test program. Based on the fact that circuit operators and samplers were fully trained and that the circuit was fully filled, a relatively short commissioning period was required. Vendors and potential investors were invited for third party testing and tours through the pilot plant.

4. SAL PROCESS

The SAL process is based on the sulphation of run of mine limonite ore using concentrated (>96%) sulphuric acid and is based on testwork carried out at SGS Minerals. It is essentially a two-stage process, the first stage involving the contacting of concentrated acid with moist limonite ore and the second stage consisting of the blending of the acid/ore mixture with crushed or ground sapolite ore. Water is added during the blending stage to produce a suitable pulp density for grinding (if required) and leaching. In the case of crushed ore, the mixing device may be a ball mill. The following (simplified) reactions take place during the limonite sulphation:

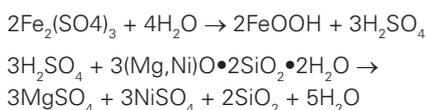


Water present in the ore, or generated by the above reactions, is largely consumed by hydration of the metal sulphate salts formed, or driven off as steam. The great distinction between this process and the atmospheric leach process described by Liu et al. (2004) is the fact that there is no water addition during the limonite sulphation stage other than water as ore moisture or contained with concentrated acid (2 or 4% water). The sulphation reactions generate a large amount of heat, increasing the temperature of the reacting mixture far above the normal boiling point of water (as high as 140°C) and accelerating the reactions, allowing the process to operate under atmospheric conditions and with a very short retention time (as little as 5 to 10 min).

It is of interest to note the work carried out by Xu et al. (2005), who investigated similar methodologies, identified as the sulphuric acid water leaching (SAWL) process of what appears to be a low grade mixed limonite and saprolite ore. Though high nickel and cobalt recoveries (>90%) were reported, a significant drawback appeared to be high iron coextraction in the range of 60 to 70%.

The acid/ore mixture, which can be of paste consistency or of a dry, powdery consistency, is mixed with water and crushed or ground saprolite in a ball mill. This mixture is then ground to a particle size sufficiently small to enable off bottom suspension in standard CSTR's. The pulp thus produced is fed to the AL circuit. The combined limonite sulphation step and mixing or grinding of the sulphated limonite with the crushed or ground saprolite ore is referred to as the Leach Slurry Preparation (LSP) stage.

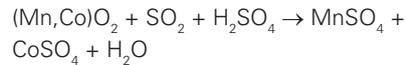
The leach feed pulp is then re-heated (or kept at) at temperatures ranging from 95°C to 105°C for a time ranging from 12 to 48 hours. At these temperatures, dissolved ferric iron is hydrolysed to form ferric hydroxide or goethite and sulphuric acid. The sulphuric acid is consumed by dissolving nickel bearing minerals in the saprolite ore. The following (simplified) reactions take place:



The three most important variables to describe the SAL process are the acid to total ore ratio (A/O), the acid to limonite ore ratio (A/L) and the saprolite to limonite ore ratio (S/L). The A/O ratio is based on solubilizing all non-ferrous elements, rejecting iron, and is greatly dependent on the magnesium content of the saprolite ore. By adjusting the saprolite to limonite ore ratio, the amount of acid produced by iron hydrolysis can be balanced with the quantity of saprolite added.

Cobalt recovery from manganese minerals in the limonite is enhanced by mild reduction using SO_2 gas during

atmospheric leaching according to the following simplified reaction:



During this project the performance of the SAL process was described by three measured parameters:

- Nickel and magnesium recovery. These were calculated by mass balance or by "Sitie". Mg recovery was used to evaluate the level of saprolite leaching. High Mg recovery in combination with low overall Ni recovery implied low nickel recovery from limonite;
- Iron to nickel ratio in the final leach liquor. During the limonite sulphation stage, goethite is almost completely digested to form ferric sulphate species. Subsequent water leaching results in very concentrated iron solutions ranging from 60 to 100 g/L Fe and Fe/Ni ratios ranging from 12 to 20. Iron hydrolysis during atmospheric leaching typically reduces the iron concentration and thus Fe/Ni ratio to values of 2 to 5. High Fe/Ni ratios indicate inefficient use of acid and lead to increased consumption of limestone in primary iron removal;
- Final free acidity. Well balanced A/O and S/L ratios lead to low residual free acidity and often less than 0.5 g/L H_2SO_4 .

Optimum saprolite to limonite ore ratios were found to depend on the "sulphatable" iron content (acid regeneration capacity) of the limonite ore and neutralizing capacity of the saprolite ore and need to be determined for each particular ore composition.

5. PILOT PLANT FLOWSHEET

Figure 4 shows the detailed flowsheet as tested in the final fully integrated pilot campaign. It is divided into three sub areas (divided by dotted lines in the flowsheet):

Leach Slurry Preparation Area. This area included the limonite sulphation and the mixing / grinding of the sulphated limonite with crushed saprolite ore. Feed for the atmospheric leach circuit was stored in drums and transferred to the AL circuit;

- Upstream Leaching and S/L Separation Area. This included atmospheric leaching, primary iron removal and counter current decantation;
- Downstream Liquor Purification and Ni Recovery Area. This included secondary iron removal (SIR), mixed hydroxide precipitation (MHP), scavenger precipitation (SP) and manganese removal (MR).

A natural boundary exists between each plant and it is important to take full advantage of such boundaries during phase wise piloting by piloting separate sections only. The LSP circuit operated at a throughput of 2300-2400 kg/day (limonite plus saprolite), which was close to 14 times higher than the equivalent throughput of the upstream plant.

A second natural boundary is located between CCD and SIR. During the phase 4 program CCD1 o/f liquor was collected in 1000L storage containers and used as feed for the downstream plant. Products for recycle, such as the SIR and SP precipitates, were stored and recycled in subsequent upstream plants.

Separate operation of the upstream and downstream plants allowed key personnel to concentrate on fewer problems at a time. Hence, once the fully integrated 10 day pilot campaign started, each circuit was debugged and operating and sampling personnel were fully trained. This allowed for a relatively short commissioning time and smooth and constant operation for extended periods of time. This in turn led to the production of representative and stable operating data, confirmation of bench test work, and most importantly the production of significant quantities of product (or tailings) streams that were representative of steady state operation. Hence, the quality of the vendor testing, such as for CCD thickener and MHP precipitation filter design, was greatly improved and future potential problems minimized or eliminated.

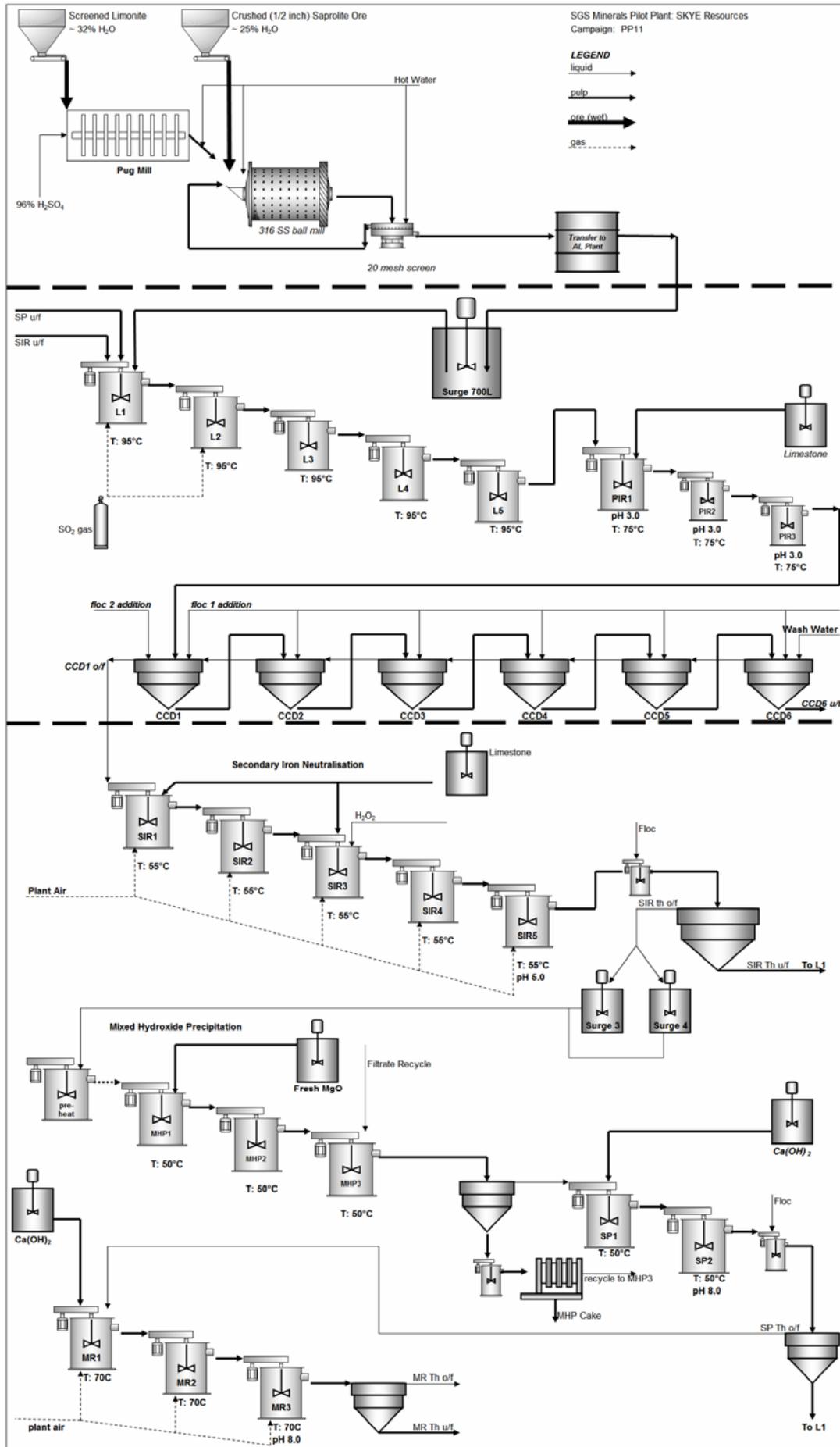


Figure 4 Integrated Pilot Plant Flowsheet

5.1. ORE PREPARATION

The bulk ore samples were prepared some weeks before the start of the pilot campaigns. Limonite ore was screened at 1 inch to remove rocks which could potentially damage the pug mill. In addition, the screening process de-agglomerated limonite ore chunks. Sapolite ore was crushed to 1/2 inch using jaw and cone crushers. Two composite sapolite samples, a "high iron" and a "low iron" sample, were prepared by blending several received sapolite samples. It was Skye's intention to approximate the effect of including or rejecting the transitional zone material with the mined sapolite. It was also intended to achieve blended nickel grades close to the estimated deposit average. The composition of these ores blends as used for the two final campaigns is included in Table 2.

One of the most important variables in the SAL process is the sapolite to limonite ore ratio (S/L). Whereas ore hoppers, equipped with vibratory feeding systems were installed, it was found in earlier Phase 3 work that control at this small scale was insufficient to maintain

a constant and on-target S/L ratio. The constant adjusting of the vibratory feeders, coupled with the occasional plugged hopper opening resulted in off-target S/L ratios. An alternative method was developed based on adding pre-weighed charges of limonite ore and sapolite ore. To keep the operations as "idiot proof" as possible, charges were prepared that matched throughput equal to 5 minutes, an easy to work with number. The operators adjusted their vibratory feeders to ensure a charge was emptied just within the 5 minute time span.

A significant drawback of this approach was that the chemistry of each campaign was actually determined at the sample prep stage, instead of during the campaign. Small changes or tweaking of the S/L ratio was not possible during the campaign without altering the weight per bag or alternatively the period of time for each bag.

5.2. LEACH SLURRY PREPARATION

A twin shafted, continuous trough-type Pug Mill built by Patterson Industries was used for the limonite sulphation stage.

A photograph of the internals is shown in Figure 5. Concentrated sulphuric acid was pumped into the feed end of the pug mill using an accurate piston pump. Limonite was fed in the same side of the pug mill in pre-weighed charges through a small hopper and vibratory feeder. The retention time of material in the pug mill was difficult to determine due to the consistency of the sulphated limonite, which varied significantly and ranged from a brown pasty "toffee" product to a dry grey powder type product. Using various assumptions relating to percent filled volume and specific gravities of limonite and acid, the retention time was estimated to range from 10 to around 30 minutes. A photograph looking into the pug mill at the point of contact of the acid with the limonite ore stream is included in Figure 6. It shows the formation of a brown toffee-like sulphated limonite, which quickly transforms into a dry, grey sulphated limonite product. Based on this photograph and other visual observations, it appears that the reaction is complete at this point and it is probable that the retention time can be further reduced.

Table 2 Ore Head Grades

	Ni %	Co %	Cu %	Zn %	Fe %	Mg %	Al %	Cr %	Mn %	Ca %	Si %	Cl g/t	H ₂ O %
LIMONITE	1.28	0.19	0.01	0.03	48.3	1.0	2.73	2.26	1.13	0.02	2.64	61	32.5
HIGH FE SAP	2.14	0.03	0	0.01	13.0	14.4	1.16	0.6	0.22	0.15	18.5	140	31.9
LOW FE SAP	2.21	0.02	0	0.01	10.3	16.7	0.77	0.45	0.16	0.19	18.6	120	22.6

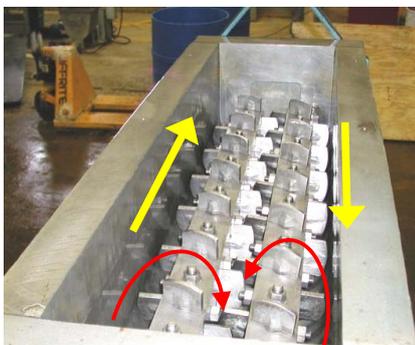


Figure 5 Photograph of Pug Mill Internals



Figure 6 Photograph of Formation of Sulphated Limonite

Since no water is added during the limonite sulphation, limonite ore is in contact with concentrated acid. The heat generated due to the sulphation reactions leads to measured temperature around 140°C in the pug mill. In the SAL process as tested at SGS, water was added to the discharge trough of the pug mill, which facilitated the feeding of sulphated limonite into a custom designed stainless steel ball mill (2 feet diameter, 3 feet length). Further additions of water based on pulp density requirements in the ball mill and leach circuit were made to the ball mill and subsequent classifying screen. The ball mill was gravity fed from the pug mill. A (top view) photograph showing the feed trough of the ball mill with crushed sapolite feed and discharged sulphated limonite is shown in Figure 7.

As indicated in Figure 4, ball mill discharge pulp was screened at 20 mesh. Screen u/s was collected as feed to the AL circuit and screen o/s was returned to the ball mill in batches. Significant procedural development was carried out during the Phase 3 and 4 stages of the project. One problem was related to the fact that the LSP circuit only operated for approximately 7 hours every two days of pilot plant operation, due to its high throughput. In early pilot campaigns (Phase 4), product pulp was collected as soon as screen u/s was produced to limit the usage of excess sample. Leach liquor from those earlier campaigns displayed higher Fe/Ni ratios (5-6) than equivalent bench experiments. No explanation was found during the early Phase 4 campaign, but after an intermediary Phase 3 type program carried out in October 2005 (see Figure 3), it was found that pulp produced within the first hours of LSP operation was different from pulp produced under steady state conditions. This was predominantly due to the build-up of rocky sapolite in the ball mill as it reached steady state. Consequently, the process produced pulp featuring significantly lower than targeted S/L ratios.

To speed up the approach to steady state, indirect steam was used to heat up the pug mill during start up and hot water was used as makeup water. Pulp



Figure 7 Photograph of Ball Mill Feed Trough

produced during the first two hours was set aside and not used in further processing. The remainder was stored in large agitated feed tanks. Other than mechanical problems associated with transfer pumps, no down time was experienced in the operation of the LSP process. Though there is the potential for hardened toffee-like paste to form in the pug mill during forced outages or during operating periods with insufficient acid addition, this can usually be resolved by the immediate flushing of the pug mill.

5.3. UPSTREAM AREA (AL, PIR, CCD)

The atmospheric leach tank train consisted of five custom designed polypropylene 100L reactors that were heated by steam coiling at temperatures around 95°C. Using temperature controllers and solenoid valves, steam (10-15 psi) was circulated through the coiling. Leach circuit down time was negligible. Retention time varied from 15 to 24 hours and was controlled by adjusting feed rate or volume level in the tanks. SO₂ gas was added to the first and second tanks to control emf (vs. Ag/AgCl) at or around 650 mV. Ventilation was supplied to all tanks and off-gases were treated in a caustic spray scrubber. The AL circuit was easy to operate as the only control parameters for the operators were feed rate and temperature.

It is difficult to construct fully sealed reactors and still have the flexibility to modify flowsheets on the fly during piloting as well as to allow for all scheduled and unscheduled sampling. Hence the tanks were not fully sealed and evaporation of process water was significant. Reflux of condensed water

was promoted by combining hot air from the reactors with 'cold' air from outside the reactor. However, to maintain a constant pulp density, water was added to the tanks to make up for evaporative losses.

In order to prevent having to pump 95°C leached pulp, the PIR circuit, consisting of 3 reactors, was located just below the AL circuit as one large cascade tank train. Retention times were approximately 4 hours in PIR1 and 1.5 hour each in PIR2 and PIR3. Ground limestone (20- to 25 % solids) was used to remove residual acid, ferric iron, and aluminium, as well as impurities such as copper and zinc. The long retention time in PIR1 ensured that the majority of iron was precipitated in PIR1, aiming to remove iron to below 1 g/L Fe³⁺.

To improve ease of pH control the limestone pulp was diluted somewhat to below maximum workable pulp density. This allowed improved fine tuning of the pumps and also compensated for some of the evaporative water losses.

A small surge tank (100L) was installed between PIR and CCD. The use of surge tanks in piloting has its pros and cons. Among its pros are that the larger the surge, the more constant the feed to the next circuit. Particularly in a challenging CCD circuit, it is important that fluctuations in feed are evened out as much as possible. Its cons include that the total circuit inventory increases and that it will take longer to complete several complete circuit turn overs. During the Skye piloting the surge consisted of a agitated 75L tank.

A CCD circuit is perhaps the most difficult and underestimated circuit in any complex hydrometallurgical pilot plant and as mentioned before can act as 'show stopper' if not properly designed and operated. A photograph of the 6-stage CCD circuit is included in Figure 8. The Skye CCD was designed conservatively with >1 m²/tpd solids to ensure that the CCD circuit would provide good quality solution to downstream processes, while still proving the operability of the circuit. Thickener and reagent vendors were invited to test fresh pilot plant streams and the intent was to establish CCD design criteria based on the vendor testing.

The successful operation and control of a pilot CCD circuit boils down to the following three critical steps, listed in order of importance:

- **Flocculation.** Without flocculation, there is no settling. So obviously, this is the single most important parameter to control. During the Skye project, this was accomplished by performing frequent 'in situ' settling tests at each thickener. Parameters like floc structure/size and settling rate can be evaluated even on a dirty 100 mL sample cup scale. Any changes in feed or floc dosage were always accompanied with more frequent tests to ensure that proper flocculation was maintained.
- **Management of solids mass.** More input than output leads to a build-up of material (usually observed by increasing mud lines) and more output than input will drain the solids out of a thickener. This is common sense, but it is more complex if there are 6 units to manage. The operators were responsible for maintaining target mud lines, while managing fluctuating feed streams.
- **Target wash efficiency vs. target product grade.** Data obtained in the pilot plant were inserted in the CCD model and a wash efficiency was predicted. Wash feed flows were adjusted on a daily basis to maintain acceptable wash efficiency, while meeting CCD1 o/f grade targets. During an integrated pilot plant the latter is of more importance as it directly affects downstream circuit performance.

Preliminary testing revealed that feed dilution and flocculation regime were key components to the successful operation of the CCD circuit. Several methods of flocculant addition / mixing were tried, such as inline mixing (coagulant first, then floc or reversed) of both reagents or mixing of reagents and pulp in a separate mix tank. Typically these were first tested on cylinder scale, then applied in the plant.

5.4. DOWNSTREAM AREA (SIR, MHP, SP, MR)

A detailed discussion of this area is beyond the scope of this paper, but a brief description is included below. The "MHP Process" was chosen for the downstream pilot plant because it is a relatively simple, commercially proven process, but other processes could be utilized. The intent was to demonstrate that SAL leach liquor could be processed successfully using conventional technology.

Secondary iron removal (SIR) consisted of a 5 reactor cascade with 1 hour retention per reactor. Its main objective was to, oxidize ferrous iron (produced in AL1/AL2) using air or oxygen, and to precipitate residual aluminium and ferric iron using ground limestone. It is usually quite difficult to oxidize ferrous using air or oxygen in pilot sized tanks. Hence a "cheater" line with hydrogen peroxide was used to achieve complete iron removal. Average conditions included a final pH of 5.0 and temperature of 55°C. The temperature in each downstream unit operation was controlled at values predicted from a commercial plant MetSim balance with natural, progressive cooling.

Thickener overflow was stored in one of two large surge tanks, which were used as feed tanks to the MHP circuit. The MHP circuit consisted of a three reactor cascade and nickel and cobalt were precipitated with freshly prepared caustic calcined magnesia. The primary control mechanism was stoichiometry based: for each full feed tank, a new MgO dosage was calculated based on its nickel and cobalt content. The pH was allowed to fluctuate within a certain range, but a secondary control strategy was followed if it exceeded that range. Ni recovery



Figure 8 Photograph CCD circuit

was targeted at 85% to minimize manganese co-precipitation.

Residual nickel was precipitated in a scavenger circuit comprising a 2 reactor pH controlled circuit. Hydrated lime was used as precipitant. Target conditions included pH 8.0 and temperature of 50°C.

The manganese removal circuit consisted of a 3 reactor cascade targeting 1 hour per reactor. Hydrated lime was added to control pH at 8.0 at a temperature of 70°C (later reduced to 50°C). Air was added to promote manganese oxidation.

The pilot plant generated approximately 200 to 300 samples per day, with solids Ni assays ranging from 0.1% to 40% and liquor tenors ranging from 1 mg/L to 100 g/L Fe. Filtration times ranged from under 1 minute for a MHP sample to more than 10 hours for a LSP sample containing 100 g/L Fe.

Piloting with 10 or more operators on a shift rotation represents the bulk of program costs and should therefore be planned carefully. In a circuit of around 24 hours retention time in the leach circuit alone, there are only a few chances to get it right. Hence, it is vital that from the start correct chemical information is available to the metallurgists. This is only possible by ensuring that individual sample processing protocols are developed prior to the start of the campaign and sampling personnel have been fully trained on them. For example, separate protocols were developed for handling leach pulp samples containing 80 to 100 g/L iron, to prevent dissolved salts

crystallizing during drying and resulting in a false, negative characterization of leach behavior.

The phase-wise approach used during the Skye project ensured that many of these issues were dealt with during earlier phases of the work, leading to a relatively short pilot plant commissioning time.

6. PILOTING RESULTS OF SAL PROCESS

6.1. LEACH SLURRY PREPARATION

As indicated above, several short LSP campaigns were carried out during Phase 3 to develop a protocol for the continuous piloting of the SAL process and to understand its chemistry. This consisted of several continuous limonite sulphation campaigns and combined limonite sulphation and saprolite grinding (LSP) campaigns. In addition, the effects of pulp density, mixing time, mixing temperature and pulp ageing (and cooling) were examined in a detailed bench scale program.

During the limonite sulphation, a dry and grey material was produced under steady state conditions in the pug mill at an acid addition of 1400 kg/t (limonite). XRD analysis of the sulphated limonite pointed to the formation of rhomboclase ($\text{HFe}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$) as the main crystalline phase. Based on an average content of 0.5% Ni, 20% Fe and 20% S, approximately 2.4-2.7 kg of sulphated limonite was produced per kg of limonite.

Measured pug mill temperature averaged around 140°C and it is of interest to note that even under those aggressive conditions, the only damage to the stainless steel internals of the pug mill was moderate staining of the steel surfaces. The combination of high temperatures and significant suction applied to the pug mill to prevent fugitive emissions, resulted in a substantial loss of water through evaporation, though a portion of the water was captured within the crystal structure of rhomboclase. Based on several campaigns, it was estimated that water evaporation was as high as 250 kg water per ton of limonite (dry) processed. In commercial practice,

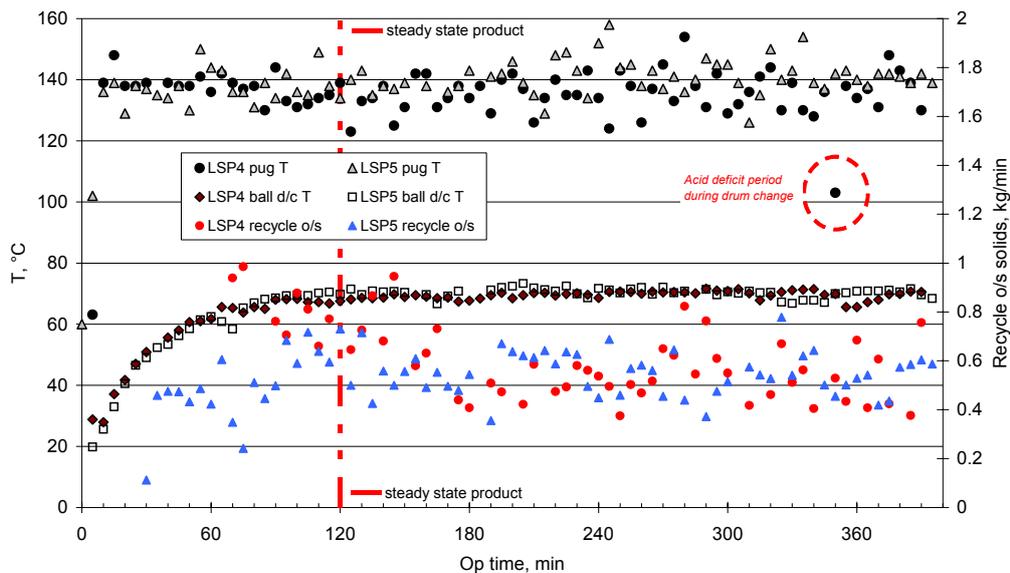


Figure 9 Temperature and Oversize Recycling in LSP Processing

it is expected that condensation of this saturated steam could be used to produce hot water for use in the process.

In Figure 9 a series of physical parameters are plotted against time. This type of graph was used to determine the period of time required to reach steady state. It can be seen that the pug mill reached steady state practically from the start. The ball mill temperature and screen o/s recycle appeared to reach constant values after 2 hours due to the mill's high thermal mass and recycling of oversize material. To ensure that a constant and on target S/L ratio was transferred to atmospheric leaching, it was decided to set aside material produced in the first 2 hours.

In the ball mill, sulphated limonite was dissolved in water and mixed or ground in the presence of crushed saprolite. Due to the elevated temperature in the ball mill some of the dissolved iron hydrolysed to leach saprolite. In fact,

average ball mill leach extractions of 52% Ni, 30% Co, 57% Fe and 35% Mg indicate that a significant portion of the saprolite was leached. The reactivity of the saprolite is further shown by the fact that measured free acidities in ball mill discharge pulp were consistently below 0.5 g/L H_2SO_4 (see also Table 3). During the pilot plant, pulp produced in the LSP circuit was drummed and transferred to the AL circuit. During the integrated piloting, the time between when a pulp was produced and the time the pulp was fed (and reheated) to the AL circuit was at times as long as several days. It is of interest to note that during the ageing of the pulp, calculated leach extraction increased to 68% Ni, 30% Co, 52% Fe and 59% Mg. Given the fact that both streams contain negligible amounts of acid, the mechanism of nickel dissolution can only be saprolite leaching by iron hydrolysis. The rate of iron hydrolysis is further shown in Table 3 and in Figure 10, which show decreasing Fe concentration and Fe/Ni ratios.

Table 3 SAL Process Liquor Composition (low Fe saprolite)

g/L	Ni	Co	Fe	Mg	H_2SO_4	Fe/Ni	Mg/Ni
Ball Mill PLS:	6.6	0.16	102	25.2	<0.5	15.5	3.8
Screen u/s PLS:	4.6	0.13	75.7	16.8	<0.5	16.4	3.6
Feed to AL:	5.3	0.13	67.0	24.3	<0.5	12.7	4.6
L1	6.9	0.37	44.7	34.7	pH 1.5	6.5	5.1
L3	6.7	0.34	25.5	36.7	pH 1.5	3.8	5.5
L5	7.2	0.37	23.0	40.6	4.0	3.2	5.6
Bench (L5)	8.0	0.4	23.9	47.0	4.5	3.0	5.9

6.2. ATMOSPHERIC LEACHING

Metal recoveries in various stages of the LSP/AL operations are presented in Table 4. The data indicate that nickel recovery in the final leach stage exceeded 87%. It also shows that the pilot plant performed very close to performance based on bench 'product leach' tests. Cobalt recovery 'jumps' from 30% in the leach feed to 86% in L1, showing the effectiveness of the SO₂ addition. Cobalt recovery further improves to around 88.6% in reactor L5. SO₂ consumption during the 10 day integrated pilot campaign averaged around 8 kg/t ore and resulted in an average ORP reading of 600 mV (vs. Ag/AgCl) and ferrous levels of below 1 g/L Fe²⁺. It is of interest to note that during periods of higher SO₂ dosages (and resultant higher ferrous levels) cobalt recovery did not improve.

Table 4 SAL Process Leach Recoveries (low Fe Sapolite)

%	Ni	Co	Fe	Mg
Ball Mill	52.0	31.1	57.0	31.5
Screen u/s	51.7	31.4	54.6	36.9
Feed to AL	68.0	30.3	52.4	58.6
L1	80.7	85.9	40.7	76.3
L3	86.4	89.0	24.0	88.6
L5	87.2	87.9	20.4	89.8
Bench (L5)	87.5	88.6	21.5	89.0

Table 5 summarizes the main SAL process parameters and the key results are included for the two final pilot plant campaigns, each using a different type of sapolite. It shows that an overall lower Ni extraction is obtained when using the high Fe sapolite. It may be feasible to optimize these results somewhat, particularly at the limonite sulphation stage. However, the higher sapolite Fe content is indicative of a higher goethitic content. While this nickel containing goethite phase may react under the aggressive limonite sulphation conditions of 1.4 t/t acid addition, this phase is not soluble under the mildly acidic AL conditions (pH 1.5 to 5 g/L H₂SO₄).

Table 5 SAL Process Comparison, High and Low Fe Sapolite Ore

SAP ORE	Ni, FEED %	Mg, FEED %	A/O kg/t	A/L t/t	S/L t/t	Fe/Ni (L5) -	Ni EXTR (L5) %
High Fe	1.8	9.0	560	1.4	1.53	2.7	84.0
Low Fe	1.8	10.2	582	1.4	1.41	3.2	87.2

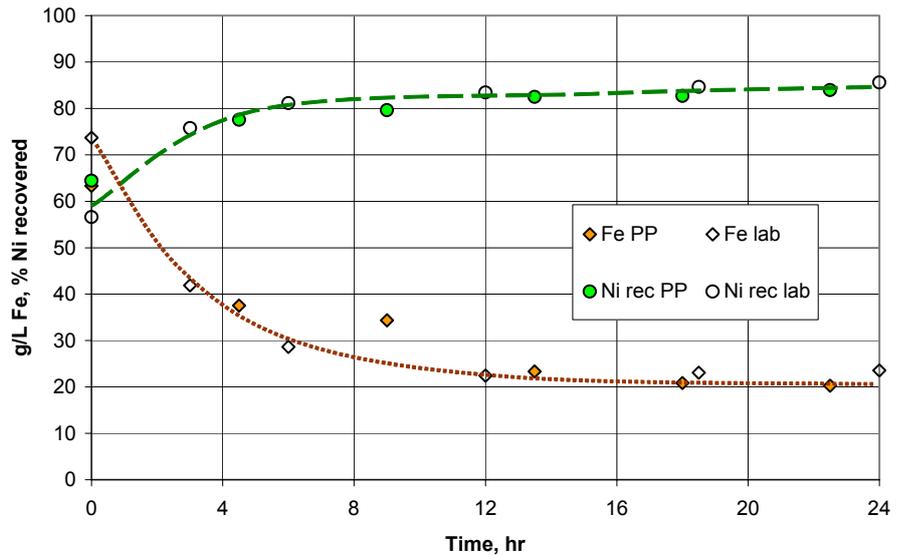


Figure 10 Kinetic Profile of Fe Hydrolysis and Ni Recovery in Pilot and Bench Tests (high Fe sapolite)

The pilot plant operated at a leach retention time of approximately 22 hours equally divided over 5 reactors. As can be seen in Table 4, nickel extraction reached 86.4% in L3 (equal to 13 hours). Kinetic iron precipitation and nickel recovery curves of pilot plant and comparative leach tests are plotted in Figure 10, showing that a leach time of around 15 hours is sufficient for optimum leaching. It also shows how closely the pilot plant performance matches the bench tests performance.

Fe/Ni ratio's of around 3 (see Table 5) indicate that some acid regeneration capacity remained in the AL circuit. A small series of bench tests showed that recycling SIR and SP precipitates to L1 (start of AL) further reduced the Fe/Ni ratio to around 1.4 while re-leaching a significant portion of the precipitates. In the pilot plant this was not implemented due to insufficient testing to confirm re-leach efficiencies. Instead, a separate re-leach reactor was used with a fresh acid addition, increasing overall A/O ratio from 580 kg/t to around 600 kg/t.

As indicated above, in a circuit with 24 hr retention time, there are only a few chances to get it right from the start.

Figure 11 illustrates the leach recovery trends for several metals during the 10 day final pilot campaign. Target leach extraction was obtained from day 1 and was maintained until the end of the campaign, resulting in the production of useful operating data and production of representative product streams (most notably CCD feed streams). Nevertheless small operating changes were made to optimize variables such as cobalt recovery. It is here where the advantages of the Phase 4 program become apparent, as those campaigns were used to resolve the bulk of problems in the up and down stream circuits.

Detailed mineralogical analyses showed that the majority (~60%) of nickel in the leach residue (L5) occurred in amorphous goethite silica gels. A moderate (26%) proportion of the nickel was also hosted by fairly crystalline iron oxides of goethite, hematite and maghemite. It is of interest to note that only 9% of the nickel was hosted by leached serpentine. This provides a strong indication that further nickel recovery may be obtained by optimizing the limonite sulphation stage, for example, by improving the acid-limonite blending.

Atmospheric leaching of limonite and sapolite consumes large amounts of acid. This is mainly due to the high acid consuming sapolite ores. Magnesium sulphate in leach liquor accounted for ~64% of the total acid addition.

However, iron co-extraction is also responsible for part of the high acid consumption, ferric sulphate accounting for ~16% of the total acid addition. Leach residues (L5) contained on average 1% sulphur, accounting for approximately 4% of the total acid addition.

6.3. PRIMARY IRON REMOVAL

Iron extraction in atmospheric leaching is significantly higher than comparative HPAL processes due to the very efficient iron hydrolysis reaction under HPAL conditions. Hence, iron needs to be precipitated in a neutralization circuit. The Skye PIR circuit operated at 75°C and was based on limestone neutralization to a target pH of 3.0. Iron and aluminium removal averaged 90% and 69% efficiency, respectively. Residual iron was all ferrous and was completely removed in SIR. Aluminium precipitation was used as a gauge for pH control. Measuring pH at high pulp densities and temperatures is difficult and it was found that a target removal of around 70% Al ensured that all of the ferric iron was precipitated, while keeping nickel and cobalt losses under control. Nevertheless, the greatest source of lost pay metals (besides un-leached metal values) occurred in PIR with 2% loss of nickel and 2-5% loss of cobalt. It was often observed that some level of re-dissolution occurred in the CCD circuit.

6.4. COUNTER CURRENT DECANTATION

One of the main drawbacks of an atmospheric leach process for laterites is the more difficult solid liquid separation step. Particularly in a pilot plant with changing conditions this may have practical implications to the overall plant performance. Despite the fact that a significant amount of solid liquid separation bench work was performed, early campaigns experienced difficult CCD operation, leading to poor wash efficiency and high levels of suspended solids in CCD1 overflow. Supportive bench test work prior to and during the Phase 4 pilot campaigns had shown that the following parameters were critical:

Pulp dilution was the single most important parameter in maintaining proper flocculation. There exists a critical pulp dilution level that, if exceeded, results in almost instant

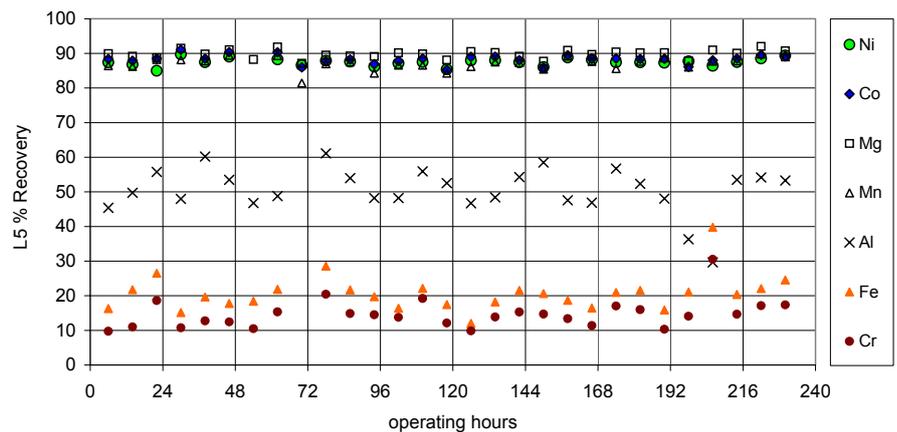


Figure 11 Leach Recovery Trends (low Fe saprolite)

loss of flocculation. The loss of flocculation resulted in unstable thickener operation and rising mud lines within 15-30 minutes. During several pilot campaigns it proved to be difficult to recover from such upsets as pulp dilution was accomplished by recirculating supernatant from the top of the thickener. However, the dilution effect was diminished during times of unstable operation as the recirculation would include unsettled solids. Based on observations gathered during all campaigns, this cut-off point was estimated to be around 3% solids. Early pilot campaigns targeted a dilution ratio fairly close (2.5 to 3%) to this cut-off point and a great deal of time and energy was spent fighting system upsets. The final fully integrated pilot campaign operated at target pulp dilutions of 1.5 to 2% solids, which resulted in a smooth and efficient CCD operation.

Reagent mixing conditions were also found to be critical. In the Skye pilot campaigns, it was found that the addition of coagulant (MF368) was critical for good flocculation to take place. It appeared to act as a pre-conditioner to the addition of flocculant. In this pilot plant coagulant was added to the thickener feed mix tank, while flocculant was added in-line into the thickener feed line. In addition, it was found that decreasing the flocculant stock strength from 1 g/L to 0.5 g/L (MF351) improved flocculation characteristics. Some key trends of the integrated pilot campaign are included in Figure 12, which show the effects of wash ratio on nickel tenors in CCD1 and CCD6. The wash ratio was decreased at 144 h to values below 2, in order to produce a higher

grade feed liquor to the MHP circuit. Also shown are the CCD underflow % solids trends, which indicate a constant CCD operation with CCD1 and CCD6 u/f densities approaching 27 and 30% solids respectively. This was accomplished at an average feed dilution to 1.7% solids in CCD1 and CCD6.

6.5. DOWNSTREAM AREA OPERATIONS

It is beyond the scope of this paper to discuss in detail the downstream circuit however some key results specific to treating a mixed limonite and saprolite ore are included below. The downstream circuits consisted of known and proven processes. However, due to the high Mg content of the saprolite ore there is a distinct solution chemistry characterised by the high dissolved magnesium levels.

The nickel and cobalt recoveries in the downstream plant were 98.8% and 99.3%, respectively. Principal Ni and Co losses occurred in the manganese removal circuit and were in the manganese removal solids. However, improved clarification of SP thickener overflow liquor would have prevented most of these losses.

Nickel and cobalt 'losses' in SIR averaged 4% and 2% respectively, while operating at a final pH of 5.0. The majority of these 'losses' were recovered in a re-leach circuit and recycled back to AL. It was found that ferrous oxidation by air in the relatively small pilot scale tanks was inefficient, despite the use of Rushton impellers for improved gas dispersion. Instead, hydrogen peroxide was used to oxidize and precipitate iron. Aluminium was also completely removed in this circuit.

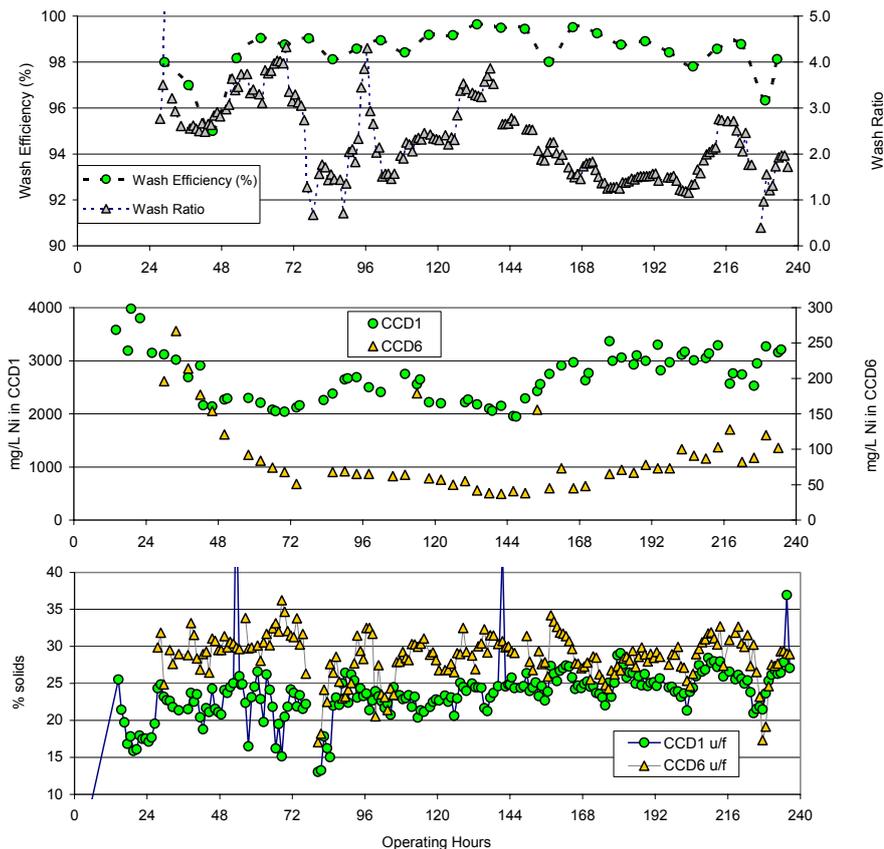


Figure 12 CCD trends

Average MHP precipitation efficiencies were 85.6% Ni and 96.0% Co leading to a final product assaying on average 41.6% Ni, 2.5% Co, 1.2% Mg, 4.5% Mn and 3.8% S. The primary control philosophy employed in the MHP circuit was based on adding a set amount of MgO to the MHP feed stream. The MgO addition factor was defined as the ratio of the kg of MgO per kg of Ni and Co in the MHP feed. During the final integrated campaign the MgO addition factor was 0.65.

Average SP recoveries were 98.0% Ni and 95.4% Co leading to a final product assaying 11.7% Ni, 0.2% Co, 5.5% Mg and 13.5% Mn. SP thickener overflow contained 2 mg/L Ni, <1 mg/L Co, 18 g/L Mg and 0.4 g/L Mn.

Manganese was selectively precipitated from the SP thickener overflow by controlled oxidative precipitation with air and hydrated lime at pH 8.0. Manganese precipitation efficiency was >98% with less than 5% Mg co-precipitation. Final effluent contained less than 10 mg/L Mn and approximately 18 g/L Mg.

7. CONCLUSIONS

The following conclusions were made during the execution of this project:

- The stage wise development approach used in this project was found to be very effective, both from an economic and a technical point of view;
- All unit processes should be tested at bench scale to estimate reagent consumption and determine optimum conditions;
- Where possible, S/L separation testing needs to be included using freshly prepared samples;
- Key unknown unit processes (such as the LSP process) require separate stand alone pre-pilot campaigns.
- Allowing one or more semi integrated pilot campaigns for process optimization allows for the de-bugging of the circuit and for the training of operating and sampling personnel;
- The SAL process as developed and piloted at SGS facilities proved to be a robust process. A well run final campaign resulted in smooth and constant operation and was used to produce product streams for vendor testing as well as for demonstrating the process to potential investors.

8. ACKNOWLEDGEMENTS

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