

# CAN COMPLEX HYDROMETALLURGICAL PILOT PLANTS EFFECTIVELY REDUCE PROJECT RISKS? PART ONE: KEY METALLURGICAL AND TECHNICAL MANAGEMENT ISSUES

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Pilot plant metallurgical testing is commonly included in feasibility studies with the intent to limit the risks associated with the commercial implementation of mining projects. Still, metallurgical problems are among the main causes leading to disappointing project outcome. The most serious difficulties often arise in the case of projects involving difficult ores, generally requiring novel and/or complex flowsheets. The paper discusses pilot plant practices proven to be effective in producing metallurgical test data of sufficient reliability to effectively minimise metallurgical project risks. The causes and remedies of poor metallurgical performance at the pilot plant level are also discussed. Broader technical management issues covered include design, construction and operation of complex hydrometallurgical pilot plants. Particular emphasis is placed on pilot plant operations with respect to their preparation, management, design, operation and most importantly, overlooked metallurgical issues. Piloting experiences discussed include flowsheets treating polymetallic feed samples containing nickel, cobalt, copper and gold as pay metals.

## INTRODUCTION - THE BIG PICTURE

Current and predicted metals prices seem to confirm a sustainable upturn in the mining business. This (long overdue) positive trend inevitably leads to increased project activity. When new projects come on line there is an increased chance that some of these new plants encounter problems during commissioning, which in turn can have quite a negative effect on the industry. There are opinions whereby metallurgical risk is among the leading causes of the failure of mining projects - some believe in fact, and with good justification, that after resource risk, metallurgical risks account for most of the failures of mining projects

[1]. This situation does not appear to be changing for the good, in spite of the fact that metallurgy should be more "controllable" compared to other areas of lower risk, such as financing, environmental, political, market, etc. The chances for lowering metallurgical risk could in fact be diminishing as the global workload is increasing while the supply of a qualified workforce remains relatively unchanged for the short to medium term. This may well be the legacy of the extended period of weak metal markets and thus characterised by a lack of interest in the extractive metallurgical profession. The root causes leading to metallurgical failures, particularly with respect to their effect on ramp-up times, have been discussed [2, 3]. The purpose of this paper is to focus on the pilot scale metallurgical testing and its role in preventing metallurgical failure. Particular emphasis is placed on pilot plant operations with respect to their preparation, management, design, operation and overlooked metallurgical issues. The underlying logic of piloting is disarmingly simple - a pilot plant contains most of the critical attributes of a commercial plant that allow for the evaluation of the metallurgical response of typical feed samples. A pilot plant is designed and built having the clear objective of trouble-free commissioning and rapid ramp-up to designed tonnage throughput and metallurgical performance. Reaching these objectives generally tends to indicate the existence of a positive metallurgical environment whereby fatal operating errors are unlikely. A modern continuous pilot plant allows for cost-effective operation while maximizing the quantity and quality of the data produced. Good operation does not necessarily preclude the existence of process flaws - the data produced will however flag these problems.

Producing data requires that the pilot plant operation starts-up and operates as planned. Key to achieving this is detailed preparation, as will be discussed below.

This paper highlights the importance of establishing the flowsheet design during the laboratory phase preceding the pilot plant, in order to maximise its chances of success.

To begin, it is important to recognise that the conclusions of the McNulty-Nice papers [2, 3] are entirely applicable at the pilot stage as in most full scale metallurgical projects. Accordingly, the main causes of pilot-plant failure include:

- Insufficient time, sample and human resources - or succinctly - not enough money;
- Work scope and expectations are poorly matched
- Over-confidence in the proposed flowsheet - generally combined with lack of key metallurgical expertise, as well as a lack of bench scale testing.

The majority of the problems occur when new processes are tested, and this is generally in relation to non-conventional feed stocks, where processing is carried out hydrometallurgically. The role of comprehensive pre-pilot testing in these situations can not be understated. This paper complements some of the ideas put forward in a recently released SGS Minerals Services paper [4], which provides an overview of issues relating to hydrometallurgical piloting.

## THE CONTINUOUS HYDROMETALLURGICAL PILOT PLANT - OVERVIEW

### OBJECTIVES

The main objectives of a hydrometallurgical pilot plant exercise include:

- Continuous and integrated testing of the metallurgical response of the sample(s) to a certain process;
- Proof of process concept;
- Generation of prefeasibility and feasibility engineering data for commercial design;
- Identification of potential operating, automation, corrosion and maintenance problems;
- Retesting and validation of optimum process parameters and new reagents;
- Securing product samples for vendor- equipment sizing, comprehensive analyses, evaluation and further testing;
- Completion of a comprehensive report that outlines all aspects of the process, including possible negative outcome and remedies, and most importantly - whether the process is technically feasible or not.

### FEATURES

The main feature of the modern pilot plant is that it is designed, built and operated in a fashion that resembles as closely as possible the future commercial plant. Features of the commercial plant that should be mimicked or modeled as closely as possible include:

- Detailed flowsheet design;
- Detailed equipment sizing and selection;
- The process chemistry - a pilot exercise is the very first instance of when the chemistry of a flowsheet is tested under conditions that simulate "real-life";
- Individual target definition for each unit operation;
- Integration of recycle streams;
- Integration of critical key unit operations and processes;
- Replication of all relevant conditions with respect to materials handling, corrosion, scaling, environmental impact and tailings disposal.

Differences between pilot plants and commercial plants include:

- Actual equipment used may be different at times;
- Data generation takes priority over the production of metallurgical samples;
- The operation time is generally significantly shorter than the preparation time (i.e. planning, design and construction).
- Extensive requirements of "managing change" as opposed to routine managing occurring in current operations;
- Pilot plants are "forgiving"; which can be either good or bad, depending on the metallurgical judgement exercised by the beneficiary of the data produced.
- Slight "over sizing" of equipment may be necessary in the pilot plant in order to be able to accommodate a range of feed samples and test conditions.

Limitations of the pilot plants include:

- The data produced by the pilot plant will tend to reflect, the behaviour of the samples tested, hence the need of sample variability testing for those projects with predicted long term mineralogically diverse deposits;
- Existence of unit operations that can not be commonly replicated in a manner that allows for generation of scale-up data - examples include oxygen mass transfer, especially at atmospheric pressure, pressure autoclaving, heat losses/balancing, long distance pipe-loop operation, etc.;
- The long term behaviour of certain equipment (pumps, compressors, drives, sensors, etc.) may not be fully ascertained through a pilot plant exercise, because pilot plants are never operated for long enough;
- Failure of materials of construction is sometimes difficult to predict based on pilot plant operations. Examples include mechanical erosion (flash vessels, choke valves), stress corrosion cracking, certain solvent extraction related scaling, anode damage.
- Replication of process control strategies is often difficult due to less automation encountered in the pilot plant, which in turn is limited by the low flowrates.

## THE ULTIMATE LIMITATION

The ultimate limitation of a pilot plant is that it can not be used efficiently and cost effectively for process flowsheet optimization testing. This limitation is very important. In fact, a pilot plant is the most expensive process optimization. Accordingly, attempting to carry out process optimization during the pilot plant operation will significantly increase the project risk. This does not preclude normal operational flexibility that a well-designed pilot plant is expected to allow for. Indeed, this is the strength of pilot operations where allowances for sample variability testing, adjustment of certain parameters within pre-planned limits, necessary corrections in recycle stream flowrates (as a result of changing conditions) are included as part of the program. In practice this means that the design, sizing and construction of the equipment, piping and peripherals should permit for adjusting of the residence time in the various circuits, as required by each individual metallurgical sample tested. The limitations of a given pilot plant should be analysed with respect to its main goal. A "proof of concept" pilot plant for example should provide more flexibility compared to a "feasibility stage" pilot plant. On the other hand, flexibility should be realistically limited such that real life conditions are maintained as close as possible.

## RAMP-UP, DURATION, EXPECTATIONS

The differences, similarities and limitations listed above clearly suggest that a pilot plant can be compared to a commercial plant in its very early commissioning and ramp up stages. But, at the same time, they highlight the main difference between the pilot plant and the commercial plant, whereby a pilot plant must reach its nominal throughput within hours, versus ramp up times of months or years in a commercial plant. The first important decision regarding the planning of the pilot plant refers to its intended duration, which in turn is determined by generally conflicting considerations, for example metallurgical versus promotional, financial and others. The average duration of the modern hydrometallurgical pilot plant has decreased in recent years to about four weeks, which includes

about two to three weeks of fully integrated steady-state operation. By comparison, the late nineties pilot plant campaigns were up to three months in duration, such as in the case of some laterite nickel testwork programmes. The pilot plant campaigns of the "eighties" were often run in several multi-month campaigns, some even spanned over a few years.

In spite of this trend of reduced duration of the pilot plants, their goals have actually expanded, both metallurgically and beyond. This is partially due to new feed resources requiring new processes, but also the increasing environmental and political pressures to which both greenfield and brownfield projects are being subjected nowadays. Undoubtedly, shortening the pilot campaigns while extending their scope implies less time is available to reach their objectives. To compensate for this situation, additional time is required for their planning. The preparation time for a successful pilot plant is from three to five months, depending on factors such as:

- Equipment availability
- Flowsheet complexity and novelty;
- Relevant and sufficient bench data;
- Representative sampling of pilot plant feed
- Clarity/definition of objectives
- Resources available - human, logistics and financial.

#### MANAGING CHANGE

The need for managing changes during a major metallurgical program is not new, but it has become more stringent recently because of the realities of modern pilot plants. Managing change begins from the inception of the pilot plant and it involves key decision making and operating people assigned to the project. In a sense, it can be stated that changes tend to become the "backbone" of the piloting business, a very unique common component that can be singled out in every pilot project.

This paper will focus on objective changes needed to successfully mitigate the metallurgical risks, with emphasis on flowsheet development:

The main elements of the positive change-management process in the case of flowsheet changes include:

- Detailed flowsheet analyses, with emphasis on its "pilotability";
- Analysis of the implications of the changes regarding generally overlooked aspects that affect operability. These may include liquid-solid separation, rheology, effect of recycle, effect on integrating downstream unit operations on residence and hold-up times; issues related to corrosion.
- Carrying out overall risk to reward ratio analysis, based on a comprehensive supporting bench scale testwork program. This step is the most difficult to put into practice. However, it is generally the only one that provides certainty and a positive outcome, should the results warrant it.

An example of positive management is provided below, and it is inspired from the realities of the routine preparations involved in a pilot plant. The underlying philosophy is that a successful pilot plant relies on a well established flowsheet, which does not require further changes during the operation. This allows for testing the response of several feed samples. This process may require a significant number of revisions of the original flowsheet. The revisions can be done generally based on extensive bench scale testwork, in conjunction with pre-pilot runs. The end result is a "pilotable" flowsheet, characterised first and foremost by operability.

#### EXAMPLE - PRESSURE OXIDATION AUTOCLAVE - DISCHARGE OPTIONS

##### SCENARIO

This example describes certain details related to the preparation (i.e. planning and designing) of a pilot plant in which sulphidic concentrates were oxidized by oxygen in a continuous autoclave. SGS Minerals Services carried out this exercise as part of an in-house development program focussing on generic pilot plant flowsheet development and planning. The goal of the study was to determine the best available autoclave discharge treatment option that would allow for smooth operation of the downstream pilot plant.

The feed sample consisted of a polymetallic sulphide concentrate containing 29% S, 24% (Cu+Ni+Co) and 11 g/t Au. The discussion provided in this particular section is limited to factors that influence the operability of the continuous pressure oxidation (POX) in a pilot autoclave, with specific focus on discharge treatment options. The continuous POX autoclaving process was followed by liquid-solid separation in conjunction with partial neutralization to pH ~ 2, using limestone slurry. Whilst quite commonly applied, this generic sequence of unit operations is still sufficiently complex to create problems during pilot plant operation, if not prepared with attention to certain details, as discussed below.

##### RISKS

POX parameters scale up - from bench ("Parr") test to continuous operation - from a pure process chemistry point of view, the optimisation of POX leaching parameters is generally a routine bench scale exercise and produces straightforward results. Scale-up problems are quite rare and they can be generally addressed successfully by carrying out a few pre-pilot plant continuous POX runs. Continuous operation allows for better understanding of the POX process, allows for "fine-tuning" of parameters such as oxygen flowrate, pressure and off-gas content. Agitator rotation speed ("RPM regime") and feed solids density for autothermal operations are also important parameters that can be determined / confirmed relatively easily during the pre-pilot runs. The process chemistry is often somewhat different in the continuous autoclave compared to batch POX, particularly related to oxidation of iron sulphides and the type of iron hydrolysis compounds formed. Reaction kinetics can also be subtly different, and because of the impact on downstream equipment sizing, optimum throughput rate in the autoclave needs to be confirmed in short pre-pilot plant runs. Other issues that can be looked at during the pre-pilot plant include corrosion related issues, analytical sampling and sample processing, data collection and reduction, with emphasis on mass balance and accountability. Corrosion testing may not always be accommodated within the fully integrated

pilot plant because of issues relating to the stability of dissolved species and the risk of contamination by the eventual corrosion products.

**FEED ISSUES** - although often a simple issue, feed characterisation is critical to the continuous POX process. Severe problems can arise for example if the pilot plant feed is different from the bench scale feed. Feed inconsistency quite frequently occurs in commercial operations. The implications can be quite grave because feed composition affects the process chemistry, which in turn affects the downstream unit operations. Avoiding some of the feed related issues can also be successfully addressed during the pre-pilot continuous runs. On the other hand, feed variability testing can be a critical aim of the pilot plant operation requiring constant attention throughout its duration.

**OPERABILITY** - autoclave feed and control issues generally take precedence during the preparation stage of the pilot plant, and pre-pilot runs allow for detailed “debugging” of any problems that may arise. The most significant overlooked aspect however relates to the handling of the autoclave discharge slurry from the perspective of liquid-solid separation and rheology (“LSR”), along with the underlying factors such as changes to the physical properties of the constituents of the slurry. For this reason it has been chosen as the prime example of how important hydrometallurgical pilot operations are.

The key interactions discussed above are depicted schematically in Figure 1.

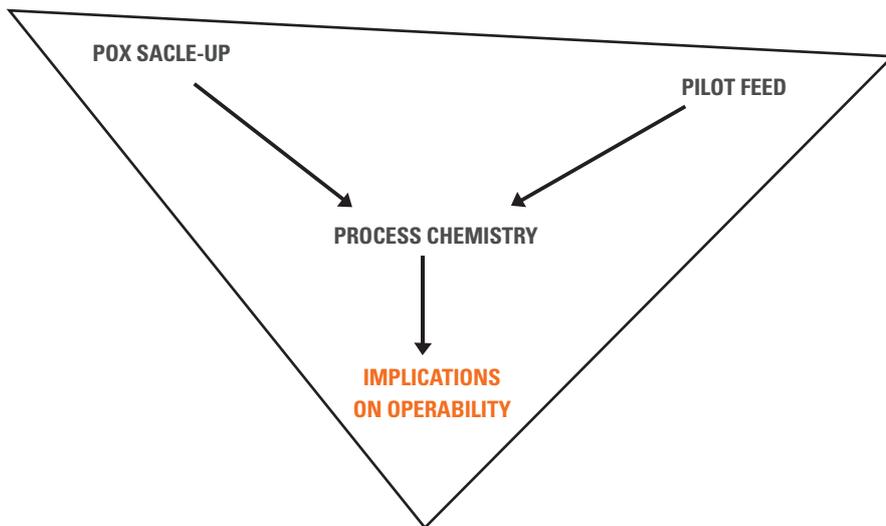


Figure 1 Key interactions

**OPTIONS**

In the example of pressure oxidation, feed characteristics and autoclave operating conditions will determine the actual process chemistry, which in turn will influence process operability, as depicted in Figure 1.

The complications arise from the fact that the process design is often based on process chemistry optimization (batch) results only, without considering their implications on operability. This is an overlooked aspect of flowsheet design that appears to be a leading cause of metallurgical problems. These problems are often detected for the first time in the pilot plant but, at a significant cost of time and money. While still better than building a real plant that would not operate, the effects of a failed pilot plant are often damaging to the project.

**DETAILS**

The autoclave discharge treatment options considered for the continuous POX process are summarized in Figure 2.

- Option A - direct discharge where POX slurry goes straight to a thickener - the liquid-solid separation and rheological response is quantified as a function of the process parameters and one set of conditions selected for further investigation.
- Option A1 is a distinct part of Version A as it involves the partial-neutralisation to pH ~2 (using limestone) of the overflow liquor produced from the Version A slurry produced under the selected conditions;
- Option B indirect discharge in the sense that the selected Version A produced POX autoclave discharge slurry is pre-neutralised to pH ~2 (using limestone) prior to being subjected to settling-thickening-rheology characterisation.

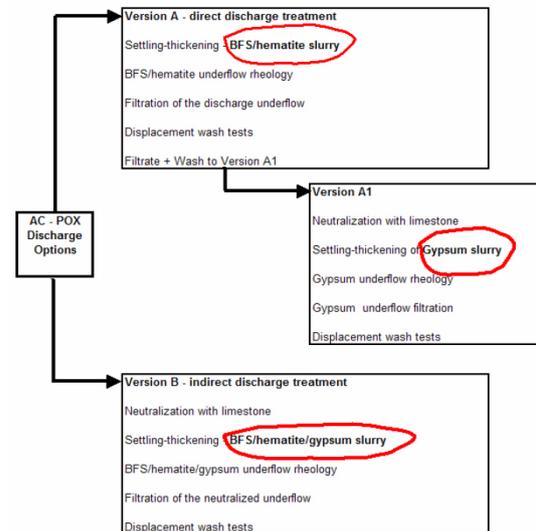


Figure 2 Discharge treatment versions considered

According to the authors' experience, there is a consistent need in hydrometallurgical pilot projects to carefully consider the physical implications of the purely chemical optimization process. This is because changing the chemical parameters (through optimization) will cause changes in the physical behaviour of the resulting autoclave discharge slurry. Hence, the need for making provisions to include specific investigative testwork in preparation of the pilot plant. The results of this testwork allow for the flowsheet to be "shaped" as required to ensure process operability. When this exercise leads to a change in an existing flowsheet proposed for piloting (and generally it does) it meets the attributes of a so called "positive change management," since it allows for planning and implementation before the pilot plant design stage.

In the case of the generic example discussed, the pre-pilot test data package normally required can be produced by a combination of short autoclave runs and parallel comprehensive bench scale work. That testwork is carried out on fresh autoclave discharge samples. Discussing the possible outcome of such a testwork program in its entirety is not the aim of this paper. However, a succinct data presentation on continuous POX autoclaving, liquid-solid separation, rheology and related issues is provided as follows:

- Continuous pre-pilot POX runs results overview - extractions vs. key process chemistry parameters;
- Version A - direct discharge thickening and underflow rheology results vs. process chemistry parameters;
- Comparative results - all versions - all relevant data under selected Version A conditions

#### OVERVIEW OF THE CONTINUOUS PRE-PILOT POX RUNS RESULTS

Bench scale testwork determined several POX conditions from which a set of three was selected for further confirmation/optimisation through continuous autoclave pre-pilot runs. The objective of the pre-pilot runs was to investigate the conditions

that would ensure autothermal operation, acceptable metal extractions and downstream operability. The runs were carried out on a representative composite feed sample, with allowance for about 20 hours of steady state operating time to test each set of parameters. Sample variability was briefly tested towards the end of the exercise, for about 6 hours operating time each. The continuous run results indicated that maximum Cu/Ni/Co extractions of about 95% each could be achieved under the following conditions: feed grind size  $K_{80} \sim 70$  microns, temperature - 210°C, 90 minutes residence time. Feed solids density of ~12% wt. was needed to ensure auto-thermal conditions for 210°C operating temperature. Lowering the feed solids density below ~12% wt. caused a drop of about 4°C per each percent solids decrease. The pre-pilot POX autoclaving was carried out with particular attention to maximising extractions (Table 1) while minimising the ratio between the basic iron sulphate (BFS) and hematite formed during the process. Hematite is generally stable at higher temperatures (>200°C) and relatively low acidity (<30 g/L at 200°C in this case). At a certain temperature, the main factor determining the BFS/hematite ratio is the free acidity produced by oxidation of the sulphur, which in turn is determined by its availability (in the feed), as well as its degree of oxidation during the POX process as to allow for autothermal conditions. In addition, the iron and free acid contained in the recycle solution streams could also ultimately become a potential source of BFS. Avoiding BFS formation is important for several reasons, including handleability of the discharge slurry, environmental stability of the tailings and optimum cyanidation process chemistry, in case of a downstream gold plant.

The following clarifications are provided in relation to the data summarized in Table 1:

The solid phase component of the autoclave discharge (and hydrometallurgical slurries in general) does not display a specific gravity ("SG") that can be determined directly using for example a nitrogen purge picnometer. Instead, the density of the solid phase is calculated based on direct determination of the pulp and solu-

tion densities. This value is defined in this text, as well as in previous SGS Minerals Services publications, as Actual Specific Gravity ("ASG").

The pH of the autoclave discharge pregnant solution does not reflect its true free acid level (i.e.  $H_2SO_4$ , i.e.  $H^+$  activity) because of the presence of metal ions (i.e. ferric), which are capable of producing excess acidity by hydrolytic dissociation. This implies that the free acid can not be calculated based on the pH - instead, it must always be determined by titration.

#### OVERVIEW OF "DIRECT DISCHARGE" (OPTION A) SETTLING - THICKENING - RHEOLOGY RESULTS

Freshly produced autoclave discharge slurry samples were subjected to bench scale flocculant screening and subsequent settling-thickening testwork.

The settling, thickening and separation results (Table 1 and Figure 3) indicated that, in general, the POX conditions that ensured the best extractions created the worst liquid-solid separation conditions, i.e. they were inverse proportional to the ionic strength of the PLS solution, as detailed below:

- The slurry samples tested displayed settling only in the presence of a very high molecular weight, cationic charged flocculant - this was a result of the overall high ionic strength of the solution of the autoclave discharge.
- The specific thickener unit area requirement ("TUFUA") for the POX conditions that produced 95% average metal (Cu, Ni, Co each) extraction was four times greater compared to the TUFUA requirements for the POX conditions that produced 77% metal extractions, i.e. 0.36 m<sup>2</sup>/t/day vs. 0.09 m<sup>2</sup>/t/day.
- The specific flocculant consumption for the POX conditions that produced 95% average metal (Cu, Ni, Co each) extraction was about 13 times greater than the consumption under the POX conditions that produced 77% metal extractions, i.e. 164 g/t vs. 13 g/t;
- The initial settling rate for the POX conditions that produced 95% average metal (Cu, Ni, Co each) extraction was

five times smaller than the initial settling rate for the POX conditions that produced 77% metal extractions, i.e. 120 m<sup>2</sup>/t/day vs. 602 m<sup>2</sup>/m<sup>2</sup> day.

Rheology testing of typical underflows produced by the settling-thickening tests indicated that they displayed a Bingham plastic behaviour, meaning that they would flow only if subjected to a shearing regime that imparts sufficient energy to overcome a critical level called the yield stress. The yield stress vs. solids density curve allows for determination of the critical solids density (CSD), defined as the solids density at which the yield stress increases significantly for insignificant changes of the solids density [5-7]. The CSD value also provides valuable indications relative to:

- Maximum underflow solids density achievable in a continuous thickener;
- Solids density value where the friction pressure losses during pumping increases considerably.

Rheology data (Table 2, Figure 4) indicated that:

- The solids densities expected in the pilot plant/commercial thickeners would be higher compared to the densities produced in the static settling tests;
- The above conclusion was in agreement with the general observation that the static settling-thickening tests tend to produce conservative results with respect to the solids density of the underflows produced;

- The flowability of the underflow that would be produced would increase in inverse proportion to the ionic strength of the autoclave discharge solution, i.e. to the solids density of the autoclave feed slurry.

<b>AC FEED SOLIDS DENSITY, % WT.</b>	11.80	8.30	6.90
<b>AC AVERAGE TEMPERATURE C1-C5, °C</b>	210	195	189
<b>AC DISCHARGE = THICKENER FEED SOLIDS, % WT.</b>	6.6	4.5	3.5
<b>AVERAGE EXTRACTIONS (Ni, Co, Cu), %</b>	95	89	77
<b>AC TEST PRODUCT</b>	As IS A/C Discharge Version A - Direct Feed to the Thickener AC Leach Residue		
<b>PROCESS VERSION</b>			
<b>MAIN NATURE OF SOLIDS IN SEPARATION TESTS</b>			
<b>DISCHARGE SOLIDS ASG AS TESTED, g/cm<sup>3</sup></b>	3.21	3.38	3.52
<b>CHARACTERISTICS OF THE AUTOCLAVE DISCHARGE PLS</b>			
<b>pH</b>	0.41	0.78	0.89
<b>FREE H<sub>2</sub>SO<sub>4</sub>, g/L</b>	49.6	29.7	20.1
<b>Me (Ni, Co, Cu), g/L</b>	36.8	23.2	19.9
<b>STATIC SETTLING-THICKENING DATA SUMMARY (KINCH TESTS WITH TALMADGE - FINCH REDUCTION)</b>			
<b>CATIONIC FLOCCULANT, g/t DRY</b>	164	41	13
<b>THICKER U/F SOLIDS DENSITY, % WT.</b>	29.2	43.1	55.7
<b>THICKENER UNDERFLOW UNIT AREA, m<sup>2</sup>/t/day</b>	0.36	0.18	0.09
<b>THICKENER HYDRAULIC UNIT AREA m<sup>2</sup>/t/day</b>	0.07	0.04	0.03
<b>INITIAL SETTLING RATE, m<sup>2</sup>/m<sup>2</sup>.day</b>	120	413	602

Table 1 Pre-pilot autoclave runs - conditions, extractions and liquid-solid separation data

LEGEND/FOOTNOTES

Common POX conditions: 210°C, 90 minutes residence time, 10 g/L initial H<sub>2</sub>SO<sub>4</sub>, 100 psi O<sub>2</sub> overpressure, 90% O<sub>2</sub> in off gas, 450 RPM.  
Feed grind K<sub>80</sub> ~ 70 microns.

Fresh (~90°C) AC discharge samples were retrieved and subjected bench testing.  
ASG - Actual specific gravity of dry solids, calculated from the measured pulp density ("Density")  
Autoclave discharge was autodiluted (with overflow) by about 50% to ensure good flocculation.

All Data reported "As Produced" - No Safety Factors Included.

AC FEED SOLID DENSITY, % WT. AND RESULTING DISCHARGE SOLUTIONS FREE ACIDITY, g/L H <sub>2</sub> SO <sub>4</sub>					
11.8 % wt. AC feed	50	8.3% wt. AC feed	30	6.9% wt. AC feed	20
Resulting discharge underflows Solids density, % wt. and Yield stress, Pa					
% wt.	$t_{yB_3}$ Pa	% wt.	$t_{yB_3}$ Pa	% wt.	$t_{yB_3}$ Pa
34	21	49	28	63	55
29	12	43	17	56	33
26	9	39	14	51	24
23	6	36	10	49	22
CRITICAL SOLIDS DENSITY (CSD), % wt. and corresponding Yield stress, Pa.					
33-35	>20	48-50	~30	63-65	>55

Table 2 Autoclave POX discharge underflow rheology data summary

Variable shearing was produced in the 0 to 600 s<sup>-1</sup> range.  
 Constant shearing was produced at 300 s<sup>-1</sup> for 300 seconds.  
 Bingham Plastic parameter of interest yield stress ( $t_{yB_3}$ ) for the specified shear rate range.

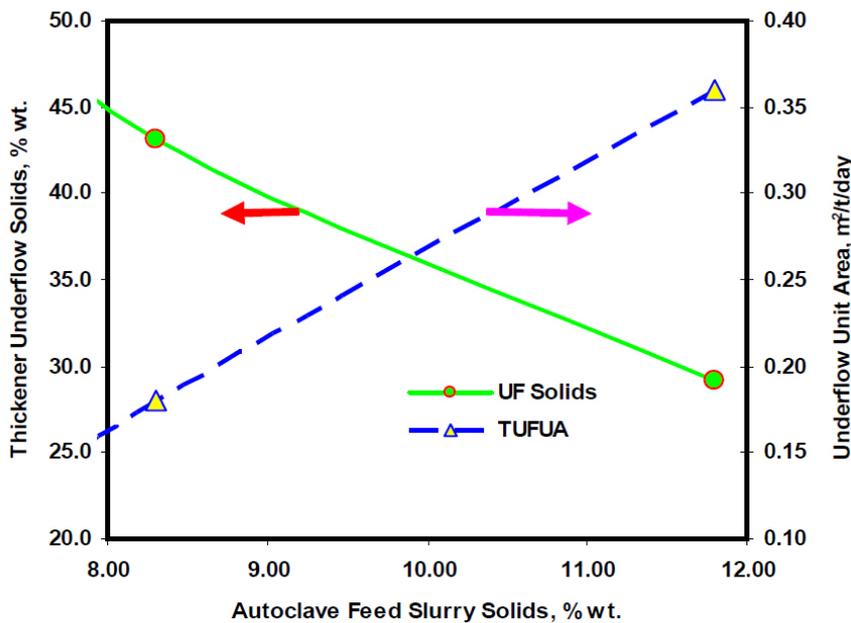


Figure 3 Settling and thickening versus POX conditions

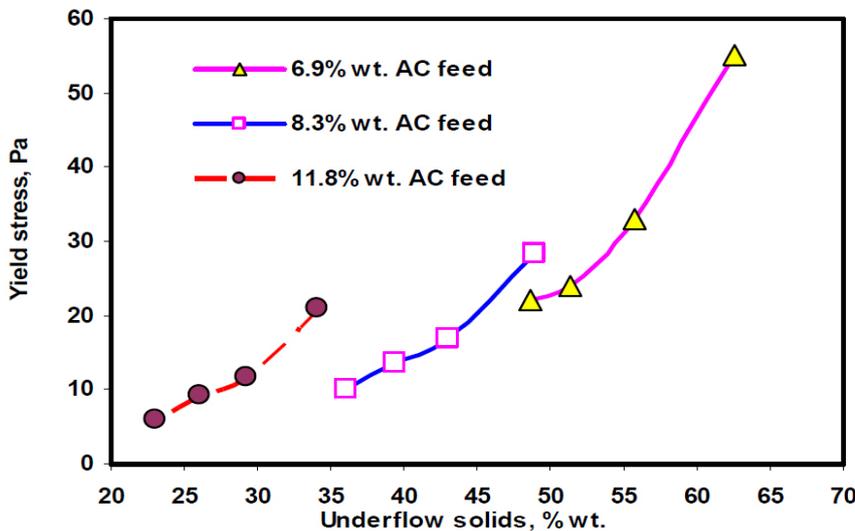


Figure 4 Autoclave POX discharge underflow rheology dependencies

### COMPARATIVE RESULTS OVERVIEW - ALL THREE DISCHARGE OPTIONS

Comparing the liquid-solid separation (settling-thickening-filtration-washing) data (Table 3) on the basis of throughput (i.e. equivalent autoclave discharge) indicated that:

- The specific thickener underflow unit area requirement favoured indirect discharge Version B, needing about 57% less thickener unit area compared to the direct discharge option (i.e. A and A1 cumulative);
- Above performance could be achieved at the cost of 80% increased flocculant consumption in the case of indirect discharge version B;
- Belt vacuum filtration data were comparable, with the main difference consisting of 29% more wash water required by version B compared to the direct version (cumulative A and A1).

Rheology data (Table 4 and Figure 5) confirmed that the best compromise was produced by the indirect discharge version B, producing a free-flowable underflow containing 32% wt. solids.

<b>SAMPLE GENERATION DETAILS BY CONTINUOUS POX RUNS AND SUBSEQUENT NEUTRALIZATION</b>			
Sample/Test Sequence ID	Selected Version A	Version A1	Version B
Discharge type vs. thickener	Direct	Subsequent to A	Indirect
Test Product	As is A/C Discharge	Part-neut PLS	Part-neut A/C Discharge
Nature of solids in separation tests	AC Leach Residue	Gypsum	AC Leach Residue + Gypsum
Solids ASG as tested, g/cm <sup>3</sup>	3.21	1.99	2.09
Discharge PLS pH	0.41	1.85	1.85
<b>STATIC SETTLING-THICKENING DATA SUMMARY (KINCH TESTS - TALMADGE - FINCH REDUCTION)</b>			
Initial Solids, % wt.	6.6	8.1	10.6
Feedwell autodiluted solids, % wt.	3.30	5.1	5.0
Cationic flocculant, g/t dry	164	19	197
Above as discharge AC residue eq'Int	164	19	329
U/F Solids Density, %, wt.	29	24	26
Thickener Underflow Unit Area, m <sup>2</sup> /t/day	0.36	0.46	0.21
Above as discharge AC residue eq'Int	0.36	0.46	0.35
Thickener Hydraulic Unit Area, m <sup>2</sup> /t/day	0.07	0.18	0.03
Initial Settling Rate, m <sup>3</sup> /m <sup>2</sup> .day	120	76	876
<b>VACUUM FILTRATION DATA SUMMARY - BELT FILTER OPTION</b>			
Filter feeds solids, % wt.	28	27.8	17.0
Discharge cake moisture, % wt.	33.2	56.9	50.1
Belt output, dry kg/m <sup>2</sup> /h	1445	6731	2555
Above as discharge AC residue eq'Int	1445	8520	1530
<b>FILTRATION DISPLACEMENT DASH TEST DATA SUMMARY</b>			
Filter/wash feed solids, % wt.	29.0	27.1	16.9
Filter feed PLS Me (=Cu+Ni+Co), g/L	31.0	29.0	31.7
Filter discharge PLS Me, mg/L	19.0	16.3	23.4
Final wash, Me, mg/L	71	80	26
Washed cake moisture, % wt.	32.1	59.0	52.1
Washed residue Me grade, %	0.7	0.03	0.39
Displacement ratio per was, L/L	1	1	1
Required displacements, "NVD"	3.9	3.2	2.7
Wash water required, t/t dry cake	0.449	1.333	1.019
Above as discharge AC residue eq'Int	0.45	1.7	0.61

Table 3 Comparative liquid - solid separation results

Selected direct discharge Versions A and subsequent A1 - corresponding to maximum extractions.

Versions A and A1 are sequential hence sizing data are cumulative.

All samples were tested fresh, i.e. "as produced" to avoid chemical bias.

Testing average temperatures: 87°C for settling/thickening, 51°C for filtration output and 21°C for displacement wash filtration.

PLS - pregnant POX leach solution

ASG - Actual specific gravity of dry solids, calculated from the measured pulp density ("Density")

Feedwell autodilution used to ensure good flocculation.

Displacement Ratio, L/L, i.e. L of water per L of liquor contained in the cake

NVD - Number of Volumic Displacements required to produced >99.5% washing efficiency

All data Reported "As Produced" - No Safety Factors Included.

SELECTED VERSION A DATA		VERSION A1 DATA		VERSION B DATA	
% wt.	$t_{yB1}$ Pa	% wt.	$t_{yB1}$ Pa	% wt.	$t_{yB1}$ Pa
33	21	29	52	32	27
29	12	24	18	27	14
26	9	22	13	25	11
23	6	18	8	23	7
Critical solid density (CSD), % wt. and corresponding Yield stress, Pa					
33-35	>20	28-30	~50	>32	~30

Table 4 Comparative rheology data

Version A - Autoclave discharge underflows having PLS free acidity of ~50 g/L H<sub>2</sub>SO<sub>4</sub>  
 Version A1 - Gypsum underflows at pH ~2  
 Version B - continuous AC-POX leach residue + gypsum blend underflow at pH ~2  
 Variable shearing was produced in the 0 to 600 s<sup>-1</sup> range.  
 Constant shearing was produced at 300 s<sup>-1</sup> for 300 seconds.  
 Bingham Plastic parameter of interest: yield stress ( $t_{yB}$ ) for specified shear rate range.

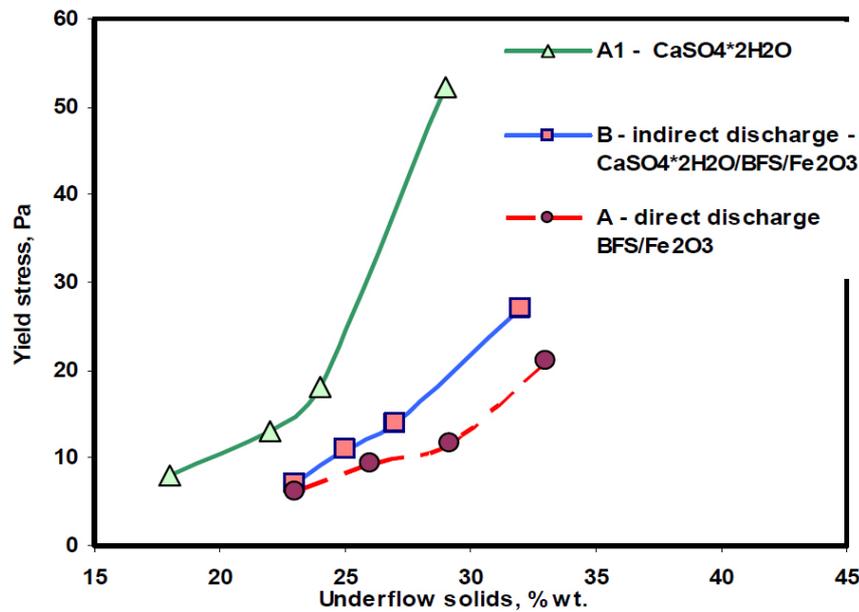


Figure 5 Comparative rheological dependencies

Example Concluding Analysis - pressure oxidation autoclave - operability

The pre-pilot results confirmed that the liquid-solid separation and rheology ("LSR") properties were interrelated and clearly determined by the chemical regime. The chemical regime determined two key underlying factors that contributed to the LSR response under the tested conditions:

- The properties of the solids produced, characterized by their BFS/hematite gangue ratio in case of the direct

discharge slurry, and by the BFS/hem tite/gypsum/gangue ratio in the indirect discharge versions;

- The properties of the solution produced, characterized by significant changes of their ionic strength. The metal ions dissolved do not only consist of pay metals, but also elements such as magnesium, aluminium, calcium, etc., whose presence in solution increase its density and particle surface-chemistry, and therefore, present the potential of negatively affecting the liquid - solid separation behaviour;
- The above two phenomena acted in

the same direction, which in this case translated into an unusually high sensitivity of the practically operating range to the process parameters (i.e. chemical conditions). This example illustrated an extreme situation, even within the complex landscape of today's hydrometallurgical processes.

**IMPLICATIONS:**

- Increased acidity allowed for good metal extractions at the expense of plant operability. The optimum chemical response did not translate into

optimum physical response. The main implication of this was the dramatic increase of the thickener specific unit area required to separate directly the autoclave discharge slurry into its liquid and solids components (treatment "Option A");

- Furthermore, the rate of filtration was found to be inversely proportional to the total ionic strength;
- While comprehensive, the conclusions of the pre-pilot testwork also revealed a rather narrow range of practical operating conditions because of the relatively low underflow solids densities produced, and their tendency to cease flowing;
- In spite of expected higher estimated operating costs, the indirect discharge Option B appeared to provide the advantage of relative insensitivity to the POX conditions. Therefore, it allowed comfortably managing the potentially large fluctuations in the discharge slurry properties. Hence it offers the best metallurgical risk to reward ratio;
- Comparatively, Option A direct discharge is less robust. For example, an accidental increase of feed sulphur grade at constant feed solids density would increase the discharge free acidity. This in turn would render the resulting underflow "too thin", hence leading to decreased washing efficiency and increased wash water demand. Conversely, a decrease of the feed sulphur grade would lead to potentially "un-pumpable", "thick" underflows, quickly causing the shutdown of the thickener and thus of the entire operation;
- A possible limitation of Version B discharge refers to certain gold applications, with concerns around lime consumption. In these cases, a variation of Version A called "hot curing" is sometimes preferred - example is provided further below;
- Version B is not applicable in case of direct leTach processes that contain gold and platinum metal (PGM) complexes, such as the Platsol™ process. This is because these complexes display increased instability with the decrease in the hydrogen ion concentration (i.e. free acidity). Under

these conditions, the central cation (Au/Pt/Pd) is reduced by the unreacted sulphide contained by the residue;

- The settling thickening data allow for the design of the counter-current decantation (CCD) wash circuits in case of all three versions discussed above. This fact is quite important in practice, as CCD circuits are widely used in most SGS Minerals Services pilot plants.

Several SGS Minerals Services pilot plants have been operated successfully under all three versions tested above. In addition, innovative solutions and modifications were proposed, investigated and applied when this was deemed necessary, as detailed below.

### EXAMPLE - OPTION B DOWNSTREAM PROCESSING OPTIONS, COMPLICATIONS AND IMPLICATIONS

This example provides an analysis and discussion of the second category of overlooked factors, namely downstream integration and the effect of solution recycle. The discussion is based on the conceptual generic flowsheet depicted in Figure 6. The flowsheet includes the continuous POX autoclaving discussed in the previous section. Quite generic, the process involves continuous pressure oxidation and partial neutralization (Option B) coupled with a 6 stage counter-current wash-decantation (CCD). The CCD underflow is directed to the gold plant, whilst the overflow goes to the copper solvent extraction and electrowinning (CuSXEW) followed by nickel-cobalt mixed hydroxide precipitation.

Typical issues regarding this generic flowsheet include:

- Optimising Cu tenor in the CuSX feed - this involves targeting a copper level that allows maximising SX efficiency whilst producing a reasonable level of acidity in the raffinate;
- Optimising Ni-Co tenor in the MHP circuit feed means increasing the Ni and Co tenors by recycling some of the iron removal thickener overflow discharge to the autoclave. The main limitation is due to the density of the resulting solution, with direct

implication on the settling- thickening-rheology behaviour in the CCD and iron removal circuits. These consequences will be detectable only during the pilot plant operation, particularly in steady state. Use of a synthetic start-up recycle solution may assist reaching early quasi-steady state operations sooner;

- Maximising the ferric to ferrous ratio in the autoclave discharge, so as to maximise the iron removed in the iron removal circuit, because of the relatively slow rate of ferrous to ferric oxidation in the iron removal stage compared to the autoclave. On the other hand, increasing the amount of ferric in the autoclave would increase the POX discharge acidity and it is usually undesirable to recycle too much ferric (and acidity) to the autoclave. Therefore, this option could be exercised only within the limits allowed by the BFS/goethite and LSR considerations detailed in the previous paragraph;
- A compromise solution would be splitting the Cu SX raffinate between the autoclave and the iron removal circuit at a well-determined ratio. The first issue arising under this scenario is that the acidity of the Cu SX raffinate returned to the autoclave needs to be at least partly neutralised. This is needed to avoid unwanted extra "ready acid" being fed into the autoclave. This neutralisation stage generates gypsum, hence requiring a thickener and a subsequent washing stage to avoid copper loss. Accordingly, the liquid-solid separation and rheology issues need to be re-addressed, especially given the fact that, as outlined in the previous section, the gypsum slurries generally tend to display diametrically opposed properties compared to the autoclave discharge slurries. At the same time, it is important to recognise that the iron contained by the raffinate would increase the acidity in the pressure oxidation autoclave. Overall, it must be ensured that recycling the copper raffinate to the autoclave, to build up nickel and copper tenor does not create more headaches (i.e. costs) than it solves.
- The overall neutralization agent (i.e. limestone in this case) requirement remains constant regardless of the point in the circuit where the iron is

being removed, since any iron recycled to the autoclave will generate acidity, which will have to be neutralised in the partial neutralization prior to the Cu SX-EW. This is the metallurgical interpretation of the term of "no free lunch".

Establishing the optimum split of the acid-bearing streams (as actual H<sub>2</sub>SO<sub>4</sub> and as iron) requires an involved metallurgical analysis. Practical experience suggests that sensible integration of downstream unit operations in conjunction with stream recycles, as defined above, requires pre-pilot test data to determine the best "split" in each stream. This allows for proper sizing of the equipment. Additional decision-support information can be produced by metallurgical modelling and simulation, in conjunction with the test data.

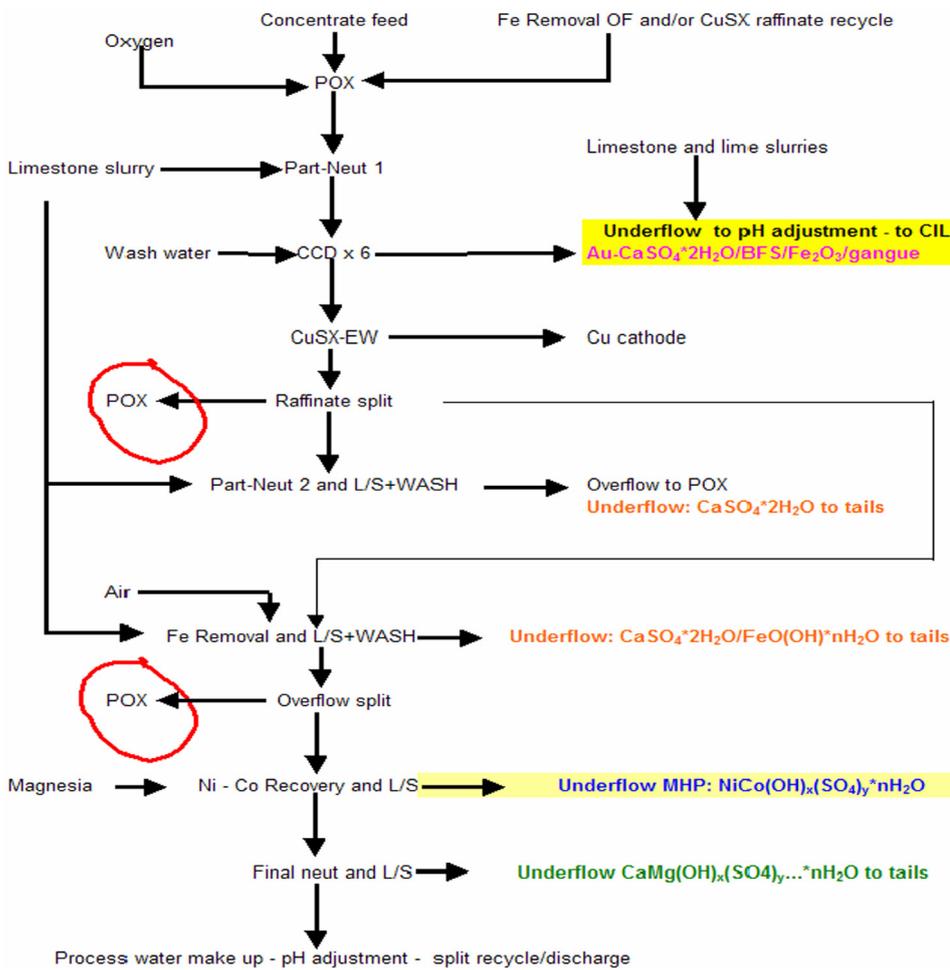


Figure 6 Example of generic conceptual flowsheet

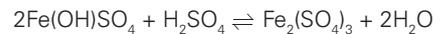
**EXAMPLES OF ALTERNATIVE ROUTES - HOT CURING, CCD, CCL**

This group of three examples discusses briefly the alternatives that are available for inclusion in any flowsheet, including the one discussed in the previous section, to address specific issues. The aim of this section is threefold:

- a.) Exemplify the extent to which a pilot flowsheet can be changed to meet certain requirements;
- b.) Flag the limitations of the changes;
- c.) Highlight what is involved in order to implement the changes.

**SCENARIO 1 - HOT CURING OF THE POX DISCHARGE OPTION A**

Hot curing is a variation of Option A which allows for the decomposition of the basic iron sulphate (BFS) by its reaction with sulphuric acid at 90°C and atmospheric pressure. The reaction equation is depicted below:



Practically, this reaction is the reverse of the BFS formation reaction, which takes place at 200°C and under oxygen pressure. The hot curing reaction is reasonably fast (several hours) at 90°C, and consumes acid while bringing iron into solution as ferric sulphate. Hot curing is considered sometimes as an option in the case of certain gold and occasionally, PGM projects.

**ADVANTAGES OF THIS ROUTE INCLUDE:**

- The sulphuric acid would be converted into ferric sulphate;
- The weight of the residue would decrease thus increasing the gold grade in the CIL feed, compared to Option B;
- The underflow would display higher solids densities (due to less BFS), requiring fewer CCD stages and less wash water;
- The main advantage of hot curing is for applications in which the autoclave solids are processed for gold recovery. If the solids contain BFS when proceeding to a gold cyanidation circuit, all of the acid in the solids (as

BFS) must be consumed by lime (which is expensive) before the pH of the slurry will stabilize >pH 10. After hot curing and CCD however, when the BFS has been converted to ferric sulphate, the acidity associated with the iron compounds can all be neutralized with limestone which is usually at least 10 times cheaper than lime.

#### POSSIBLE DISADVANTAGES INCLUDE:

- An additional circuit needed;
- Heating and mixing costs;
- Need for closed tanks to contain/limit evaporation;
- Silver recovery usually decreases, due to stable silver jarosite formation during hot curing

Analogous to considerations discussed in the previous section, the decision would require preparatory metallurgical testwork as part of the pre-pilot program, because the hot curing feed sample must be fresh. Conversely, sudden implementation could negatively affect the process. Past pilot plants produced good results when employing hot curing.

### SCENARIO 2 - VERSION B RECYCLING OF IRON PRECIPITATES UNDERFLOW INTO THE CCD TO RECOVER CO-PRECIPITATED BASE METALS

Regardless of how efficiently the iron removal circuit is operated, some nickel and cobalt losses occur. The origin of the losses could be chemical (co-precipitation), physical (entrapment) or both.

Physical entrapment can be reduced by washing, whereas co-precipitation losses are more difficult to deal with. Re-leaching of the Ni-Co basic hydroxide is an option, but it requires lowering the pH. Within certain limits, the goethitic precipitate appears to be relative stable, hence opening the theoretical opportunity of recycling the underflow to the acidic CCD circuit to redissolve co-precipitated nickel and cobalt. Practice shows however that this approach may not be the best option, and therefore, it needs careful evaluation.

#### PERCEIVED ADVANTAGES OF THIS APPROACH INCLUDE:

- Plenty of wash water is available to "rinse out" the Ni-Co values if the iron precipitate is recycled into an advanced CCD stage, i.e. >2;
- Capital cost savings;
- Reduced Ni-Co losses.

#### DISADVANTAGES AND POTENTIAL RISKS INCLUDE:

- Possible re-leaching of some of the iron;
- Increased "hold-up" of the iron product in the circuit;
- Potentially adverse physical behaviour (settling, underflow densities, rheology) of the blend of underflow, containing practically all compounds relevant to the flowsheet: basic iron sulphate, gypsum, goethite, hematite and gangue.

According to the authors' previous experience, the settling - thickening - rheology behaviour of the "blended underflow" is unpredictable at best, in spite of the fact that, individually, each of the streams blended may display good operability on their own. The explanation relates to the complexity of the CCD operation, requiring the following:

- Solids mass balance between each pair of successive stages;
- Overall mass balance across all stages;
- Adequate wash water flowrate to ensure >99% wash efficacy;
- Closed volume flowrate, i.e. adequate design and piping to prevent spillage;
- Proper flocculation regime, which could be different for each particular stage, depending on the nature of the solids and ionic strength of the wash water.

Since pilot plants operate with relatively low flow rates, their measurement is limited by the industrial instrumentation available. In addition, a key parameter that must be measured in conjunction with the underflow flowrates is their solids density. The practicalities involved in measuring multiple (i.e. six) sets of parameters generally lead to unwanted sophistication of this unit operation in a pilot plant. This is because the CCD circuit should ideally be controlled as much as possible in real time to avoid complication

arising from losing control over the solids mass balance. Typical CCD problems, remedies and sequence of implications are exemplified below:

- Improper flocculation in one stage can rapidly produce a "thin underflow," in another stage which in turn causes turbid overflow;
- Trouble shooting the above by adjusting the autodilution allows for recovery of stage X, however stages x+1 and x-1 need also to be recovered, as they received thin underflow and turbid overflow during the stage x upset and repair;
- Underflow bed height can be maintained constant under sufficient rake rotation to maintain the underflow in flowing state. Being a Bingham fluid, its undershearing will cause plugging, whilst overshearing will favour channelling ("rat holes");
- Any disruption in one stage will quickly reverberate through the entire circuit via the previous and next stages. Hence, the CCD circuit requires constant attention and real-time control;
- Clarity generally is required in the first CCD stage. However, if it is not achieved, clarifiers are needed, and in conjunction with a polish (cartridge) filter. Stages 2 and higher do not pose

In the light of the above, it is easy to explain why bringing a second feed into a pilot plant CCD circuit will most likely compromise its operability. This conclusion is consistent with SGS Minerals Services experience.

SCENARIO 3 - counter current leach (CCL) for pre-neutralization of discharge Options A/A1 CCL is a well established commercial unit operation which can be used as a stand-alone operation or in conjunction with counter-current decantation washing. It generally requires the availability of an oxidized feed stock or reasonably clean flotation tailings containing acid neutralising gangue minerals (limestone, magnesite etc.) and some pay metals that should be recovered.

**ADVANTAGES:**

- Significantly reduced acid consumption;
- Use of the neutralized acid for leaching of pay metals;
- Reducing operating cost compared to direct discharge Version A due to renouncing to limestone;
- Possible lower weight gain during neutralization (if the neutralising mineral is magnesite);
- Smaller tailings pond requirement;
- Reduced environmental discharge in the gaseous phase;
- Avoiding the deleterious effects of gypsum formed in the equipment (mineral);
- Capital expenditure similar or comparable to direct discharge Version A;
- Lower operating expenditure.

**DISADVANTAGES:**

- Scarce availability of suitable oxidised feed;
- More sophisticated operation required to controlling the CCL compared to the co-current limestone pre-neutralization;
- Specific thickener unit area requirements must be comparable to the direct discharge Version A.

The many advantages of the counter-current leaching justify its inclusion when oxidized feed is available, such as in the case of many tropical ores, including laterites.

**VENDOR TESTWORK**

It is important to point out that the results produced during the short run and associated bench test sequences do not preclude the need and opportunity for specialized testwork to be carried out by equipment vendors. They include suppliers of liquid-solid equipment, pumps, instrumentation and control, etc. The pre-pilot testwork creates the premises necessary to design and operate the pilot plant. This allows for the pilot plant to be operated under optimum conditions, which in turn secures representative vendor samples. The vendors should be called in only at this stage of the pilot plant. The vendors' role is to produce specific data under robust operating conditions. These data allow for the most cost

efficient equipment design. It is quite common that poorly prepared pilot plants face insurmountable liquid-solid separation problems, and the vendors are called to resolve the problem. The vendors will generally help provide a solution during panic, so they are quite welcome to relieve the pressure. The downside however is that the solution provided does not reflect the optimum operating conditions of the pilot plant. The net result in most instances is excessive flocculant addition and oversized thickeners. The implication is increased metallurgical risk due to technical and economic implications. The metallurgical implications are that the parameters under which the sizing data were produced were far from being optimised. The subsequent economic implications are that the project runs out of both money and "real estate," due to the cost and size of the thickeners. To conclude, vendors are important and they should be called in when the operation is running as desired, so fresh, representative samples can be provided for adequate equipment sizing testwork.

**MANAGING**

Complex hydrometallurgical pilot plant management has come a long way at SGS during the past few years. This trend was in response to the realities of the modern pilot plants, as outlined in this paper so far. The SGS pilot plant management pods are illustrated in Figure 7.



Figure 7 SGS overall pilot plant management

Recent improvements in pilot plant management effectiveness included:

- Streamlined project budgeting - the cost of any testwork program can be estimated rapidly based on individual cost modules. This feature also allows for changes in scope to be accommodated within a day. The entire costing process has also been standardized to allow for consistent quality review;
- Focus on pilot plant planning, pre-pilot testwork and flowsheet development;
- Wealth of high level technical expertise, covering all specific areas required for pilot plant design and operation;
- Diverse workforce recruited during the last two decades from about 30 countries - mainly individuals with significant international project exposure;

- SGS Global Metallurgy initiative ensures that additional resources are made available when needed for the Canada, Perth, Santiago and Johannesburg operations;
- IT support has been centralized. Wireless communication devices have been installed in pilot plant locations, allowing for instant data capture, even where the automated data control system (DCS) is not “wired in”;
- The LIMS data management system has been updated with an enhanced analytical data collection system;
- Pilot plant construction is supported by a permanent maintenance crew, as well as by a pool of contractors. About 80% of the construction work is carried out by the technicians and technologists who operate the plants.

NEWLY ESTABLISHED OR RECONSTRUCTED STATE OF THE ART FACILITIES AVAILABLE INCLUDE:

- Refurbished continuous autoclave;
- Several CCD circuits, thickeners, filter;
- Pressure oxidation laboratory;
- Liquid-solid-separation laboratory;
- Solvent extraction and electrowinning circuits of various sizes.

The planning, preparation and construction of the pilot plant is generally scheduled in a sequence as detailed in Table 5.

**TABLE 5 PLANNING, PREPARATION AND CONSTRUCTION**

1. Transfer Metsim data/test data
2. Determine pilot plant flow rates
3. Verify mass balance
4. Define: Residence time, Tank size and numbers, Surge volume, Cycle time
5. Produce detailed unit operations sequence
6. Select “off the shelf” equipment - tanks, pumps, sensors, peripherals..
7. Define control requirements
8. Selection of adequate flowrate measurement devices for gases and liquids
9. Design project - specific equipment - tanks, thickness/ccd, “exotics”, etc.
10. Define reagent requirements and order, include gas reagents
11. Produce floor plan - request client review if necessary
12. Final review before construction - include client input
13. Construction
14. Attach instrumentation and control
15. Define operating data logging requirements
16. Design sampling schedule, include sampling types
17. Design material management sheets
18. Define IT requirement
19. Produce first draft of pilot plants operations plans and submit to client for approval
20. General review - include client input
21. Define Health and Safety requirements, including HAZOP
22. Engineers training session
23. Key personnel training session
24. All personnel training session and HAZOP

## CONCLUSIONS

- Pilot plants can make a huge difference in preventing metallurgical failure, provided they are prepared (planned, designed, constructed), operated and managed properly;
- Key to success is detailed flowsheet design based on pre-pilot testwork, which combines continuous operating sequences with bench scale testwork on freshly produced samples;
- Flowsheet options analysis and selection should be made with careful consideration to all aspects relating to the effect of process chemistry and plant operability;
- Emphasis should be placed on liquid solid separation and rheological properties of all streams, as well as on the effect of downstream integration and recycle streams;
- Effective pilot plant management involves the availability of a complex and sophisticated infrastructure, consisting of expertise, facilities and a trained workforce.

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