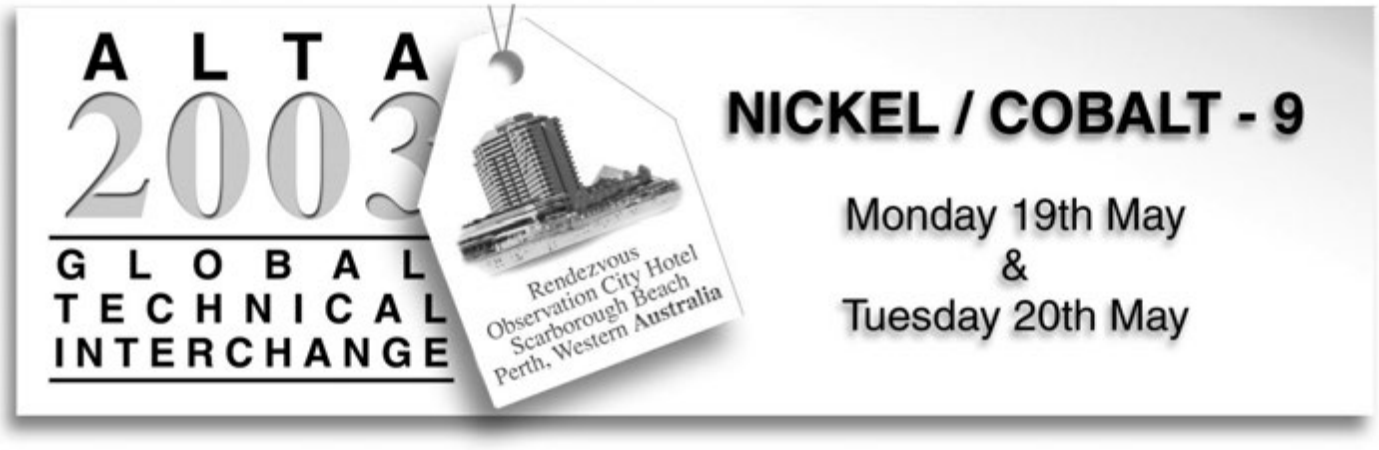
Welcome to



Welcome to another great ALTA Nikel/Cobalt Conference. Following its success and popularity at ALTA 2002, we are again running the event together with our annual Copper Conference. However this time we are including two mid-week one day events, the SX/IX World Summit and the Process Plant Materials of Construction Forum. These are being held in parallel and provide for the varying interests of the delegates attending both the Nickel/Cobalt and Copper Conferences.

We are delighted to have Ivor Kirman, President of the Nickel Development Institute as our Key Note Speaker at the Conference Dinner. He is in a strategic position to provide a keen insight into the overall nickel scene.

We are also pleased to welcome Rod Evans to open the conference. Rod is Executive Director Investment Attraction, for the Department of Industry & Resources, Western Australia.

Once again, we have a highly topical international program of papers, originating from Russia, Finland, Brazil, South Africa, Botswana, the Netherlands, USA, and Canada, as well as from Australia. We greatly appreciate our presenters being willing to spend the considerable time and effort required. We also thank our sponsors and exhibitors who play a key role in the conference, many of whom have supported ALTA events over a number of years.

Our post conference tour this year takes us interstate to one of the world's great metallurgical complexes, Western Mining's Olympic Dam copper/uranium operation in South Australia. The tour also includes a visit to South Australia's famous Barossa Valley wine region.

Have a productive and enjoyable conference.

Alan Taylor

Convenor

ALTA - Our Vision

'Serving the global mining and metallurgical industries with technical excellence and proven Integrity'

ALTA Metallurgical Services have convened annual international Nickel/Cobalt and Copper Conferences and Trade Exhibitions for the past nine years. During this time, we have seen these events grow into important international forums, bringing together top industry representatives and technical experts in exciting and innovative conference programs.

These events have provided and continue to provide the global mining and metallurgical community with the opportunity to exchange valuable technical information, meet with key contacts and showcase products and services to strategic industry organizations, facilitating the exchange of ideas and best practice.

Managing Director Alan Taylor established ALTA Metallurgical Services in Melbourne in 1985 and has continued as Principal to the present time. With over 30 years global experience in the mining, metallurgical and chemical industries, his areas of expertise involve consulting, project development, project audits, engineering, operations, start-up and technology development. These services are now provided through International Project Development Services. Prior to ALTA, Alan spent many years in senior positions with major international engineering firms.

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Future events - May, 2004

ALTA 2004 NICKEL/COBALT-10

May 10th & 11th

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or

HEAP LEACH TESTWORK SCALE-UP & DESIGN SYMPOSIUM

May 12th

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May 13th & 14th

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19-20 May 2003

Perth, Australia

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ALTA Metallurgical Services was established by metallurgical consultant **Alan Taylor** in 1985, to serve the worldwide mining, minerals and metallurgical industries.

Conferences: ALTA conferences are established major events on the international metallurgical industry calendar. The event is held annually in Perth, Australia. The event comprises three conferences over five days: Nickel-Cobalt-Copper, Uranium-REE and Gold-Precious Metals.

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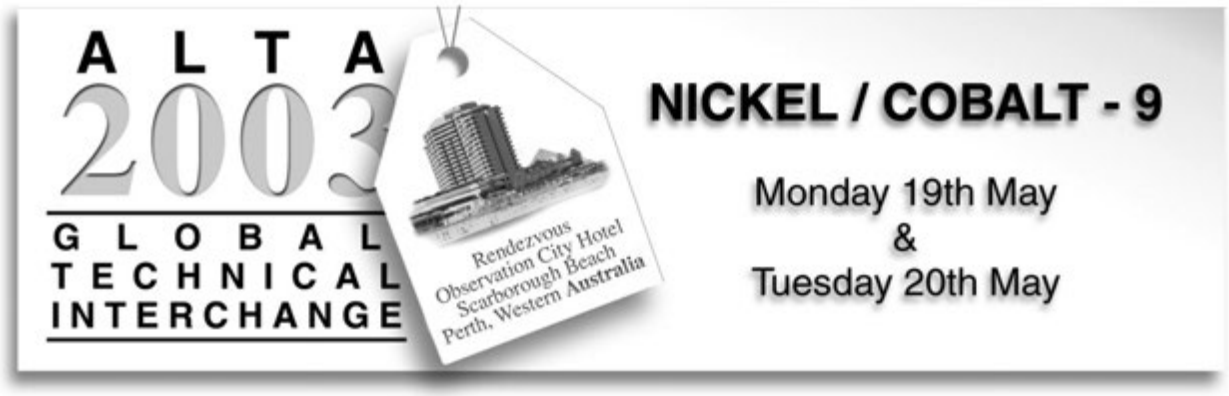

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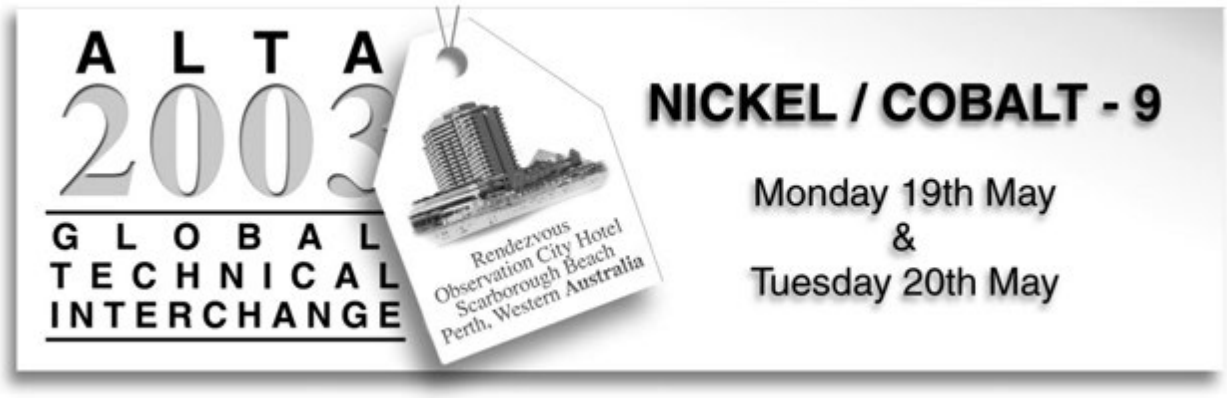

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PRESSURE ACID LEACHING OF NICKEL–COBALT LATERITES: STATUS AND LIKELY DEVELOPMENTS

By

John O'Shea
John O'Shea & Associates Pty Ltd
<john_oshea@bigpond.com>

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Outline of presentation

Pressure acid leaching of nickel–cobalt laterites

- Operating PAL plants (four)
- Committed PAL projects (two)
- PAL projects at feasibility-study stage (six)

1

Four operating PAL plants

- Moa Bay
- Bulong
- Cawse
- Murrin Murrin

2

Moa Bay: the 1950s pioneer

Very large, relatively high-grade resource

Fully decoupled flowsheet (mixed sulphide sent to refinery in Louisiana)

Comprehensive piloting

Development of innovative process equipment

Developed by Freeport, a major USA mining and chemicals company

3

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Moa Bay: the first thirty years

- Freeport's commissioning interrupted by the Cuban revolution in 1960
- Commissioning resumed under Cuban management, with assistance from Russia and Eastern European allies
- Ten-year ramp-up to around 20,000 tonnes per year of nickel and cobalt in mixed sulphide

4

Moa Bay: the past decade

- Operations badly affected by the collapse of the Soviet Union (from1991)
- Revitalised by the partnership with Sherritt from 1994
- Mixed-sulphide intermediate now processed at Fort Saskatchewan
- Steady increase in production to a record 33,382 tonnes of nickel and cobalt in mixed sulphide in 2002

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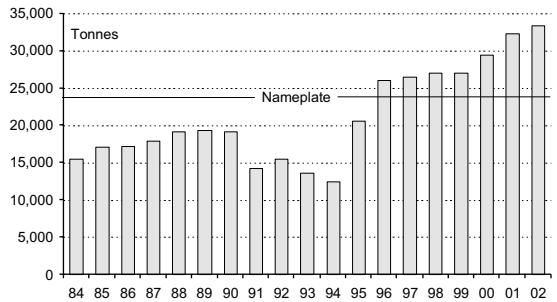
Moa Bay: annual production of nickel and cobalt



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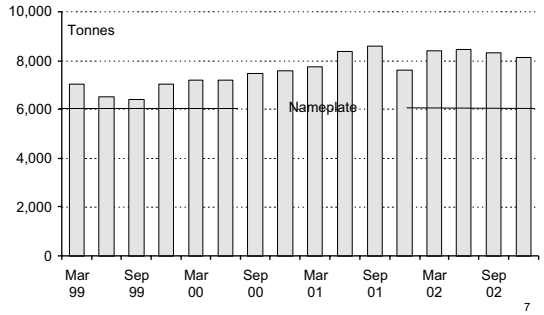
Moa Bay: quarterly production of nickel and cobalt



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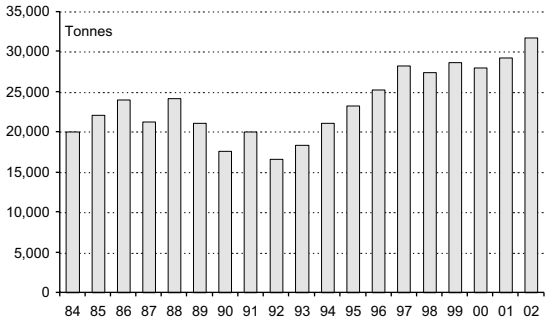
Fort Saskatchewan: annual production of nickel



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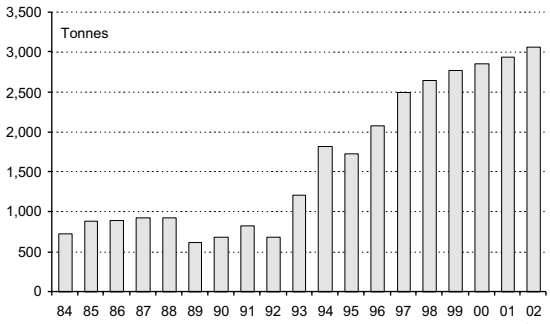
Fort Saskatchewan: annual production of cobalt



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Lessons from Moa Bay, the 1950s pioneer

- The front-end PAL process works, and works well
- It may take a front-end PAL plant many years to reach close to nameplate capacity
- Significant in-process storage increases overall plant availability
- In the long run, resource quality is critical to project success

10

Bulong: distinctive features

- Fully integrated PAL operation (no intermediate product)
- Innovative flowsheet to recover nickel and cobalt from neutralised PAL product liquor
- Modest size (single autoclave train, 537,000 t/y of autoclave feed)
- Relatively low-grade orebody after first few years of operation

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Bulong: the first four years

- Quarterly nickel production reached around 80% of nameplate output within two years of startup
- In years 3 and 4 the plant has struggled to maintain 80% of nameplate output
- Project is yet to achieve consistent positive cashflow
- Project was unable to meet scheduled debt-service payments
- Project now 95% owned by lenders

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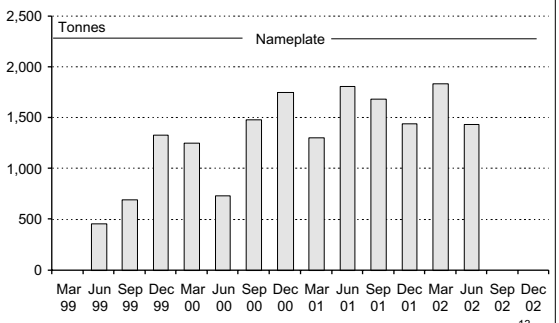
Bulong: quarterly nickel production



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Cawse: distinctive features

Decoupled operation producing nickel–cobalt hydroxide intermediate

Innovative refinery flowsheet (ammonia releach of mixed hydroxide)

Modest size (500,000 t/y of PAL feed)

Low-grade resource; however, limonite ore is upgraded prior to leaching

Cobalt sold as cobalt-sulphide intermediate

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Cawse: the first four years

Quarterly nickel production reached 83% of nameplate output within two years of startup

In years 3 and 4 the plant has struggled to maintain 80% of nameplate output

Project was unable to meet scheduled debt-servicing payment

Sold to OM Group in December 2001

Now sells mixed-carbonate intermediate

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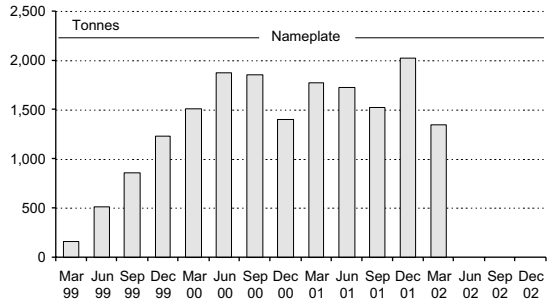
Cawse: quarterly nickel production



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Murrin Murrin: distinctive features

- Decoupled operation producing nickel–cobalt sulphide intermediate
- Front-end PAL flowsheet similar to Moa Bay, back-end refinery flowsheet similar to Harjavalta
- Large size (3,750,000 t/y of autoclave feed)
- Low-grade resource

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Murrin Murrin: the first four years

- Quarterly nickel production reached 65% of nameplate output within three years of startup
- In 2002 (year 4), nickel production was 30,009 tonnes (67% of nameplate)
- In February, Anaconda extinguished A$819 million of debt by paying its noteholders A$188 million
- Future ownership of Anaconda is uncertain

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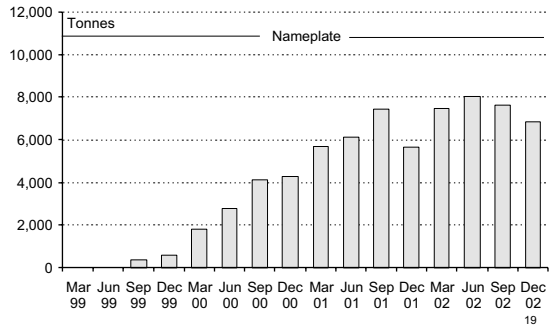
Murrin Murrin: quarterly nickel production



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Lessons from the three WA second-generation PAL plants

- Process chemistry works, but side-reactions cause problems
- Decoupled operation is easier to commission and operate
- Close-coupled plant is more difficult to operate
- Operating costs are much higher than feasibility-study estimates
- All three operations failed financially

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Four operating plants: prognosis

- Moa Bay, Bulong, Cawse and Murrin Murrin should continue to operate
- Bulong's survival requires sustainable fixes to problems in its back-end refinery
- The operating cashflow of all four plants will be significantly impaired by a low cobalt price
- Any operating-cashflow surplus will probably be reinvested in the operation

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Two committed PAL projects

- Goro
  - Located in the far south of New Caledonia
  - Construction underway
  - Ownership yet to be finalised, but likely to be Inco (65%), Japanese consortium (25%) and New Caledonia (10%)
- Rio Tuba
  - Located in the south of Palawan, Philippines
  - Construction not yet underway
  - Ownership is Sumitomo Metal Mining (54%), Mitsui (18%), Nissho Iwai (18%) and Rio Tuba Nickel Mining Corporation (10%)

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Goro: distinctive features

Large, high-grade resource

Large-scale project (54,000 t/y nickel, with potential for brownfield expansion)

Innovative flowsheet includes:

- Direct and indirect heat-exchange
- Nickel and cobalt recovery from treated PAL product liquor by solvent extraction using Cyanex 301
- Recovery of nickel as nickel oxide by pyrohydrolysis of nickel chloride

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Goro: enhancing attractiveness

Inco has enhanced Goro's investment attractiveness by:

- Incorporating novel processes and equipment to lower cash-operating costs
- Implementing a fully integrated flowsheet (no front-end intermediate product)
- Conducting extensive and comprehensive piloting
- Producing semi-finished nickel and cobalt
- Learning lessons from the four operating PAL plants

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Goro: status

Work suspended in December 2002 because of blowout in estimated capital cost to about US$2 billion

Review to ensure at least 9% rate of return is underway, on three fronts; completion expected by July 2003

One team, looking afresh at Goro's flowsheet, is assessing the option of a front-end only plant producing a mixed-sulphide or mixed-hydroxide intermediate for processing elsewhere

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Rio Tuba: distinctive features

Resource is primarily stockpiled low-grade limonite ore mined as overburden at Rio Tuba over many years

Modest scale (10,000 t/y of nickel in mixed sulphide)

Front-end only plant will produce mixed sulphide to be shipped to Japan

Rio Tuba's mixed sulphide will be processed at Sumitomo Metal Mining's Niihama nickel–cobalt refinery

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Rio Tuba: enhancing attractiveness

Sumitomo Metal Mining has enhanced Rio Tuba's investment attractiveness by:

- Processing stockpiled ore
- Incorporating a front-end plant only, producing nickel–cobalt sulphide
- Having an existing nickel-refinery operator as project sponsor, majority equity participant and buyer of Rio Tuba's intermediate product
- Learning lessons from the four operating PAL plants

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Rio Tuba: status

EPCM contract awarded in August 2002 to JGC Corporation, with SNC–Lavalin Australia as JGC Corporation's technical partner

Construction scheduled to be completed in mid-2004

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Two committed projects: prognosis

Goro

- **Goro should ramp up to nameplate output over two years, as scheduled**
- **Goro is likely to (at least) come close to meeting its expected cash-operating cost per pound of nickel in nickel oxide product**
- **Goro's measured return on investment will depend on how much of Inco's pre-commitment expenditure is incorporated in the calculation**

Rio Tuba

- **Appears promising, based on the limited information available**

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Six projects at feasibility-study stage

- **Ravensthorpe (Australia)**
- **Syerston (Australia)**
- **Niquel do Vermelho (Brazil)**
- **Weda Bay (Indonesia)**
- **Gag Island (Indonesia)**
- **Ramu (Papua New Guinea)**

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Ravensthorpe: distinctive features

Located in southern Western Australia

Able to significantly upgrade PAL-autoclave feed

Decoupled operation

- **Ravensthorpe front-end produces mixed-hydroxide intermediate**
- **Expanded Yabulu refinery processes mixed hydroxide**

Annual production to average around 35,000 tonnes of nickel and 1,300 tonnes of cobalt

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Ravensthorpe: enhancing attractiveness

BHP Billiton is enhancing Ravensthorpe's investment attractiveness by:

- **Incorporating a front-end only plant at Ravensthorpe, producing nickel–cobalt hydroxide**
- **Incorporating atmospheric leaching**
- **Expanding its existing refinery at Yabulu to process the mixed-hydroxide intermediate**
- **Conducting extensive and comprehensive piloting**
- **Learning lessons from the four operating PAL plants**

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Syerston: distinctive features

Located in central New South Wales

Large resource, relatively low in nickel and high in cobalt

PAL-autoclave feed grade of 1.0% Ni and 0.28% Co over the first five years

Decoupled operation incorporating production and releaching of mixed nickel–cobalt sulphide

Annual production of up to 20,000 tonnes of nickel and 5,000 tonnes of cobalt

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Syerston: enhancing attractiveness

Black Range Minerals is enhancing Syerston's attractiveness by:

- Maximising the benefits to be gained from existing infrastructure
- Incorporating beneficiation of PAL-autoclave feed
- Developing a mining plan to maximise PAL-autoclave feed grade in the early years of operation
- Seeking offtake agreements for up to 5,000 t/y of cobalt (in intermediate product or in finished products)

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Weda Bay: distinctive features

Located on Halmahera Island, Indonesia

Large resource, not yet fully delineated

Front-end-only PAL operation, producing mixed nickel–cobalt sulphide for sale

OM Group has (until December 2002) funded most of the feasibility-study work

Deep-sea tailings placement

Annual production of 60,000 tonnes of nickel and 5,000 tonnes of nickel in mixed sulphide

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Weda Bay: enhancing attractiveness

Weda Bay Minerals Inc, as majority joint-venture partner and operator, is enhancing Weda Bay's investment attractiveness by:

- Developing a flowsheet that incorporates both saprolite neutralisation and atmospheric-pressure leaching, in addition to high-pressure leaching (PAL)
- Learning lessons from the four operating PAL plants

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Gag Island: distinctive features

Located close to the westernmost point of Irian Jaya, Indonesia

Investigation began in 1969 (by PT Pacific Nikkel Indonesia)

Large, relatively high-grade resource of 240 million tonnes grading 1.35% Ni and 0.08% Co

At present, development is blocked by legislation banning open-pit mining in areas designated as protected forest

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Gag Island: enhancing attractiveness

BHP Billiton, as the majority joint-venture partner and operator, is enhancing Gag Island's investment attractiveness by:

- Developing a flowsheet using atmospheric-pressure leaching in addition to high-pressure leaching (PAL)
- Developing a flowsheet using atmospheric-pressure leaching instead of high-pressure leaching (PAL)
- Investigating a front-end-only project
- Learning lessons from the four operating PAL plants

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Niquel do Vermelho: distinctive features

Located near Carajas, Brazil

Large (250 million tonnes) but low-grade (0.9–1.0% Ni) resource

Ability to beneficiate PAL-autoclave feed to around 1.7% Ni

Low acid consumption and favourable slurry rheology

Annual production of 40,000–45,000 tonnes of nickel and 3,500 tonnes of cobalt

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Niquel do Vermelho: enhancing attractiveness

CVRD, as owner, is enhancing Niquel do Vermelho's investment attractiveness by:

- **Maximising the benefits to be gained from existing infrastructure**
- **Incorporating beneficiation of PAL-autoclave feed**
- **Conducting comprehensive resource-evaluation and metallurgical-testwork programs**
- **Learning lessons from the four operating PAL plants**

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Ramu: distinctive features

Located in inland Papua New Guinea

Large, relatively low-grade (1.0% Ni, 0.10% Co) resource with low acid consumption

135-km-long slurry pipeline to transport ore to a front-end PAL plant and a back-end refinery on the coast

Decoupled flowsheet, with a nickel–cobalt hydroxide intermediate product

Mid-sized (33,000 t/y Ni, 3,200 t/y Co)

Deep-sea tailing placement of residues

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Ramu: enhancing attractiveness

Highlands Pacific Limited, as majority joint-venture partner and operator, has enhanced Ramu's investment attractiveness by:

- **Incorporating a flowsheet similar to the Cawse flowsheet**
- **Minimising operating costs by locating the PAL plant and the refinery at a deep-sea location**
- **Maximising benefits stemming from Ramu's low strip ratio and favourable mineralogy**
- **Carrying out extensive metallurgical testwork**
- **Learning lessons from the four operating PAL plants**

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Six projects at feasibility-study stage: prognosis

It is unlikely that any company or any joint venture will commit to development of a PAL project until Goro has achieved close to nameplate output. Mining companies are becoming increasingly risk-averse.

Most projects require significant revenue from byproduct cobalt to meet a hurdle rate of return on investment. Owners of projects dependent on cobalt revenue are unlikely to commit to developing these projects until there is a sustained recovery in the price of cobalt.

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Six projects at feasibility-study stage: prognosis (cont.)

Many major companies are reluctant to invest in nickel projects because of the historical volatility of the nickel price and the historical inadequate profitability of nickel-producing companies.

The development of Gag Island and Weda Bay is, at present, blocked by Indonesian legislation banning open-pit mining in areas designated as protected forest. It is not at all clear when this block to development will be removed.

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Six projects at feasibility-study stage: prognosis (cont.)

Although Ravensthorpe is by far the most advanced of the six projects considered in this presentation, BHP Billiton might well consider that the potential rewards from developing Ravensthorpe are insufficient to offset the risks currently associated with implementing PAL technology.

If BHP Billiton does not commit to the development of Ravensthorpe, the next committed PAL project might well be a brownfield expansion of Goro.

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Outlook for PAL operations and projects

- All four operating plants should continue to operate, although Bulong may not yet be out of the woods
- Committed projects should have a less-troubled ramp-up towards nameplate capacity
- Projects at feasibility-study stage will continue to struggle to secure commitment and financing
- Goro's ramp-up performance will be critical to determining support for other projects

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Pressure acid leaching of nickel–cobalt laterites

John O'Shea

John O'Shea & Associates
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NI/CO LATERITES: WHERE TO NOW?

By

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ABSTRACT

Laterite deposits grading less than 1.5% Ni contain substantial resources of both Ni and Co and will inevitably be required to satisfy future world demand for primary Ni. To date, however, projects based on such low-grade deposits have generally not been commercially successful and this paper examines some options that could be used to remedy this situation.

Some lean-ores are presently beneficiated by the removal of relatively coarse gangue particles but product grades would be improved still further if micron-scale ore and gangue minerals could be separated. The separation of such fine particles, especially since both ore and gangue comprise oxides and silicates, remains a substantial technical challenge.

For some deposits beneficiation is compromised because of substantial Ni and Co lost to the reject stream. A low-capital, low unit-cost technique to recover this Ni and Co would allow beneficiation to be maximised. The author has identified one such technique that warrants evaluation.

1. INTRODUCTION

For many years laterite ores have supplied some 40% of the world demand for primary Ni and the identified laterite resources are ideally sufficient to sustain present production rates for many years into the future. Given the current rate of growth in primary Ni consumption and the absence of any significant new sulphide-Ni discoveries, expansion of the Ni-laterite industry is therefore inevitable.

Countering this are the limited high-grade laterite resources and the generally poor commercial records to date of projects based on the lower-grade resources; i.e., the goethite and smectite clay-rich resources. Possible solutions to this situation include:

- real rises in the prices of primary Ni and Co,
- more cost-effective methods of Ni and Co extraction than the current Pressure-Acid-Leach (PAL) and Ammonia-Leach processes.

These are substantial requirements but resource characteristics vary and the commercial ranking of some low-grade deposits can be improved by upgrading the leach-feed. However, without an accessory process to recover Ni and Co from the reject fractions, the benefits of such beneficiation would be seriously reduced.

A possible accessory method of Ni and Co extraction has been identified but has yet to be tested. If practicable, this process would materially assist the development, within the current commercial parameters, of a generation of projects based on suitable low-grade deposits. However, resources that cannot be beneficiated or are unsuited to the accessory process may have to remain undeveloped until well into the future.

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2. WORLD LATERITIC Ni RESOURCES

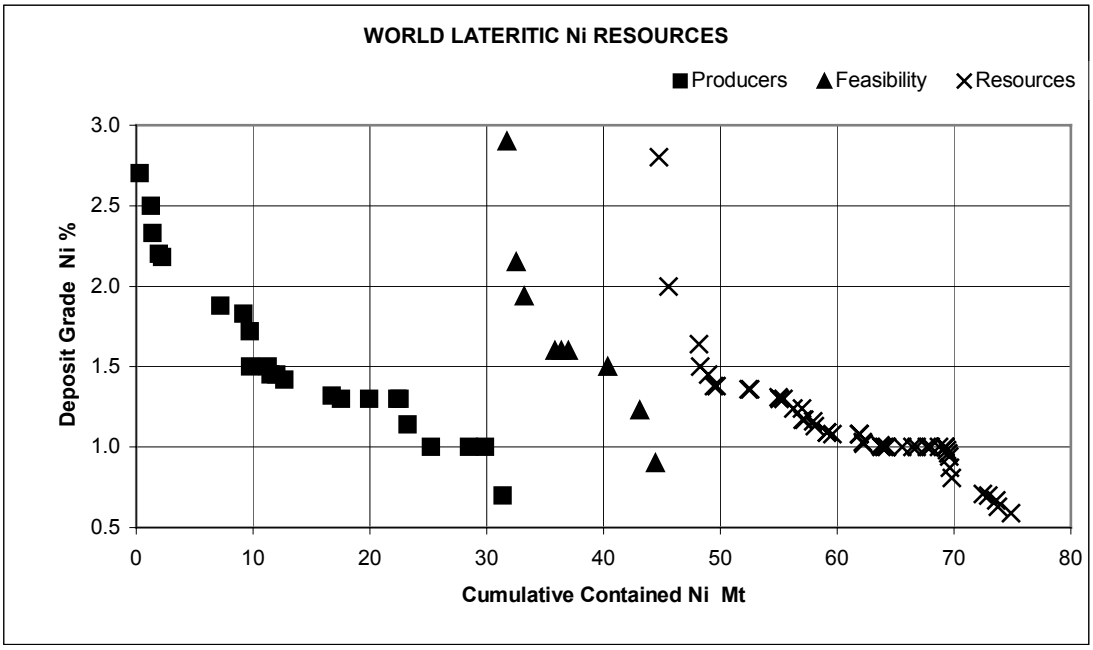

FIGURE 1. Laterite deposits: Ni grades and contained Ni tonnages

Figure 1 shows Ni grades and tonnages of contained Ni for current mines, deposits presently subject to feasibility studies and other identified resources. It is significant that the resource/reserve tables on which Figure 1 is based show that:

- Deposits with grades above 2% Ni contain a total of only some 4.5mt of Ni metal and of this 75.1% is held by operating mines or is subject to feasibility studies.

- Deposit grading 1.5-2.0% Ni host some 14.6 million tonnes of Ni of which 82.2% is contained in the reserves of established mines or deposits subject to feasibility studies.

- Some 47.4 million tonnes of Ni are hosted by deposits grading 1.0-1.5% Ni. This is almost 2.5 times the combined contained Ni resources of all deposits grading above 1.5% Ni, and more than 3 times that of the >1.5% Ni resources currently considered to have commercial potential.

- The <1% Ni deposits contain a modest 8.5 million tonnes of Ni metal. However, as little exploration effort has been directed towards these lean resources the actual resource is probably much higher.

If laterite resources were mined in sequence by diminishing head-grades, at current production rates (approximately 0.45mt/annum) the >2% Ni resources currently considered to have commercial potential would be exhausted within 7 years and the >1.5% Ni deposits some 34 years later. However, the laterite industry continues to expand and it is therefore likely that well before this 41 year theoretical limit the <1.5% Ni resources will be increasingly required to help satisfy world demand.

However, much of the <1.5% Ni resource is at present arguably sub-commercial. Fortunately some low-grade deposits have characteristics that can be used to improve their commercial ranking.

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3. COMMERCIAL FACTORS

Co is not important to the higher-grade (saprolite) ores but can be significant to the economics of lower-grade deposits. Nevertheless, project economics are primarily dependant upon mill-feed Ni grades, which, in some deposits, can be increased by raising the mining cutoff grade or by the beneficiation of as-mined ore.

Increasing the cutoff grade is rarely a valid long-term option because of the geometric relationships between cutoff grade, resource tonnage and resource grades. The resource tonnage may be dramatically reduced and the high-grade shoots can also be comparatively small, irregular and discontinuous. Waste □ ore ratios can be high and the high-grade shoots may be impossible to mine without severe ore loss and dilution penalties.

By contrast, at comparatively low cutoffs the ore horizon can be comparatively thick, continuous and readily mined. Included waste can sometimes be removed by screening and the beneficiated product may be more readily processed than equivalent grade whole ores mined to a higher cutoff. However, substantial proportions of the Ni and Co contained in the as-mined ore can report with the reject stream and be lost: degree to which beneficiation is practicable may be limited by the Law of Diminishing Returns.

4. BENEFICIATION

The Australian dry Ni-laterite ores commonly comprise a mix of coarse gangue particles and a finer ore-grade fraction that in turn is a mix of very fine-grained waste and high-grade particles, respectively. “Macro-beneficiation”, the removal by screening of coarse (approximately >50μ) gangue particles, has been practiced for many years but “micro-beneficiation” (i.e., the separation of micron-scale gangue and ore particles) is an exciting possibility that still awaits commercial development.

4.1 MACRO BENEFICIATION

The coarse gangue fraction includes sand to boulder-sized remnants of bedrock, accretions of chert and chalcedonic silica and also the sand-sized spinel (chromite and magnetite) heavy mineral fractions. The bedrock and siliceous particles can be readily removed by screening and the spinels by gravity separation techniques; e.g., spirals.

4.1.1 Beneficiation by Screening

The potential for loss of Ni and Co to the rejected oversize fraction can limit the degree of beneficiation that is feasible. This is apparent from the relationships between particle size and Ni and Co grades of two deposits. See Figures 2 –5.

In these graphs, to accommodate the broad range of particle sizes, the screen aperture is represented by the phi scale where:

phi = -(logn)/log2, where n = particle size in millimeters

.

For example; -4phi = 16mms, -3phi = 8mms, -2phi = 4mms, -1phi = 2mms,
0phi = 1mm, 1phi = 500μ, 2phi = 250μ, 3phi = 125μ, 4phi = 62.5μ

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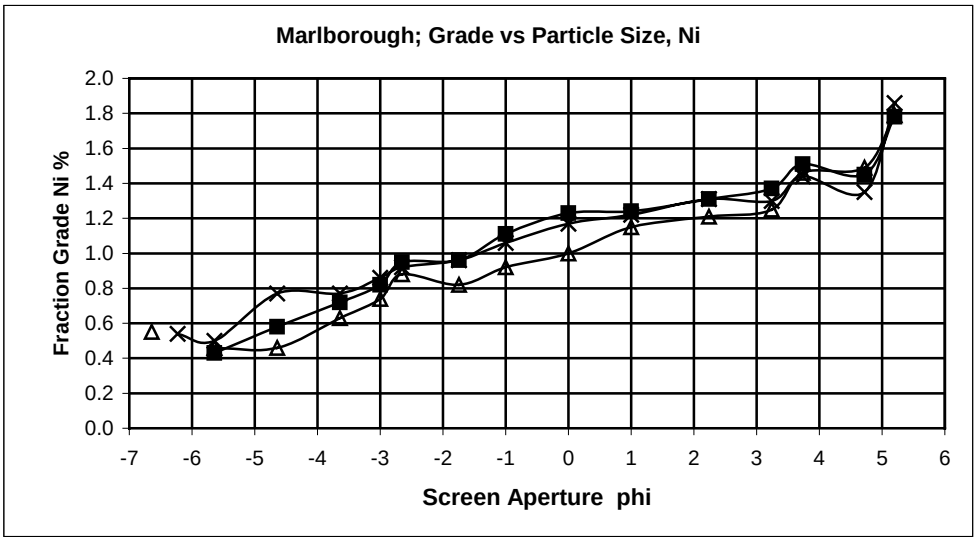

FIGURE 2. Marlborough, Queensland. Particle size vs Ni grade for 3 ore blends.

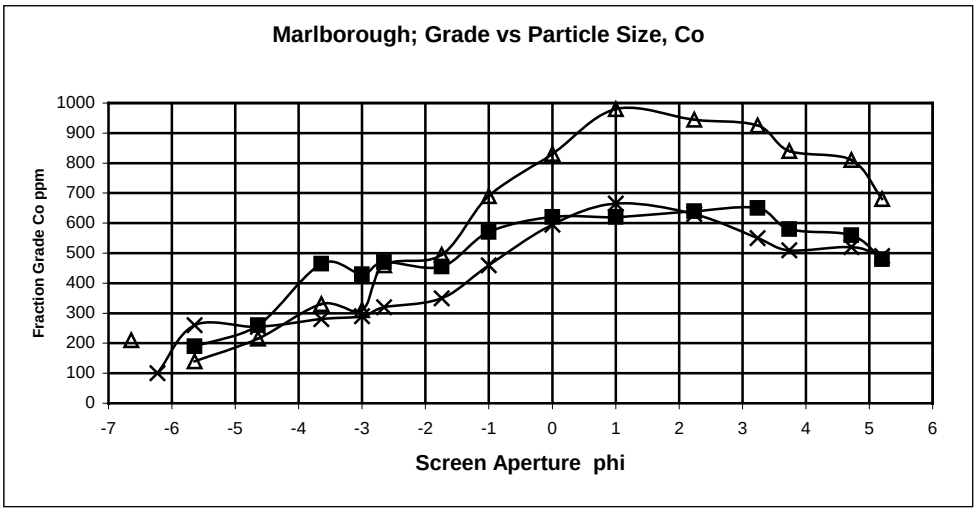

FIGURE 3. Marlborough, Queensland. Particle size vs Co grade for 3 ore blends.

Figures 2 and 3 show the size/grade analyses for 3 bulk samples from the Marlborough, Queensland deposits. Head grades of the samples tested ranged from 1.04-1.25% Ni and 460-550ppm Co. Although the samples contained different proportions of different ore types drawn from different deposits, their size/grade relationships are remarkably similar. Ni grades are highest in the finest fractions while the highest Co grades occur in the intermediate sizes, approximately 100µ - 1mm, (approximately 0-3.5phi). A dilemma is immediately apparent: both Ni and Co grades cannot be maximised in any single screen product.

The potential for Ni and Co losses presents a second dilemma. Screening at 100µ yields a >1.65% Ni undersize product but some 60% of the Ni and 65% of the Co report with the screen oversize. Screening at 1mm yields an undersize product around 1.55% Ni but some 40% of both the Ni and Co still report with the oversize.

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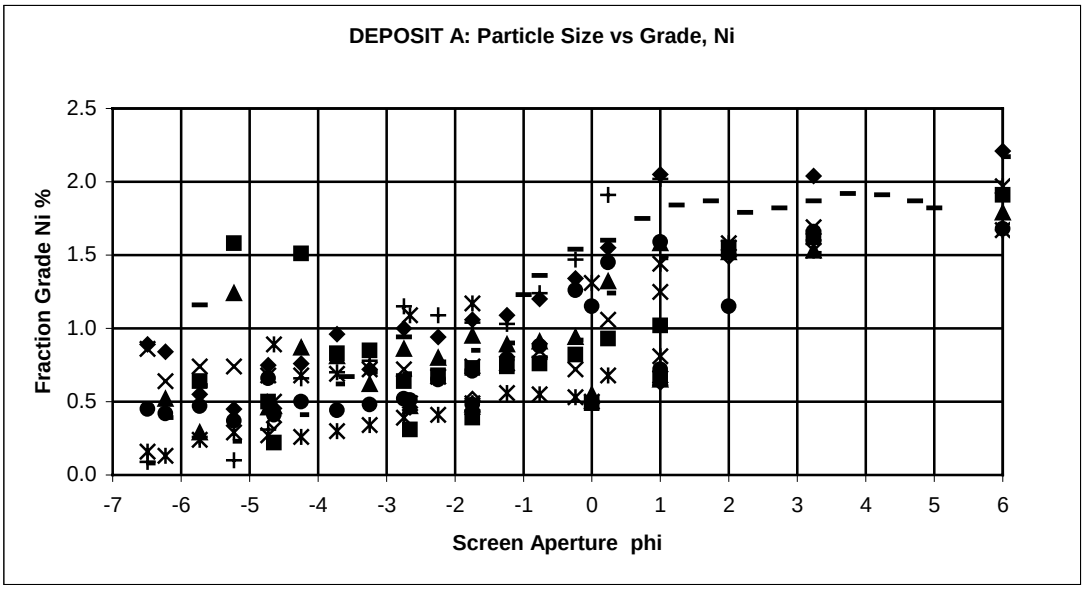

FIGURE 4. Deposit A: particle size vs Ni grade.

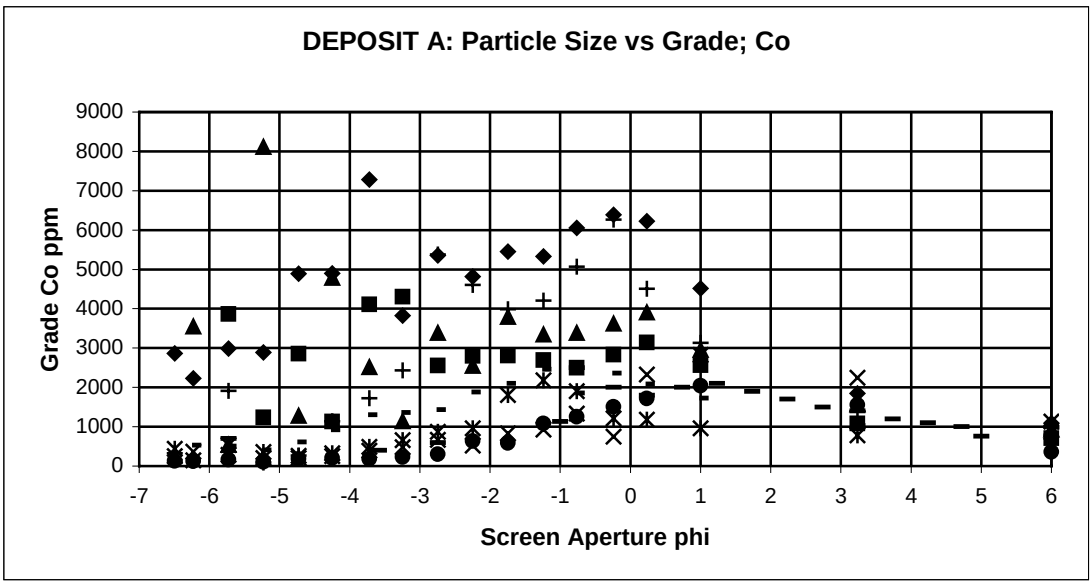

FIGURE 5. Deposit A: particle size vs Co grade.

Figures 4 and 5 also illustrate the dilemmas screening creates for another deposit. Screening 1mm or finer would again yield a Ni-enriched undersize product but losses to the oversize, particularly for Co, would again be very high. The variability in the size **:** grade relationships in Deposit A would also make selection of an optimum screen size difficult. Possible solutions to these dilemmas are discussed in 5 below.

4.1.2 Spinel Separation

The most common ore minerals of the <1.5% Ni laterite ores are:

Ni-rich goethite {Ni,FeO(OH)},
Ni- and Co-rich Mn-oxides {MnO_2},
Ni-nontronite {$Fe,Ni(AlSi)_8O_{20}(OH)_4$,
partially oxidized Ni-serpentine {$(Ni, Mg, Fe)_3Si_2O_5(OH)_4$.

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The individual minerals are commonly finer than 10μ and because of their spongiform, acicular or platy habits, they settle slowly in water. The spinel minerals, chromite {$(Fe,Mg)O.(Fe,Al,Cr)_2O_3$} and magnetite {$(Fe,Mg)Fe_2O_4$}, although only marginally more dense than the ore-grade minerals, are typically equant, coarser than 50μ and settle more readily. Because PAL-feeds are slurried it is relatively easy to remove the spinels from the leach feed by gravity concentration processes such as spirals. Because ammonia-leach feeds are dried, spinel extraction, although feasible, would be less simple.

The spinel minerals contain negligible Ni and Co and in some deposits spinel extraction can increase leach-feed Ni grades 0.05-0.2% Ni. However, in some deposits the spinels are coated with Mn-oxides that have very high Ni and Co grades and heavy mineral extraction can be counter-productive.

4.2 MICRO BENEFICIATION

Even after waste components are effectively removed by screening and gravity separation, the beneficiated product still comprises a mix of ore and gangue particles. Figure 6 shows Ni grades determined by electron microprobe analyses of the ore fractions from 2 substantially different deposits. One deposit could be beneficiated by screening, the other not. The determinations for spinel and Mn-oxide minerals were culled from both populations: the Mn-oxides commonly exceeded 5% Ni while the spinels were <0.2% Ni.

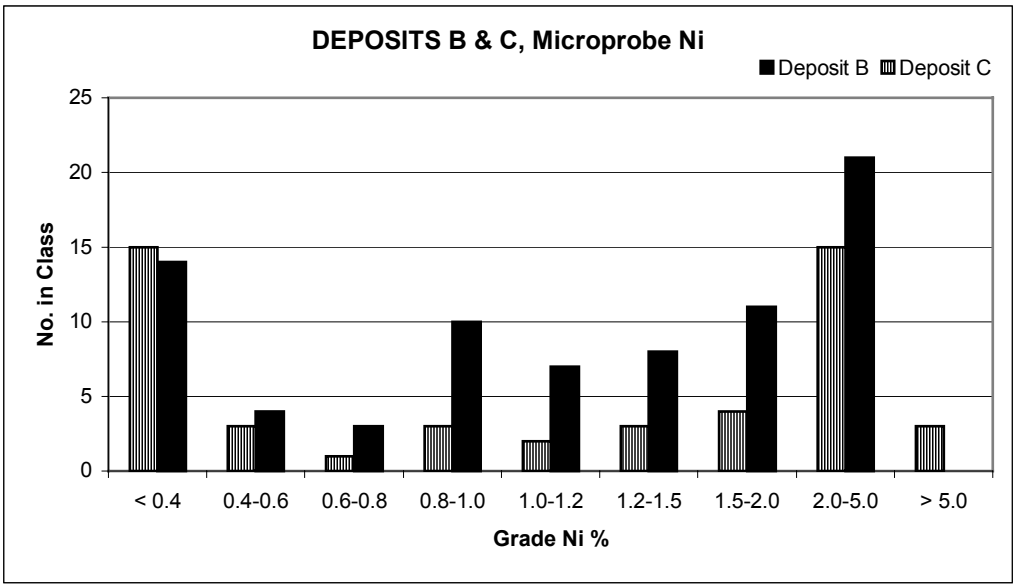

FIGURE 6. Electron microprobe determinations of Ni grades for discrete minerals in 2 deposits.

The Ni distribution of both probe populations is strikingly bimodal. Comparatively few minerals in deposit B grade 0.4-0.8% Ni and comparatively few in Deposit C grade 0.4-2.0% Ni. In both deposits the Co grades of minerals grading <1% Ni were insignificant. If the (Ni/Co)-poor minerals could be removed the resulting beneficiation effects for both these deposits would be very substantial. However, for separation to be feasible the “ore” and “gangue” fractions must be mineralogically distinct and significantly, in both these deposits they are.

Figures 7 and 8 show Fe and MgO grades of the minerals probed for each of these deposits. Interestingly, the Fe vs MgO distributions in Figures 7 and 8 resemble the patterns for whole drill samples from typical laterite deposits. A high proportion of the Ni-poor minerals in both deposits grade above 25% MgO; i.e., near-fresh serpentines (Mg-silicates). In Deposit C (Figure 8) a substantial proportion of the Ni-poor minerals also

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grade above 45% Fe, i.e., haematite, Fe_2O_3. The Ni-poor fractions of both deposits are therefore mostly minerals that are either insufficiently oxidized, or too heavily oxidized, to contain significant Ni. Minerals in the intermediate oxidation states are mostly Ni-rich. Significantly, none of the samples in either of these populations were taken immediately adjacent to the respective upper or lower limits of the possible ore horizons

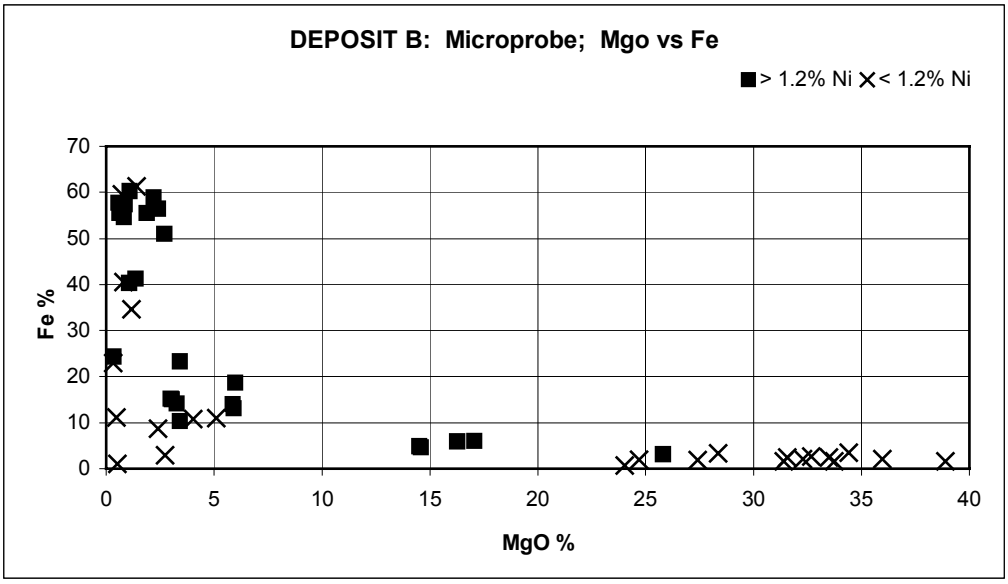

FIGURE 7. Deposit B: electron microprobe determinations of MgO and Fe.

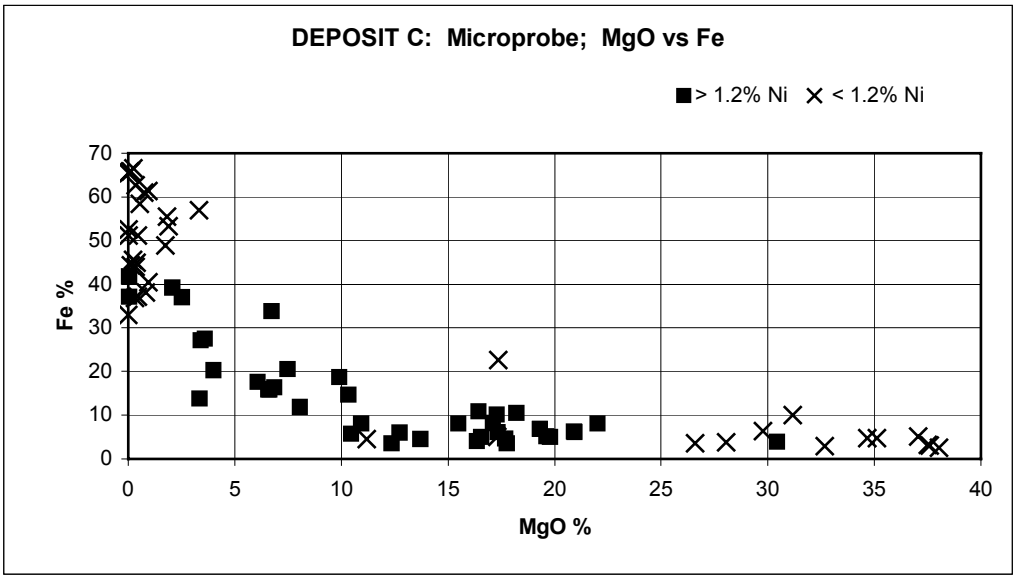

FIGURE 8. Deposit C: electron microprobe determinations of MgO and Fe.

Because there are reasonable differences between the Ni-rich and Ni-poor minerals (Figures 7 and 8) separation may feasible. The challenge is, however, to separate micron-scale oxides and silicates from other micron-scale oxides and silicates. Although industry have been aware of this challenge for at least a quarter of a century, a commercial separation technique has yet to be developed. However, if such a process were possible, the effects could be almost as profound as those of flotation for sulphide ores.

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5. SOLUTION TO THE Ni/Co LOSS DILEMMA

The Ni and Co loss dilemma created by screening and gravity separation would be resolved if an accessory process were available to extract Ni and Co from the reject fractions. Leach-feed grades could be maximized, the capital cost per unit of Ni and Co production minimised and high overall metal recovery rates maintained.

The value of the contained (Ni + Co) of the accessory process feed would be comparatively modest and the process would therefore need to be cost effective. However, unlike typical as-mined laterite ores, the reject fractions would be permeable; and therefore amenable to heap leaching. This immediately opens the field to potentially low-cost, bulk treatment techniques, such as heap leaching.

The author believes he has identified a low unit-cost technique to recover Ni and Co from the reject fractions from some laterite resources. Some laboratory test work has been completed but a joint-venture partner is now required to sponsor the development of the concept to commercial status.

6. CONCLUSION

- The Ni and Co resources of the goethite- and smectite clay-rich laterite deposits grading 1.0-1.5% Ni are substantial and these deposits will increasingly be required to satisfy world demand in the near future.

- To date few projects based on <1.5% Ni deposits have been commercially successful.

- Although some low-grade resources can be beneficiated by screening and spinel extraction, substantial proportions of the Ni and Co resource are commonly lost to the reject stream.

- Beneficiation effects could be maximised if an accessory process were available to recover Ni and Co from the reject fractions.

- The screen oversize fractions would be permeable and therefore amenable to heap-leaching.

- Even after screening and spinel extraction, laterite ores comprise a mix of micron-scale Ni/Co-rich and Ni/Co-poor minerals.

- If these Ni/Co-rich and Ni/Co-poor minerals were separated leach-feed grades would be markedly improved.

- A possible heap-leach technique has been identified but requires development.

- Given an efficient beneficiation and an accessory Ni/Co recovery process a it should be possible for a generation of projects based on <1.5% Ni resources to be competitive under current economic conditions.

REFERENCE

Eckstrand, O.R. and D.J. Good, 2000. World Distribution of Nickel Deposits. Geological Survey of Canada, Open File Report 3791a.

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ACKNOWLEDGEMENTS

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NIQUEL DO VERMELHO PROJECT – METALLURGICAL UPDATE

By

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Presented by

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ABSTRACT

Cia. Vale do Rio Doce's Mineral Development Center is currently developing Niquel do Vermelho Project. The laterite deposit is located at Carajas Mineral Province, near Brazil's major iron ore mine and copper deposits. It can take advantage of the available infrastructure, which includes paved roads, large capacity railroad and port complex owned by CVRD, power supply and good quality water.

The deposit was first discovered in 1974. Early work (1980–1995) focused on smelting saprolite ore to ferronickel, but in the late 1990s, CVRD switched its attention to investigating the possibility of using pressure acid leaching at Niquel do Vermelho. CVRD is now assessing the attractiveness of developing Niquel do Vermelho as a pressure acid leach (PAL) project. CVRD has completed the Niquel do Vermelho scoping study on March 2002, and the project is currently on its prefeasibility phase.

CVRD has designed the prefeasibility study to be thorough, with the aim of reducing risks as well as the time and effort needed for the final feasibility study, scheduled for completion by end-2004. This paper outlines the results of the recently completed pilot testwork, as well as the methodology being applied for project development.

1. INTRODUCTION

Companhia Vale do Rio Doce (CVRD), is carrying out the development of its wholly owned Niquel do Vermelho nickel–cobalt laterite project in southern Para State, northern Brazil. Niquel do Vermelho has excellent existing infrastructure. It is 55 kilometres south-east of CVRD's very large iron-ore mining centre of Carajás; it has excellent transportation links to a major port (Ponta da Madeira); it has easy access to hydro-electricity; and fresh water is readily available. The Carajás region is highly mineralised; in April 2002, CVRD began construction of the Mineração Serra do Sossego copper–gold project some 15 kilometres from Niquel do Vermelho.

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Project location and infrastructure of the deposit is shown in Figure 1.

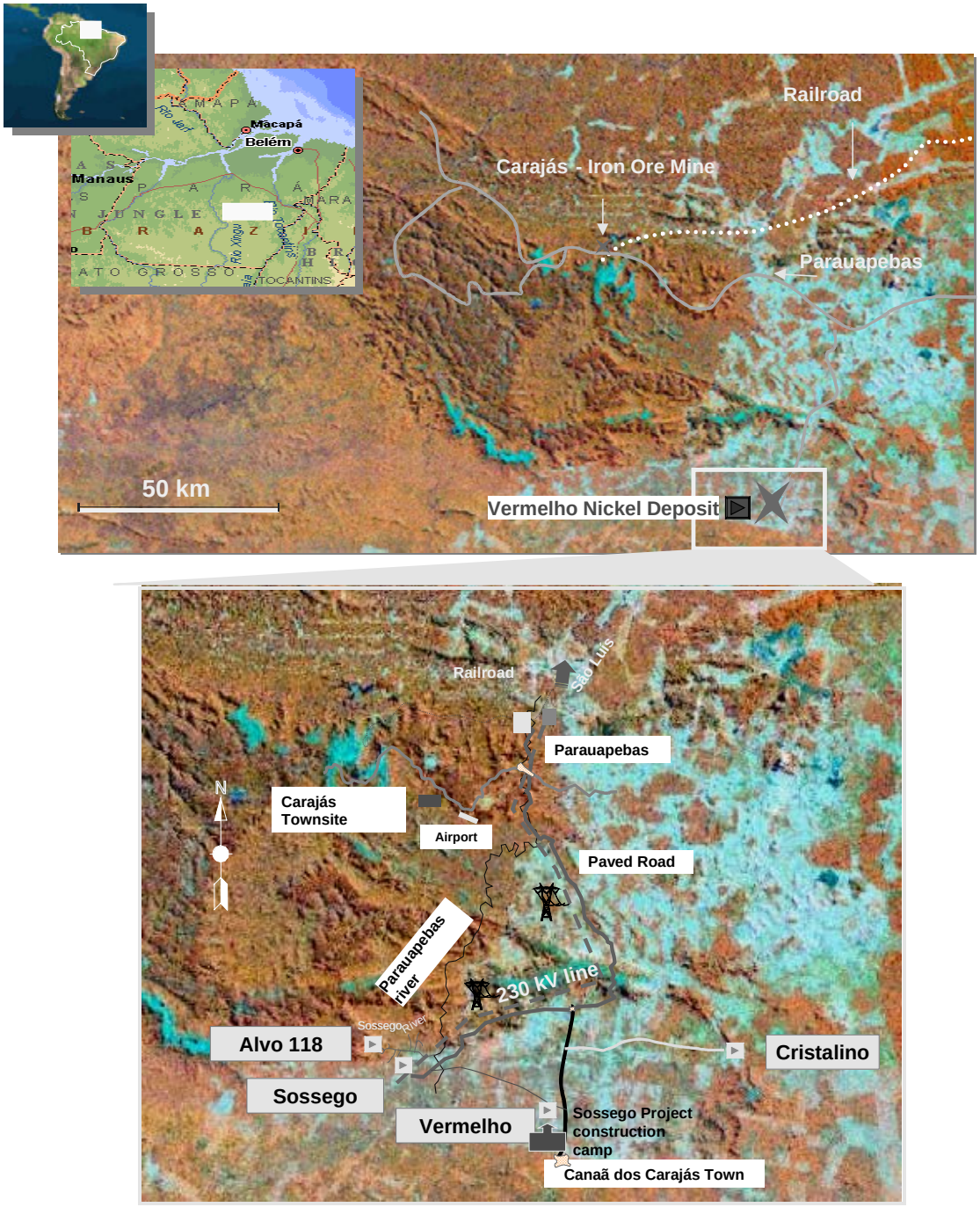

Figure 1 – Location and Infrastructure of Niquel do Vermelho Project

The Niquel do Vermelho nickel–cobalt laterite resource is estimated to contain around 270 million tonnes grading around 0.9–1.0% Ni (no cut-off), as shown on Table 1; it has a nickel-to-cobalt ratio of around 12. The resource occurs in two low hills (V1 and V2), that contain three zones of potentially economic mineralisation:

- a high-grade saprolite zone (garnierite) ;
- a siliceous-saprolite zone grading (limonite + quartz) ;
- a ferruginous-saprolite zone grading (iron-rich limonite) .

The saprolite zone (around 15% of the resource) is the only mineralisation suited to smelting to ferronickel; however, material from all three zones of the resource are

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considered to PAL processing. Geology details have been reported previously in more detail (Ribeiro et alli, Alta 2001). Figure 2 shows an overview of the deposit.

Figure 2 – Deposit overview: main orebodies (V2 and V1) and nearby construction camp (Sossego Project)

The deposit was first discovered in 1974. Early work (1980–1995) focused on smelting saprolite ore to ferronickel, but in the late 1990s, CVRD switched its attention to investigating the possibility of using pressure acid leaching at Niquel do Vermelho. CVRD is now assessing the attractiveness of developing Niquel do Vermelho as a pressure acid leach (PAL) project. CVRD is considering either Niquel do Vermelho will be a front-end only plant or will be an integrated plant incorporating a back-end refinery producing finished nickel and cobalt. The project is expected to produce around twelve times as much nickel as cobalt.

CVRD has completed the Niquel do Vermelho scoping study on March 2002. The scoping study phase comprised significant batch scale testwork programs performed at Dynatec, CVRD and Lakefield Oretest facilities. This testwork had the following scope:

- Preliminary testwork (Dynatec);
- Variability and preliminary optimization testwork - beneficiation and acid leaching - with 72 samples (CVRD);
- Mixed Sulphide Precipitation (MSP) and Mixed Hydroxide Precipitation (MHP) batch testwork (Lakefield Oretest);
- Pressure leaching batch optimization testwork (Lakefield Oretest).

One of the key factors for the project atractiveness is the mineralogy of most of the deposit, which comprises a combination of coarse cristalline silica (quartz) and very fine iron-rich limonite. This mineralogy allows ore beneficiation and significant upgrading, raising the R.O.M. grade from 1.2% Ni (after 0.8% Ni Cut-off) to around 1.7%, as shown on Table 2. The beneficiated limonite shows good rheology and relatively low acid consumption (between 200 and 300 kg/t of ore).

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Table 1 – Total resources (2001)

Geological Resources			
V1 + V2	**Tons (x1000)**	**Ni%**	**Co%**
Meas+Ind+Inf	**269.741,61**	**0,87**	**0,05**
Meas+Ind	**222.724,66**	**0,86**	**0,05**
Measure	**71.956,68**	**0,93**	**0,06**
V1 body	**Tons (x1000)**	**Ni%**	**Co%**
Meas+Ind+Inf	203.385,71	0,80	0,05
Meas+Ind	184.225,44	0,80	0,05
Measure	55.399,73	0,84	0,05
V2 body	**Tons (x1000)**	**Ni%**	**Co%**
Meas+Ind+Inf	66.355,90	1,08	0,06
Meas+Ind	38.499,22	1,13	0,05
Measure	16.557,26	1,24	0,07

Table 2 – Mineable reserves and mine schedule (2002)

Year	Mine (kTon)	ROM		Autoclave feed		
		kTon	Ni%	kTon	Ni%	Prod. Ni (t)
1	7818	2581	1,15	1546	1,61	23298
2	10612	4065	1,23	2444	1,71	39149
3	14465	4801	1,21	2759	1,74	45000
4	15426	4719	1,23	2761	1,74	45000
5	15477	4598	1,25	2759	1,74	45000
6	14037	4965	1,15	2760	1,70	44000
7	14269	4736	1,19	2760	1,70	44000
8	13490	4787	1,19	2760	1,70	44000
9	13433	4673	1,21	2760	1,70	44000
10	12021	4769	1,19	2760	1,70	44000
11 - 12	22712	9353	1,21	5520	1,70	88000
13 - 14	21657	9526	1,19	5520	1,70	88153
15 - 16	21038	9498	1,15	5520	1,64	84840
17 - 18	16164	9707	1,14	5520	1,65	85376
19 - 21	17678	13767	1,14	8280	1,58	122909
22 - 26	Stock	22638	0,78	13800	1,07	138257
27 - 31		23810	0,60	13800	0,86	111846
32 - 36		24762	0,45	13800	0,67	86132
37		3778	0,41	2082	0,61	11850
Mine life	230298	171534	0,93	99911	1,32	1234812

2. PREFEASIBILITY STUDY

The combination of reasonable head grade, low acid consumption and available mining infrastructure justified the start of a prefeasibility study on July 2002, which is scheduled for completion by end-2003. The prefeasibility study, which is looking at annual production of 40,000–45,000 tonnes of nickel and around 3,500 tonnes of cobalt, includes comprehensive resource-evaluation, metallurgical-testwork and engineering programs. CVRD has designed the prefeasibility study to be thorough, with the aim of reducing risks as well as the time and effort needed for the final feasibility study, scheduled for completion by the end 2004.

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The activities currently underway includes a drilling program of 80,000 m percussive drilling and 2,500 m large diameter (6.5") drilling. Percussive drilling samples, in addition to around 20,000 m of previous drilling, will be used for resource estimation and large diameter drilling will be used for metallurgical testing.

Comprehensive hydrological, geotechnical and hydrogeological programs have started on 4thQ 2002, and are expected to be completed by July 2003, to support prefeasibility engineering and environmental studies.

One of the main objectives of the prefeasibility study is to provide a good technical and economic basis for the comparison of available nickel refining routes and possible nickel products (intermediates or nickel metal), in order to maximize the project value and make the best use of the available infrastructure, related projects and markets that CVRD group currently operates. GRD Minproc has been awarded the prefeasibility engineering contract.

Another important objective of this phase of the project is to minimize key uncertainties typical of laterite nickel deposits, which, among others, are related to geological variability and process behavior.

3. METALLURGICAL DEVELOPMENT - PILOT PLANT

Metallurgical testing at prefeasibility was designed to be comprised of two main programs:

– Pilot plant program – for process development and engineering data collection
– Variability program – for variability evaluation of beneficiation, leaching, settling and rheology within the orebody.

Much attention was devoted to the samples that would be used for the testwork. Both programs used large diameter drill core samples, distributed throughout both orebodies. The variability program used 10% of the 2500 samples available, collected every 10 meters of the mineralised laterite profile, which ranges tipically from 40 to 80 m. In this way, the samples would represent the deposit and would intercept all ore types and subtypes.

The remaining samples were used to compose four large volume samples for pilot and process development testwork:

- "LowSi" sample, representing mainly a ferruginous saprolite oretype (SapFe), comprised of limonite with a small amount of silicates or quartz;
- "HighSi" sample, representing mainly a siliceous saprolite oretype (SapSi), comprised of roughly 50% relatively coarse SiO_2 (as quartz) with the remaining mass as fine limonite;
- "MidSi" sample, representing an intermediate oretype SapFeSi, comprised of roughly 35% coarse SiO_2 with the remaining mass as fine limonite and fine SiO_2;
- A blended sample, composed of amounts of the three samples above, and included all major oretypes.

Small amounts of the saprolite oretype (garnierite), found in the contacts and immediately below the main oretypes were blended within the samples, in order to simulate some blending of the oxidized ore with a low Mg garnierite that will occur at the mine.

The sampling composition scheme used allows direct correlation of pilot plant and variability samples, in the way that each of the bulk pilot samples tested had its variability counterparts. Also, sub-samples of the pilot samples were used for the same bench scale tests of the variability study, in order to assess scale-up and validate the methodology.

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The pilot plant program was performed at Lakefield Oretest, Australia from November 2002 to March 2003, and the variability testwork was started in November 2002 at CVRD's Mineral Development Center, located close to the main project office. Cross checks of bench scale test procedures between both labs was performed with sub-samples of the pilot plant bulk samples.

The pilot plant program was designed considering that pressure acid leaching of limonitic laterite nickel ores is a commercialized process and plant design and operating experience exists from four operating industrial plants. It also considers that the process flowsheet and its engineering is rather complex and variation between laterite ores requires a significant amount of pilot testing for new projects to quantify process parameters and collect engineering data.

Equipment and reagent vendors, namely Ciba, Cognis, Delkor, Eimco, Filtres Philiipe-RPA, Larox, Ondeo-Nalco, Outokumpu and SNF-Floerger participated in the pilot program to collect specific reagent selection and equipment sizing data.

Pilot testing comprised four beneficiation and three high pressure acid leaching (HPAL) runs with a total of 21 days of operation for both MHP (14 days) and MSP (7 days) downstream options. The MHP circuit pilot plant included all unit operations up to production of nickel cathode, whereas the MSP circuit pilot plant produced mixed nickel-carbonate sulphide for bench scale refining to nickel powder.

Beneficiation

The beneficiation flowsheet tested at pilot scale was quite simple, comprising crushing up to 2 inches top size, followed by scrubbing and classification. Product top sizes from 0.075 mm to 0.15 mm were achieved with good recoveries and nickel grades of the reject well below mine cut size.

Each sample was beneficiated separately. The blended sample product was used for the first HPAL run, and the products of the LowSi, MidSi and HiSi beneficiation samples were blended to produce the feed for the other two HPAL runs.

Table 4 – Beneficiation products - characterization

BeneRun No.	Ore Type	Feed			Product										
		Ni %	SiO2 %	(Si) %	Ni %	SiO2 %	pgrade	Co g/t	Cr %	Cu g/t	Al2O3 %	MgO %	MnO %	Fe2O3 %	Zn g/t
1	Blend	1,1	**38,7**	18,1	1,45	20,1	**132%**	800	0,62	240	2.57	4.44	0.45	63,9	240
2	Low Silica	**1,1**	**8,1**	3,8	**1,15**	6,9	**105%**	840	0,64	180	3,08	1,18	0,63	76,9	290
3	Mid Silica	**1,2**	**39,1**	18,2	**1,61**	18,8	**134%**	756	0,71	210	3,29	3,78	0,54	62,6	295
4	High Silica	**1,1**	**52,3**	24,4	**1,80**	19,6	**164%**	890	0,57	151	1,66	5,5	0,5	62,8	429

Pilot beneficiation confirmed previous bench scale variability results (Ribeiro et all, 2001), with a good correlation between nickel upgrade and silica content, as can be seen in Figure 3.

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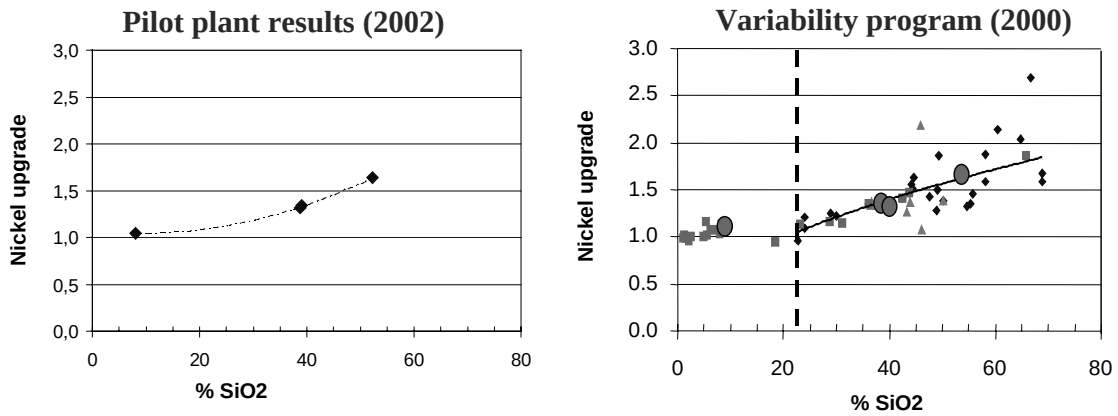

Figure 3 – Beneficiation results: prediction of nickel upgrade by SiO2 content

The silica upgrading algorithm is currently being improved by the 2003 variability program, which is coupled with a statistic geometallurgical data assessment which considers geology and mineralogy as inputs. The new algorithm will be the basis for prefeasibility mine planning as well as for the design of a beneficiation demonstration campaign scheduled for the bankable feasibility phase, which will focus on scale-up and variability behavior of mineable oretypes of the deposit.

The beneficiation products were thickened in a pilot paste thickener, which produced underflows with densities ranging from 42 to 47% solids under steady state (but not optimized) conditions. All samples were also submitted to bench scale testing by an independent consultant and thickener vendors. An independent company was also hired to assess ore rheology. Pilot and bench test results indicates that good underflow densities – around 45% - can be consistently achieved for the ore types tested.

Settling and viscosity parameters are also under evaluation with the current variability program, with the assessment of 50 samples distributed throughout the deposit.

High pressure acid leaching (HPAL)

Considering that the three pilot HPAL runs were the first ever performed with Niquel do Vermelho ore, with limited bench testwork performed prior to the runs, the operational and process results obtained at Lakefield Oretest can be considered remarkable. Plant availability of 98.5% was achieved in the preliminary pilot run with Niquel do Vermelho ore, while 99.7 and 99.0% were achieved in the following two prefeasibility runs, resulting in around 465 hours of steady state operation with no ore-process related problems or incidents. Figure 4 illustrates the operational profile of the second pilot plant run.

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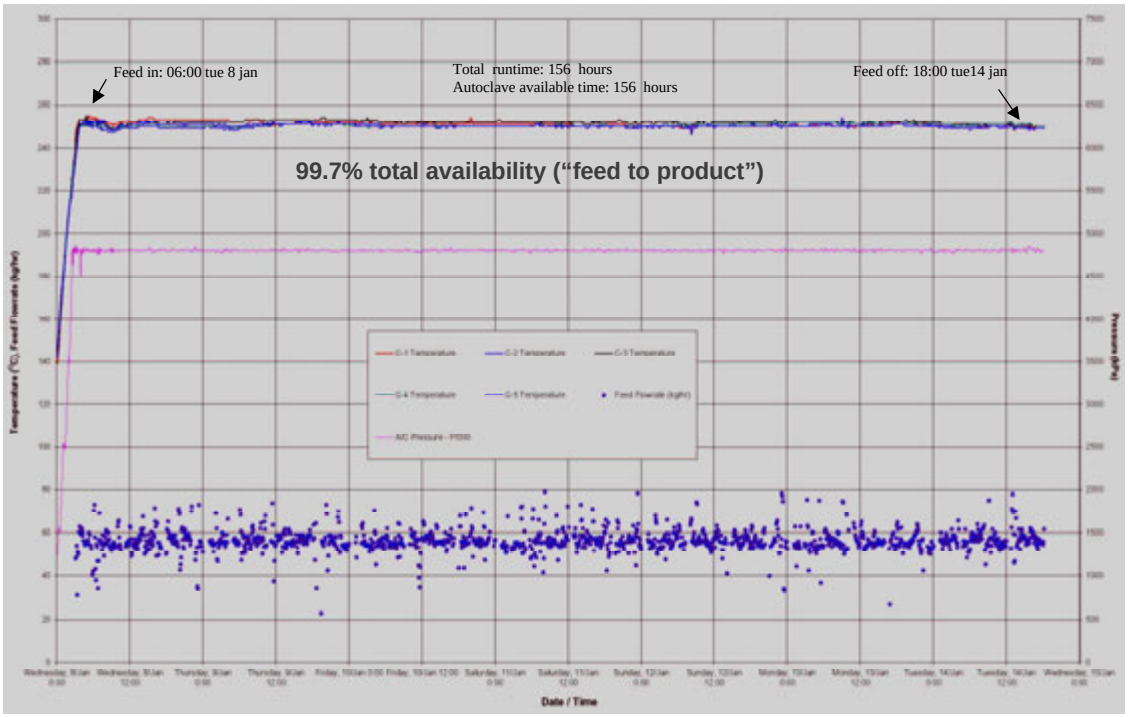

Figure 4 – Operational profile – HiPAL run#2 (Lakefield Oretest)

The ore rheological behavior and use of fresh water (available at the project site) contributed to the smoothness of the runs, which were characterized by good metallurgical results. Nickel extractions in excess of 96% were achieved consistently after three days of operation and remained high until the end of the campaign, with average recoveries of 96.5% on runs #2 (MHP) and #3 (MSP), as illustrated by Figure 5. Cobalt recoveries were around 95%.

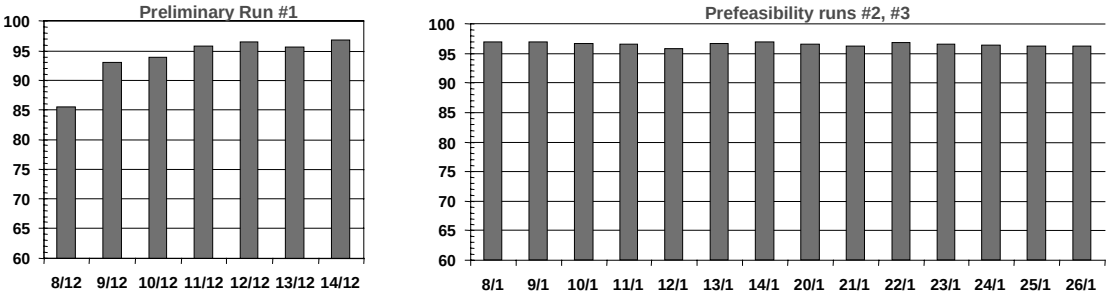

Figure 5 – Nickel recoveries – HPAL pilot runs

Other key performance indicators of the pilot runs are outlined below:

- Acid consumption of 280 kg/t (feed samples with ~2.5% Mg and ~1.3% Al);
- CCD thickener underflow densities between 50 and 60% at the pilot runs;
- Average washing efficiencies of 99.6 and 99.9% with 5 CCD stages for the MHP and MSP circuits respectively;
- Recycle re-leach efficiencies in excess of 99% (MHP circuit)

Nickel Refining

The selection of refining routes for testwork considered only routes that are already proven in commercial / industrial scale. This guidance considers the minimization of operational risks for the project and aims to provide basis for estimating project ramp-up at least as series #2 of McNulty assessment (Nice R., 2002). Therefore, considering current nickel industry status, mixed hydroxide and mixed sulphide precipitation options were selected.

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The evaluation of both options at pilot scale aimed to provide a fair basis of technical comparison between both routes, identify possible fatal flaws on process flowsheet for this particular ore, evaluate effluent treatment options and obtain data to sustain the engineering studies of the project's current phase.

MHP route was piloted through to the production of nickel cathode, with the production of nickel hydroxide, carbonate and oxide intermediates. MSP was piloted through to continuous precipitation of nickel sulphide, followed by bench scale refining and production of nickel metal by hydrogen reduction.

Nickel refining testwork is underway at the time this paper is being written, so results cannot be reported yet.

4. PROCESS ECONOMICS FOR THE SELECTION OF REFINING OPTION

The characteristics and objectives of the project makes it suitable for a *process economics* approach, shown on Figure 6, and applied previously by CVRD on the development of other projects (Torres et al, Alta 2002).

The main aim of using such methodology is to develop a consistent *project concept* at the engineering stage which can minimize project risks while maximizing project profitability. The end result of this approach is the maximization of the ***project value to the owner*** and related stakeholders, since development of projects without proper risk identification and mitigation can indeed undermine project profitability and ultimate value.

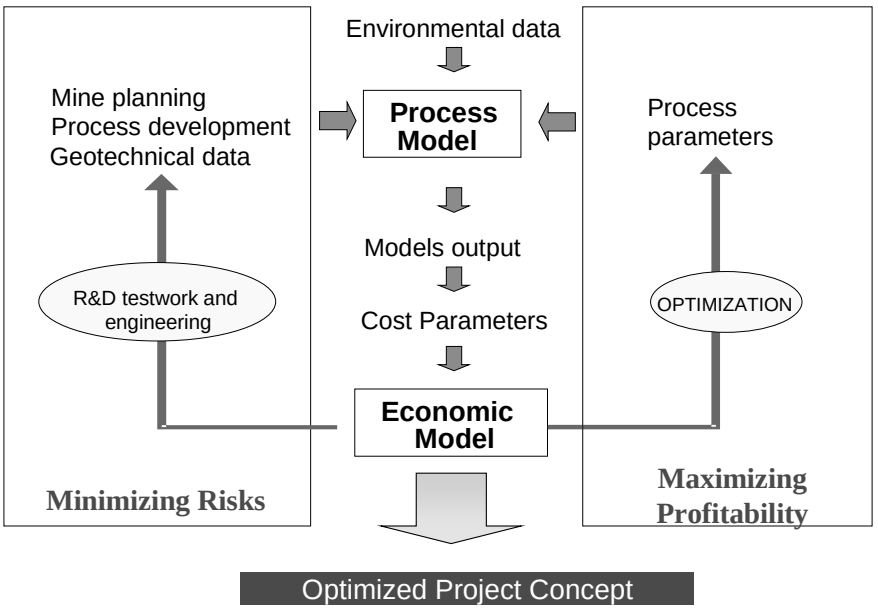

Figure 6 – Process Economics approach

The use of the process economics methodology on this project started early on its 2001-2002 *Scoping Study,* when preliminary process and economic models were custom developed by SNC-Lavalin. At that time, the MSP refining route was used for cost estimation due to more availability of operational and costing data, which is key in order to provide valid cost estimates at the scoping study level, when scarce testing and engineering data is available. The project costs were also estimated independently by Dynatec, with reasonable agreement between the estimates.

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For the prefeasibility study, the approach will be used more thoroughly, with the development by Minproc and CVRD of process and economic models which will based on pilot testwork data for the refining routes being currently considered for the project. The models will use more local cost data than previously and will compare production costs of metal and intermediates, as summarized on Figure 7. Market studies and strategic guidelines will also back up the cash flow models for each particular project configuration.

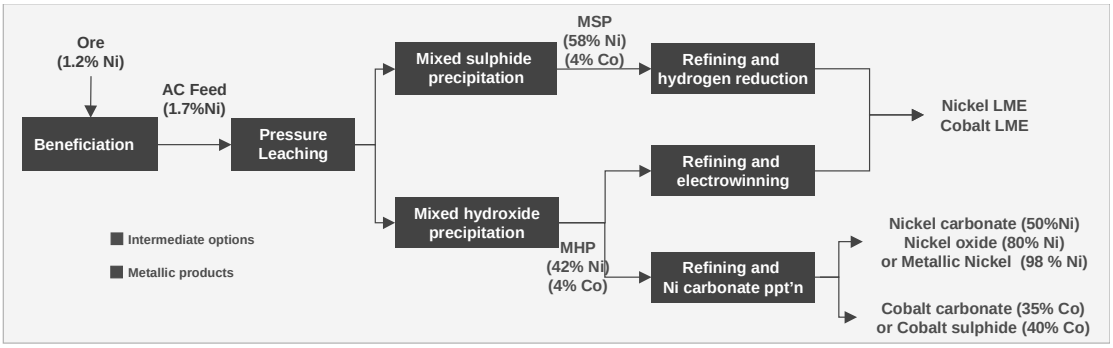

Figure 7 – Possible refining routes and nickel products for Niquel do Vermelho

If needed, prefeasibility engineering will be conducted for two or three process/product options. The following deliverables are sought at the end of the prefeasibility phase:

- A technical and economic assessment that can effectively capture the project economic value to the shareholder;
- Technical, economic, operational, commercial and strategic assessment of possible refining routes which can select the route to be detailed at the bankable feasibility study with a sound basis of information and knowledge, thus avoiding re-work and minimizing operation risks;
- Infrastructure, logistics and environmental plans that can capture the synergies of the project with Carajás Iron Ore operations and the nearby Copper Projects.

5. CONCLUSIONS

A benchmarking of Niquel do Vermelho metallurgical results with other nickel laterite deposits position the deposit well due to the following characteristics:

- ability to upgrade at beneficiation with good predictability;
- good rheological characteristics at reasonable thickener underflow densities;
- high nickel and cobalt extractions;
- relatively low acid consumption;
- very good CCD washing efficiency;
- preliminary positive response to both MHP and MSP refining routes.

The combination of the characteristics above with reasonable mineable grades and existing infrastructure can contribute to good capex efficiency, as well as to operating costs in the lowest quartile among nickel laterite plants.

The project development approach at the engineering phase is based on the dual objective of minimizing risks while maximizing project profitability and value. Considering the issues associated with mineral projects in general and nickel laterites in particular, thorough resource assessment, process development and engineering programs are well justified towards the goal of delivering a successful operation.

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“DESIGN OF HORIZONTAL AUTOCLAVES, A PILOT PLANT EVALUATION OF SOLIDS SUSPENSION, BLENDING & RESIDENCE TIME DISTRIBUTION”

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ABSTRACT

Mixing theory is based on applying many fundamental correlations to standard baffled dished bottomed vertical cylindrical vessels with high degrees of symmetry. Horizontal autoclaves have been used for more than two decades to rapidly complete chemical reactions at elevated temperatures. All compartments and especially the first and last compartments are asymmetrical. To date no comparison of the effects of the asymmetric geometry on mixing system design and scale-up has been published. The results of investigating solids suspension, mixing time, momentum of impeller to feed slurry and feed location in the first autoclave compartment are presented as influences on residence time distribution. CFD modeling was developed and verified with the test apparatus.

1. INTRODUCTION

Hydrometallurgy processing has adopted the use of horizontal autoclaves divided into compartments or stages to rapidly complete chemical reactions at high temperatures as the dissolution step in metal recovery.[1][2] The horizontal autoclaves are operated as continuously stirred tank reactors (CSTR) with each compartment being a reactor stage. Scale-up into production from lab scale autoclaves, used to develop the process, has had varying degrees of success and in some cases metal recovery is less than expected. [3]

Most published information about mixing systems presents data from batch simulations in vertical cylindrical tanks.[4] There are also presentations identifying new impeller evaluations claiming enhanced performance at minimum capital and operating cost. Vertical cylindrical tank mixing systems have high degrees of symmetry. Horizontal autoclave compartment mixing systems have high degrees of asymmetry.

This presentation identifies that the impeller system is important to impart kinetic energy into the total slurry volume of asymmetric compartments. However, more important to successful scale-up is to address the dynamic mixing system in total. Historically horizontal autoclave design has typically been divided into the engineering of the autoclave vessel and the process design separately from the design of the agitators.

EKATO defines the total mixing system for the horizontal autoclave compartments into individual components: the compartment geometry, the impeller system, baffle geometry, process feed inlet locations and kinetic energy from the feed components. Each component has an influence on the mixing system performance and must be carefully selected and considered for full-scale operation to meet expectations.

2. OBJECTIVES

The overall performance of horizontal autoclaves is significantly influenced by the mixing efficiency within the first compartment or compartments where reactants are added. The following mixing tasks must be achieved:

- Blending of chemical reactants such as minerals feed slurry and acid,
- Solids suspension, and
- Gas dispersion.

This work focuses on blending and solids suspension.

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A 1:10 scale model of a typical first compartment of a horizontal autoclave was used to observe and measure the effectiveness of fulfilling each mixing task. A photo of the autoclave compartment model can be seen in Figure 1.

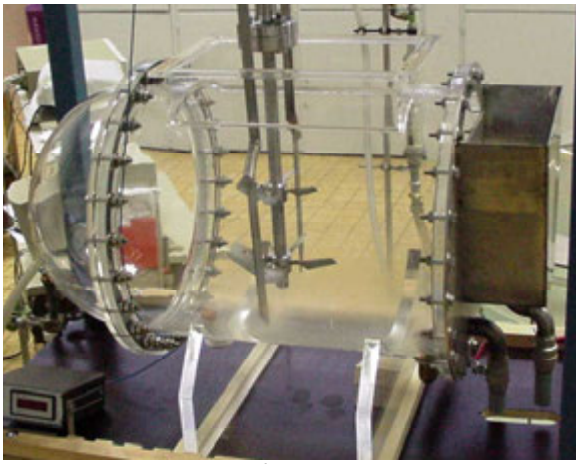

Figure 1
1:10 Scale Autoclave Model

Blending efficiency and the residence time distribution (RTD) of the reactants are impacted by the flow patterns produced by the impeller system and inlet locations of reactants such as feed slurry and sulfuric acid. Homogeneous solids suspension is required so that sufficient residence time is maintained to achieve the desired reaction completion. Settled solids will not only be isolated from chemical reactions, but will also reduce the active compartment volume. Poor blending and poor solids suspension contribute to loss in compartment efficiency and can reduce overall residence time and can cause metal recovery to be less than expected.

The impeller flow patterns developed in an autoclave compartment are asymmetrical and deviate significantly from a standard baffled dished bottom vertical cylindrical vessel. The first and last autoclave compartments are the most asymmetric, and whether the 'know how' for vertical cylindrical vessels can be applied is an open question.

The kinetic energy from feed streams must be optimized in addition to the energy supplied by the impeller system in a way that the net influence of the feed streams and mixing are synergistic and do not act as opposing forces.

In view of optimizing performance in the first compartment, measured as homogeneous blending of reactants and homogeneous solids suspension, EKATO engineers

- First developed an optimized impeller configuration to produce reliable solids suspension,
- Measured blending and suspending characteristics typically used in design correlations,
- Identified optimum feed locations that minimized concentration gradients and short circuiting and produced a near ideal RTD, and
- Confirmed that test results are duplicated by CFD simulation for scale-up.

Only results using the optimized impeller system are presented.

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3. EXPERIMENTAL INVESTIGATION

3.1 SOLIDS SUSPENSION

Hydrofoil type axial impellers are ideal for solids suspension in standard baffled dished bottomed vertical cylindrical vessels. The solids suspension "problem zone" is the center of the vessel. The mixing system generates symmetrical axial flow patterns directed toward the center of the vessel bottom. The flow is inverted off the bottom and lifts the solid particles, distributing them over the slurry height. The design basis is to transmit sufficient energy into the slurry with the impeller system to overcome the settling velocity of the solids.

EKATO engineers observed that an axial impeller system does not produce reliable solids suspension in a horizontal autoclave compartment. The solids suspension "problem zones" in a horizontal autoclave compartment are moved from the center of the vessel to inside the dished end and near to the bottom of the compartment division wall.

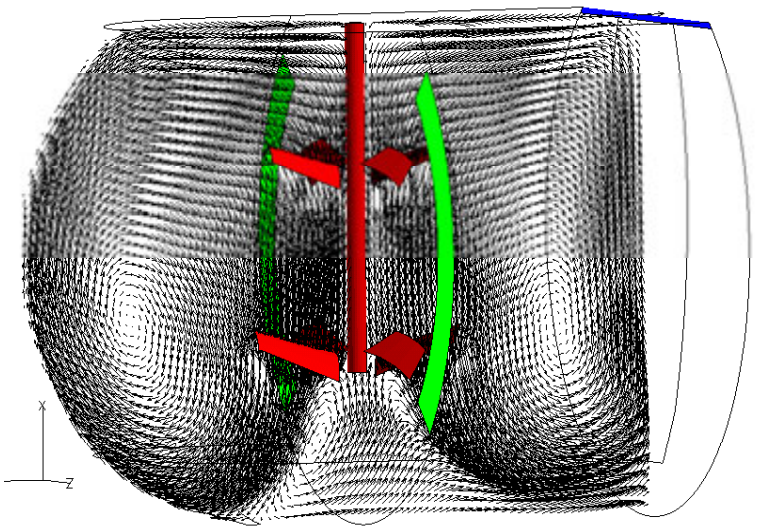

Figure 2 Velocity Distribution at the Longitudinal Center Line

Figure 2 exhibits the flow patterns of an optimized impeller system design, that develops both axial and radial flow components directed at the locations of the two problem zones. Reliable solids suspension is achieved with the EKATO VISCOPROP hydrofoil impeller system. Power savings in the order of 30% were achieved with this impeller system versus the power required by other impeller systems to obtain a similar degree of reliable homogeneous solids suspension.

A review of mixing literature failed to identify the existence of correlations for the design for solids suspension in horizontal vessels. Therefore EKATO selected to make measurements and use the well-known Zwietering correlation that can be applied to vessel volumes up to 100 m^3.[5] The use of the correlation allows for the design of off-bottom suspension of relatively large particles that are found in some nickel laterite ores or are produced by scale formation.

Zwietering Correlation

$$n_{js} = S \cdot \nu^{0.1} \cdot \left(\frac{(\rho_s - \rho_l) \cdot 9.81}{\rho_l} \right)^{0.45} \cdot d_p^{0.2} \cdot c_g^{0.3} \cdot d_2^{-0.85}$$

Using S.I. units the Zwietering correlation is dimensionally consistent. The S-value or geometrical constant was measured using two different sizes of glass beads with

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diameters of 1.2 mm and 0.215 mm. An S-value of 4.5 was found for a homogenous solids distribution in this mixing system.

3.2 MIXING TIME MEASUREMENT

Every mixing system has a unique mixing time characteristic ($n \times \theta$) that is a function of the combination of mixer and compartment geometry and the impeller system's transfer of kinetic energy into the slurry. The mixing time characteristic definition is the impeller speed and blend time required to produce minor gradients in temperature and/or concentration throughout the compartment volume.

Mixing Characteristic Equation

$$n \times \theta = c \text{ x } N_e^{0.33} x \left(\frac{d_1}{d_2} \right)^2 x \frac{h_1}{d_1}$$

Batch measurement of the mixing time characteristic was made. The following method was used.

A deep blue Iodine-Starch complex was decolorized by the addition of a stoichiometric amount of thiosulfate solution. The measurement was repeated 3 times for three different mixer speeds. Under turbulent flow conditions in a baffled tank the mixing time characteristic is a constant.

The mixing time characteristic constant for the autoclave compartment model is ($n \times \theta = 25$). An ideally mixed compartment volume is achieved with a residence time to blend time ratio of $\tau / \theta > 10$. The horizontal autoclave compartment model had a $\tau / \theta > 80$.

3.3 MOMENTUM COMPARISON

Another correlation used to produce ideal blending is to compare the ratio of the momentum created by the impeller system to the momentum created by the incoming feed streams. The correlation is identified by the equation below.

$$\frac{M_{imp}}{M_{feed}} = \frac{Q_{imp}^2 \cdot n^2 \cdot d_j^2 \cdot d_2^4}{q_{feed}^2}$$

$$Q_{imp} = \frac{q_{imp}}{n \cdot d_2^3} = 0.83$$

A ratio of $\frac{M_{imp}}{M_{feed}} > 10$ is used so that the mixing system controls and an ideal mixing will exist in a standard baffled dished bottomed vertical cylindrical CSTR vessel. The impeller system in the autoclave compartment model produced momentum ratios of $\frac{M_{imp}}{M_{feed}} = 18 \rightarrow 50$.

3.4 RESIDENCE TIME DISTRIBUTION (RTD)

Correlations used in symmetrical CSTR vessels to achieve ideal blending and homogeneity were used to design the autoclave compartment model mixing system. The ratio of residence time to blending time (τ / θ) and the ratio of impeller momentum to feed

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momentum $\left(\frac{M_{imp}}{M_{feed}}\right)$ were measured to be much greater than required in a symmetrical CSTR vessel. The mixing system design should produce an ideal RTD in the horizontal autoclave compartment model.

3.4.1 Measurement

The mixer speed used for the RTD measurement was the mixer speed that produced homogeneous solids suspension and had a clockwise rotation. A two component mixing system was selected where the chemical reaction kinetics would be very rapid. Thus eliminating reactions kinetics as an influence in evaluating various mixing systems. Typical feed rates were used and reduced to the 1:10 scale to simulate the feed momentum.

Component A was 0.2 m NaOH solution simulating ore slurry feed and was fed through an 8 mm diameter pipe at a rate of 300 l/h.

Component B was 1 m H_2SO_4 solution simulating a lixiviant used to dissolve minerals in component A and was fed through a 2 mm diameter pipe at a rate of 30 l/h.

Figure 3 exhibits how the two components were continuously fed into the compartment at different feed point locations.

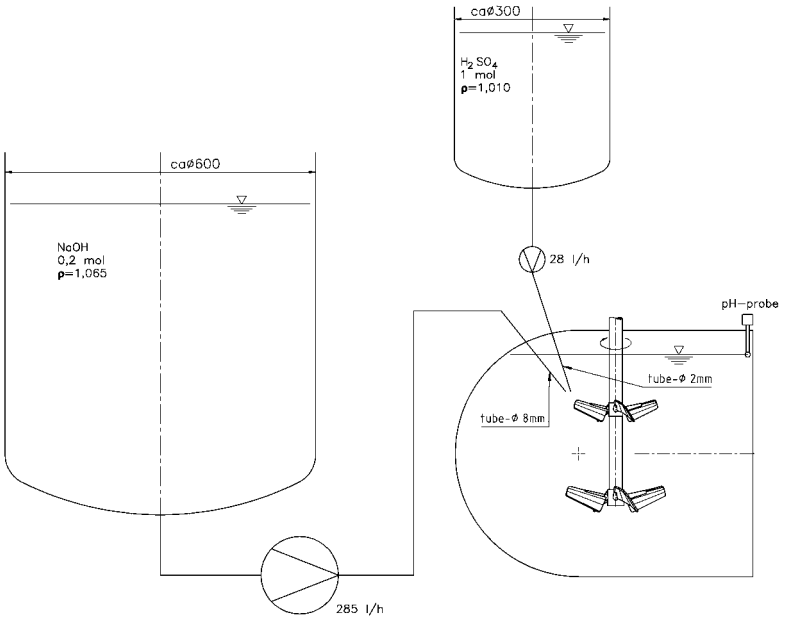

Figure 3 Continuous Feed System

Phenolphthalein was used to be able to visualize variation in the RTD. In addition, the degree of homogeneity was measured by pH differences with a pH meter. Ideally no color or pH difference should exist throughout the compartment volume, with exception of the feed point area.

The mixing system was allowed to stabilize or come to steady state defined as no measurable change in the size of colored zones or pH values at four different feed locations.

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- Feed location 1. Component A inlet is located on the right side of the mixer with rotation toward the dished head and component B inlet is located 180^{o} opposite on the left side of the mixer with rotation toward weir.

- Feed location 2. Components A and B inlets are located on the right side of the mixer with rotation toward the dished head. Discharge flow from each pipe is parallel.

- Feed location 3. Components A and B inlets are located on the right side of the mixer with rotation toward the dished head. Discharge flows from each pipe are directed as impinging jets, however one is in the same direction of the mixer rotation and the other is opposite to the mixer rotation.

- Feed location 4. Components A and B inlets are located on the right side of the mixer with rotation toward the dished head. Discharge flows from each pipe are directed as impinging jets directed in the same direction of the mixer rotation.

3.4.2 Discussion of Results

Visual color gradients from red to clear identified steady state asymmetric flow patterns in the autoclave compartment model, very different from flow in vertical cylindrical vessels. There are two strong tangential flow components developed on either side of the mixer. From a view at the end of the dish, the component on the left impacts into the compartment wall and the component on the right flows into the dish head.

3.4.2.1 Feed location 1

Figure 4 exhibits components A and B divided into two different sections of the compartment. The sulfuric acid, component B, is swept toward the weir and the sodium hydroxide component A is swept toward the dish end creating a stationary concentration gradient of ~4 pH units. Increasing the impeller speed to 220 rpm (>200% increase in power) did not improve the situation.

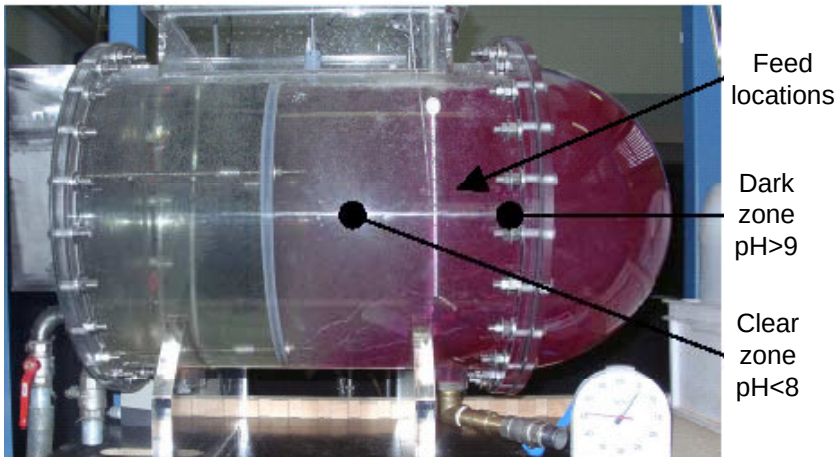

Figure 4 Horizontal Autoclave Compartment Model (Feed Location 1)

The process impact was poor RTD of the reactants or a reduced residence time and short-circuiting of sulfuric acid.

3.4.2.2 Feed Location 2.

The concentration gradient of Components A and B was less severe. Increasing impeller speeds up to 185 rpm had little affect on a stationary concentration gradient of ~2 pH units.

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The process impact was an improvement in RTD, but the active compartment volume remained reduced as well as residence time. Acid short-circuiting was reduced.

3.4.2.3 Feed Location 3.

The feed orientation had a dramatic affect on improved blending. Impeller speeds of 170 rpm and 150 rpm produced a stationary concentration gradient of 0.5 pH units.

3.4.2.4 Feed Location 4.

This feed orientation produced a near ideal RTD and is shown in Figure 5. Impeller speeds of 170 or 150 rpm produced no stationary gradient in pH. A small zone of ~pH 9 appears in the bottom dish area on the left side only.

The process impact was an almost ideal RTD with a high homogeneity and component residence time as expected.

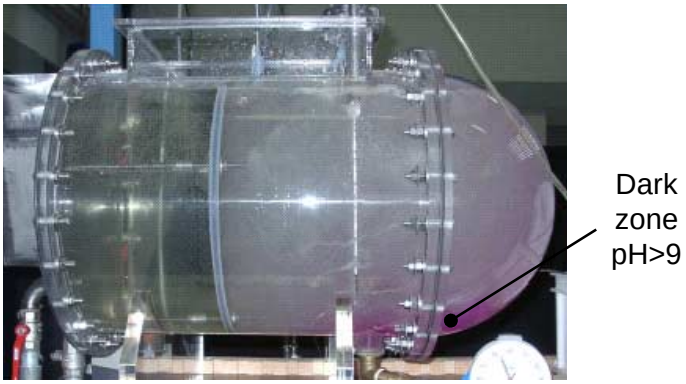

Figure 5
Horizontal Autoclave Compartment Model
(Feed Location 4)

4. CFD SIMULATION

A CFD model simulating the first compartment was developed and validated with the experimental results and observations. Scale up by a geometrical factor of 10 was made to full size. The point of origin is at the bottom of the vessel under the impeller axis. The inlet nozzles are not modeled, however spherical mass sources were applied in the calculation instead.

Continuous mode single-phase calculation was used. The slurry was modeled with a representative density and viscosity. Impeller rotation was implemented by a sliding mesh and modeling of the turbulent nature of the flow field used a standard k-ε turbulence model. Transient solution of the unsteady Navier-Stokes equations was applied to a block-structure mesh with 100,000 control volumes.

4.1 FLOW PATTERNS

Figure 2 shows the resulting velocity field for the single-phase slurry calculation using the optimized EKATO VISCOPROP hydrofoil impeller system. Strong upward directional flows are found on the compartment walls so solids overflow into the next compartment or stage is realized.

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Figures 6, 7 and 8 exhibit vertical velocity profiles at three cross-sectional locations in the compartment; in the dished head, near the impeller, and at the compartment wall

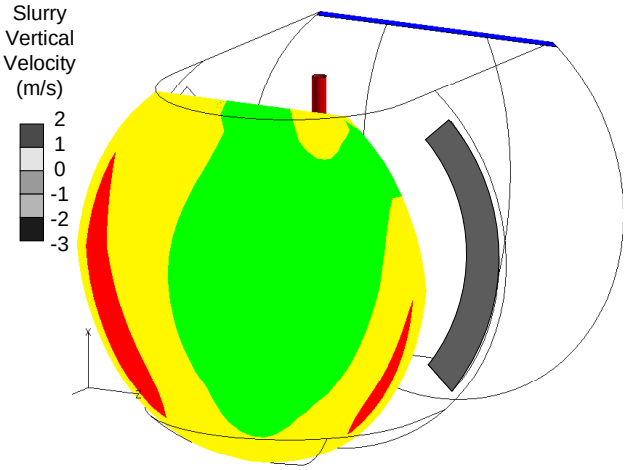

Figure 6 Velocity Profile Cross Section +1.5m from Shaft

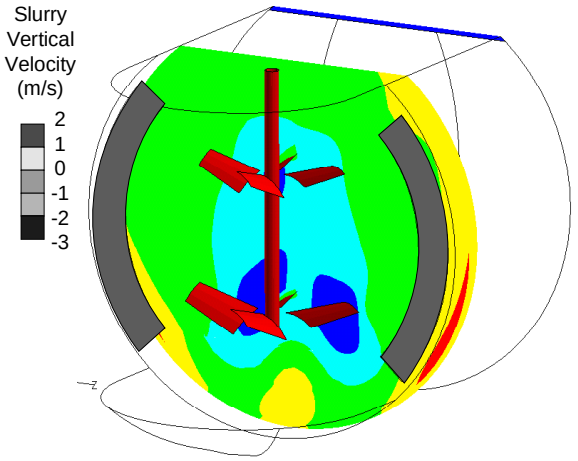

Figure 7 Velocity Profile Cross Section -0.5m from Shaft

respectively.

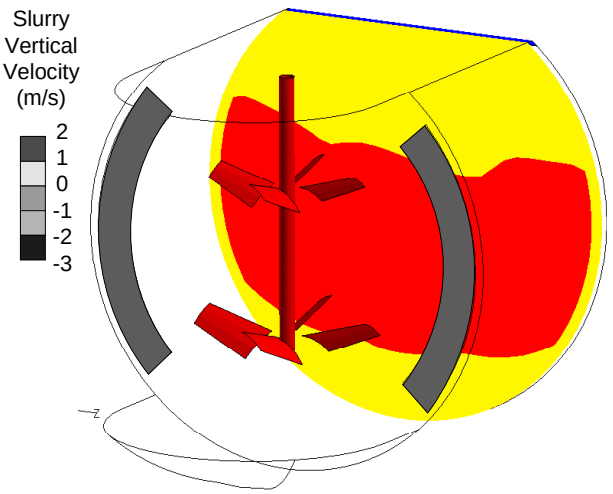

Figure 8 Velocity Profile Cross Section at Wall

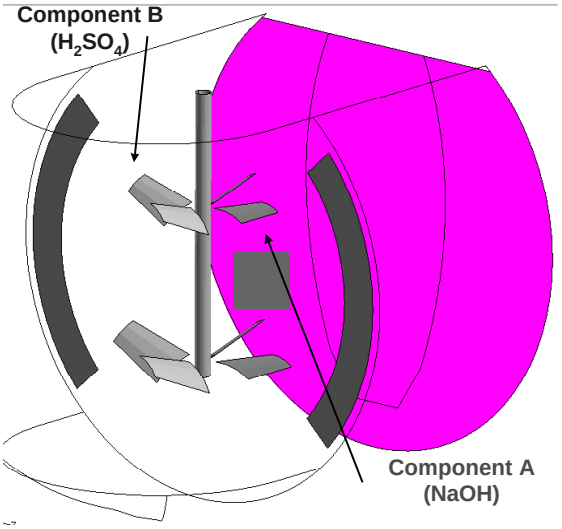

Figure 9 CFD Model - Feed Locations

4.2 CONCENTRATION PATTERNS

The colorization used in the experimentation provided vivid pictures that could be used to in the development of the CFD model. Ultimately the CFD model matched the concentration gradients measured in continuous simulation in the 1:10 scale model.

Figure 9 exhibits the approximate feed inlets of both components A and B for location 1. Both components A and B inlets for locations 2, 3, and 4 both components were at the component A location in Figure 9.

4.2.1 CFD Simulation - Location 1

Figure 10 exhibits the CFD simulation of the same concentration gradient exhibited in figure 4 obtained by the solution of the differential equations for the convective transport of components A and B. The same division of components is identified in more detail by the CFD simulation.

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Figure 11 exhibits flow paths of the components A and B starting from the feed locations. The flow of component B to the compartment wall overflow and component A to the dish end is visible.

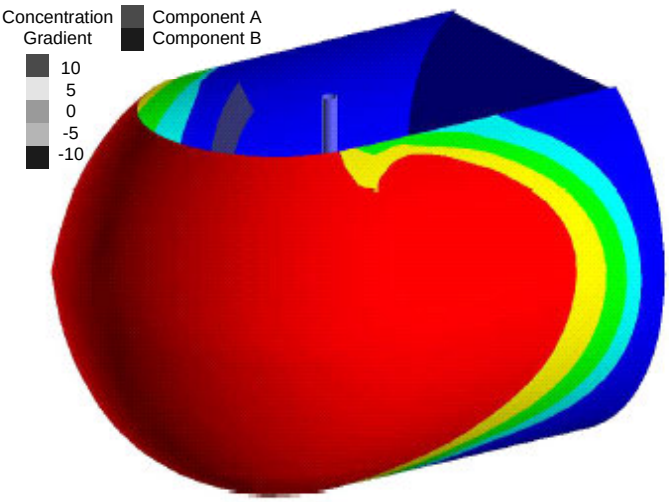

Figure 10 Concentration Gradient Profile (Location 1)

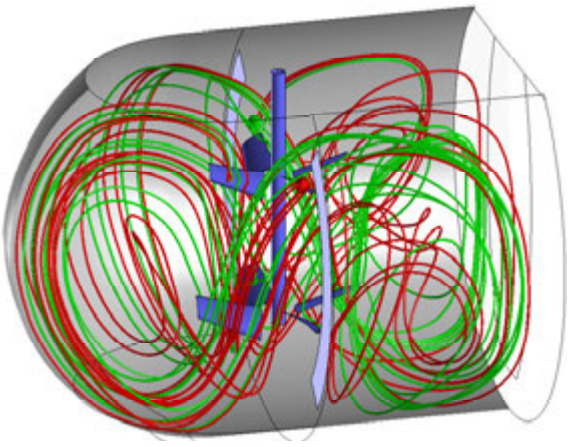

Figure 11 Component Flow Paths (Location 1)

4.2.2 CFD Simulation - Location 2

Figure 12 exhibits the affect of locating the two feed components close together. A reduction in concentration gradient is apparent, but the RTD is still less than ideal.

Figure 13 exhibits the flow paths of both components directed into the dish end but still separated.

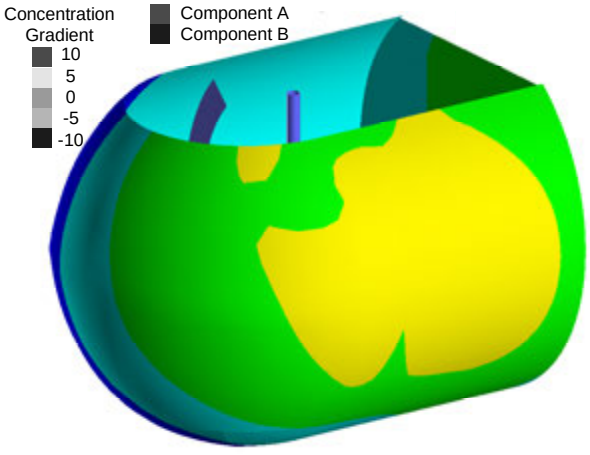

Figure 12 Concentration Gradient Profile (Location 2)

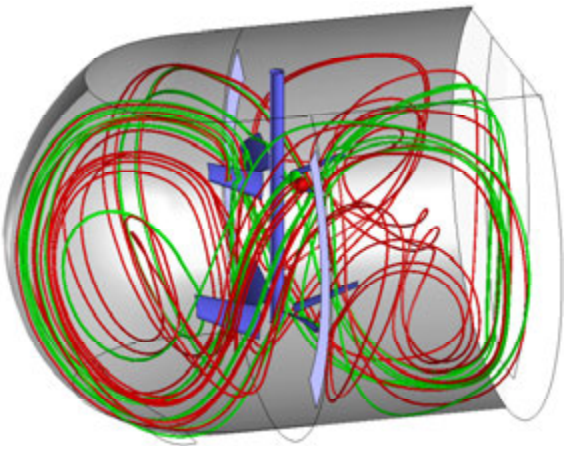

Figure 13 Component Flow Paths (Location 2)

4.2.3 CFD Simulation - Location 4

Figure 14 exhibits the results of total optimization of the mixing system. Near ideal RTD was obtained with a small zone in the dish head containing small concentration gradients. Most important is there was no short-circuiting of the sulfuric acid component B into the next compartment.

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Figure 15 exhibits that the flow paths of the two components are together ensuring almost instantaneous contact of the reactants creating the maximum possible residence time.

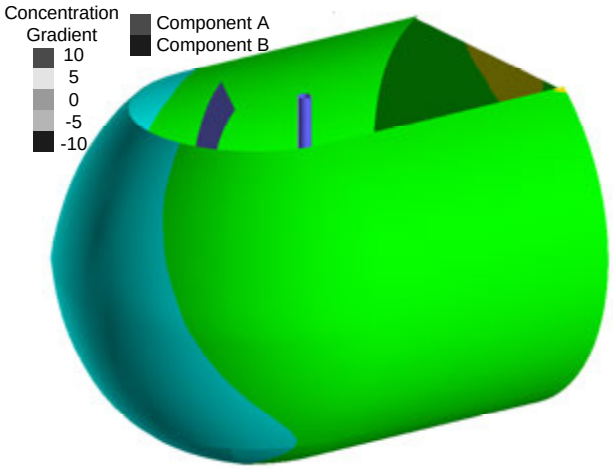

Figure 14 Concentration Gradient Profile (Location 4)

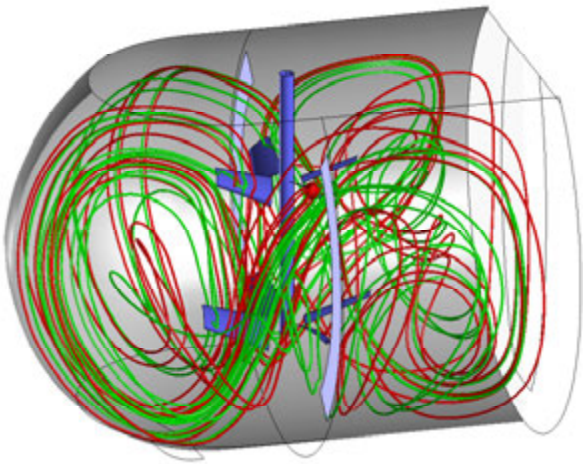

Figure 15 Component Flow Paths (Location 4)

5. CONCLUSIONS

5.1 Slurry flow patterns in horizontal autoclave compartments are asymmetrical and very different from the symmetrical flow patterns in standard baffled dished bottom vertical cylindrical vessels.

5.2 Correlations used to design mixing systems for solids suspension, blending time, and residence time distribution can be used with corrected constants to predict performance in each compartment.

5.3 Two solids suspension problem zones exist, one in the dish and one near the bottom of the compartment separation wall. Standard hydrofoil axial impellers developed for symmetrical vertical cylindrical vessels are not ideal for solids suspension in the asymmetrical geometry of horizontal autoclave compartments. An adapted impeller shape with both significant radial and axial flow components produces reliable solids suspension at power savings compared to the power input required by other impeller systems to achieve the same level of reliable solids suspension.

5.4 Feed streams inlet locations, flow rates and direction with respect to the mixer rotation significantly affect the RTD of the components, even if other blending correlations relating the ratio of (residence time / blending time) and (impeller system momentum / feed momentum) are satisfied.

5.5 CFD simulations of mixing in compartments of horizontal autoclaves have been developed and can be used as a powerful tool to confirm that the total mixing system design will meet expectations identified in pilot plant demonstrations.

5.6 Successful scale-up of hydrometallurgical processes using compartmented horizontal autoclaves requires a coordinated engineering effort with process design, vessel design and agitator design to produce a total mixing system that meets performance expectations.

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6. NOMENCLATURE

n_{js} = Agitator shaft speed for off bottom solids suspension (s^{-1})

S = constant (dimensionless)

ν = Liquid kinematic viscosity (m^2/s)

ρ_s = Solids density (kg/m^3)

ρ_l = Liquid density (kg/m^3)

d_p = Mean particle diameter (m)

c_g = Solids mass (%)

d_2 = Impeller diameter (m)

n = Agitator shaft speed (s^{-1})

θ = Blend time (s)

c = Constant (dimensionless)

N_e = Impeller power number (dimensionless)

d_1 = Compartment diameter (m)

h_1 = Slurry height (m)

$$Q_{imp} = \frac{q_{imp}}{n \cdot d_2^3} = 0.83$$

M_{imp} = Momentum from impeller $\left(kg\,m/s\right)$

M_{feed} = Momentum from feed slurry1 $\left(kg\,m/s\right)$

Q_{imp} = Pumping number (dimensionless)

q_{imp} = Impeller pumping capacity $\left(m^3/s\right)$

n = Agitator shaft speed $(1/s)$

d_j = Component A nozzles diameter (m)

d_2 = Bottom impeller diameter (m)

q_{feed} = Component volumetric feedrate $\left(m^3/s\right)$

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Jarosite / Alunite in Nickel Laterite Leaching - Friend or Foe?

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Presented by

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1. INTRODUCTION

Iron is the fourth most abundant element in the earth's crust, and occurs as a major gangue element in many ores and minerals. For example, gold often occurs with pyrite (FeS_2), pyrrhotite ($Fe_{1-x}S$) and/or arsenopyrite (FeAsS), nickel in pentlandite ($[Fe,Ni]_9S_8$) and violarite (Ni_2FeS_4), copper in chalcopyrite ($CuFeS_2$), and zinc in sphalerite ([Zn,Fe]S). Iron also occurs as a major component of oxide minerals such as iImenite ($FeTiO_3$), ferrotantalite ($FeTa_2O_6$), chromite ($FeCr_2O_4$), wolframite ($[Fe,Mn]WO_4$) and nickel laterite ores (Fe[Ni,Co]OOH).

The type of iron minerals present in the ore will have an important effect on the leaching process and the residual solids and solutions formed. For example in gold processing using cyanide, pyrrhotite is highly reactive requiring the addition of reagents to control its activity, whereas pyrite and arsenopyrite are much less reactive, and will remain in the residue. In fact, gold encapsulated in these latter minerals needs to be processed by a suitable oxidation process such as pressure oxidation or bacterial leaching to destroy the sulphide minerals and render the gold amenable to cyanidation.

In zinc processing using oxidative ammonia leaching, the iron content of the sphalerite governs its reactivity with high-iron dissolving faster than low-iron sphalerites. The oxidized iron minerals are generally precipitated as hematite Fe_2O_3, goethite FeOOH or various jarosite-type compounds $MFe_3(SO_4)_2(OH)_6$ during this process, depending on the chemical environment of the oxidation process. In many cases the form in which the iron precipitates can be controlled, but in others it cannot.

Iron is the major metallic component of nickel laterite ores, ranging from 10% to over 50%, and processing of these ores is "*essentially a process of controlling the behaviour of iron and separating it from the nickel and cobalt*" (Queneau and Weir, 1986). In the hydrometallurgical processing of nickel laterite ores, either by high pressure sulphuric acid leaching or the more recent atmospheric leaching processes, the iron in the ore is transformed into either hematite or a jarosite. The form of this residue is very dependent on the leaching temperature, the composition of the ore and the process water used. This paper investigates how the temperature and process solution composition govern the form of iron in the final residue, and the implications of this for product purity and processing costs.

2. THE ALUNITES

The jarosites are iron-containing members of a large mineral family called the Alunites having the generic formula $AB_3(SO_4)_2(OH)_6$. The cation A can be H_3O^+, Na^+, Rb^+, Ag^+, Tl^+, K^+, NH_4^+, $Pb_{1/2}^{+}$ or $Hg_{1/2}^{+}$. The metal ion B can be Al, Cu or Fe. The alunites commonly form solid solutions with each other. They have been well studied by Dutrizac and co-workers (Dutrizac and Kaiman, 1976, Dutrizac, 1983 and 1984, Dutrizac and Jambor, 1985).

Only six jarosite minerals are known to occur naturally, but a further nine have been synthesized in the laboratory. The six naturally occurring minerals are listed below in Table 1 together with the two main aluminium analogues.

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Table 1 Naturally Occurring Alunites

Name	Formula
Hydronium Jarosite	$(H_3O)Fe_3(SO_4)_2(OH)_6$
Ammoniojarosite	$(NH_4)Fe_3(SO_4)_2(OH)_6$
Argentojarosite	$AgFe_3(SO_4)_2(OH)_6$
Jarosite	$KFe_3(SO_4)_2(OH)_6$
Natrojarosite	$NaFe_3(SO_4)_2(OH)_6$
Plumbojarosite	$PbFe_6(SO_4)_4(OH)_{12}$
Hydronium Alunite	$(H_3O)Al_3(SO_4)_2(OH)_6$
Natroalunite	$NaAl_3(SO_4)_2(OH)_6$

The relative stabilities of the most common jarosite minerals are reported to be $K^+ > NH_4^+ > Na^+$. Jarosite minerals can be synthesized by precipitation from a ferric or aluminium sulphate solution containing the appropriate salt at low pH. They are typically found in gossans, siliceous limonitic veins in limestones, acidic soils and sulphide-rich mineral dumps. They are formed naturally during the oxidation of pyrite, and are commonly found intergrown with "limonite" (goethite and other hydrated forms of iron oxide) or less commonly hematite. During weathering, they break down to goethite, hematite and other hydrated iron oxides (Chen and Cabri, 1986). Also important in sulphuric acid leaching of nickel laterite ores are the aluminium-containing members of the alunite family, hydronium and natro-alunite. Much less has been written about these minerals.

3. STABILITY RANGES OF GOETHITE, HEMATITE AND JAROSITE

The relative stability of jarosite and goethite can be expressed in terms of an E_H-pH diagram. Figure 1 (Brown, 1971) shows that jarosite is only stable in a highly acidic and oxidizing environment (pH 0–3 and $E_H > 600$ mV).

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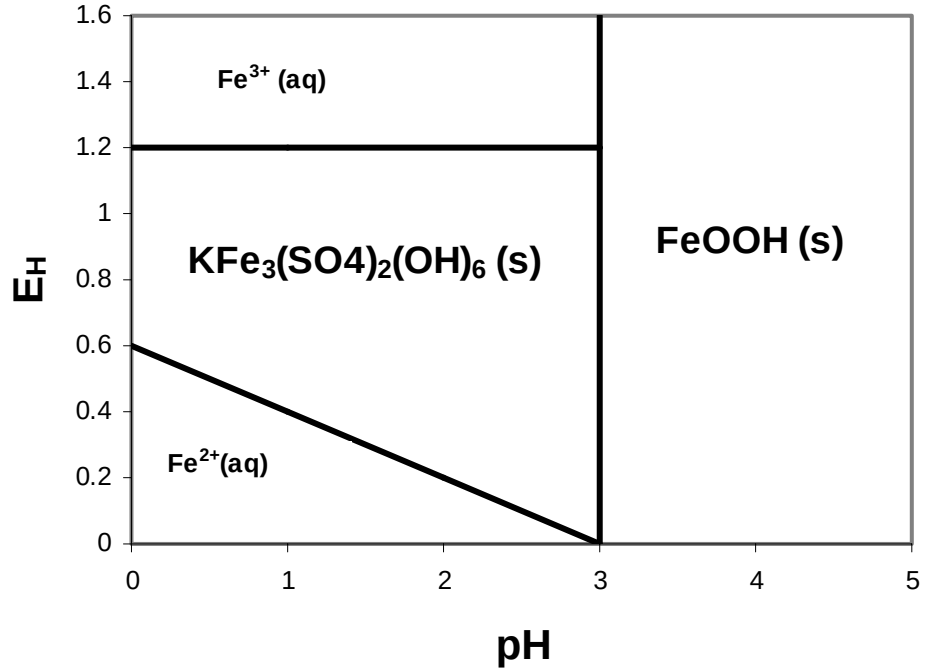
Figure 1 E_H-pH Stability Diagram for Jarosite (after Brown, 1971)



The relative stabilities of jarosite, goethite and hematite in terms of pH and temperature are shown in Figure 2 (after Chen,1986). It can be seen from this diagram that jarosite is only stable below pH 3, and that at higher temperatures in this pH region jarosite is favoured over $Fe^{3+}_{(aq)}$ ion in solution. This is a preferred method for iron removal from solution, and is used in high pressure sulphuric acid leaching and also in high temperature pressure oxidation. It can also be seen that at higher pH's, goethite formation is favoured at lower temperatures, and hematite at higher temperatures.

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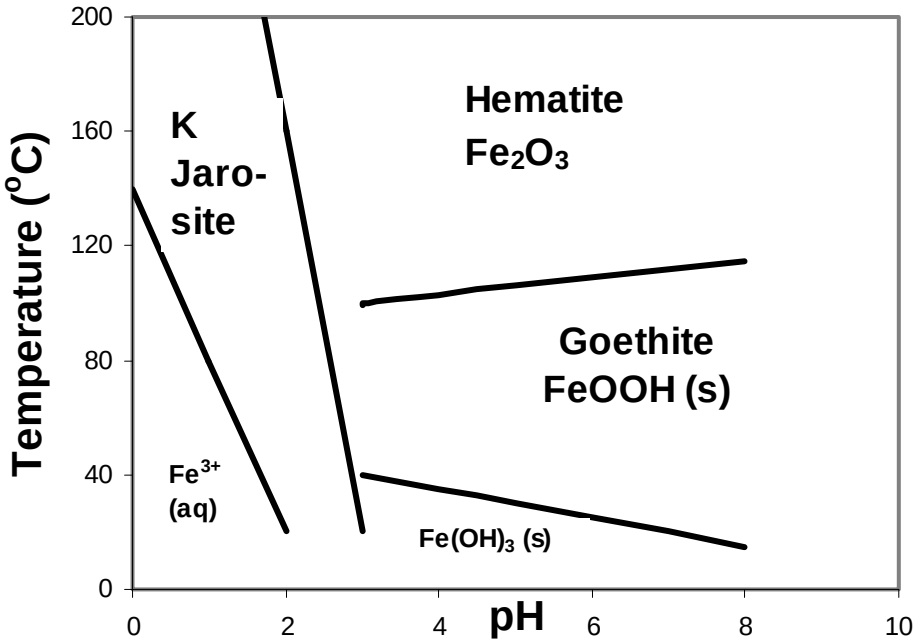
Figure 2 Temperature-pH Stability Diagram for Jarosite (after Chen, 1986)



In tailings dams where the pH has generally been adjusted to neutral, jarosite will tend to break down over time to goethite, releasing acid. The rate of this process, however, is extremely slow, as jarosites have been found as natural minerals.

e.g. $NaFe_3(SO_4)_2(OH)_6 \rightarrow 3\ FeOOH + \frac{1}{2}\ Na_2SO_4 + H_2SO_4$

4. CHEMISTRY OF GOETHITE, HEMATITE AND ALUNITE/JAROSITE FORMATION

4.1 GOETHITE AND HEMATITE FORMATION

In the absence of sulphate, the main stages in the thermal hydrolysis of iron can be described as (Feitnecht and Schindler, 1963):

Soluble iron	-->	Active Amorphous	-->	Inactive Amorphous	→	Crystalline Goethite	→	Hematite
$Fe^{3+}(aq)$		$Fe(OH)_3$		$Fe(OH)_3$		FeOOH		Fe_2O_3

Goethite is stable at ambient temperature but dehydrates to hematite above 130°C. The transformation of goethite to hematite results in zero net acid consumption.

$$2\ FeOOH + 3\ H_2SO_4 \rightarrow Fe_2(SO_4)_3(aq) + 4\ H_2O$$
$$Fe_2(SO_4)_3(aq) + 3\ H_2O \rightarrow Fe_2O_3 + 3\ H_2SO4$$

In the 1980s, the focus of research in the zinc industry changed from jarosite and goethite to hematite, as jarosite, in particular, became more difficult to discard, and hematite was seen as a more stable, inert and a possibly saleable product (Umetsu *et al*, 1977, Dutrizac *et al*., 1983a, 1983b, 1984,1985). This involved examining the hydrolysis of iron at temperatures higher than 100°C.

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Arauco & Doyle (1986) reported on pressure oxidative leaching at 110 to150^{o}C of zinc concentrate at Trail (The demonstration plant at Trail started around 1975 and the full scale plant went into operation in the early 1980's). In the presence of jarosite seed, jarosite was the major form of iron in the residue. However, losses of silver, copper and lead into the residue decreased markedly with increasing precipitation temperature.

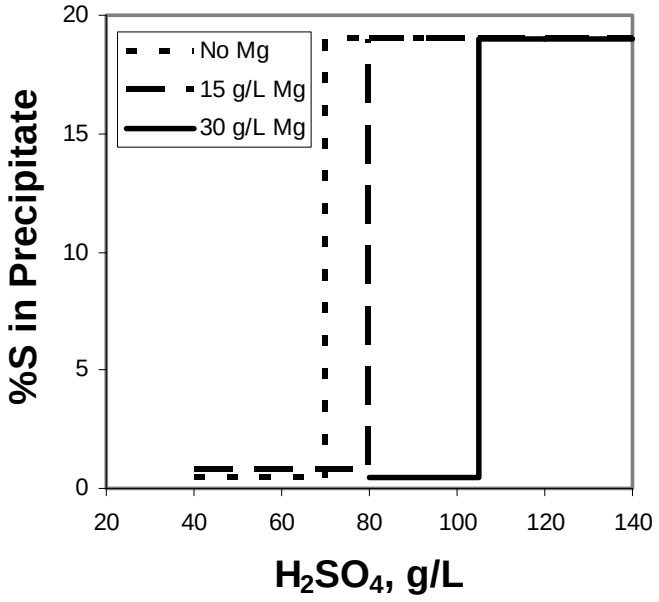
Tozawa and Sasaki (1986) examined the hydrolysis of ferric sulphate above 170^{o}C, and found that the product formed depended on acidity. At high acidity, a basic ferric sulphate $FeSO_4OH$ was favoured, whereas at low acidity hematite was the preferred product.

High Acidity ***Low Acidity***

$$FeSO_4OH + H_2SO_4 \leftarrow 2\,H_2O + Fe_2(SO_4)_3 + 3\,H_2O \rightarrow Fe_2O_3 + 3\,H_2SO_4$$

The addition of sulphate salts (Zn, Cu, Mg or Na) increased the stability range of hematite into higher acid regions. This resulted in lower sulphur content in the leach residue and lower overall acid consumptions (see Figure 3). Papangelakis *et al.* (1994) later showed that this was due to the effect of these ions on the acidity of the solution at temperature.

Figure 3 Effect of Magnesium and Acidity of Sulphur in Precipitates at 200^{o}C (Tozawa and Sasaki, 1986)



4.2 JAROSITE FORMATION

In acid ferric sulphate solutions, the equilibrium iron concentration in solution decreases sharply with increasing temperature (Walter Levy and Quemeneur, 1964,1966), due to hydronium jarosite formation at 50 to 140^{o}C, and basic ferric sulphate $Fe(SO_4)OH$ and hematite Fe_2O_3 formation at 150 to 200^{o}C .

The formation of both basic ferric sulphate and hydronium or metal jarosites involve a positive acid consumption –

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one mole of acid per mole of goethite for basic ferric sulphate:

$$2\ FeOOH + 3\ H_2SO_4 \rightarrow Fe_2(SO_4)_3(aq) + 4\ H_2O$$
$$Fe_2(SO_4)_3(aq) + 2\ H_2O \rightarrow 2\ FeSO_4OH + H_2SO_4$$

Two thirds of a mole of sulphuric acid per mole of goethite for hydronium jarosite:

$$6\ FeOOH + 9\ H_2SO_4 \rightarrow 3\ Fe_2(SO_4)_3(aq) + 12\ H_2O$$
$$3\ Fe_2(SO_4)_3(aq) + 14\ H_2O \rightarrow 2\ (H_3O)Fe_3(SO_4)_2(OH)_6 + 5\ H_2SO_4$$

Half a mole of sulphuric acid per mole of goethite for natrojarosite:

$$6\ FeOOH + 9\ H_2SO_4 \rightarrow 3\ Fe_2(SO_4)_3(aq) + 12\ H_2O$$
$$3\ Fe_2(SO_4)_3(aq) + Na_2SO_4 + 12\ H_2O \rightarrow 2\ NaFe_3(SO_4)_2(OH)_6 + 6\ H_2SO_4$$

The optimum conditions for, and the main factors affecting the formation of jarosite at atmospheric pressure have been determined by Dutrizac (1983a and 1983b). The optimum conditions for complete reaction in a relatively short time are 100^{o}C and pH 1.5.

At lower temperatures the reaction rate is very slow taking many hours for completion. This is because the hydrolysis of iron appears to be dominated by the relatively slow kinetics of hydrolysis, and the formation of metastable species, rather than by thermodynamic equilibrium. The simple iron oxides appear to form by gradual polymerization of semi-hydrolysed iron species, and the slow elimination of water and hydrogen ions from the hydroxo bridges (Dousma and de Bruyn, 1976, 1978). For jarosites, the sulphate present in iron-hydroxy-sulphato complexes probably becomes incorporated into the polymeric structure.

Other factors include counter-ion concentration (>1.2 times stoichiometric), seeding and to a lesser degree sulphate and iron concentration, ionic strength and agitation.

The ions Na^+, K^+ and NH_4^+ can be used to maximize iron precipitation as in the jarosite process, as these jarosites are less soluble than hydronium jarosite and more stable at increased temperatures (Haigh, 1967).

4.3 JAROSITE TO HEMATITE CONVERSION

The stability relations of jarosite are shown below in Figure 4. This figure indicates that jarosite should exist at acidities intermediate between those required to produce hematite and basic ferric sulphate. Stoffregen (1993) showed that at 150 to 250^{o}C, a sulphuric acid activity of 0.25 to 0.63 molal (about 20 to 60 g/L) was required to stabilize jarosite over this temperature range. He also showed jarosite could be formed from hematite under appropriate conditions at 200^{o}C but not at 250^{o}C.

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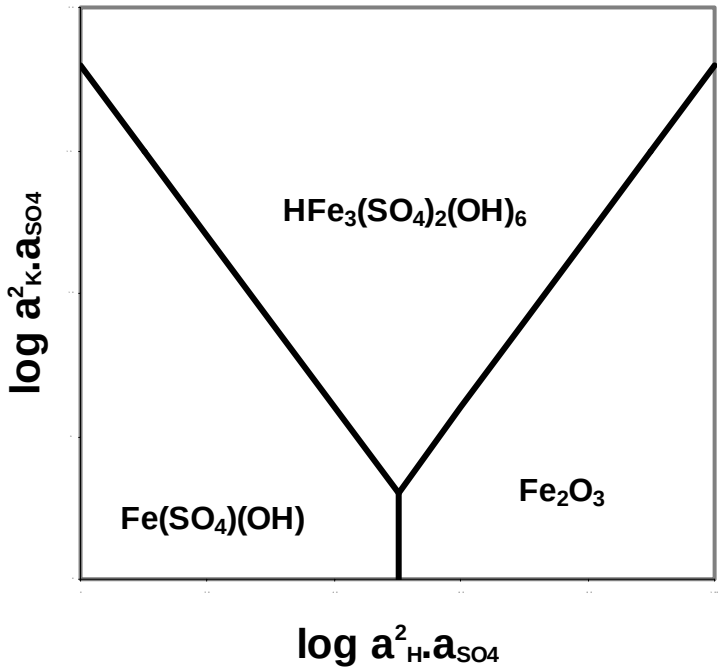
Figure 4 Stability relations of Jarosite, Hematite and Basic Ferric Sulphate (from Stoffregen, 1993)



Dutrizac (1990) has studied the conditions required for the hydrothermal conversion of sodium jarosite into hematite at temperatures greater than 220°C. With no free acid added, the jarosite was readily converted to hematite, and seeding with hematite assisted this process.

As the initial free acid concentration increased, the degree of conversion decreased until, at about 50 g/L free acid, the amount of hematite formed decreased rapidly and basic iron sulphate formation increased. In all cases studied, no sodium jarosite remained. Additions of sodium (46 g/L) as sodium sulphate appeared to have no effect on the decomposition reaction.

5. HIGH PRESSURE SULPHURIC ACID LEACHING OF NICKEL LATERITES

5.1 MOA BAY

High pressure sulphuric acid leaching (PAL) of nickel laterites has been carried out at Moa Bay in Cuba since 1959. The plant was started by the Freeport Sulphur Company and for a short time, until political changes caused this to cease, the nickel/cobalt sulphide product was shipped to their New Orleans Port Nickel Refinery for conversion to nickel and cobalt metals. The Moa Bay plant is still operating and has, since 1994, been managed by a Joint Venture with Sherritt , which includes the Sherritt refinery operations at Fort Saskatchewan in Canada. Until recently, these two processes remained the only proven processes for the total hydrometallurgical treatment of nickel laterites. The Moa Bay ore contains about 70% goethite, with which most of the nickel is associated. The other main gangue mineral is gibbsite (aluminium trihydrate, $Al_2O_3.3H_2O$), which comprises about 10% of the ore.

The ore is leached at about 45% solids with sulphuric acid at 246°C (475 °F). At this temperature, about 95% of the nickel and cobalt are dissolved as soluble sulphates, and over 99% of the iron is hydrolysed to hematite and rejected to the leach residue. Because of the

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low salt content of the process water, jarosite formation by the iron is minimal. Aluminium is mostly rejected to the leach residue as alunite, $MAl_3(SO_4)_2(OH)_6$, where M is either H_3O^+ or K^+. However, net aluminium extraction is 10 - 15%, and aluminium in solution in PAL discharge is 2-3 g/L. Temperatures of more than 240^{o}C are required for the efficient thermal hydrolysis of the iron and aluminium (Queneau and Weir, 1986).

5.2 AUSTRALIAN OPERATIONS

In recent times, three high pressure sulphuric acid leaching plants have been constructed in the Eastern Goldfields of Western Australia; at Bulong, Cawse and Murrin Murrin. The planning for these projects, and their histories following construction, have been well documented in the ALTA Nickel and Cobalt Hydrometallurgy Conferences from 1995 to the present, as well as in other places (see bibliography for a range of papers in this area).

These ores differed from Moa Bay ore in that they were classified as '"dry" laterites and contained significantly more nickel associated with aluminosilicate clays such as nontronite than the "wet" variety. In the Bulong and Murrin Murrin ores, more nickel is associated with nontronite than with goethite (Burger, 1996, Monti and Fazakerley, 1996) whereas at Cawse most of the nickel is associated with iron oxides rather than nontronites. The nontronitic nickel lay below the orebody and was generally sub-economic (Brand *et al,* 1996).

The following table indicates some typical compositions of gangue elements of Australian laterites compared to Moa Bay laterite (Kyle, 1996).

Table 2 Chemical Composition of Australian Laterites

	Bulong %	**Cawse %**	**M. Murrin %**	**Moa Bay %**
Fe	20.8	18.0	21.3	45
SiO_2	42.9	42.5	41.7	8.3
Al	2.75	1.71	3.16	4.8
Mg	4.62	1.58	3.28	0.55
Moisture	Up to 35%	Up to 10%	~30%	~35%

The main differences are the lower iron content, and higher silica and magnesium contents, of the Australian ores. This is due to the higher magnesium aluminosilicate and lower goethite content, especially in the Murrin Murrin and Bulong ores. In addition, the aluminium content has a differing mineralogy in the Moa Bay ore, being essentially the aluminium trihydrate gibbsite. The differing mineralogy of these ores, together with the use of saline to highly saline process water at Cawse and Bulong, has led to a different behaviour of iron and aluminium in the leaching process.

5.3 JAROSITES/ALUNITES IN RESIDUES

Whereas in the Moa Bay leach iron is precipitated mainly as hematite, Fe_2O_3, in the Australian operations using saline waters (Bulong, Cawse), hydronium and natrojarosites can be a significant component of the leach residue (Kyle, 1996). The amount of iron in the leach residue existing as natrojarosites has been estimated as about 3%, in 3% salinity process water, to 65% in 20% salinity process water (Kyle, 1999). The relative amounts of hydronium and natrojarosites formed will depend on the chemical composition of the process solutions in the autoclave. In addition, the Australian ores can contain a significant amount of iron(II),

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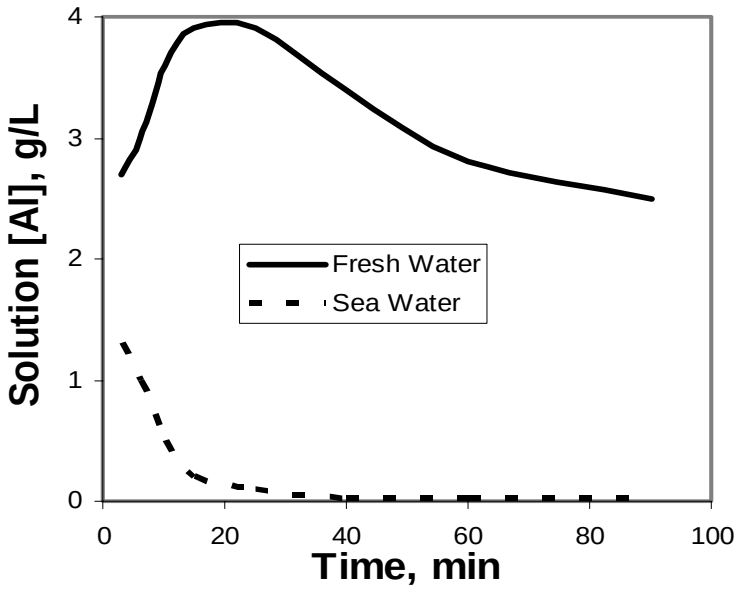
which needs to be oxidized and hydrolysed to hematite or jarosite, if possible, in the autoclave, to alleviate downstream processing problems.

The nature of aluminium in the leach residue also depends on the salinity of the process water. At Moa Bay, the aluminium exists in the ore mainly as gibbsite, and this is partly solubilised (about 30%) and partly hydrolysed to potassium and hydronium alunite (70%) in the PAL process.

In saline process waters, the insoluble natro-alunite appears to be the dominant aluminium hydrolysis product in the leach residue, also leaving less aluminium in the soluble form, with solution aluminium levels being reduced from 3 g/L to less than 0.5 g/L (see Figure 5, Krause *et al.,* 1998).

Figure 5 Differing Solubility of Aluminium in Fresh and Saline Water (Krause, 1998)



5.4 JAROSITES/ALUNITES IN SCALE

Moa Bay has always reported a significant scaling problem with their process, attributed mainly to hydronium alunite. Mass ratios of Al:Fe in scale at Moa Bay are often four times that found in the leach residue, with the iron being present mainly as hematite. In testwork at Lakefield Oretest on a tropical laterite, using potable process water, a concentration factor for aluminium in scale of five times was reported. In highly saline process waters, however, the ratio of Al:Fe remained the same in the feed, residue and scale (Czerny and Whittington, 1999). This indicates that jarosites, as well as alunites, are scale forming, and that the relative amounts of iron and aluminium in scale are dependent on the process conditions being suitable for the formation of jarosites and alunites rather than any other factor.

As the amount of jarosites and alunites formed during the high pressure sulphuric acid leach process increase, so do the scaling rates (Czerny and Whittington, 1999). This appears to be due to the greater scaling ability of natrojarosite, compared to hematite, and the greater precipitation of aluminium as natroalunite. However, the Australian operations have not experienced anywhere near the high scaling rates found at Moa Bay. This has been

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attributed to the higher degree of agitation in modern autoclaves as compared with the pachuca-style autoclaves at Moa Bay (Sobol and Zinoviev, 1984).

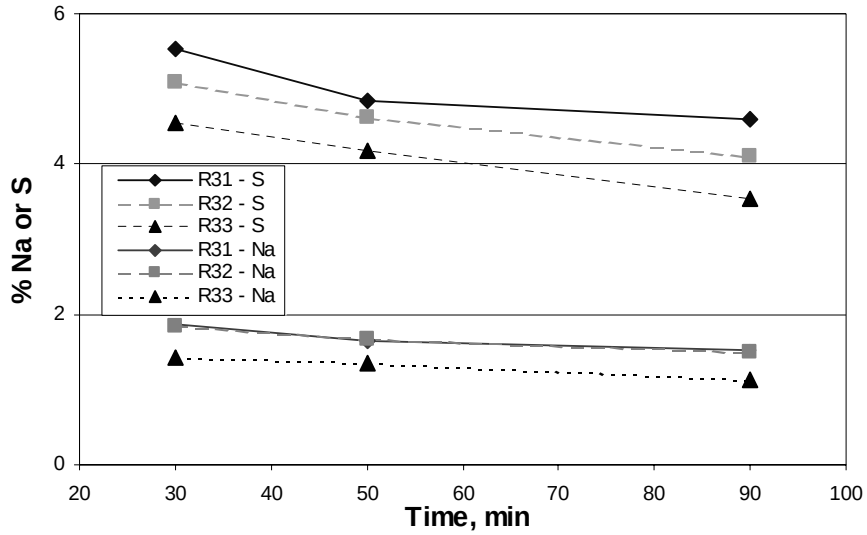
5.5 RESIDUE FORMATION IN PRESSURE ACID LEACHING

Chou *et al.* (1977) showed that the initial hydrolysis products on acid pressure leaching limonite ores were primarily basic iron and aluminium sulphates, which became unstable as leaching progressed, transforming into hematite and alunite. Johnson *et al.* (2002) confirmed that hematite in limonite acid pressure leaching residues increased with leaching time, and in saline waters alunite/jarosite was quickly formed but decreased in concentration with time, except at the highest salinities.

In recent PAL testwork on limonite ores from the Ravensthorpe Project (D White, 2003), timed samples of PAL residue at 30, 50 and 90 minutes were assayed for sulphur and sodium content, both indicators of natro-alunite/jarosite formation. The samples showed a slight decrease in both sodium and sulphur content in the residues with time, as shown in Figure 6. This slow change in composition with time is attributed to a slow transition of natro-alunite/jarosite to alunite and hematite. The fact that aluminium levels in solution continued to decline, while free acid continued to increase, is also indicative of this transformation occurring.

The transformation of goethite into hematite in the temperature range 220 to 250^{o}C has been studied by Tindall and Muir (1996). The transformation was negligible over 90 minutes at 220^{o}C, but complete within about 60 minutes at 250^{o}C. Increasing acidity aided the dissolution of goethite but hindered the hydrolysis of iron(III) and hematite precipitation.

Figure 6 Sodium and Sulphur in Residue versus Time (White, 2003)



The ideal acidity is therefore dependent on the interaction between these two competing processes, but also on other factors that can govern the “acidity at temperature”, such as dissolved salt concentrations (Papangelakis, 1994).

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The addition of salts such as sodium (~45 g/L Na) and magnesium (~6 g/L Mg) sulphate was found to enhance the rate of transformation of goethite to hematite, reducing reaction times at 250^{o}C from 60 minutes to less than 15 minutes. For sodium, the transformation occurring *via* the more stable intermediate natrojarosite appears to also have assisted in increasing the reaction rate.

Unexpectedly, the addition of aluminium (~10 g/L) and chromium (~0.5 and ~3 g/L) sulphate, however, completely stopped the reaction, even though these ions were significantly hydrolysed and precipitated, limiting their concentrations in solution. This was explained as both Al(III) and Cr(III) ions adsorbing strongly onto the goethite surface and most likely inhibiting the protonation and dissolution of goethite. These results need to be considered when optimising leaching conditions for real laterites, especially the effects of soluble aluminium and chromium on hematite formation.

Umetsu *et al.* (1977) also demonstrated that a range of iron compounds are formed during the acid pressure leaching of zinc ores, and that the sulphur content of the precipitates decreased at higher temperatures as more hematite, and less jarosite, was formed. This was critical for acid recovery. However, at very high temperatures (>280^{o}C), the high sulphur compound, basic aluminium sulphate Al(SO_4)OH, tended to form. As expected, hematite formation was also affected by high free acid concentrations (>55 g/L in the absence of zinc and 80 g/L in the presence of zinc).

5.6 METAL INCORPORATION INTO JAROSITE

As well as aiding in iron removal, jarosite precipitation can also lead to metal losses due to incorporation into the jarosite structure. This has been studied at 90^{o}C and at 97^{o}C (Dutrizac, 1984). Losses of divalent metals such as zinc and nickel to jarosite increase with increasing concentration of the divalent metal or iron(III), and increasing pH. This is due to the divalent metals replacing iron in the jarosite structure, with the charge balance being maintained by the loss of hydroxyl groups. Losses are greatest for the more stable jarosites ($K^+ > NH^{4+} > Na^+$) because incorporation of the divalent metals leads to a weakening of the structure. Losses vary for different metals but rarely exceed 3%. The order of stability for metals most prone to incorporation is the same as the hydrolysis constants:

$$Cu^{2+} > Zn^{2+} > Co^{2+} > Ni^{2+} > Mn^{2+} > Cd^{2+}$$

At temperatures greater than those studied, the loss of metals may be significantly different due to the differing relative stabilities of the various jarosites, and the varying solution composition. For example, Arauco and Doyle (1986) found that less silver, copper and lead were incorporated into jarosite residues from the pressure oxidation of zinc at 150^{o}C as opposed to 110^{o}C.

Incorporation of nickel and cobalt into jarosite/alunite scale has not been extensively investigated, but a survey of some of the available data for scale (see Table 3 below) indicates that it does occur in scale, and possibly more so in low salinity waters than high salinity waters, even though the proportion of alunite/jarosite in the low salinity scale is less (after Czerny and Whittington, 1999).

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Table 3 Nickel and Cobalt Content of Alunite/Jarosite Scale

Ore	Water	Major Scale Phases	Ni ppm	Co ppm	Reference
Limonite	**Fresh**	Hematite (40%) and Alunite (60%)	690	56	Sobol, 1966 and 1969.
Not stated	**Fresh**	Hematite and Alunite	700-2,000	<200	Krause et al., 1998
Limonite Nontronite Saprolite	**Hyper-Saline**	Natrojarosite/Natroalunite (>90%), Hematite (~5%)	330-390	<50	Czerny & Whttington, 1999
Tropical Limonite	**Fresh**	Hydronium Alunite (63%), Hematite (34%)	680-800	<50	Czerny & Whttington, 1999

6. SALINITY AND RESIDUE COMPOSITION IN PRESSURE ACID LEACHING OF LATERITES

6.1 SCALE COMPOSITION

A comparison of the scales formed in a continuous autoclave pilot plant by a Western Australian “dry” laterite processed in saline water (52 g/L Na) and a “tropical” laterite processed in potable water (0.08 g/L Na), has been undertaken by Lakefield Oretest and CSIRO (Czerny and Whittington, 1999). The major scale components, listed below in Table 4, show the dramatic difference in the scale-forming species in saline as opposed to potable water. Whereas in potable water, hydronium alunite is the main scale-forming element followed by hematite, they are virtually totally replaced in saline water by natrojarosite and natroalunite in solid solution. Hematite and silica are only minor scale components.

Jarosite/alunite scale was predominantly formed and deposited where the acid was added, in the first two compartments of the autoclave. Scaling was much lower in latter compartments. This is consistent with the fact that high acidity is required for the formation of these species, and that their formation can be controlled to some extent by staged addition of acid to the autoclave (Feteke *et al*., 1978).

Table 4 Major Scale Components in Saline and Potable Water

	Saline Water %	Potable Water %
Natrojarosite/alunite	92	0
Hematite	4	34
Hydronium alunite	0	63
Other (Silica etc.)	4	3

Hydrodynamics strongly influenced where scale was formed but did not significantly influence it’s composition. It formed on both static (autoclave walls) and moving (agitator blades) surfaces, with the latter experiencing 20-30% faster growth rates. Less scaling occurred with

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low salt content process water than for saline water. The scale occurred as large naroalunite/natrojarosite crystals with the interstices between crystals being filled with microcrystalline hematite and amorphous silica.

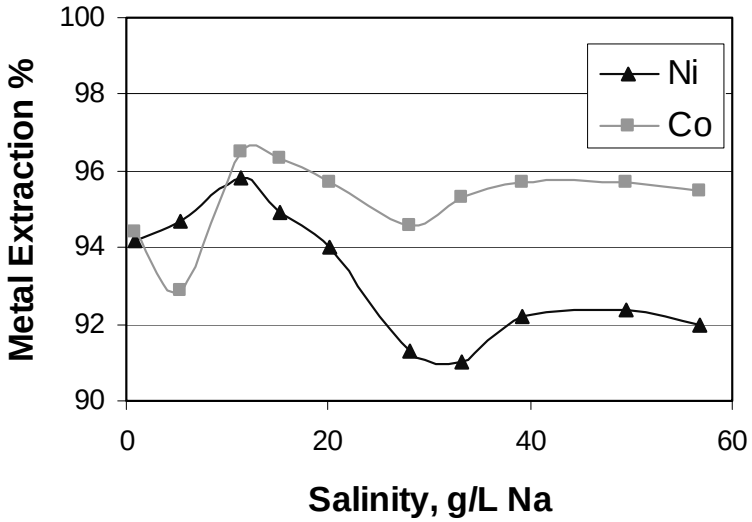
6.2 SALINITY, ACID CONSUMPTION AND METAL EXTRACTION

Recently, Johnson *et al.* (2002) have reported a systematic study into the effects of process water salinity on the leaching kinetics, element extractions, acid utilisation and residue composition during high pressure acid leaching of Cawse nickel laterite ore. They found that free acid levels in solution after leaching decreased as process water salinity increased, and concluded that acid usage increased due to the formation of alunite/jarosite, rather than hematite, in the leach residue. Their results showed that when moderately saline water is used in the leach (~15 g/L Na) rather than fresh water, about 16% (or 40 kg/t) more acid is used, rising to 45% (80 kg/t) at 40 g/L Na. This compares to previous work by Kyle (1999), who calculated, based on alunite/jarosite formation, that highly saline water increased acid consumption by around 40 kg/t at 25% solids.

Johnson *et al*. (2002) added the same amount of acid to leaches performed in fresh and saline waters and determined acid usage as acid added minus residual free acid at the completion of the leach. Acid usage changed because free acid changed.

However, the extraction of nickel and cobalt from the ore did not fall, except for nickel extraction at very high salinities (>28 g/L Na), which decreased from 94% to 91-92%. Up to 15 g/L Na, the extraction of nickel and cobalt in fact increased to 95-96%.

Figure 7 Metal Extraction versus Salinity (Johnson *et al*., 2002)



White (2003) has confirmed the above effects of process water salinity on free acid and metal extraction for Ravensthorpe beneficiated ore at relatively low salinities (9 g/L Na). In a set of tests on both limonite and saprolite ores, at 32% solids, the acid additions were kept constant while the water type was varied from tap water (potable) to saline water (75% seawater salinity or 9 g/L Na). For these tests, the results, summarized below in Table 5, demonstrate that at these salinities, metal extraction is not reduced even at the lower free acid levels

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present in the saline water. In fact, the similar metal extractions at lower residual free acid concentrations, is a bonus as it means lower processing costs through lower use of neutralization reagents.

Table 5 Metal Extractions at Constant Acid Addition in Saline and Tap Water

Sample	Head Assays				Acid Added Kg/t	Water Type	Free Acid g/L	Diss. Ni %
	Ni	Mg	Al	Fe				
Lim 01	1.58	0.65	4.69	29.2	295	Tap	42	98
						Saline	30	98
Lim 02	1.51	5.45	1.54	25.0	393	Tap	57	96
						Saline	52	95
Lim 03	0.62	4.76	5.46	19.4	426	Tap	32	90
						Saline	28	87
Lim 04	2.10	4.47	2.11	19.1	345	Tap	35	87
						Saline	22	90
Sap 01	1.05	12.2	2.25	14.2	620	Tap	39	96
						Saline	21	96
Sap 02	1.05	10.3	0.57	13.4	666	Tap	82	94
						Saline	90	90
Sap 03	1.33	9.00	1.48	20.6	546	Tap	75	94
						Saline	60	94

6.3 SALINITY AND RESIDUE COMPOSITION

The formation of alunite/jarosite leads to an increased mass of the residues, due to the incorporation of extra water and sulphate into the jarosite structure, compared to the dehydrated hematite structure. This is well demonstrated in a separate series of PAL tests on eleven samples of ore from the Ravensthorpe deposit, carried out in both saline and potable (tap) water. The test conditions, except where stated, were at 32% solids for 90 minutes, with acid additions (except for Tests R46 and R47) varying for the tap and saline water tests (White, 2003). The data are shown in Figure 8.

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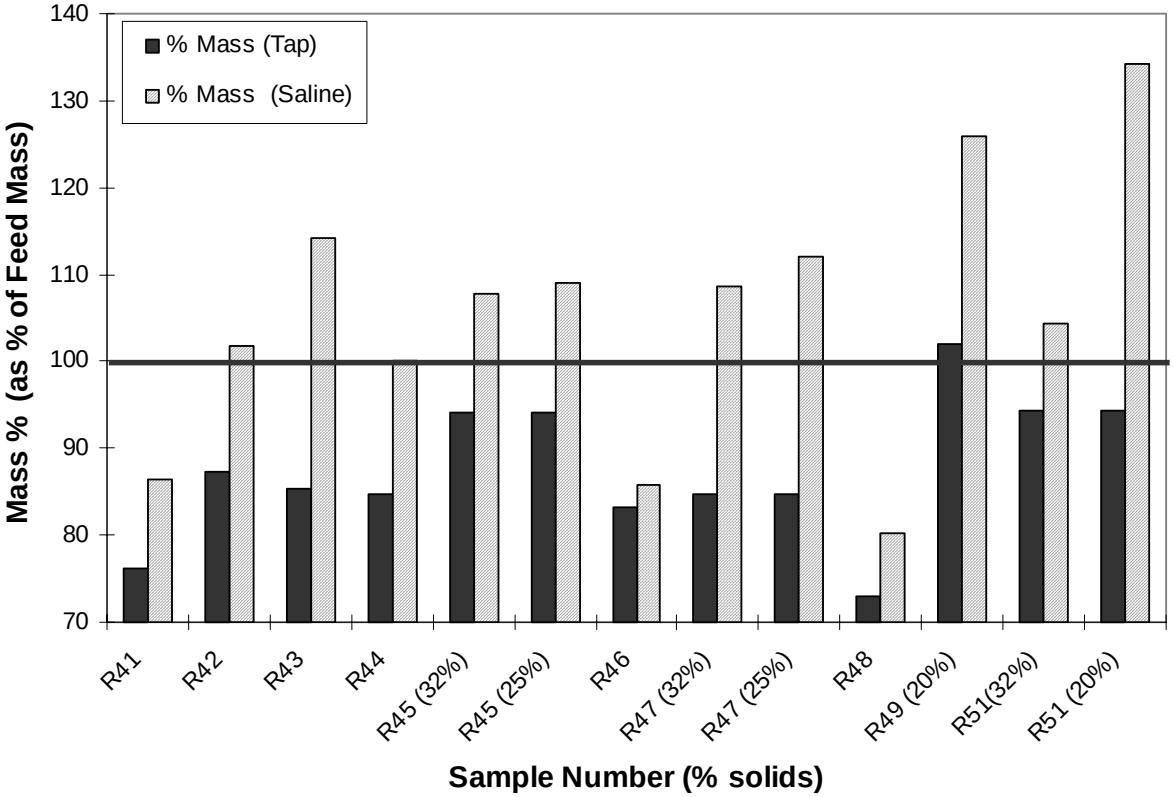
Figure 8 Mass of PAL Residues for PAL Tests in Tap and Saline Water



A number of tests, all in constant salinity water (~9 g/L Na) were done at lower percentage solids (20% and 25% versus 32% w/w). In all cases, the amount of solids produced was greater relative to the feed solids mass than in tests done at higher salinity. This was due to the greater amount of saline water in the leach relative to the solids content, leading to an increased amount of alunite/jarosite formation, as evidenced by an increase in sulphur and sodium levels in the residues (see Table 6).

Table 6 Residue Composition for PAL Tests at Various Percent Solids

Sample	Solids Conc % w/w	Acid Added Kg/t	Free Acid g/L	Water Type	Residue Composition		
					% Mass Increase	Na %	S %
R51	32	385	16	Saline	4	2.65	8.43
	25	417	11	Saline	42*	2.90	9.76
	20	449	11	Saline	34	3.41	10.2

* This result is possibly too high due to experimental error.

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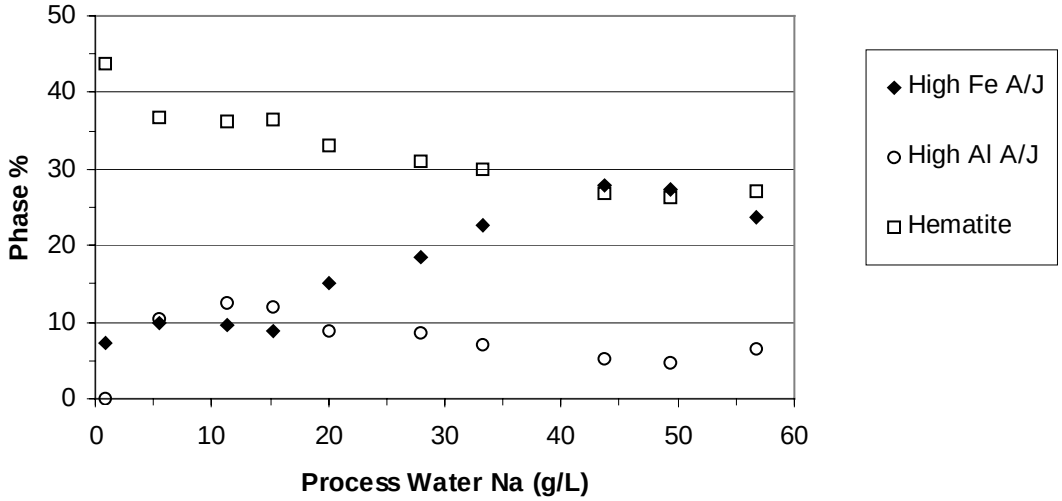
Johnson *et al*. (2002) found that the formation of hematite and high-iron alunite/jarosite phases in the residue were clearly affected by process water salinity (see Figure 9). Hematite formation was greatest in low salinity process water, but decreased with increasing salinity as more high-iron alunite/jarosite was formed. The amount of high-aluminium alunite/jarosite remained relatively constant, but the percentage decreased slightly as the total weight of residue increased.

Figure 9 – Effect of salinity on residue composition (after Johnson *et al.*,2002).



The pivotal role of aluminium content in the formation of alunite/jarosite is well demonstrated in a series of 16 batch PAL leach tests performed on a range of limonite ores from the Ravensthorpe deposit (White, 2003). The tests were carried out under nitrogen at 36.4% w/w solids density in water of 75% seawater (~9 g/L Na) salinity at 250^{o}C for 90 minutes. Other details of the test conditions are outlined below:

Ni %	Mg %	Al %	Acid Kg/t	Mass Loss %	Free Acid g/L	S in Residue %
0.98–1.93	0.9 – 4.0	0.5 – 4.4	135 - 331	0 – 15.8	22 - 46	0.9 – 4.6*

*This amount of sulphur implies the residue contains 7 - 35% alunite/jarosite.

In general terms, the acid addition was directly related to magnesium and aluminium content, as was the sulphur content of the residue. The mass loss was inversely related to residual sulphur. In terms of the residue composition, the most definitive relationship was between the aluminium and sulphur contents in the residue (see Figure 10 below).

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Figure 10 Mole% Aluminium vs Mole % S in PAL Residues

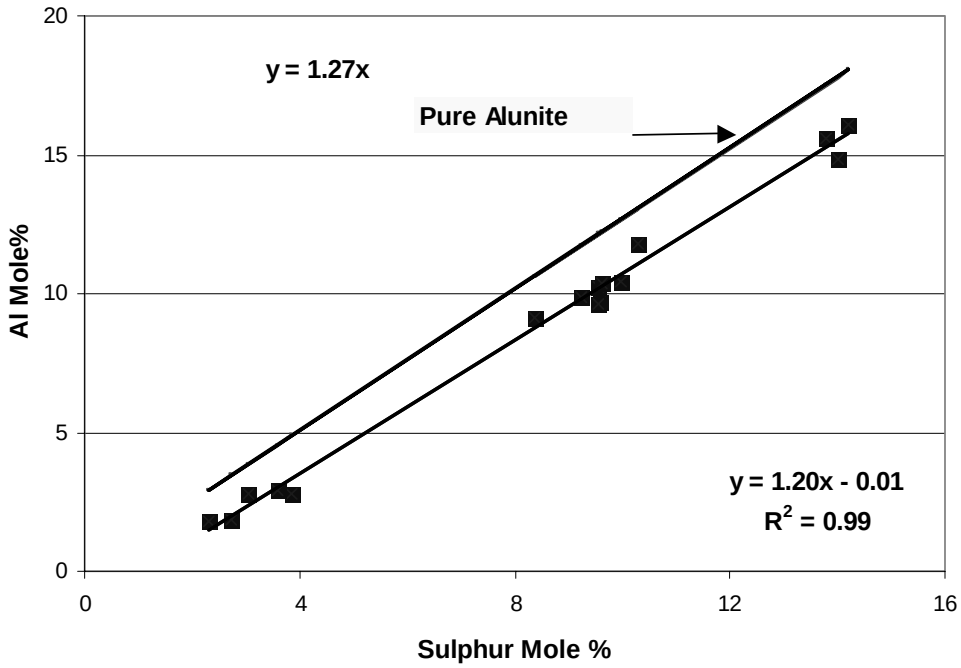

Figure 10 shows the very strong linear correlation between aluminium and sulphur in residues, and is compared with the theoretical relationship for pure alunite. This shows that the slope of the line of best fit, and the mole percent aluminium, was slightly lower than that required for pure alunite, indicating some iron was present in the alunite structure, in solid solution with the alunite.

This strong correlation, together with the poor correlation between iron and sulphur in residues (not shown), also indicated that the aluminium content determined the amount of alunite/jarosite formation. The jarosite was not formed independently of the alunite but was rather formed by iron substituting for aluminium in the alunite structure.

The mole percentages of iron and aluminium in alunite/jarosite as a function of the mole percent alunite/jarosite are shown below in Figure 11. This assumes all aluminium and sulphur in the residue are present as alunite/jarosite, and any sulphur not associated with aluminium is associated with iron in the alunite/jarosite matrix.

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Figure 11 Mole % Fe and Mole % Al in Alunite/Jarosite

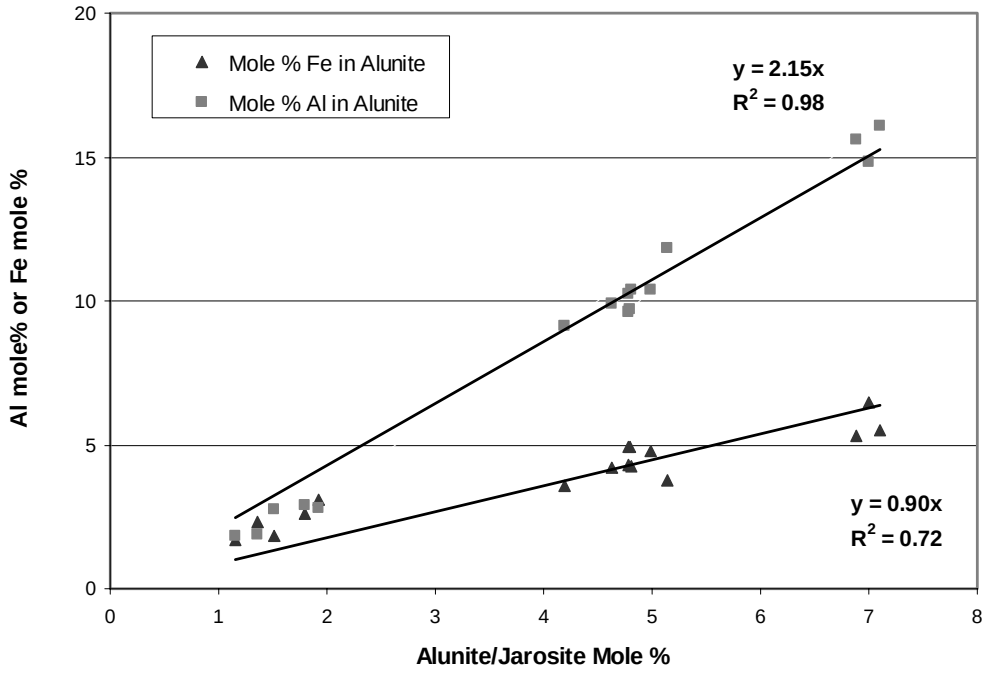

Figure 11 indicates that the molar ratio of aluminium to iron in alunite/jarosite was relatively constant at about 2:1, except at low aluminium levels. At low aluminium levels, iron levels were higher relative to aluminium, so that the ratio was closer to 1:1.

A strong correlation also existed between sodium and alunite/jarosite in residues (see Figure 12). The sodium content is lower than is required for pure alunite/jarosite by about 30% at low alunite/jarosite content to about 10% at high alunite/jarosite concentrations, indicating most of the aluminium and also iron were present as the natro-alunite/jarosite, and the remainder existed mostly as the hydronium alunite/jarosite. Mass balancing indicated the sodium deficit averaged only 13% over the 16 tests.

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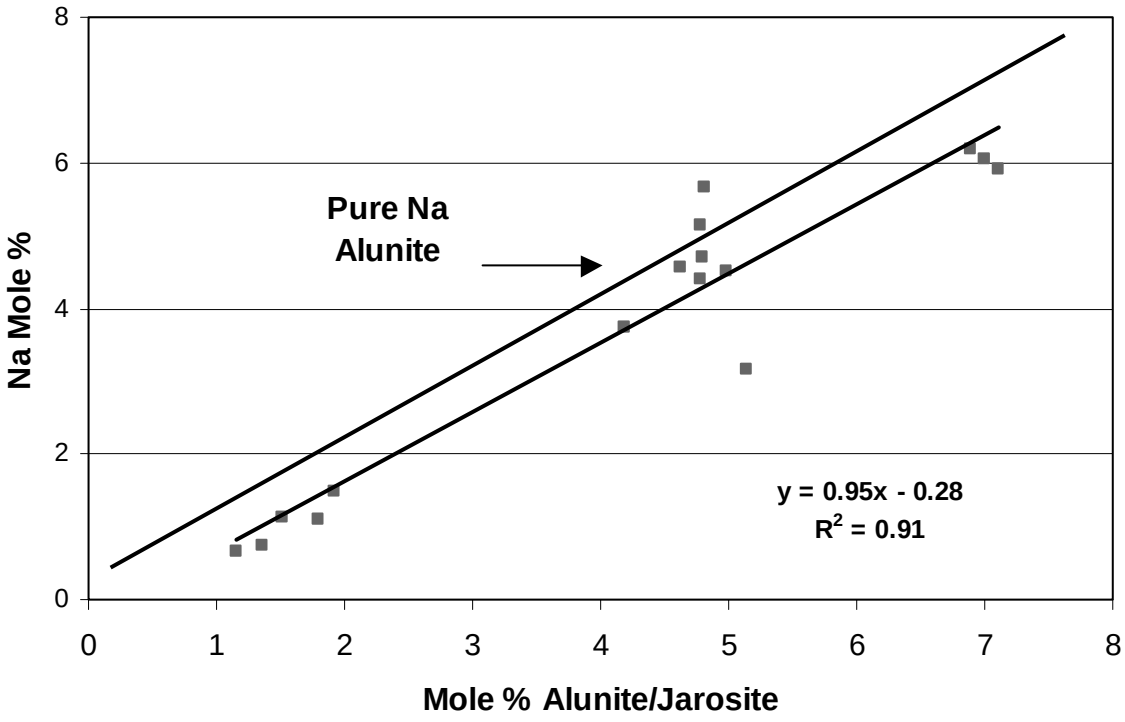
Figure 12 Mole % Sodium vs Mole %Alunite/Jarosite



6.4 RESIDUE COMPOSITION VERSUS FREE ACID

White (2003) also investigated the effect of residual free acid concentration on the residue composition. One sample of Ravensthorpe beneficiated ore was subjected to a number of PAL tests at different acid additions producing different free acid levels in the residual solution. There was only a slight increase in sulphur and sodium, and therefore alunite/jarosite, in the residue as the residual free acid levels increased from 18 to 23 g/L. Above 23 g/L, the amounts remained unchanged as free acid levels increased (see Figure 13).

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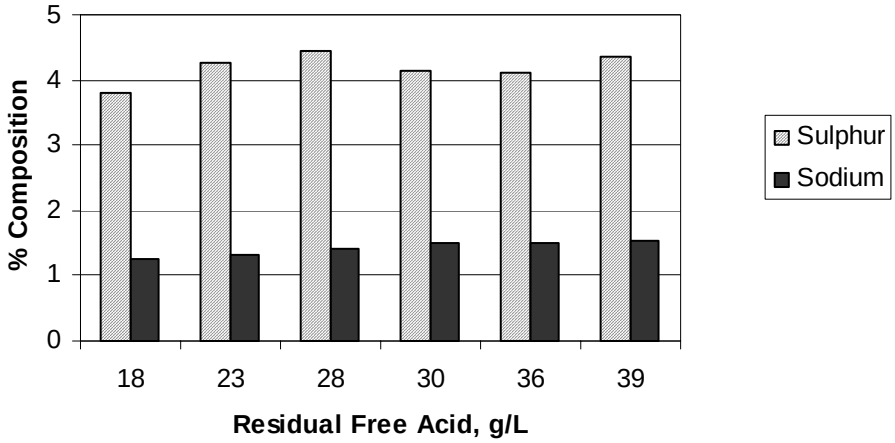
Figure 13 Sodium and Sulphur versus Residual Free Acid



6.5 RESIDUE MORPHOLOGY

SEM and microprobe studies showed relatively large crystals of alunite/jarosite compared to fine particle agglomerates of hematite and silica. Alunite and jarosite existed in solid solution but hematite and silica were not intimately associated (see Figure 14, from Johnson *et al,* 2002).

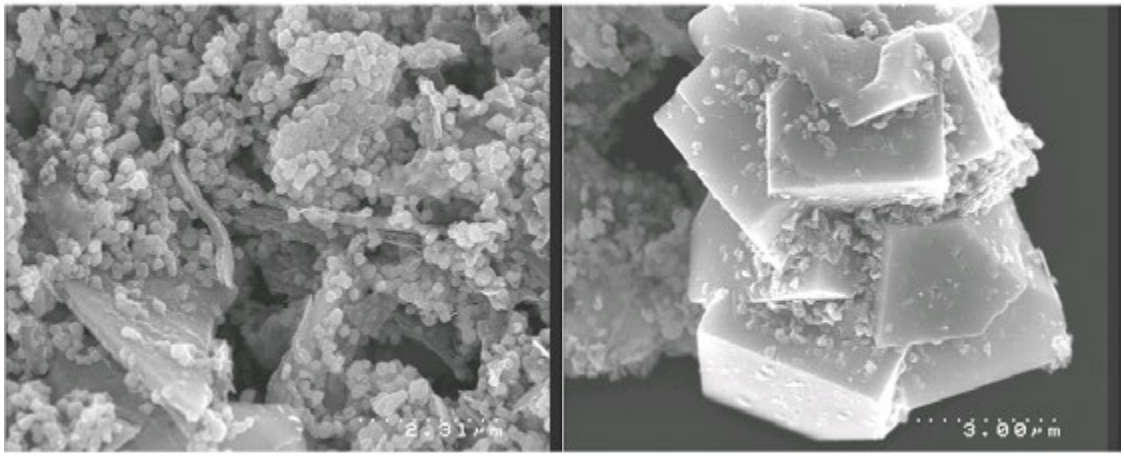


Figure 14a – Typical SEM Micrograph of Leach Residue (tap water)

Figure 14b – Typical SEM Micrograph of Leach Residue (hypersalinewater)

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Rubisov and Papangelakis (2000) have investigated the effect of terminal solution acidity on the morphology of the hematite residue at 270°C in potable process water. They found that the platelets of hematite, similar to those shown above in Figure 14a, and about 350 mm in diameter, that are formed at lower terminal acidities (~20 g/L), were replaced by smaller and more spherical shaped particles of about 200 mm diameter.

The shape of the particles is important for their settling and filtration behaviour. Platelet particles settle and filter more slowly than spherical particles. In comparing different laterite blends, the *"acidity at temperature"*, which depends on the solution composition and is derived from a solution model developed by the authors, was found to be a more sensitive measure of hematite shape than terminal acidity. This *"acidity at temperature"* needed to be greater than the point of zero charge (PZC) of the hematite in order to prevent platelet hematite formation that occurs when there is a predominance of hydroxyl ions on the surface.

7. ATMOSPHERIC PRESSURE LEACHING OF LATERITES

The use of jarosite precipitation for iron removal at atmospheric pressure has been known for some time, and has been extensively used in the zinc industry (Gordon and Pickering, 1975; Arregui *et al*., 1980).

Until recently, the use of atmospheric leaching and jarosite precipitation for nickel laterites has been relatively poorly understood. Hatch and Dunn (1983) showed that high extractions of nickel and cobalt could be obtained by atmospheric leaching high magnesia serpentine fraction of laterite ores with sulphuric acid in the presence of a reducing agent such as sulphur dioxide gas.

Maraboutis and Kontopolous (1988) investigated iron removal from atmospheric leach solutions by jarosite precipitation from both synthetic and real solutions obtained by atmospheric heap leaching of low-grade Greek laterites. They confirmed that iron removal by jarosite precipitation was feasible from such solutions at pH 1.6 and 95°C. The lowest residual iron levels of less than 1 g/L were obtained using potassium as a counter-ion, although the use of sodium gave similarly low levels. When pH was controlled at 1.5 to1.6 with limestone, reaction times were less than 4 hours. Losses of nickel and cobalt were generally less than 3% and 5% respectively, while more manganese (~10%) and aluminium (~40%) were incorporated into the jarosite precipitate. The filtration characteristics of the jarosite precipitates were described as "excellent in all cases".

More recently, the atmospheric tank (Arroyo and Neudorf, 2001) and heap (Duyvesteyn *et al.,* 2001) leaching of nickel laterite ores with sulphuric or other acids (Duyvesteyn and Liu, 2002), as a stand alone process or in conjunction with PAL (BHP, 2001, Arroyo and Neudorf (2001), Arroyo *et al.* (2002) has been the subject of significant research and a number of patents by BHP, now BHP-Billiton, and others (e.g. Curlook, 2002).

Arroyo and Neudorf (2001) initially leached limonite ore (usually >25% Fe and 0.5-0.6% Mg) in seawater at about 95°C with high concentrations of sulphuric acid (>1:1 by weight). Following this, saprolite ore (usually 5-20% Fe and at least 8% Mg) was added to lower the acidity to about 5 g/L while leaching nickel and cobalt from the ore. The lower acidity and presence of sodium ions, together with the addition of jarosite seed, induced the formation of natrojarosite from the iron sulphate solution. Further saprolite was added as acid was released to solution by the jarosite precipitation reaction. If the redox potential in the pulp was controlled to less than 900 mV with sulphur dioxide then nickel and cobalt dissolution was greater than 90%.

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Total reaction times for this process, including limonite leaching, pH adjustment and jarosite precipitation were generally 8 – 9 hours, and optimum nickel and cobalt extractions from Gag Island ore were 91% and 95% respectively. Metal concentrations in the jarosite residue were low compared to zinc industry jarosites at 0.15% Ni and <0.01% Co, and residue leach tests showed virtually zero release of nickel, cobalt and chromium over 16 leach cycles. The residue leachant was not detailed in the paper, however, as calcium, magnesium and manganese were released over about seven cycles, it is assumed the leachant was water (BHP, 2001).

Atmospheric leaching, in conjunction with PAL, has also been extensively investigated by BHP Billiton and a process patented (Arroyo *et al.,* 2002). In this process, the limonite ore is subject to PAL and the saprolite ore is leached at atmospheric pressure with the PAL discharge slurry to solubilise contained nickel and cobalt, and precipitate soluble iron in the presence of an alkali ion as a jarosite. BHP-Billiton propose to use this process to exploit the Ravensthorpe deposit.

Heap leaching of laterite ores has been the subject of BHP patents, including a process for palletizing the ore prior to sulphuric acid leaching (Duyvesteyn *et al.*, 2001) and heap leaching using microorganisms (Duyvesteyn and Liu, 2002).

BHP Billiton see the main advantages of this atmospheric leaching process over the conventional high pressure acid leaching as having a lower implementation risk (even though it is a commercially untried process), better economics and lower capital requirements, and also having a better utilization of the laterite ore, in that both limonite and saprolite ores could be used as part of the process.

Atmospheric heap leaching of a number of laterite ores is currently being studied for a number of projects, but to date little has been published in this area.

8. SUMMARY

The first plant incorporating PAL of nickel laterite ores produced predominantly hematite residues with hydronium alunite in both the scale and residues. Two of the recent Australian projects incorporating PAL have utilized saline (Cawse) and hypersaline (Bulong) process water resulting in significant changes to the iron and aluminium compounds formed in the residue. For Cawse, although hematite is still dominant, due to the moderate salinity and low aluminium content of the ore, there is significant natrojarosite and natroalunite in solid solution. For Bulong, however, natrojarosite is the dominant form of iron in the residue, and aluminium exists in solid solution in both high iron natrojarosites and high-aluminium natroalunites. Testwork on the Ravensthorpe deposit confirms that at seawater salinity, the residue will be similar to the Cawse residue.

The use of hypersaline water has resulted in higher acid consumptions, through more jarosite formation, and increased acid costs, but advantageously lowers aluminium levels in solution.

Lower salinity waters (seawater quality or less) appear to offer not only lower aluminium in solution, due to natroalunite formation, but also slightly improved metal extractions at the same acid additions as freshwater. In addition, terminal free acids will be less than in fresh water, leading to decreased neutralization costs.

These advantages of lower salinity process waters have led to sea water being considered for the Goro plant in New Caledonia and being chosen for the Ravensthorpe project in Western

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Australia which involves both pressure acid leaching combined with atmospheric leaching, and iron removal by natrojarosite formation.

Although jarosite processes have been extensively studied and used in the zinc industry for many years, there has over the last twenty years been a move to the more environmentally friendly hematite processes. At the same time, nickel laterite processing appears to be moving from hematite to jarosite processes.

Although there are significant costs involved in forming jarosite rather than hematite residues, including extra acid consumption (in some instances), more thickener area, and increased tailings volumes, the overall costs of processing are lowered by using the cheaper (and often only available) saline waters.

The jarosites produced by nickel laterite processing have less contaminants than those produced in the zinc industry. However, the movement from hematite to jarosite residues does appear to contradict the movement toward overall waste reduction in metals processing, and the formation of usable by-products in the place of tailings. This may limit the use of jarosite processes to regions where tailings disposal is cheap and large land areas are available for the disposal of residues and the evaporation of excess saline process waters.

9. ACKNOWLEDGEMENTS

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PRACTICAL ASPECTS OF RHEOLOGY AND THE APPLICATIONS IN NICKEL PROCESSING

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ABSTRACT

Quantifying and understanding the rheological performance of slurries is critical to optimising plant performance, particularly in relation to milling, screening, pumping, agitation and thickening applications. Most laterite and sulphide nickel processing plants try to achieve optimum throughput rates based on a solids throughput rate, which is always related to a slurry solids concentration. The rheological performance of the slurry is what drives the optimum solids concentration and will vary across each process area within the plant.

This paper presents a series of rheological trends that have been generated aimed at optimising the solids throughput rate for several different operations. Applications will include both limonite and saprolitic ores as well as sulphide ore types. Emphasis will be placed on how these trends can then be applied to practical plant situations to improve the operating efficiency. The importance of rheological characterisation of slurries during the design phases of a project will also be discussed.

Factors that significantly effect rheology such as water quality, clay mineralogy, clay dispersion and hydration, effect of slurry temperature, effect of flocculated versus non flocculated systems and the effect of varying degrees of applied shear on slurries will all be presented.

The paper will also attempt to dispel some of the myths around rheology in the mineral processing industry, particularly importance of shear history and temperature effects.

1 INTRODUCTION

Probably the most misunderstood rheological term is "**viscosity"**. Many people think of viscosity as a physical property such as SG, temperature or pH.

Viscosity is a **measure** of the **resistance to flow** and is in effect the proportionality between shear stress and shear rate. This clearly implies that viscosity will vary with shear rate. Shear rate is the **rate** of deformation of a fluid and is dependent on the force or energy applied to the slurry. Therefore viscosity should be considered as a variable dependent on shear rate and temperature and should be quoted at a given shear rate and temperature.

For measurement purposes, shear stress is measured at known shear rates and the viscosity is calculated at each shear rate. These individual points can be graphically represented as a viscosity curve which allows prediction of shear stress or viscosity at non – measured shear rates.

From a practical perspective, a viscosity curve (sometimes referred to as a rheogram) allows us to determine the resistance to flow through various stages of the plant and therefore allows, for example, a prediction of pressure drop in pipes or the critical slurry viscosity through a set of screens. These curves are drawn for a range of solid concentrations and should be matched to process design criteria.

This paper looks at several viscosity trends that have been generated from laboratory and plant data and the process implications that arise from understanding and interpreting these trends correctly.

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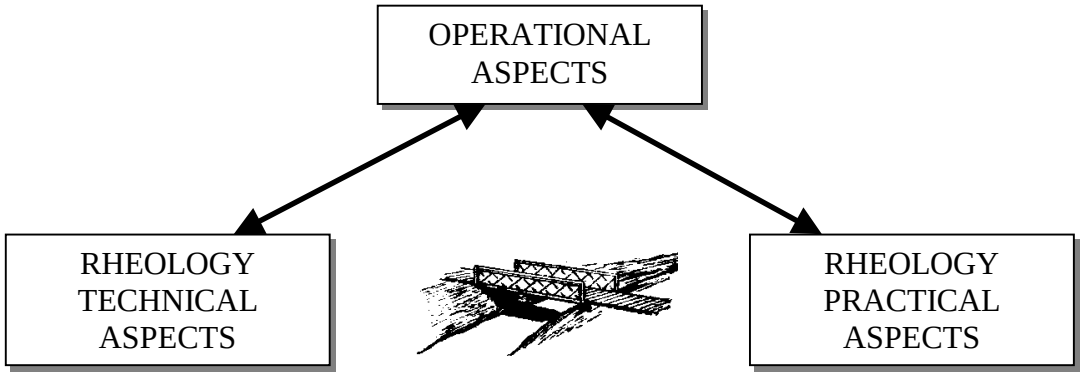
2 RHEOLOGY CONCEPTS

Rheology is too high or out of control. This is a common phrase used in the mining industry to describe a fluids rheological . How can you understand a slurry which sometimes gets thinner when you apply energy, then gets thicker, when the energy is removed, or vice versa. For a mineral slurry add this phenomenon with other variables such as particle type, particle size distributions, geochemistry, mineralogy and some infinitely expandable modeling concepts and you can begin to understand the rheology of the slurry and what aspects you can and can't control.

Rheological data would be easier to interpret if all the data was relative, collected from the same type of instrument using the same conditions and the same theoretical model. In the minerals industry, there are currently several types of instruments producing data for a diverse range of slurries. This data can be interpreted using a variety of mathematical models with very different results.

Different viscometers or viscometer configurations are appropriate for various viscosity ranges in the same way that pressure or temperature gauges have an operational range. To gain an understanding slurry rheology, it is necessary to first determine the appropriate measuring device and technique. The produced data should then be fitted to a mathematical model that accurately describes the data.



It is easy to identify where rheology affects plant operations but linking the measurement of rheology to process design or improving plant performance is the biggest bridge to cross.

From a rheological perspective, slurry processing can be divided into three main areas, high shear, low shear and ultra low shear applications. High shear applications involve vigorous slurry movement such as pumping and pipeline transport, where as low shear applications mostly involve areas of the plant with slow slurry movement such as thickeners and screens.

High shear rheology data is often extrapolated for applications which require much more precise low shear measurement. Similarly, how a slurry behaves at high shear cannot be accurately modeled using low shear data.

A case in point is yield stress. All pseudoplastic (or deformable) slurries have a yield stress (τ_y). It is also referred to as yield value and yield point. This point is defined as the torque or power required to initiate flow of a slurry, yield stress, and is determined using either direct measurement techniques or modeled data.

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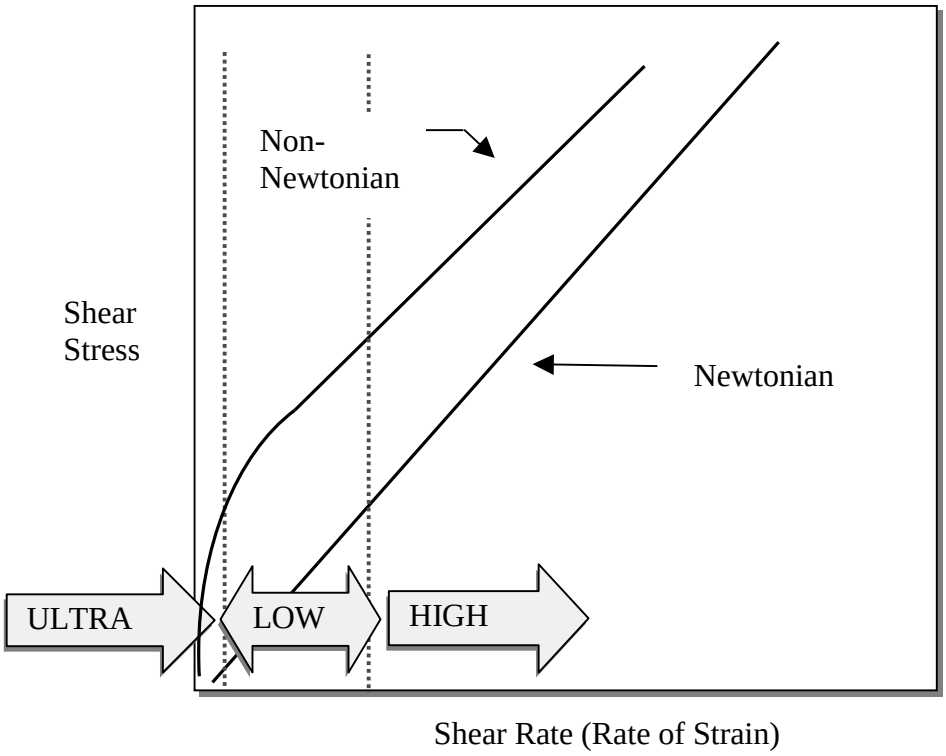
Fitting the same rheological data to different models can lead to different yield stress or comparative values. The accuracy of the determined yield stress is dependent on the applicability of the model and the derivation of the value should be carefully considered before utilising a curve derived model parameter for engineering calculations.

The rheogram of a Newtonian fluid is a straight line which passes through the origin. The viscosity is defined by the slope of the line. Rheograms for non Newtonian fluids are more complex. Mineral slurries are non Newtonian and are often described as “plastic” fluids, see Figure 1.

Figure 1 Rheograms for Non Newtonian versus Newtonian Fluids



The simplest model is the Bingham Plastic model which predicts that flow will not begin until applied shear stress overcomes a limiting value defined as yield stress. For this model the viscosity is defined by the tangent to the line as shown in the following graph. The Bingham model extrapolates high shear values towards the y axis.

This translates linear behavior in the high shear region into the low shear zone. This results in the y intercept value or Bingham Yield point overestimating the true yield stress value and it does not accommodate for non linear behavior in the low shear zone as shown in Figure 2.

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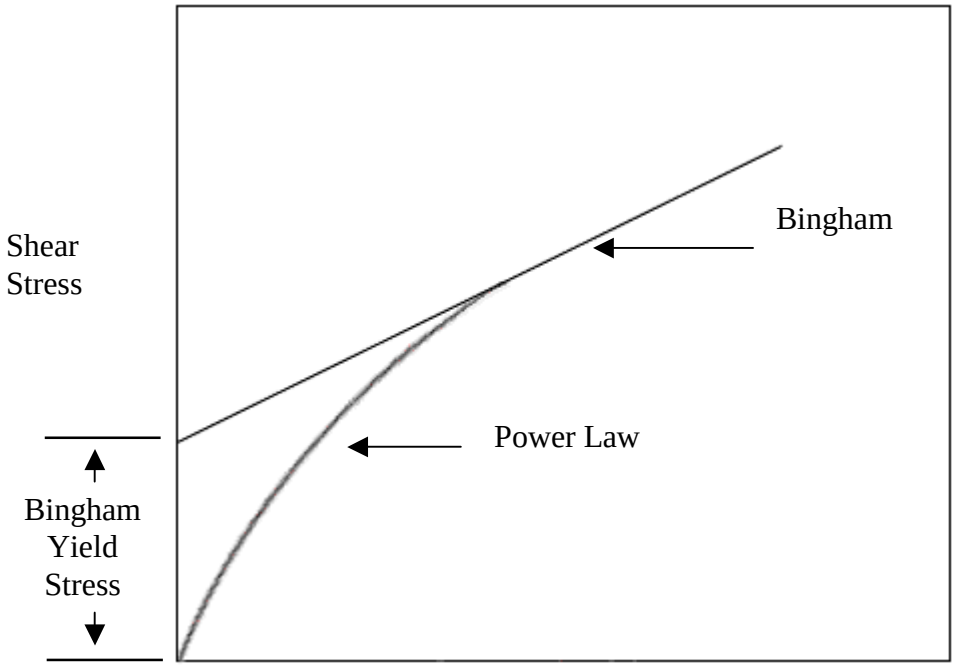
Figure 2 Bingham versus Power Law Rheograms



The Power Law model provides much greater accuracy in determining the shear stress at low shear rates. This model assumes all slurries are pseudoplastic.

The main disadvantage with the power law model is that it says any fitted curve must pass through the origin ie when the shear rate is 0 sec^{-1} the shear stress is 0. This doesn't fit as any fluid has an inherent viscosity in the static state (zero shear). Therefore the power law model better describes the low shear rate zone up to 1 inverse second but not between 0-1 sec^{-1}.

The Herschel Bulkley and Casson models are extensions of the power law model and allows for non linear shear stress-shear rate relationships. These models acknowledge that a slurry may have a yield value at very close to zero shear rates. All models that utilise data generated from cup and bob configurations will suffer from slippage at the bob/cup slurry interface in the low shear zone, particularly for thicker slurries. This ultimately effects the reliability of the data.

Rheochem have found that no single model can be used to predict the true yield stress of a slurry across a range of sample types. It is for this reason that the Vane yield stress method is recommended to determine the true yield stress of a slurry, particularly at high solids concentrations. This technique ensures that slip does not occur between the vane and the slurry surface, therefore providing a measurement of the true stress required for the slurry to irreversibly deform and yield.

Table 1 shows a comparison of "true" yield stress and modeled yield stress values obtained for a number of different limonite and saprolite nickel slurry samples.

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Table 1 Comparison of True Yield Stress versus Modeled Yield Stress

Sample	True Vane Yield Stress Pa	Modeled Yield Stress from Rheograms Pa		
		Bingham	Herschel Bulkley	Casson
L1	121	191	131	144
L2	100	140	41	123
L3	105	181	32	127
L4	126	203	102	158
S1	110	136	98	106
S2	100	115	101	88

In all cases the Bingham model has over estimated the yield stress to varying degrees. The Herschel Bulkley or Casson models sometimes provided a better correlation to the true yield stress, but no single model could be used to predict the yield stress for the samples tested.

Once users of rheology information understand the basic concepts of slurry at different points in the plant then testwork considerations can be implemented to best represent plant conditions.

3 RHEOLOGY AND PROJECT DESIGN CONSIDERATIONS

Most process plants are designed to handle a certain flow rate at a given solids concentration.

For each section of a process there is a limiting flow rate dictated by pressure drop. If, for instance, the allowable pressure drop is exceeded in a thickener, the end result is often a “donutting” effect. For pumps this can cause cavitation by either suction or discharge pressure drop over a given length of pipe work. In tanks it is usually a mixing efficiency issue and for screens it is a flooding problem.

In recent years due to the move towards treatment of lateritic ores, viscosity issues have become more prevalent. Conflict only arises when the plant cannot maintain desired solids throughput.

Understanding the limitations of each unit process through understanding the rheology mechanisms involved is critical for correct plant design and operation.

3.1 ORE MINERALOGY

A basic understanding of an ore type’s mineralogical components is highly recommended to assist with rheological testing. Particular areas of interest include a breakdown of any clay components present including platelet orientation as shown in Figure 3.

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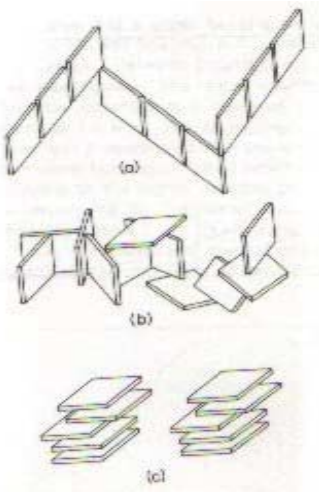
Figure 3 Clay Platelet Orientation

Edge to Edge

Edge to Face

Face to Face Stacks



This information, combined with zeta potential and cation exchange capacity data is a very useful tool for characterising slurries prior to testing.

3.2 PARTICLE SIZING

Particle size analysis and shape, particularly at the sub 10 micrometer level is an important tool that can be used to distinguish between various clay components that can affect the rheological performance of a slurry. Among the factors that determine the viscosity of the slurry are the fineness of the clay particles in the natural state and the degree to which the particles and aggregates are cleaved or dispersed during the hydration process.

Often a determination of the particle-size distribution may indicate only the degree of disaggregation and not represent the particle size distribution of the material in its original state. Combined with sub micron size analysis, measuring the “volume fraction” in a dispersed slurry can be a useful tool for determining the way a particle will behave (swell) from the initial stages of hydration through to full hydration.

3.3 TEMPERATURE

The effect that temperature has on slurry viscosity needs to be considered particularly around the low shear range experienced in screening and thickening circuits and particularly if there is potential to introduce heat into a circuit utilising excess steam capacity. Factors such as time dependent properties, shear dependency, and effect of temperature on flocculation need to be taken into account as these will effect the type of measurement that needs to be used to quantify the effect that temperature has on rheology.

3.4 WATER QUALITY/CLAY HYDRATION

The viscosity of clays can be significantly affected by the presence of soluble salts in the process water. It is critical to use the correct process water during the testwork phase.

The adsorption of water by ores leads to swelling of the clay particles which effects the rheological performance of the slurry. The magnitude of the hydration varies significantly depending upon the kind of clay, size of the clay particles, amount of clay present and the salt

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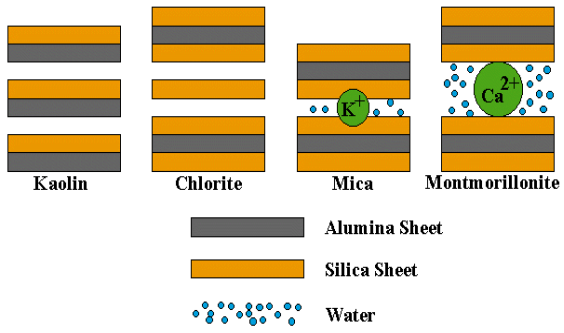
content of the water. As examples, sodium nontronite clays show a high rate of expansion or swelling while kaolinite clays typically show low levels of swelling.

3.5 ORE DRYING

Work conducted by Greene and Kelly [Ref 2] and separate studies by Rheochem have shown that drying an ore can affect the ultimate viscosity of the slurry once the ore is hydrated.

For most "plastic" type clays (that is, clays that show swelling characteristics when hydrated) the drying process will generally shrink the clay particles [Ref 1]. Water essentially evaporates from the outer surface and the particles of clay are drawn closer together by surface tension forces. Eventually the clay particles will come into mutual contact and form a loosely packed assembly where, it is not possible for the particles to pack more closely, and the critical moisture content is achieved. Further drying will result in loss of water by evaporation from the pores of the body and water being drawn to the surface by capillary attraction. This would typically result in a further improvement in rheological performance. For certain ore types air drying will only cause partial dehydration and higher temperature drying would be required for full dehydration. The natural salinity of the process water and cation make-up of the process water during any hydration and/or rehydration process plays an important role in how the clay particles swell as shown in Figure 4.

Figure 4 Clay Hydration/Cation Exchange



Typically the removal of any of some or all of the adsorbed water from the "smectite", or montmorillonites will result in improved rheology performance but some laterite ore's have shown the reverse effect, suggesting that the majority of the clay particles present are of the non swelling type.

The effect of reduced water adsorption in to the dried clay lattice has also been shown to be amplified when organics are present in the ore. As water is removed by drying, the organics form a hydrophobic coating on the clay particles, reducing their capacity to adsorb water during re-hydration.

3.6 SHEAR HISTORY

Mineral slurries in general often exhibit some type of gelling response under low shear. Natural aggregation within a sample will cause localised sedimentation, and inconsistent properties within the network of particles. Hence the need to properly homogenise samples even prior to preliminary characterisation tests.

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The amount and type of shear that a sample receives prior to testing for rheology is referred to as “shear history”. The measurement protocols must be identical to ensure repeatability. The amount of mixing prior to measurement and the time the sample has been left standing all need to be considered and need to reflect the particular plant or process conditions that are being sought. The low shear viscosity of many mineral slurries is highly time dependent and exhibits a high level of hysteresis dependent on which direction it has been measured from. Many lateritic slurries develop a strong gel structure with time. This has a major impact on yield stress measurements.

3.7 SHEAR THINNING

The viscosity of a slurry is highly dependent on the level and duration of shear it experiences.

The term shear thinning basically means that as more shear or work is put into a slurry, its apparent viscosity drops. Most “laterite” slurries are shear thinning and viscosity decreases as shear rate increases, however the term should not be taken too literally.

Another term often referred to is the thixotropic effect. This is usually reversible and the slurry will thicken to its original viscosity when the shear has ceased. The act of shearing a slurry to produce a non-recoverable change in the viscosity is usually referred to as rheodestruction, although it can be argued that the material will eventually return to its original state provided sufficient time is allowed. These effects are important parameters to determine and understand when dealing with any slurry system.

As the shear rate increases interparticle bonds are broken and the slurry appears to thin. At high shear, the kinetic energy is too great and inter-particle bonding is no longer the major contributor to viscosity. At high shear rates particle numbers or more accurately particle volume fraction plays an important role. As particle numbers increase, they become crowded and must be physically dragged past each other. As the particle volume approaches its maximum packing density there is physically no room to move.

This should not be used to infer that high shear devices could be used to permanently thin a slurry. High shear mixers or pumps will reduce the viscosity whilst the fluid is in shear, but may cause particle size degradation which can lead to an overall increase in viscosity. When the slurry returns to rest, it may have a higher baseline viscosity than prior to shearing.

In flocculated slurries it is imperative not to exceed the limits of the optimal shear rate range. Too low a shear can result in gel structures forming, while too high a shear may create undesirable centrifugal forces.

The reversibility of the thinning and thickening is the key to understanding the true nature of the slurry. If you assume that the measurement does not physically alter the slurry, and that after measuring it resumes its original properties, further concepts are more readily understood.

4 PRACTICAL APPLICATIONS

The following section provides practical examples of how various operating conditions and parameters can effect the rheology of a slurry.

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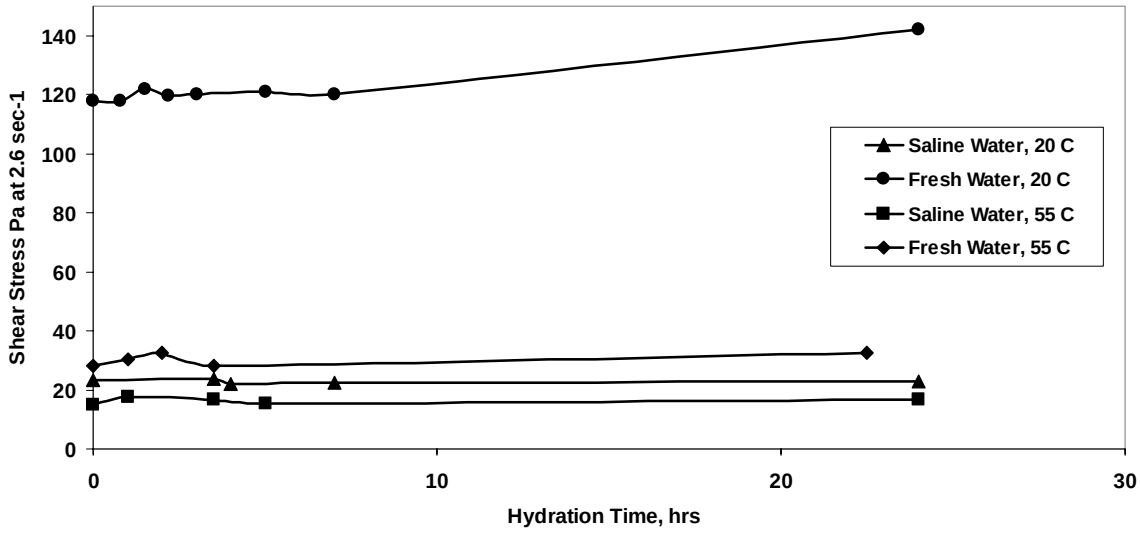
4.1 WATER QUALITY

An example of the effect of fresh versus saline process water on slurry rheology is shown in Figure 5 for an ore type that contained a significant quantity of smectite clay mineral (high swelling characteristics).

Figure 5 Variation in Shear Stress with Water Quality for L4A Ore Type at 35% Solids



Smectite is a alumina/silicate clay mineral where substitution takes place in the central alumina layer through exchange of aluminium ion by magnesium ion. Hydrated cations occupy the spaces between the alumina/silica sheets (refer to Figure 4) The type of cation and its associated charge density will determine the hydrated ion diameter which determines the amount of water bound in the structure which in turn dictates the amount of "swelling" present.

The data for this particular sample highlights the following;

- The adverse effect on rheology when using fresh water
- The improvement in rheology when increasing slurry temperature from 20 to 55 ^{o}C even in the fresh water system.

4.2 TEMPERATURE

In most cases with laterite slurries, an improvement in slurry viscosity with increasing temperature is experienced as shown in Figure 6 which was measured from slurries from an existing operation. The critical temperature at which this occurs is dependent on a number of factors including mineralogy, process water quality and the ore preparation and shear conditions.

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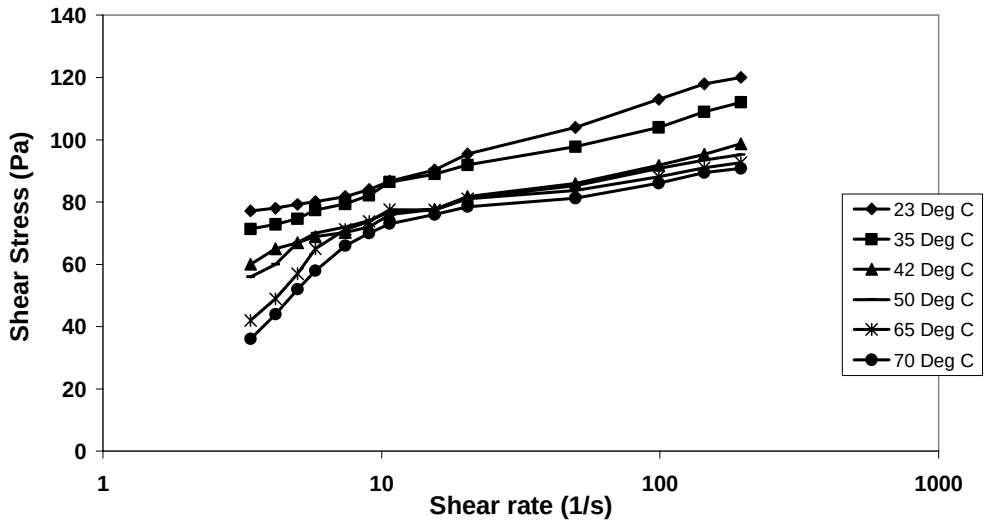
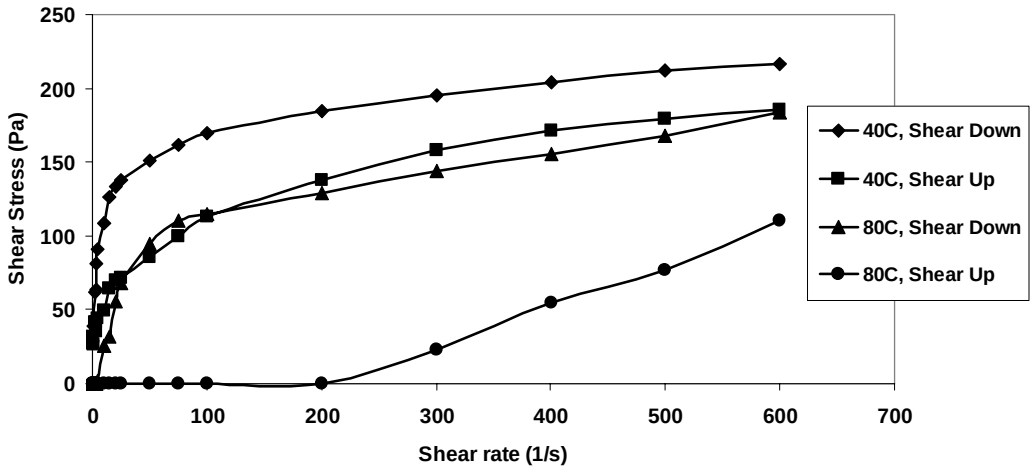
Figure 6 Shear Stress Profile at Varying Slurry Temperatures for a Milled Laterite Slurry



It can be seen that for this particular ore the effect of increasing the slurry temperature beyond 50 ^{o}C has a significant effect on reducing the shear stress of the slurry particularly at the low shear rate range of 5-10 inverse seconds where the shear stress has been reduced by up to 50%. This low shear rate range would be very relevant for screening applications.

Figure 7 shows the effect of temperature on a precipitated nickel sulphide sample that had been flocculated and thickened.

Figure 7 NiS 1 SampleRheograms at 40% Solids
Comparisons at 40C / 80C for shear up and down



The results show the reduction in viscosity when the slurry temperature is increased from 40 to 80 ^{o}C. The results also show localised gelling at 80 ^{o}C in shear up mode to 200 inverse seconds. Since the sample had been flocculated, this could possibly be explained by increased particle agglomeration in the static and low shear environment. The effect was not

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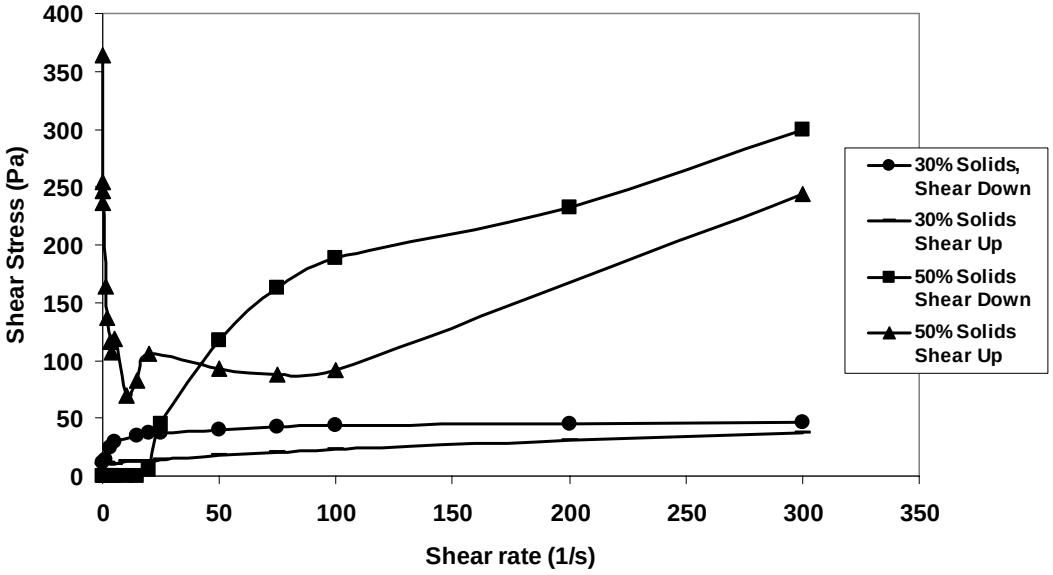
seen at the lower solids concentration of 30% but was very evident at the higher solids concentration of 50% solids as shown in Figure 8.

Figure 8 NiS 1 SampleRheograms at 80 C
Comparisons at 30/50% Solids for shear up and shear down



Combining the two graphs shows that for this sample, increases in slurry temperature above 40-45% solids accentuates the issues of slurry homogeneity. This is most likely due to increased activity of particle interaction, which increases the speed and extent of natural agglomeration and/or gel formation.

4.3 SLURRY MODIFICATION

Many mines use dispersants to reduce viscosity. The logic behind this thinking is that if the viscosity is reduced more tonnes per hour can be processed. This premise becomes flawed when the operator does not understand the true plant limitations.

In some instances dispersants can be used to modify the rheological behavior of a slurry when treated through a certain area of the process. It is important to test the performance of the dispersant over the correct shear rate range that would be applied in practice. Often Yield stress measurement is more appropriate, particularly for low shear applications, than determining shear stress versus shear rate rheograms. Examples of the effect of a dispersant on a milled laterite slurry is shown in Figure 9.

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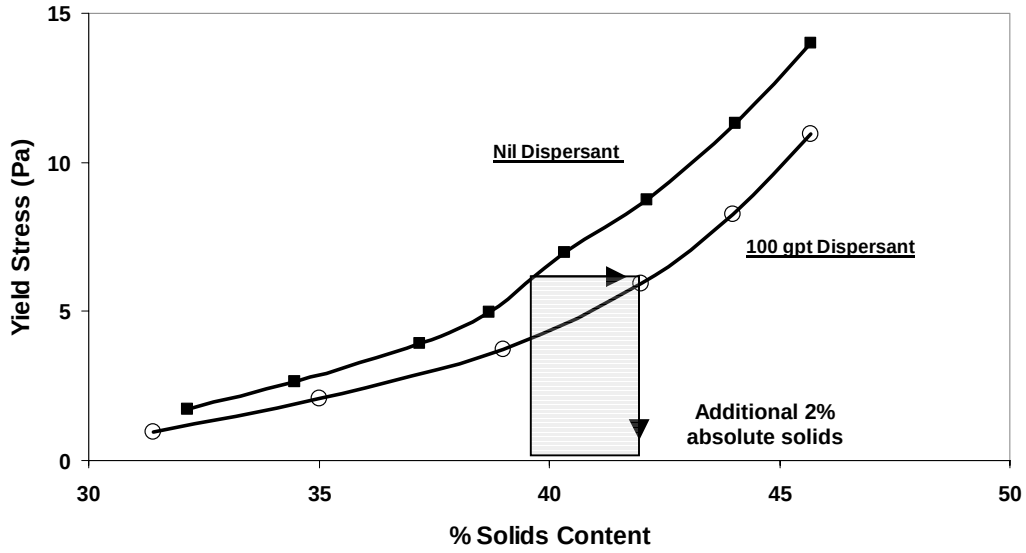
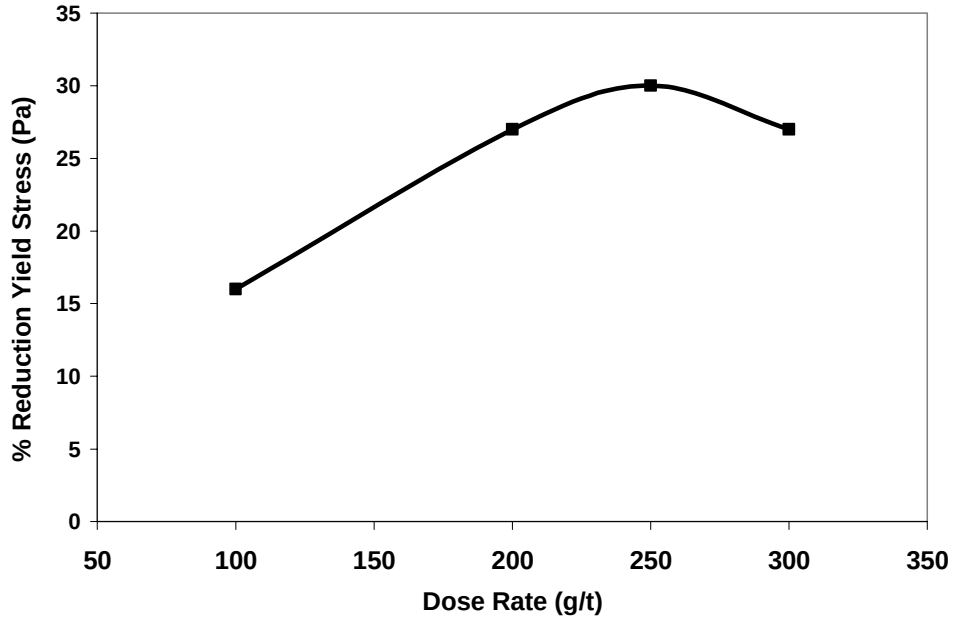
Figure 9 Vane Yield Stress vs % Solids for Evaluating Dispersant Application on a Milled Laterite Slurry



Over the yield stress range from 5-10 Pa an additional 2% absolute solids concentration can be achieved by using a dispersant at a dose rate of 100g/t. The effect of dose rate also needs to be optimised as shown in Figure 10, as overdosing can often lead to a misleading result.

Figure 10 Dispersant Dose Rate vs % Reduction in Yield Stress of Milled Laterite Slurry at 40% Solids



Work conducted by Klein and Hallborn [Ref 3] has shown that surface properties of goethite particles present in a laterite sample had a dominant effect on the rheological responses. Increasing the electrostatic repulsion by adding a dispersant or lowering the pH, caused decreases in the apparent viscosity and reduced the rheopectic (shear thickening) effect seen in the samples tested. This implied that increasing the electrostatic repulsion reduces shear induced particle aggregation.

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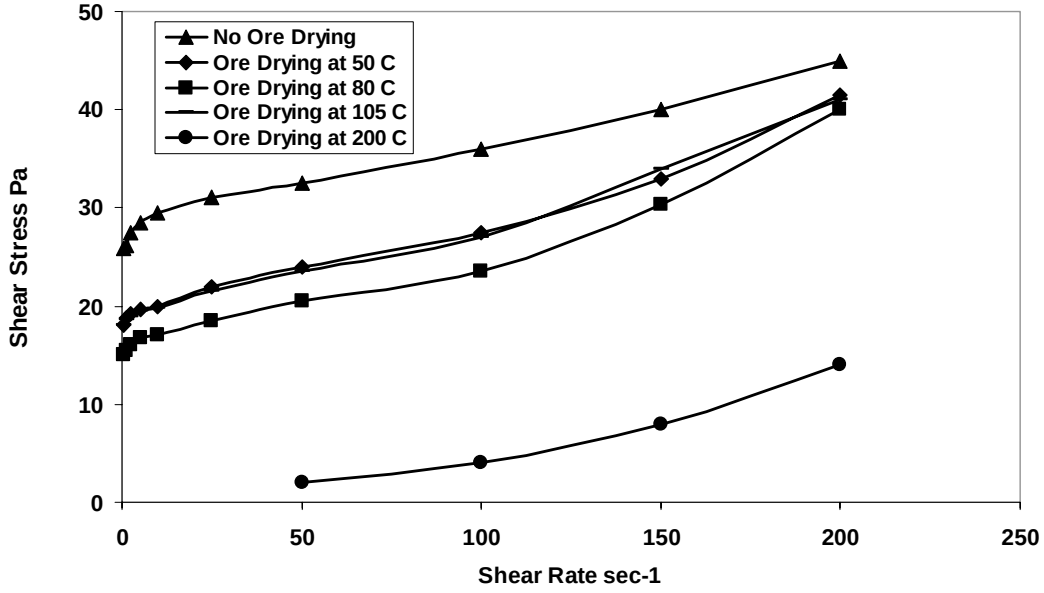
4.4 ORE DRYING

The interstitial water or water bound within an ores lattice structure can be removed by drying the ore, particularly at temperatures above 100 ^{o}C, prior to re-hydration. Figure 11 shows the effect that drying ore at a range of temperatures up to 200 ^{o}C has on slurry viscosity once the ore is re-hydrated.

Figure 11 Variation in Shear Stress with Ore Drying Temperature for L4A Ore Type Re-hydrated at 35% solids in saline water



The results clearly show the improvement in slurry rheology once the ore is dried and then rehydrated. This is mostly due to the physical limitation of water re-entering the reduced spacing between the clay layers. If the drying temperature is sufficient to remove all the free and bound water, the clay absorbs less moisture and acquires lower viscosity as a result. The results for this particular sample which contains a significant quantity of “smectite” clays shows a steady improvement in viscosity at drying temperatures of up to 80 ^{o}C where the drying process is mostly limited to the removal of free moisture in the ore. It is possible in practice that the removal of free moisture could be achieved by solar drying. The results at 105 ^{o}C show higher re-hydrated viscosity values than the results at 80 ^{o}C, which could indicate that other mechanisms are at play in this temperature range.

There is a large improvement in viscosity when the ore is dried at 200 ^{o}C due to the removal of bound water within the clay lattice.

The natural salinity of the ore and the salinity of the process water also plays an important role during the rehydration process. If the ore has a low natural salinity and saline process water is used (as is the case for the above results) then, fresh water is removed from the clay structure and replaced by saline water during rehydration. The saline water increases the osmotic pressure required for rehydration which effectively reduces the amount of water adsorbed.

Ore drying may not be beneficial for all laterite samples. Solar drying trials conducted at ambient temperatures using Windrow drying techniques have shown the results in Figure 12.

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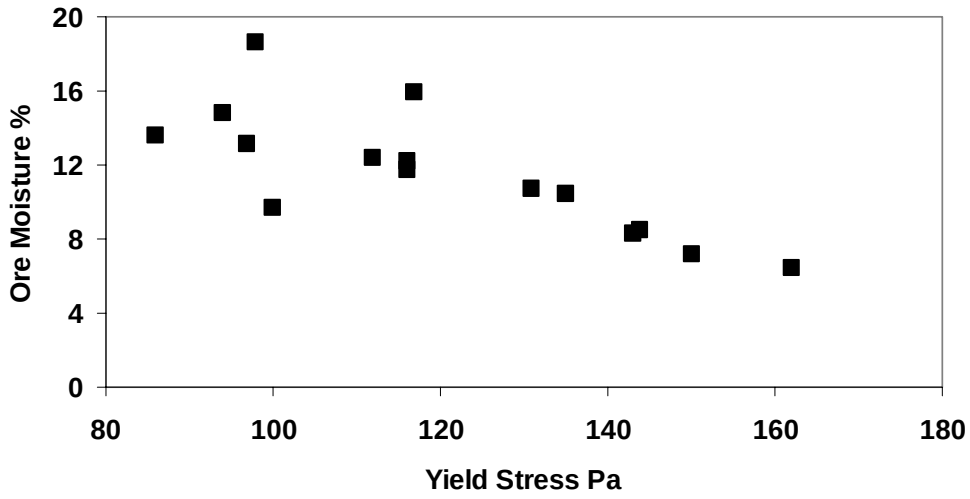
The sample was a quartz and goethite limonite material that contained moderate levels of kaolinite and nontronite clay types.

Figure 12 Ore Moisture vs Yield Stress at 38% Solids
Limonite 5 Ore Type



The results on this particular sample show the opposite effect to the L4A ore type in that viscosity of the rehydrated slurry increases as moisture is removed by air drying. The effect seen would be equivalent to a reduced absolute solids content of 2% which would have a detrimental impact on ore throughput rates.

This particular ore did not contain active or swelling clays, therefore one would not necessarily expect an improvement in viscosity, however, further investigative work is required to determine the mechanism behind the increase in slurry viscosity.

4.5 ORE VARIABILITY

Figure 13 highlights the large variations in yield stress that can be found across a range of ore types from clay bearing limonites, saprolitic material and transition ores to a sulphide concentrate.

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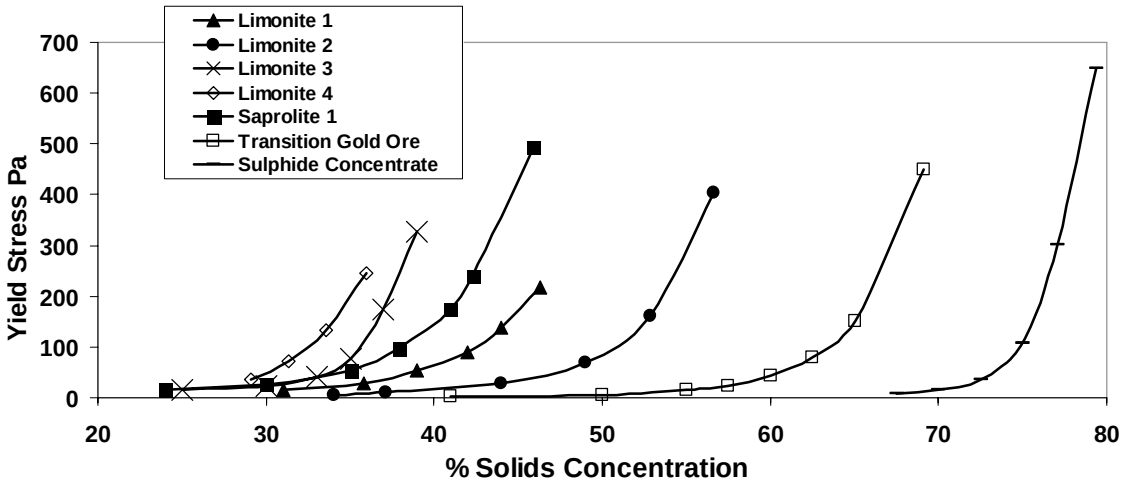
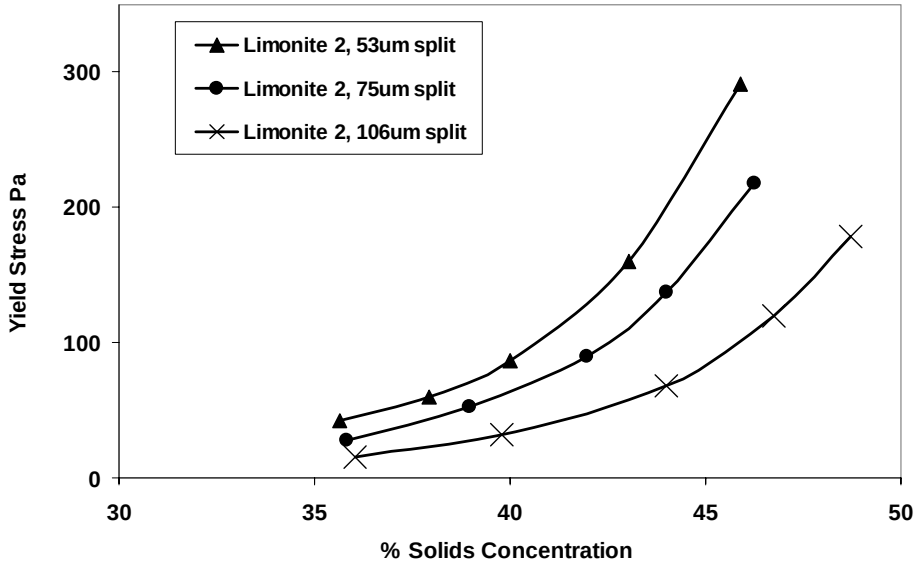
Figure 13 Yield Stress vs Solids Concentration for a Range of Ore Types



The results typically show that most samples will display an exponential fit, with yield stress increasing dramatically once a critical solids concentration is achieved. This is an important parameter to determine for design purposes.

The variation in yield stress as the grind size or split size of a slurry changes is shown in Figure 14.

Figure 14 Yield Stress vs Solids Concentration for a Range of Ore Types



In this instance variations of up to 5% solids can be experienced by lowering the size distribution of the slurry from 106 to 53 micrometers. This is significant if an ore shows metal upgrade potential and coarser material can be rejected. The relationship between metal upgrade/recovery, rheology and subsequent solids through put rate would need to be established.

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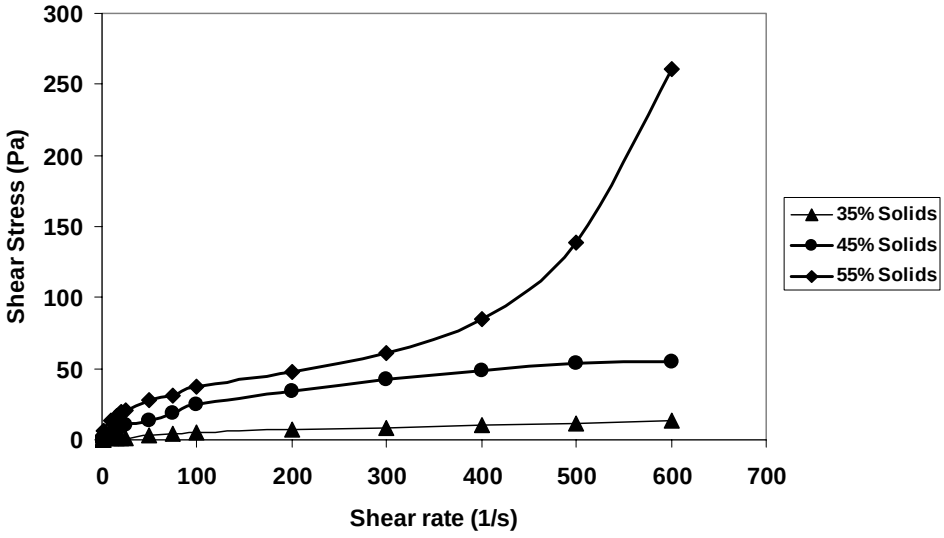
4.6 EFFECT OF SHEAR

Figure 15 shows rheograms (shear down sweep) prepared at varying solid concentrations for a laterite ore slurry.

Figure 15 Rheograms at 40 C for Limonite 5 Sample



In this instance the reduction in shear stress as solids concentration decreases at the high shear range is appreciable when compared to the lower shear range. For this particular sample, the results show that slurry dilution decreases slurry yield requirements (as expected) but has a marked effect on the high shear viscosities which would have major design ramifications if high shear mixing or pumping in the range of 400-600 inverse seconds was to be considered for the process. It is possible that some rheopectic (a reversible increase in apparent viscosity with time) effects are being seen at the higher solids concentration and would need to be examined further.

Worked conducted by Klein and Hallbom[Ref 3] showed a nickel laterite sample to display rheopectic time dependent properties. Pre-shearing at a low rate produced a curve in which the apparent viscosity increased with time and eventually became constant, whereas pre-shearing at a higher rate caused the apparent viscosity to decrease with time before remaining constant.

Shear time dependency work conducted by Rheochem has also shown some samples to display rheopectic behavior. Figure 16 shows the shear time dependent effect seen for sample Lim C, a Western Australian limonite.

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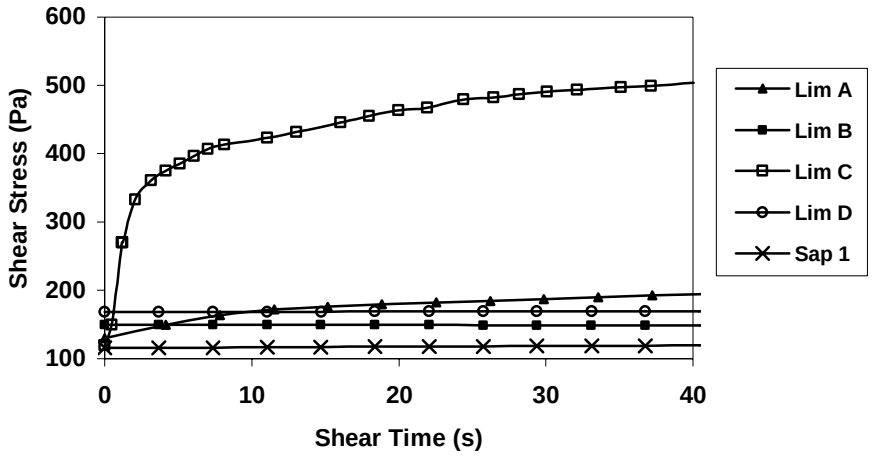
Figure 16 Shear Time Dependence Curves at 600 sec-1

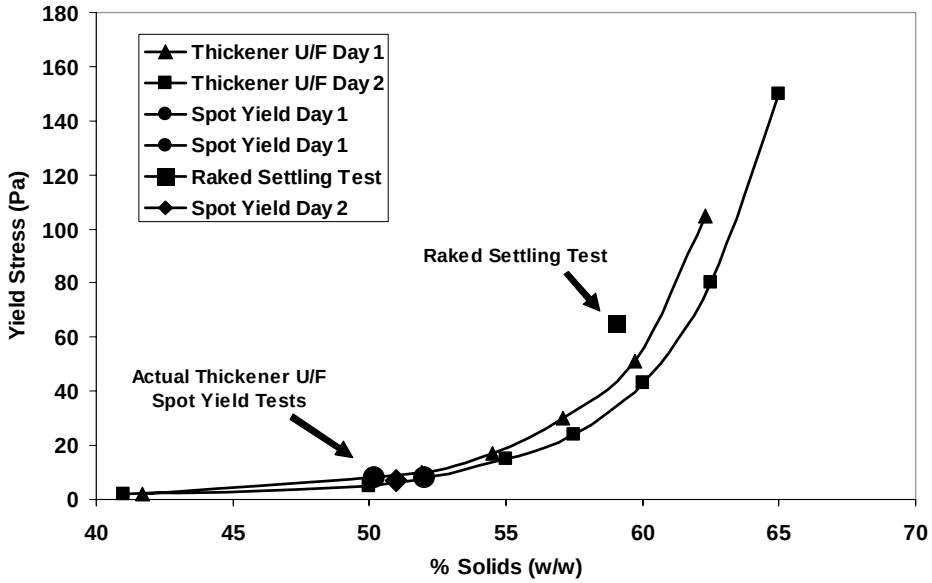


The results show that Lim C displayed strong rheopectic behavior when compared with other samples which either showed no shear thickening effect or mild shear thickening as shown by Lim A. Note that the effect seen with Lim C was reversed when a low shear regime was applied over a period of time, that is the slurry returned to its original equilibrium state.

4.7 THICKENER OPTIMISATION

An example of a thickener being operated under capacity is shown in Figure 17.

Figure 17 Vane Yield Stress vs % Solids for a Tailings Clarifier Application



The curves show the yield stress value of the thickener underflow to be in the lowest region of the curve. A standard raked settling test indicated that a higher ultimate settling density could also be achieved.

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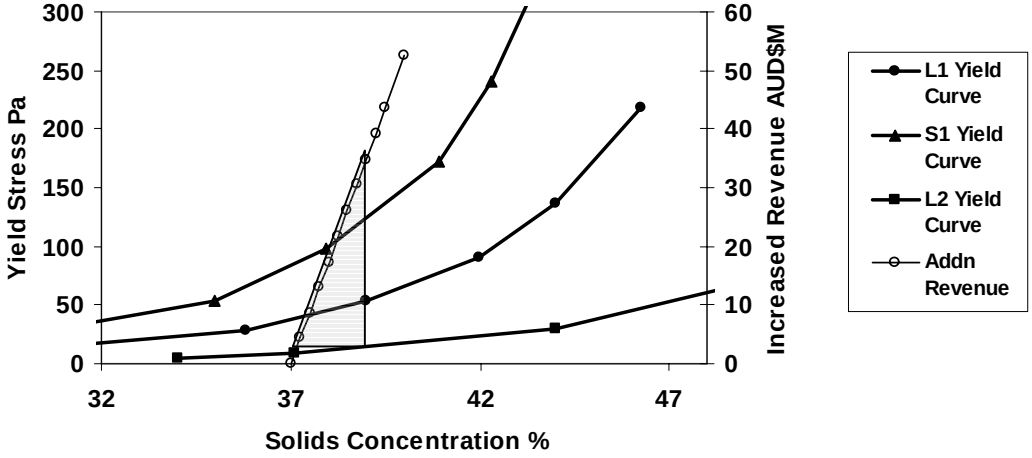
This suggests the relatively low thickener underflow density is probably not due to direct slurry rheology effects in the compression zone, but is more likely the result of other factors which could include:

- poor or over flocculation,
- loss of compression zone due to chaneling
- disturbance of the compression zone by entrained air,
- process control factors; e.g. low density setpoint for underflow discharge pump or,
- preferential discharge, chaneling in the compression zone.

5 ECONOMICS

The full impact of increasing the solids concentration for various laterite slurries through any of the means presented in this paper can be seen in Figure 18. A 1,100m^3/hr acid pressure leach process has been used, feeding L1, L2 and S1 samples to the PAL circuit.

Figure 18 Effect on Economics of Varying Slurry Solids Concentration



1,100m3/hr, Ni grade 1.2%, Ni price US$7,500/t, Availability 75%, Exch rate 0.55, Addn Op cost 20%

Figure 18 shows that optimising the solids throughput rate is directly related to the rheology of the slurry and if this can be improved or modified by methods such as ore drying, blending, temperature, controlling water quality, or addition of dispersants, then potentially, significant gross savings can be made. For instance, Improving the solids concentration feed of the L2 sample by 2% absolute solids would add an approximate $A32 million to total revenue even after removing 20% of the additional revenue as an operating cost component.

The results also highlight the importance of operating at the critical viscosity level for a particular ore type. In this case it would be easier from an operational view point to achieve an additional 2 % solids increase from the L2 ore than the S1 ore which is operating in the steeper region of the yield stress/solids curve.

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6 CONCLUSIONS

Understanding and quantifying the rheological behavior of a slurry is critical to optimising plant performance. Maximising the solids throughput rate by determining the critical viscosity limits through various areas of the plant can have dramatic improvement on the overall economics of a project.

Factors which can significantly effect rheology include;

- ore mineralogy
- process temperature
- water quality
- ore drying
- use of dispersants

The appropriate measuring devices and techniques need to be carefully considered for each application and then fitted to the correct models that best describe the data.

7 ACKNOWLEDGEMENTS

The authors would like to thank those companies that gave permission to publish the data used in the paper.

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NORILSK PLATINIFEROUS PYRRHOTITE CONCENTRATES - NEW INSIGHT INTO COMBINED PROCESSING

By

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1. INTRODUCTION

In first half of the 21st century the global civilization development will be in many respects determined by the level of precious metals production. These minor elements perform the most versatile functions; just to name a few of them: banking (currency and pledge), cultural and aesthetic and technological. The unique physicochemical properties of platinum group metals (PGM) and their alloys are responsible for the rapidly growing consumption of these metals in all leading industries, new technologies, space engineering, medicine, etc.

Platinum group metals play an extremely important role in the programs for ecocatastrophes prevention. Platinum and palladium catalysts open a wide range of opportunities in this field. The projects for ecologically clean new generation vehicles are based on their application.

The long-term outlook for PGMs demand in all industries considered is positive. At the same time, the world production of platinum group metals has not provided for the growing demand for them recently.

2. MAJOR WORLD PGM DEPOSITS

The uniqueness of commercial PGM deposits and the constant decline in their mining output exceeding stock addition results in, on the one hand demand for an intensive search of new unconventional sources of PGM-bearing feedstocks to expand the mineral base, and on the other hand for more effective utilization of available resources by modernization of working operations and development of advanced complex processes and technologies. Known deposits of platinum group metals in the world are estimated approximately at 95 000 t. More than 90% of them are concentrated in three large regions: the Bushveld Complex (South Africa), Norilsk (Russia) and the Great Dyke (Zimbabwe).

The main PGM suppliers in the world market are South Africa (63,3% of the world's platinum production and 23,8% of the world's palladium) and Russia (23,8 and 65,3% respectively).

The structure of PGM hosting deposits and mining in South Africa and Russia differ drastically. South Africa's PGM ore bodies (the Merensky and UG2 reefs in the Bushveld Complex) are for the most part associated with chromite layers occurring in the layered intrusions of basic and ultrabasic rocks. Platinum prevails in South African ores, its production output approximately twice as much as palladium production (in 1998, 118.2 and 57.0 t respectively). The main sources of PGM extraction in Russia are sulphide copper-nickel ores of the Norilsk deposits. At the same time reserves occurring deep in the earth in the Russian deposits as distinguished from South African ores are more than 3 times as great as platinum reserves. This circumstance allows Russia to influence the global palladium market actively.

According to the forecasts, the increase in the platinum group metals production in the main producing countries (the South African Republic, Russia, USA, Canada, Zimbabwe) in the near term will be limited. By estimate it will begin no sooner than in 2-3 years and it will amount barely to 40-55 t per year.

The extremely favorable market situation for PGMs producers requires prospecting, exploration and development of new deposits, expansion of working mines, increase of PGM output, involvement of the industrial PGM-bearing feedstocks and a more integrated approach to ores and concentrates treatment.

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3. SITUATION AT NORILSK

In recent years, in view of the decrease in the content of rich ores in the mass of commodities in Norilsk deposits, PGM grade and mining output in JSC Norilsk Nickel MMC (JSC NN) have been steadily decreasing.

Strategies are required to expand the mineral base for PGMs in JSC Norilsk Nickel the foremost Russia's enterprise in reserves and production output of PGM.

Among promising sources of PGM in JSC NN are considered: PGM bearing low sulphide deposits, stockpiled pyrrhotite concentrates, magnetite concentrate and impounded mill tailings of sulphide copper-nickel ores. They represent man-made formations with significant content of non-ferrous and platinum group metals. What is more, an important reserve of PGM output enhancement lies in the improvement of the integrated approach to the processing of the current nickel-pyrrhotite concentrate produced from the rich Talnakh sulphide ores.

It is known, that the reserves of the rich copper-nickel ores at the Talnakh and Octyabr'sky deposits are represented by pyrrhotite varieties (more than 90%). These types of ores are characterized by the high content of non-stoichiometric sulphides (30-60%) belonging to the pyrrhotite group (Fe1-x S) and the extremely close intergrowth of sulphide minerals, that complicates their separation by conventional beneficiation processes. In particular, close binding is marked between the main nickel mineral pentlandite and pyrrhotite, up to a solid solution forming nickeliferous pyrrhotite.

Pyrrhotite in the ores from Talnakh cluster is represented by two varieties: hexagonal (Fe_8S_9 - $Fe_{13}S_{14}$) and monoclinic (Fe_7S_8). At that the share of monoclinic pyrrhotite constitutes about 20% of its general content in the ore. The prevalence of the hexagonal (non magnetic) pyrrhotite modification notedly distinguishes Norilsk ores from sulphide copper-nickel ores treated at other enterprises elsewhere, in particular in Canada. Monoclinic (ferrimagnetic) pyrrhotite has widespread occurrence there. And that fact has allowed Canadian operators to implement electromagnetic methods of enrichment, successfully providing pyrrhotite extraction into a highly selective concentrate beginning from the middle of 70-ties of the last century.

4. NORILSK INTERMEDIATE NICKEL-PYRRHOTITE CONCENTRATE

Processing of rich sulphide ores at Norilsk is based on flotation concentration methods. A separate sulphide intermediate product is produced in the flotation process, nickel-pyrrhotite concentrate, into which, depending on the conditions of nickel and pyrrhotite flotation, is recovered %: Ni 13-28; Cu 4-6; Co 15-30, and PGMs 15-30 of their content in the ore. Chemical, mineralogical and size compositions of the nickel-pyrrhotite concentrates depend on the make-up of the raw ores and the beneficiation conditions. Usually, concentrate includes the following minerals: pyrrhotite (60-75 %); pentlandite (6-9%); chalcopyrite(1,5-3,5%); magnetite (up to 8-10%), with minor talnakhite and cubanite. In currently produced nickel-pyrrhotite concentrates, 1 t of contained nickel accounts for up to 20-25 t of iron and 12-17 t of sulphur. Direct processing of such material for matte is considered uneconomical, in spite of the significant progress and efficiency of modern methods of autogenous smelting.

Pyrrhotite concentrates selected in the process of sulphide copper-nickel ores enrichment are considered rather a new source of PGM bearing feedstock. At the same time the contents of PGMs in Norilsk nickel-pyrrhotite concentrates and old pyrrhotite concentrate are by higher a factor of two in comparison with similar concentrates selected at other enterprises elsewhere.

In terms of PGMs content, Norilsk nickel-pyrrhotite concentrates and stockpiled pyrrhotite concentrate are on a par with the richest ores in the Bushveld Complex (South Africa) –

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the ores in the Merensky Reef and UG-2, and they appreciably (3-5 fold) surpass this figure in the ores at Platreef. The total value of metals in pyrrhotite concentrate is more than an order of magnitude higher than in the rich South African ores, owing to the considerably higher (by 25-30 times) content of non-ferrous metals, nickel first of all. Comparing the mining and technical conditions and level of expenditures in the sphere of preparation to metallurgical production, Norilsk pyrrhotite concentrates as a raw source of PGMs also have unquestionable advantages over similar sources among the leading platinum group metals producers worldwide. Pyrrhotite concentrates are characterized by high degree of dispersion (80-90% minus 44 micron), and they are essentially a flotation co-product, while ores in the Bushveld Complex (Merensky Reef and UG-2) for example, are produced by underground extraction at a depth of 1500-2000 m and require enormous expenditures for mining and beneficiation.

While making assessments of current processes and prospects for alternative trends, it is necessary to take into account that Norilsk nickel-pyrrhotite concentrates are extremely metallurgically complex feedstocks. The produced concentrate genetically derives all the basic features of the raw ore, but it differs by the finest association of non-ferrous, platinum group metals and pyrrhotite bearing minerals; advanced isomorphism and essential predominance of hexagonal (non magnetic) pyrrhotite modification over monoclinic variety and troilite.

5. CURRENT TREATMENT OF NICKEL-PYRRHOTITE CONCENTRATE

Presently the existing nickel-pyrrhotite concentrate is treated by a pressure oxidizing process with production of sulphide concentrate, commercial sulphur and final iron hydrate tails (fig. 1).

In the pressure-chemical enrichment of nickel-pyrrhotite concentrate, non-ferrous metals content in the final sulphide concentrate is increased 4-6 fold, at a recovery of about 85-88%. Besides, roughly 30% of the sulphur content of the nickel pyrrhotite concentrate is recovered into commercial elemental sulphur, and about 50% is discarded to the tailings pond as iron hydrate tails. As a result, the sum of non-ferrous metals to sulphur ratio in the sulphide concentrate sent to pyrometallurgical process, in comparison with that in the raw nickel pyrrhotite concentrate, increases by approximately 5 times, which improves the ecological conditions of non-ferrous metals production from pyrrhotite feedstocks significantly. Practically all gangue material and 80-90% of the iron are rejected into tailings. The degree of achieved non-ferrous and precious metals enrichment of the sulphide concentrate predetermines highly favorable technical and economic parameters for its further processing.

Despite a variety of unquestionable advantages in comparison with other trends in the nickel-pyrrhotite concentrate processing, the current technology also has serious demerits. One of them is impermissibly high level of rare platinum group metals lost with the final tailings, amounting to 60-80%. In this regard, it must be emphasized, that this situation is in many respects caused not by deficiencies in autoclave hydrometallurgy, application of which actually allows the PGMs recovery to be raised up to 90-95%, but by initially inaccurate priority ranking.

By virtue of national technological traditions and existing price lines, the priority in the development of autoclave technology was given to nickel recovery; while all platinum group metals were considered by-products and the process of their production was rigidly linked to non-ferrous metals production. The result of such an approach was the following: combining diverse requirements, for example while optimizing pyrrhotite decomposition figure, the major condition was to provide maximum nickel recovery to the detriment of PGM recovery, and the issue of PGMs loss reduction was not given due attention in the process development.

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The low level of platinum group metals recovery in the autoclave process for nickel pyrrhotite concentrate treatment is to a considerable extent caused by non-design (high temperature) conditions in the oxidizing leaching stage. This stage has excluded the possibility of implementing the design (so-called "complete") production circuit, which provided a special aggregating stage for platinum group metals recovery into a separate intermediate product.

6. ANHYDRIDE LEACHING PROCESS

One of the promising technologies for solving the problem of non-ferrous and platinum group metals loss reduction in the autoclave hydrometallurgy of nickel-pyrrhotite concentrate is the transition from "oxygen" leaching to a more selective "anhydride" one based on application of sulphur dioxide as an oxidizer. The idea of the implementation of similar processes was expressed for the fist time in 1932-33 by Makovetsky E.A. Later it was developed in Professor Sobol's works. An autoclave process for "anhydride" pyrrhotite leaching is unique in the fact that elemental sulphur is produced from two sources: sulphides and gas-reagent. Here the reagent simultaneously acts as the second raw source of sulphur:

$$2FeS + SO_2 + 2H_2SO_4 = 2FeSO_4 + 2H_2O + 3S^\circ \quad (1)$$

$$FeS + 2SO_2 = FeSO_4 + 2S^\circ \quad (2)$$

In this case both platinum group and non-ferrous metals practically do not pass into solution; rhodium, iridium and ruthenium are not found in it, platinum and palladium recovery amounts to less than 1%, gold up to 5%.

Strong sulphur-containing gases from the smelting units can be a source of sulphur. In this respect the Norilsk industrial site, where hydrometallurgical and pyrometallurgical operations are situated in close proximity, provides a unique opportunity to use SO_2 contained in the exhaust gases from autogenous smelting (15-25% SO_2) as an oxidizer in the high pressure hydrometallurgical processes for pyrrhotite treatment. A serious problem in all "anhydrite" process circuits is ferriferous effluents treatment. The most preferable solution in this case, taking into consideration the long term Nadezhda Metallurgical Plant development, is an alternative jarosite process. In this case, the formation of Fe (II) sulphate solutions can be considered a positive factor, as it provides a solution to the problem of recycling sodium sulphate effluents which are generated in the carbonate stage in the process of hydrometallurgical converter matte treatment.

Thus the main advantage of the technology higher for "anhydrous" leaching of nickel-pyrrhotite concentrate, in addition to high recovery of PGM (up to 95%) into a high-grade concentrate, lies in the opportunity to establish on the basis of the above technology balanced low-waste chemical and metallurgical complexes which ensure the opportunity for mutual neutralization of environmentally dangerous components. In this regard, it is important to highlight the option of combining sulphuric acid (non-oxidizing) leaching of nickel-pyrrhotite concentrate and the catalytic Claus-process for desulphurization of waste gases from autogenous smelting (SO_2) with elemental sulphur generation. In this chart (Fig. 2), along with high-grade sulphide concentrate production (~9% Ni), a large amount of hydrogen sulphide (H_2S) is liberated:

$$FeS + H2SO4 = H2S + FeSO4 \quad (3)$$

Hydrogen sulphide can be used for recovery of gaseous sulphur dioxide according to the following equation:

$$H_2S + SO_2 = 3S^o + 2H_2O \quad (4)$$

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In the process of nickel-pyrrhotite concentrate leaching, PGM recovery into a sulphide concentrate is over 95%, and sulphur recovery into sulphide-ion H_2S is 60-70%. According to the calculations, 1 ton of nickel-pyrrhotite concentrate (30% S) ensures utilization of ~0,5 t SO_2, resulting in 0,24 t commercial sulphur. The degree of gas desulphurization in this process tops 99,5%.

7. OTHER PROCESSING OPTIONS

In the near future the problem of complex processing of Norilsk pyrrhotites will become a vexed one. On the one hand this results from the necessity to improve the quality of copper-nickel concentrates for the purpose of cost saving in the major metallurgical operations, and on the other hand from the increment of high-sulphur pyrrhotite and chalcopyrite-pyrrhotite ores mined with pyrrotite content of ~50-60% on average. The prospective trend of growth of the pyrrhotite portion in the processed ore will inevitably lead to increase in nickel-pyrrhotite concentrate extraction at the stage of concentration, and finally it would result in a higher conversion of nonferrous and precious metals into this product.

At the same time nickel-pyrrhotite concentrate processing at operating and expanding smelting plants of “Norilsk Nickel” is an issue of the day, due to a high sulphur content in concentrate, where 7-8 tons of sulphur are accounted for each ton of the total nonferrous-metal value. Annually 350-400 kilotons of sulphur dioxide will be emitted into the atmosphere during the process of oxidizing roasting and smelting of nickel-pyrrhotite concentrate in the installations of the Sintering Plant, Nickel Smelter and Nadezhda Metallurgical Plant (NMP) for lack of waste-gas desulphurization systems. At present the technology of the NMP hydrometallurgical section and because of sulphide autoclave oxidation it is possible to convert max ~85% of pyrrhotite concentrate sulphur into sulfate and elemental forms, which are transferred for storage (gypsum) and sold as marketable products (elemental sulphur). The sulphide concentrate yielded from the Nadezhda hydrometallurgical section carries only 1,8-2,0 tons of sulphur per 1 ton of nonferrous metals and sulphur dioxide emission into the air caused by processing of nickel-pyrrhotite concentrate constitutes at most 50-60 tons per year. Hence direct smelting of nickel-pyrrhotite concentrate for matte without recovery of generated SO_2 would increase annual sulphur dioxide emissions by 300-350 kilotons.

The above data leads to two conclusions:

- firstly, nickel-pyrrhotite concentrate is a valuable platinum-bearing metallurgical raw material, unacceptable to be taken out from production;

- secondly, development of an alternative technology for nickel-pyrrhotite concentrate processing requires a comprehensive approach, combining complete recovery of all the valuable components into marketable products while keeping a high level of environmentally appropriate production.

Optimization of the “Norilsk Nickel” metallurgical complex, based on the concept of high pyrrhotite recovery into a low-Ni product at the stage of concentration, and rejection of this product for long-term storage, in the context of PGM losses is considered to be unacceptable. Such a concept, typical of Canadian nickel operations, is unsuitable for Norilsk pyrrhotite because of its distinctive property - a far higher content (~ 20 times more) of the total PGM value. It is common knowledge that some portion of the palladium and all rare PGMs occur exclusively in the form of solid solutions in pyrrhotite and pentlandite, and are found in both minerals in equal concentrations. Since the amount of pyrrhotite in Norilsk ores is 5-7 times higher compared with pentlandite, the bulk of rare-platinum metals (RPM) is structurally associated with pyrrhotite and there is no way to extract it by methods of mechanical upgrading into copper-nickel concentrates. Therefore rejection of a low-Ni pyrrhotite product containing up to 2,5 g/t of the total PGM value (comparable with

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PGM content of the ore from Platreef, South Africa) for storage will inevitably lead to an economically unjustified decrease in palladium and RPM production.

High PGM content, the finest association of valuable components with pyrrhotite, and much more developed isomorphism, eliminates the possibility applying experience of foreign companies for solving the problem of Norilsk pyrrhotite, and requires elaboration of domestic non-traditional solutions suitable to the nature of the processed raw material.

Norilsk Nickel specialists propose several options for Norilsk pyrrhotite concentrates processing.

One of the main options under consideration is combined processing of Norilsk pyrrhotite concentrates together with oxidized nickel ores (ONO) outside the Norilsk industrial region. This method was first suggested by the Norilsk Nickel working team which participated in the "Kupey" project concerned in development of an alternative process for Cuban lateritic ore treatment at "Las Kamariocas" site. Figure 3 shows the process flowsheet for Norilsk nickel-pyrrhotite concentrates and Cuban lateritic ore collective treatment. The proposed process comprises pre-separation of laterite into high-magnesia (serpentinite) and ferrous (limonite) fractions by screening. Norilsk pyrrhotite concentrates together with the serpentinite ore fraction (as a fluxing additive) are fed to autogenous smelting to produce matte (in a two-zone Vanukov's furnace). Generated SO_2 is transferred to sulphuric acid production; the latter is used in autoclave leaching of the ferrous ore fraction. At this stage waste steam generated during nickel-pyrrhotite concentrates smelting in Vanukov's furnace is utilized for heating pulp up to operating temperature (240-250^{0}C). The main advantages of this flow-sheet are the high recovery of non-ferrous and platinum metals into rich matte, utilization of pyrrhotite concentrates sulphur in laterite autoclave leaching which have a very high H_2SO_4 consumption (300-400 kg/t of ONO) and recovery of heat generated in pyrrhotite oxidation.

Combined technology based on using Norilsk nickel-pyrrhotite concentrates to process ONO from domestic nickel-cobalt deposits was defined as a promising approach in the course of investigations performed on high-magnesia ore samples of (Burukhtalsky, Serovsky and Sakharinsky) deposits in the South Urals.

The proposed technology offers several advantages at a time:

- efficient processing of nickel-pyrrhotite concentrates outside Norilsk Industrial Region with maximum non-ferrous and PGM recovery, accompanied by sulphur utilization and exothermal heat recovery;

- lower processing cost of ONO with high magnesium oxide and aluminium content due to utilization of cheap sulphuric acid and waste steam produced in nickel-pyrrhotite concentrates smelting;

- lower ecological impact of nickel-pyrrhotite concentrates processing;

- production of high-quality nickel-cobalt intermediate products needed for future full load operation of the Severonickel Combine.

A hydrometallurgical method for combined separate nickel-pyrrhotite concentrates and ONO processing was also developed. The process provides up to 95% recovery of PGMs into sulphide concentrate. This process involves sulphuric acid leaching of nickel-pyrrhotite concentrates and ONO in separate cycles with addition of sulphur-containing products derived from pyrrhotite decomposition at ONO processing stage.

Another method of nickel-pyrrhotite concentrates processing comprises concentrate smelting in autogenous unit (flash smelter or Vanukov's furnace) with matte production and

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sulphur recovery as gypsum. After calcination gypsum may be used as a binding agent for hardening backfill mixtures in mining.

There are two possible sub-variants for gas desulphurization. **Sub-variant 1** involves gas desulphurization from autogenous smelting with intermediate sulphuric acid production (Fig.4); the latter is then neutralized with calcium alkaline agent pulp in a bank of reactors. In the proposed flow-sheet nickel-pyrrhotite concentrates can be smelted alone or mixed with nickel (copper) concentrate; nickel-pyrrhotite concentrates processing with bulk copper-nickel-pyrrhotite concentrate being the most preferable. Nepheline slime or limestone can be used as an acid neutralizer. The use of lime, though possible, is uneconomic due to its high price.

In sub-variant 2 (Fig.5) furnace gas is desulfurized by absorption through direct contact of sulphur dioxide with a neutralizer pulp (nepheline slime, limestone or lime). The resulting pulp is treated with air for undefined sulphides oxidation to sulphate-ions and well-decrystallized gypsum dihydrate formation. Similar processes are used worldwide in desulphurization of industrial gas with low SO_2 content.

Individual elements of the processes under review are commercially implemented and widely used in combination with different processes. Operating experience and investigation results show that all selected ways for PGMs recovery are highly comprehensive. Combined nickel-pyrrhotite concentrates and ONO processing is the most attractive and practically feasible option, as it provides a basis for innovative pressure acid leaching of oxidized nickel-cobalt ores in Russia.

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ALTERNATIVE FLOWSHEET AT LAS CAMARIOCAS PLANT (Cuba)
Option 1

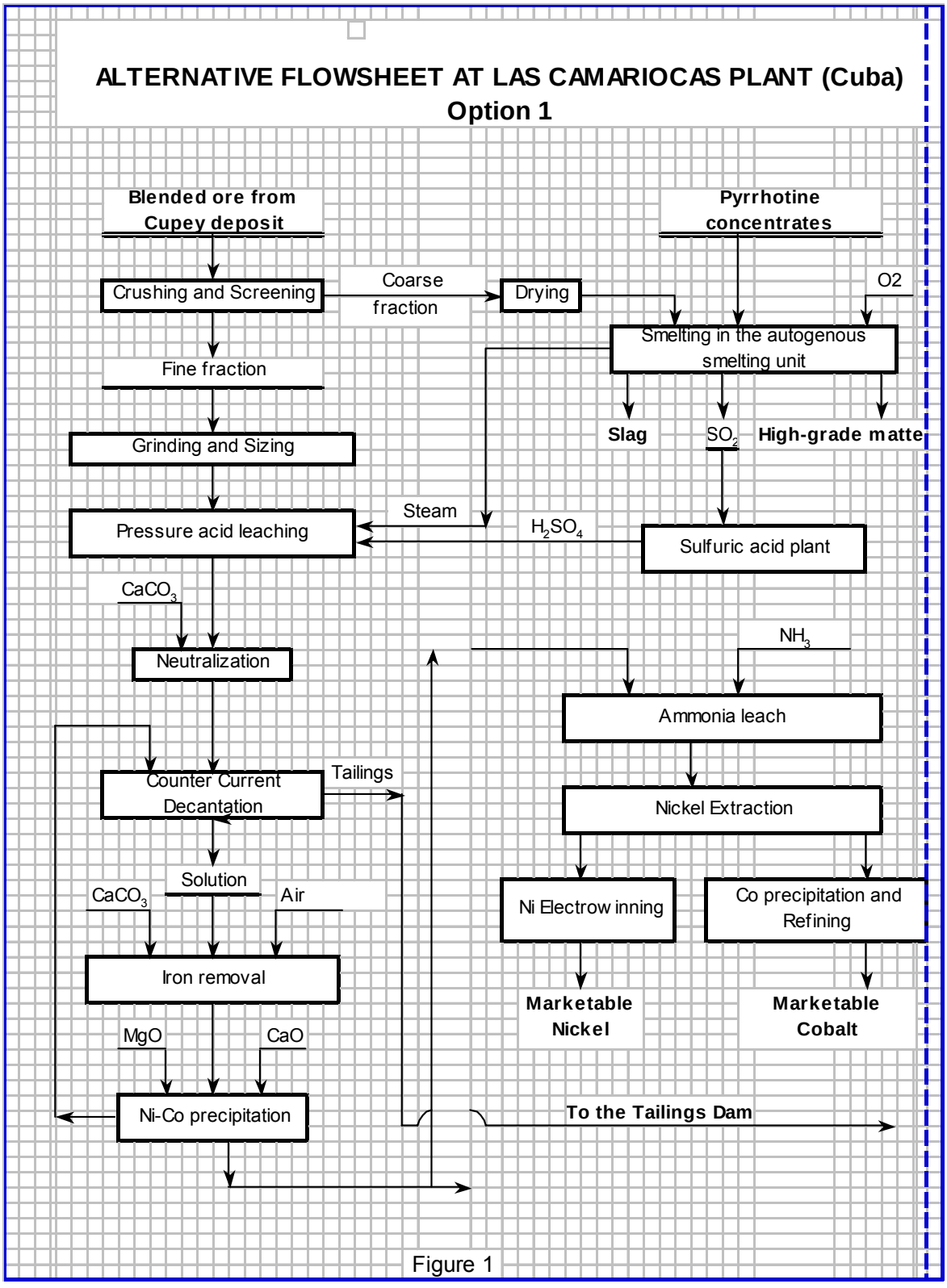

Figure 1

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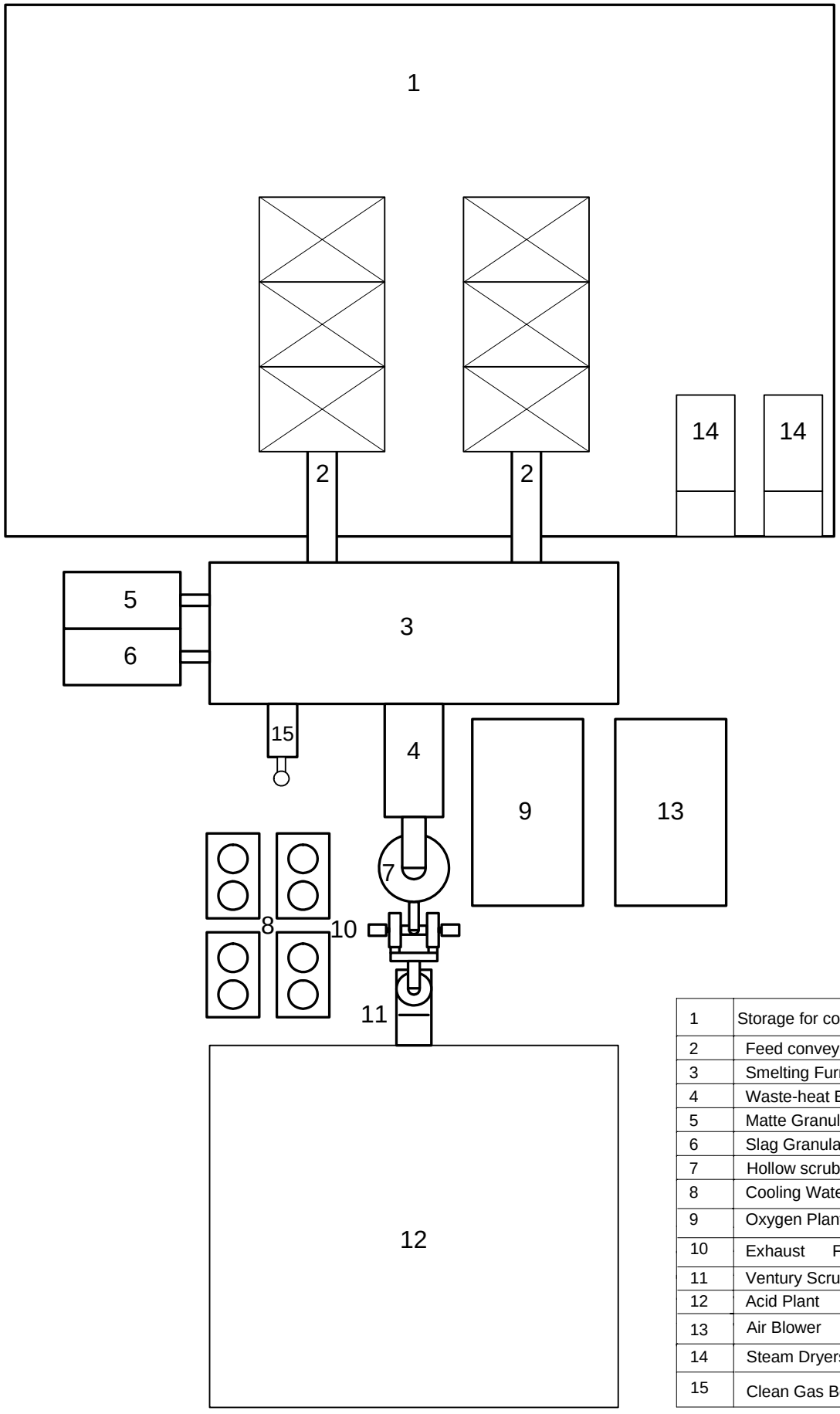

1	Storage for concentrate, ore, sand
2	Feed conveyors
3	Smelting Furnace Room
4	Waste-heat Boiler
5	Matte Granulation Pool
6	Slag Granulation Pool
7	Hollow scrubber
8	Cooling Water System
9	Oxygen Plant
10	Exhaust Fans
11	Ventury Scrubber
12	Acid Plant
13	Air Blower
14	Steam Dryers
15	Clean Gas Boiler with a Stack

Fig.2 Equipment Layout of Pyrometallurgical Process According to Alternative Flowsheet Proposed for Las Camariocas Plant

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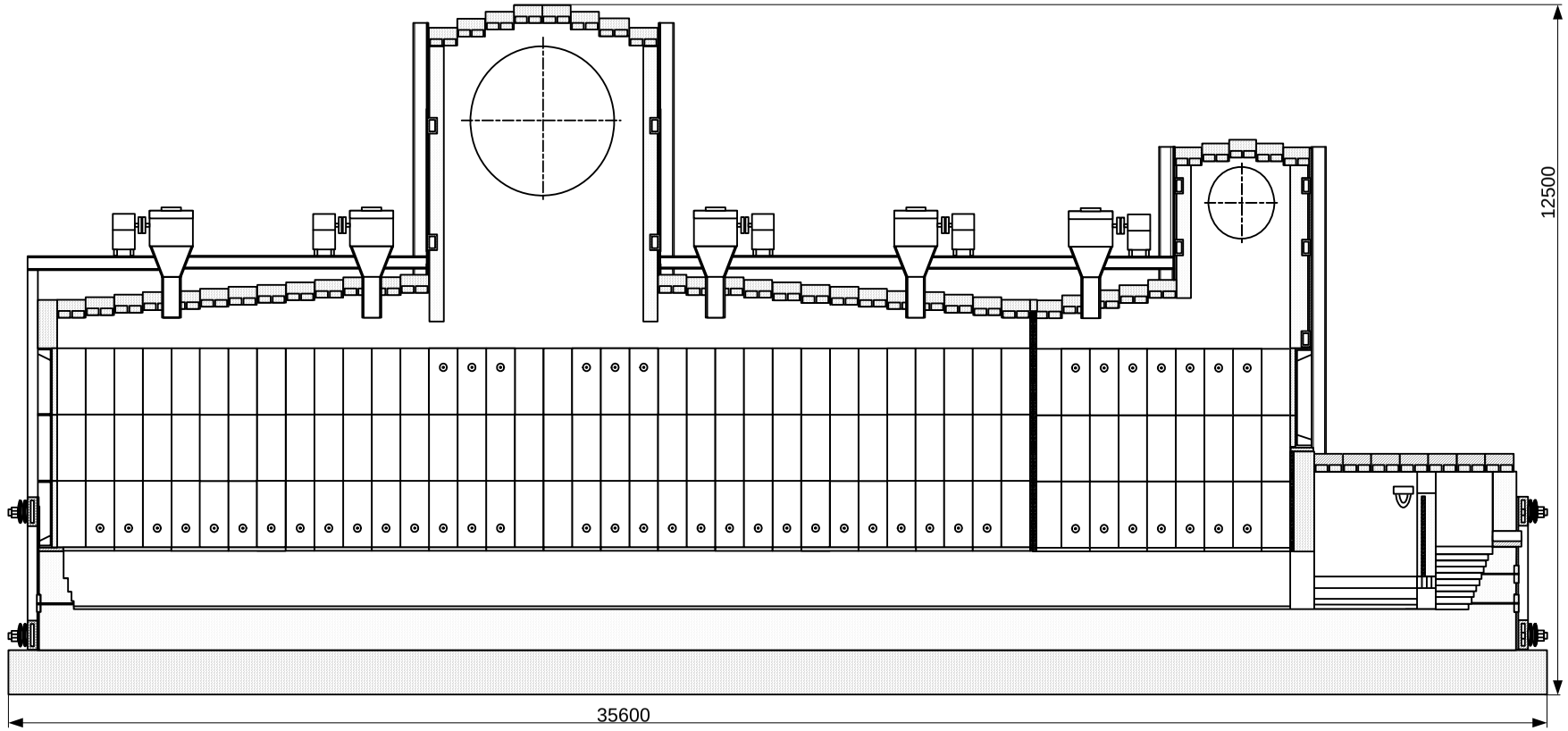

Fig. 3 Schematic Figure of Two-zone Vanyukov's Furnace for High Grade Matte Production from Norilsk Pyrrhotine Concentrate and Magnesia Fraction of Oxidized Nickel-Cobalt Ore

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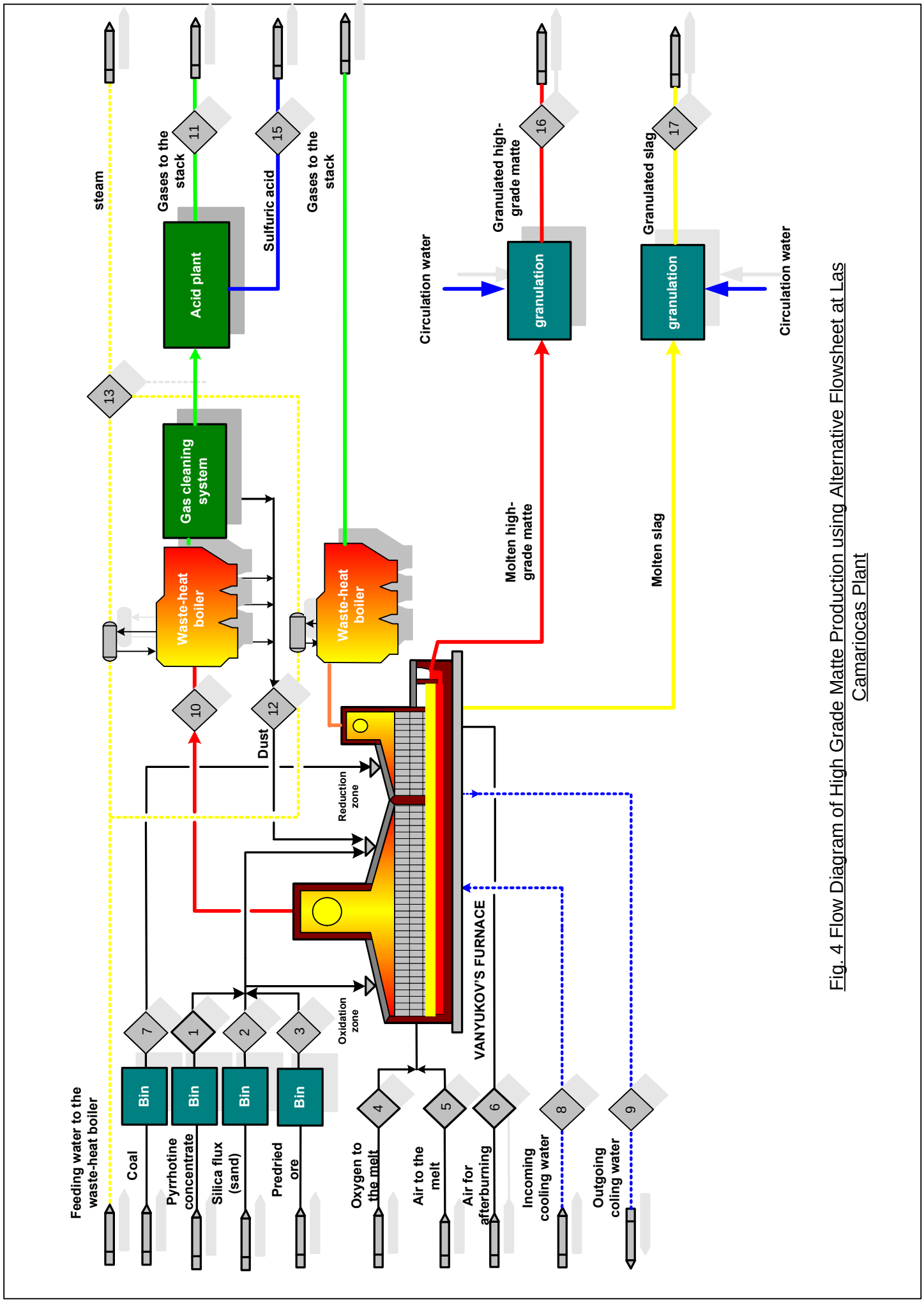

Fig. 4 Flow Diagram of High Grade Matte Production using Alternative Flowsheet at Las Camariocas Plant

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ALTERNATIVE FLOWSHEET AT LAS CAMARIOCAS PLANT (Cuba)

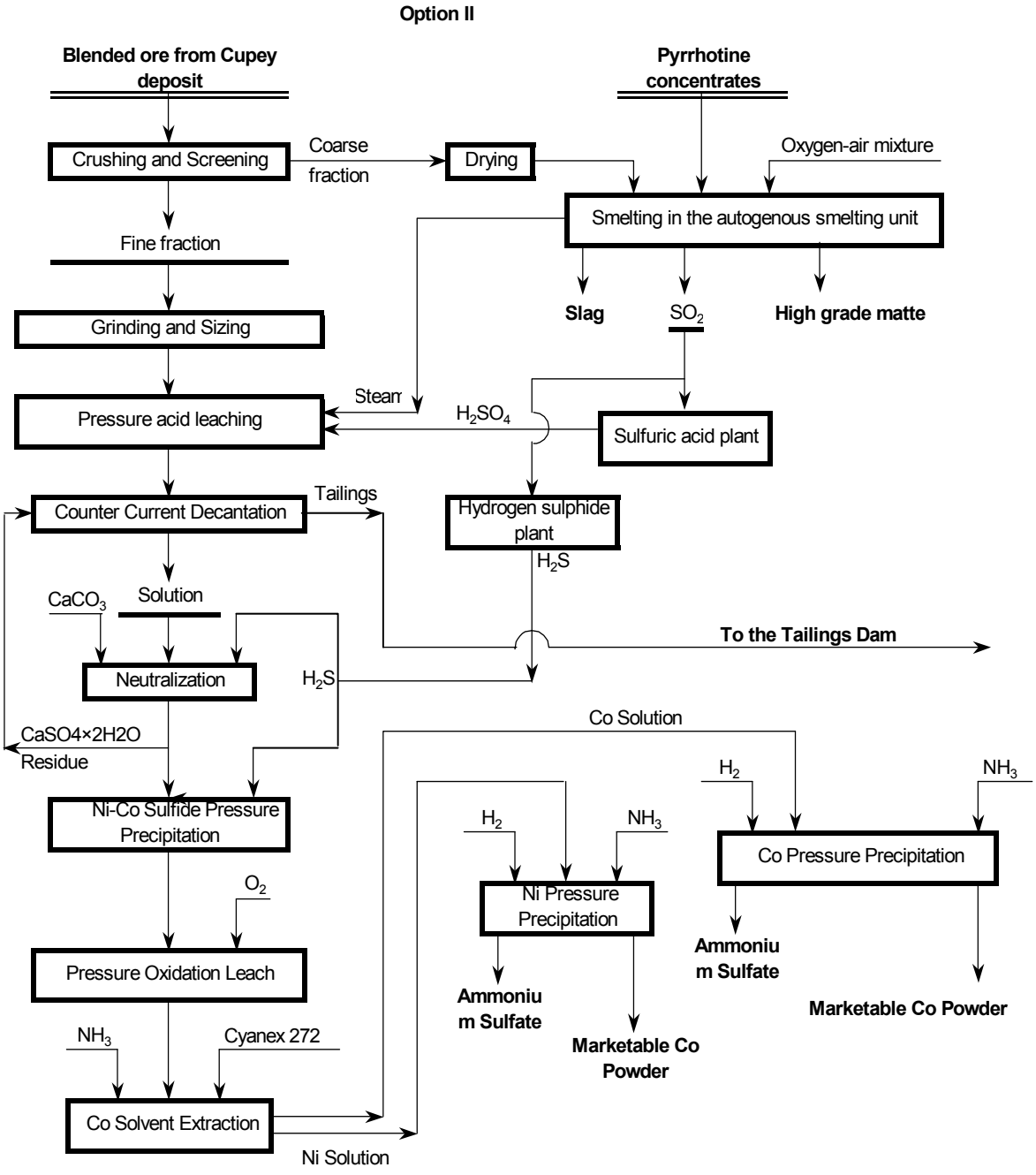

Figure 5

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NEUTRALIZATION OF ACID LEACHATE AT A NICKEL MINE WITH LIMESTONE

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ABSTRACT

Pyrites-rich discard is produced as waste during mineral processing. During oxidation of pyrites in a tailings dam, acid leachate is produced which contains high concentrations of acid, sulphate and metals. In this paper an integrated approach is proposed for dealing with the treatment of discard leachate. The approach consists of the following stages: $CaCO_3$ handling and dosing; $CaCO_3$-neutralization; and gypsum crystallization to achieve partial sulphate removal. The following conclusions were made during the investigation: (i) Powdered calcium carbonate placed in a pile can be slurried to a constant density and applied for treatment of acid water; (ii) Acid water can be treated with calcium carbonate for neutralization and removal of metals (e.g. iron(II)).

Key words: Discard leachate, calcium carbonate, limestone, gypsum crystallization.

1. INTRODUCTION

Mine waste discard, that contains pyrites, is produced as a waste during mining operations. When pyrites-rich waste ore is exposed to oxygen and water in the presence of iron oxidising bacteria, acid leachate is produced which contains high concentrations of acid, sulphate and metals due to oxidation of pyrites (Reaction 1).

$$2FeS_2 + 7½O_2 + H_2O \rightarrow Fe_2(SO_4)_3 + H_2SO_4 \quad (1)$$

BCL Limited, a copper-nickel mine in Botswana, mines and processes 450 t/d of ore and experiences such a problem. The operations consist of underground mining, concentration of the copper and nickel part of the ore by means of flotation, and smelting of the concentrate to produce copper and nickel. The main flows of water into the underground workings include cooling water (with high NaCl content from the ice plant), groundwater (fissure water) and water recycled with the coarse waste backfill. These streams are currently mixed and returned to surface where the combined stream of 350 m^3/h is neutralised.

Central to the water network is the Mill Return water Sump (MRWS). The used-water streams are recycled to the MRWS, from where the concentrator circuit is supplied with water. Lime is used to adjust the pH of the return water to 8.5 in the MRWS. This water is used in the concentrator circuit as transport medium and to facilitate separation. The pH of the water is the main quality consideration for the concentrator - high salinity levels do not pose a problem. In the copper-nickel concentration processing plant, solid waste material containing 5% pyrite is produced. The coarse fraction of the solid waste material is discarded as backfill underground, while the fine waste is discharged onto a tailings waste dump. These wastes give rise to acidic leachate due to pyrite oxidation. Lime is used to neutralise 350 m^3/h of underground mine water (with an acidity of 235 mg/l as $CaCO_3$) and 60 m^3/h of tailings dump seepage (with an acidity of 5000 mg/l as $CaCO_3$). Excess water is used for the cooling and granulation circuit in the smelter. The smelter intake water chloride concentration should be limited to 5 mg/l to prevent pitting corrosion in the smelter cooling jackets, so for this purpose raw water is imported from a local dam.

BCL currently experiences the following water-related problems:

- Neutralised water is discharged into a public stream at a rate of 300 m^3/h. The effluent quality does not meet the permitted level of 500 mg/l sulphate.
- The neutralisation cost is high due to the use of imported lime.
- Excessive acid seepage has resulted in deterioration of the land area adjacent to the tailings dump.

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- The water intake of 300 to 400 m^3/h is expensive.

A modelling exercise was carried out during 1999 to audit and simulate the water network of BCL with the aim to identify the optimum water management strategy (van Tonder, *et al*, 2000). It was found that discard leachate could be neutralized with limestone to minimise chemical cost and that it should be treated, before being mixed with less polluted streams, to achieve maximum sulphate removal through gypsum crystallization and precipitation. The latter will result in reduced scaling of gypsum in the metallurgical plant.

The purpose of this study was to:

- Determine through laboratory studies, pilot-scale studies and full-scale experience gained at Navigation Section of Landau Colliery the most suitable effluent treatment configuration for neutralization of discard leachate at BCL. An integrated process existing of the following stages was investigated: $CaCO_3$ handling and dosing; $CaCO_3$-neutralization; and gypsum crystallization to achieve partial sulphate removal.
- Develop design criteria for the construction of a plant to treat 50 m^3/h of discard leachate.

2. MATERIALS AND METHODS

2.1 FEEDSTOCK

Powdered calcium carbonate, a by-product from the paper industry, was used for neutralization of acid water. It contained 25% moisture and 10% impurities (dry mass) which was mainly silica. Coal discard leachate or a synthetic solution of similar chemical composition was used as feed water for studies on iron(II)-oxidation at low pH and $CaCO_3$ neutralization.

2.2 EQUIPMENT

The three stages of the integrated process were studied separately.

The *$CaCO_3$ - handling and dosing system* (Photo 1) was evaluated on the first full-scale plant of its kind. It has a capacity of 23 t/d $CaCO_3$. The plant consists of the following:

- A sloped concrete slab onto which the $CaCO_3$ powder is dumped and stored. The $CaCO_3$ powder is slurried with a water jet and collected in a slurry tank through gravity flow.
- Float valve in the slurry tank to maintain the water level at a specific height.
- $CaCO_3$- recycle slurry pump that withdraws slurried $CaCO_3$ from the slurry tank or clear water through a water jet onto the $CaCO_3$ dump to maintain a constant $CaCO_3$ concentration. The slurried $CaCO_3$ is returned by gravity via the sloped concrete slab back to the slurry tank. The $CaCO_3$ concentration is controlled by a density meter which directs the water jet on to the $CaCO_3$ dump when the slurry density is below a set value and onto a clean section of the slab when the density is equal to or above the set slurry density. A side-stream from the delivery side of the recycle pump is passed through the density meter. The density meter works on the basis where the mass of a fixed slurry volume is measured on a continuous basis with a load cell.
- Transfer pump, feeding slurried $CaCO_3$ to the neutralization reactor.

Iron(II)-oxidation at low pH was studied by doing laboratory studies under batch conditions. The solutions in the beaker reactors were stirred continuously and aerated with compressed air through diffusers (porosity no. 2, 210 x 8mm (OD)). The air to the container reactors and box reactors was distributed through small holes punched into a perspex pipe situated at the bottom of the reactor. Varius support media were evaluated to

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identify the most suitable medium for iron(II)-oxidation at low pH. Photo 2 shows Geotextile as support medium.

The *$CaCO_3$ neutralization stage* consisted of a fluidised-bed reactor with a sludge separator. The $CaCO_3$-neutralization stage was a continuous laboratory-scale plant. The dimensions are indicated in Table 1. Compressed air was used for iron(II) oxidation when powder $CaCO_3$ was used for neutralization..

Table 1. Dimensions of $CaCO_3$ neutralization pilot plant.

Parameter	Value	
	Fluidised-bed	**Solids separation**
Feed rate (l/h)	24	
Recycle rate (l/h)	200	
Diameter (m)	0.20	0.53
Water height (m)	4.99	0.35
Specific surface area (m^2/m^3)	20.2	-
Up-flow velocity (m/h)	6.37	0.91
Residence time (h)	6.53	3.22

2.3 EXPERIMENTAL

The performance of the various stages (iron(II)-oxidation and $CaCO_3$ neutralization) were evaluated by determining the chemical composition of the feed and treated water during batch experiments and during continuous operation.

Batch studies were carried out in beakers at atmospheric pressure to determine the rate of iron(II) biological oxidation. The following steps were followed:

- Water in the reactor was replaced with new feed water. The same support medium was used repeatedly during consecutive batch runs. During the first run the reactor was inoculated by adding 5% discard leachate from Navigation Mine to the volume of the beaker.
- Samples were taken at different intervals, filtered and analysed for iron(II) concentration and pH (beginning and end of the experiment).
- The run was stopped when iron(II) was completely removed.
- The procedure was repeated for several iterations until the rate of iron(II)-oxidation had stabilized.

2.4 ANALYTICAL

Samples were collected regularly and filtered through Whatman No 1 filter paper. Sulphate, sulphide, alkalinity, calcium, iron(II), mixed liquor suspended solids (MLSS), volatile suspended solids (VSS), acidity and pH determinations were carried out manually according to procedures described in Standard Methods (APHA, 1985). Calcium was analysed using atomic absorption spectrophotometry. Acidity was determined by titrating the solution to pH 8.3 using NaOH. The COD samples were pre-treated with a few drops of H_2SO_4 and N_2 to strip off H_2S gas.

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3. RESULTS AND DISCUSSION

3.1 $CACO_3$ HANDLING AND DOSING SYSTEM

Waste $CaCO_3$ from the paper industry was used for neutralization of acid water in the Primary Neutralization plant at Navigation since July 2001 and for neutralization of acid leached from the coal in the Coal Processing Plant since 12 June 2002 (Strobos, *et al*., 2002). During the first 12 months of operation the limestone throughput was limited to 2.5 t/d as only the Primary Neutralization Plant was served with limestone. Since July 2002, the throughput was increased to 20 t/d as limestone was also used to neutralize acid leached from the Coal Processing Plant. During operation of the Limestone plant at a high through-put of 20 t/d it was learned that the following operational guidelines need to be followed to allow smooth operation:

- Slurry limestone on slab to run-off completely into the slurry tank. No obstacles (e.g. sieves) should be positioned on the slab with the aim to separate stones from the fine particles) as it works against the slurry process. Such obstacles place a limit on the slurry density. A slurry density of only 1.09 can be achieved with sieves on the slab, while a density of 1.5 would be required once the through-put has increased to the designed rate of 40 t/d (when discard leachate plant is in operation).
- The density of the limestone slurry needs to be monitored and controlled on a continuous basis. A density meter was developed for this purpose, as described under Materials and Methods.
- Stones need to be separated completely from the limestone to prevent blockages in the slurry pipelines and in the nozzles.

3.2 IRON (II)-OXIDATION AT LOW PH

Figure 1 shows the rate of iron(II) removal at low pH with brown Geotextile as medium (Nengovhela, *et a.l*, 2002). It is noted that the rate of iron(II)-oxidation stabilised after 14 repeated batch studies. Bacterial growth has increased to the level where further bacterial growth is controlled by the available surface area of the Geotextile, oxygen in solution and the temperature. A maximum rate of 16.1 g Fe/(l.d) was determined for iron(II)-oxidation with Geotextile as medium. This is significantly higher than achieved with other support media (after 8 repeated batch studies) (Table 2). Du Preez and Maree (1994) showed that the rate of iron(II) oxidation is related to the surface area of the medium.

3.3 CACO3-NEUTRALIZATION, IRON(II)-OXIDATION AND GYPSUM CRYSTALLIZATION AT NEUTRAL PH

Limestone can be used in the integrated process for treatment of acid water (Maree, 1997). In this process powdered $CaCO_3$ is used for iron(II)-oxidation at pH 5.5, neutralization, metal precipitation (e.g. Fe^{3+} and Al^{3+}) and gypsum crystallization, in the same reactor. The novelty of this development lies in the fact that conditions were identified where iron(II) can be oxidised at pH 5.5, by the addition of $CaCO_3$. Previously, lime was used to raise the pH to 7.2 where the rate of iron(II)-oxidation is rapid. Table 3 shows the results obtained when synthetic discard leachate was treated with limestone. The water was neutralised effectively and sulphate was reduced from 8 342 to 1 969 mg/l (as SO_4) (Maree *et al.*, 1998). It was possible to achieve complete iron(II) oxidation by using only $CaCO_3$ as the neutralization agent.

It was determined that the rate of iron(II)-oxidation is not only influenced by the iron(II), hydroxide and oxygen concentrations as suggested by Stumm and Lee (1961), but also by the suspended solids concentration as suggested by Maree *et al.* (1998). In order to achieve complete iron(II) oxidation sufficient reaction time was allowed for gypsum

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crystallization to reach its saturation level (2 h). Aeration and sludge recirculation were applied to maintain a suspended solids concentration at 50 g/l.

Table 2. Comparison between support media (pH=2.5; after 8 repeated batch studies)

Support medium	Iron(II)-oxidation rate (g Fe/(l.d))
Control	1.44
Rings (100g/l)	1.68
Pellets(100g/l)	2.64
Discard(100g/l)	2.88
Sand(100g/l)	4.32
Anthracite(100g/l)	4.49
Activated Carbon (100 g/l)	5.16
White geotextile (100 g/l)	6.25
Brown geotextile (100 g/l)	6.34
Grey geotextile (100 g/l)	6.98

Table 3. Chemical composition of feed (synthetic discard leachate) and $CaCO_3$ treated water.

Parameter	Feed	Treated
pH	1.8	6.6
Acidity (mg/l $CaCO_3$)	7 300	100
Sulphate (mg/l SO_4)	8 342	1 969
Ortho phosphate (mg/l P)	2.9	0.0
Chloride (mg/l Cl)	27	30
Iron(II) (mg/l Fe)	2 500	<56
Total iron (mg/l Fe)	2 500	<56

3.4. FULL-SCALE PLANT

The information discussed above was used for the design of the BCL plant with a capacity of 50 m^3/d. The design was required to be flexible so that either crushed limestone, powder limestone or a mixture thereof could be used, depending on the availability of limestone and the iron(II)-concentration of the feed water. The process consists of the following stages:

- $CaCO_3$ handling and dosing system.
- Biological iron(II)-oxidation stage. Initially water with a low iron(II)-concentration (Red Lake) will be used as feed water. Red Lake water contains only 100 mg/l iron(II) due to natural oxidation while stored for several months in the Red Lake. Trench water will be fed later directly to the neutralization plant and the Red Lake will be used for storage of waste. This reactor is designed to treat a small stream of trench water to obtain design criteria for treatment of the total stream.
- Fluidised-bed reactor for neutralization of acid water with crushed limestone. No aeration is provided in this reactor as crushed limestone particles get scaled with gypsum and ferric hydroxide in the presence of aeration.
- Complete-mix reactor and thickener for limestone neutralization with powder $CaCO_3$, iron(II)-oxidation and gypsum crystallization.

Table 4 shows the dimensions of the various stages of the plant. The construction of this plant is complete and it has been commissioned during November 2002 (Photo's 3 to 7).

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The treatment approach offers the following benefits:

- The least expensive alkali, chrushed limestone, is used for neutralization of the acid.
- Removal of the bulk of the sulphate concentration through gypsum crystallization.
- Reduced scaling in the metallurgical plant due to separate treatment of discard leachate to the level where it is saturated with respect to gypsum.

Table 5 shows results collected during commissioning of the plant when red lake water (low iron(II) concentration of 100 mg/l) was used as feed water. It is noted that:

- Acidity was reduced from 2 100 to 50 mg/l (as $CaCO_3$).
- PH was raised from 1.9 to 6.6.
- Iron(II) was removed from 100 mg/l to <20 mg/l (as Fe).

4. CONCLUSIONS

The following conclusions followed from the investigation:

- Powdered calcium carbonate in a dump can be slurried to a constant density and applied in the treatment of acid water.
- Acid water, rich in iron(II), can be treated with calcium carbonate for neutralization and, removal of metals (e.g. iron(II)).

5. ACKNOWLEDGEMENTS

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- **National Research Foundation (NRF)** who provided funding through their Technology and Human Resources for Industry Programme (THRIP) for CSIR projects on neutralisation and sulphate removal.
- **CSIR** who provided substantial financial support for the research programme.
- **Golder Associates Africa** who did the detail engineering design for the full-scale plant.
- **Thuthuka Project Managers** who did project management for the full-scale plant.
- **Anglo Coal (Navigation Section of Landau Colliery)**, where experience obtained in the operation of the first full-scale limestone handling and dosing system and neutralization of acid mine water with powder $CaCO_3$, was used for the design of the full-scale plant at BCL.
- **Water Research Commission** for their financial support during the idea stage of development of the technology.

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Photo 1. Limestone makeup and dosing system at Navigation

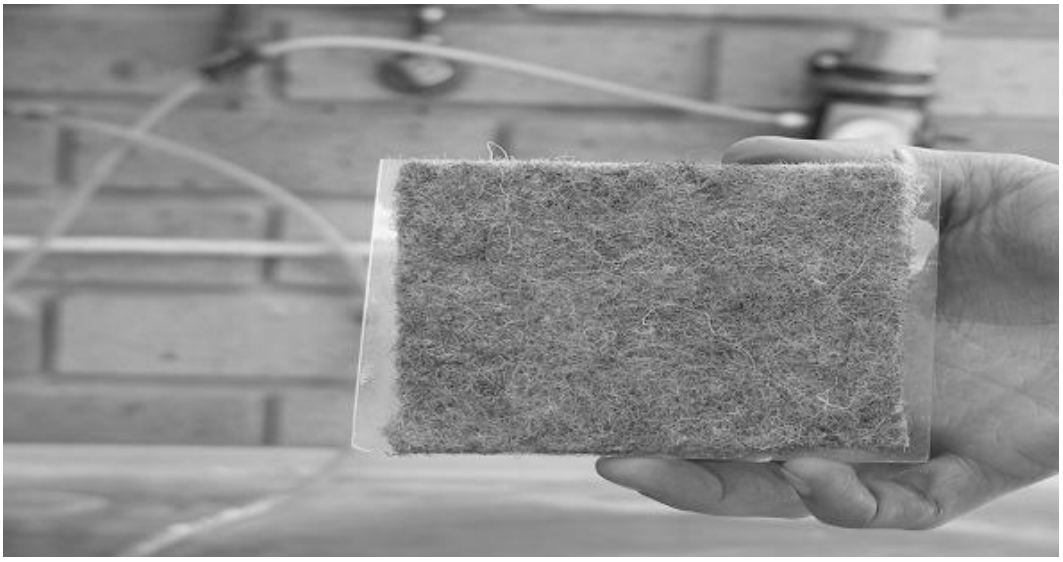

Photo 2. Geotextile mounted on a perspex plate

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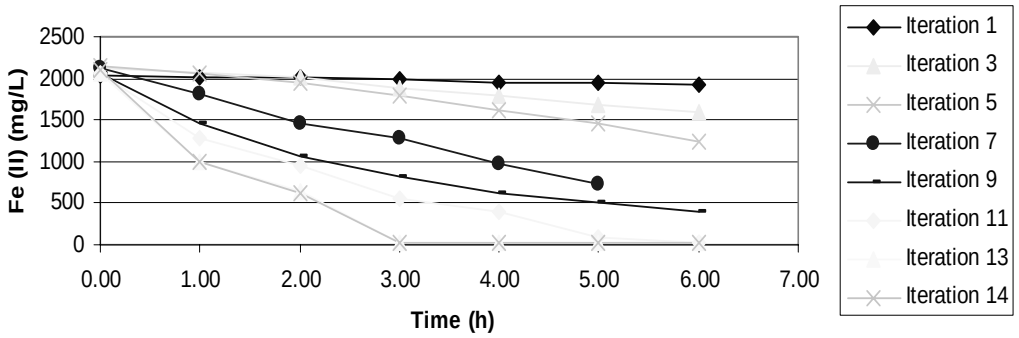

Figure 1. Effect of Iteration on the rate of iron(II)-oxidation using brown geotextile.

Photo 3. BCL neutralisation plant: Red lake

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Photo 4. BCL neutralisation plant: Limestone slurry tank, feed and recycle pumps

Photo 5. BCL neutralisation plant: Iron(II) – oxidation and fluidised-bed reactors

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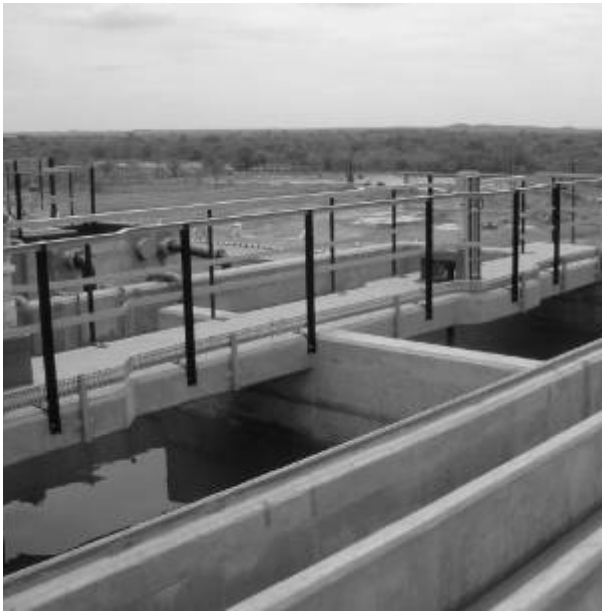

Photo 6. BCL neutralisation plant: Complete-mix reactor for gypsum crystallization

Photo 7. BCL neutralisation plant: Thickener

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Table 4. Dimensions of the various stages of the integrated limestone neutralization process.

Parameter	Stage				
	Iron(II)-oxidation	Limestone neutralization		Gypsum crystallization	Solids separation
		Column section	Cone section		
Flowrate (m^3/h)	50.0	50.0	50.0	50.0	50.0
Length (m)	3.2	9.3	2.4	10.0	15.0
Width (m)	3.2			5.0	5.0
Diameter (top) (m)		1.9	4.6		
Diameter (bottom) (m)		1.9	1.9		
Height (m)	4.0	9.3	2.4	4.0	3.4
Area (m^2)	10.0	2.7	16.6	50.6	75.0
Volume (m^3)	40.0	25.0	39.6	202.5	255.0
HRT (h)	0.8	0.5	0.8	4.1	3.8
Upflow velocity (m/h)		18.6	3.0		0.7

Table 5. Chemical composition of feed and treated water.

Parameter	Feed	Treated
Flow rate (m^3/h)	50	50
pH	1.9	6.6
Acidity (mg/l $CaCO_3$)	2 100	50
Iron(II) (mg/l Fe)	100	<20

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HIGH DENSITY RESIDUE TRANSPORT AND DISPOSAL

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1. INTRODUCTION

Hydraulic conveying of solids is common in mining and processing. Mineral slurry pipelines transport products in an environmentally acceptable and economically efficient manner over long distances ranging from a few kilometres to several hundred kilometres. Tailing disposal pipelines transport waste materials distances ranging a few hundred meters to several kilometres. From a disposal viewpoint it is no longer environmentally and economically acceptable to transport waste materials at low concentrations to large tailing catchments.

Thickened tailings disposal systems and stope filling procedures are now actively sought and require much higher concentrations to be transported than can be delivered by conventional pipelines. High concentration, fine suspension (paste) pipelines require different design methods. The processing and pipeline transport of much higher concentrations requires special thickening and pumping equipment. GEHO PUMPS has played an active role in the development and realisation of high concentration tailings disposal systems. The sharing of the experience and lessons learned may further enhance current pipeline practice for existing and future applications.

The practice has been to transport tailings and waste from industrial processes by pipeline, trucks, conveyor or gravity. For the production facility, tailings transport is primarily a logistics function, which must be ensured to sustain production. From that perspective the preferred method of transport handles the tailings/waste consistency with a minimum of preparation and at the lowest cost. Any requirement needed by the deposition ads costs and complexity. It should be emphasised that a principally different approach is required to make thickened tailings disposal a success.

2. TRIPARTITE SOLUTION

The pioneering first systems were the result of combined efforts involving a few key equipment suppliers in the development of the concept, laboratory testing, process design, equipment lay out, process control, commissioning, in-situ verification and adjustment of parameters and the instruction and training of operators. The prerequisites for a high density tailings disposal system to be effective are that the specialised pump supplier, thickener supplier and the disposal expert have overall system knowledge and experience, that they work closely together and that the interaction is managed. GEHO PUMPS has the experience and testing and analysing capability to verify that a stackable paste consistency is indeed pumpable with appropriate flow behaviour and to decide which type and size of pump is the most suitable.

The more conventional approach whereby the disposal system is pre-engineered and pumps, thickeners or mixers are merely supplied against an equipment specification involves avoidable risks and has in a few instances resulted in inadequate system performance. If one component in the system does not meet the specified duty (as may be amended during the plant commissioning) operational problems with other equipment such as the main pumps will likely result. Inadequate performance usually results in lower solids concentration and density than design and is manifested by erosion channels on the tailings deposit and flatter than expected stack slopes, as opposed to desired slopes in the range of 3 to 6%. Most importantly the concentration must be controlled within a rather narrow range. A tendency to increase the margin between the actual pipeline operating pressure and design pressure by lowering the flow velocity and solids concentration may cause segregation and sedimentation of particles. The operator, therefore, needs to understand the rheological mechanisms involved and cannot rely solely on automation systems and controls.

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Tailing materials and disposal sites are all different and each requires a unique solution and a different approach to engineering and its operation. This results in relatively complex systems, a fact that has at times been underestimated by some vendors and engineers. The associated equipment, instrumentation and control systems are more sophisticated. These systems are not so forgiving of variations in tailing characteristics, concentration and flow rate, poor equipment performance and operator intervention. In a co-ordinated approach vital aspects will not be overlooked or underestimated and satisfactory system performance is ensured.

The work scope for a conceptual pipeline transport system requires material characterisation, slurry chemistry, slurry rheology and slurry concentration from a sufficiently wide range of representative product samples. It includes testing and calculations for pipeline operation, stoppage and restart pressures with definition of the operating envelope in terms of flow rate, concentration, pressure etc. Occasionally it may be concluded that a particular product should not be pumped at high density since the risk of non-performance is considered unacceptable.

For dry stack disposal and back fill, it is important that the solids and fluid be immobilised so as to minimise or prevent leaching and drainage of deposits. This is normally achieved by increasing the solids concentration such that the fine particles combine with the conveying fluid to form a non-Newtonian suspension. In the case of cemented backfill slurries this is achieved by adding fine and chemically reacting particles. With 'stable' suspensions (true pastes), the yield stress is sufficiently high to support the coarser particles and they do not require turbulent support during storage and transport provided the particle concentration and distribution is controlled. This is in contrast to conventional turbulent heterogeneous suspensions where the solids and fluid are separated and successful transport requires that a minimum transport velocity be maintained. An increasing number of industrial (stabilised) suspensions, however, are typified by high solids concentrations with a wide particle size distribution and, despite being statically stable, coarse particles will settle i.e. migrate to the bottom of a horizontal pipe when pumped and travel slower than the superficial bulk velocity. Generic research is undertaken to better understand the fundamentals involved in coarse particle actions and their effect on laminar, transitional and turbulent flow. GEHO PUMPS understands the various flow regimes and the nature of coarse/high concentration flows and has an extensive rheology database of industrial samples tested and of systems built and operating.

3. HIGH DENSITY RESIDUE TRANSPORT AND DISPOSAL SYSTEMS

The drawbacks of transporting waste materials at low concentrations to large tailing catchments can be overcome with High Density Residue Transportation and Disposal Systems. The aim of this technology is to prepare the slurry at a concentration where it behaves as a viscous fluid which will distribute over the disposal site by gravity flow, but from which, because of the concentration, there is none or very little water runoff to form a pond.

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3.1 SYSTEM DESCRIPTION

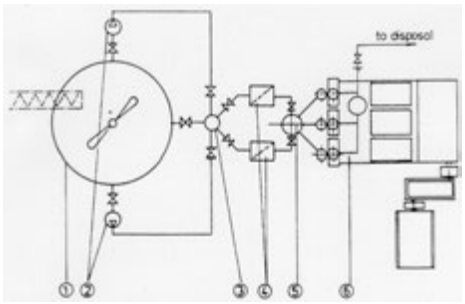

Fig. 1 Typical layout of high density ash slurry pumping station.
1. Slurry tank, 2. Booster pumps,
3. Lobster pot, 4. Sieve, 5. Suction dampeners, 6. GEHO pump.

GEHO PUMPS proposes technology based on high-density residue transportation and disposal as is commonly used in the mining and mineral processing industry (i.e. at alumina refineries). Depending on the application slurry mixture / filter residue is discharged into a intermediate slurry tank (Fig. 1) which allows for a minimum residence time or thickener underflow is directly discharged into the suction of a centrifugal booster pomp. The slurry content of the tank is continuously agitated to maintain a homogeneous mixture. The GEHO Piston Diaphragm Pump takes its suction from this tank/thickener via a booster pump and transports the high-density slurry to the disposal area. A variable speed motor controlled by the slurry tank level drives the pump. The slurry consistency is verified by a density and pressure drop measurement in the suction line or separate control system on the tank. These signals can be used to re-adjust the mixing ratios, thickener performance or for adding additional water into the intermediate slurry tank. The solids concentration is carefully controlled, as it should be as high as possible, to reduce required water quantities. Above a certain solids concentration in some instances the slurry tends to suddenly increase in viscosity, as a result of which the friction loss in the discharge pipeline increases dramatically.

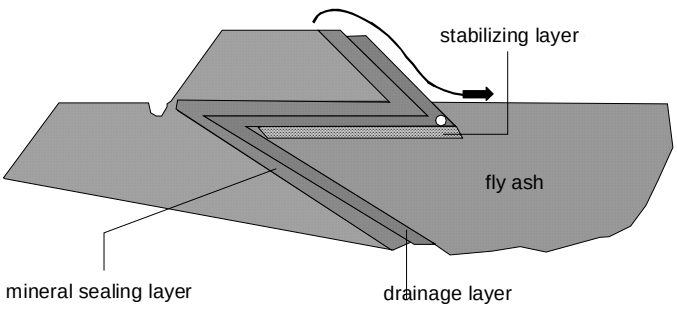

Fig. 2 High-density ash slurry disposal site.

The resulting rheological behaviour is completely different from the low concentration slurry disposal systems. The produced slurry is (almost) homogeneous and viscous. The slurry discharged as such has a homogeneous, more viscous character. As a result its flowing character, when compared to low concentration slurry disposal, is substantially reduced. For that reason a solid, expensive pond construction, like in the case of low concentration slurry disposal, is not required (Fig. 2).

3.2 DISPOSAL SITE

When disposing slurry just below its "critical" concentration at a disposal site, it will separate hardly any or no water. It is therefore often referred to as "Dry Stacking". The slurry is distributed in thin layers promoting rapid moisture loss by evaporation. This ensures that the solids rapidly consolidate to a high density, forming a deposit, which has sufficient bearing strength to support machinery used to progressively rehabilitate the site as it is filled. The dry stacking is easier of course, where solar drying is better available. During drying time the discharge of the slurry can be removed to another discharge point at the disposal site through a ring line/distribution line system. The deposit slope forms at 3-6%, providing a surface, which will discharge rainfall at a non-erosive velocity. It is

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therefor also referred to as "Sloped Disposal". The consolidated deposit has a low permeability. Caustic/salts retained in the deposit when water is lost by evaporation are locked into the deposit, minimising the risk of leaching, and associated contamination of ground water.

3.3 TECHNICAL FEATURES

Many features of this system can be mentioned:

- Low initial disposal site costs as the introduced tailings slurry has a much stronger consistency, eliminating the need for a strong dam construction. In addition, the introduced flow rate is reduced to a minimum (more solids instead of water), for which reason the size of the disposal site can be minimised. This also applies to the size of the discharge pipeline.
- Power costs savings can be realised as a result of the strongly reduced flow rates as well as the high efficiencies of the GEHO Piston Diaphragm Pumps (up to 94%). This efficiency will not change over the service life of the pump.
- No need for an overflow or water return line system, eliminating maintenance and power consumption for this system.
- Due to the high solids concentration for certain applications it is experienced and proven that the scaling effect in the discharge line is almost eliminated. Therefore the need for turning a discharge line or disassembling and cleaning is eliminated.
- The use of the GEHO Piston Diaphragm Pump for this application eliminates almost all maintenance to the pump station. The pump, designed for handling abrasive slurries, allows for a availability ≤ 98% with scheduled short maintenance stops approximately every 10-15 weeks. The rest of the time being in continuous 24-h/day service, if required. Maintenance and operator costs are substantially reduced when compared to the low concentration slurry disposal system.

3.4 SLOPED DISPOSAL SITE OPERATING PARAMETERS

The aim of a sloped disposal site operation is threefold:

- Solids should be distributed over the site with a minimum of operator intervention.
- Solids should consolidate by drying.
- Water (or slurry) accumulation at the toe of the deposit should be minimised.

To achieve these aims the slurry quality must be controlled, and the deposit must be managed.

3.5 SLURRY QUALITY

Slurry quality, principally its concentration to achieve a rheology where the slurry will flow as a relatively homogeneous fluid with minimum of water separation, must be controlled at the slurry preparation plant. If the slurry is too "thin", the slurry runs too freely over the deposit at relatively high velocity. This causes erosion of the deposit, and a pond forms at the toe of the deposit. Channels eroded in the deposit promote further erosion from rainfall, resulting in a gradual deterioration of the deposit and conversion from a "sloped" disposal site to a "pond" site.

3.6 DEPOSIT MANAGEMENT

The sloped deposit must be managed to achieve a relatively uniform distribution across the site, and to achieve the deposit drying and consolidation aims. This principally involves the management of slurry discharge spigots to distribute slurry over the site at a rate, which is consistent with the prevailing site conditions. This includes:

- Management of the number of locations of the discharge spigots.

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The frequency of switching spigots will vary with total deposition rate, the development of the deposit, and the environmental conditions.

- Environmental Conditions
  The sloped disposal design concept is based on the deposited solids being consolidated by water loss through evaporation. Evaporation rates are controlled by:
  . The daily sunlight hours
  . Wind
  . Slurry conditions.
  While there is sufficient water in the slurry for the surface to be maintained wet by capillary rise, the evaporation rate is equivalent to the prevailing free surface evaporation at the site. Once moisture levels fall and capillary rise no longer occurs, moisture loss is controlled by more complex mechanisms, and the rate of loss rapidly falls. Surface cracking caused by deposit shrinkage on drying can open a surface to the bottom of a slurry layer, and increase the evaporation rate.

4. TYPICAL OPERATIONAL QUESTIONS

4.1 WINTER CONDITIONS

On many possible locations where such High Density Residue Transportation and Disposal Systems may find acceptance, winters can be cold with temperatures below 0°C for longer periods of time. Although there is a heat generation in the pipeline due to friction and there is a limited residence time of the slurry in the pipeline it may be required to protect the slurry pipeline from freezing.
There are several ways of protection:

- Underground Pipeline
- Thermal Isolation of the Pipeline
- Thermal Isolation with Heating (Tracing) of the Pipeline

All three solutions have proven to be working to full satisfaction.

4.2 HARDENING OF SLURRY

High amounts of CaO or other chemicals in the slurry may result in quicker hardening of the slurry with the risk of pipeline blockages. During the design stage of the installation this chemical behaviour needs to be researched. The residence time in the entire installation (mixer, slurry tank and pipeline) needs to be shorter than the acceptable hardening time. This, again, may have an influence on the pipeline diameter selection. Alternatively a higher water content may be selected to maintain a sufficiently low viscosity in the pipeline and on the disposal area. It is obvious that special requirements on the pipeline diameter and on the water content of the slurry will have an impact on the pipeline pressure loss and rheological behaviour of the slurry.

4.3 PIPELINE FLUSHING

For certain slurry types it is important to clean (flush) the pipeline on a regular basis and prior to a major shut down. The cleaning is very easy and is generally done with water. To increase the cleaning results, "pigging" is recommended. In that case cleaning balls (so-called "pigs") are introduced to the pipeline with help of pigging stations. These cleaning balls are moved through the pipeline by water. The GEHO Piston Diaphragm Pumps can generate the water pressure. In case an amount (of water of the content of the pipeline) is run through the system, an additional volume of water will be let onto the disposal area, which will be drained from the disposal site in the same way as rainwater. This amount of water can be considered minimum, compared to a short rain shower.

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4.4 LABOUR REQUIREMENTS

Once the installation is properly installed and commissioned, it will run almost fully automatic and can be controlled from the plant's Central Control Room. With respect to maintenance requirements, it can be stated that the design of the installation is based on rigid and wear resistant components reducing labour requirements to a minimum.

4.5 RELIABILITY

The proposed installation is based on a combination of proven technologies: high density slurry pumping with piston diaphragm pumps, including slurry preparation and mineral waste disposal. Although the GEHO Piston Diaphragm Pumps have an availability up to 98 %, standby equipment is often provided. Further all equipment is amply sized with adequate safety margins with respect to capacities, as well as operating conditions.

5. SPECIFIC ADVANTAGES OF THE PROPOSED SYSTEM

For more than 15 years it has proved possible to transport high-density slurries (practically without free water) through a long distance pipeline. Subsequently it is possible to dispose (waste) slurry in an environmentally safe and sound way. These possibilities became economically viable through the development of high-pressure piston diaphragm pumps. Compared with the conventional ways of transportation by means of conveyor belts, trucks or low density slurry pumping, this proposed technology offers following advantages:

5.1 ENVIRONMENTAL ADVANTAGES

- No ground water pollution because of the solidification of the slurry on the disposal site.
- No dusting because of slurry solidification.
- Easy drainage of rainwater.
- Hills can be easily constructed in order to re-create the original landscape.
- Possibility of recultivation/revegetation.
- No noise on the transportation track or in the disposal site.
- No spoilage and clean transportation track due to a fully enclosed pipeline system.
- No dirty exhaust gases from trucks on the transportation track or at the disposal site.
- The pipeline can be buried underground and remains invisible.
- No topographic limitations to the pipeline systems.

5.2 OPERATIONAL ADVANTAGES

- The slurry preparation, transportation and disposal are largely automated.
- The slurry preparation, transportation and disposal are continuous and trouble free.
- The system is not influenced by weather conditions (low temperatures, storms, heavy rainfalls etc.)
- No additional manpower or machinery is required to spread the waste over the whole area of the disposal site.
- Therefor no accommodations for personnel or equipment on the disposal site are required.
- Flexibility: a later change of the pipeline routing is possible at low costs.
- The pipeline system allows for more freedom of layout (routing).
- The installation offers high availability and reliability in summer as well as in winter seasons.
- The installation requires little maintenance.
- Easy cleaning possibilities of installation and pipeline.

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5.3 FINANCIAL ADVANTAGES

- The pump system is designed to operate 25-30 years, thus reducing the depreciation per year.
- Storage capacity disposal area (typical lifetime comparison 4.5 years (low density) – 12.5 years (high density)).
- Design and preparation costs disposal area.
- The energy consumption of the entire pumping system will generally be lower.
- The expensive operating costs (high spare parts consumption and labour) of trucks and belts is eliminated.
- No roads for trucks or observation roads for conveyor belts are to be constructed and maintained.
- Costly cleaning of trucks or conveyor belts is eliminated.
- A separate service crew (for cleaning and repair) for trucks or belts is not necessary, neither are truck drivers.
- No energy consumers (infrastructure!) on the disposal site.

6. OPERATING EXPERIENCE

The following comments refer to a few of the many operations, which have adopted the principles and have converted to thickened tailings disposal.

At the Orapa Mine in Botswana, a GEHO PUMPS diaphragm pump transports high-density Kimberlite tailings (from the thickener underflow) to a stable tailings dump without the need for water recovery. The percentage of slimes is controlled to support the coarse grit and

Picture 1: Spigot discharge of high-density kimberlite tailings. (Photo by courtesy of Paterson & Cooke, South Africa).

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stabilise the flow behaviour. Recirculation ensures that any settled coarse grit are thoroughly remixed (codisposal). A screen prevents blockages in the valves due to ingress of large oversize particles. Earlier installed plunger pumps were stripped often due to (clay) clogging of the valves. Previous test work had shown that conveyor belt transfer, even at extreme high densities, would be problematic. The slurry liquefied, spillage was observed at the transfer points and slurry contaminated the roller assemblies. (Picture 1)

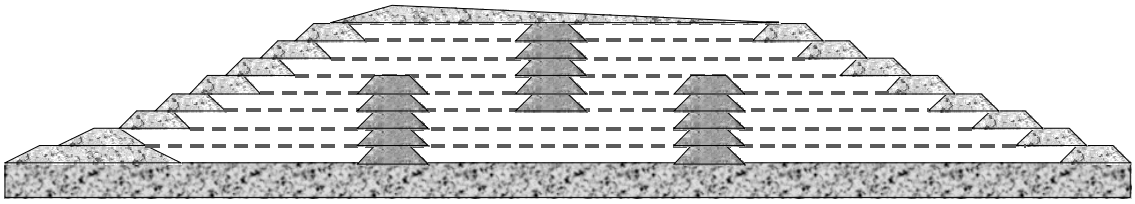

Fig. 3: Steepened stack design.

Picture 2: Red mud disposal area, Pingguo Alumina Plant, China.

China's Pingguo alumina refinery operates vacuum filters to recover a high percentage caustic soda from the bauxite residue. The thixotropic red mud is shear thinned and diaphragm pumps transport the viscous mud to the steepened stack where layers are allowed time to dry. As the soil-sealed stack reaches the top of the surrounding dam, it is a stable base for the next progressively stepped-in dam tier until it will be 80 m in height. GEHO PUMPS was charged with the design of the system, including the shear thinning and pumping plant, the pipeline and disposal site, which is monitored for seepage and ground water pollution by caustic substances. (Fig. 3)

Picture 3: Red mud disposal, Nalco, India.

At the Nalco alumina refinery operation in Damanjodi, India, GEHO PUMPS diaphragm pumps transport the red mud underflow of EIMCO Deep Cone Thickeners, through a 6700 meters pipeline to the existing conventional wet pond. After the pond is completely filled with thickened mud, the precipitation run off will channel out and the pond can be progressively reclaimed, covered with soil and vegetated. The transfer from lean phase disposal to dry stacking extends the life of the existing disposal area from 2002 until 2017!

The power industry's use of dense fly ash disposal has resulted in less pollution from seepage, ash spillage, dust storms and alkaline water discharge associated with lean phase disposal. Rek Velenje in Slovenia commenced dense phase pipeline transport to the lignite mine for backfill in 1986. In Australia, Bayswater Power Station has almost land filled the first Ravensworth Mine void by pumping over 10 Mt (dry) of fly ash over a 10km

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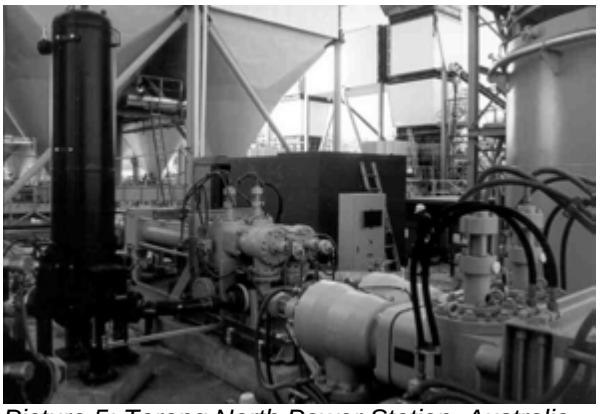
distance. The operation confirmed the diaphragm pump ability to handle the 74% concentration determined in GEHO's laboratory and that the pipeline can be shutdown daily without flushing.

Picture 4: Solidified disposal area, Ledvice Power Station, Czech Republic.

At CEZ Ledvice power station in the Czech Republic, residue from coal combustion and flue gas desulphurisation is deposited at a distance of 4,800 m into a former open pit lignite mine in a manner which meets Czech environmental legislation. GEHO PUMPS met the challenge to develop a concept, design, built, commission, performance test and warrant the preparation, (diaphragm) pumping, transportation, distribution and dry disposal of a hardening (from CaO reaction) slurry with entrained crushed slag in a rather humid and temperature climate.

Placer Dome South Deep Mine in South Africa has operated since 1993 a series of hydraulically driven transfer tube type piston pumps transporting backfill over 1,250 m from the underground crushing plant to the stopes where the 85% (Cw – concentration by weight) mixture of crushed waste and aggregate helps prevent rock bursts. Other backfill projects such as Galmoy in Ireland are using valved hydraulic piston pumps. Kali+Salz in Germany ensures the structural integrity of salt caverns with paste backfill, which is a cemented high density, and corrosive mixture of incinerator ash and superfluous caustic. An example of a large size hydraulically driven piston pump which meets the 5% pressure pulsation's specified is at Tarong North Power Station in Queensland handling 75% Cw fly ash which is on the verge of being pumpable with a diaphragm pump. The pump with actuator assisted valves must be complemented by a level controlled air vessel, associated compressor and air receiver.



Picture 5: Tarong North Power Station, Australia.

7. CONCLUSIONS

Most tailing residues do satisfy the homogeneity required for a thickened tailings disposal system. Conversion to thickened disposal by discharging a sloping tailings cap on an existing flat disposal pond facilitates reclamation and increases the storage capacity. The current knowledge, design methodology and experience available with expert companies enable the design of a disposal pipeline with a high degree of confidence. The most appropriate mainline pump type is best selected by a qualified pump supplier having relevant know how and experience. It must similarly be ensured that the available thickener technology will produce the paste consistency and quality required for disposal and pipeline transport.

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The selection of a suitable mixer, as applicable, from a broad range of different types remains a concern and an agitated tank is normally used for homogenisation. A sample loop takes slurry to a density meter which signal is used to adjust the addition of water.
It is believed that previous difficulties experienced with pumps and system functioning/performance would largely have been avoided with current technology and a co-ordinated approach to project execution, calling on expertise and experience available within the three major disciplines.

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Versatic 10 as an Extractant for Nickel and Cobalt

By

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ABSTRACT

The Versatic™ Acid 10 by Resolution Performance Products (previously Shell Chemicals) is based on a mixture of highly branched-C10 carboxylic acids. It is a cheap and stable chemical with a number of applications outside the mining industry. As an extractant it is used for nickel, but it is only slightly selective for nickel over cobalt. Also calcium may be co-extracted with nickel. Furthermore, the reagent has as an inherent drawback that when pH > 7 it becomes slightly water-soluble and when pH > 8.4 micellar aggregates are formed in the aqueous phase. Due the very sensitive selectivity and the solubility problems the Versatic system requires an accurate pH control and good understanding of the physico-chemical phenomena taking place in the process.

The paper reports experimental results and observations for the extraction of nickel from sulphate solutions in the presence of cobalt. The extractant Versatic 10 is used partly pre-neutralized with aqueous ammonia. That means that the metal cation is extracted through a cation exchange mechanism, e.g. $2\ NH_4^+ \rightarrow Ni^{2+}$. It is a common practice in the industry to use the acidic extractants in their salt form in order to facilitate the pH control. However, when using the extractant in the saponified form, it becomes extremely interfacially active and the extractant molecules form amphiphilic aggregates as has been previously reported by the authors at the ISEC-conferences.

In the current paper, a kinetic approach is applied to study the selectivity of the nickel and cobalt extraction. A high-shear mixer cell followed by a PTFE-membrane for fast phase separation is used to monitor the extraction also at very short contact times (< 5 s). It is shown that nickel is extracted very fast. However, if both nickel and cobalt are present, cobalt is extracted more rapidly while the kinetics of nickel extraction is slower. Nickel replaces the cobalt (at about 2 min) from the organic phase. The final equilibrium and selectivity is slowly approached. The amount of ammonia was continuously monitored in the organic phase and it was found that the ammonia is very rapidly transferred from the organic phase into the aqueous phase during metal extraction. The paper emphasizes the importance of well controlled pH and residence time distribution in the mixing device of a solvent extraction plant.

1. INTRODUCTION

The commercial product Versatic™ Acid 10 by Resolution Performance Products (previously by Shell Chemicals) is a synthetic saturated tertiary monocarboxylic acid. It consists of a mixture of highly branched isomers of C_{10} carboxylic acids also known as neodecanoic acids. Similar carboxlic acids were used already in the 1970's as extractants for nickel /1/ and the Versatic 10 is among the oldest commercial metal extractants still in use in the mining industry. It is a cheap and stable chemical having a number of large volume applications as a chemical intermediate outside the mining industry.

As a metal extractant it is selective for nickel over cobalt - unlike the organophosphorus extractants (e.g. the phosphinic Cyanex 272), which are selective for cobalt over nickel. Unfortunately Versatic 10 is only slightly selective for nickel over cobalt (see Fig. 1). Also calcium may be co-extracted with nickel, which has been reported to be a problem at Bulong /2,3/. To solve the selectivity problem there have been attempts to use Versatic acid in synergistic combination with a co-extractant /4,5/.

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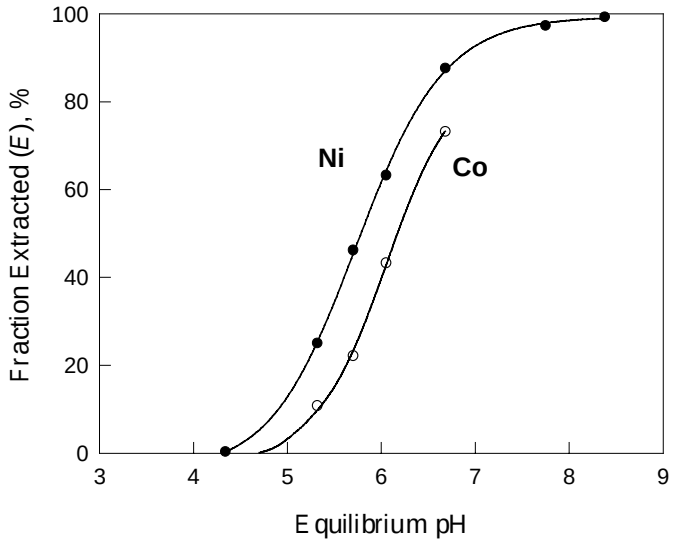


Figure 1: Isotherms of simultaneous extraction of nickel and cobalt from sulphate solutions using 40 wt.% Versatic 10 in isooctane at 25 °C.

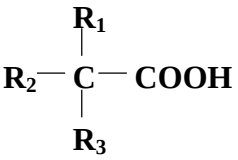
The reagent has as an inherent drawback that it is water-soluble at pH > 6 and an extra scrub stage is needed to recover the extractant from the raffinate for example by contacting it with the stripped organic at mildly acid conditions /2/.

Due the very sensitive selectivity and the solubility behavior the Versatic system requires an accurate pH control. It is usually made with ammonia or sodium hydroxide. However, when the extractant is in salt form it behaves as a surfactant that forms micellar aggregates. This paper reports some fundamental data and observations about the physico-chemical phenomena in connection with nickel extraction from a sulphate solution containing also cobalt. The aim is to offer information that helps to design and operate an SX-plant using pre-neutralised acidic extractants.

2. PROPERTIES OF THE VERSATIC 10 MOLECULE

2.1 MOLECULAR STRUCTURE

The molecular structure of Versatic 10 may be represented as:



The molecule contains a total of 10 carbons atoms. However, the reagent is a mixture of branched C_{10} isomers as well as some longer and shorter homologues (C_8 – C_{12}). In the as-received sample that we have analyzed by GC-MS, 92 mol-% of the carboxylic acids were as the C_{10}-isomers /6/. One of the alkyl groups is predominantly a methyl group (> 96 mol-%).

The highly branched structure of the tertiary carboxylic acids at the α-position determines their extraction properties as demonstrated with model carboxylic acids by du Preez and

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Preston [7]. The strong steric hindrance of Versatic 10 gives also good thermal and hydrolytic stability, resistance to esterification and low acidic strength (pK_a = 7.33) /8/.

2.2 AQUEOUS SOLUBILITY

The high solubility of Versatic 10 in the aqueous phase during the extraction process is a known drawback of this reagent. The dissolution of Versatic 10 in the water phase increases with pH. The solubility of Versatic 10 in the aqueous phase increases linearly from 0.03 to 0.25 mol/l as pH is increased from 7.0 to 8.3 (Fig. 2). At pH > 8.4 the Versatic concentration increases dramatically in the raffinate due to the formation of normal micelles.

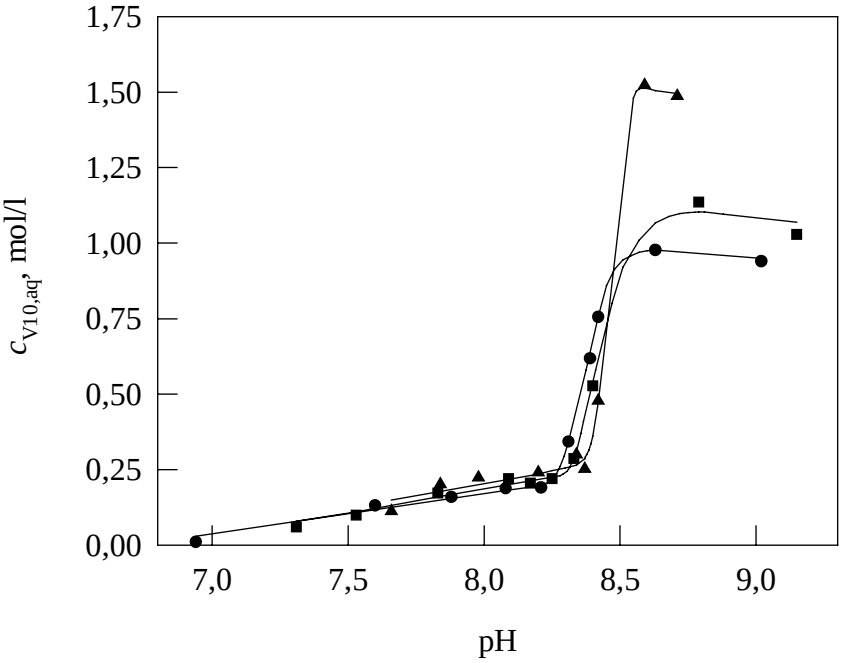

Figure 2 Dissolution and solubilization of Versatic 10 in water as a function of pH in the water(NH_3)/Versatic 10/isooctane system. Symbols: (●) 30 wt-% Versatic 10, (■) 40 wt-% Versatic 10, and (▲) 50 wt-% Versatic 10.

The different isomers and homologues have different water solubility as we have shown by chemical analysis /9/. The shorter as well as the less branched structures give higher water solubility. Although we have no proof from plant samples, it is suspected that the composition of the reagent in an extraction circle gradually shifts with time due to solubility losses.

3. PRE-NEUTRALISATION OF THE EXTRACTANT

3.1 DEGREE OF PRE-NEUTRALISATION

Acidic extractants can be partially changed to their salt form prior to contact with the metal loading aqueous solutions in order to adjust the pH. This process is here called pre-neutralisation (sometimes called pre-equilibration). The degree of neutralisation (*N*) is defined as the ratio of the amount of extractant in salt form to the total amount of extractant. In this paper the neutralisation is done in ammonia water and it can be described with the reaction formula

$$NH_3 + qH_2O + y/2(\overline{HA})_2 \leftrightarrows \overline{NH_4A \cdot (y-1)HA \cdot qH_2O} \qquad (1)$$

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where $(HA)_2$ is a Versatic acid dimer. For example $q \approx 3$ for concentrated 14 M aqueous ammonia and $y = 2$ when $N = 50\%$. Naturally, q = 0 if gaseous ammonia is used.

3.2 PHASE EQUILIBRIA IN THE PRE-NEUTRALISATION STAGE

The Versatic acid is in its ammonium salt form an anionic surfactant capable of solubilizing large amounts of water in the organic phase. The maximum of water/ammonia ratio in the extractant phase (i.e. $n_{H_2O}/n_{NH_4A} = 14.2$) is observed when N is 50% (i.e. when the ammonia/Versatic molar ratio is 1:2). Visually this is seen as a single liquid phase. The pre-neutralised extractant is in the form of a hydrotropic solution when gaseous ammonia or concentrated aqueous ammonia is used ($n_{H_2O}/n_{NH_4A} = 3$) and it becomes a microemulsion when diluted with water.

The regions of formation of the one-phase microemulsion are depicted in the series of phase diagrams shown in Fig. 3. These partial phase diagrams are for four different fixed ammonia concentrations between 2.4 M and 14 M NH_3 in water. The number of liquid phases that co-exist in equilibrium are indicated in the figures as L and 2L. Concentrations are given as percentage by weight. Notice that with increasing amount of water in the system, the one-phase region shrinks and moves towards the water corner. The location of the one-phase region follows the specific ammonia/Versatic molar ratio of 1:2 (i.e. N = 50%) shown with dotted lines in the phase diagrams. On the right hand side of the one-phase region there is a two-phase region where a “water-in-oil” microemulsion is in equilibrium with a small excess aqueous phase. The structural change and the increase of water solubilization is reflected in an exponential growth in the viscosity and electrical conductivity of the solutions /9/.

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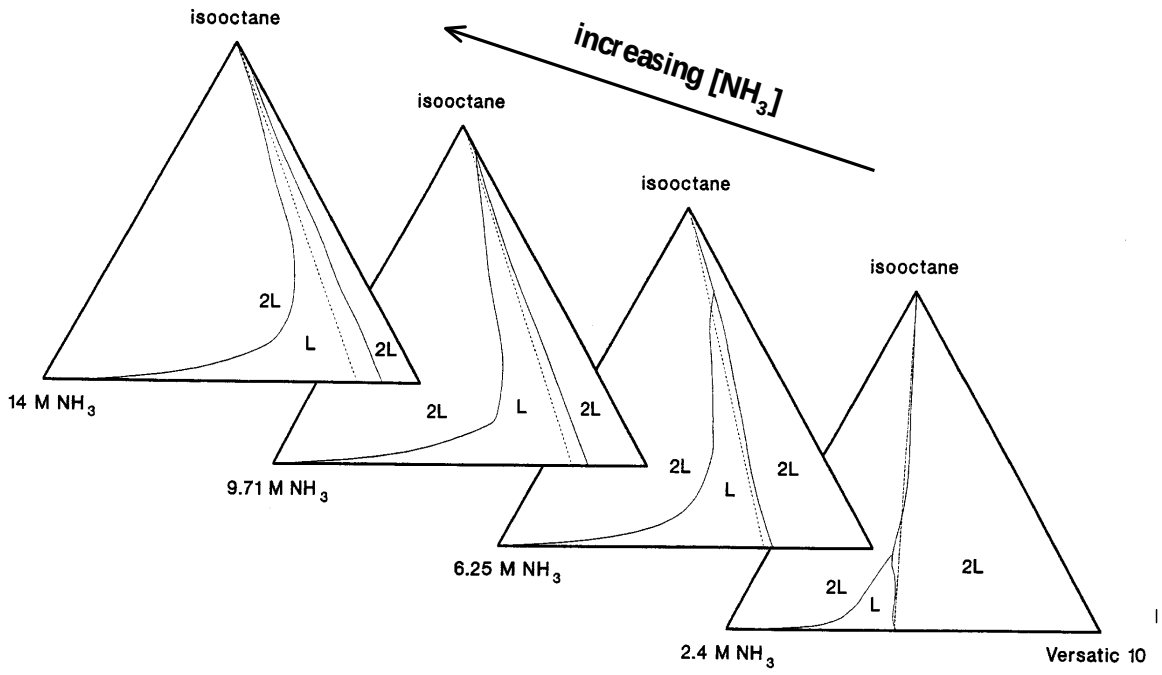

Figure 3: Partial pseudo-ternary phase diagrams for the water(NH_3)/Versatic10/ isooctane system for four different ammonia concentrations at 25 °C.
L = one liquid phase, 2L = two liquid phases. The dotted lines show the ammonia/Versatic molar ratio of 1:2. /10/.

The micellar formations are dynamic units that are constantly forming and dissociating on a time-scale of μs to ms range. Therefore phase equilibria takes place very rapidly in the pre-neutralisation stage /11/ and it may be carried out in-line prior to the SX-mixer. However, sufficient macroskopic mixing and heat removal must be arranged.

4. KINETICS OF EXTRACTION

4.1 THE RATE OF ION EXCHANGE

The kinetics of the extraction was studied using a small (V = 42 cm^3) co-current column contactor with a high speed stirrer and a settler on top of the column. The column is divided into nine compartments with individual stirrer blades as well as individual inlet ports for the second phase. Depending on the point of entrance the length of the column and consequently the contact time could be varied between 2 and 252 s. The organic phase was sucked though a hydrophobic filter located in the settler for analysis of the metals, NH_3 and H_2O (see ref. 10 and 12 for experimental details). Since the rate of extraction depends in such an apparatus on the stirring intensity, it does not give well defined kinetic data. However, the method is very convenient to measure relative extraction rates as is demonstrated with the following pictures.

The rate of nickel extraction is extremely fast when using Versatic 10 in the pre-neutralised form. About 90 % of the extracted nickel is transferred in the organic phase in the first 15 s (Fig. 4). At first contact between the aqueous feed and the pre-neutralised extractant phase, the complexation takes place on the extremely large liquid-liquid interface of the

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microscopic amphiphilic aggregates. The extractant binds Ni^{2+} through a cation exchange reaction according to the following reaction stoichiometry:

$$Ni^{2+} + (2+m)/y\,\overline{NH_4A \cdot (y-1)HA \cdot qH_2O} \quad \leftrightarrows \quad 1/x\,(\overline{NiA_2 \cdot mHA \cdot pH_2O})_x + (2+m)/y\,NH_3 + ((2+m)q/y - p)H_2O + 2H^+ \qquad (2)$$

where $y = 1/N$ and $m = 2(y\text{-}1)$.

The experimental kinetic data in Fig. 4 show the cation-exchange reaction between $2NH_4^+$ and Ni^{2+}. Simultaneously, water is transferred from the organic phase as the Ni-Versatate looses its surface activity and the micellar aggregates break up. Consequently the rate slows down and finally we have a normal organic phase and a normal aqueous phase. All ammonia is rapidly transferred to the aqueous phase but some residual water remains with nickel in the organic phase.

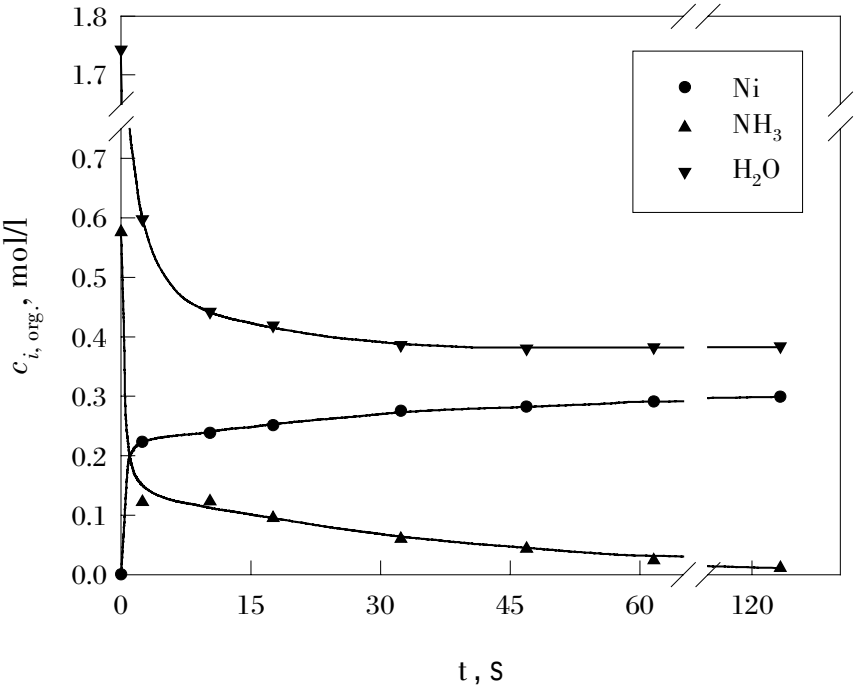

Figure 4: Extraction of nickel into organic phase and the simultaneous transfer of NH_3 and H_2O out of the organic phase. The extractant was 40 wt.% Versatic 10 in isooctane and it was partly (N = 33%) pre-neutralised with 14 M NH_3. The aqueous feed contained 0.34 mol/L Ni at pH = 3.4. T = 25 ^oC.

4.2 THE RATE OF NICKEL AND COBALT EXTRACTION

When contacting the pre-neutralised Versatic 10 with an aqueous sulphate solution containing both nickel and cobalt, it is interesting to observe that cobalt is first extracted very fast (see Fig. 5) but then an exchange reaction between nickel and cobalt takes place. Finally the same equilibrium conditions are achieved as when using Versatic 10 in acid form and adjusting the pH during the extraction. When using Versatic 10, which is pre-neutralised with aqueous ammonia, the extractant phase is electrically conductive and the pH can be measured with a combination electrode. Typically the pH is about 8, but it naturally depends on N /8/. When this solution meets the acidic aqueous feed, a pH drop

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takes place during the first seconds after phase contact as seen in Fig. 5. This partly explains the reversal of Ni/Co selectivity during the extraction.

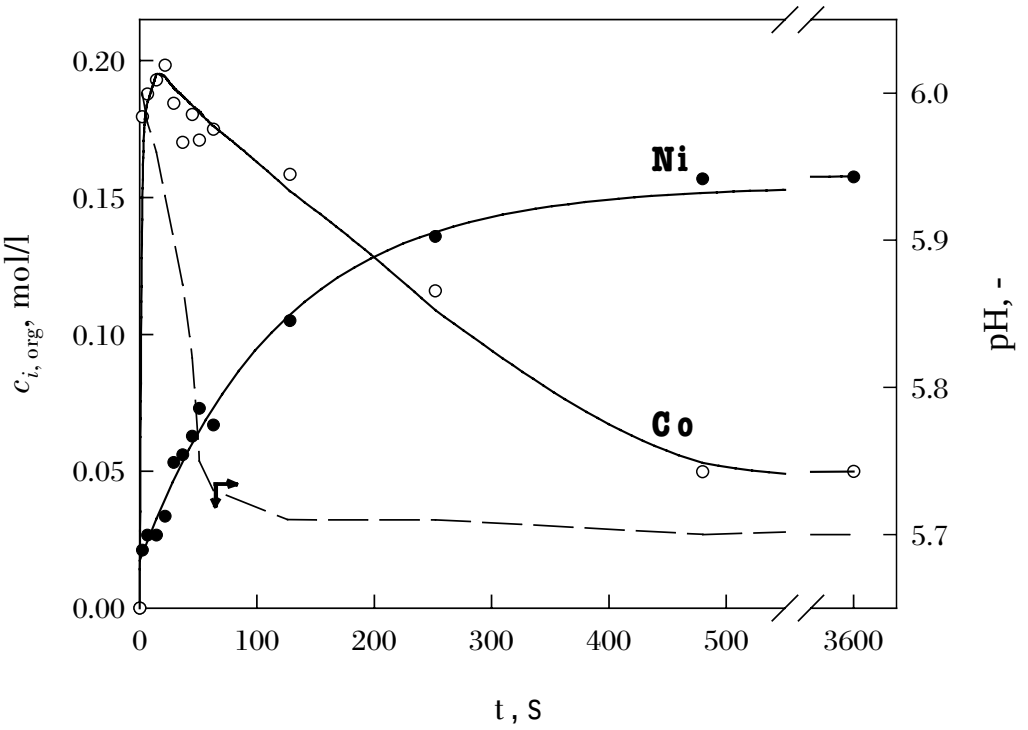

Figure 5: Kinetics of nickel and cobalt extraction using Versatic 10 pre-neutralised with aqueous NH_3. The dash line shows the pH of the raffinate. Aqueous feed: 0.34 mol/L Co and 0.34 mol/L Ni, pH = 3.43. Organic feed: 40 wt.% Versatic 10 in isooctane (*N* = 20%). T = 25 ^{o}C.

The kinetics was further studied from a solution containing nickel, cobalt and magnesium at different degrees of pre-neutralisation (*N*). The same reversal of Ni/Co selectivity is seen in Fig. 6. Furthermore we can see that the total amount of Ni, Co and Mg extracted into the organic phase follows the stoichiometry of $Me^{2+} \approx 2\ NH_4^+$.

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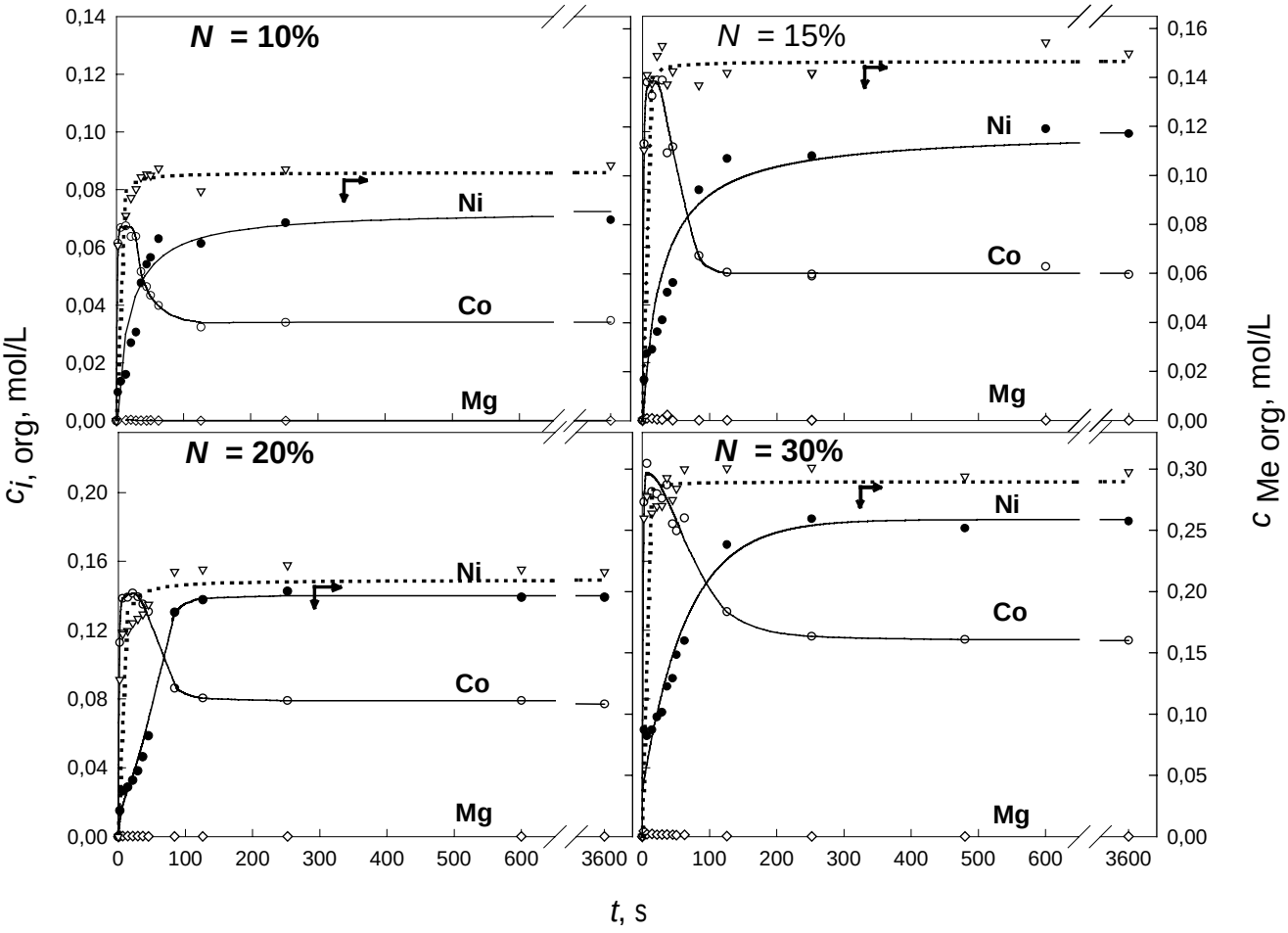

Figure 6: Kinetics of nickel, cobalt, and magnesium extraction using pre-neutralised Versatic 10. The dotted lines show the total metal concentration in the organic phase. Aqueous feed: 0.34 mol/L Co, 0.34 mol/L Ni and 0.0.082 mol/L Mg, pH=3.38. Organic feed: 40 wt.% Versatic 10 in isooctane at different degrees of neutralisation *N.*

5. CONCLUSIONS

(1) The extraction of nickel with ammonia pre-neutralised Versatic 10 is very fast and it takes place trough an ion exchange reaction according to the stoichiometry of $2NH_4^+(org) = Ni^{2+}(aq)$.

(2) With pre-neutralised extractant the Ni/Co selectivity is reversed after short contact times. Co is fist extracted and then it is replaced by Ni in the extractant phase so that the mole balance $2NH_4^+(org.feed) = Me^{2+}(aq.\ feed)$ is fulfilled in the system.

(3) If the degree of pre-neutralisation is in excess compared to the amount of extractable metals the organic phase remains micellar and the residual water and ammonia concentrations in the organic phase are high.

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(4) In a solvent extraction plant the method of pH control is important for obtaining good selectivity and for reducing extractant losses to raffinate. This requires good mixing and a narrow residence time distribution in the contacting equipment.

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SEPARATION, EXTRACTION, AND REFINING OF COBALT AND NICKEL FROM BASE METAL FEED STREAMS USING MOLECULAR RECOGNITION TECHNOLOGY (MRT)

By

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ABSTRACT

Many mine base metal ores and concentrates can be quite complex, and consequently, extraction and refining of the value metal components can be difficult and expensive. Development of a low cost process technology for treatment of cobalt, nickel, copper, and other impurity bearing feed materials, has long been of particular interest.

IBC Advanced Technologies, Inc. (IBC) has developed a range of SuperLig® products which are highly effective and economic in the selective separation and extraction of cobalt, nickel, copper and iron from leach, process and waste streams. These products are effective in both sulfuric and nitric acid matrix solutions. The value metals are recovered as salts of high purity. Various flow sheet separation alternatives based on Molecular Recognition Technology (MRT) are provided, depending on relative concentrations of each metal ion, and what is the primary target separation. A number of available options for commercial treatment are also discussed.

1.0 INTRODUCTION

Economic extraction and purification of cobalt from a wide range of potential feed materials containing nickel and other base and ferrous metal impurities has long been a major objective of the industry. Cobalt, nickel, and copper all have significant value in metallic or higher purity salt form, and the first ideal objective of any hydrometallurgical separation process should be to produce a separation product or products of maximum added value at minimum cost. Whether this can be achieved for all significant metal components depends on many factors such as concentration of each component, relative concentrations of each component, separation and elution chemistry for each component, and relative costs of the separation and elution chemistry for each component. For example, if cobalt is the major value component, it might seem logical to separate the cobalt from the other impurities, including the other metal value components such as copper, nickel, etc. This does, indeed make sense if the cobalt can be separated efficiently, and economically in a high value added form, using the appropriate chemistry. However, if the cobalt separation proves to be technically less efficient, or a higher cost, then the approach is usually to initially remove the significant impurities and leaving essentially only the target cobalt element in solution. The IBC MRT process offers considerable flexibility in the separation process sequence that can be used in practice, depending on the above factors that apply to the specific situation. In this paper we review the experimental results for separation and recovery of copper, nickel, iron, and cobalt from sulfuric acid leach solution. Basic flow sheet alternatives for this process are also provided. Similar processes are also applicable to a nitric acid matrix.

2.0 MOLECULAR RECOGNITION TECHNOLOGY

The Molecular Recognition Technology (MRT) process utilizes “lock and key”, or “host guest” chemistry. It is a highly selective, non ion exchange system, using especially designed organic chelators, or ligands that are chemically bonded to solid supports such as silica gel, or polymer substrates. Alternatively, these ligands can be used as unattached complexing agents. The solid phase system consists of the ligand material, SuperLig® packed into fixed bed columns that can be built in skid mounted modular form, and can be fully automated for continuous operation. The feed solution is passed through the column and the specific ion of interest is removed from the solution in a "lock and key” fashion.

By utilizing "host-guest" chemistry, SuperLig® products are designed to selectively bind with ions based on multiple parameters such as size, coordination chemistry, and geometry. In contrast, conventional separation methods such as precipitation, ion exchange, and solvent extraction often recognize differences between ions based only on a single parameter (i.e. charge, solubility, size). SuperLig® products can bind ions even

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when they are present at extremely low levels in the presence of very high concentrations of competing ions and/or in highly acidic or basic solutions. The MRT process exhibits high selectivity, high binding factors, and rapid reaction kinetics, resulting in a very efficient separation. The elution chemistry is quite simple, thus producing highly concentrated solutions that can be easily treated to produce marketable products of high added value with minimal environmental impact. The SuperLig® materials exhibit long life.

Due to high selectivity, high loading capacities, and rapid loading and release kinetics of the SuperLig® materials, application of IBC MRT results in substantially lower capital and operating costs than competitive technologies like ion exchange, solvent extraction, classical pH adjustment, and chemical precipitation procedures. [1-17] Because relatively small quantities of the appropriate SuperLig® product are required, the scale of installation can be smaller, solution wash and elution chemical requirements and volumes are substantially less, and higher feed solution flow rates are possible. Higher efficiencies are possible due to single pass quantitative removal. Typically, if an existing ion exchange system is already in place, SuperLig® material can replace ion exchange resin with minor modifications to equipment. SuperLig® materials have a long life and do not introduce any contaminants into the process.

MRT can be used to accomplish separations to very low levels that are not possible using other technologies. MRT can produce a 99+% pure product that can be sold or recycled. This is an important factor from the standpoints of cost, the environment and waste disposal. A wide choice of eluent formulations is usually available to ensure compatibility with particular plant requirements. Highly concentrated eluent solutions can be produced from which the simple recovery of a high purity, high value added product is possible. The result is a readily marketable product and provides significant financial credit to the process. Eluent by-product produced will also be quite pure and can often be sold. Refer to the following references for more detailed information on the SuperLig® chemistry. [1-20]

3.0 SUPERLIG® MATERIALS

Table 1 provides a summary of the key operating parameters for the SuperLig® materials discussed in this paper.

Separation and Extraction of Copper and Iron Impurities from Cobalt Bearing Sulfuric Acid Leach Solutions

4.0 EXPERIMENTAL

IBC has solid phase SuperLig® resin technology capable of removal and separation of Ni, Cu, Fe, and other impurities from concentrated cobalt solutions. Laboratory scale test work has been performed at IBC to demonstrate the capabilities of the SuperLig® resins. Tests were based on use of separate SuperLig® in each column. Two columns in series were used for individual removal of each impurity. In some cases, joint removal of specified impurities is also possible using the same SuperLig® material. These tests are described below.

For the copper and iron removal, two sets of tests were run for two different feed pH values (pH 1 and pH 2).

pH 1 Feed

For copper removal at pH 1, a single column containing 0.17g of SuperLig® 86 was used at a feed flow rate of 0.10 ml/min. Analysis of the feed solution was : Co: 85 g/l, Cu: 23 mg/l, Ni: 235 mg/l, Fe: 26 mg/l, and Zn: 300 mg/l. The wash was 0.1 M H_2SO_4. The eluent used was 8 M H_2SO_4. For iron removal at pH 1, a single column containing 0.5g of SuperLig® 14 was used at a feed flow rate of 0.3 ml/min. The wash was 0.1 M H_2SO_4. The eluent used was 37% HCl.

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Table 1: Operating Parameters for Various SuperLig® Materials Used for Separations from Acidic Cobalt Sulfate Solutions

Target Element	SuperLig® Type	Effective Target Element Concentration Range	Solution Range pH	Eluent
Cu	SuperLig® 132	Cu can be removed at virtually any Cu concentration. Optimum Ni/Co rejection	Down to pH 1.7	1-2 M H_2SO_4, or other acid
Cu	SuperLig® 152	Cu can be removed at virtually any Cu concentration	2-3 M strong acid	4-5 M strong acid
Cu	SuperLig® 86	Cu can be removed at virtually any Cu concentration	Very strong acid	Requires chelant elution
Cu, Fe	SuperLig® 145	Binds to Cu & Fe (III)	~ pH 2	2 M H_2SO_4
Fe	SuperLig® 14	Binds to Fe (III) only.	Maximum pH of 2.0-2.2 preferred for Fe(III) solubility	1-2 M H_2SO_4
Ni	SuperLig® 199	Optimum Co/Ni ratios are 200/1 or less. Final Co/Ni ratios can be 10,000/1 and greater, depending on flow rate, starting solution viscosity, and number of Ni polishing columns.	Down to just below pH 1. Co rejection best by running feed near pH1(0.1M strong acid)	2-5 M strong acid, depending on acid used
Co	SuperLig® 138	Co can be removed at virtually any Co concentration. Selectivity over Cu is <u>not</u> present.	~pH 0.5	2-5 M strong acid, depending on acid used. Reducing agent must sometimes also be present to elute any Co(III).

pH 2 Feed

This test was performed by removing the Cu and Fe together on a selective basis. For the Cu and Fe removal at pH 2, a single column containing 0.5g of SuperLig® 145 was used at a feed flow rate of 0.15 ml/min. The feed solution analysis was: Co: 85 g/l, Cu: 23 mg/l, Ni: 240 mg/l, Fe: 31 mg/l, and Zn: 310 mg/l. The wash was 0.01 M H_2SO_4. The eluent used was 2 M H_2SO_4.

4.1 RESULTS AND DISCUSSION

4.1.1 pH 1 Feed Tests Copper/Iron Removal

Both the Cu and the Fe were readily and separately removed using the two SuperLig® materials in series. A typical loading cycle for the Cu separation using SuperLig® 86 is shown below in Figure 1. The test indicated excellent Co rejection. A single column was used with approximately ½ capacity loading since this configuration is usually more economic at relatively low impurity concentrations in the feed solution than using two columns in series and fully loading the first column.

Figure 2 below shows the elution curve for the copper stripped off the column. The curve demonstrates how the Cu can be collected as a small volume concentrate with minimal impurities present. At laboratory scale a concentrate of approximately 3 g/l Cu was obtained. At larger scale a concentrate of ~ 20 g/l is typically obtained from a ~ 50% loaded column. The tail, or second half of the elution can be recycled as the front half of the next elution. This particular SuperLig® material offers the advantage of successfully

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removing Cu at any pH level with a varying feed. It also offers the highest Cu over Co selectivity of the resins presently available from IBC that use H_2SO_4 for elution.

Figure 1: Partial Loading Profile for Cu Binding at pH 1 on a Single 0.17g SuperLig® 86 Column at Flow Rate of 0.10 ml/min.

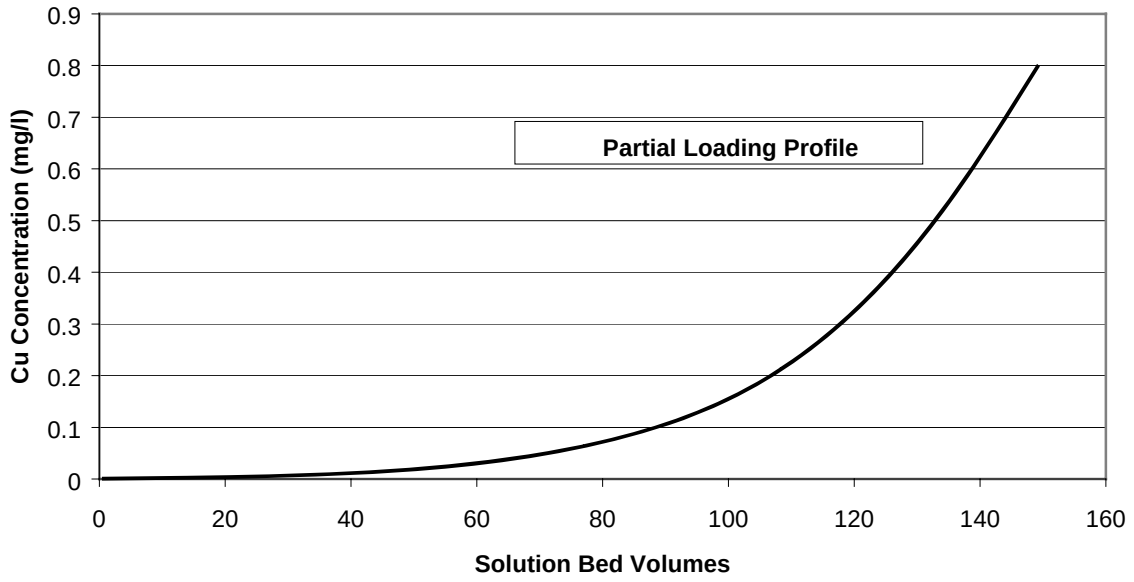
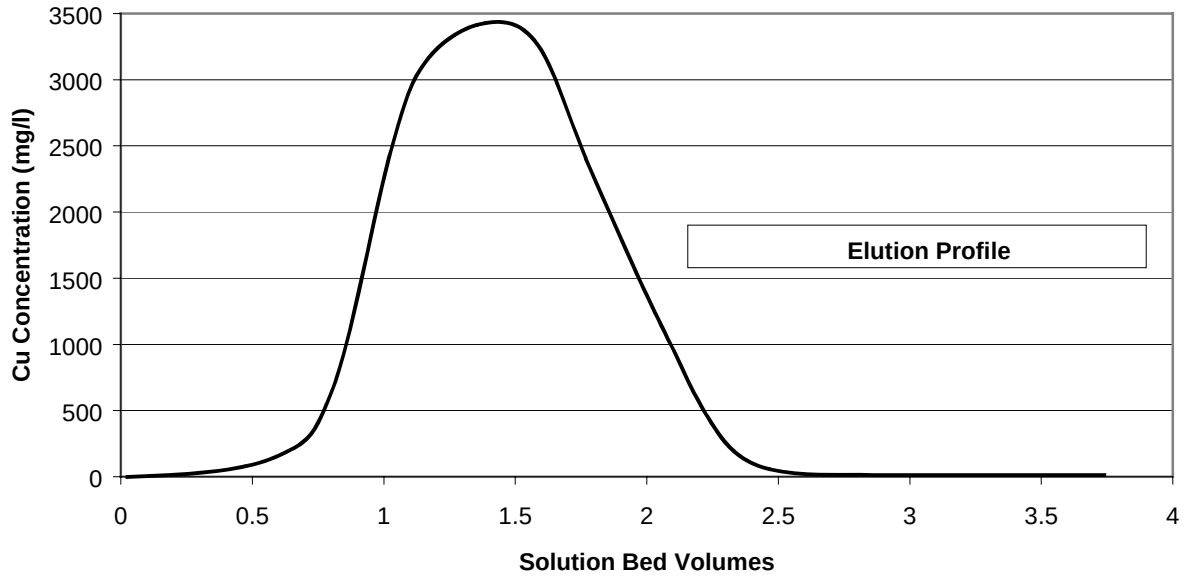


Figure 2: Elution Profile at Room Temperature, 8 M Sulfuric Acid at 0.1 ml/min. Flow Rate



A similar test was then performed to remove the Fe (III) using SuperLig® 14. The loading curve for the Fe (III) is shown in Figure 3. The results confirm that the Fe (III) can be successfully singly removed. There was no sign of Fe breakthrough through 60 solution bed volumes.

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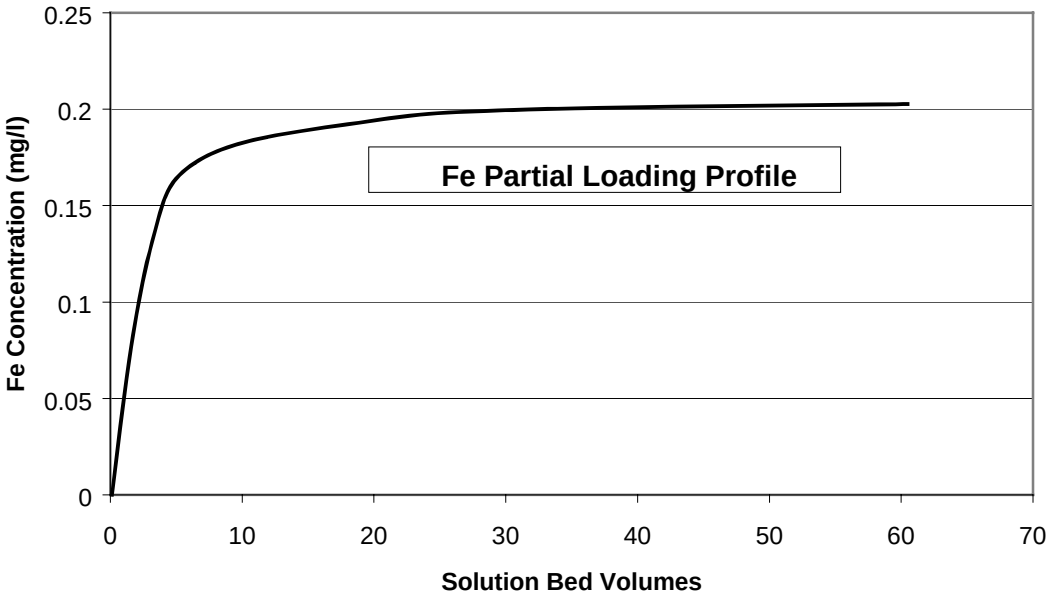
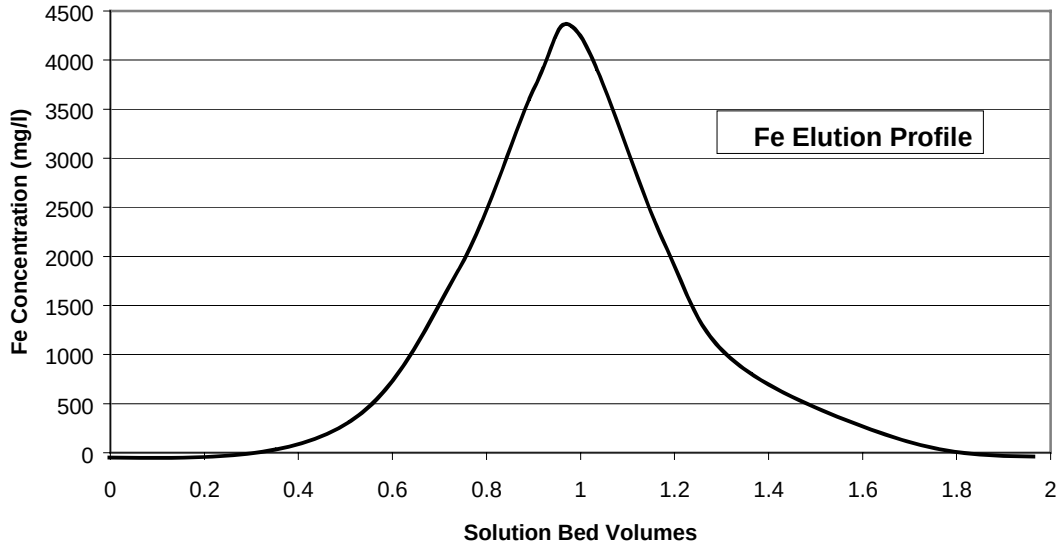
Figure 3: Fe Partial Loading Profile for Feed Solution at pH 1 on a 0.5g SuperLig® 14 Column Using a Flow Rate of 0.3 ml/min.



The elution curve is shown in Figure 4. Two options are available to elute Fe (III) from SuperLig® 14. The first option utilizes a strong HCl solution (30% or higher). This can be used at room temperature and is the option used in the laboratory tests. The second option is a hot 1-2 M H_2SO_4 , 0.5 M SO_2 elution. This option requires a significantly reduced amount of acid and avoids HCl, but it does require temperature and a reducing agent.

Figure 4: Fe Elution Profile, 37% HCl at Room Temperature at 0.1 ml/min. Flow Rate



4.1.2 pH 2 Feed Tests Copper/Iron Removal

This test set involved taking the Cu and Fe (III) selectively together out of solution at ~pH 2. A 0.5g column was used for this test. Hence, the void volume of the column approaches 0.75 ml. A slower flow rate, (0.3 l/min/kg versus 0.6 l/min/kg for the pH 1 tests) and a lesser loading volume per mass of SuperLig® were achievable with this material prior to Fe and Cu breakthrough. This also leads to a less concentrated Cu and Fe in the eluent.

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Again, the second half of the elution should be used for recycle to increase the concentration. This material does reduce the acid requirement as a 2 M H_2SO_4 can be used. Figures 5 and 6 below show the loading and elution curves for the combined Cu and Fe removal.

Figure 5: Partial Loading Profiles for Combined Selective Fe and Cu Binding from pH 2 Solution on a 0.5 g SuperLig® 145 Column at a Flow Rate of 0.15 ml/min.

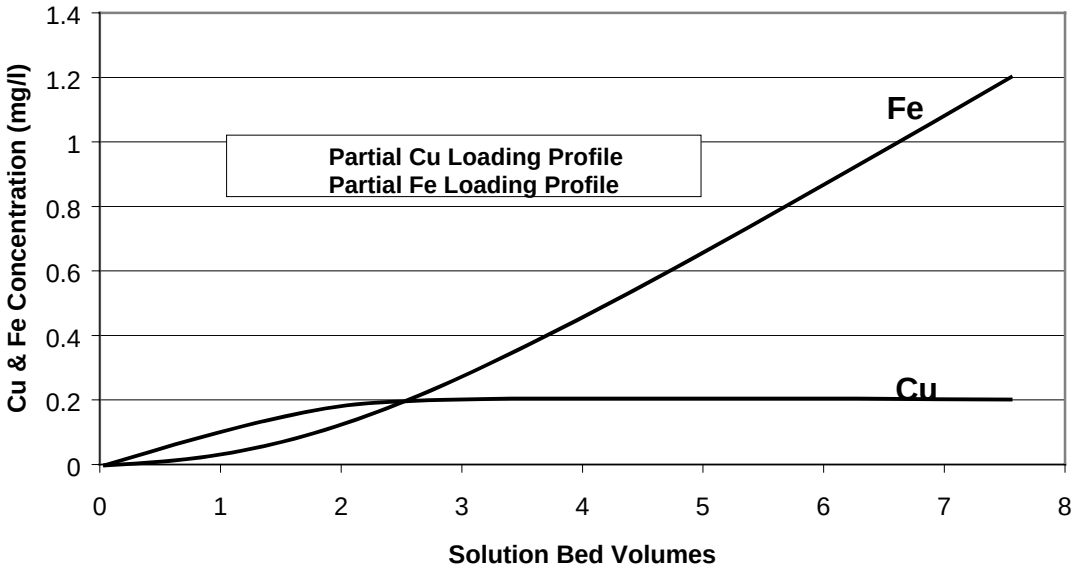
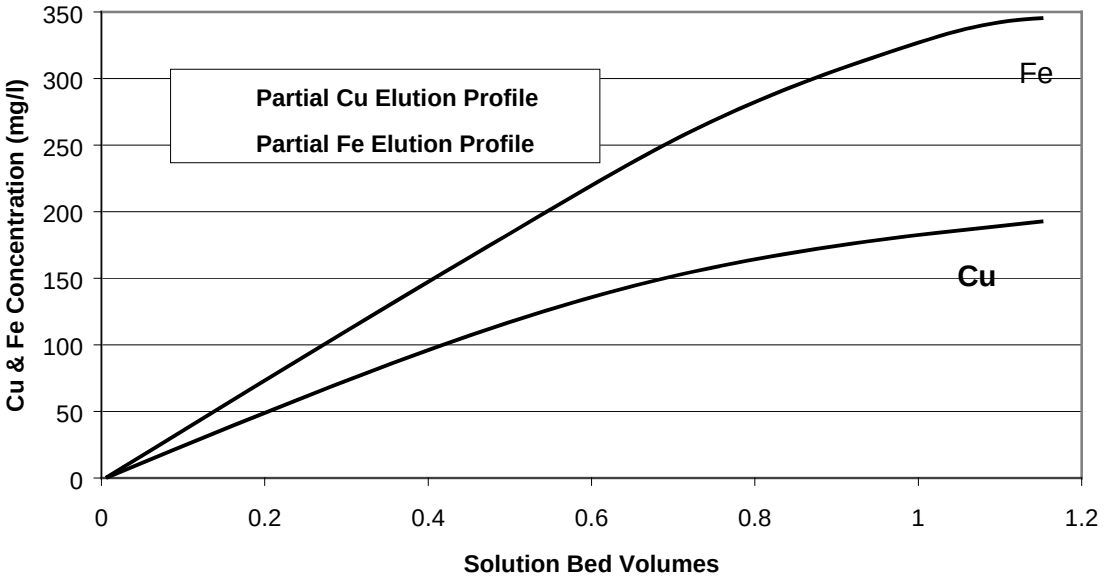


Figure 6: Partial Elution of Cu & FE at Room Temperature, 2 M Sulfuric Acid at 0.4 ml/min. Flow Rate



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4.1.3 Summary

The Cu and Fe can be separated, polished, and concentrated out of the solution at either pH tested, or at varying pH conditions. The best separation test was that conducted with the feed ~ pH 1 due to the higher flow rate and longer loading volume possible. The combined Fe and Cu removal at pH 2 has the advantage of a more dilute H_2SO_4 elution. However, as the elution chemical requirements are rather small in any case, the separate Cu and Fe removal option at pH ~ 1 would seem to be the better option.

4.2 SEPARATION AND EXTRACTION OF NICKEL IMPURITY FROM COBALT BEARING SULFURIC ACID LEACH SOLUTIONS

Table 2 below provides cobalt and nickel concentrations in feed, wash, and eluent solutions for a complete cycle for extraction of nickel from excess cobalt in a 10g/l free H_2SO_4 feed solution containing 8.1 g/l cobalt and 0.71 g/l nickel. The cobalt/nickel ratio in the feed solution is approximately 11.4/1. The SuperLig® material is loaded into three columns operating in series.

Table 2: Example of Data for Ni From Excess Cobalt Separation from a 10g/l Free H_2SO_4 Feed with 8.1 g/l Co and 0.71 g/l Ni

Sample Description[c]	Lead Column		Third Column[a]	
	Co (g/l)[b]	Ni (g/l)[b]	Co (g/l)[b]	Ni (g/l)[b]
Feed	8.1	0.71	8.1	0.71
0-16 Bed Volumes Raffinate	7.6	0.23	6.6	<0.03
0-0.4 Bed Volumes Wash[d]	8.0	0.56	8.1	<0.03
0.4-0.8 Bed Volumes Wash	3.9	0.35	8.1	<0.03
0.8-1.2 Bed Volumes Wash	0.44	0.10	8.1	<0.03
1.2-1.6 Bed Volumes Wash	0.19	0.10	8.1	<0.03
0-0.4 Bed Volumes Elution[e]	0.017	0.091	a	a
0.4-1.2 Bed Volumes Elution	0.019	4.6	a	a
1.2-2.0 Bed Volumes Elution	< 0.004	2.0	a	a
2.0-2.8 Bed Volumes Elution	< 0.004	0.68	a	a

Notes:

a: Three columns in series during loading and washing. Only the lead column is in line during elution.
b: ICP/Flame AA analysis
c: All sample volumes adjusted for void volume offset
d: 0.1 M H_2SO_4
e: 2 M H_2SO_4

Note that the nickel concentration in solution exiting the third or polishing column is reduced to less than 0.03 g/l through 16 Bed Volumes (BV) of feed solution. The cobalt/nickel ratio in the exiting raffinate solution is approximately 270/1. The data indicates that there was a small amount of co-loading of the cobalt with the nickel. The nickel elution in the first column was rapid and sharp. The elution was essentially complete in three BV. The peak nickel concentration measured in the eluent solution was approximately 4.6 g/l. The co-loaded cobalt was eluted along with the nickel.

Figure 7 below provides examples of two Flow Sheet options for removal of copper, iron, and nickel from acidic concentrated cobalt solution.

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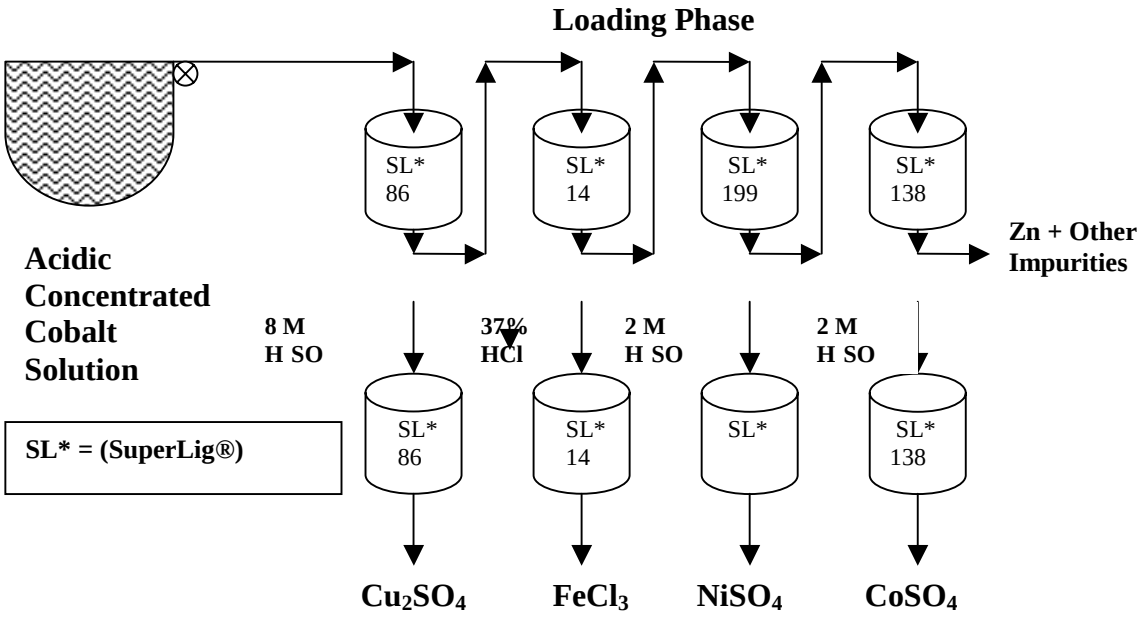
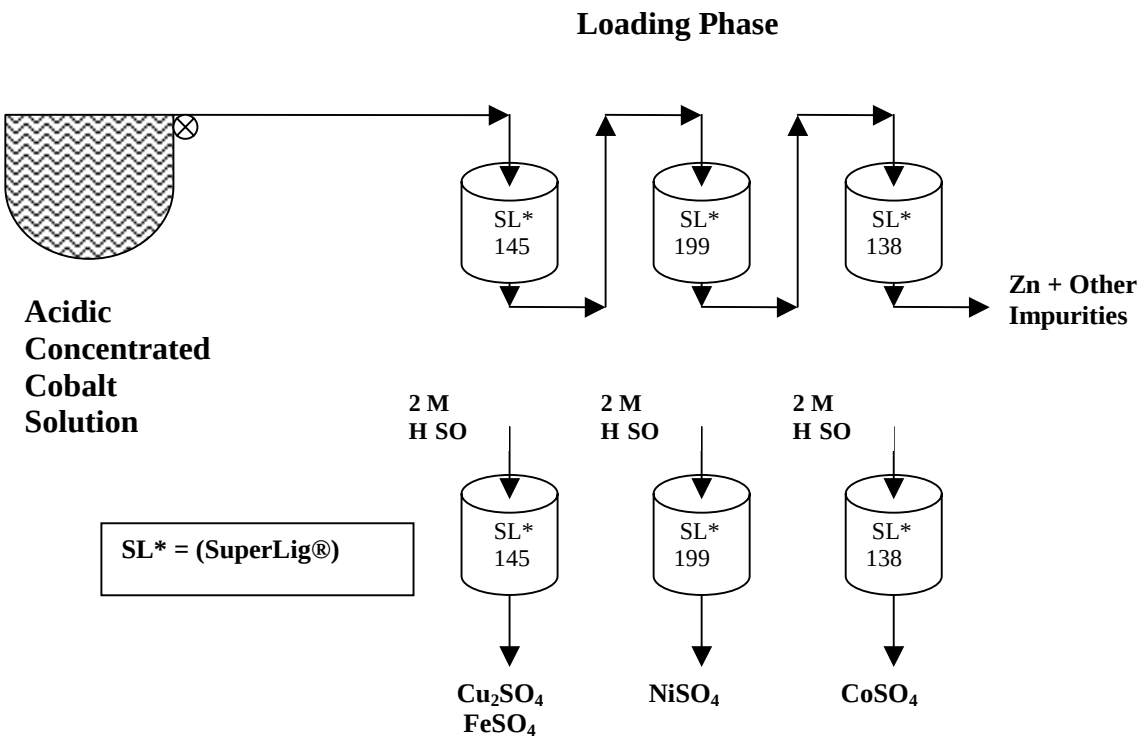
Figure 7: Examples of Basic Flow Sheet Options – Removal of Copper, Iron and Nickel from Acidic Concentrated Cobalt Solution

Option 1: pH ~ 1



Option 2: pH ~ 2



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5.0 SUMMARY

IBC has demonstrated that nickel, copper, and iron can be successfully separated from acidic cobalt solution using MRT. Two process options were presented at pH 1 and at pH 2. In the examples, cobalt is the key metal of value and the objective was to produce a pure cobalt solution for cobalt salt or metal recovery. Depending on the impurity content, it may or may not be necessary to use a separate cobalt separation if the deleterious impurities have been removed to satisfactory levels. If a highly pure cobalt solution is required, the cobalt specific SuperLig® material can, of course, be used. IBC has SuperLig® materials and systems available for removal of various other impurities such as Zn, Ca, Pb, As, Bi, from similar sulfuric and nitric acid matrices.

The low capital and operating costs applicable to the MRT SuperLig® systems make it feasible for companies to acquire cobalt bearing feed materials, with high impurity levels, at considerable market discounts, and process these materials economically. One key feature of the MRT system is the flexibility and ability to process a wide range of feed materials, at various impurity concentration levels, with the same equipment. The technology also provides the opportunity to convert the impurities to high purity salt products with a significant added value. The MRT systems also offer the flexibility to readily increase capacity at a low cost at any time, and to readily add processing capacity to an existing flow sheet to deal with major changes in feed and impurity specifications.

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NEW DIAPHRAGM MEDIA FOR NICKEL ELECTROWINNING PROCESSES

By

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Tamfelt Corp,

Presented by

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ABSTRACT

In the nickel electrowinning processes, nickel is precipitated from a nickel sulfate solution into metallic form by electric current. The desired cathodic reaction is the reduction of nickel ions on the cathode plate and the main anodic reaction is the oxidation of water on the surface of the lead anode.

Due to the presence of the secondary hydrogen reaction on the cathode, it is necessary to keep the concentration of hydrogen ions in the cathode compartment as low as possible. The hydrogen bubbles that form due to the secondary reaction adhere to the surface of the deposit, especially if the current efficiency is high. This inhibits the deposition of metal at that point and, as the metal continues to deposit around it, causes a pit at the location of the bubble.

To minimize the hydrogen evolution, the pH of the solution should be sufficiently high to have a good current efficiency for nickel electrodeposition. At pH values 2.5 and higher, hydrogen evolution is low and the main reaction is deposition of nickel.

Industrial scale nickel electrowinning operations around the world use tightly woven diaphragm fabrics to enhance the quality and quantity of nickel production. The diaphragm cloth resists the electrolyte flow from cathode compartment to anode compartment and by doing so forces the electrolyte to accumulate into the cathode compartment until the liquid level is high enough to overcome the fabric flow resistance. The flow resistance of a woven fabric is proportional to the fabric thickness, tortuosity and pore structure (i.e. the size, shape and number of pores). Hydrostatic pressure effectively works against the back-diffusion of hydrogen ions and other impurities back to the cathode compartment.

By the same token the fabric forms an extra resistance for electrical current from anode to cathode. The less permeable and more solid the fabric the higher the specific resistance which in turn increases the power consumption for nickel production. From the electrical resistance point of view the desired diaphragm should have as open and thin structure as possible to allow for lowest possible resistance to the electricity.

Consequently, a diaphragm fabric should at the same time have low permeability to allow for high hydrostatic head build-up and sufficienctly open structure to allow for low specific resistance. These two requirements are contradictory and difficult to combine in one single fabric design.

This work studies the correlation between diaphragm fabric permeability, hydrostatic head, net ionic transport and nickel production efficiency in an experimental cell equipped with a new media invention which combines low media permeability and low electrical resistance. The results are compared with a conventional PET multifilament woven diaphragm fabric performance. The results confirm that the new media makes it possible to run a nickel electrowinning process at high current efficiency, high hydrostatic head and lower power consumption.

1. INTRODUCTION

In the electrowinning process, the desired cathodic reaction is the reduction of nickel ions on the cathode plate and the anodic reaction is the oxidation of water on the surface of the lead anode. Simplified cell reactions can be described by the following overall reactions.

Cathode reaction:	$Ni^{2+} + 2e^- = Ni$	E^o=-0.23 V
	$2H^+ + 2e^- \rightarrow H_2$	E^o=0.00 V
Anode reaction:	$H_2O = 2H^+ + ½\ O_2 + 2e^-$	E^o=1.229 V

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Cell reaction: $NiSO_4 + H_2O = Ni + H_2SO_4 + 1/2O_2$

The production of hydrogen gas occurs because nickel is less noble than the hydrogen ion, as can be seen from the standard reduction potential of nickel. Due to the presence of the secondary hydrogen reaction, it is necessary to keep the concentration of hydrogen ions in the catholyte as low as possible. The hydrogen bubbles that form due to the secondary cathode reaction adhere to the surface of the deposit, especially if the current efficiency is high. This inhibits the deposition of metal at that point and, as the metal continues to deposit around it, causes a pit at the location of the bubble.

To minimise the hydrogen evolution, the pH of the solution should be sufficiently high to have a good current efficiency for nickel electrodeposition. For a typical industrial scale nickel electrowinning operation the curves show that the higher the hydrogen ion concentration, the higher the reduction rate of hydrogen within the potential values between –600 and –650 mV vs. SCE. The reduction peak demonstrates the activity of hydrogen ions. At pH values 2.5 and higher, hydrogen evolution is low and the main reaction is deposition of nickel. The current efficiency studies at different pH values confirm this phenomenon. At pH 3.0 the current efficiency for nickel is 97 % and 94 % at pH2.5, when the current density is 200 A/m^2. At pH 2.0 or less the current efficiency decreases drastically (Knuutila, 1997).

Industrial scale nickel electrowinning operations around the world use tightly woven diaphragm fabrics to enhance the quality and quantity of nickel production. The diaphragm cloth resists the electrolyte flow from cathode compartment to anode compartment and by doing so forces the electrolyte to accumulate into the cathode compartment until the liquid level. i.e. hydrostatic head, is high enough to overcome the fabric flow resistance. The formation of the hydrostatic head effectively prevents the diffusion of hydrogen ions and other impurities back to the cathode compartment.

The fabric also forms an extra resistance to the flow of electricity in the cell. Diaphragm electrical resistance is a function of the fabric pore structure and yarn material. The less porous the fabric is the higher its specific resistance. High resistance increases the power consumption for nickel production. As a result, an optimal diaphragm fabric should at the same time have a low permeability and a very open structure.

2. FLUX OF IONS THROUGH A DIAPHRAGM FABRIC

Ions in electrolyte can move between the catholyte and anolyte by means of diffusion, migration and convection. Migration is the movement of ions under the electric field generated between the electrodes. Under this force, the positive cations are attracted to the negative cathode and the anions to the anode. Migration is generally negligible if excess inert salt is present in the solution. Diffusion is the movement of any species due to a concentration gradient and convection is the movement of any species due to the bulk flow of solution. The higher is the feed flow and flow through the fabric the higher is the convective mass transfer. Similarly the higher the concentration difference between the anode and cathode compartment the higher the diffusive mass transfer other things being the same. Without a diaphragm fabric the back-diffusion off acid is significant and the optimal reduction pH in cathode is distorted.

Mass transfer to an electrode is governed by the Nernst-Planck equation. As shown by Rodrigues (Lenthall et al., 1997), equation (1) could be used to model the net flux of species i through porous diaphragm due to migration, diffusion and convection.

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$$j_i = v_d c_i(x) - \frac{IFD_i z_i c_i(x)}{RT\,\kappa} - D_i A_{diap} \frac{dc_i(x)}{dx} \qquad (1)$$

Which, when integrated between the boundary conditions $c_i(0)=c_c$ (catholyte concentration) and $c_i(d)=c_a$ (anolyte concentration), yields the following equation for the flux of species i through the diaphragm

$$j_i = -\alpha_i D_i \frac{\left(c_{i,a} - c_{i,c} \exp\left(\frac{-\alpha_i d}{A_{diap}\,\varepsilon} \right) \right)}{1 - \exp\left(\frac{-\alpha_i d}{A_{diap}\,\varepsilon} \right)} \qquad (2)$$

where

$$\alpha_i = \frac{IFz_i}{RT\,\kappa} - \frac{v_d}{D_i}$$

In order to be able to use equation (1) for diaphragm mass transfer simulations one would have to have accurate values for diffusivity D_i of each ionic species in the system and the average conductivity of the solution κ in the diaphragm. As shown by Awakura et al. (Awakura et al, 1988) the diffusivity of $NiSO_4$-$H2SO_4$ solution depends on the concentration of both the acid and the salt. Similarly the conductivity of the solution is dependant on the concentration of the solution in the diaphragm.

Since the purpose of this paper is to test how two different diaphragm fabrics effect the performance of a nickel electrowinning test cell performance it was concluded sufficient to note the above equation as a basic model explaining the three simultaneous physical transport phenomena affecting the flux of ions through the diaphragm fabric.

3. LABORATORY SCALE TESTING OF THE DIAPHRAGM FABRICS

3.1 TESTED DIAPHRAGM FABRICS

To test the effectiveness of a new innovative polyaniline based coating on the diaphragm fabric two types of fabrics, namely A and B, were tested for three different levels of hydrostatic head. In an industrial scale nickel electrowinning operations there is a continuous feed of nickel sulphate solution to the cathode compartment and pH control to keep the process condition optimal for nickel reduction. In the experimental cell it was found out that in order to be able to compare the performance of two fabrics with different voidage and porous structure it is necessary to test the fabrics for several different levels of hydrostatic head to find out the hydrostatic head level which ensures that no significant back-diffusion from anolyte to catholyte is taking place.

Both tested fabrics had the same multifilament basic weave and yarn structure. Fabric B was the reference diaphragm with standard PET multifilament yarns and 581 grams/sqm weight. The measured air permeability of B was 0,27 qm/sqm/min (at 200 Pa). The polyaniline coated fabric A had a slightly higher weight, 585 grams/sqm, and little lower air permability 0,23 qm/sqm/min. Both fabrics were 0.71 mm thick and the voidage of fabric A was 0,41 and fabric B 0,42. Fabric A electrical resistance was 40 % less than with fabric B due to a special patented polyaniline based coating.

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3.2 TESTING PROCEDURE

The diaphragms were soaked in catholyte for 24 hours before each experiment to ensure thorough wetting of the fibres and the structure. A test run was started out by filling identical electrolyte to the anode and the cathode compartments. Hydrostatic head was maintained by overflowing the anolyte and catholyte to the respective tanks. Both the anode and cathode circulations were equipped with temperature controls to make sure that the testing conditions were isothermal. Electrolytes were pumped back to the respective compartments at a constant rate of 35 ml/min to ensure effective mixing and even ionic concentration.

Before each experiment the electrolyte Ni- (AAS), Sulphate (AAS) and sulfuric acid (titration) concentrations were analysed. The anolyte and catholyte circulation pumps and the heating circuits were started. After the electrolyte circulations were stabilised (roughly after few minutes) the electrical current was switched on to 2.05 A and the potentiometric measurements were started.

After 24 hours of operation the total anolyte overflow and anolyte and catholyte volumes were measured and 50 ml samples were taken out of each solution. For each sample the Nickel, Sulphuric acid and Sulphate concentrations were analysed. The amount of reduced nickel was determined gravimetrically and the total power consumption was recorded.

3.3 EXPERIMENTAL APPARATUS

To study the flux of ions in a laboratory scale a 120 x 190 x 200 mm polycarbonate cathode-anode cell was equipped with necessary instrumentation and a diaphragm fabric specimen as show in figure 1. The cell had 70 x 70 mm lead anode and 70 x 70 mm stainless steel cathode blanketed from the back side. The anode and cathode compartments were connected via a 70 x 70 mm (0,0049 m2) aperture for the fabric specimen. At the beginning of each experiment the electrolyte volume was 4500 ml out of which 362 ml was in the anode side and 4038 ml was in the cathode circulation. The balance was in the capillaries of the potentiometric equipment.

Both the anolyte and catholyte had independent circulation tanks out of which the overflow anolyte and catholyte were circulated back to the cell at the flow rate of 35 ml/min. In each experiment a desired hydrostatic head was maintained by opening the respective anode and cathode compartment overflow channels. Both the anolyte and catholyte circulations had also separate heating elements to maintain the electrolyte temperatures constant at 50 ^{o}C.

The electrolyte used in the experiment was a commercial grade nickel electrowinning process electrolyte with initial pH 2.8 and initial nickel concentration of 136 g/l. There was practically no sulfuric acid present in the initial electrolyte and only minor concentrations of other components.

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Fig 1. Experimental apparatus.

3.4 TEST DATA

The purpose of this work was to compare two different diaphragm fabrics in terms of nickel electrowinning process performance. The test cell operation efficiency was measured in terms of potential drop across the diaphragms, the cell voltage, bulk flow through diaphragm, anolyte and catholyte ionic concentrations before and after the test runs, and gravimetric nickel production for three (3) different hydrostatic heads. In addition the anolyte and catholyte pH and conductivities were measured before and after each test run.

Measured average electrolyte flow through the diaphragm, average cell voltage, average potential drop across the diaphragm and total amount of reduced nickel for each test are presented in table 1.

Table 1. Average flow through the diaphragm, cell voltage, potential drop across the diaphragm, electrolytic nickel production and calculated current efficiencies for the test runs.

	Diaphragm A			Diaphragm B		
Hydrostatic Head, cm	3	5	10	3	5	10
Flow through diaphragm, ml/hr	3,96	16,17	42,46	21,79	39,83	87,92
Cell voltage, mV	4956	5173	5311	5321	5724	5945
Diaphragm potential drop, mV	602 (12 %)	658 (12,7 %)	762 (14,3 %)	785 (14,8 %)	1246 (21,8 %)	1300 (21,9 %)
Produced nickel, grams	49,12	49,81	52,18	51,46	52,82	53,04

Current efficiency and permeability from table 1 are plotted against the hydrostatic head in graphs 1 and 2.

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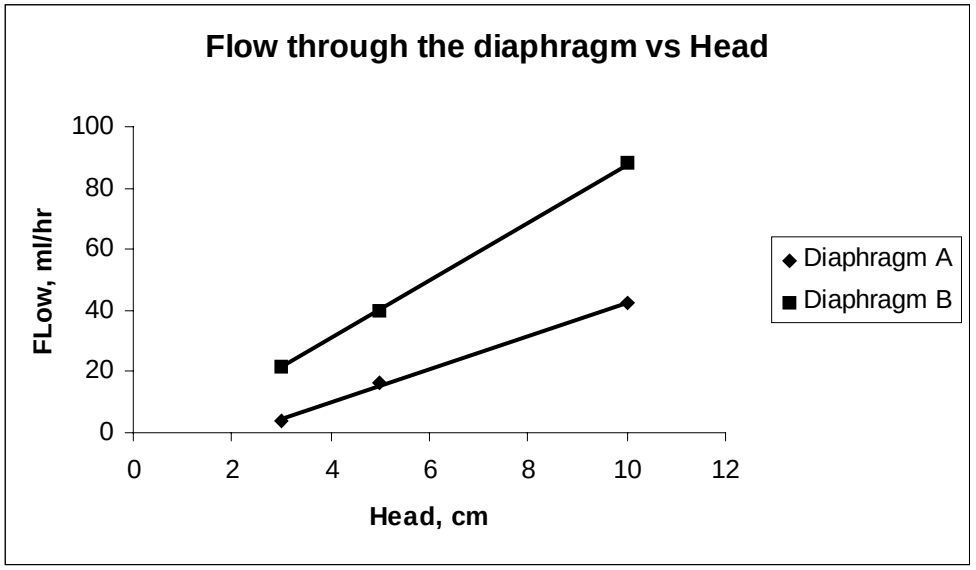
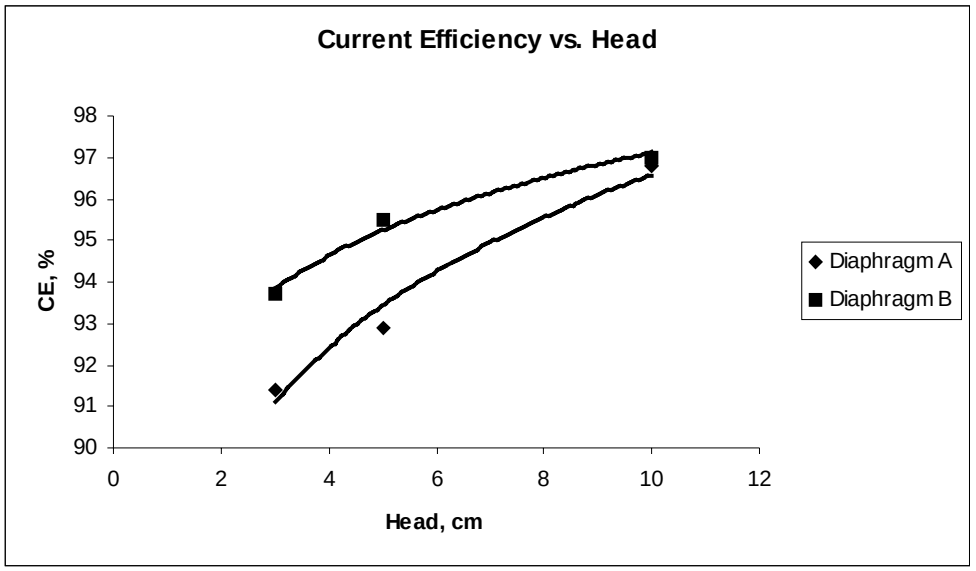


Graph 1. Flow through the diaphragm vs. three different levels of hydrostatic head for diaphragm fabrics A and B.



Graph 2. Current efficiency vs. three different levels of hydrostatic head for diaphragm fabrics A and B.

Analyzed ionic concentrations before and after each test run allow calculation of the total ionic fluxes through the diaphragm within the 24 hour run. These fluxes are presented in graphs 3 through 5.

As can be seen in graph 1 having a high hydrostatic head means a high flow through the diaphragm. For the more open reference fabric B the flow was significantly higher resulting also in higher flux of sulphate and nickel through the fabric as can be seen in graphs 3 and 4. As can be seen in graph 5 the hydrogen flux was also clearly higher than with the polyaniline coated fabric A with less voidage.

As explained for example by Sermyagina et al (Sermyagina et al. 1990) there exists a critical level of hydrostatic head for a given diaphragm, which prevents the influx of hydrogen to the cathode compartment. In fact the only case in which the hydrogen ions where not flowing from the anode to the cathode for the fabric A was the case of 10 cm

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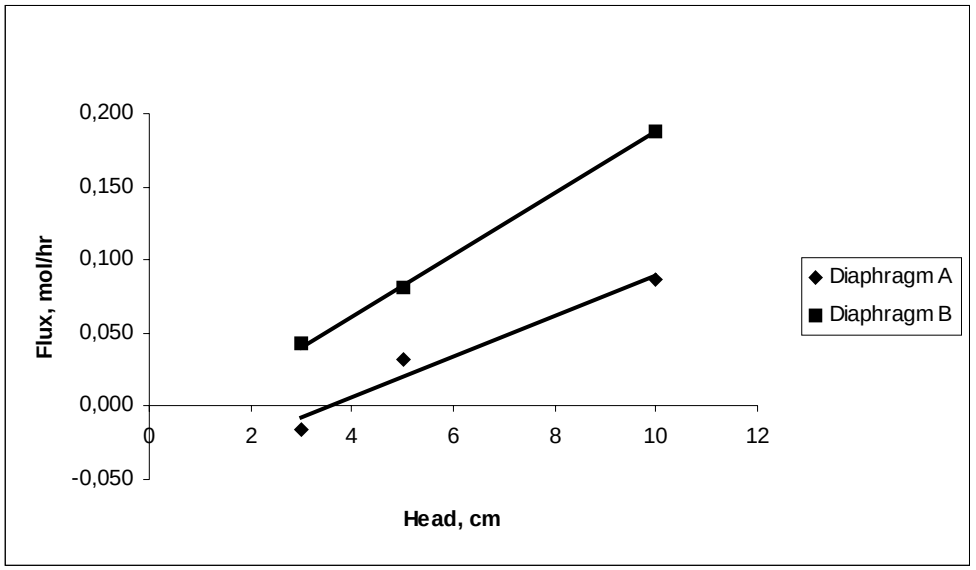
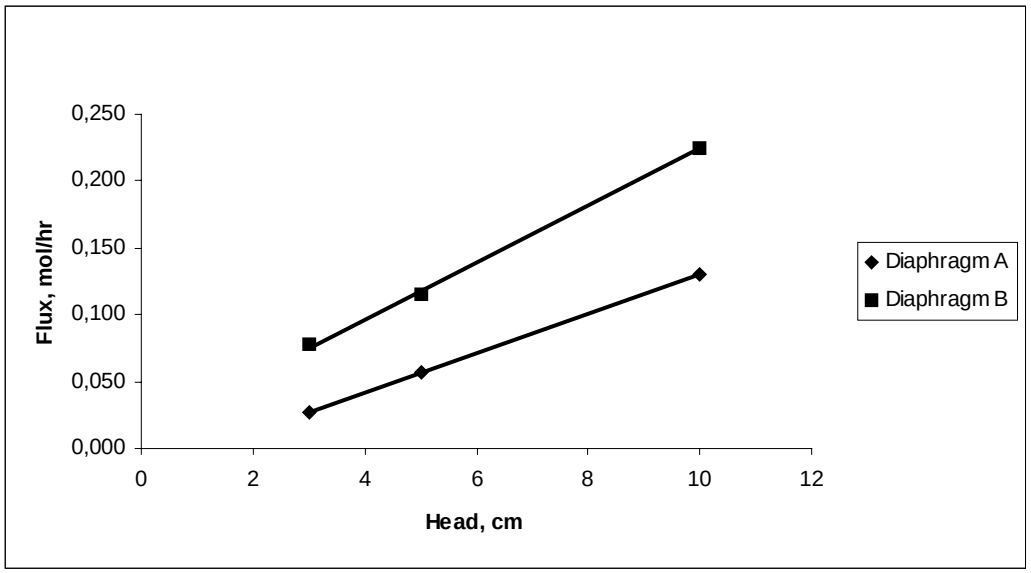
head. Consequently, the performance comparison between the reference fabric B and the new fabric A performance was based on the diaphragm fabric A 10 cm head case and the respective measured flow through the fabric i.e. 42.46 ml/hr.



Graph 3. Nickel flux through the diaphragm vs. three different levels of hydrostatic head for diaphragm fabrics A and B.



Graph 4. Sulphate flux through the diaphragm vs. three different levels of hydrostatic head for diaphragm fabrics A and B.

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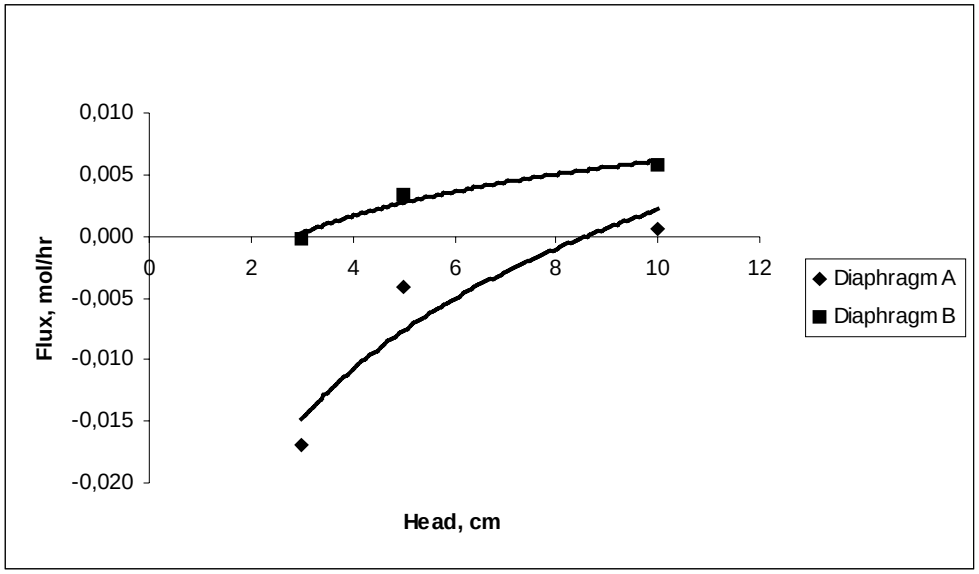
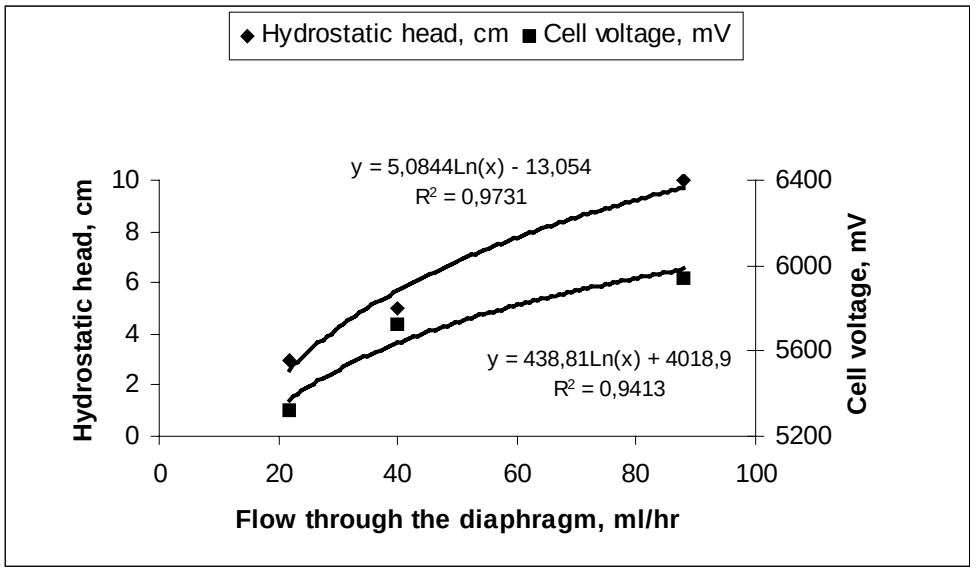


Graph 5. Hydrogen flux through the diaphragm fabric vs. three different levels of hydrostatic head for diaphragm fabrics A and B.

Process performance values for the reference fabric B for the operating point with 42.46 ml/hr flow through the diaphragm fabric were then estimated by fitting the table 1 performance results against the measured flow rates and extrapolating the 42.46 ml/hr flow rate performance values. Graphical presentation of the main performance characteristics for the reference fabric B together with the fitting results are presented in graphs 6 and 7.



Graph 6. Reference fabric B hydrostatic head and cell voltage vs. flow through the fabric and the related fit curves.

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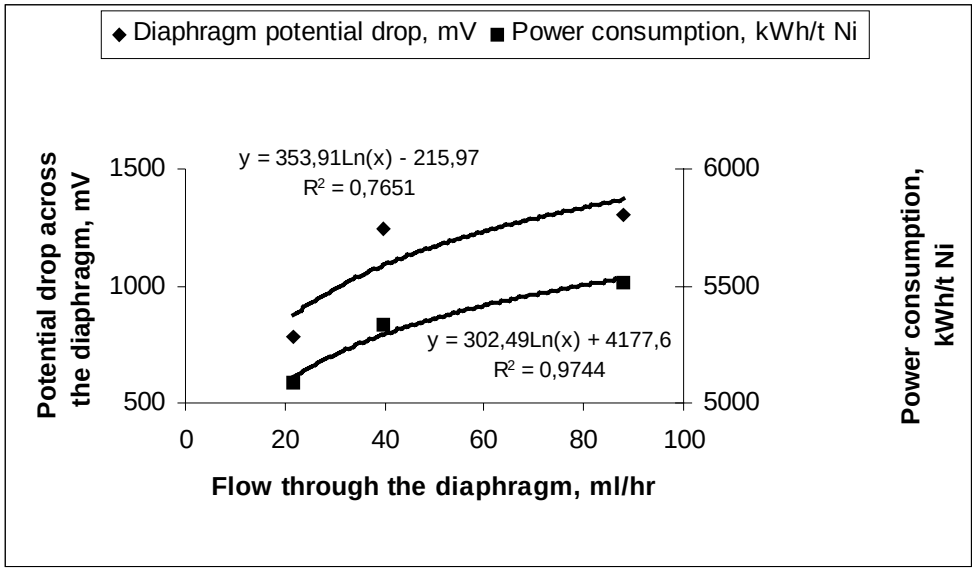

Graph 7. Reference fabric B nickel production and current efficiency vs. flow through the fabric and the related fit curves.

Comparison of the two diaphragm fabrics performance at the selected operating point is presented in table 2.

Table 2. Average flow through the diaphragm, cell voltage, potential drop across the diaphragm, electrolytic nickel production and calculated current efficiencies for the test runs.

	New diaphragm A	Reference diaphragm B
Flow through diaphragm, ml/hr	42.46	42.46
Hydrostatic Head, cm	10	6
Cell voltage, mV	5311	5664
Diaphragm potential drop, mV	762 (14,3 %)	1111 (19,6 %)
Power consumption, kWh/t Ni	5008	5312

As can be seen in table 2 the new polyaniline coated diaphragm fabric A performance is better than with the reference fabric B in terms of power consumption per ton of nickel produced. This is because both the cell voltage and potential drop across the diaphragm are less than with the more permeable reference fabric B. As a conclusion of this test work it can be concluded that the fabric A consumes less energy for the nickel production than the conventional fabric B. The absolute amount of nickel produced is almost the same for both fabrics and the only difference is the higher hydrostatic head with fabric A.

4. DISCUSSION AND CONCLUSIONS

One single laboratory scale nickel electrowinning cell can not accurately predict the behavior of a full-scale tankhouse with hundreds of cells and cathode bags. Flow dynamics, electrolyte preparation, bag construction etc.. are all different in a full-scale plant compared to laboratory scale equipment not to mention the fact that the space for hydrostatic head build-up is always strictly limited.

The value of the results obtained from this laboratory scale nickel electrowinning cell test is in making it possible to compare the two different diaphragm fabrics in strictly controlled process environment. More specifically, this work allows to study experimentally how a diaphragm fabric surface material modification changes the performance characteristics of an electrowinning process in which the fabric is used.

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Normally a nickel electrowinning plant is operated by having the electrolyte feed flow to one cell as constant as possible at around 10-30 liters/hr. The diaphragm cloth resists the electrolyte flow from cathode compartment to anode compartment and by doing so forces the electrolyte to accumulate into the cathode compartment until the liquid level difference between cathode and anode compartments (hydrostatic head or head) is high enough to overcome the fabric flow resistance. Naturally the lower the diaphragm fabric electrolyte permeability is the higher the hydrostatic head will be.

This work confirms that electrowinning cell electrolyte flow from cathode compartment to anode compartment, and consequently the diaphragm fabric electrolyte permeability, is one of the most important control parameters for the electrowinning process. The higher the flow is the less is the hydrogen flow from anode to cathode which in turn means that the optimal conditions for nickel reduction are maintained.

The aim of this study was to compare a new diaphragm fabric innovation with a conventional woven diaphragm fabric in terms of nickel electrowinning process performance. Both fabrics were first woven using same PET multifilament yarns and same weave. The new fabric was then coated with an intrinsically conductive polyaniline polymer to reduce the fabric electrical resistance and also the electrolyte permeability roughly 40 %.

The measured cell voltage and potential drop across the diaphragm fabric data confirm that the new invention reduces the total cell voltage and potential drop across the diaphragm fabric by 6 % and 31 % respectively. Provided that the hydrostatic head for the new fabric A can be high enough to allow for sufficient electrolyte flow from cathode compartment to anode compartment the absolute amount of nickel production is not affected by the polyaniline coating. This means that it is potentially possible to produce the same amount of nickel with less power by using the polyaniline coated PET diaphragm fabric.

These results are for one single experimental nickel electrowinning cell and they have to be confirmed for full scale production units before any final conclusions can be drawn.

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PROCESS IMPROVEMENTS AT BULONG OPERATIONS PTY LTD

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ABSTRACT

Bulong remains the only hydrometallurgical process plant in the world that recovers nickel and cobalt directly from High Pressure Acid Leach (HPAL) laterite leach liquors without the need for precipitation of an intermediate product (e.g. sulphide, hydroxide or carbonate). Direct Solvent Extraction (DSX) of both nickel and cobalt in combination with electrowinning of nickel has been in operation at Bulong since early 1999.

Since commissioning, a number of significant process problems have been resolved, the most difficult being the formation of gypsum in the nickel SX circuit.

The metallurgical focus in the first years of operation was on ramp-up and resolving numerous commissioning issues. More recently the focus has been on plant de-bottlenecking and process enhancement.

In the two years since ALTA 2001, the performance of the process plant has significantly improved. All parts of the plant now consistently operate well above design capacity (when on-line) and has on several occasions reached the tankhouse metal winning limit (at approximately 11,000 tpa Ni). Overall metal recovery and nickel cathode quality has also improved.

1 INTRODUCTION

Bulong Operations Pty Ltd (BOPL) commenced mining operations on 3 April 1998. First ore was fed to the Ore Preparation plant on 12 November 1998, with “acid on” on 21 December 1998. Tankhouse current was first applied on 28 February 1999 with a starter sheet harvest on 2 March 1999. First commercial metal production was in April 1999. The background to the project is well described by Nofal et al [(1)] and other authors [(2) (3)]. A flowsheet showing the overall Bulong process is attached.

Several occurrences of major plant equipment failure and the “discovery” of gypsum formation in the Ni SX circuits have resulted in a lengthy ramp-up to design capacity and metal production. These commissioning problems have been previously documented by Nofal et al [(1)] and others [(2) (3)]. The gypsum problem is also well described by Nofal.

In the last six months, BOPL has consistently achieved greater than design plant capacity in both HPAL and Refinery circuits. However, long-term metal production continues to be restricted by poor equipment availability and unexpected lost time due to the increased frequency and duration of shutdowns. However, improved condition monitoring and the adoption of other modern maintenance practices is addressing the problem of poor equipment availability. The combination of increased plant capacity and higher availability should see improved metal production in the future.

This paper is focused on recent process and process equipment modifications which have resulted in substantial improvements in capacity, recovery and product quality.

2 MINING

Mining commenced on 3 April 1998 and mining practices are largely unchanged from those described by Oram [(4)].

Bulong ore is blended in the following lithological proportions:

60 – 70 % nontronite
20 – 30 % serpentinite
10% limonite

Five pits have been completed and three are currently in operation.

A total of 5.54 Mt of ore (4.2 MBCM) and 5.31 MBCM of waste has been mined since the project commenced.

2.1 Grade Reconciliation

The reconciliation between run-of-mine (ROM) grades predicted by grade control and that back-calculated from HPAL feed grade was generally poor. Grade control sampling is now conducted using drill rigs instead of trenching by "ditchwitch".

The back-calculated ROM grade uses HPAL feed grade and data from several reject streams from the Ore Preparation Plant. Two of these reject streams contain rocks with >30 mm particle size. The sample size for these streams is probably too small and is currently being re-assessed. Bulong has benefited from a significant upgrade in leach feed grade (not predicted by the feasibility study), however, the nature and number of the rejected streams makes metal reconciliation between mine and plant difficult. It is not clear if this is a unique Bulong issue. However, future laterite projects

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treating ores with a significant mass rejection of coarse rock should be aware of this problem.

Assaying for Ni, Co, Mg and other elements continues to be conducted by digest - ICP, however, Bulong is planning to use pressed powder XRF for all grade control and exploration samples in the near future.

2.2 Pre-blending of Ore

A major change in stockpiling and ore feeding practices from that reported by Oram [4] is that Bulong now pre-blends ores. Pre-blending commenced on 10 July 2002 with a 20,000 tonne parcel of ore and continues to this day.

The primary objectives for pre-blending are the stabilisation of HPAL performance in terms of:

1. Maintaining consistent leach discharge free-acid (FA). Since there is a strong relationship between leach free acid and ferric concentration, stabilising acid addition and FA levels also stabilises the ferric levels in solution enabling the downstream iron removal circuit to function smoothly. Erratic FA and ferric iron causes erratic performance in subsequent neutralisation and iron removal circuits.

2. Maintaining consistent ferrous levels. There appears to be a broad relationship between free acid and ferrous levels in leach discharge, which is thought to be due to the amount of serpentine type minerals (ferrous containing) being leached. Hence maintaining consistent free acid indirectly helps stabilise the subsequent downstream ferrous oxidation process. Blending of serpentine minerals should also assist in smoothing out the input of ferrous.

3. Maintaining consistent leach feed grade. This allows better control of the nickel metal input into the process. Bulong has a limited ability to store nickel in inventory, hence consistent mass flow of nickel into the plant should improve overall plant control.

The effects of pre-blending are discussed in the Ore Preparation and HPAL sections below. It is clear that pre-blending does give some benefit in terms of ferrous levels in leach discharge. However, for the other parameters like Ni feed grade and leach discharge FA, variation due to inherent process instability within the leach circuit itself appears to be greater than geo-chemical and/or mineralogical variability in the ore.

3 ORE PREPARATION (OP) PLANT

3.1 General

The Bulong orebody is being mined “pit by pit”, hence ore delivered to the plant displays considerable lithological variability. Lithological variability within pits is also significant. Other variability in terms of geochemistry, hardness of ore, fraction of “rocks” and fraction of “sand” also exists.

The OP circuit was originally designed for a “constant” mass rejection of 5%. However, due to natural ore variability, this can vary between 5% and 45%. Rejection is on the basis of particle size. Coarse, harder rocks are rejected which are generally lower in grade than ore in finer particle sizes. This rejection naturally leads to an upgrade in HPAL feed grade. The ore is beneficiated.

Bulong continues to exploit the natural beneficiation effect displayed by most ore types, and a considerable number of process changes have been made since commissioning to further improve leach feed grade without rejecting excessive amounts of Ni and Co.

While there is some nickel and cobalt lost during beneficiation, the upgrade in HPAL feed grade is far more important from a cash-flow and metal production perspective.

Leach feed grade continues to dominate the economics of nickel production at Bulong, however, operation with lower leach feed grades will become viable as plant performance continues to improve and costs are further reduced.

3.2 Recent Changes to Process and Process Equipment

Recent changes to process configuration, equipment or operating practices within the Ore Preparation plant are described below.

3.2.1 Ore Crushing

The original “toothed” rolls MMD sizer has been replaced by a larger unit and modified to generate a top size of 80mm. The new unit has the facility to reject oversize rock if required. Previous crusher product top-size was >100mm. Reduced wear and hence reduced maintenance costs and improved availability have been noticed in the downstream OP equipment.

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Bulong is planning to further reduce the product top-size to 50 mm.

3.2.2 Ball Mill

The ball mill has the capability of being operated as a fully autogenous scrubber (FAS) (no ball charge at all) or as a semi-autogenous scrubber (SAS) (some ball charge) depending on the blend being fed to the plant. Until very recently the mill has been operated in the FAS mode, however, changes in the ore blend have necessitated operation in SAS mode allowing some grinding of ore to maintain plant throughput. Based on ore blends of the last two years and general operating experience a mass reject rate of approximately 25% appears optimum in terms of nickel upgrade factor and equipment capability. It is clear that while some "grinding" does occur the primary function of the mill remains scrubbing (of rocks) and attritioning (of clay lumps). A large fraction of the 40-60 mm mill feed is unbroken and leaves the mill as ball mill scats.

The opening size of the mill feed chute was increased in September 2002 to accommodate larger rocks and clay balls from the primary attritioning device – logwasher. In conjunction with this change a new "Sanki" conveyor was installed to accept the increased tonnage of ball mill scats.

Recently a process water spray bar has been installed to wash the ball mill scats leaving the mill. An incremental increase in recovery is expected.

3.2.3 Sizing Screen

The sizing screen is a twin deck vibrating device.

The top deck of the sizing screen has been changed to 2mm (from 3mm) and oversize from this screen is now being rejected as "sands" scats.

The bottom deck has been recently changed to 1.6 mm aperture (cf 600 micron design) to improve the density of the autoclave feed and increase Ni and Co recovery. At this stage no substantial reduction in leach Ni or Co extraction has been detected and minimal "sanding" of the heater circuit has been observed.

3.2.4 Pre-blending of Ore

Pre-blending of ore appears to have assisted in OP performance. Mineral sizer and logwasher wear rates are more consistent and predictable. "Scats" reject rates are more consistent and in general the OP operator has to make less adjustments. Pre-blending also allows less frequent ball mill power adjustments.

3.2.5 Thickening of Ore

The ultimate leach feed slurry density achieved in the thickener is essentially a function of ore slurry viscosity and hence is largely ore blend related.

However there are a number of mechanical/operational issues that affect the actual density achieved including:-

1. Limitations with underflow (U/F) pump power. The pumps trip at high viscosity and density.
2. Undersized thickener O/F piping. Restricts water removal from slurry.
3. Overfloccing of ore and generally poor flocculant control.
4. Frequent startup and shutdown of the OP circuit generates lower thickener densities.

Historically OP operators have targeted a leach feed slurry density irrespective of actual slurry viscosity.

To assist operating personnel maximise density, "slump ring" readings are now taken to measure slurry "yield stress". On-line calculation of apparent viscosity using pipeline pressure drop is also being implemented. To maximise leach feed slurry density Bulong is moving towards rheological control of thickening and pumping of leach feed slurries.

An interesting feature of flocculated Bulong ores is that they are strongly shear-thinning. Since slurry pipework size is normally fixed, operation at low flow (low shear rate) increases the apparent viscosity in pipework and makes pumping more difficult. Operation at high flowrates indirectly enhances the leach feed density since the shear rate is increased and apparent viscosity is reduced. This aspect of practical U/F pumping operation as well as startup and shutdown of non-Newtonian slurry systems needs to be carefully considered by future HPAL plant designers.

3.2.6 Leach Feed Slurry Surge Tank Agitator

Until upgraded in September 2002, agitation in the leach feed slurry surge tank was grossly inadequate. Increases in bottom deck aperture

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size on the OP sizing screen (as well as holed screen decks) allowed some coarse ore into the tank which promptly settled due to poor agitation. This effectively halved the active capacity of the vessel. A larger agitator was installed and the motor size was upgraded from 15 kW to 45 kW. Allowance was made for non-Newtonian slurry rheology in the agitator sizing. This has effectively increased surge capacity by 50% or 6 hours.

Prior to the agitator upgrade, leach throughput was often restricted due to upstream OP equipment failure. While OP plant failure still occurs, the impact on HPAL has been greatly reduced and leach is now rarely slowed due to upstream OP problems.

3.3 Ore Preparation Plant – Metallurgical Performance

Metallurgical performance following the numerous changes made in the OP plant is described below.

3.3.1 Mass Rejection

Figure 3.1 shows OP plant mass rejection. These values are calculated from monthly data.

A significant increase in mass rejection rate occurred in July 2001 when oversize from the top deck of the sizing screen was rejected.

A recent large increase in mass rejection has resulted in some shortfalls in leach feed ore. This has been addressed by introducing a charge into the ball mill (SAS mode).

While mass rejection could be maintained or even increased to further improve nickel upgrade this would require a higher overall OP plant capacity. Since the equipment is already operating well beyond the original design capacity any further increases would require capital injection.

3.3.2 Nickel Upgrade

Figure 3.2 shows OP plant nickel upgrade factor. These values are calculated from monthly data.

A significant increase in nickel upgrade factor was achieved when oversize from the top deck of the sizing screen was rejected in July 2001.

3.3.3 Nickel Recovery

Figure 3.3 shows OP plant nickel recovery. These values are calculated from monthly data.

Once again the change caused by rejection of oversize material from the top deck of the sizing screen is noticeable with an additional 3% loss in Ni recovery in OP.

The July 2001 change in rejecting top deck oversize was particularly successful since it increased the upgrade factor from ~5 to ~15% with only a minor drop (3%) in nickel recovery.

3.3.4 Cobalt Upgrade

Figure 3.4 shows OP plant cobalt upgrade factor. These values are calculated from monthly data.

There has been no detectable change in upgrade factor with recent modifications.

3.3.5 Cobalt Recovery

Figure 3.5 shows OP plant cobalt recovery. These values are calculated from monthly data.

It is clear that with the July 2001 change of rejecting the top deck of the sizing screen to waste there was a significant reduction in OP Co recovery with no noticeable improvement in upgrade factor. This probably indicates that the mineralogy of the ore containing Co is significantly different from that containing Ni.

By plant observation it appears that there is considerable quantities of coarse and heavy black high Co-Mn material in leach feed. Hence it is possible that coarse high-Co ore is being preferentially rejected into the sizing screen top deck reject stream explaining the significant drop in Co recovery.

It is possible that this cobalt could be recovered by spirals or some other mode of separation based on density and/or size. This remains an area that is yet to be investigated by Bulong metallurgical staff. At current cobalt prices there is little incentive to improve cobalt recovery. If the price improves then this project could be quickly progressed.

3.3.6 Leach Feed Density

Figure 3.6 shows OP plant leach feed density. These values are monthly averages of four-hourly spot samples.

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The average leach feed density is reasonably consistent and is generally above the design value of 31% w/w.

To avoid overfloccing, a device which directly measures feedwell solids settling velocity has recently been installed (March 2003). The unit is still undergoing commissioning but it is clear that improved flocculant control is achievable.

Warman have recently developed a new impeller – a "scoop impeller" which may allow pumping of high yield stress slurries. It is planned to install and test one of these devices at Bulong in the near future.

3.3.7 Effect of Pre-Blending on HPAL Feed [Ni], % w/w Variability

Figures 3.7 and 3.8 show frequency distributions for HPAL feed grade, % w/w Ni, before and after the introduction of pre-blending.

It is clear that pre-blending has had no noticeable impact on stabilising leach feed grade. Hence there must be other causes of variation more significant than that induced by the natural variability of the ore.

3.3.8 Effect of Pre-Blending on HPAL Feed Slurry Density, % w/w

Figures 3.9 and 3.10 show frequency distributions for HPAL feed slurry density, % solids w/w before and after the introduction of pre-blending.

It is clear that pre-blending has had no noticeable impact on stabilising leach feed slurry density.

As for leach feed Ni grade it appears that there must be other causes of variation more significant than that induced by the natural variability of the ore.

To maximise cash flow at Bulong, HPAL feed grade in terms of (tonnes Ni)/(m^3 of leach feed slurry) is "king". This parameter can be improved by **beneficiation of ore** and by **increasing leach feed density**.

3.4 Ore Preparation Plant – Other Issues and Potential Future Enhancements

- Logwasher mechanical availability continues to be poor and the machine is expensive to maintain. To accommodate high wear and general maintenance the OP plant is shut down every Thursday for 6-8 hours helping to minimise downtime associated with plant breakdown. Reduced crusher product top-size is also expected to improve availability.

- To eliminate the logwasher from the OP circuit some preliminary testwork on drum scrubbing has been completed with encouraging results. However, to install the machine would require a significant capital injection.

- Currently +1.6 mm material from the bottom deck of the sizing screen re-circulates to the ball mill. Rejection of this stream may also lead to further increases in the nickel upgrade factor.

- Reduced crusher top-size may allow operation without the scalping screen. This would eliminate continual and expensive maintenance of the screen.

- A flowsheet has been developed which would allow pre-heating of leach feed slurries using the first direct heater in the leach plant. Preheated slurries would then be thickened to higher densities at reduced viscosity. This process change would allow rejection of additional water from leach feed effectively increasing the capacity of the existing autoclave. This concept is still undergoing development by Bulong metallurgical staff.

4 HIGH PRESSURE ACID LEACH (HPAL)

4.1 General

Apart from some well documented mechanical failures (nozzles, agitator hubs) [2] [3] [4] the metallurgical performance of the HPAL plant is close to that predicted in the feasibility study.

Leach metal extractions (Figure 4.1) and acid/steam usage (Figures 4.7 and 4.8) have been close to design.

Bulong has found that the key to consistently good metallurgical performance is stable operation. Instability in autoclave and flash/heater vessel level and pressures has been identified as a major contributor to erratic metallurgical performance in terms of throughput, leach extraction and free acid control.

4.2 Recent Changes to Process and Process Equipment

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Changes to process configuration, equipment or operating practices within the HPAL plant are described below.

4.2.1 Leach Discharge – De-bottlenecking of Flash Tank Discharge (VSP109)

Due to low pressures in the final flash tank the discharge of slurry from this vessel is by gravity. High operating levels in this vessel is a cause of acid carryover into the heater circuit.

To overcome a hydraulic restriction in the discharge pipework the internal "upcomer" pipework 90^{o} bend was altered from a mitre bend to a short radius bend. The inlet to the upcomer was "fluted" to reduce hydraulic losses.

The changes were successful and have allowed an increase in normal autoclave flowrate from 260 m^3/h to 280 m^3/h (autoclave feed flowrate).

4.2.2 Installation of an Actuator on the Choke on the last flash tank – VSP109

Until November 2002 the variable choke on VSP109 was manually controlled. This led to sometimes poor control of the upstream flash tank level. Remote actuation is now possible. In the near future, level in the upstream flash vessel VSP108 will be measured and controlled using automatic control of the choke on VSP109.

4.3 HPAL Plant – Metallurgical Performance

Recent metallurgical performance of the leach plant is described below below.

4.3.1 Leach Extractions

Figure 4.1 shows average leach extraction data for Ni and Co from mid 1999. These values are derived from monthly data.

Poor monthly extractions are typical in planned shutdown months. The combination of low temperature and inadequate acid addition during autoclave start-up results in poor extraction.

Multiple leach plant "box-ups" (where leach plant is stopped but not de-pressurised) also lead to erratic leach extraction.

Poor leach extraction is also been caused by:

1. Insufficient downstream acid neutralisation capacity thereby forcing reduced free acid levels in leach.

2. Problems with acid supply necessitating reduced free acid in leach.

3. Problems with high pressure acid pumping systems leading to poor acid pump availability.

Neutralisation capacity was recently upgraded in December 2002 by adopting partial neutralisation on leach discharge slurry. This has allowed higher free acid (FA) in leach discharge directly improving leach extraction.

4.3.2 Effect of Pre-Blending on HPAL Discharge Free Acid (FA), g/L

Figure 4.2 shows the frequency distribution for leach FA prior to the introduction of pre-blending.

The distribution is distinctly "bell shaped" and has an estimated standard deviation of 6.0 g/L.

Figure 4.3 shows the corresponding frequency distribution for HPAL discharge FA after the introduction of pre-blending. Once again the distribution is distinctly bell shaped and has an estimated standard deviation of 6.0 g/L.

The distributions show that there was no reduction in FA variability after pre-blending was introduced. This was not expected.

There are two possible reasons:

1. The pre-blending method as currently practised may not be sufficient to blend out mineralogical variation. While this may be partially true, some reduction in variability should have been detected.

2. That FA variability in leach discharge is <u>not</u> overly sensitive to ore FA demand. There are other more significant causes of FA variability.

Other potential causes of FA variability are (amongst many):

a) Variability associated with leach feed density and flowrates and the actual acid/ore (A/O) ratio.

b) Operator/Supervisor/Metallurgist (Human) induced error - making changes that actually contribute to the variation detected.

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c) In-plant variability eg heater-flash variable dilution and flows due to unstable process control

Statistical process control (SPC) "Run Charts" are now being used in an attempt to eliminate human induced error in FA control.

Investigations are continuing into the elimination of internal leach plant induced FA variability.

4.3.3 Effect of Pre-Blending on HPAL Discharge Ferrous Fe(II), ppm

Figure 4.4 shows the frequency distribution for leach discharge Fe(II), before pre-blending. The distribution is very flat with no obvious “bell shaped” character.

Figure 4.5 shows the frequency distribution for leach discharge Fe(II), ppm after the introduction of pre-blending. This distribution shows more “bell shaped” characteristics. The lower limit is 0 ppm (leach is fully oxidising). Clearly the introduction of pre-blending has improved Fe(II) stability in leach discharge which in turn has improved the downstream recovery of Fe(II). Since there has been no change in FA variability, the improvement in ferrous stability must be due to greater pre-blending of ferrous containing minerals.

4.3.4 HPAL Mass and Energy Balance

Due to the complexity of the Bulong Flash/Pre-heat circuit, a mass and energy balance model has been created in a spreadsheet allowing modeling of changes to the energy recovery circuit. The model has also allowed Bulong to estimate the heat of reaction for most Bulong ore blends and has been used for changes in choke valve sizing.

A dynamic model of the heater/flash circuit would be useful for operator training and is a future project for Bulong

4.4 HPAL Availability

While not directly related to individual process plant improvement, leach availability is an important production parameter. Leach instability due to poor equipment availability does contribute to erratic leach extraction and erratic CCD settling (particularly with unleached ore).

Figure 4.6 shows monthly leach plant availability as measured by high pressure acid pump utilisation. Major shutdown events have been excluded. While utilisation remains erratic, monthly average utilisation has improved as the plant has ramped up.

4.5 HPAL Pant – Other Issues and Potential Future Enhancements

Control of the HPAL plant in terms of vessel pressures and levels remains a major hurdle to optimising throughput and metallurgical efficiency. Planned changes include:

- Improved autoclave pressure control by separating the safety vent duty from the pressure vent duty through the installation of a smaller vent control valve.
- Improved autoclave level control by providing continuous compartment level detection. Point detection leads to unstable level control and flows out of the autoclave to the first choke valve.
- Continuous level control on VSP108 (by controlling VSP109 choke) will result in improved flash/heater train performance.
- Conversion of the first heater to pre-heating of leach feed slurry may allow improved leach feed densities.
- Continued mass and energy model development to include dynamic modelling.
- Installation of an orifice plate into the flash steam line from the last flash tank to the first heater providing back pressure to force slurry flow out of the last flash tank.

5 NEUTRALISATION PLANT

5.1 General

The neutralisation plant and associated infrastructure (limestone grinding and lime slaking) prepare leach discharge liquor for solvent extraction (SX) of Ni and Co. A pH of approximately 5.5, combined with Fe, Al and Cr at levels <1-2 ppm is required for SX feed.

Free acid in leach discharge liquor is neutralised using Loongana limestone milled to approximately 90% passing 45 micron. Typical limestone slurry density is 25-35% w/w solids. Loongana limestone is a high quality limestone with minimal impurities and a high percentage $CaCO_3$.

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Fe(III), Cr(III) and Al(III) are hydrolysed and precipitate as hydroxides (possibly some iron-jarosite in primary neutralisation) as the pH is raised. Fe(II) is removed by first oxidising with hydrogen peroxide (H_2O_2). The ferric is subsequently removed by hydrolysis at the high pH.

Limestone is used for the bulk of the neutralisation/hydrolysis and achieves a maximum pH of ~ 4.5 - 5.0 within the residence time limitations of the Bulong equipment. Given greater residence time the limestone may achieve a higher pH.

The final pH adjustment to 5.5 to 5.8 is achieved by the addition of dilute slaked lime slurry.

The original neutralisation circuit at Bulong consisted of liquor neutralisation on CCD No.1 overflow (O/F) and was named PN for Partial Neutralisation. As installed, the circuit was grossly inadequate having less than one hour reactor residence time and a gypsum thickener about half the diameter actually required to settle and remove gypsum solids. This lack of capacity has been a major production bottleneck resulting in the loss of hundreds of tonnes of nickel production over the last four years.

5.2 Recent Changes to Process and Process Equipment

The neutralisation circuit at Bulong has undergone two major upgrades in the last two years. Firstly an upgrade to the pH Control System was implemented in February 2002 and secondly a complete re-configuration of the neutralisation circuit in December 2002 when slurry neutralisation facilities were installed and commissioned.

A brief description of these changes follows.

5.2.1 pH Control System Upgrade – February 2002

A number of equipment changes were made to improve PN performance.

- Rubber diaphragm on/off limestone slurry control valves were removed and ceramic control valves were installed (allowing continuous limestone addition). This change was successful and allowed stable limestone addition and good pH control in PN. As well as the ceramic control valve, a limestone flowmeter was installed allowing cascade pH control from pH probes to limestone flow control. The on/off valves originally installed surged the flow of limestone and failed regularly (within 3-4 days).

- The addition of limestone to CCD No. 1 underflow slurry was part of the original flowsheet. Several attempts were made to commission this facility, however, the addition of limestone could not be controlled. In the February 2002 upgrade a ceramic control valve and flowmeter was installed with the same pH control strategy as installed in PN. However, in this case the pH probe was relocated to CCD No. 2 feedwell. The changes were successful and partially de-bottlenecked the process.

- The original installation of air sparging into the main neutralisation tanks was with a single open pipe outlet into the tank directly under the agitator with air controlled manually. These lines frequently blocked and the flow of air could not be controlled. Furthermore the single point addition led to flooding of the agitators and inefficient dispersal of air. To solve this problem a new air sparger was installed. Air flow control valves were also installed. These changes were successful and have also minimised build-up of solids at the bottom of tanks that was seen previously. The improved air dispersion has allowed operation of the tanks without mechanical agitation for weeks. Previously this would not have been possible and would have led to a plant shutdown to rectify.

- A de-gassing tank was originally installed between the last PN vessel and the final PLS clarifier as part of the initial design. However, a fatal flaw in the initial piping installation required that the vessel was removed from service. Changes made in February 2002 allowed the vessel to be recommissioned. New pipework allowed gas to escape from pipework (preventing gas hold-up and restricted flow) and on-line gypsum cleaning. These changes were very successful and clarifier overflow solids levels have dropped significantly. The clarifier is no longer affected by highly turbulent and gaseous flow entering the vessel.

5.2.2 Slurry Neutralisation – December 2002

Slurry neutralisation has been previously attempted at Bulong. However, the facilities installed were inadequate and failed to

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continuously supply limestone to the leach discharge tank.

Following some additional batch testwork at Bulong, a series of pilot runs were completed at Lakefield-Oretest in early-mid 2002. While these tests successfully demonstrated that acid could be neutralised and that the bulk of the solids would settle, two potential problems were found:-

1. Foaming of neutralisation reactors appeared excessive and uncontrollable.

2. The clarity of CCD overflow solutions was poor indicating that a coagulant would be required.

Despite these potential problems, planning for installation of slurry neutralisation commenced.

By mid 2002 it was clear that the existing leach discharge surge tank would have to be replaced. Attack by SO_2 fumes (from SMBS addition) and general acid and chloride corrosion meant the vessel had to be completely replaced. Since a new tank was to be built anyway it was decided that it should be capable of operating as a neutralisation reactor.

The reactor was installed in December 2002 with the following features.

- A single reactor/tank of the same size as the original was installed.

- The reactor operates at 60-70% level providing approximately 60 minutes residence time and allows for sufficient disengagement space for foam and entrained liquor and solids that may be present in vented water vapour/CO_2.

- The reactor operates with the pre-existing bottom discharge pumps allowing operation with disengagement space and surge volume if required (approximately ½ hour of surge is provided).

- Significantly upgraded agitator (size increased from 15 to 75 kW). A high agitator tip speed (6.2 m/s) was originally installed for removal of entrained CO_2, however, this was later reduced to 5.0 m/s to avoid excessive wear on agitator blades.

- Installation of differential pressure (dP) and radar level detection. Both forms of level detection have proven successful. The radar system appears more robust as it does not readily lose calibration.

- An increased tank overflow line size was installed to accommodate potential foaming in the reactor. Fortunately, no foam related problems have occurred thus far.

- Limestone slurry is added to the reactor in CSTR (continuously stirred tank reactor) mode, whereby the limestone is added to main reactor contents (already at a higher pH) and does not directly contact the acidic slurry as it enters the tank. Sufficient residence time is available for this mode of operation to allow gypsum particle growth and gypsum growth on leach residue slurry particles. Limestone utilisation at the design residence time has been good.

- A 3m high vent stack has been installed to enable steam and CO_2 to discharge away from the top of the vessel improve the general working conditions near the top of the vessel.

- The existing leach discharge slurry flowmeter is used to control level in the reactor.

- pH probes were installed in CCD No. 1 feedwell to allow cascade pH control to limestone addition on the new reactor. This control strategy is still to be fully commissioned.

- A ceramic control valve and flowmeter was installed to control limestone addition to the reactor.

5.2.3 Upgrade of Clarifier Underflow (U/F) Pumping Capacity

Prior to September 2001, the capacity of the clarifier U/F pumps was a severe throughput limitation. As the upstream gypsum thickener was grossly undersized, it frequently (sometimes continuously) “slimed” solids into the clarifier. While the bulk of the solids settled in the larger clarifier, the U/F pumps were sized for a much smaller duty and operators frequently “dumped” the thickener to the sump to recover the thickener.

To minimise wastage and avoid environmental problems associated with spillage of liquors the pumping capacity was increased by installing a diesel driven pump. This almost completely eliminated the practice of dumping and allowed improved overall production.

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5.3 Neutralisation Plant – Metallurgical Performance

5.3.1 Total Suspended Solids (TSS) in PLS Clarifier Overflow

TSS is a measure of clarifier performance in terms of solids removal. Solids present are precipitated $Fe(OH)_3$ and carry-over gypsum from the gypsum thickener.

Performance statistics before and after the February 2002 pH control changes are shown below.

Bulong PLS Clarifier Overflow Solids, ppm prior to February 2002 pH Control Changes

Mean	144
Median	70
Standard Deviation	544

Bulong PLS Clarifier Overflow Solids, ppm after February 2002 pH Control Changes

Mean	61
Median	40
Standard Deviation	154

Solids levels in PLS have more than halved since February 2002 and variability has also substantially improved. These changes have been so successful that operation of Co SX without the PLS sand filters being on-line is possible. The sand filters have historically scaled with gypsum and cleaning and maintaining the filters has been costly.

While operation without PLS filters is not ideal, the improved performance of the clarifier has allowed Bulong to save substantial amounts of money in cleaning and maintenance.

It is still too early to establish if the conversion to slurry neutralisation has affected TSS levels since the modifications were made in mid December 2002 and poor plant availability in January 2002 caused numerous process upsets. However, based on limited assay data available it appears that clarifier TSS has not been affected.

5.3.2 pH of PLS – Clarifier Overflow

Stable pH control is critical for good SX performance and for removal of minor impurities Fe, Al and Cr.

Performance statistics before and after the February 2002 changes are shown below.

Bulong PLS Clarifier Overflow pH prior to February 2002 pH Control Changes

Mean	5.4
Median	5.5
Standard Deviation	0.6

Bulong PLS Clarifier Overflow pH after February 2002 pH Control Changes

Mean	5.4
Median	5.4
Standard Deviation	0.3

Clearly pH control has improved since February 2002 halving the standard deviation of PLS pH.

It is still too early to establish if the conversion to slurry neutralisation has affected the control of pH in PLS. However, based on limited assay data it appears that pH control has not been affected.

5.3.3 Impact of Slurry Neutralisation

Slurry neutralisation has allowed more stable operation of the secondary neutralisation (SN) circuit (old PN circuit) and has largely prevented sliming of the gypsum thickener into the PLS clarifier. This has translated into increased volumetric throughput to the PLS pond since greater leach flowrates are possible.

Despite pilot plant testwork indicating a potential problem with liquor clarity in the CCDs, actual plant CCD O/F clarities appear no different to pre-slurry neutralisation. It appears that no coagulant will be required at Bulong.

There has been no observable impact on CCD U/F density. This may not be the case for other ores/projects since Bulong densities are normally very low (19-25% w/w solids).

There have been some negative effects with the introduction of slurry neutralisation:

- Motor power for CCD No. 1 U/F pumps is clearly undersized for the new duty (and the higher tonnes in general) and requires upgrading. While envisaged in the initial design, no up-front allowance was made. Design for upgraded power supply is in progress.

- In the original liquor neutralisation mode CCD No. 1 O/F liquor is not saturated in $CaSO_4$ (considerable Ca precipitation

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occurs in the HPAL heater circuit). This is not the case with slurry neutralisation and scaling of the overflow pipework has meant that frequent cleaning is required to maintain flows. Since discharge from the CCD is by gravity any scale formation quickly reduces flow.

Work has commenced to install open launders for on-line cleaning. Reduced flows translates into reduced wash water and wash recovery is suffering.

Once these problems are solved it is expected that consistently high flowrates will be maintained.

5.4 Other Issues and Potential Future Enhancements

- The pH control scheme to control CCD No.1 feedwell pH by controlling limestone addition to the leach discharge tank remains to be fully commissioned. This is required since consistent leach chemistry has been found to be important in CCD No. 1 & 2 for optimum flocculant/settling performance.

- Several new types of pH probes including units which continuously purge electrolyte will be tested within the neutralisation and SX plants at Bulong. pH control is a considerable problem at Bulong. Continuous drifting of probes, scale and general wear and tear translate into a significant cost burden for the plant. Effectively one full-time instrument fitter continuously calibrates the dozens of pH probes used at Bulong.

- The U/F density of the gypsum thickener is considerably lower than design (~ 6 - 9% vs 30% w/w solids) and seeding of gypsum has been attempted several times with mixed results.

  Some improvement in gypsum thickener U/F density has been observed using limestone conditioning of gypsum thickener U/F slurry. In this approach, limestone slurry is intimately mixed with recycle gypsum seed and the combined slurry is directed to the acid neutralisation circuit. In theory, the limestone coats the gypsum particles forcing additional growth of the gypsum particle. Unfortunately high recirculation rates of seed could not be produced to take maximum advantage of this concept (25% recycle rate only could be achieved). Bulong plans to attempt another plant trial of this technology within the next year.

- The addition of limestone and acid liquor has created considerable debate within the metallurgical group at Bulong. As designed, limestone slurry and acid liquor are mixed in a feedbox to the side of the first SN (PN) tank. This approach leads to the maximum rate of acid neutralisation since limestone is reacting with free acid ~ 15 g/L, however it is also believed to be conducive to the formation of huge amounts of fine gypsum particles due to the very large supersaturation of gypsum. Addition of limestone to the tank in CSTR mode (slurry already at pH 2.5) should lead to the generation of larger crystals of gypsum having better solid-liquid separation properties, however the kinetics are reduced.

  At Bulong it appears to make little difference how the limestone is added and no noticeable improvement in thickener performance was detected using either approach.

- The problem of lack of thickener U/F pumping capacity has been extreme at Bulong. Future projects containing like equipment must allow for :-

  a) Operation at U/F densities considerably lower than design. Hence a large range of U/F flowrate and density must be accommodated.

  b) Operation at solids feed rates considerably greater than design to accommodate fluctuations in leach discharge mass flux of free acid and variations in PLS flowrate

  c) Normal thickener “catch-up” capacity.

  Future designers must carefully consider the design envelope for any gypsum thickener or PLS Clarifier.

- Slurry neutralisation is probably only a temporary solution to the neutralisation of excess acid in leach discharge. Solution neutralisation with separate filtration and washing of coarse gypsum would appear to be the preferred long term solution. Separate removal of gypsum and leach residue would allow higher on average U/F densities within the CCD plant and

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effectively expands the CCD circuit since a significant solids stream is removed.

- Future designers should be generous in the sizing of tankage and thickeners in neutralisation circuits. Considerable variation in all aspects of plant performance has been found in practice at Bulong.

- Due to continual sliming of the gypsum thickener at Bulong, the downstream PLS clarifier has never been operated in true clarification mode since this requires a solids bed to be maintained in the vessel. Due to the continual surging and upsets seen in the circuit in the past it was not possible to operate in this mode without the considerable risk of carrying the clarifier bed into the overflow (PLS). Hence understandably operators run with no or little bed level. Only recently with the bulk of the neutralisation load shifted to slurry neutralisation is Bulong in a position to consider operation in clarifier mode.

  It is clear from previous testwork and surveys that operation in clarifier mode will generate substantially lower solids in PLS and should assist in helping removal residual aqueous silica in solution.

- Considerable scale has been noticed in the first tank of SN (PN). Seeding and limestone addition direct to CCD O/F seems to have reduced the rate of scale growth.

6 PLS POND & PLS SAND/ANTHRACITE FILTERS

The PLS pond is nominally 48 hours capacity but due to carryover is of solids it is reduced in capacity to effectively 70% of the original volume.

The pond has been cleaned at least twice using a small dredge a very effective process that removes the bulk of the settled solids.

The PLS filters are not currently used since they quickly scale with gypsum. The high pressure water cleaning process is aggressive and destroys the rubber lining of the internal fittings. This in turn leads to considerable maintenance costs.

It is planned to re-introduce the filters if the addition of anti-scalant to PLS as part of an on-going plant trial is successful.

7 COUNTER CURRENT DECANTATION (CCD)

7.1 General

A seven stage CCD circuit was installed at Bulong. There are provisions and foundations available for an eighth thickener if required in the future.

At best the settling performance of Bulong leach residue/gypsum can be described as mediocre. At design thickener U/F densities of between 21 and 23% w/w solids, very high wash ratios are required to achieve reasonable Ni and Co recoveries.

Ni SX raffinate is used as wash water for the CCDs with make-up process water (PW) as required. Because the Bulong CCD and SX circuits are intimately linked (with some minimal surge in the PLS pond) the higher the average flowrate through SX the higher the average wash flow through the CCDs.

Each CCD thickener has an agitated mixing tank where n+1 thickener O/F is combined with n-1 thickener U/F solids.

Flocculant make-up at Bulong is industry standard.

Neat flocculant distribution is made via a ring-main. Testwork has shown that some flocculant activity is lost when using a ring-main due to shear within pipework and control valves.

Flocculant dilution for final addition to individual thickeners is via washer “n” O/F liquor. Individual flocculant dilution pumps take O/F and mix with neat flocculant in-line. Gross amounts of solids in the O/F will reduce flocculant effectiveness and thickeners are recovered using PW. However, the use of PW for long periods of time impacts on CCD metal recovery as this effectively backs out wash water that would normally be added to the last CCD washer.

7.2 Recent Changes to Process and Process Equipment

With several years of operating experience the following parameters have been identified as critical to achieving consistently high metal recovery:

1. Consistent and high wash rates. Any reduction in wash flowrate for downstream problems in SN (PN) or SX will immediately and substantially impact metal recovery.

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The plant trial application of anti-scalent since June 2002 has allowed consistently higher PLS flowrates through SX and some marginal improvement in CCD recovery has been observed. De-bottlenecking of neutralisation has also improved wash flows.

2. Consistent and stable operation. The Bulong CCD circuit, like others, requires consistent operation. Good operator training has been found to be very important. Inconsistencies between operating shifts have led to major circuit imbalances in terms of solids levels and solution flows. After wash ratio this aspect of plant operation has been found to be the most important for achieving high metal recoveries with minimal flocculant consumption. Bulong continues to develop and implement training strategies aimed at providing consistency of operation.

3. Feedwell density. Bulong CCDs were designed to achieve a 7.5% w/w solids density in the feedwell at design wash flows. Until recently design flows have not been achievable due to SX gypsum problems and much greater densities have been observed in practice. High feedwell densities translate into viscous pulps in the feedwell and very poor solids flocculant contact. Poor CCD O/F clarities and very high flocculant usage are the end result.

   Compounding the on-average low wash flows for most of the operating life of the CCDs, the wash flowrates have also been unstable. Since the CCDs use e-duc devices for solids dilution (drawing in supernatant clear liquor into the feed pipe), unstable flows cause unstable dilution further adding to the overall instability of the thickener operation.

   It has also been confirmed in recent testwork that the design value of 7.5% is too high to allow good contact between solids and flocculant and that values between 5 and 6% are required. Operation at the lower density has been found to more than halve the specific flocculant usage.

   To partially overcome the high feedwell density problem, additional thickener O/F liquor is pumped back into the feedtanks of individual washers using the spare CCD O/F pump. At low flows this has been found to be very effective. However, at high flowrates the washer gets hydraulically overloaded. Plant trials have shown reduced flocculant consumption and improved clarities with additional external dilution of solids, confirming bench scale testwork.

   Additional dilution in the feed tank has also allowed the contact of dilute flocculant in the feed tank itself, providing additional time for flocculant solids contact.

   Other means of enhancing feedwell dilution are being considered by Bulong.

4. Flocculant dilution. Consistent dilution of neat flocculant is essential for good solids-flocculant contact and Bulong has trialed numerous alternative approaches. Neat flocculant addition into the n+1 O/F line has been plant trialed and found to be an improvement on current flocculant addition strategies. An engineered system will be installed in the future.

5. Process control. To maintain consistent and high metal recoveries a good process control strategy is required. Due to the erratic flows and unstable behaviour in the CCDs, much of the existing instrumentation has not been utilised and has been neglected. Interface level probes, bed pressure, U/F density and flow meters have in the past been unused.

   In February 2002 and with further refinements in January 2003 a cascade bed pressure control strategy was implemented to stabilise and increase U/F densities. This approach has been largely successful and has resulted in improved U/F densities.

   A wash ratio control strategy has also been devised and partially installed. This will allow control of the CCD wash ratio to near optimum levels at all times. Further enhancements and expansion of this control strategy are planned for Q3 of this year.

7.3 CCD Plant – Metallurgical Performance

Figure 7.1 shows CCD Ni (and hence Co) recovery since early 2000. These values are monthly values. Recovery continues to be below the design level (>98%) and currently averages approximately 94%. However, it is also clear that CCD recovery is slowly improving with time.

It is still too early to determine the impact of recent feedwell density changes on flocculant consumption, however, on individual days flocculant consumption has been halved. Bulong is confident that flocculant consumption

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can be consistently and substantially reduced in the future.

8 COBALT SOLVENT EXTRACTION (Co SX)

8.1 General

The function of Co SX is to separate Co from Ni producing a "Co free" raffinate for Ni SX. Cyanex 272 (C272) extractant @ ~ 15% v/v in Shellsol 2046 diluent is used. C272 also extracts other impurity elements not removed in the neutralisation section of the plant notably Mn, Zn and Cu. Mg is also partially extracted but can be largely scrubbed from the organic. Ni is also extracted in small quantities.

A flowsheet of the Bulong Co SX circuit is attached.

8.2 Recent Changes to Process and Process Equipment

Recent changes in Co SX include:

1. Use of anti-scalant (AS) to minimise gypsum formation in plant equipment

2. Modifications to the strip circuit such that the strip liquor is added in a "single pass" arrangement. This minimises Co entrained in stripped organic which will re-extract in organic wash and/or Co E5.

3. Substantial upgrade of ammonia addition facilities to Co E5, enabling higher pH operation and lower Co raffinates at above design flowrates.

4. Installation of additional ammonia vapourisation facilities to prevent condensation of ammonia and freezing of control valves and pipework.

5. Re-configuration of Co SX raffinate diluent wash and C272 recovery plant and equipment to maximise the recovery of C272 and prevent ingress of C272 into Ni SX.

8.3 Co SX Plant – Metallurgical Performance

8.3.1 Anti-Scalant (AS) Plant Trial

Gypsum formation creates a hydraulic restriction in pipes and valves which limits flow through both SX circuits. While some flexibility has been installed to allow bypassing for cleaning, frequent flow changes create inefficiency and metal loss.

The frequency of cleaning, particularly in Ni SX, also leads to high cleaning and maintenance costs.

A low capital cost solution to minimising the impact of gypsum has been the addition of anti-scalant (AS) to SX as part of a plant trial. The trial is approximately a year old and thus far has proven successful. There has been no adverse impact on SX or in any other part of the Bulong process. Scale formation has been considerably reduced allowing extended operation before cleaning.

The primary advantage of AS is that it allows higher average flowrates of wash water through the CCD circuit since Ni SX raffinate is used as wash. Poor SX flowrates directly translate to poor CCD metal recovery.

Figure 8.1 shows weekly Co SX flowrates. Since June 2002 the average flowrate has increased and overall Ni and Co recovery have improved.

Prior to the introduction of AS the average Co SX flowrate was approximately 275 m^3/h (March 2000 to May 2002). There were periods of higher flowrates after major gypsum cleaning but flow would reduce within a few weeks of restarting.

From the period when AS was introduced to December 2002 the average Co SX flowrate was 315 m^3/h (May 2002 to December 2002), a 13% increase in average flow. In December 2002 major changes were made to the neutralisation circuit which further de-bottlenecked solution flows and the average flowrate through Co SX is now 335 m^3/h, representing a 20% increase in average Co SX flow since the introduction of AS. Part of this increase in flow could be attributed to a clean circuit. However, Figure 8.1 clearly shows a consistently improving Co SX flow performance over many months of operation.

8.3.2 Modifications to the Co SX Strip Circuit

The Bulong Co SX strip circuit was initially configured with a strip liquor tank that circulated strip liquor with make-up sulphuric acid and water added to the tank. However, entrained strip liquor in stripped organic containing aqueous at the Co concentration of the strip liquor led to re-extraction in the organic wash stage and/or in Co E5. Effectively Co was being

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inadvertently added to barren organic restricting the ability of the extraction circuit to recover Co down to low raffinate levels.

The circuit has now been modified such that strip liquor is single pass only. Dilute acid contacts organic in a countercurrent arrangement hence the fresh strip liquor contacts an organic that has been largely stripped in preceding strip stages and any entrained aqueous contains only low levels of Co. The advance flowrate of strip acid is significantly lower than installed and aqueous/organic ratios are maintained on individual stages by internal recirculation.

A flowsheet showing these changes is attached.

The focus of this change is improved Co recovery and the reduction in Co in raffinate. Co in raffinate directly impacts on Ni cathode product quality.

Figure 8.2 shows Co contamination in Ni cathode product. A step change occurred after the introduction of single pass stripping and variation in Ni product quality certainly improved.

This modification was made with no capital injection requiring DCS programming changes only.

There is no doubt that other low capital process improvements are possible within the Bulong flowsheet via the application of modern process control principles.

Ni cathode quality statistics before and after the Co strip circuit changes were:

Co in Ni cathode	**Before**	**After**
Average, ppm Co	5,918	4,828
Median, ppm Co	5,752	4,570
Standard dev, ppm Co	1,928	1,719

While these sets of data do not appear significantly different, the Co strip circuit changes occurred at the same time when substantially increased SX flowrates were achieved due to the presence of AS. Historically Ni product quality has been very sensitive to increased flowrates with a noticeable reduction in quality at higher throughput. It should be noted that additional C272 was added to the circuit in the latter half of 2002 also providing conditions for improved product quality.

Further investigation of the performance of the strip circuit is required. Numerous cobalt containing streams continue to be added to the acid make-up tank for the strip circuit and these extraneous sources of Co need to be redirected to fully realise the benefits of “clean” aqueous entrainment.

8.3.3 Upgrade to Co E5 Ammonia Addition

Ammonia is added to the extraction circuits in both Co and Ni SX to control pH during the recovery of metals. Ammonia is added as an anhydrous gas directly into the advance organic pipeline effectively pre-equilibrating C272 prior to entering the mixer.

Apart from periods of low Co SX PLS flowrate the pH in E5 could never be increased above ~4.5 to 4.8 due to restrictions in ammonia supply. With increased flow from the addition of AS and the desire to eventually achieve LME grade Ni (<1500 ppm Co) it was decided to upgrade the ammonia addition facilities to Co E5.

Due to the poor selectivity of C272 for Co over Mg, substantial Mg and Ca extraction occurs in E5, which is then later crowded (or scrubbed) in E1-E4. Hence substantial quantities of ammonia are required to achieve a final pH of ~5.8 in E5.

Part of the ammonia addition problem is that high ammonia gas flows also tends to back up organic flow since a finite amount of time is required for the ammonia equilibration reaction to occur.

Ammonia piping and valving was increased in size to accommodate higher flows and a second ammonia sparger was added to the organic line splitting the ammonia flow and allowing staged pre-equilibration, minimising the hold-up of organic due to gas.

These modifications were successful and have allowed consistently higher pH operation in Co E5 even at significantly higher flowrates than previously experienced. The impact of this change is also clearly evident in Figure 7.2.

Statistics for Ni cathode quality after these changes are as follows:

Average, ppm Co	1,775
Median, ppm Co	1,500
Standard dev, ppm Co	800

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Product quality has substantially improved. The variability of Co in metal has also reduced.

Bulong believes that further optimisation of the extraction and strip circuits will allow the LME specification of <1500 ppm Co in Ni cathode to be consistently achieved in the future.

8.3.4 Installation of Additional Ammonia Vapourisation Facilities

The supply of gaseous ammonia to the SX circuits has been surprisingly difficult at Bulong. Liquefied ammonia stored in "Bullets" is vapourised in a unit supplied by steam. However, this vapouriser only generates saturated ammonia gas. In winter there is substantial condensation of ammonia gas in supply pipework causing unstable flows. Unstable flow has (and continues) to cause erratic ammonia supply to all SX extraction stages.

As well as condensation in pipework, significant condensation also occurs at any point where there is a pressure drop in the ammonia supply system, particularly the control valves. This Joule-Thomson effect creates additional liquid ammonia causing two-phase flow and instability.

To overcome the condensation problems a secondary ammonia vapouriser was installed in August 2002. This unit is designed to superheat ammonia. However, problems with control of steam addition have plagued the operation of the unit and further modifications are required to ensure stable vapouriser operation.

Attention to detail during the initial design of these facilities would have eliminated the subsequent operational problems. These problems could have been avoided.

8.3.5 Re-Configuration of Co Raffinate Diluent Wash (DW) and Cyanex 272 Recovery (C272-R) Circuits

Nofal [(1)] referred to the commissioning of a diluent wash (DW) process whereby raffinate from the Co SX (containing entrained C272) circuit would be washed using fresh diluent.

A flowsheet of the DW and C272-R circuits is attached.

The process and plant equipment were commissioned in May-June 2001.

Recirculating diluent removes entrained C272 from raffinate. All fresh diluent make-up to Co SX goes through DW. The existing Jameson cell recovers dilute entrained organic from the DW unit.

However, due to the large amount of entrained organic and the need to maintain low levels of C272 in the Versatic Ni SX circuit a separate C272 recovery circuit is required.

Bulong has developed and refined a process whereby C272 can be selectively extracted from an organic phase into an aqueous phase thereby allowing recovery of the organic extractant. The diluent is unaffected.

DW organic is contacted with a dilute ammonia stream at pH~9.5-10.5 which converts the C272 extractant into an aqueous form. To recover C272 the reaction is reversed by contacting the aqueous phase with a low pH aqueous liquor. Excess strip acid is used to recover the C272.

The initial installation for DW was successful in recovering entrained organic. However, the C272-R circuit suffered from a number of metallurgical problems which required equipment modification. Unfortunately these modifications were only completed in December 2002 due to cash flow constraints.

The December 2002 modifications were successful and the capacity of the C272-R process has been doubled with improved pH control. The process is still undergoing metallurgical optimisation with the aim of further improving recovery and efficiency.

Not only do the DW and C272-R circuits prevent major contamination of the V10 circuit, C272 is recovered for re-use representing an important cost saving. C272 is an expensive reagent.

8.3.6 Crud Filter

No crud filters in either Co and Ni SX were installed as part of the original installation.

The movement of crud from Co SX to Ni SX has been identified as an important contributor to C272 contamination in Ni SX. A new crud filter was installed in May/June 2001 and after some initial teething problems has been successful in removing crud from the Co SX circuit.

Bulong metallurgical staff and others continue to work on minimising crud formation. Bulong has identified that soluble silica appears to be the primary cause. However, excessive flocculant and solids carry-over as well as low pH PLS are also known contributors to crud formation.

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9 NICKEL SOLVENT EXTRACTION (Ni SX)

9.1 General

The function of Ni SX is to recover and concentrate Ni for the purposes of final metal recovery by electrowinning. Versatic 10 (V10) extractant @ ~ 18% v/v in Shellsol 2046 is used. Four stages of extraction are installed at Bulong. Extraction of any remaining impurity metals (in Co SX raffinate) like Co, Cu, Zn also occurs. Some Mg extraction also occurs but can be readily scrubbed from the organic. Most importantly, Ca extraction occurs when barren organic contacts Ca saturated aqueous in E4, particularly at higher pH (>6.8). Crowding of Ca with Ni in E2 and E3 within the extraction circuit supersaturates an already Ca saturated solution. This mechanism is responsible for a significant proportion of the gypsum formed in Ni SX.

Any contamination of V10 with C272 also increases Ca extraction particularly at the pH's used for Ni extraction. It is important that C272 concentration is kept below about 0.7-0.8% v/v in Ni SX.

To minimise the extraction of Ca and deportment into EW, good pH and O/A control is critical. pH is used to minimise Ca extraction while organic/aqueous (O/A) ratio is used to minimise the number of available sites for Ca to load. Due to variable aqueous flowrates and input Ni metal throughput, Ca extraction is also variable. Therefore to ensure that Ca concentration on loaded organic is minimised, the circuit is operated so as to leave some Ni in raffinate. The target Ni in raffinate at Bulong is currently 80 ppm.

A flowsheet of the Bulong Ni SX circuit is attached.

9.2 Recent Changes to Process, Plant and Equipment in Neutralisation

Apart from minor "tweaking" of pH and O/A ratios the major change in Ni SX has been the addition of an anti-scalant (AS) into Ni SX.

A plant trial commenced in May 2002 and has seen a dramatic reduction in the rate of scale formation throughout the extraction circuit. The operating time between de-scales has been substantially increased. De-scaling costs have been reduced and average flowrates increased.

9.3 Ni SX Plant – Metallurgical Performance

Figure 7.1 shows the improvement in Co SX PLS flowrate with substantially improved flows since the introduction of AS into Ni SX in May last year.

Table 1 below shows [Ni] in Ni SX raffinate since the beginning of 2001. Prior to a complete V10 change-out in November 2001, average [Ni] in raffinate was 226 ppm. Assuming a design PLS [Ni] of 3200 ppm, this equates to a Ni recovery of 92.9%. After the V10 change-out (C272 "free") and combined with the commissioning of DW and C272-R, the average raffinate grade dropped to 101 ppm improving Ni recovery to 96.8%. Since the AS plant trial commenced, raffinates have been further reduced to 80 ppm or approximately 97.5% Ni recovery.

Additional significant improvements in [Ni] in raffinate will only occur if the selectivity between Ni and Ca can be improved. This would most likely require a change in the Ni extractant.

Table 1 : BOPL Nickel Raffinates

	Jan-Nov 2001 [Ni] Raff. ppm	*Dec 01-May 02 [Ni] Raff. ppm*	*Jun 02-Mar 03 [Ni] Raff. ppm*
Mean	226	101	80
Median	180	60	60
Std Dev	264	209	115

The period January 2001 - November 2001 was an operating period prior to a complete Versatic inventory changeout. Versatic contained high levels of C272.

The period December 2001 - May 2002 included fresh V10 as well as a commissioned DW and C272-R process in May/June 2001

The period June 2002 - March 2003 incorporates the AS plant trial.

Only minor modifications to the Ni SX circuit have been attempted thus far, leaving room for considerable optimisation and improvement in the future.

9.3.1 Ni SX – The Future

Bulong has shown that Ni recovery from laterite leach liquors is possible using V10. However, the problem of gypsum formation is significant and must be addressed during the design process if the Bulong flowsheet is to be adopted in future projects.

Any extractant that enables Ni recovery without Ca (and to a lesser extent Mg) would represent a significant enhancement to the technology and should be pursued.

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As mentioned by Nofal [1], the use of Synergistic Extractants (V10 + S = V10S) for the extraction of Ni is an exciting development and Bulong intends to fully explore the potential of V10S. Particularly interesting is the potential to extract Ni at a lower pH thereby further reducing Ca co-extraction caused by C272 contamination.

A further enhancement would be to reverse the SX circuits and have V10S extract both Ni and Co while minimising co-extraction of Ca, Mn and Mg. Co would be recovered from a more concentrated Co stream containing fewer impurities. Co recovery and Ni cathode quality would also be improved. Avoiding the extraction of Ca, Mg and particularly Mn would lead to substantial savings in ammonia and strip acid. The extraction of Mn by C272 represents a considerable cost burden.

In combination with Zn and Cu IX it may also be possible to recover Co as cathode directly and hence not require the use of messy and expensive sulphide precipitation to separate Co from impurity elements Mg and Mn.

It is disappointing that little commercial progress (on synergistic extractants) has been made since May 2001 and the industry in general needs to "move on" quickly if direct SX (DSX) is going to be seen as a viable technology for the winning of Nickel. Bulong has a considerable commercial incentive to ensure this technology is developed and intends to play a leading role in demonstrating the process.

10 NICKEL ELECTROWINNING (Ni EW)

10.1 General

Electrowinning is used for Ni metal recovery at Bulong. Bagged cathodes and starter sheet technology is used.

The primary changes in the tankhouse have not been process related but more focused on supervision/management. A separate Tankhouse Supervisor has been appointed to improve overall quality and good housekeeping.

The Bulong tankhouse, like most others, can produce a consistent quality product if attention to detail and housekeeping is practised.

A flowsheet of the Bulong Ni EW circuit is attached.

10.2 OH&S

OH&S issues within the tankhouse are important. Most of Bulong's injuries and accidents occur in the labour intensive tankhouse. Some recent innovations are:-

1. Improved protective equipment to minimise lacerations.

2. Improved ventilation and acid aerosol protection for tankhouse workers.

10.3 Tankhouse Automation

The Bulong tankhouse has very little automation and requires a considerable labour workforce.

A recent change has been to install a machine which allows faster and more efficient hanger strap attachment to starter sheets. The machine rivets the hanger strap to the starter sheets using a hydraulic press. Previously hanger bar attachment was done using a less efficient spot welding machine. The press is still undergoing commissioning but at this stage is performing well.

10.4 Cobalt EW Cell Conversion

At the current low Co prices it is not commercially viable for Bulong to produce Co metal cathode.

It is possible to convert the existing Co EW cells to Ni cells and hence improve the capacity of the Ni tankhouse by several tonnes per day. This conversion is currently in progress. An important design consideration is the ability to quickly convert back to Co if the price of Co rises in the future.

10.5 Current Efficiency (CE)

Monthly CE data is shown in Figure 10.1.

The CE of the Bulong tankhouse currently averages approximately 85% having risen substantially in the last 12 months from 78-80%. Bulong is aiming for a world's best practice CE of 95-97%.

Projects aimed at minimising impurity contamination in electrolyte and improved pH control should enable substantially improved CE in the future.

11 COBALT REFINERY

11.1 General

The Co refinery process has been described by others. [1] [2] [3]

As previously mentioned, the price of Co does not warrant the operation of the cobalt refinery and cobalt is recovered as a sulphide and sold

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as a bagged wet CoS filter cake. Hence the only operating sections of the Co refinery are the CoS precipitation reactors, CoS thickener and filter press as well as associated tankage and pumps.

CoS is precipitated from Co SX strip liquor using dilute sodium hydrosulphide (NaHS) and using dilute caustic soda (NaOH) for pH control. Precipitated solids are then thickened, filtered and washed prior to bagging in "bulki-bags" for shipment and sale.

11.2 Recent Changes to Process and Process Equipment

Originally the sulphide precipitation process used sodium sulphide (Na_2S) and sodium carbonate (Na_2CO_3) and while the process worked, it suffered from the following:

1. Generation of excessive H_2S from CO_2 stripping generated by the addition of Na_2CO_3.

2. Sometimes very fine "unfilterable" precipitates.

3. Unstable and erratic pH control

To improve the process the following changes have recently been made:

A. Use of NaHS instead of Na_2S. This has improved the quality of final product in terms of better physical size distribution of precipitate and reduced chemical impurities.

B. Use of NaOH instead of Na_2CO_3 for pH control. This eliminates CO_2 generation and reduces the load on the ventilation scrubber. OH&S issues associated with H_2S generation have reduced since this change was made.

C. As in any sulphide precipitation, how and where the reagents are added is critical. This is a trial and error process and Bulong, after numerous attempts, has identified a reagent addition configuration which gives a readily filterable precipitate with high Co recovery and with below-design barren liquor (BL) Co values. This "near optimum" configuration was established in December 2001 and has not changed since.

D. Addition of NaHS using a feed-forward control strategy.

   NaHS is a naturally caustic reagent being essentially a mixture of caustic soda and H_2S. Upon reacting with $CoSO_4$ to precipitate a CoS the "basic" component of NaHS does some neutralisation of acid-forming Na_2SO_4. The remainder of the acid is neutralised with dilute NaOH

   i.e.

   $2CoSO_4 + 2NaHS \rightarrow 2CoS + Na_2SO_4 + H_2SO_4$

   and

   $H_2SO_4 + NaOH \rightarrow Na_2SO_4 + H_2O$

   Clearly there is some interaction between NaHS addition (which also contains some excess base) and caustic addition for pH control. This interaction led to sometimes poor and erratic pH control.

   To overcome the inherent conflict, a feed-forward control strategy was adopted whereby the amount of Co in incoming feed was analysed. Combined with measured flowrate the mass of Co to be precipitated was determined. Then a predetermined NaHS to total metals (TM) stoichiometric ratio controls the addition of sulphide reagent. The current ratio is set very close to 1:1 and minimal excess sulphide reports to BL. Very high Co recovery is achieved. pH is exclusively controlled by dilute NaOH addition. This control strategy is particularly suited to Bulong since there is ample upstream storage of strip liquor and the concentration of Co in strip liquor does not change greatly.

11.3 Cobalt Sulphide Precipitation – Metallurgical Performance

Since the changes in reagent addition configuration were made in December 2001 and more recent changes to feed-forward sulphide addition control (~ June 2002) the performance of the sulphide circuit has dramatically improved.

Soluble cobalt levels before and after changes in CoS precipitation are shown below.

Cobalt in Solution - ppm			
Jan 01 - Nov 01		***Dec -01 - Mar 03***	
Mean	259	Mean	91
Median	85	Median	29
Std Dev	568	Std Dev	246

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Solids losses (due generally to fine CoS particles being lost with solution or due to holed filter cloths) are shown below.

"Safety Tank" TSS - CoS mg/L			
Jan 01 - Nov 01		*Dec -01 - Mar 03*	
Mean	1649	Mean	267
Median	190	Median	40
Std Dev	5611	Std Dev	1714

These improvements in both soluble Co and solids losses have translated into significantly improved cobalt recovery.

12 LEACH RESIDUE STORAGE FACILITY (LRSF)

12.1 General

Since commissioning, the disposal of leach residue has been to a conventional earth dam constructed "paddock style", common to the WA goldfields. Recovered decant water from the LRSF is then sent to nearby evaporation ponds. The climate of the WA goldfields allows the evaporation of vast amounts of water. Only remnant cyclones from northern Australia can substantially reverse the water balance and this is of course only temporary. The existing LRSF is approximately 6 km from the plant.

Leach residue and gypsum solids are neutralised to a pH of 7 using dilute slaked lime slurry prior to pumping to the LRSF.

12.2 Recent Innovation – In-Pit Leach Residue Storage Facility

In Q2 2002 Bulong was faced with an expensive lift of the LRSF earth wall, however, the option of in-pit leach residue storage was examined and found to be the preferred means of leach residue storage. Several pits had been previously exhausted with the closest being the Criterion Pit, less than 2 km from the plant. After gaining approval from relevant State authorities installation of facilities began. These facilities consisted of:

1. Diversion of existing leach residue disposal pipeline to the Criterion Pit. Several valved "off-takes" were installed. However, once running, only one discharge point was used.

2. The lease (and eventual purchase) of a return or decant water pump and floating pontoon. The pump was designed to return up to 300 m^3/h of decant water.

3. Extension of the existing overhead electrical power system for the pontoon pump.

4. Pipework to allow decant water connection into the existing LRSF line out to the evaporation ponds.

5. Re-use of the old LRSF line.

6. Installation and use of anti-scalant in the decant water line.

7. Installation of several monitoring bores to allow regular sampling of surrounding ground water to detect any ingress.

It was initially predicted that the Criterion pit would take about nine months of plant residue.

12.3 Performance of Criterion In-Pit LRSF

Criterion Pit operated for approximately seven months only partially filling the pit. Leach residue was then diverted back to the old LRSF to complete the filling of that facility.

The operation of Criterion went smoothly with solids settling as anticipated. The pontoon pump and associated decant facilities operated without fault and the anti-scalant addition was successful.

Criterion Pit has about three months remaining storage capacity and leach residue will be diverted back to the pit in May/June 2003 to complete filling.

12.4 Federal In-Pit LRSF

Following the success of the Criterion In-Pit LRSF it was decided to pursue the option of using the old Federal Pit for future leach residue disposal. This pit is significantly larger than Criterion and will store approximately 2.5 years of leach residue. The Federal Pit is approximately 6 km from the plant site

Monitoring bores have been drilled and a new leach residue pipeline has been installed. The pontoon pump has been purchased and will be relocated to Federal once Criterion is full.

12.5 Future LRSF

There are a number of mined-out pits close to the plant and each pit is potentially capable of being used for In-Pit LRSF. Each pit will be assessed on its own merits in terms of environmental and cost considerations. At some point in the future it is conceivable that a lift on the original LRSF may be more cost effective.

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From an environmental perspective filling old pits with neutralised leach residue seems more attractive than building larger and larger earth wall paddock style storage facilities. The nature of laterite mining and processing means that a large number of near surface pits will be generated and it seems logical that these pits be progressively filled during the life of the project.

The Criterion In-Pit LRSF is probably the first application of re-filling old mined-out areas with leach residue generated from a laterite HPAL facility. This application is not only more environmentally acceptable but more cost effective and may allow for a considerable reduction in up-front capital and operating costs for future laterite projects.

13 UTILITIES

13.1 Process Water

No change from installed equipment.

13.2 Potable Water

The demand for potable water on-site has increased recently and is due almost entirely to the increased leach throughput and the increased demand for steam. Various alternatives for supply of water are being pursued. On-site conservation of valuable potable water is also being actively pursued.

13.3 Power

No change from installed equipment

14 REAGENTS

14.1 Sulphuric Acid

Recently BOPL was informed by WMC Resources that supply of acid from the nearby Kalgoorlie Nickel Smelter (KNS) would be reduced from the end of 2003. Additional acid is required for operations at the Mt Keith Nickel Mine.

BOPL is currently investigating alternative supplies of acid and is confident that a cost effective supply of additional acid will be found.

From a technical perspective, the supply of acid to the plant has been dominated by one single issue – High Pressure (HP) Acid Pumps.

While the pumps themselves are of sound design and construction, spillage and subsequent ingress of acid under the concrete bund has led to movement of pumps and generated stress on connected pipework. The pumps appear to have been installed on foundations containing swelling clays and carbonates.

Pipework has been de-stressed several times and it is planned to relocate the pumps in Q3 or Q4 2003.

14.2 Limestone

Crushed limestone from Loongana is used for leach discharge acid neutralisation at Bulong.

Dry limestone is milled in a conventional ball mill in closed circuit with cyclones. Cyclone O/F reports directly to a limestone slurry storage tank. The milling circuit is oversized and operates for only 10-14 hours/day.

Limestone is ground to 90%<45 micron and a slurry with a density of between 25 and 40% solids is produced.

Due to the inherent design of the limestone milling circuit a widely varying size distribution of limestone is produced. The cyclones are operated at a high cyclone feed density to get good O/F densities. This in turn leads to poor classification. This design is typical of that used within the WA goldfields for oxide gold ores, where the size distribution or ore does not greatly affect gold extraction. In hindsight, it was not appropriate to extrapolate this approach to the preparation of a major reagent for a nickel process.

In the neutralisation process at Bulong there is some residual slow reaction of coarse limestone. This creates bubbling and poor settling within thickeners and leads to a delayed pH response. Any delayed reaction of even small quantities of coarse limestone around a pH ~4.5 to 5.0 can affect the pH, making control of pH more erratic. Limestone is a major reagent which is not being correctly prepared for the neutralisation and impurity removal process.

To improve the quality of limestone from a size distribution perspective a thickener is required. The cyclones could then be operated as classifiers and the thickener used to remove excess water.

The limestone milling circuit also produces a variable slurry solids density. This translates into a variable flowrate of limestone slurry to neutralise the same mass rate of acid. This causes flow surging within the neutralisation circuits. The recently installed slurry neutralisation circuit is particularly sensitive to limestone solids density since the pH control

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loop originally envisaged for limestone addition has not yet been fully implemented.

Considerable thought must be given to the limestone preparation circuits of any future laterite plant. Classification and thickening must be considered separately to achieve a consistent grind size and solids density.

15 CONCLUSIONS

Apart from the formation of excessive gypsum scale in Ni SX, the original process flowsheet essentially performs as per the design intent.

The Bulong flowsheet is a commercially viable option for the winning of nickel and cobalt.

Additional process enhancements as described in this paper have directly improved the financial viability of BOPL and of the direct solvent extraction (DSX) process in general. Many additional enhancements are also potentially viable.

Capital cost savings imposed during EPCM and construction resulted in many parts of the plant being "undersized" from an equipment perspective. This was particularly evident in neutralisation, but occurred across the entire process plant. In spite of this and with very limited funds, Bulong personnel at all levels of the organisation have persisted and have made changes and improvements in process configuration/operation that have overcome many of the inherent mechanical deficiencies.

Direct solvent extraction DSX of metals from HPAL laterite leach liquors does have a role to play in the future winning and nickel and cobalt. However further enhancements and refinements that have been identified by Bulong personnel and others must continue to be explored and developed to ensure the long-term and low cost supply of these metals to the world.

Acknowledgements

I would like to acknowledge the support and assistance of all BOPL personnel in the compilation of this paper. However, special mention must be made of Dale Oram – Mining Manager and Simon Donegan/Paul Gilman – Metallurgists for their assistance with data compilation.

I would like to thank the management of Bulong Operations Pty Ltd for their permission to publish this paper.

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FLOWSHEETS AND FIGURES

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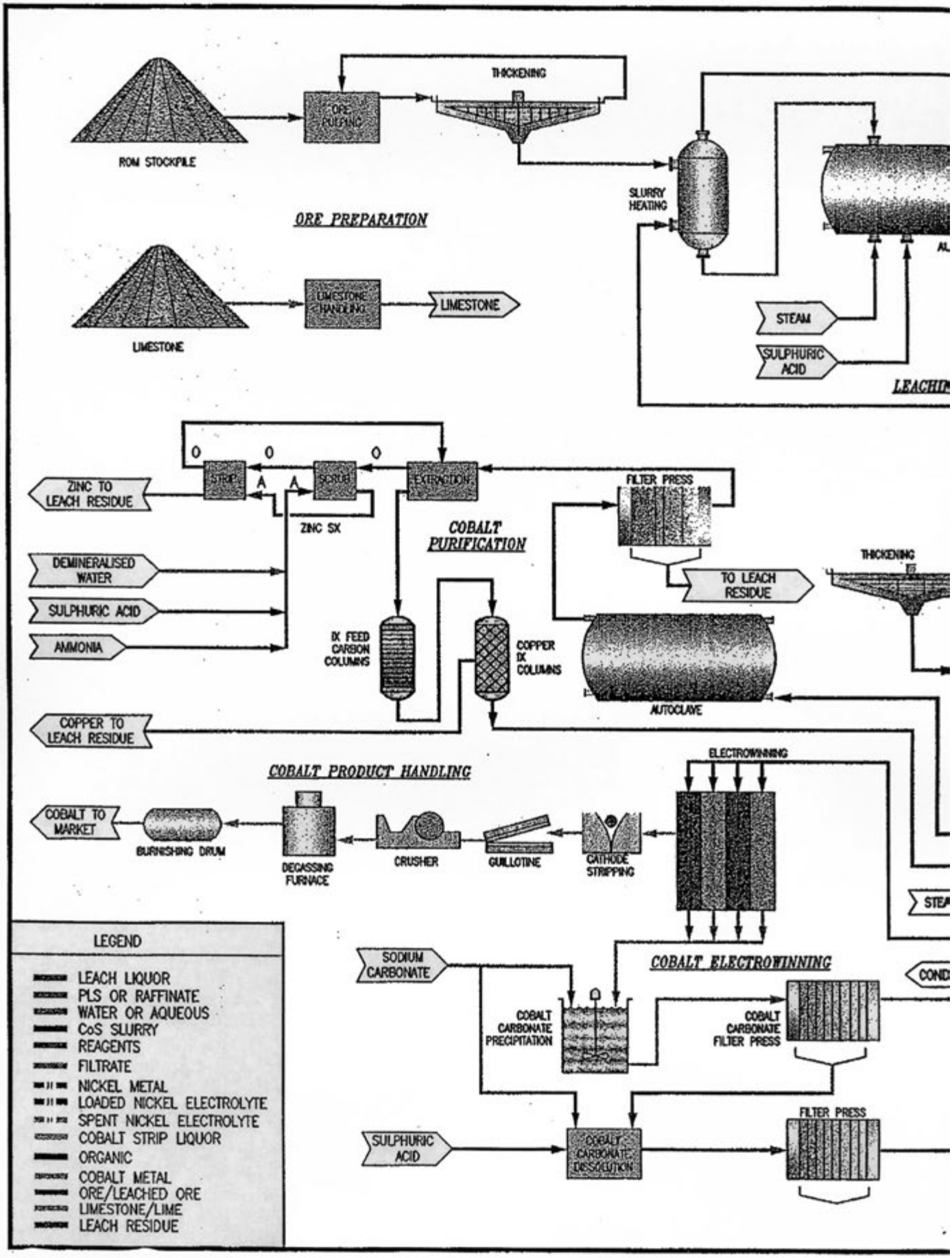
Flowsheet 1 : Overall Flowsheet of Bulong Operations Pty Ltd



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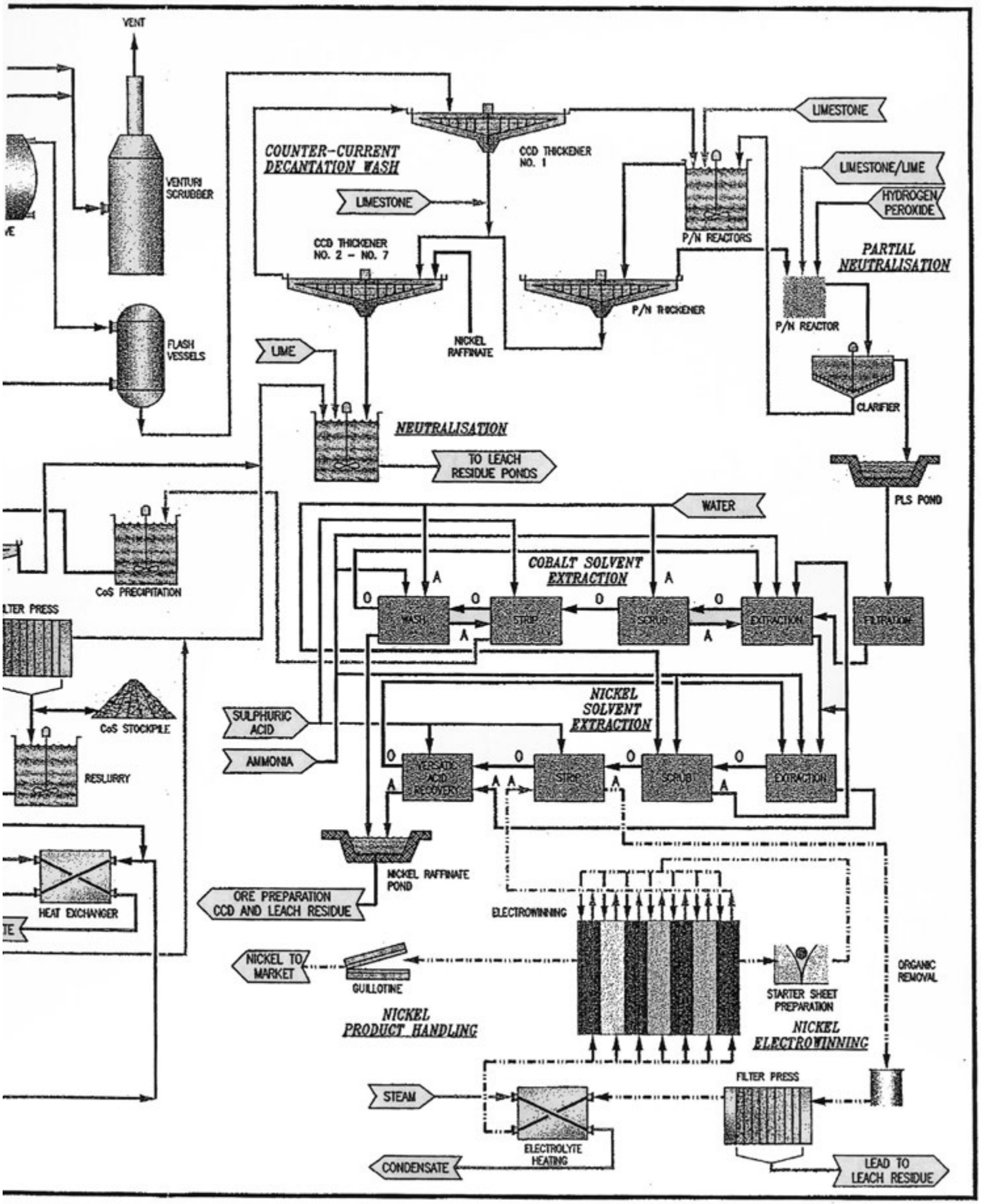

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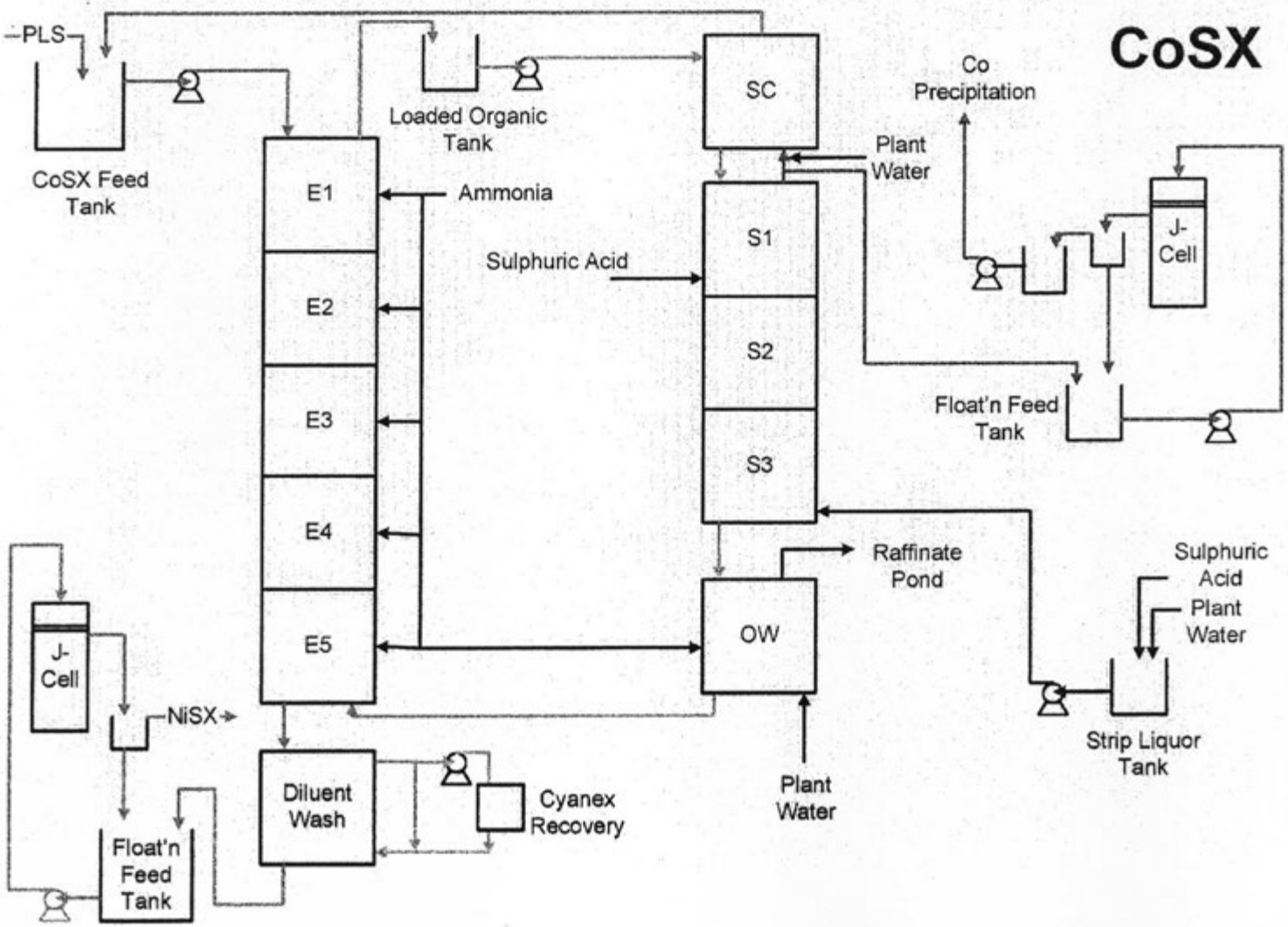
Flowsheet 2 : Cobalt Solvent Extraction at Bulong Operations Pty Ltd



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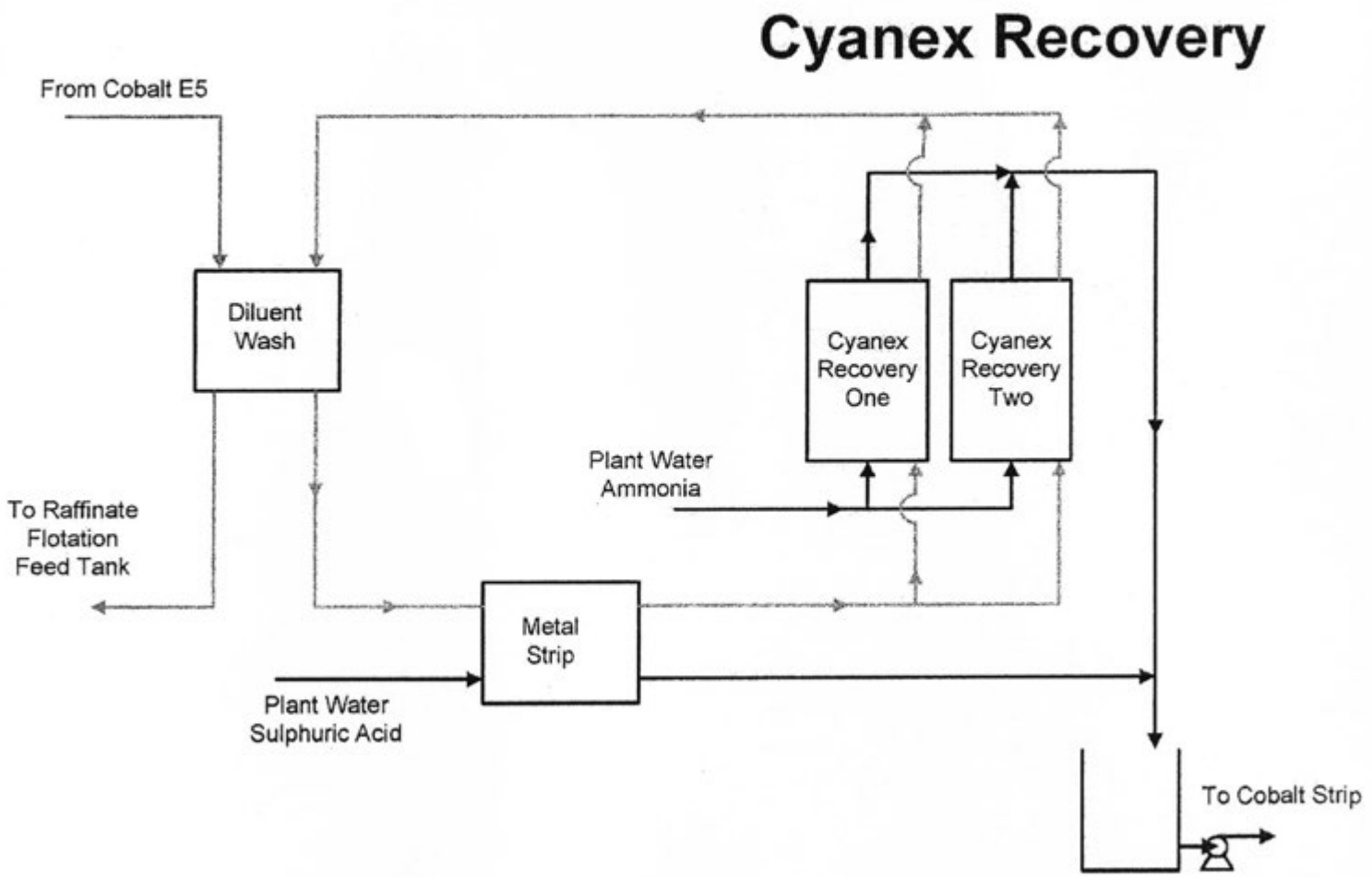
Flowsheet 3 : Cyanex Recovery at Bulong Operations Pty Ltd



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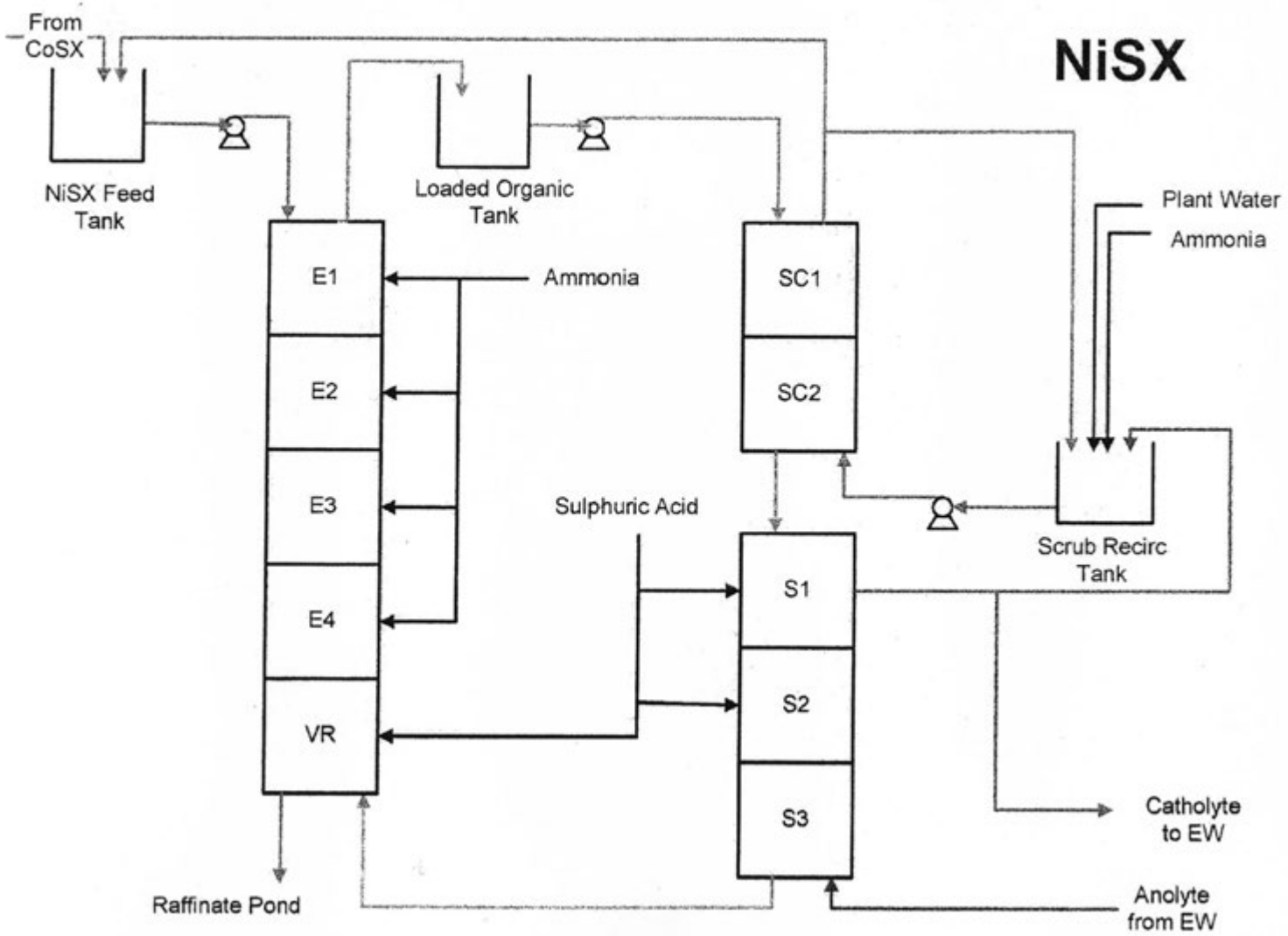
Flowsheet 4 : Nickel Solvent Extraction at Bulong Operations Pty Ltd



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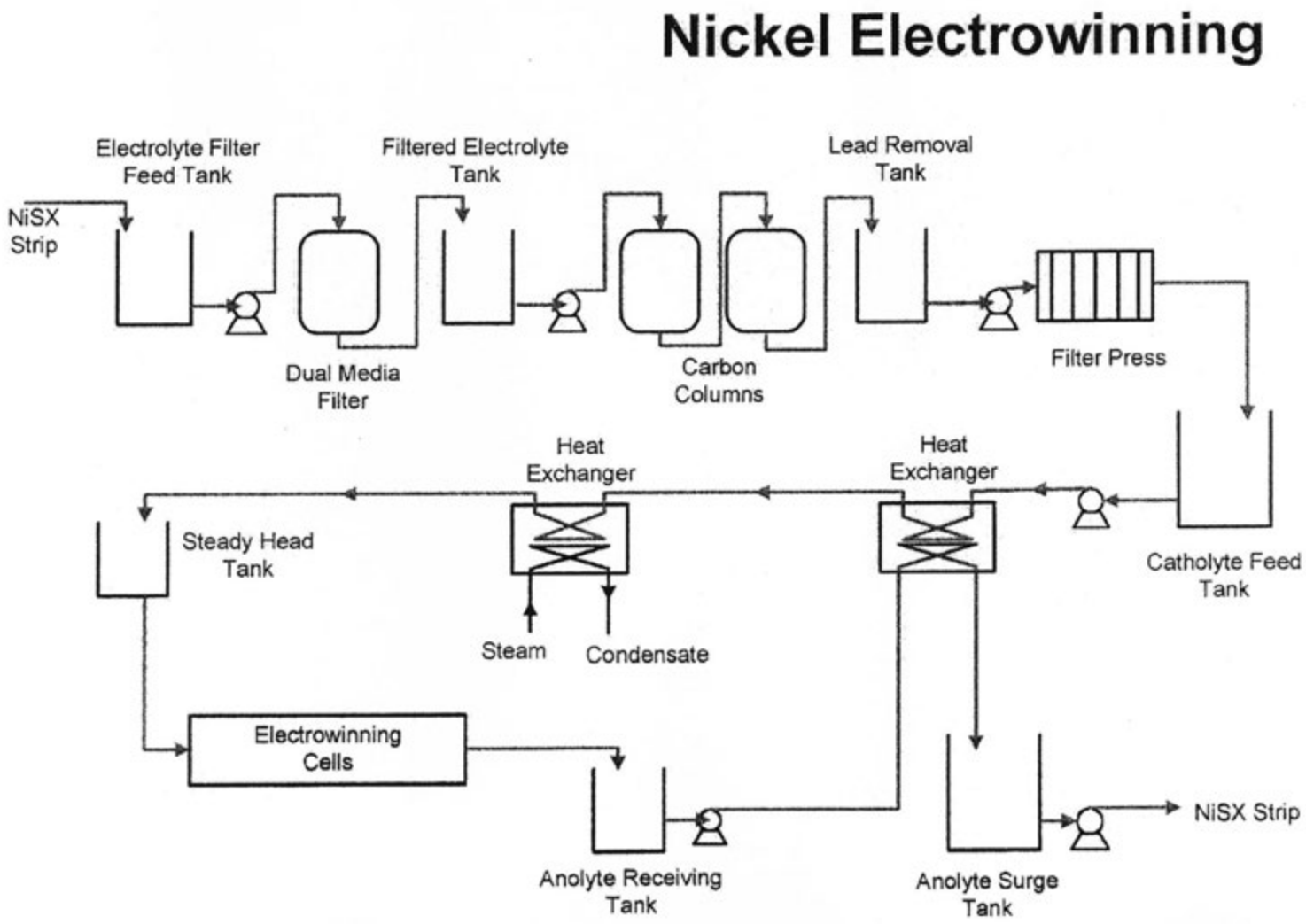
Flowsheet 5 : Nickel Electrowinning at Bulong Operations Pty Ltd



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Flowsheet 6 : Cobalt Strip Circuit at Bulong Operations Pty Ltd

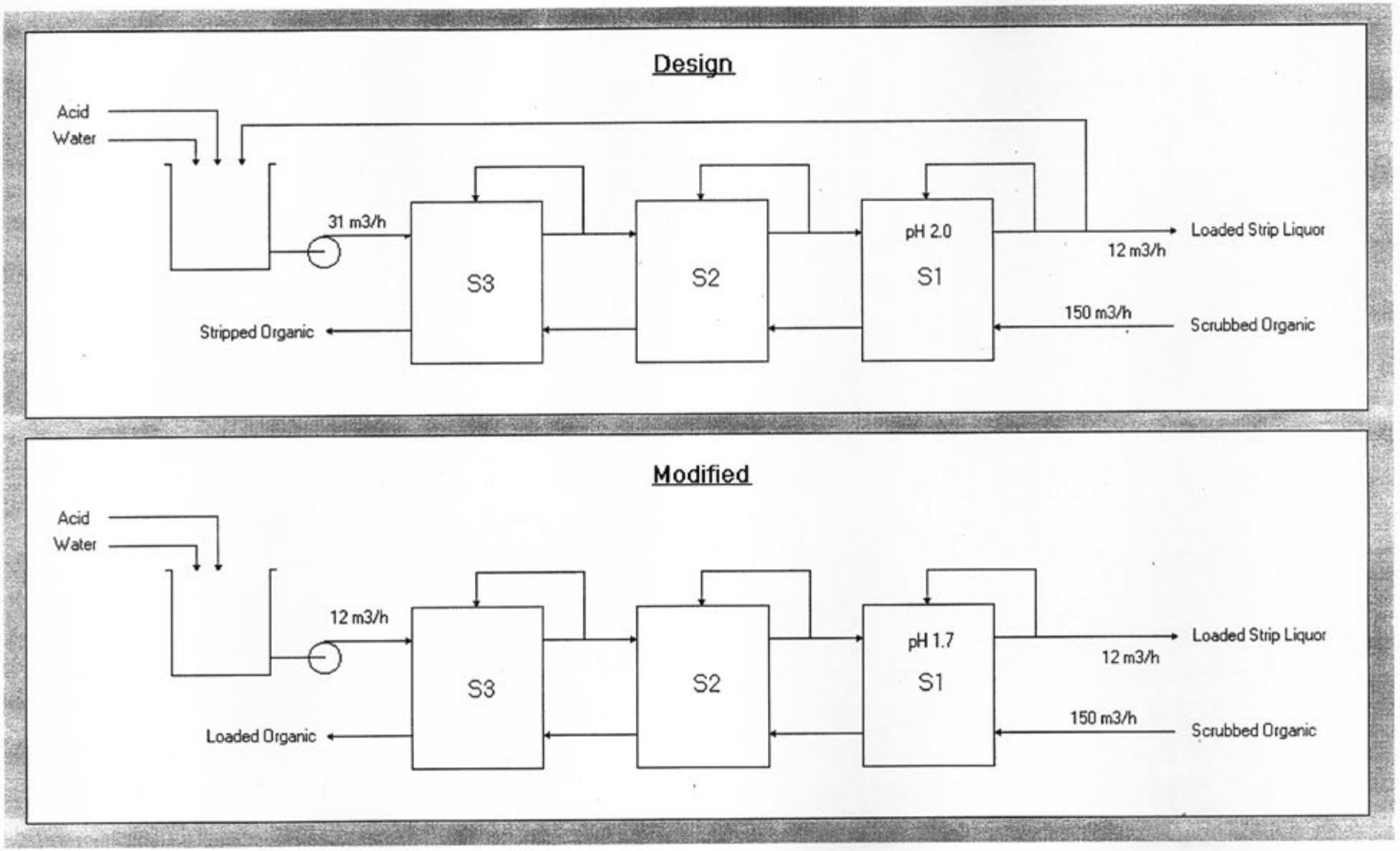

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Figure 3.1 Bulong Operations Pty Ltd Ore Preparation Plant - Mass Rejection

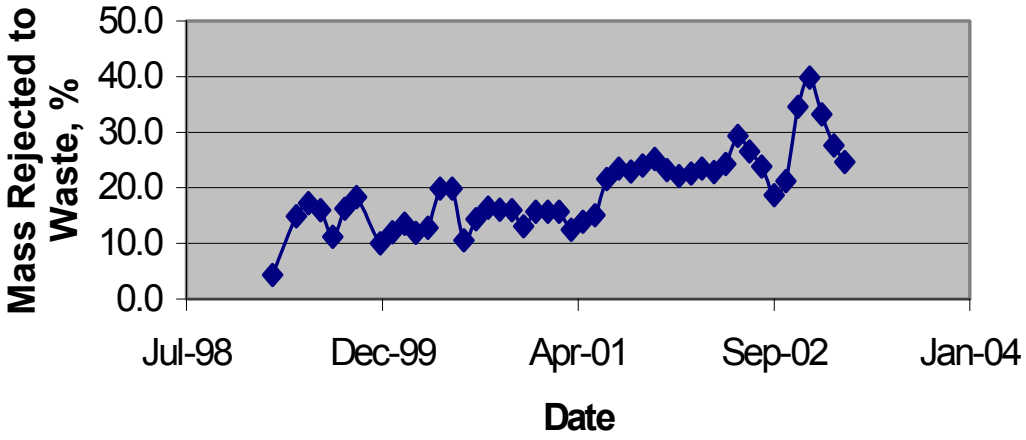

Figure 3.2 Bulong Operations Pty Ltd Ore Preparation Plant Upgrade Factor for Nickel, %

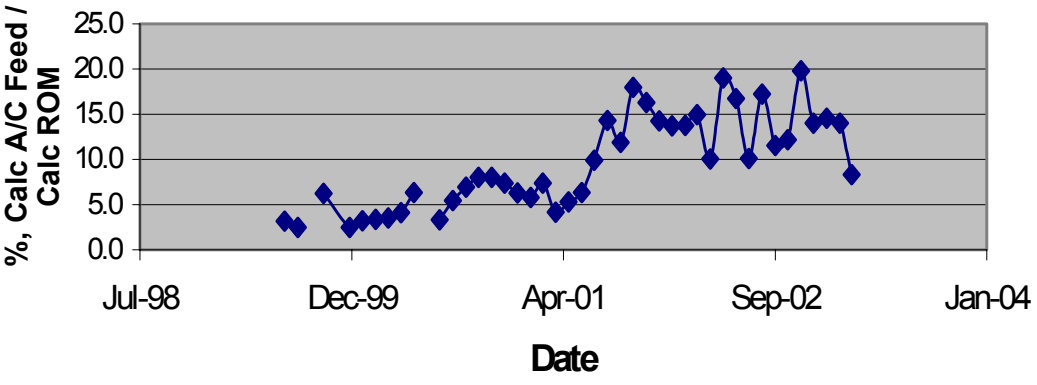

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Figure 3.3 Bulong Operations Pty Ltd Ore Preparation Plant Nickel Recovery, %

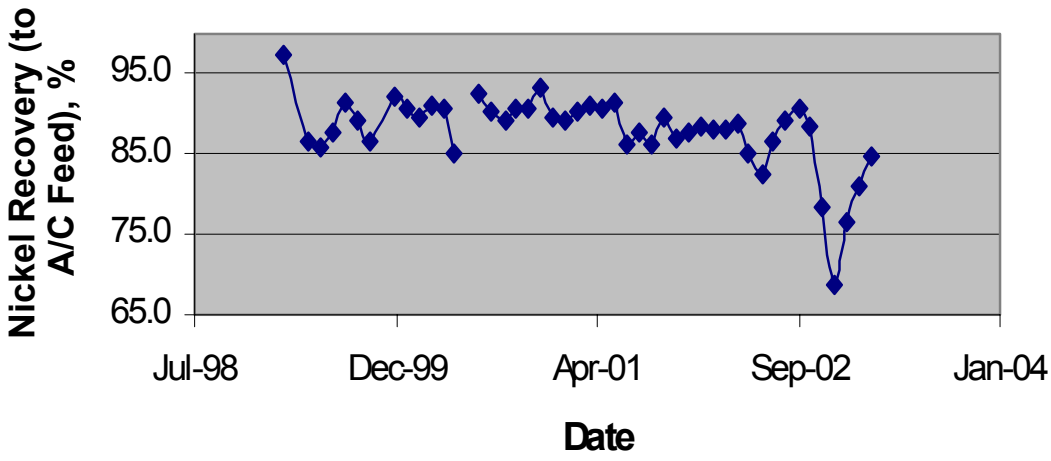

Figure 3.4 Bulong Operations Pty Ltd Ore Preparation Plant Upgrade Factor for Cobalt, %

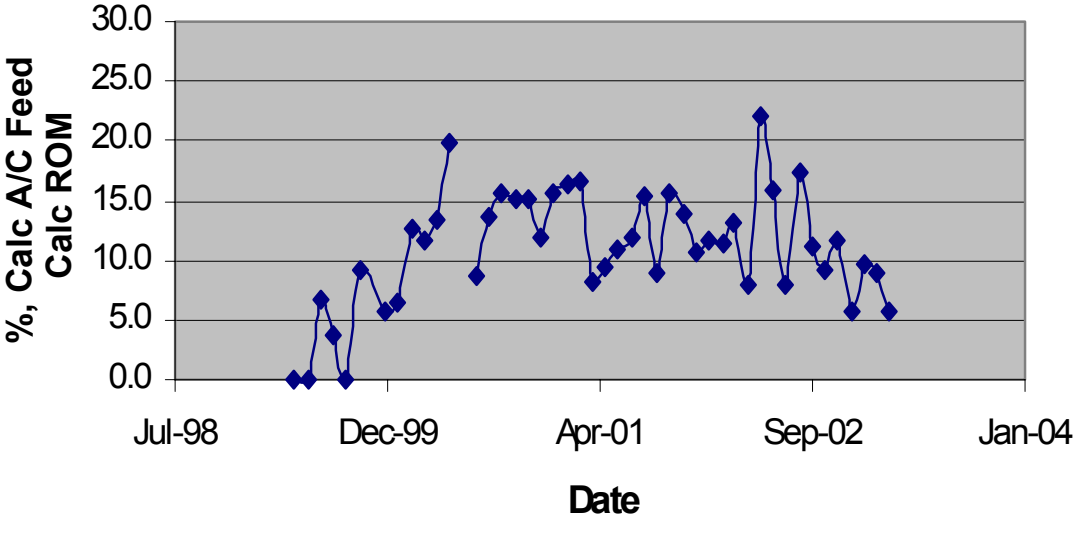

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Figure 3.5 Bulong Operations Pty Ltd
Ore Preparation Plant Cobalt Recovery, %

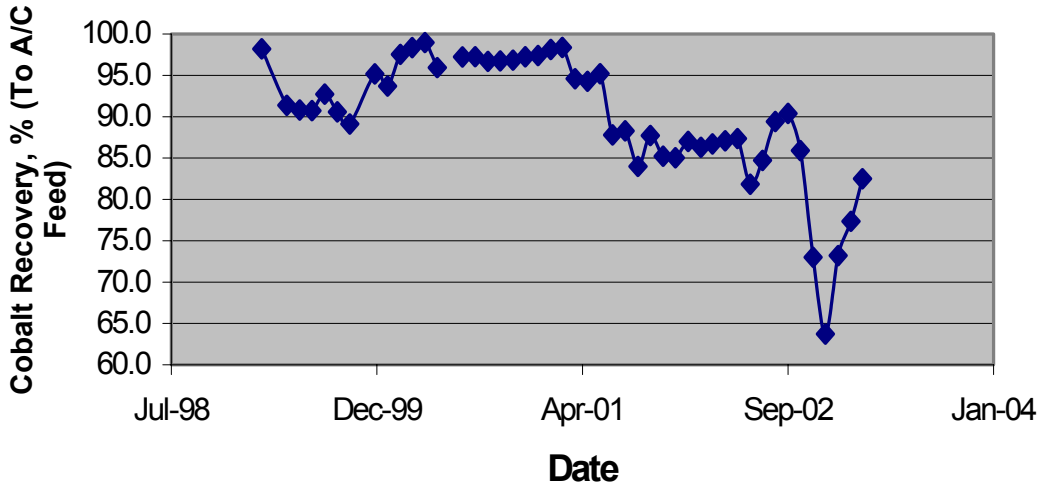

Figure 3.6 Bulong Operations Pty Ltd
Leach Feed Thickener U/F Density, % w/w

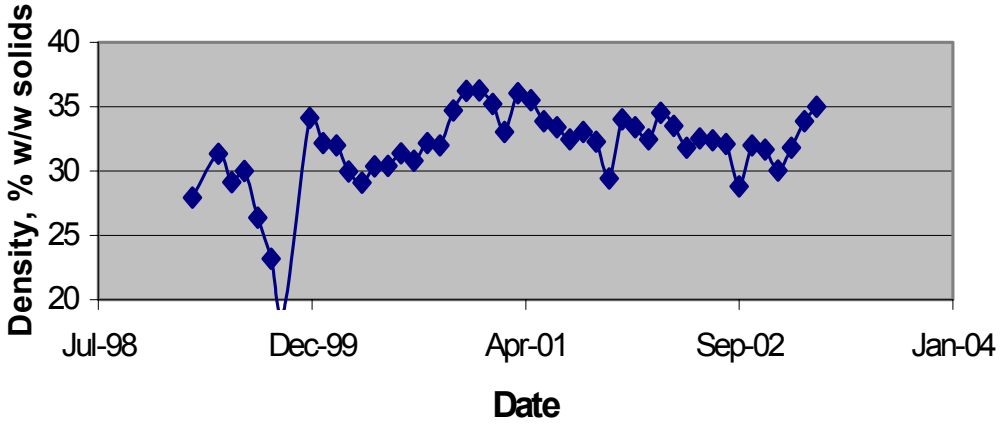

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Figure 3.7 - Bulong Operations Pty Ltd
Ore Preparation Plant
HPAL Feed Ni, % w/w Frequency Distribution
no Pre-blending

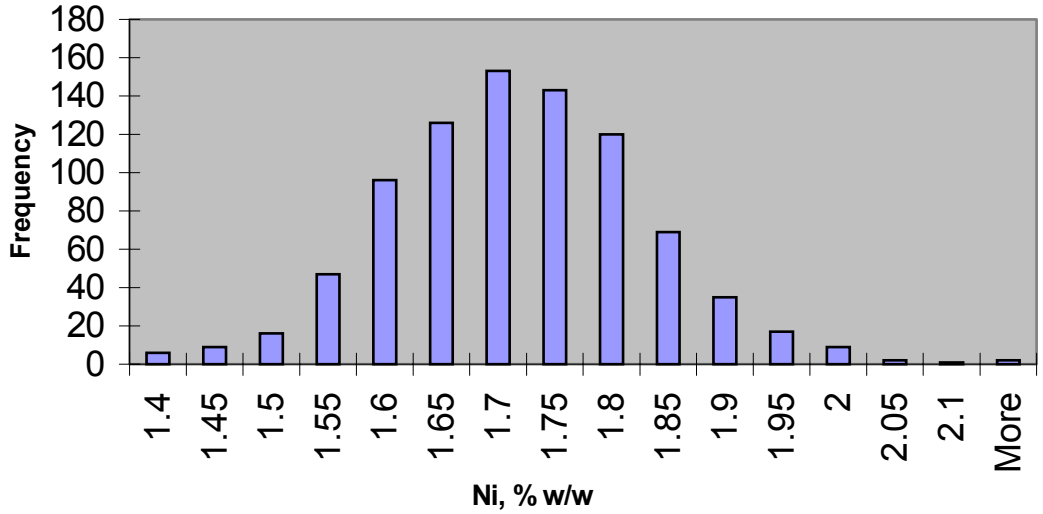

Figure 3.8 - Bulong Operations Pty Ltd
Ore Preparation Plant
HPAL Feed Ni, % w/w Frequency Distribution
post Pre-blending

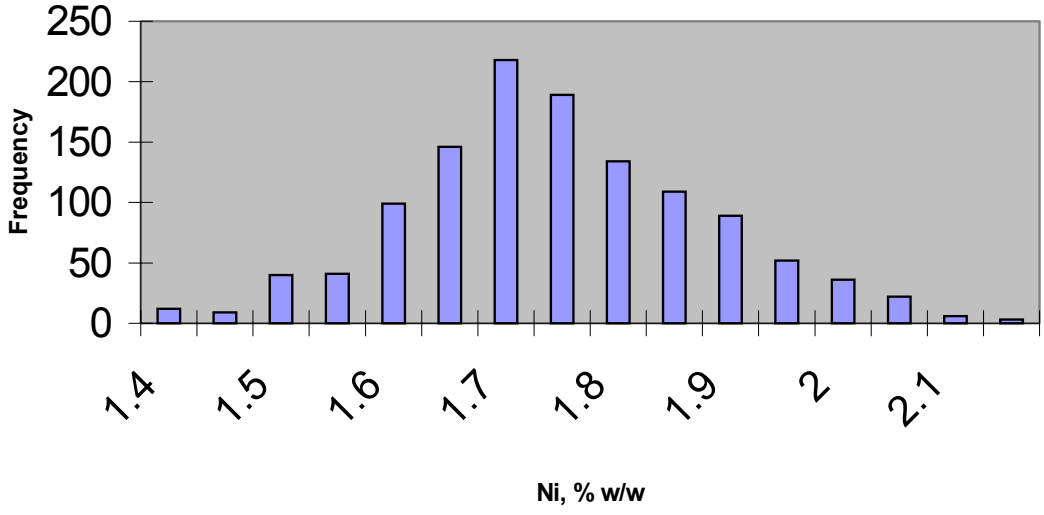

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Figure 3.9 - Bulong Operations Pty Ltd
Ore Preparation Plant
HPAL Feed Slurry Density, % w/w Frequency Distribution
no Pre-blending

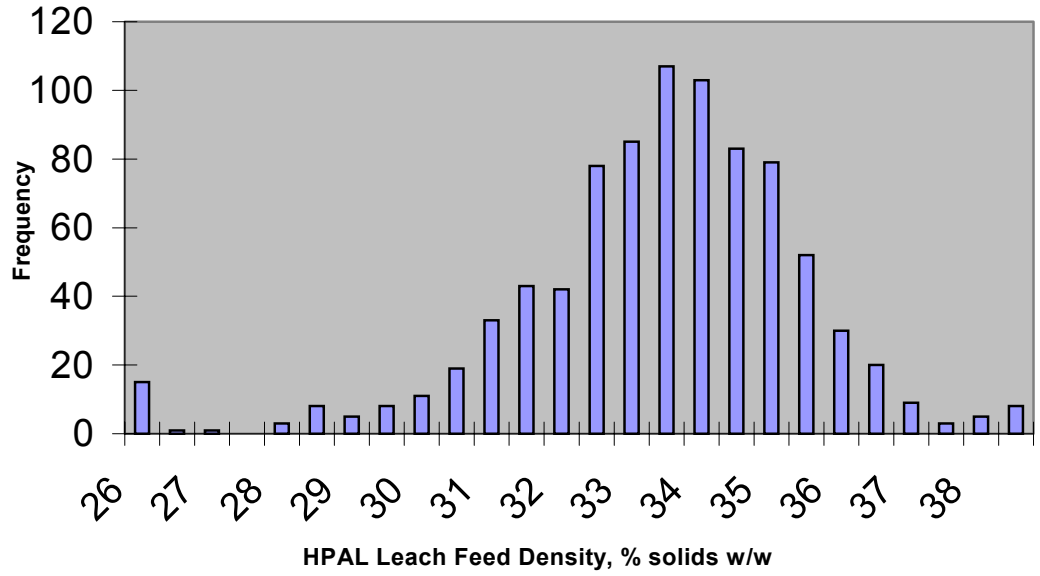
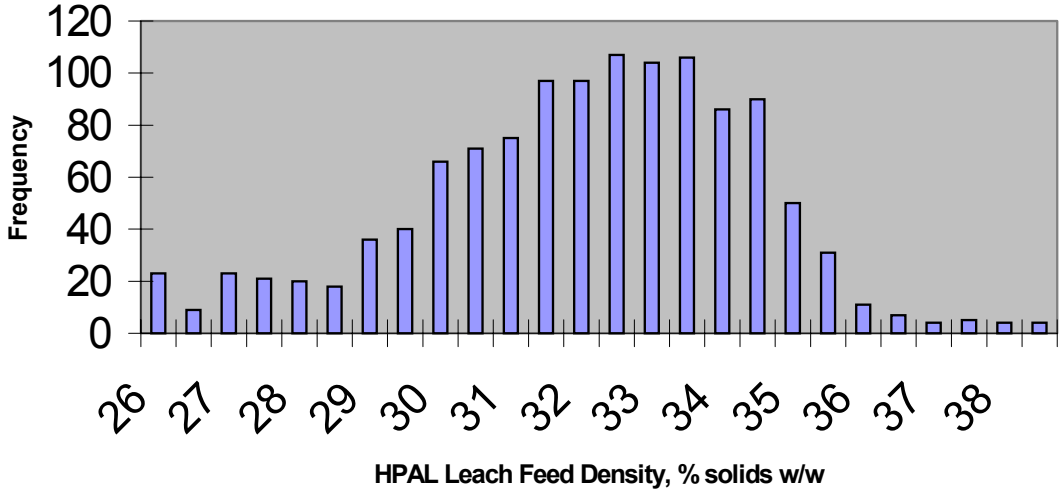


Figure 3.10 - Bulong Operations Pty Ltd
Ore Preparation Plant
HPAL Feed Slurry Density, % w/w Frequency Distribution
post Pre-blending



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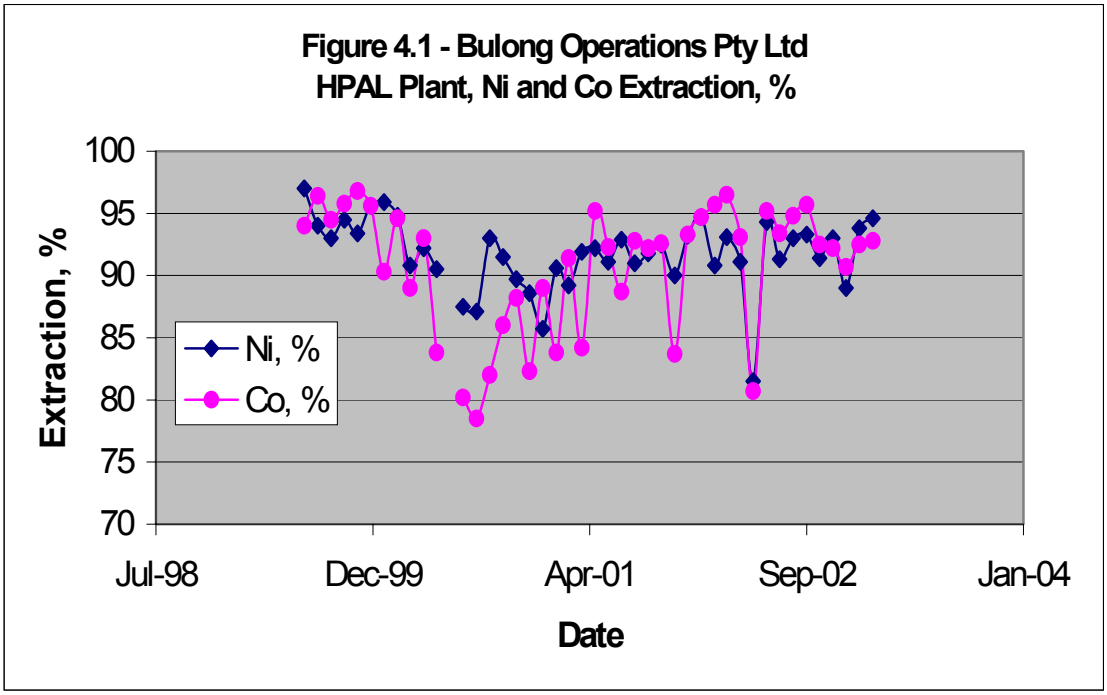

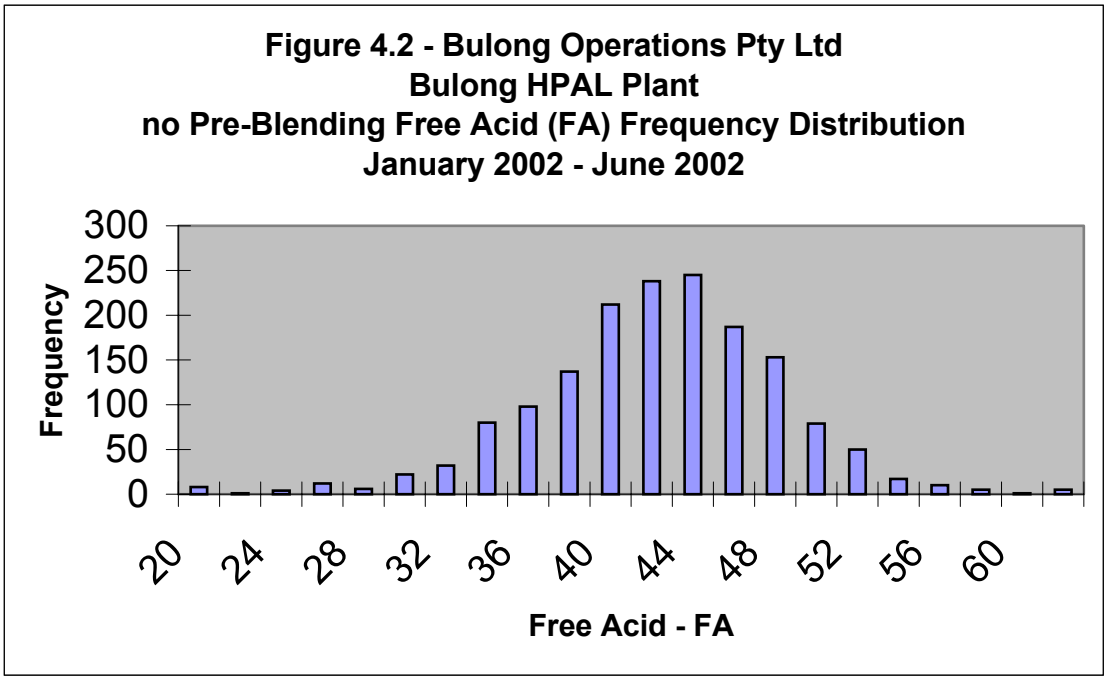

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Figure 4.3 - Bulong Operations Pty Ltd
Bulong HPAL Plant
post Pre-Blending Free Acid (FA) Frequency Distribution
July 2002 - February 2003

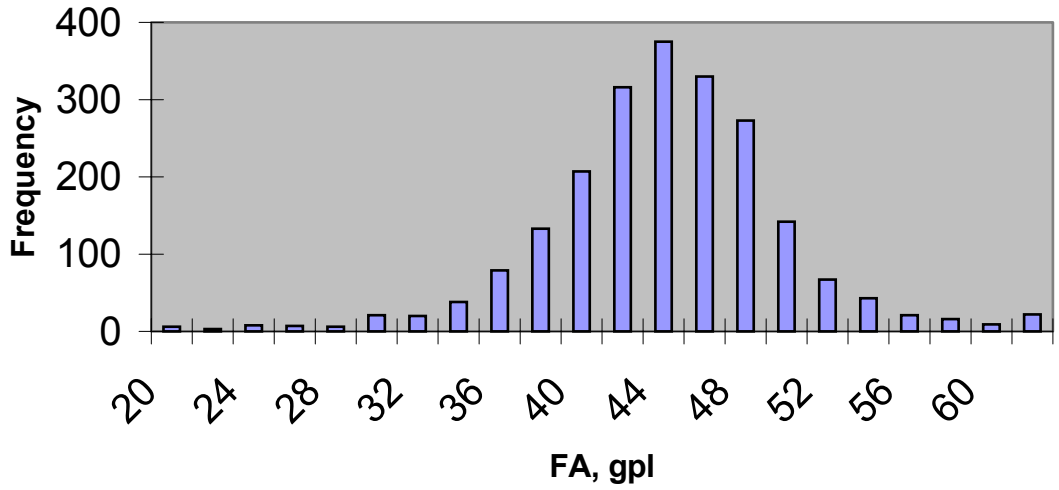
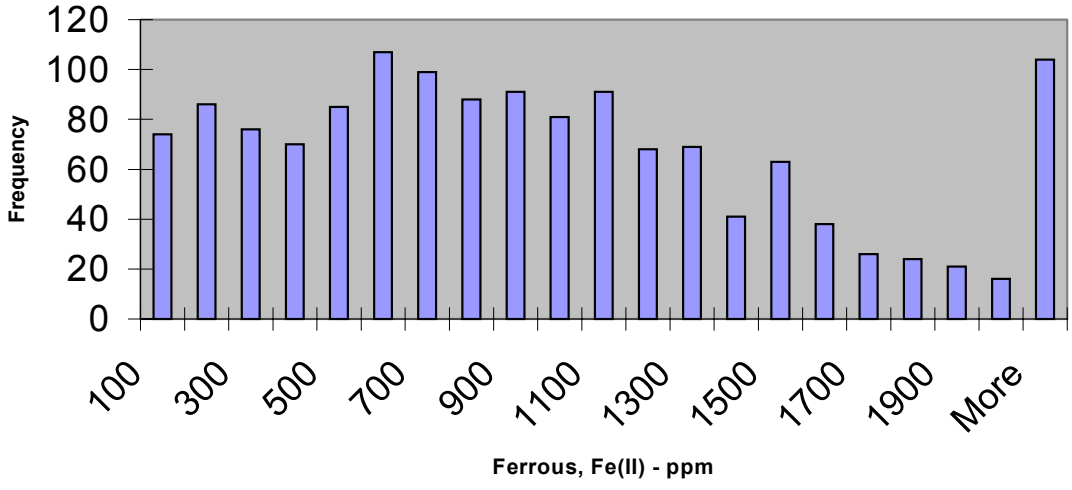


Figure 4.4 - Bulong Operations Pty Ltd
Bulong HPAL Plant
no Pre-Blending Frequency Distribution
Ferrous - Fe(II) in HPAL Discharge



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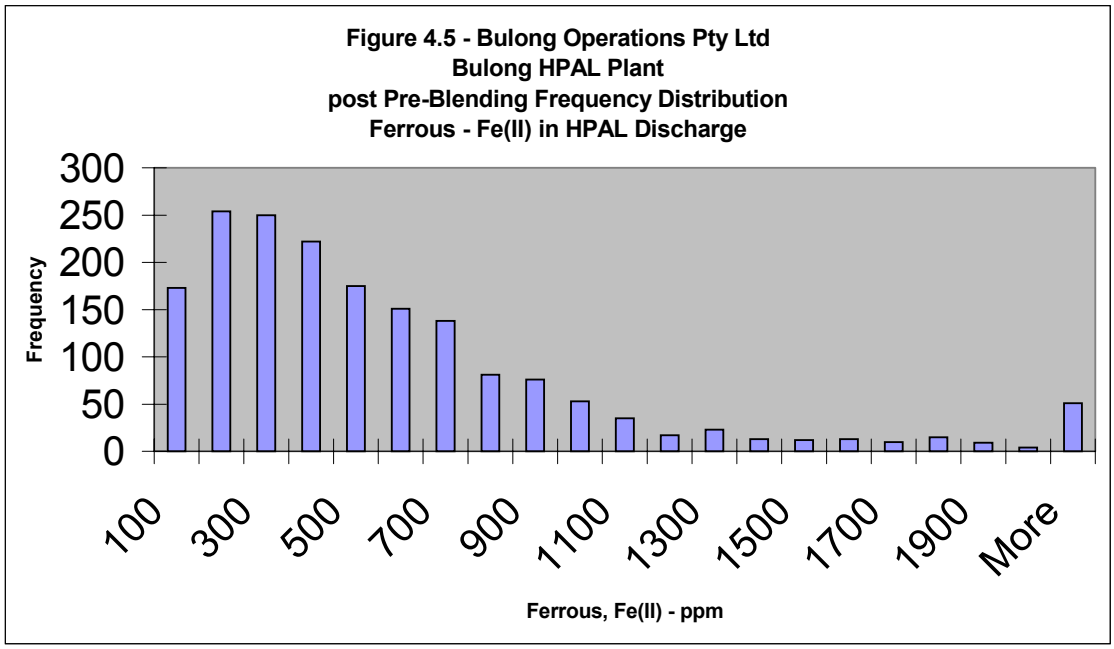

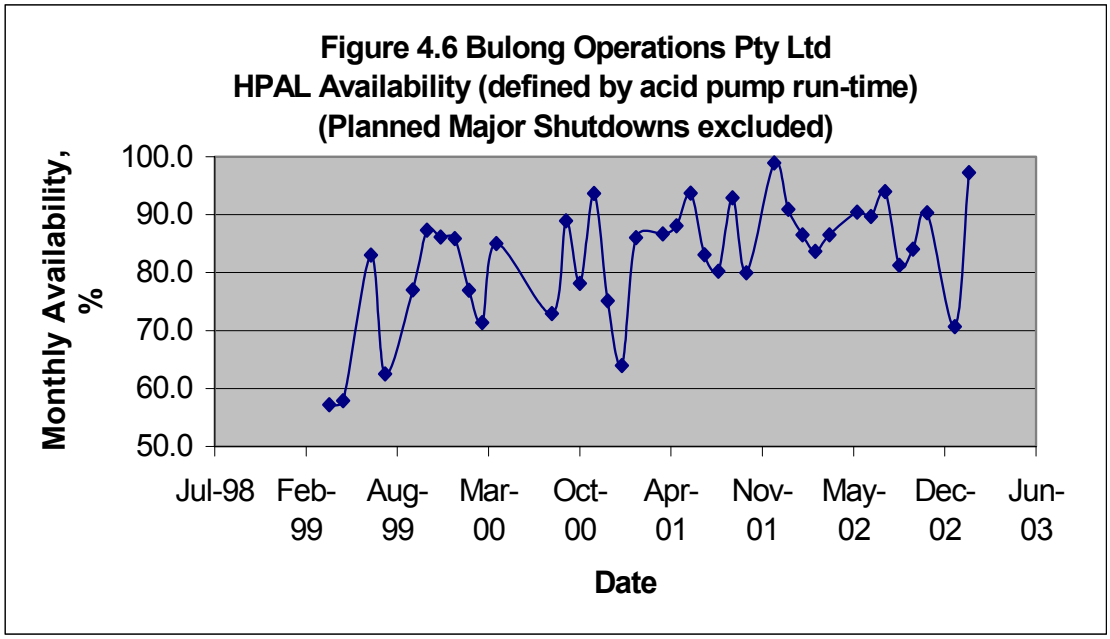

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Figure 4.7 Bulong Operations Pty Ltd
HPAL Acid/Ore Ratio

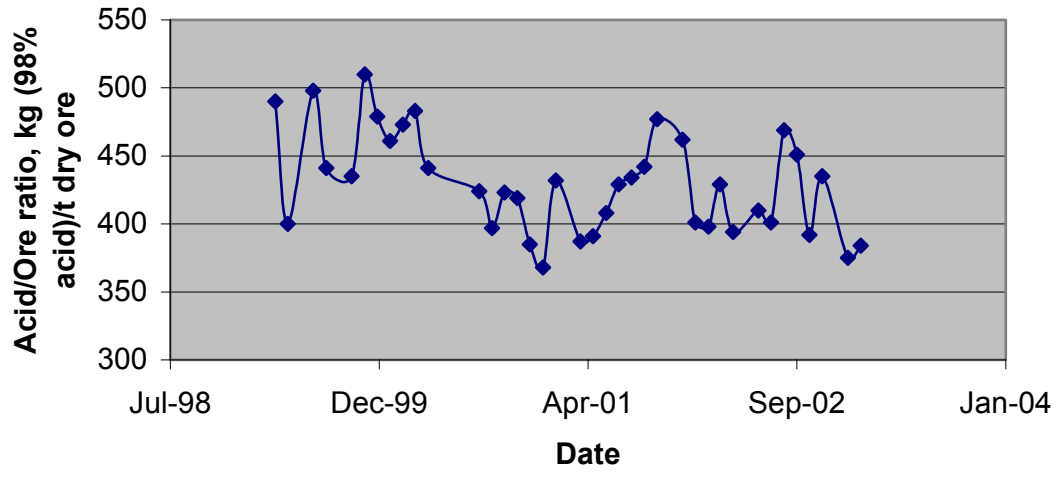

Figure 4.8 Bulong Operations Pty Ltd
HPAL Steam/Ore Ratio

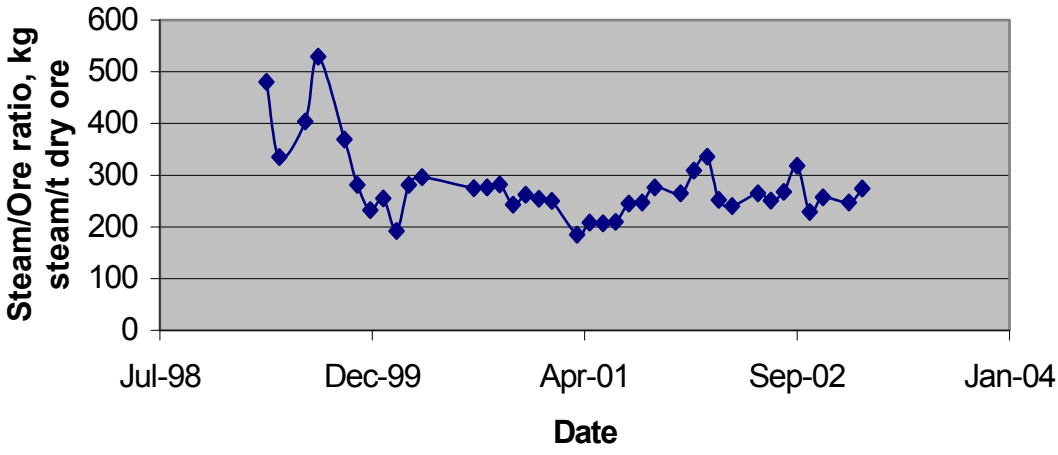

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Figure 7.1 Bulong Operations Pty Ltd
Neutralisation & CCD Plant
Ni Recovery %

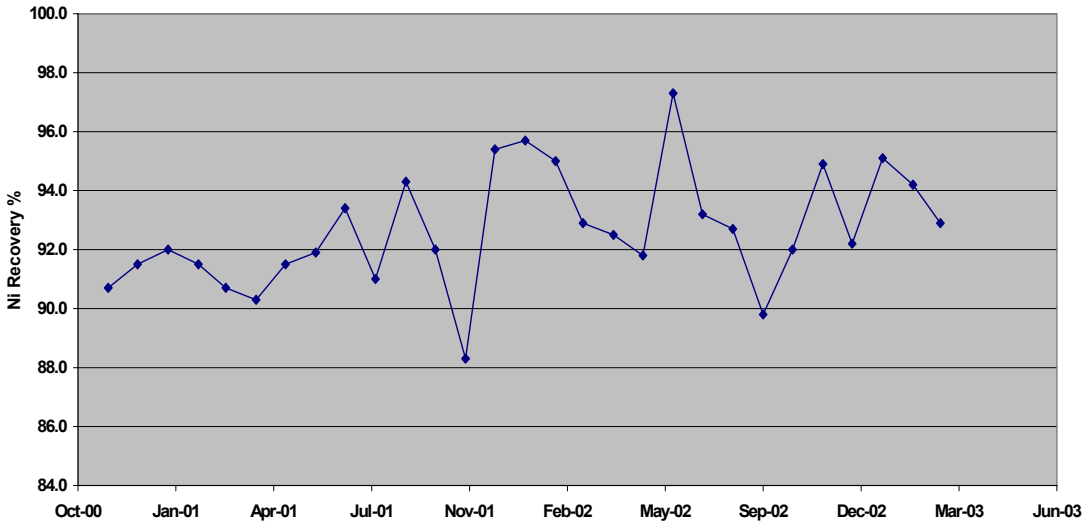

Figure 8.1
Bulong Operations Pty Ltd
Co SX Flowrate, m^3/h (weekly data)

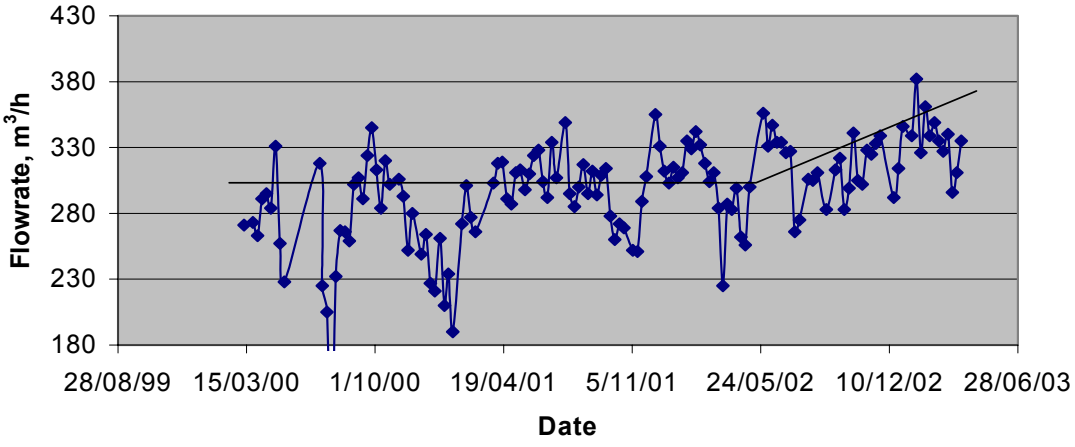

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Figure 8.2 Bulong Operations Pty Ltd
Ni Cathode Quality since July 2001
Co ppm (LME<1500)

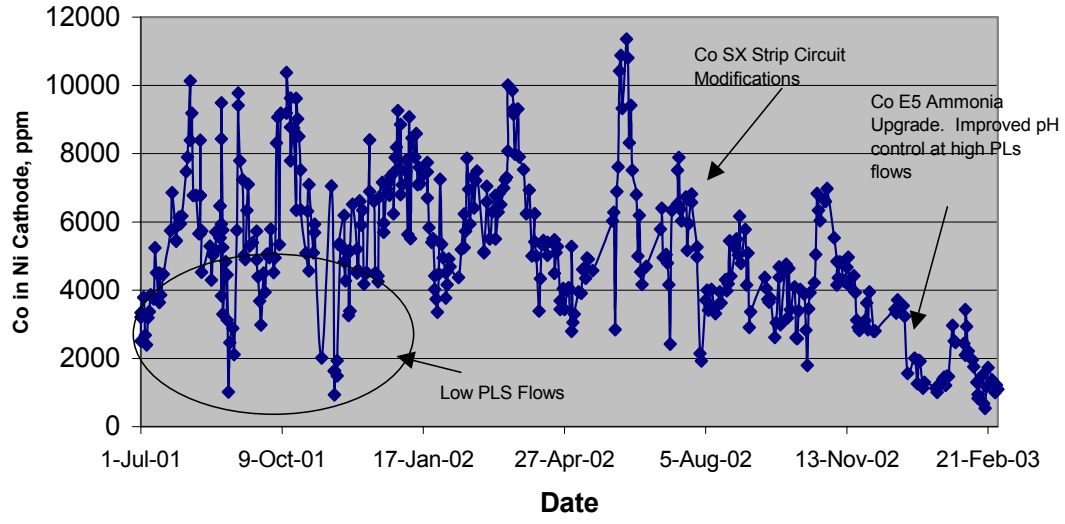
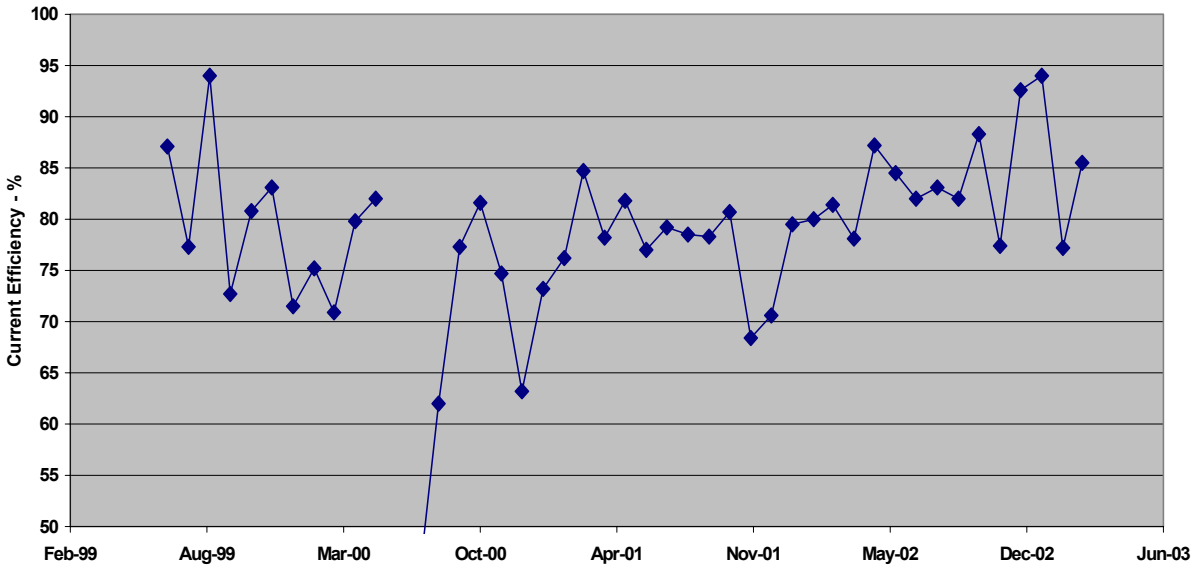


Figure 10.1 Bulong Operations Pty Ltd
Nickel Electrowinning Plant
Current Efficiency, %



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ALTA 2003

NICKEL /COBALT - 9

SERVING THE GLOBAL MINING & METALLURGICAL INDUSTRIES WITH TECHNICAL EXCELLENCE & PROVEN INTEGRITY

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CHALLENGES ARISING FROM THE INTEGRATED OPERATION OF DUAL AUTOCLAVE PAL/MSP PILOT CAMPAIGNS AT SGS LAKEFIELD ORETEST

By

Evan Matthews, Mark Benson, Dwight van der Meulen, John Turner and Jeff Robinson
Lakefield Oretest Pty Ltd

Presented By

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ABSTRACT

Since commissioning of the high pressure acid leaching (PAL) pilot plant was completed in early 2001, a number of PAL piloting campaigns have been successfully completed integrating several downstream flow sheet options including mixed hydroxide and mixed sulphide intermediate products (MHP and MSP).

With the recent addition of a 35 litre, six-compartment titanium autoclave - commissioned during March 2003 for the precipitation of a MSP - a number of materials, design and operational issues were overcome to ensure the successful operation of future integrated piloting campaigns.

This paper will cover these challenges including lessons learnt from batch optimisation and pilot commissioning MSP test work carried out at SGS Lakefield Oretest.

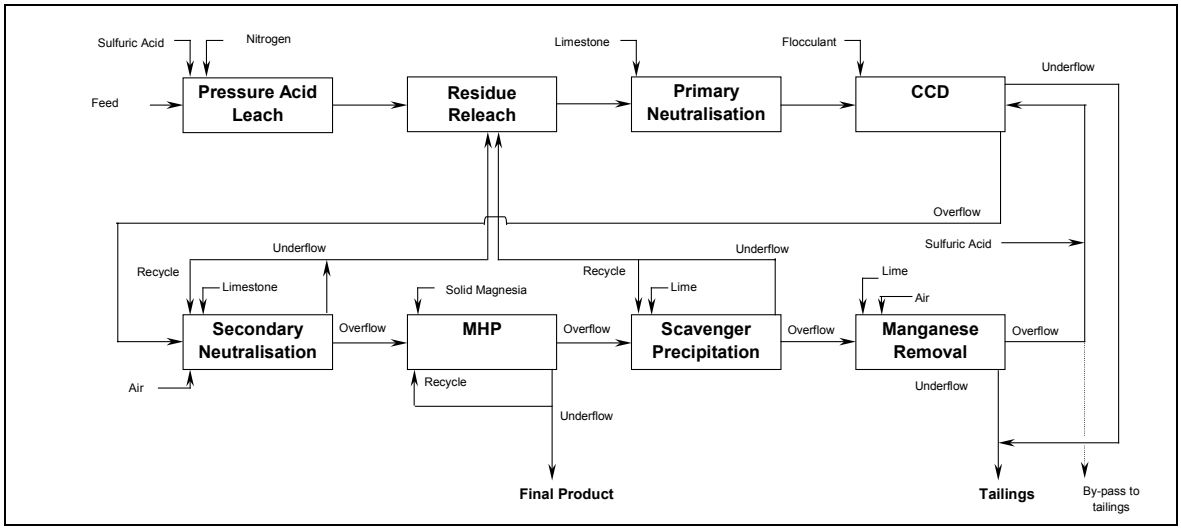
1. INTRODUCTION

The PAL process for all Nickel Laterites consists of extracting nickel and cobalt values into an acidic solution along with a number of unwanted impurities.

Further downstream processing can follow several alternative routes recovering both nickel and cobalt, while rejecting these unwanted impurities. Two of the more popular processing routes follow the production of either a mixed hydroxide precipitate (MHP) or a mixed sulphide precipitate (MSP), both as unique intermediate products.

The decision on the processing option will depend on a number of metallurgical and economic variables and risk evaluations undertaken during a project's feasibility study.

Nickel laterite testwork conducted at SGS Lakefield Oretest over the past years has primarily focused on PAL through the various downstream unit operations to the production of a mixed hydroxide intermediate product (MHP). A typical MHP piloting flowsheet is shown below.



Metallurgical challenges associated with the second of these downstream processing routes, mixed sulphide precipitation (MSP), may now be fully evaluated at a purposely-built mixed sulphide precipitation pilot plant facility at Lakefield Oretest. A typical MSP piloting flowsheet is shown below.

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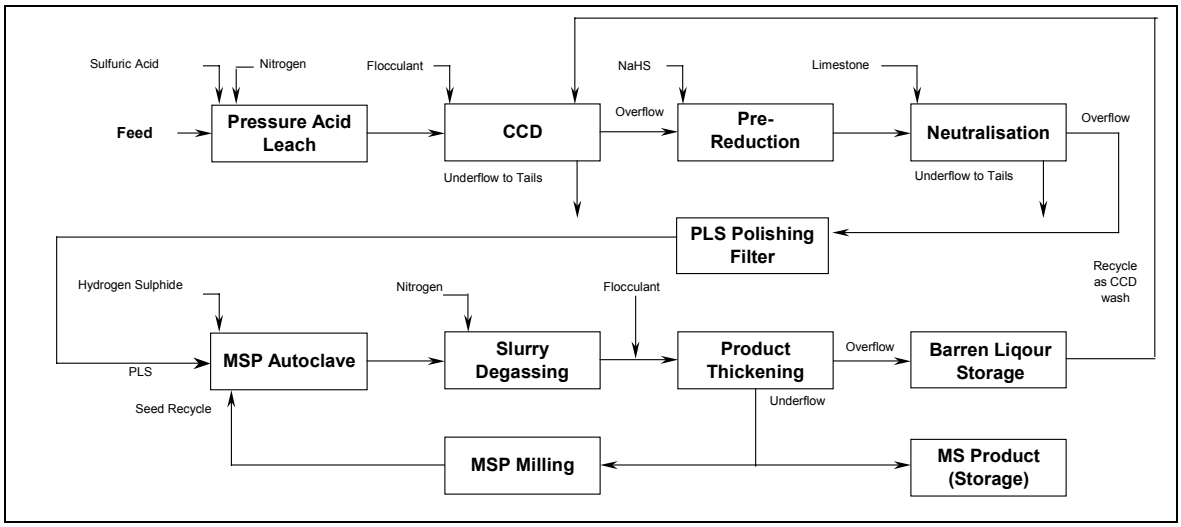

This paper outlines the challenges associated with the commissioning and continuous operation of a pilot plant configured to investigate the pressure acid leach of a nickel laterite ore through the downstream unit operations to production of a mixed sulphide intermediate precipitate using hydrogen sulphide gas.

2. PROCESS FUNDAMENTALS

2.1 PRE-REDUCTION

A CCD circuit separates the pressure leach solution (PLS) from barren PAL residue in a series of thickeners with counter current flow of solids and liquor. Wash liquor consists of recycle barren liquor from the mixed sulphide circuit (or a similar synthetic solution), which is added to the final thickener. The washed and thickened residue reports to the tailings storage system, while clarified CCD1 overflow proceeds to the Pre-reduction stage.

PLS produced from the pressure acid leach can be relatively high in oxidation-reduction potential (ORP) due to the presence of the Fe^{3+} and Cr^{6+} ions. In a commercial operation, this PLS would typically be treated in a pre-reduction circuit with the addition of H_2S gas reducing Fe^{3+} and Cr^{6+} to Fe^{2+} and Cr^{3+} respectively. Copper will also precipitate as a sulphide when reacted with H_2S gas. However at the pilot scale, NaHS can be used successfully in place of H_2S.

Copper, which has a much lower solubility than Ni or Co, was preferentially precipitated using NaHS to acceptably low levels without excessive co-precipitation of nickel or cobalt. Retention time however was important as found in the pilot plant. Co-precipitation of Ni and Co within pre-reduction was observed at around 3%, perhaps due to the longer retention times in this circuit – about 100 minutes. A pilot scale pipe reactor could overcome this issue.

The main testwork objectives successfully demonstrated during continuous pre-reduction piloting were:

- Reduction of ferric ion (Fe^{3+}) to ferrous ion (Fe^{2+}) consistent with decrease in solution ORP from 470 mV to <350 mV (vs. Pt-Ag/AgCl)
- Precipitation of copper as sulphide using NaHS

In pre-reduction the ferric ion was reduced to form ferrous sulphate, elemental sulphur and sulphuric acid by the following reaction:

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$$2Fe_2(SO_4)_3 + 2NaHS \rightarrow 4FeSO_4 + 2S^\circ + H_2SO_4 + Na_2SO_4$$

Hexavalent chromium in the PLS is also reduced by the following reaction:

$$H_2Cr_2O_7 + 2NaHS + 4H_2SO_4 \rightarrow Cr_2(SO_4)_3 + 2S^\circ + 6H_2O + Na_2SO_4$$

The copper present in the PLS reacts with NaHS to form copper sulphide:

$$2CuSO_4 + 2NaHS \rightarrow 2CuS + H_2SO_4 + Na_2SO4.$$

2.2 SOLUTION NEUTRALISATION

Free acid in the PLS along with acid generated through precipitation of coper sulphide, the conversion of ferric to ferrous and the reduction of chrome results in a free acid concentration of about 25 g/L in the pre-reduction overflow. Solution neutralisation is therefore required to ensure high recoveries of nickel and cobalt in the sulphide precipitation autoclave are maintained, as the sulphide precipitation reaction will also generate acid as metals are precipitated from solution.

The pre-reduced solution was neutralised in a series of cascaded tanks at about 70 ^oC using limestone slurry to achieve a discharge pH of 2.5, which is equivalent to an acid content of about 1 g/L. Solution neutralisation chemistry is straightforward and is the neutralisation of sulphuric acid with calcium carbonate in the limestone slurry.

$$H_2SO_4 + CaCO_3(s) + H_2O \rightarrow CaSO_4.2H_2O(s) + CO_2(g)$$

At higher pH, nickel and cobalt may co-precipitate as hydroxide or may be absorbed on freshly precipitated gypsum:

$$NiSO_4 + 2H_2O \rightarrow Ni(OH)_2 + H_2SO_4$$

$$CoSO_4 + 2H_2O \rightarrow Co(OH)_2 + H_2SO_4$$

Ferric ion precipitation also occurs by the following reaction:

$$Fe_2(SO_4)_3 + 5H_2O \rightarrow 2Fe(OH)_2 + 3H_2SO_4 + ½\ O_2$$

The impact of high ORP (i.e. incomplete pre-reduction) can lead to high amounts of ferric ion in the feed solution to the sulphide precipitation autoclave. Any ferric ion in solution would be converted to ferrous under the severely reducing conditions present within the autoclave. Elemental sulphur produced by ferric ion reduction would lead to contamination of the mixed sulphide intermediate product and is reflected in an increased sulphur to metals ratio. This would be detrimental in further downstream refining processes, especially those involving solvent extraction.

Re-dissolution of precipitated copper sulphide was observed within the neutralisation circuit. The conditions promoting re-dissolution of the copper sulphide could be caused by high levels of ferric ion providing strong oxidising conditions, which would oxidise sulphide to sulphur.

$$2Fe^{3+} + CuS \rightarrow 2Fe^{2+} + Cu^{2+} + S^0$$

The concentration of the ferric ions could determine how strongly oxidising this solution could become and thereby the amount of copper sulphide re-dissolution. Pilot plant observations indicate that temperature effects may also play a role here and need to be further investigated.

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2.3 SULPHIDE PRECIPITATION

Neutralised solution feed into the MSP pilot plant autoclave had the following typical analysis:

H_2SO_4	PH	ORP	Ni	Co	Fe^{2+}	Fe^{3+}	Mg
1 g/L	2.2–2.6	320 mV	4-5 g/L	0.2 g/L	10 g/L	< 0.1 g/L	12 g/L
Al	**Mn**	**Cu**	**Zn**	**Ca**	**Si**	**Cr**	**Na**
3 g/L	2 g/L	7.5 mg/L	100 mg/L	625 mg/L	150 mg/L	70 mg/L	1500 mg/L

The Sulphide Precipitation autoclave precipitated >99% of nickel and cobalt as their respective sulphides while rejecting a number of contaminants, including Mn, Cl, Fe, Mg, Cr, which remained in solution. The remaining copper (not removed through Pre-reduction) was also precipitated along with a major proportion of the zinc, which are both considerably less soluble than cobalt or nickel. Typical precipitation efficiencies achieved at 95^{o}C, 400 kPa(g) and 30 minutes retention were as follows:

Ni	Co	Cu	Zn	Fe	Al	Mn	Mg
>99%	>99%	98%	85%	1%	2%	< 1%	0%

The sulphide precipitation reactions are represented by the generalised expression below:

$$MSO_4 + H_2S_{(aq)} \rightarrow MS + H_2SO_4$$

where M = Ni, Co, Cu or Zn

These reactions are reversible and equilibrium conditions can drive the reaction in either direction. High acidity limits the extent of precipitation, while precipitation is enhanced by high H_2S concentrations. There are a number of factors that affect the completeness of the precipitation process and subsequent incidence of scale formation.

The gaseous H_2S must dissolve into the liquor phase. This was achieved through vigorous agitation within each autoclave compartment using 6-bladed pitched blade turbines (PBT). Agitator selection was performed during commissioning using a clear plastic autoclave model to investigate various speed and agitator configurations.

$$H_2S_{(g)} \leftrightarrows H_2S_{(aq)}$$

The H_2S dissolved in the process liquor must then dissociate to form the sulphide ion, which reacts with the metal ions in solution. This is a two-stage process represented by:

$$H_2S_{(aq)} \leftrightarrows H^+ + HS^-$$

$$HS^- \leftrightarrows H^+ + S^{2-}$$

The sulphide ion concentration has a major influence on the reaction rate and the levels of nickel and cobalt remaining in solution in the barren liquor. The reason why this occurs is due to the solubility product of metal sulphides. The table below shows a number of relevant metal sulphides and their solubilities.

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Metal Sulphide	Solubility Product at 25 ºC Ks	log Ks	at 100 ºC log Ks
CoS	$[Co^{2}] \times [S^{2-}] = 5.01 \times 10^{-22}$	-21.3	-19.2
NiS	$[Ni^{2+}] \times [S^{2-}] = 3.98 \times 10^{-20}$	-19.4	-18.6
CuS	$[Cu^{2+}] \times [S^{2-}] = 7.49 \times 10^{-37}$	-36.1	-29.7
Cu_2S	$[Cu^{+}] \times [S^{2-}] = 2.00 \times 10^{-48}$	-47.7	
FeS	$[Fe^{2+}] \times [S^{2-}] = 7.94 \times 10^{-19}$	-18.1	-15.6
ZnS	$[Zn^{2+}] \times [S^{2-}] = 2.00 \times 10^{-25}$	-24.7	-20.9

This table shows that copper and zinc will precipitate preferentially over nickel and cobalt due to their significantly lower solubilities. Also, a high sulphide ion concentration in solution favours precipitation.

2.3.1 Effect of Temperature

Raising the process temperature reduces the solubility of H_2S in solution and also increases the solubility of the metal sulphides, an apparent contradiction of where we want to be from an equilibrium point of view:

$$\log [S^{2-}] = \log 1.5 \times 10^{-22} + 2pH \quad \text{at } 25^{o}C \text{ \& 1 atm}$$

$$\log [S^{2-}] = \log 1.2 \times 10^{-20} + 2pH \quad \text{at } 90^{o}C \text{ \& 1 atm}$$

However these opposing effects are partially overcome by the fact that the dissociation of H_2S and the bisulphide ion are enhanced at higher temperature. Higher temperature also enhances mass transfer. This is critical at the low concentrations of nickel and cobalt that exist in the final compartments of the precipitation autoclave.

2.3.2 Effect of Pressure

The solubility of an ideal gas is directly proportional to its partial pressure above the solution into which it is dissolving. For this reason, while the pilot precipitation autoclave operated at 400 kPa(g) it has a rating of 3,000 kPa(g), enabling much higher pressures to be used if desired. Increasing the H_2S pressure significantly increases the gas consumption rate and makes H_2S recycle or destruction an even larger problem

While H_2S gas has a high solubility in pure water (108 g/L at 95°C), it is the partial pressure that affects solubility and any build up of other gases in the reaction vessel vapour space will have an adverse effect on the H_2S solubility. H_2S contains a number of "inert" impurities (eg hydrogen, carbon dioxide, methane), which can build up in concentration in the autoclave vapour space over time, as H_2S is consumed. For this reason a continuous bleed from the autoclave via a vent line was maintained to ensure an essentially pure H_2S atmosphere predominated within the precipitation autoclave.

H_2S gas was supplied in commercially available "G" size bottles. Each bottle contained about 31.5 kg hydrogen sulphide at a purity of not less than 99.5%. During piloting, two bottles were connected to the precipitation autoclave as a duty and standby gas supply.

2.3.3 Effect of pH

The sulphide ion concentration has a major influence on the residual concentrations of nickel and cobalt. There is a direct relationship between pH and sulphide ion concentration:

$$[S^{2-}] = K \,.\, [H_2S]_{(aq)} \,/\, [H^{+}]^2$$

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Doubling the hydrogen ion concentration results in a fourfold decrease in the sulphide ion concentration. This is the reason the pH is increased in the neutralisation circuit to about 2.5. Of the three main factors that affect the sulphide ion concentration, namely, pH, partial pressure and temperature, the pH of the solution is the most significant.

2.3.4 Seeding

Seed recycle has a number of effects on the process, these are:

- Reduces scale formation
- Improves product settling
- Reduces residual nickel and cobalt concentrations
- Enhances initial reaction rates

The addition of seed solids lowers the required activation energy such that higher reaction temperatures are not necessary to initiate the reaction. Nickel and cobalt precipitation reactions occur both homogeneously and heterogeneously. This reduces scale formation and provides solids of a larger particle size than would be generated by precipitation without using seed but at much higher pressures.

If no other surfaces are present, nucleation and precipitation will occur on vessel and pipe walls, resulting in unacceptably high scaling rates. This presents a challenge during piloting runs, particularly during startup. Rapid precipitation was also demonstrated during a power outage when seed was lost as scale due to rapid temperature losses and lack of agitation within the autoclave. Subsequent operation once power was restored proved futile with high barren liquor tenors, low recovery and very difficult operation.

To limit scale formation, new surface area was regularly provided by a constant recycle of ground-precipitated sulphides. The mixed sulphide product was removed from the thickener and ground in stirred mill to about 80% passing 10 µm, resulting in an available surface area of about 1.75m²/L PLS during the piloting testwork.

At some point during precipitation, the concentrations of nickel and sulphide become so low (the later being hindered by the rise in the H^+ concentration) that spontaneous precipitation no longer exists. At this point the precipitate and recycled seed solids are able to catalyse the reaction further, thereby allowing a closer approach to equilibrium concentrations than the residence time in the circuit would otherwise allow. By providing seeding, other process conditions such as H_2S partial pressure or temperature can be less onerous, whilst providing the same result. This was demonstrated during pilot plant operation when >99% precipitation was maintained at a lower partial pressure of hydrogen sulphide, when seed recycle was used.

2.4 H_2S DESTRUCTION

In the pilot plant, autoclave vent gases and fumes from the degassing tanks and thickener containing unreacted H_2S, were ducted to a scrubber.

The scrubber consists of an extraction fan in series with a vertical packed bed scrubber. The base of the scrubber tower acts as sump for recirculation of caustic solution to sprays located above the packed bed. Hydrogen peroxide is injected into the recirculation line with a dosing pump. The packed bed consists of loosely packed packing material, which enhances the contact between the entrained H_2S and the counter current alkaline hydrogen peroxide sprays. The packed bed is followed by a mist eliminator to remove entrained solution, prior to discharge.

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In alkaline conditions, H_2S in solution reacts predominately with peroxide as follows:

$$4H_2O_2 + H_2S \rightarrow H_2SO_4 + 4H_2O$$

Sufficient sodium hydroxide is added to neutralise the acid generated according to the following reaction:

$$2NaOH + H_2SO_4 \rightarrow Na_2SO_4 + 2H_2O$$

The barren autoclave discharge liquor was also treated with hydrogen peroxide to destroy any residual H_2S. In a fully integrated piloting operation, barren liquor is recycled as CCD wash solution after H_2S destruction. In acidic conditions peroxide reacts with H_2S to form elemental sulphur.

$$H_2O_2 + H_2S \rightarrow S^0 + 2H_2O$$

As the residual levels of H_2S were low (typically <30ppm) the amount of elemental sulphur formed was slight.

3. OPERATIONAL CHALLENGES

The MSP autoclave at Lakefield Oretest consists of a 35 L, 6-compartment titanium pressure vessel. The autoclave consists of three segments, each with two compartments and can be configured either as a 2, 4 or 6-compartment reactor. Each compartment is vigorously agitated via a 6-bladed PBT giving excellent gas – solid – liquid mixing.

PLS produced by the upstream leach circuit is injected in the first compartment via a positive displacement pump and heated on route to the autoclave through a indirect electric heat exchanger. Milled MSP solids are injected under pressure into the feed line.

H_2S gas is injected into the vapour space within each autoclave segment to maintain an over pressure of 400 kPa(g). Discharge slurry passes from the autoclave through a standpipe into a letdown vessel where initial cooling and degassing occurs. The slurry then passes to a degassing circuit where the barren liquor is purged with nitrogen before solid/liquid separation and barren liquor containment.

It was soon apparent that while the degassing circuit was very effective at removing H_2S, nickel tenors in the barren liquor increased through the circuit reaching a maximum in the thickener underflow. It was apparent that the nickel sulphide precipitate was redissolving. Typically, the nickel tenor would fall from 5 g/L in the PLS to <20 mg/L in the barren liquor. However, the nickel tenor increased to about 100 mg/L in the thickener overflow and about 200 mg/L in the thickener underflow. This effect observed during piloting can be explained by examining the sulphide precipitation diagram (after Mohemius) below.

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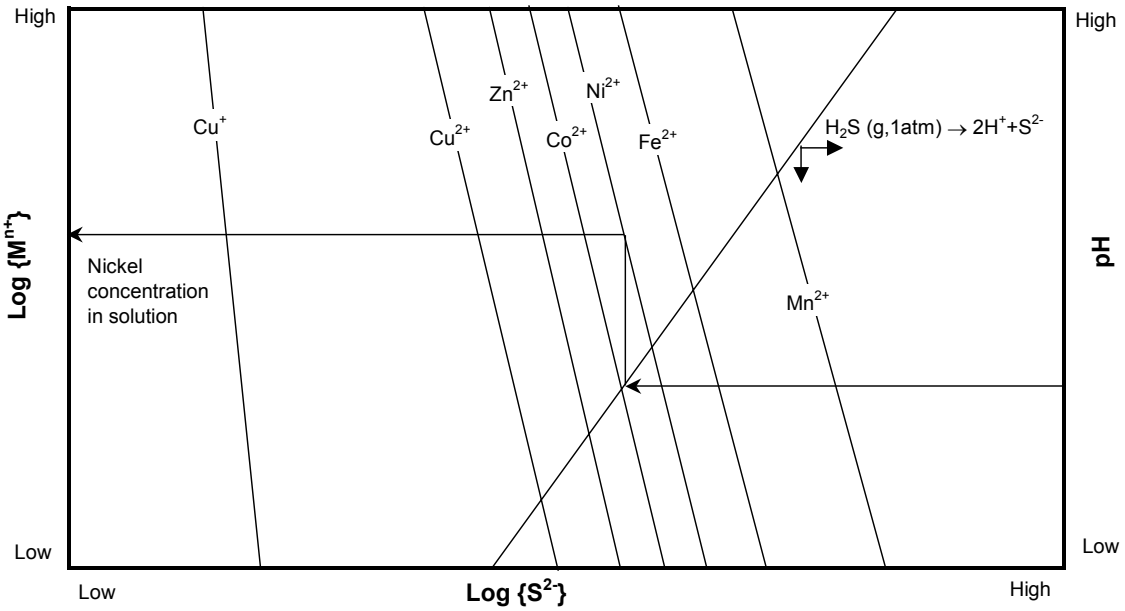

The sulphide precipitation reaction is reversible and nickel and cobalt sulphide will redissolve at low pH (high free acid) or conditions were the sulphide concentration is low. High nickel tenor in the PLS generates high free acid in the barren liquor while scrubbing H_2S reduces the sulphide concentration, both reactions drive re-dissolution. The issue was resolved in the pilot plant by discontinuing nitrogen scrubbing and minimising contact time between solids and liquor after letdown.

Re-dissolution is rarely encountered during batch scale autoclave test work due to solids and liquors being separated soon after the conclusion of the testwork. Nitrogen is passed through the batch autoclave at the conclusion of the test while it is cooling for 15 minutes prior to the solids being filtered on discharge. This is a much shorter residence period than in the pilot plant.

In general, 30 minutes retention, 95 ^{o}C and 400 kPa(g) H_2S pressure, was found to give nickel precipitation exceeding 99 %, consistent with the batch testwork results. Increasing retention to 45 minutes and increasing the H_2S pressure to 500 kPa(g) did not yield a demonstrable improvement in nickel recovery.

The most significant operational issue, accounting for most of the plant downtime was scaling of the feed and seed inlet port to the first compartment of the autoclave and a section of the feed line leading into this port.

The PLS and seed pumps used in the pilot plant were peristaltic hose pumps. However, due to the inherent design of hose pumps a slight back flow from the autoclave into the feed line was possible due to oscillating feed flows caused by each ½ revolution of the pump. With back flow occurring up the feed line causing ingress of H_2S, plating of sulphides on the feed line internals resulted. Scale formation extended from the autoclave inlet back up the feed line for a distance of 400–500 mm.

Scale was predominantly deposited as concentric layers that progressively reduced the diameter of the feed line. With the reduction in the feed line diameter, flaking of platelets of scale and the continuous addition of seed particles, the feed line eventually became blocked.

Pumping of ground mixed sulphide seed in a separate seed line was also tried and presented challenges to ensure constant flow and that sufficient seed addition was achieved. Low flow rates gave line velocities of less than 0.05 m/s and combined with the

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high specific gravity of the solids, continuous pumping of the seed to the autoclave created problems. Introduction of the seed to the feed line on the downstream side of the heat exchanger enabled longer periods of seed operation due to the short distance from the seed pump discharge to the feed line. However, seed pump performance dropped off rapidly as the feed line became restricted.

Seed addition to the suction side of the feed pump to remove the possibility of back flow was also evaluated. This resulted in a more consistent seed addition rate, however a downside was increased scaling in the heat exchanger tubes.

Regrinding of a portion of the thickened product for seed recycle was performed using a 6 L ultra fine mill with alumina media to increase the surface area of the seed to typically 2.3-4.7 m^2/cc.

Solid liquor separation was a process aided by the high specific gravity of the mixed sulphide solid (5.3 g/cc). It was however, difficult to obtain clear thickener overflow, consistent with off line testwork. The barren liquors were decanted and filtered prior to further treatment.

4. SPECIAL CONSIDERATIONS

Due to the inherent hazardous nature of H_2S gas certain safety management systems and precautions were put in place to ensure a sufficient level of response and containment could be achieved if a catastrophic failure were to occur. With continuous piloting spanning several weeks and being undertaken in a reasonably well-populated area, the pilot plant design and H_2S destruction systems needed to be robust and reliable. This was achieved by containing the autoclave and downstream sections in a purpose designed container attached to a custom-made scrubber tower. After several design reviews the pilot plant was subject to a rigorous and thorough HAZOP study, prior to commissioning and operation.

Essential to safe operations in the event of a failure are the emergency shutdown devices. The autoclave container contains two fixed zone alarms, one by the entrance and the other adjacent to the degassing tanks. The alarms are configured at a 15 ppm warning level and a 25 ppm H_2S shutdown level. At 25 ppm the alarms activate the emergency shut down (ESD) system via hard wire voltage free contacts. The main focus of the ESD is to shut off the feed systems to the autoclave, namely the PLS, seed recycle and H_2S gas supply. The scrubber is also an essential part of the safety systems and triggers the ESD if there is a failure of either the scrubber fan or recycle pump.

Operator training for working in H_2S environments was conducted both in house, and by an external safety company. External training consisted of self-contained breathing apparatus (SCBA) training for emergency response. Special procedures were implemented for the control and monitoring of personnel accessing and leaving the restricted H_2S area. For personnel required to enter the H_2S area it was necessary they comply with the following:

- Have successfully completed the hydrogen sulphide training module, and have been issued with an access permit.
- Carry a personal H_2S detector (to be worn within the breathing zone).
- Carry a half-face (or full face mask) respirator with A, B, E, K, P3 cartridge.
- Place their access permit on a permit board before entry into the H_2S area and remove it on exit.
- Carry a radio (1 per workgroup) switched to the appropriate operations channel.

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5. LESSONS LEARNT

Improving plant operational availability at a pilot scale is always difficult due to the constant challenges of the “unknown”. Lessons learnt from the commissioning runs will greatly increase the understanding for future MSP pilot campaigns. Plant improvements surrounding feed lines, seed injection, materials selection and circuit retention time will improve overall plant performance. The lessons learnt:

- The PLS and seed pumps should deliver a consistent flow with minium reverse flow.
- An external heating source on the autoclave shell is required if higher temperature campaigns are to be undertaken.
- Seed delivery position needs to be optimised. Direct injection of seed into compartment 1 is preferable and this is easily achieved via a spare port. Pumping distance also needs to be minimised to prevent line sanding or scaling.
- The contact time after letdown from the autoclave and separation of a barren liquor thickener overflow needs to be minimised to reduce the potential for re-dissolution.
- Removal of solids in the thickener overflow may not be critical to the plant availability but is essential in obtaining accurate data for metallurgical accounting purposes. More efficient flocculant contact with the incoming stream from the degassing tanks is required to reduce suspended solids reporting to the barren liquor.
- Sufficient seed surface area is required to minimise scaling.

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DEVELOPMENT OF COMBINED TECHNOLOGIES FOR COUPLED TREATMENT OF OXIDIZED NICKEL - COBALT ORES AND NICKELIFEROUS PYRRHOTINE CONCENTRATES

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1. INTRODUCTION

On the boundary between the XX and XXI centuries two challenges have specifically emerged in the metallurgy of nickel and cobalt: improvement of integrated approach and environmental acceptability of pyrrhotine concentrates treatment that were produced in the enrichment of sulfide Cu-Ni ores, and development of a cost efficient autoclave process for metal recovery from oxidized nickel-cobalt ores with high MgO content. Until recently, hydrometallurgical processes for pyrrhotine concentrates and oxidized nickel-cobalt ores treatment were developed as two independent branches, which limited the possibility of a profound solution of the indicated problems. Therefore to reduce sulfur dioxide emissions enterprises are compelled to remove from production to long-erm stockpiling a considerable proportion of the pyrrhotine concentrates containing base and precious metals. At the same time, in the treatment of oxidized nickel-cobalt ores by high pressure acid leaching (HPAL-process), elemental sulfur is consumed (up to 40 % of the cost price of the commodity output) to produce great volumes of acid, which makes nickel and cobalt production from high magnesia types of oxidized ore unprofitable. A fraction of oxidized nickel-cobalt ore, enriched with magnesia, is extracted by classification into a separate product, which goes to waste.

Unique, practically unlimited, possibilities for further progress in the production of base and platinum metals from pyrrhotine concentrates and oxidized nickel-cobalt ores provide options of combined designs, in which the processing of sulfide and oxidized nickel-cobalt feedstock is coupled in an integrated chemical-technological system.

A number of pressure oxidation leach technologies for oxidized nickel-cobalt ores processing have been proposed, in which acid forming minerals (pyrite, pyrrhotine) generating H^+ and HS^-_4 ions during ferriferrous sulfides oxidation are used instead of free sulfuric acid. The above mineral components effectively replace elemental sulfur and allow the range of MgO content in the processed oxidized nickel-cobalt ores to be expanded. At the same time, the joint leaching of the oxidized nickel-cobalt ores with pyrite and pyrrhotine concentrates is not always economically justified, as the above products can have high precious metals content. During iron sulfide oxidation, precious metals practically completely pass into the iron hydrate leach residue and will be lost. Therefore for the technologies considered, only pyrite and pyrrhotine concentrates are appropriate as the content of precious metals in them is not of any commercial concern. On the other hand, the low content of valuable metals in these concentrates is insufficient to make up for their transportation costs and treatment at the expense of additional metal production. In this context, the use of poor pyrite and pyrrhotine concentrates is effective only when their deposits are situated in the immediate proximity to the nickel - cobalt operation.

2. OPTIONS FOR NORILSK PYRRHOTINE CONCENTRATES

Norilsk′s pyrrhotine concentrates contain 2- 3 % Ni and 6-10 g/t of platinum group metals. Currently the pyrrhotine concentrates at Norilsk are treated by a pressure oxidation process. Nickel and cobalt recovery into sulfide concentrate according to this route amounts to only 84-86% and 80-85% respectively, and platinum group metals (PGM) about 70%. Pyrometallurgical treatment of such feedstock allows nickel and PGM recoveries to increase up to 95 % and more. However their usage in Norilsk is complicated by the geographic isolation of the Norilsk industrial region and the seasonal nature of transport communications with the "mainland", which makes it economically inadvisable to recover the generated SO_2 in the form of sulfuric acid, and other kinds of commercial sulfur products. High total value of metals in Norilsk pyrrhotine concentrates provides payback of their shipment expenditures for considerable distance. It creates a real possibility for its coupled treatment with the oxidized nickel-cobalt ores in the most distant regions of the world. It is quite apparent that in this case application of the conventional technologies of

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joint pressure processing of pyrrhotine concentrates and oxidized nickel-cobalt ores is inadvisable, as Norilsk concentrate is characterized by the increased PGMs contents.

Engineering personnel at the Polar Circle Division of JSC Norilsk Nickel Mining and Metallurgical Company proposed various options of combined treatment of pyrrhotine concentrates and oxidized nickel-cobalt ores over a wide range of feedstock compositions. One of the most interesting options involves a flowsheet which combines oxidizing smelting of pyrrhotine concentrates with the high magnesia fraction of oxidized nickel-cobalt ores in an autogenous smelting unit (for example, Vanyukov Furnace) along with the processing of a ferriferrous fraction of the oxidized nickel-cobalt ores using high pressure leach technology (HPAL). During oxidation of sulfides in the pyrrhotine concentrates, sulfur dioxide is formed and plenty of exothermal heat is generated, which are recovered and used as sulfuric acid and superheated steam to treat the ferriferrous fraction of the oxidized nickel-cobalt ore. Nickel, cobalt and PGM recovery from both of the raw materials in the target products of the head operations (high grade matte, nickel-cobalt solution) in this route will attain at least 95%. The further processing of these products is possible by any of the known methods. Sulfuric acid and steam utilization as by-products of the pyrrhotine concentrates smelting increases the cost effectiveness of the oxidized nickel-cobalt ores processing by HPAL , including MgO-rich ores.

3. ALTERNATIVE PROCESS FOR LAS CAMARIOCAS

An example of how coupled processing of the pyrrhotine concentrates and oxidized nickel-cobalt ores can serve an alternative process, is the process developed by Norilsk researchers for Las Camariocas Project (Cuba). Laterite ore from Cupey deposit which is used as raw material at the plant contains 5,6 - 9,4 % MgO, thus its treatment by HPAL technology is not at all simple. Therefore, the original project involved the ammonia leach technology for oxidized nickel-cobalt ores (Caron Process), characterised by high operating costs and a low level of target metals recovery, especially cobalt.

The alternative process proposed for the Las Camariocas Project (fig. 1), envisages the use of Norilsk pyrrhotine concentrates as a source of sulfuric acid and heat, consumed in the pressure leaching of laterite ore. Pyrrhotine concentrate and serpentinite (high magnesia) type of laterite ore are subjected to joint smelting in a Vanyukov furnace to produce nickel - copper high-grade matte, high strength sulfur dioxide off-gases and disposal steam. The high-grade matte, into which the bulk of base and precious metals is recovered, is either routed to Norilsk Nickel Mining and Metallurgical Company subsidiaries to treat it through to commercial metal products, or treated by tolling agreements. The hydrometallurgical stage of the proposed process involves the following basic operations (refer to Figure 1):

- Ore dressing;
- Pressure acid leaching;
- Counter current decantation in thickeners;
- Hydrolytic iron removal;
- Nickel and cobalt hydroxide stage precipitation;
- Nickel and cobalt ammonia leach;
- Ni/Co extraction separation;
- Cathode nickel and cobalt electrowinning.

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This part of the flowsheet is similar to the commercially applied process at Cawse (Australia).

Pyrometallurgical process portion of the alternative flowsheet at Las Camariocas plant has not been implemented on commercial scale at any plant elsewhere in the world. Equipment layout of the pyrometallurgical process at the Las Camariocas plant is illustrated in Figure 2.

The ore fed to smelting is dried in steam dryers (14) to 6-8 % moisture content. The steam for drying is generated by the waste-heat boilers installed for cooling of the smelting furnace gases. The mixture constituents (pyrrhotine concentrate, oxidized nickel-cobalt ore, sand and coal) from the piles in the open storage (1) are transported by loading truck into separate bins. The bins are equipped with belt feeders to provide controlled discharge of each component onto independent conveyors (2) for loading into the furnace. Preliminary blending of the components is not applied.

Nominal feed rate of mixture constituents:

Pyrrhotine concentrate	127 t/h;
Silica flux	52 t/h;
Oxidized nickel-cobalt ore	78 t/h;
Coal (for reduction)	6,5 t/h.

The smelting furnace is located in a separate room (3). This room is designed only for protection of the furnace refractories against the effect of precipitations and is constructed of light materials. The heavy lifting equipment is not used in the room. The maximum load-carrying capacity required for repair works amounts to 5 t.

The melts from the furnace are routed to granulation in two granulation pools (5), (6) for slag and matte respectively.

Nominal handling capacity for granulation:

High-grade matte	5,5 t/h;
Slag	209 t/h;

The dust-laden gases containing about 40 % SO_2 exit the oxidation zone and enter a waste-heat boiler (4) for cooling. Nominal steam production is 40 t/h at 8500 kPa. Reduction zone gases do not contain metal-laden dust and enter the clean gas boiler (15), of nominal steam production 30 t/h at 8500 kPa. After cooling in the waste heat boiler the reduction zone gases are discharged into the atmosphere through the stack.

After cooling in the waste-heat boiler, the oxidation zone gases are sent for after-cooling and rough cleaning in a water-spray hollow scrubber (7), and further by one of the working exhaust fans (10) they are pumped into a Venturi scrubber (11) and after that sent to the acid plant (12).

There's also an oxygen plant (9) on the smelter site with the capacity of 40000 Nm^3/h (95 % O_2), air-blowing installation (13) with the capacity 35000 Nm^3/h of air at 150 kPa, and also water-air heat exchangers (8) for cooling water circulation utilized in the cooling system for the furnace and auxiliary equipment.

Nominal capacity of the water-air heat exchangers is 14 Gcal/h.

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Fig. 3 shows a schematic figure of two-zone Vanyukov furnace.

The plan area of the oxidation zone shaft in the two-zone Vanykov furnace at the tuyere level is 49 m^2. The oxidation zone is equipped with 60 tuyeres for melt blowing, which provides feed rate from 3000 to 7200 t of mixture per day. Nominal furnace capacity is 6100 t per day. The plan area of the reduction zone at the tuyere level is 12 m^2. 14 tuyeres for blowing the melt and 14 tuyeres for CO afterburning in the gas space are installed In the reduction zone.

Nominal feed rate into the furnace:

- Process oxygen 37000 Nm^3/h;
- Blowing air 14000 Nm^3/h;
- Air for CO afterburning 22000 Nm^3/h.

The furnace is located on a solid concrete foundation.

The melts from the furnace are discharged continuously by gravity to granulation through slag and matte wells located at the end wall of the furnace equipped with tapholes. The slag taphole is 2050 mm above the bottom. Matte taphole is located 1660 mm above the bottom. The above mentioned tapholes location allows the furnace foundation to be positioned at zero level in the room. The gases from the oxidation zone exit through an uptake, situated in the center of the furnace roof. The gases from the reduction zone exit through an uptake in the reaction shaft. The furnace zones are separated from each other by a leak-proof partition, which prevents mixing of the oxidation and reduction zone gases.

The mixture is continuously fed into the oxidation zone through 4 chutes in the furnace roof. The reduction zone is also equipped with a loading chute for lump coal, which serves as reducing agent for both slag and fuel. The coal feed rate is 6 t/h.

The block diagram of raw materials treatment in the two-zone Vanyukov furnace is shown in fig. 4.

Considering Cuba's geographical position, specifics of it's commodities and power base, and considering available infrastructure, perennial practice of pressure processes operation and a number of other technical and economical factors, Norilsk researchers investigated the possibility of developing another hydrometallurgical process option for Las Camariocas Plant involving nickel and cobalt hydrogen pressure reduction to metallic state from ammoniacal - sulfate solutions. The proposed flowsheet is shown in fig. 5. In this option, the pyrometallurgical process is similar to the one reviewed above (fig. 1) except for the sulfur dioxide recovery stage: according to this flowsheet part of the SO_2 is reduced to H_2S, which will be used in the hydrometallurgical process for nickel and cobalt precipitation from the solution. The new process flowsheet at Murrin Murrin plant (Australia) went on stream in April 1999 can serve as a prototype for this process. The process of metallic nickel and cobalt powder production from ammoniacal-sulfate solutions by direct hydrogen reduction is run at Ft Saskatchewan (Canada), Quinana, Murrin Murrin (Australia), Harjavalta (Finland), Port Nickel (USA) etc. An important advantage of the proposed process consists in simultaneous production of the marketable ammonium sulfate - a valuable fertilizer that is seels well in the agricultural regions of South America.

The alternative flowsheet considered in the hydrometallurgical route at the Las Camariocas plant (fig. 5) involves the following basic operations:

- Laterite ore beneficiation by screening;

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- Pressure acid leach;
- Tails counter current decantation;
- Solution neutralization;
- Nickel and cobalt sulfides precipitation by hydrogen sulfide;
- Pressure-oxidation leaching of nickel - cobalt intermediate product;
- Cobalt and metal impurities solvent extraction ;
- Hydrogen precipitation of metallic nickel in autoclaves;
- Hydrogen precipitation of metallic cobalt in autoclaves;
- Sintered nickel and cobalt briquette production;
- Marketable ammonium sulfate production.

The choice of the final flowsheet for the Las Camariocas plant demands the comprehensive efficiency study of the proposed options and comparative technical and economical estimations.

4. OTHER APPLICATIONS

During investigations using high magnesia samples of Russian oxidized nickel-cobalt ores (from Burukhtal and Serovskoye deposits in the Urals), it was found out that the developed combined technology has the prospects of usage not only for Cuban laterites, but also for treatment of oxidized ore of the domestic nickel - cobalt deposits. It opens up new vistas for replenishing nickel feedstock shortage at Norilsk Nickel Mining and Metallurgical Company. It is of the utmost importance for implementing the adopted decisions on the formation of ore reserves source forthe subsidiary Severonickel Combine that has no ore reserves of its own. Given well-developed infrastructure in the Urals region and availability of fuel, energy and raw resources, the most efficient flowsheet for processing of the oxidized ores from Burukhtal, Serovskoye, Sakharinskoye and other deposits seems to be the flow diagram adopted for the Marlborough Project (Australia), which involves marketable cathode nickel and cobalt. Here, as well as at the Las Camariocas plant it is possible to propose a coupled arrangement of two process stages with the installation of a Vanyukov furnace in the pyrometallurgical stage. Local pyrite ores or Norilsk pyrrhotine concentrates can be utilized as sulfide feedstock. This flowsheet requires carrying out a detail technological study.

The proposed processes also look rather attractive as an alternative for processing Western Australia's "dry" laterite ores. Given that Second Generation Australian laterite projects are in many cases situated in the vicinity of the existing operating plants, it seems logical to develop at sulfide-laterite nickel deposits combined metallurgical operations involving autogenous smelting of sulfide feedstocks (for example, in a Vanyukov furnace) and the HPAL process for laterite ore treatment.

The considerable stockpiles of man-caused pyrrhotine raw material inventories that have been accumulated for several decades in Russia and Canada, along with the huge world resources of sulfide and oxidized nickel - cobalt ores, a provide raw materials base for the combined processing of pyrrhotine concentrates and oxidized nickel-cobalt ores for the long-term future.

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ALTERNATIVE FLOWSHEET AT LAS CAMARIOCAS PLANT (Cuba)
Option 1

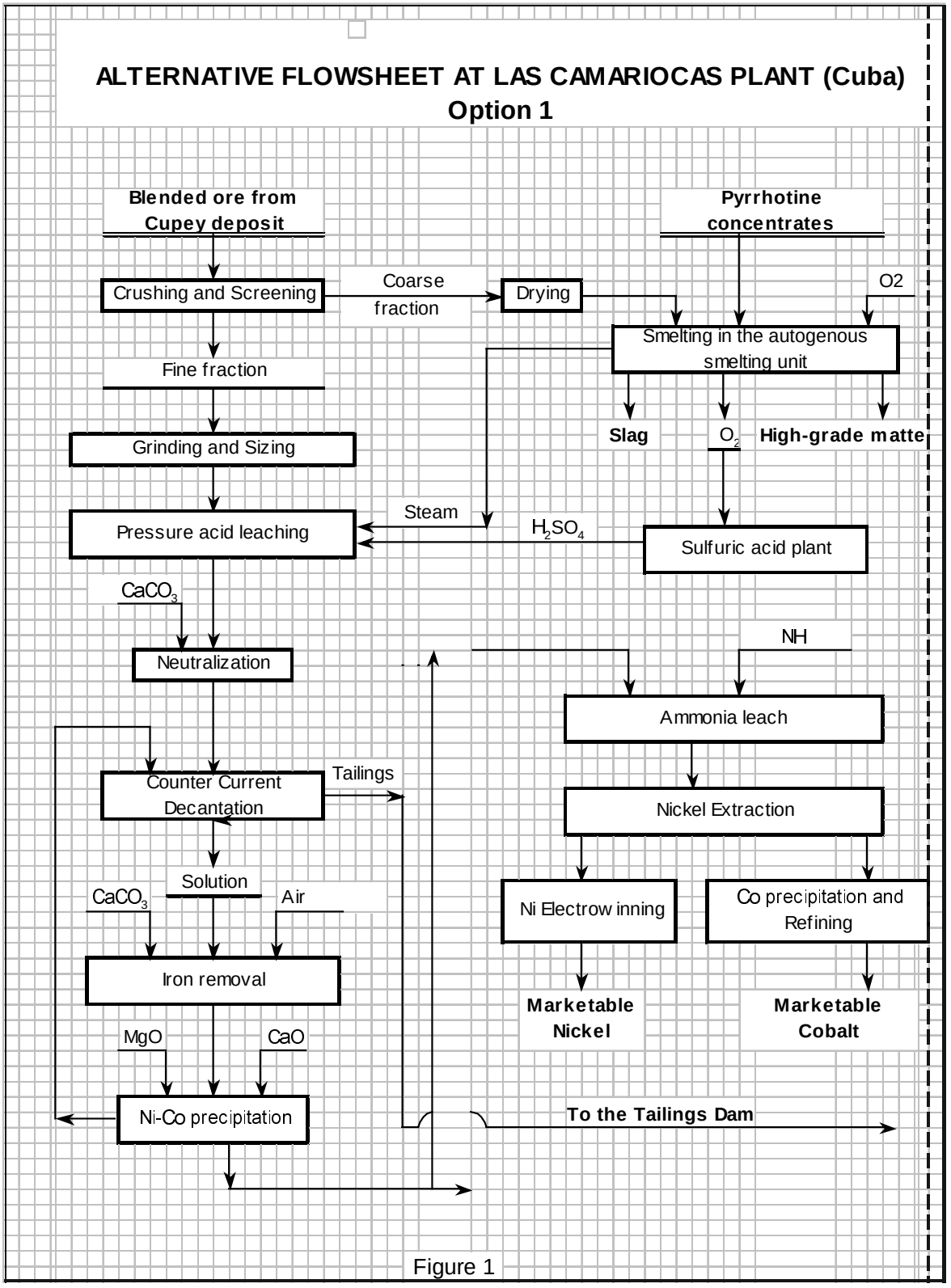

Figure 1

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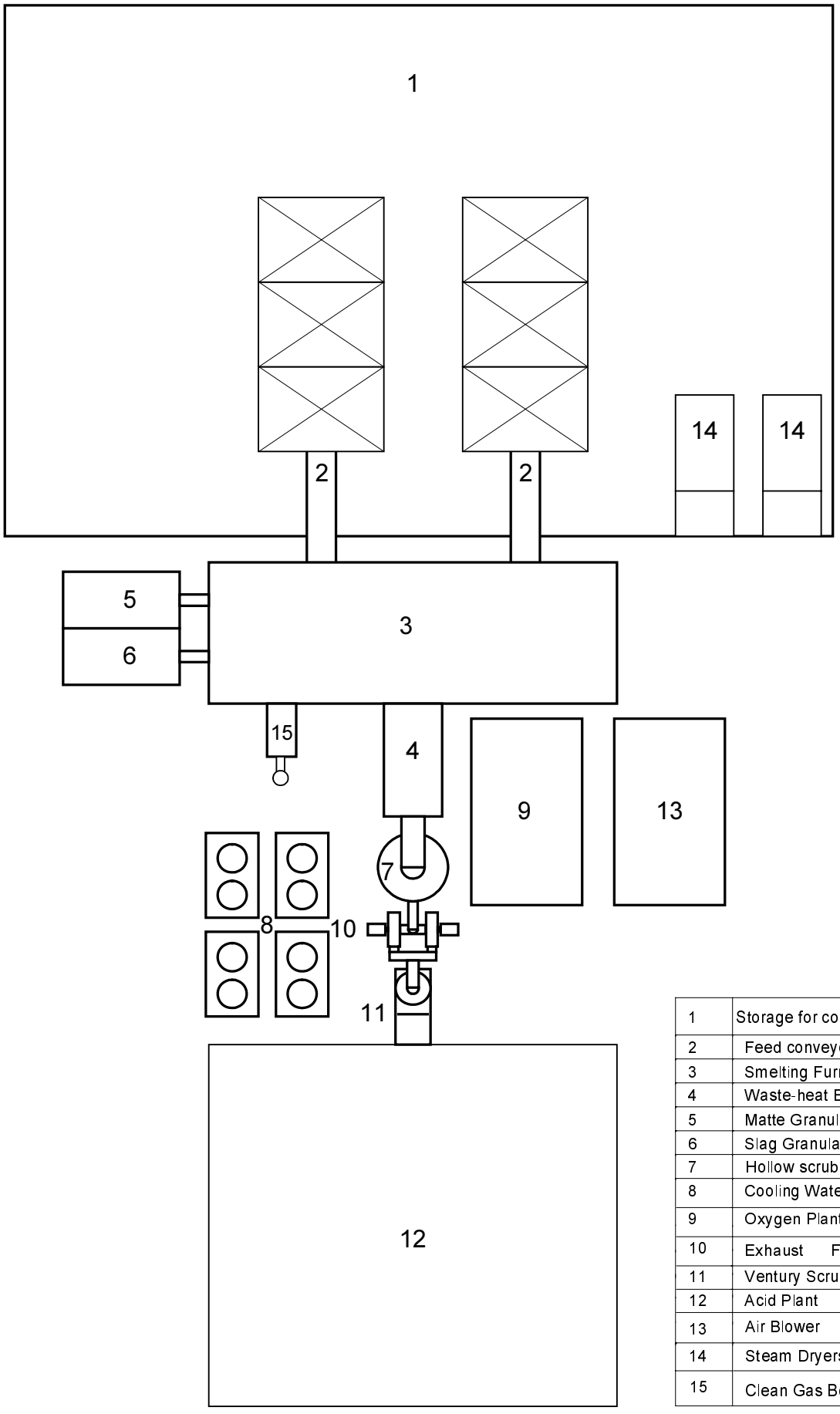

1	Storage for concentrate, ore, sand
2	Feed conveyors
3	Smelting Furnace Room
4	Waste-heat Boiler
5	Matte Granulation Pool
6	Slag Granulation Pool
7	Hollow scrubber
8	Cooling Water System
9	Oxygen Plant
10	Exhaust Fans
11	Ventury Scrubber
12	Acid Plant
13	Air Blower
14	Steam Dryers
15	Clean Gas Boiler with a Stack

Fig.2 Equipment Layout of Pyrometallurgical Process According to Alternative Flowsheet Proposed for Las Camariocas Plant

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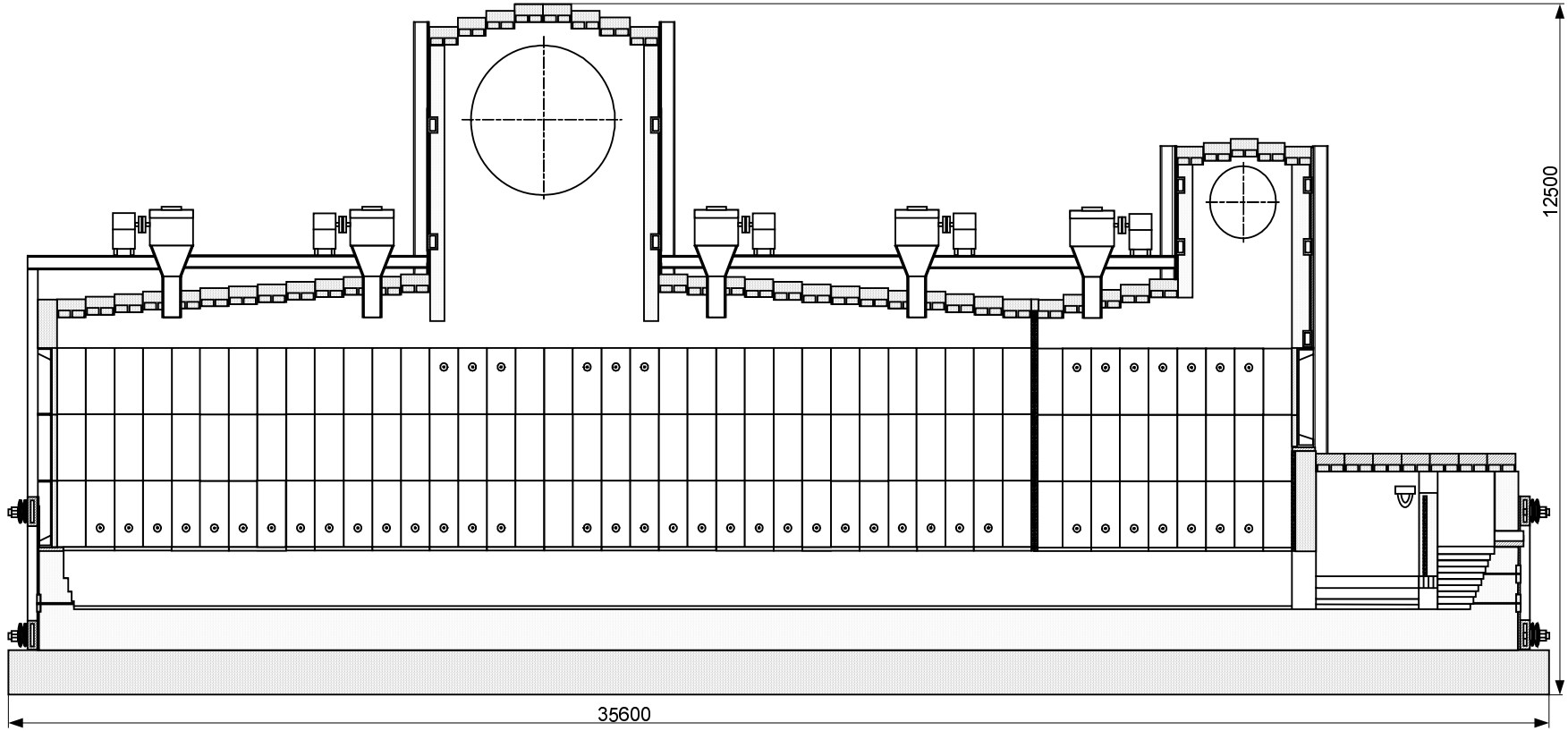

Fig. 3 Schematic Figure of Two-zone Vanyukov's Furnace for High Grade Matte Production from Norilsk Pyrrhotine Concentrate and Magnesia Fraction of Oxidized Nickel-Cobalt Ore

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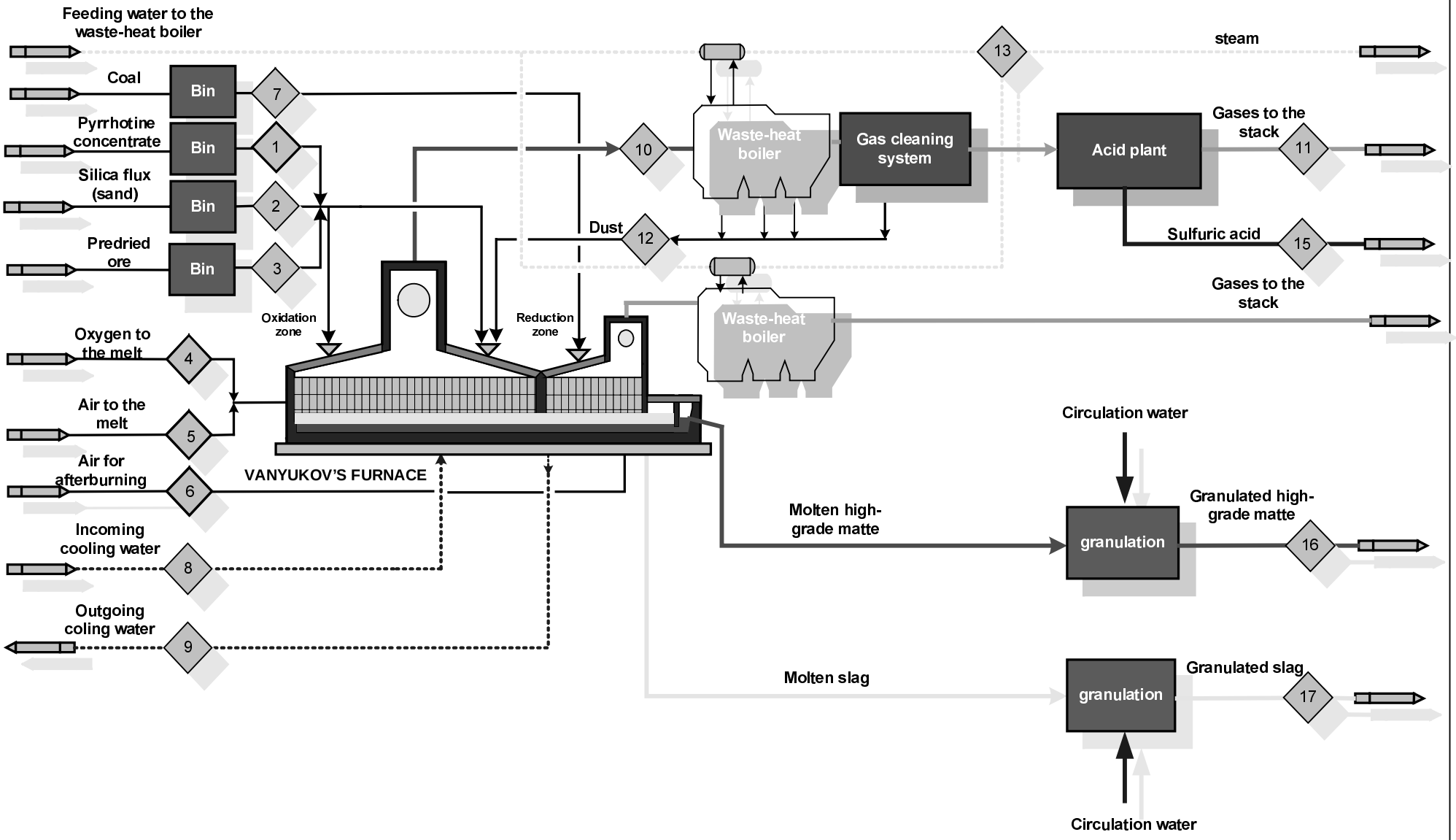

Fig. 4 Flow Diagram of High Grade Matte Production using Alternative Flowsheet at Las Camariocas Plant

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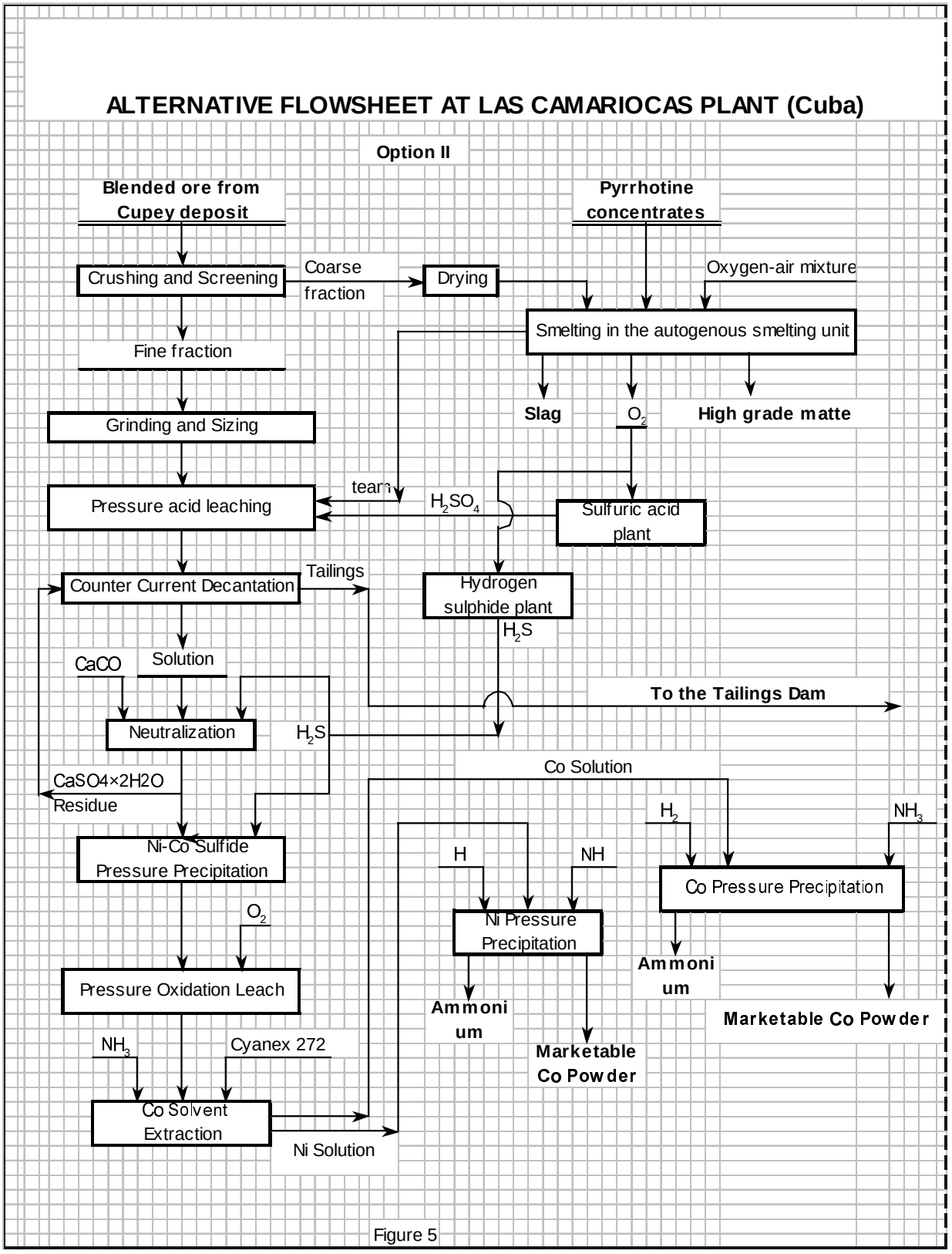

Figure 5

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SERVING THE GLOBAL MINING & METALLURGICAL INDUSTRIES WITH TECHNICAL EXCELLENCE & PROVEN INTEGRITY

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BEYOND PAL: THE CHESBAR OPTION, AAL

By

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ABSTRACT

Chesbar Resources Inc. is developing a nickel-cobalt laterite project in Guatemala, Central America, by applying existing atmospheric chloride technology to a known resource. The atmospheric acid leach (AAL) process represents the next generation of hydrometallurgical process recovery technology for nickel laterite deposits.

The tropical laterite project has an inferred resource of 133 million tonnes grading 1.51% nickel. Within its boundaries, the Sechol area has a measured resource of 14 million tonnes grading 1.46 % nickel and 0.08% cobalt, and an indicated resource of 23 million tonnes grading 1.34% nickel and 0.08% cobalt. Notwithstanding the impressive global resource, the company has taken an innovative approach to a laterite project and is concentrating on El Inicio, a high grade starter pit with 5 million tonnes grading 2.1% nickel and 0.08 % cobalt. The process flowsheet includes ore beneficiation with initial test results suggesting a 30% to 40% increase in head grade. A metallurgical scoping study has established preliminary capital and operating costs for a production facility at a proposed rate of 10,000 tonnes per year of nickel as an intermediate mixed nickel-cobalt hydroxide. A preliminary assessment of producing a magnesium oxide by-product has also been carried out.

This paper reports on the current development of the project and highlights the advantages of working in a chloride medium at atmospheric pressure and slightly elevated temperatures, demonstrating the robust financial returns of this exciting project.

1. INTRODUCTION

Chesbar Resources is a Canadian Exploration Company listed on the Toronto Stock Exchange, Canada. Minera Mayamerica S.A., a joint venture between Intrepid Minerals Corporation and Chesbar (75/25), was originally signed in January, 1998 with the objective of exploring for gold and silver; prior to that, Chesbar had been involved in gold exploration in Venezuela, In December, 1998, Minera Mayamerica S.A. was granted the original nickel licences in Guatemala for properties adjacent to Inco's Exmibal deposit, and in 2001, Chesbar subsequently acquired 100% of Mayamerica with the objective of establishing a nickel/cobalt production facility.

2. GEOLOGY OF THE GUATEMALAN LATERITE BELT

2.1 GENERAL DESCRIPTION

The Sechol group of deposits comprises several large lateritic pockets, which are delimited by tectonic faults and hosted by ultramafic rocks, mainly dunites and pyroxenites, and usually strongly serpentinized. The basement of the region is Paleozoic, mainly composed of schists and other metamorphic rocks. Some 300 million years ago, during the Carboniferous era, there was a deposition of marine sediments and conglomerates near the beach, followed by sandstones and shales at greater depths. Parallel to this, there was intrusion of granitic and dioritic batholiths into the Paleozoic basement.

During the Permian to the Lower Cretaceous Period, deposition of limestones and other carbonate rocks occurred. This was interrupted by a hiatus of nearly 51 million years during the Triassic Period, when the sea retreated and no significant deposition occurred. Exposure to oxidizing conditions in a tropical environment may account for the formation of the Upper Jurassic red beds.

The Upper Cretaceous Period was a very active one, with the deposition of more clastic sediments and the intrusion of granitic, dioritic, and ultramafic bodies. The ultramafic intrusives occurring along the major Potochio and Malagua Faults divide the region into the

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Maya Block belonging to the North American Plate to the north, and the Chortis Block belonging to the Caribbean Plate to the south. The Paleocene Period witnessed the deposition of more marine sediments, mainly conglomerates, near the shores and sandstones and shales at greater depths. During the Eocene Period, ten million years later, the formation of more red beds occurred, which probably indicates another period of sea-regression.

Finally, during the Holocene period, the principle rock formations are represented by quaternary alluvial and deluvial material, as well as by lavas and tuffs from active volcanoes. Ultramafic rocks that had undergone serpentinization before, were now being oxidized, resulting in the formation of the Ni-Co laterite zones, such as Sechol, the current main target.

As a result of the study of several laterite profiles in the area, and using the definitions established by the North American Stratigraphic Code, a new pedostratigraphic unit was defined, The Izabal Geosol, which is composed of five pedological horizons:

- Gossan or Iron Hat (not always present)
- Limonitic Horizon
- Stone Line
- Mottled Zone Horizon (also known locally as Transition Zone)
- Saprolite Horizon.

The saprolite horizon marks the bottom part of the Izabal Geosol, and lies directly over the saprock horizon which continues into the less weathered bedrock. The most complete profiles are usually present over weathered dunites and serpentinites, while on top of the less altered pyroxenites, there is usually the formation of only a limonitic zone.

All of the laterite deposits in Guatemala are associated with an ophiolitic belt that probably intruded during the Tertiary Era. The young age and the petrologic composition of these ultramafic rocks, together with the climatic conditions of the area, are responsible for the formation of these immature profiles, shown in Figure 1.

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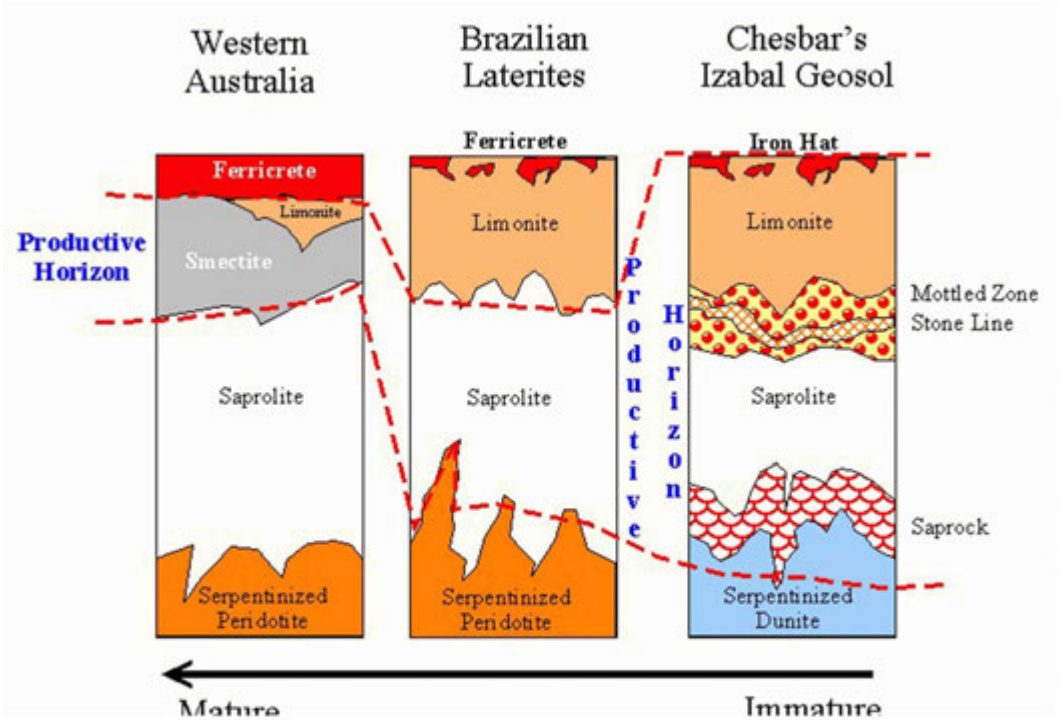

Figure 1. Comparison of the immature profile of the laterites from the Izabal Geosol with wet and dry laterites.

2.2 CHESBAR PROPERTIES

The extensive regional position held by Chesbar Resources Inc. within the Guatemalan Ophiolitic Belt provides the potential for similar or even larger-scale deposits than the Sechol Group which have already been identified. The 2002 exploration program recently completed by Chesbar has identified several potential deposits within the Sechol property and in other adjacent licences. Including the work completed in the Marichaj, San Lucas and Sechol properties, Chesbar has already identified resources of 133 M tonnes grading 1.51% Ni and 0.08% Co. Currently, the company has a total of 15 claims, representing 741 km^2 of potential lateritic targets, as shown in Figure 2.

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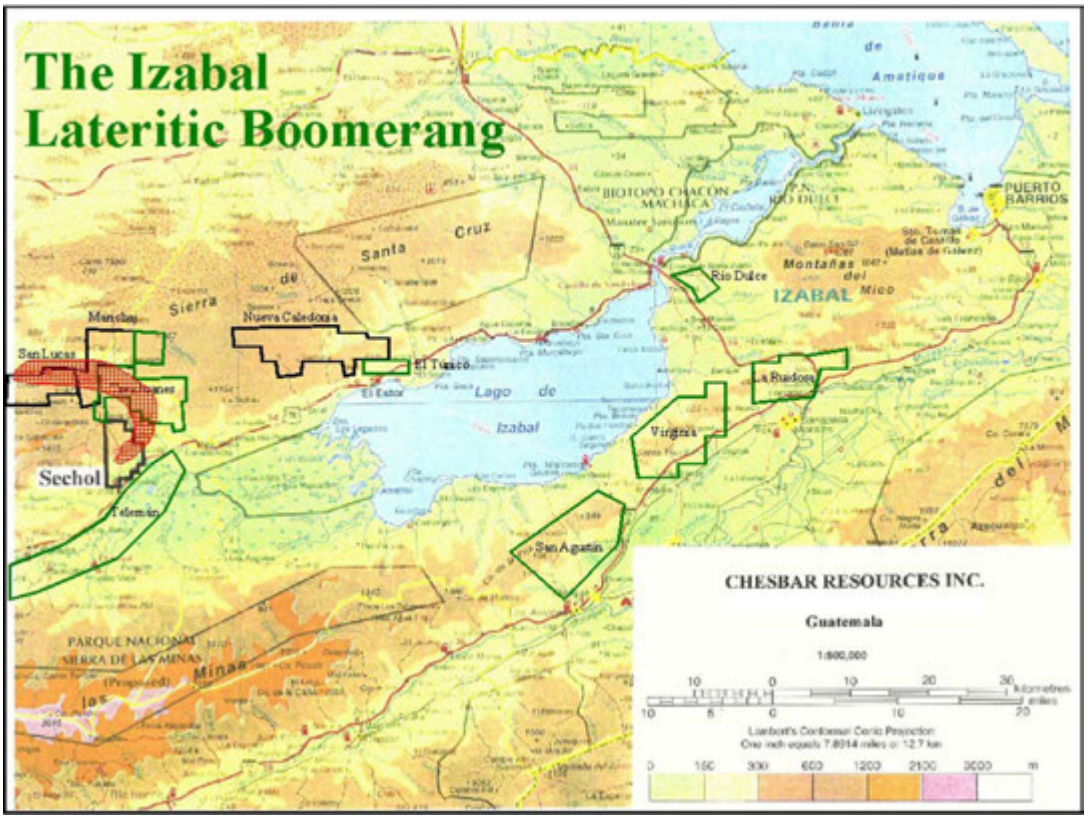

Figure 2. Location of other Chesbar licences of around Lake Izabal

2.3 MINERAL RESOURCES

In accordance with the NI 43-101 legislation, Chesbar has identified the following resources (Table **I**) in the Sechol Property, using a 1% Ni cut-off, a minimum thickness of 2 metres, and a dry density of 1 g/cm^3.

Table I. Summary of Mineral Resources of the Sechol Project as of May 22, 2002

Category	Million Tonnes	% Ni	% Co	% MgO
Measured	14.00	1.46	0.08	13.85
Indicated	23.00	1.34	0.08	11.79
Total	**37.00**	**1.39**	**0.08**	**12.57**

Within the measured resources, the start-up pit, El Inicio, with more than 5 M tonnes grading 2.1% Ni and 0.08% Co has been identified, with material from this pit being used to prove the metallurgical flowsheet.

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3. METALLURGICAL FLOWSHEET

3.1 BACKGROUND

Over the past decade, nickel laterites have received a great deal of attention in terms of hydrometallurgical flowsheet development. All of the processes planned or so far commercialized have been based on a sulphuric acid leach at high pressure, the Pressure Acid Leach (PAL) or High Pressure Acid Leach (HPAL), and from published information, there appears to have been very little attention given to evaluating a chloride-based approach. Various reasons have been advanced for this, such as exotic materials of construction being needed, chloride is an aggressive lixiviant with a consequent high acid consumption, difficulties with controlling iron leaching, and how to handle magnesium leaching. All of these are valid concerns, but neither are they insurmountable problems.

The first, and probably most successful, laterite leach plant is that at Moa Bay in Cuba, which first commenced operations in 1959 [1]. The process is currently based on a sulphuric acid pressure leach at around 240^{o}C in pachuca autoclaves, and precipitation of a mixed nickel/cobalt sulphide intermediate for further refining at the Corefco (formerly Sherritt) nickel-cobalt refinery at Fort Saskatchewan, Canada. Until relatively recently, this remained the only operating pressure acid leach process for nickel laterites.

An alternative process, a modified version of that first described by Caron in 1950 [2,3] and known by his name, based on ammonia/ammonium carbonate leaching of reduction-roasted laterite, has seen some commercial success, notably by Níquel Tocantins in Brazil and Queensland Nickel (BHP-Billiton) in Australia. The major disadvantage of this process is that cobalt recovery is limited, often as low as 40%.

4. FLOWSHEET DEVELOPMENT

The Chesbar Sechol development in Guatemala is next to the Exmibal property, owned by Inco, and which was operated as a nickel matte smelter until it shut down in 1980. These properties border Lake Izabal (Figure 2), which is increasingly becoming a resort spot, and therefore, it was considered undesirable to consider any kind of smelting operation in such a setting. Despite the relatively high MgO content of the ore, this not only eliminated options such as ferronickel or matte smelting, but also the roasting step which is a pre-requisite for the Caron ammonia leach process. With this in mind, attention was then turned to investigating a hydrometallurgical flowsheet, with the original intention being to follow the well-developed sulphuric acid pressure leach (PAL, HPAL) processes coming on-stream in Western Australia.

4.1 PRELIMINARY SCOPING ACID LEACH TESTS

Initial scoping testwork, carried out at different testing laboratories (and hence on slightly different materials), showed that both the saprolite and the limonite horizons of the orebody were amenable to leaching with either sulphuric or hydrochloric acid, as shown in Table II.

Table II. Results of Initial Scoping Leach Tests on Sechol Samples

Test Conditions	Type	Acid kg/t	Solution Analysis, g/L				Extraction, %			
			Ni	Co	Mg	Fe	Ni	Co	Mg	Fe
PAL, H_2SO_4 250^{o}C, 2h	Limonite	850	8.83	0.39	27.4	13.9	98.4	92.4	95.6	8.0
	Saprolite	500	4.1	0.073	26.0	5.29	99.3	93.2	99.2	11.0
H_2SO_4, 95^{o}C, 2h	Limonite	236	2.80	0.13	12.8	3.35	42.0	39.0	65.0	3.0
	Saprolite	390	4.91	0.059	27.4	1.42	72.0	45.0	62.0	3.0
HCl (20%), 80^{o}C, 3.5h	Limonite	285	15.0	0.29	34.4	113.0	94.9	9.2.2	74.4	84.4
	Saprolite	350	13.1	0.15	57.0	77.4	94.1	94.5	87.3	91.5

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These tests were carried out in order to give pointers as to the most attractive route to follow, and the results can be summarized as follows:

- The highest extractions were obtained with the pressure sulphuric acid leach system, although, as can be seen, the atmospheric hydrochloric leach yielded very satisfactory recoveries of both nickel and cobalt.

- The atmospheric sulphuric acid approach was not successful, and could only be used in conjunction with a subsequent pressure leach.

- Magnesium extraction in all instances was high.

- Iron extraction, as would be expected, was highest in the atmospheric chloride leach, but even with the PAL system, there were significant iron values left in solution which would require either an additional processing step such as jarosite precipitation or a high level of neutralisation.

- Although not shown in Table II, solid/liquid separation from the PAL process was very poor, especially for the saprolite phase.

The nature of the Sechol ores is such that there are relatively high magnesium and iron values in each of the saprolite and limonite, and it is clear from the scoping tests that both horizons are highly amenable to acid leaching, whether at atmospheric in a chloride medium or with PAL in a sulphate medium. However, whichever system was ultimately chosen, it was clear that there remained a number of issues which had to be addressed.

The Ore: Hydrometallurgical flowsheets to date have generally targeted mainly the limonite horizons of laterite orebodies, since these are generally low in magnesium content (nominally <5% Mg). Such areas are also lower in nickel content than the saprolite horizons, which have higher magnesium, but lower iron. On the other hand, saprolites have traditionally been treated by smelting methods to make ferronickel or nickel matte, where the magnesium can be slagged off, and is thus not a serious problem. No processes currently operating are able to treat both horizons for some of the reasons outlined below. The Sechol ore, as noted above, has appreciable levels of magnesium and iron in both horizons, and both are equally amenable to acid leaching, so that this differential was not especially marked from a chemical composition point of view.

Iron: Iron is a problem in all hydrometallurgical processes. In an atmospheric sulphate system, there is the option of removing the bulk of the iron as a jarosite, as practiced in the zinc industry. In the ammonia (Caron Process) system, the iron largely does not leach or is very easily precipitated. In pressure oxidation processes, whether in chloride or sulphate, it can be precipitated at high temperatures in a number of forms, the most common of which is hematite. However, apart from the ammonia system, all of these processes leave some iron behind in solution, which requires additional conventional neutralization and precipitation with a base such lime or magnesia, and such appeared to be the case with the Sechol ores.

Magnesium: Magnesium is a particular problem in sulphate-based flowsheets, since there is no logical outlet for it except as effluent at the end of the circuit. There is a small market for Epsom Salts ($MgSO_4 \bullet 7H_2O$), but nothing of sufficient magnitude to encourage production of magnesium sulphate. The alternatives are thus to lime, or to crystallize the sulphate salt and roast to magnesia, recovering SO_2 in the process, which can then be routed to an acid plant for recovery of sulphuric acid. Inco are understood to have examined this route for one of their operating properties, but did not adopt it [4]. Whilst magnesium also leaches in a chloride medium, it is somewhat easier to handle by direct

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pyrohydrolysis, which also permits recovery of the hydrochloric acid for recycle, and generates a magnesia product for recycle within the flowsheet or for sale. Pyrohydrolysis is well known in the steel pickling industry for iron chloride solutions for recovery of the acid [5,6].

4.2 CHOICE OF FLOWSHEET

With the above in mind, Chesbar was faced with the following, given the results of the initial scoping leach tests:

- Both iron and magnesium would have to be dealt with irrespective of whether in sulphate or chloride. In terms of iron, this appeared to be somewhat less onerous in a sulphate PAL circuit, although still significant, but for magnesium, an atmospheric chloride-based leach circuit appeared to be much more attractive, allowing for the production of magnesia and recycle of the acid.
-
- Solid/liquid separation of the leach slurry, and especially the saprolite fraction, was extremely poor in sulphate, necessitating what would have been a multi-stage CCD circuit, whereas in chloride, the slurry can be filtered relatively easily

.

- Both the saprolite and limonite horizons can be treated equally effectively, which is a major advantage, since this not only significantly simplifies the mining plan, but also renders the whole deposit an economic orebody rather than just one horizon.
-

Since going with the sulphate approach would entail high pressure autoclaves, importing sulphuric acid, a significantly greater residue disposal area due to the final neutralizing of magnesium sulphate generating gypsum solids, and a CCD circuit because of the poor solid/liquid separation characteristics, a decision was therefore taken to pursue a chloride-based flowsheet.

The chloride option was seen to offer the following advantages:

- Both limonite and saprolite horizons were amenable to atmospheric leaching, with >90% recovery of nickel and cobalt, thereby, as noted above, simplifying the mining plan and rendering the whole orebody economic.

- By incorporating pyrohydrolysis into the flowsheet, acid could be recovered and recycled, unlike in the sulphate system where it is mostly lost.

- Pyrohydrolysis also offers the potential for generating a valuable revenue stream in the form of high grade magnesia.

- Additionally, pyrohydrolysis eliminates any final effluent stream, and provides reagents HCl and MgO) for recycle within the flowsheet. Thus, whilst pyrohydrolysis, as a stand alone unit operation, might appear to be capital and operating cost intensive, the elimination of an effluent treatment, elimination of the need to buy fresh lixiviant (HCl), and elimination of the need to purchase neutralizing reagents (MgO) means that it is a very attractive proposition.

A further, crucial, decision was taken in that no attempt would be made to make metal, but rather an intermediate hydroxide product, which is highly suitable to further refining by others. There is significant spare refining capacity in the world, and Chesbar has received a number of approaches with a view to taking this intermediate product. The costs to build and operate a refinery are significant, and Chesbar wanted to both minimize the initial capital investment, as well as bringing the process into operation at the earliest possible date.

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A chloride-based atmospheric leach process, whilst unusual, is far from unique. Noranda operated the Brenda Leach Process, employing a high temperature (105-110^{o}C), high-strength chloride ($CaCl_2$+NaCl+HCl) atmospheric leach of copper-molybdenum concentrates until the mine shut down in the 1990s [7]. Falconbridge operates a chloride process at its nickel-cobalt refinery in Kristiansand, Norway, which was initially a hydrochloric acid leach, but more latterly a chlorine leach [8,9,10]. Whilst these processes treat matte as a feed, their operation, and those of other plants such as operated by Jinchuan [11], Sumitomo [12] and SLN [13] in a mixed chloride-sulphate medium, clearly demonstrate that chloride-based circuits are perfectly feasible, and that any material-handling issues associated with a chloride environment can and are being overcome.

However, perhaps the nearest operating circuit to the one conceived is that of Noranda's Magnola plant in Quebec [14]. Whatever difficulties this plant may have had with the production of pure magnesium metal, the chloride leach circuit has always performed up to expectations. This circuit operates at 100-105^{o}C (atmospheric conditions) on a serpentine-type mineral feed, and its success lends considerable confidence that a similar circuit is practical with a nickel-cobalt laterite feed.

The other major unit operation envisaged in the flowsheet is pyrohydrolysis of the final magnesium chloride liquor. Over the years, this has become a very mature technology, particularly in the steel pickling industry. There are essentially two ways of operating, in a spray roaster or in a fluid bed [15]. The former generates a fine, caustic powder, suitable for use as a neutralizing and precipitating agent within the process, whereas the latter generates coarser particles, more suitable for handling and sale. Significant work has been recently reported on the pyrohydrolysis of solutions containing magnesium chloride, which is not quite so straightforward as it is for iron chloride solutions, but is nevertheless operating successfully on predominantly magnesium chloride liquors at Rio Tinto Iron and Titanium (QIT) in Quebec [16,17]. Thus, as with the leach circuit, there is considerable confidence that pyrohydrolysis practical. It is also worth noting that at its Goro plant, Inco plans to use pyrohydrolysis for the production of nickel oxide and to recycle HCl within the solvent extraction circuit [18].

4.3 THE CHESBAR APPROACH – AAL IN A CHLORIDE MEDIUM

With the decision to proceed with a chloride-based atmospheric acid leach process, attention was then turned to examining the behaviour of iron and magnesium in the flowsheet. Clearly, the simplest and most obvious approach technically was to recycle pyrohydrolysed magnesia as a neutralizing and precipitating agent for iron as "ferric hydroxide" or amorphous ferrihydrite. However, preliminary mass balance calculations indicated that with the extent of iron leaching as reported in Table II, then each unit of magnesium would effectively have to undergo pyrohydrolysis 2-3 times. Whilst this is technically feasible, from a capital and operating cost viewpoint, it makes no sense.

Similar such circuits have been proposed previously. BHP Minerals developed a chloride-based heap leach process [19], although this was never commercialized. Nickel and cobalt were recovered from the chloride leach liquor via ion exchange with a very expensive resin, Dowex M4195, or via precipitation with recycled magnesia from pyrohydrolysis. Iron was either precipitated as a hydroxide with recycled magnesia, or itself subjected to pyrohydrolysis after solvent extraction. Magnesium was recovered as magnesia, also by pyrohydrolysis. These methods of dealing with iron, based on preliminary mass balance calculations carried out by Chesbar, as noted above, whilst perfectly technically feasible, do not appear to be so economically.

A somewhat similar process has also been proposed by Gibson and Rice [20], in which iron is removed by solvent extraction and pyrohydrolysis, similar to one of the BHP options above, and magnesium recovered by pyrohydrolysis. No data were provided on this circuit, since the focus of the paper was on the solvent extraction of nickel with Versatic 10.

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However, whilst the solvent extraction processes for iron may be technically feasible, extracting such large quantities of iron generates a significant amount of acid which still has to be neutralized, usually by recycled magnesia. As noted above, recycling magnesia equivalent to the iron leached appears to be prohibitively expensive.

Recently, another chloride-based circuits has been proposed for the recovery of nickel and cobalt amongst others [21,22]. The circuits are somewhat complex, and propose the control of iron by selective spray roasting, wherein the iron will be converted to hematite. Other metals such as magnesium, contained in the product are then recovered and removed by washing, since they remain in the chloride form. Water balance issues do not appear to have been addressed in these circuits, and the costs associated with redissolving the chloride salts from hematite also appear to render this approach unattractive.

A more acceptable method of controlling iron is to form FeOOH, either β-FeOOH (akagenite) or α-FeOOH (goethite) [23]. This approach is based to some extent on controlled supersaturation precipitation [24], and is far more attractive than, for example, the turboaeration process proposed by Great Central Mines in their chloride copper process [25].

4.4 DESCRIPTION OF FLOWSHEET

After careful consideration of all of the factors discussed above, the flowsheet of Figure 5 was arrived at. Beneficiation studies are currently underway, and initial results indicate that upgrading of about 30% should be possible. However, such is not necessary for the flowsheet, and all metallurgical testwork is being carried out on as-received ore. Of considerable interest is that leach tests on the saprolite component show that a highly magnetic material, analyzing ~50% Fe, 1-2% Ni and 4-5% Mg, and comprising 15-20% of the feed mass remains after the initial stage of leaching. Whilst Chesbar has no immediate plans for this material, it would appear, at the very least, to be suitable as a feed for a ferronickel plant, and possibly directly for stainless steel production. This material also has a potential additional benefit in that being able to market it would eliminate the need for its disposal, and would, therefore, result in a significantly reduced residue disposal area.

Figure 3 and Figure 4, generated from preliminary leach testing on the saprolitic fraction of the ore, show the effects of total chloride and initial acidity on the extraction behaviour of nickel, magnesium and iron. It is clear that there is an optimum concentration of both free acid and total chloride concentration, wherein most of the nickel is extracted, bur virtually no iron is taken into solution. The fraction of nickel that is not extracted corresponds to the magnetic fraction referred to above, with the net result that over 95% of the nickel contained in the feed ore can be recovered in one form or another.

The results also suggest that a certain fraction of the magnesium in the ore is leached, and appropriate manipulation of the initial and final leach conditions can, to a large extent, determine the amount of magnesium to be leached. Consequently, the process has been designed so that as much magnesium as is required (either for recycle or for sale) is leached, and as such, worldwide patents have been applied for. This can be achieved by recycling a significant proportion of the final magnesium chloride liquor, where the high magnesium concentration suppresses magnesium leaching. This is not the case for nickel and cobalt, however, and provided that a specific overall chloride concentration is maintained, the hydrogen ion activity from the HCl can be appropriately adjusted, with the result that the nickel and cobalt can be selectively leached.

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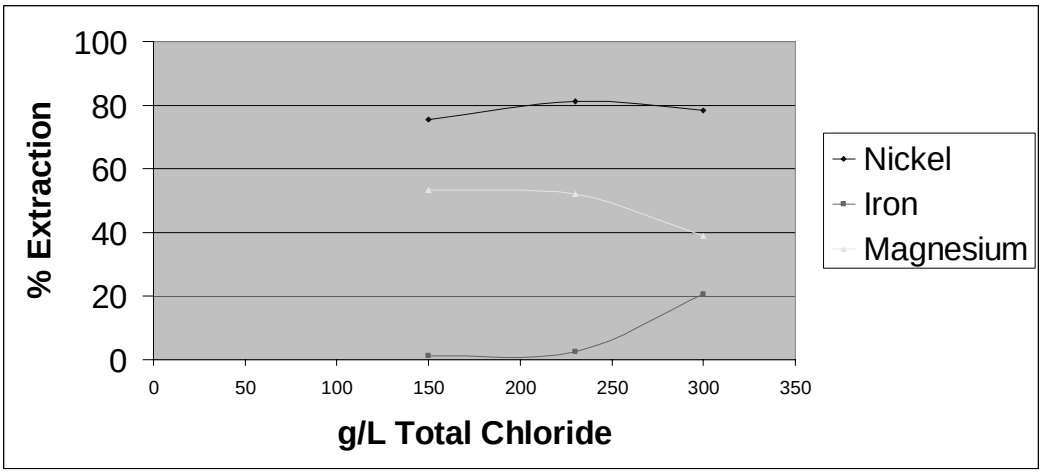

Figure 3. Metal extraction as a function of chloride at 2N initial HCl

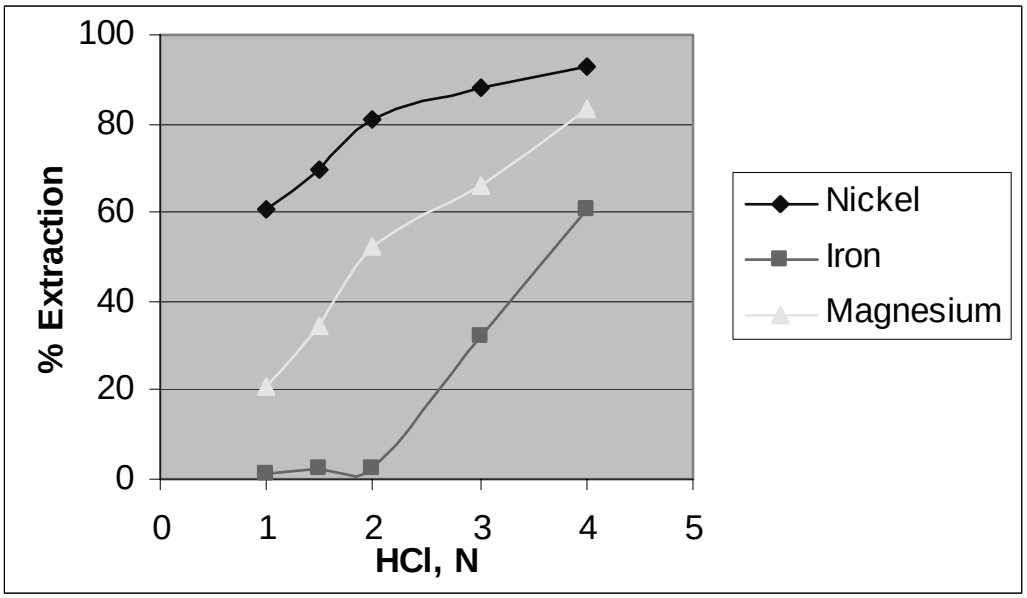

Figure 4. Metal extraction as a function of HCl concentration at 230 g/L chloride

Additionally, a countercurrent leach circuit has, at present, been adopted, as this potentially offers a further measure of control for both iron control and the degree of magnesium leaching. By appropriate control of pH and redox potential, with the ore itself, then the iron can either be prevented from leaching, or can immediately be precipitated as one of the forms of FeOOH referred to previously. A more rigorous discussion of the leach process, with additional results, will be forthcoming in a subsequent publication.

The leached material settles very rapidly, and hence solid/liquid separation is not a problem, especially if iron is leached and re-precipitated. Final purification of the leach liquor can be achieved by suitable pH adjustment with recycled magnesia to remove traces of iron, aluminium, chromium and copper, which solids can be recycled to the leach. Further increase of pH permits the precipitation of a mixed nickel/cobalt hydroxide for sale, and a very pure magnesium chloride liquor. At the present time, Chesbar is studying the market for pure magnesia, and if warranted, will make as much as can reasonably be sold within the limits of making and selling the mixed nickel/cobalt hydroxide.

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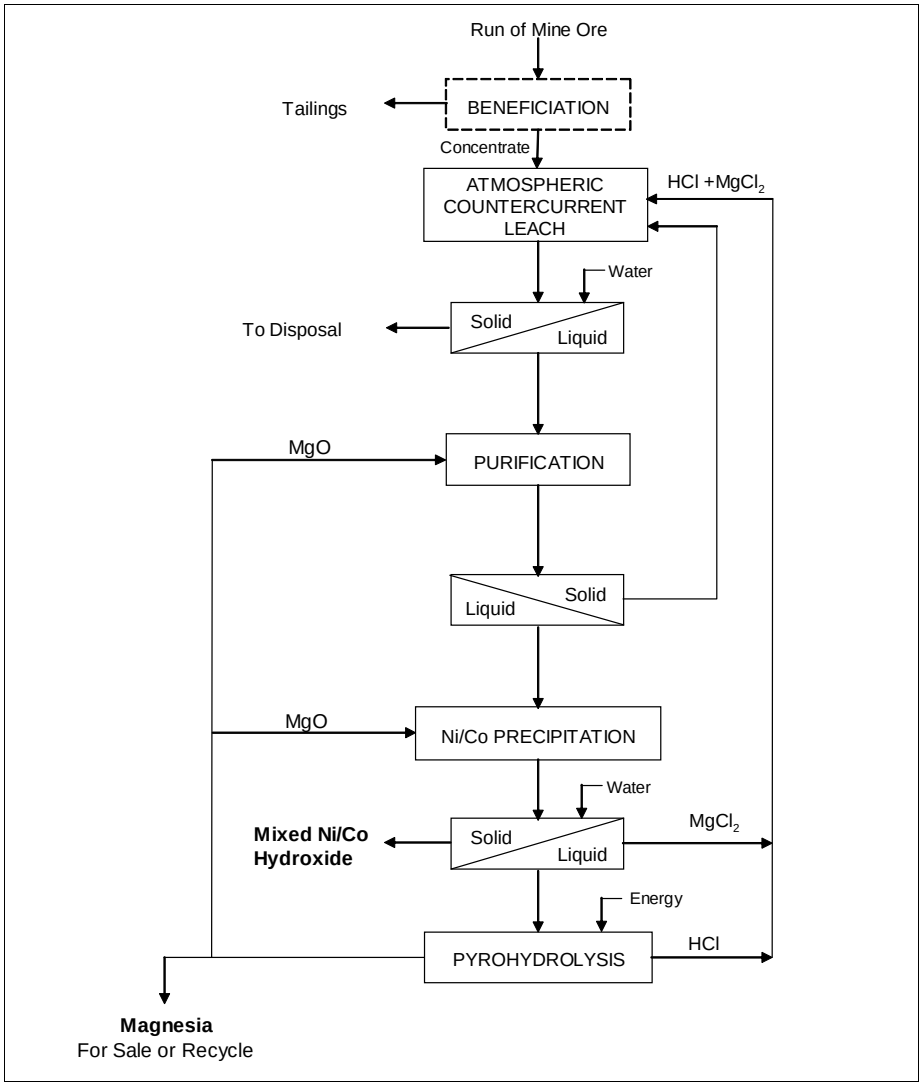

Figure 5. Chesbar Atmospheric Chloride Acid Leach Circuit - AAL

5. THE WAY FORWARD

At the time of writing, a scoping study is being completed by Hatch in Montreal. Preliminary figures indicate that both the capital and operating costs will be in the lowest quartile of current nickel industry operating plants, exclusive of any credits for cobalt, ferronickel or magnesia. These numbers suggest that the Sechol Project will therefore be extremely viable, and with this in mind, the company is moving forward towards piloting of the process in the second half of 2003. Concurrently, samples of the mixed nickel/cobalt hydroxide are being prepared for end-user evaluation, with a view to entering into off-take agreements. Most of the major refiners have expressed interest into entering into such arrangements.

6. CONCLUSIONS

Chesbar is developing an acid chloride atmospheric leach process for its Sechol property in Guatemala. These laterite deposits are geologically young and immature, and as such are highly amenable to the chloride process, which is considered to be evolutionary rather revolutionary in concept. Nevertheless, the process is sufficiently different from existing laterite circuits that Chesbar has applied for worldwide patent protection, and has the further advantage of being able to treat in one process both the saprolite and limonite horizons of the deposit.

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Preliminary cost numbers suggest that the commercial plant, slated for 2006, will be in the lowest quartile of both capital and operating costs in the nickel industry. The potential also exists for marketing a high purity magnesia for additional credits, and in so doing, turn what would normally be considered to be a problem into a considerable asset.

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NICKEL /COBALT - 9

SERVING THE GLOBAL MINING & METALLURGICAL INDUSTRIES WITH TECHNICAL EXCELLENCE & PROVEN INTEGRITY

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THE DEVELOPMENT OF THE INTEC NICKEL PROCESS TO TREAT A LOW-GRADE NI/CU/CO/PGM CONCENTRATE

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1. INTRODUCTION

Intec Ltd has patented a mixed halide hydrometallurgical process for the extraction of base metals and associated by-products from sulfidic mineral concentrates. The application of this technology has primarily focused on the Intec Copper Process (ICP), which has been developed to commercial readiness through the operation of a 350tpa demonstration plant [1]. The same mixed halide chemistry has been applied to the development of Intec Zinc Process (IZP) [2] that is the subject of a separate patent.

Most recently, the Intec process chemistry has been applied to complex ore bodies, where it permits maximum metal recovery into a low-grade bulk concentrate. All metals are leached into solution at high efficiency and subsequently converted to high purity metal and metallic by-products.

Nickel ore bodies are often mineralogically complex with economic levels of base metals (Cu and Co) and precious metals (Pt, Pd, Au, etc) often associated with the nickel. The capacity to treat low-grade bulk concentrates has the potential to significantly improve returns from complex nickel deposits. The need to find a low-cost processing route for such ores has driven the recent development of the Intec Nickel Process (INP). A laboratory-scale development program was completed in November 2002 based on a particularly low-grade nickel (3.1%)/copper (2.2%) concentrate produced from a complex nickel ore containing significant precious metals values.

The chemistry of the INP flowsheet is described in the context of the experimental results and preliminary capital and operating cost data are also presented.

2. INTEC NICKEL PROCESS FLOWSHEET

A major portion of nickel production is from nickel sulfide concentrate that is firstly smelted to nickel matte. The matte is subsequently converted to high purity nickel cathode via a number of hydrometallurgical routes, which includes well-established chloride-based hydrometallurgical processes such as Sumitomo's Matte Chlorine Leach Electrowin [3] and Falconbridge's Chlorine Leach Process [4]. The INP flowsheet shown in Figure 1 has a number of similar features to these processes and thus benefits from the existing commercial application of chloride hydrometallurgy to nickel production. In particular, conventional purification and electrowinning operations are readily applicable, however the INP differs from these processes in that the electrolyte contains both sodium chloride and sodium bromide. This mixed-halide electrolyte has the following advantages over straight chloride-based and sulfate-based electrolytes:

- Br^- is a stronger ligand than Cl^-, which in conjunction with a counter-current leach configuration allows the use of mild conditions (80-90^oC and atmospheric pressure) to leach relatively refractory minerals such as pentlandite ($NiFeS_2$) and chalcopyrite ($CuFeS_2$). Due to these mild conditions, energy requirements benefit from the use of air to control iron rather than oxygen, eliminating the need for an oxygen plant, whilst materials of construction are predominantly low-cost FRP and plastics; and
- The relatively strong oxidant $BrCl_2^-$ (referred to as Halex™) with an Eh>1000mV is generated during electrolysis. Halex facilitates the extraction of precious metals (PMs), which include gold and platinum group metals (PGMs).

The INP eliminates the need to smelt nickel concentrate to matte as high base metal and PM extractions can be achieved directly from the sulfide concentrate. Concentrates containing high levels of pyrrhotite are readily processed with the iron oxidized to form hematite with air.

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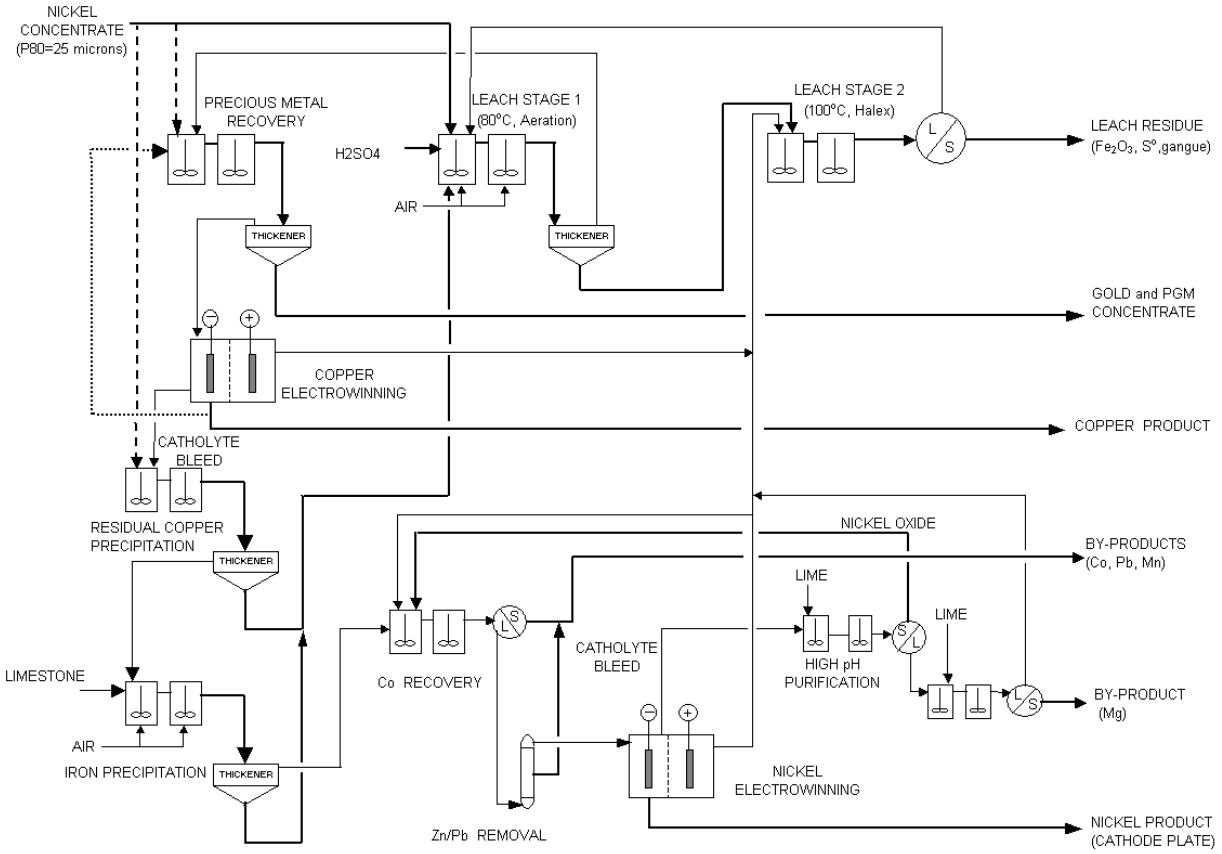
Figure 1: Simplified flow diagram for the Intec Nickel Process



In overview, the INP consists of the main circuits of leaching, precious metal recovery, copper electrowinning, purification and nickel electrowinning. The leach circuit is of a two-stage counter-current configuration with concentrate primarily fed to Leach Stage 1, whilst oxidant from electrowinning is fed to Leach Stage 2. Precious metal recovery is via precipitation to an enriched intermediate product. Copper electrowinning employs high intensity diaphragm cells with continuous product removal as is practiced in the ICP, whilst nickel is electrowon as conventional cathode plate. A degree of flexibility exists in the purification of the pregnant liquor stream, as either precipitation reactions and/or solvent extraction can be used.

2.1 LEACHING

The main functions of each of the two leach stages are as follows:

- *Aeration Leach*. The bulk of the nickel and copper are leached in this stage, with the required oxygen input for maintaining both a high solution oxidation potential (Eh>580mV versus Ag/AgCl) and rejection of iron, provided by air addition at atmospheric pressure with the temperature maintained at 80^{o}C.
- *Halex Leach*. Residual PMs are extracted in this stage by maintaining high solution oxidation potential (Eh>600mV versus Ag/AgCl), low pH (<0.5) and high temperature (100^{o}C).

2.2 PRECIOUS METAL RECOVERY

Precipitation of an intermediate PM by-product is achieved by lowering the solution oxidation potential in the presence of an active sulfide-bearing material. These conditions

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are generated by the addition of nickel concentrate rich in pyrrhotite and electrowon copper dendrites according to the following reactions:

$$FeS + 2Cu^{2+} \rightarrow Fe^{2+} + 2Cu^{+} + S \quad (1)$$
$$Cu + Cu^{2+} \rightarrow 2Cu^{+} \quad (2)$$

PMs precipitate in either their elemental state or as sulfides. An alternative source of sulfide-bearing material can be a reagent such as NaHS (NASH).

2.3 COPPER ELECTROWINNING

After the reduction leach, the clear liquor stream is fed to the cathode chambers of the copper electrowinning cells where copper dendrites are deposited at the cathode. The copper tenor of the spent catholyte is maintained at 6g/l to minimise the residual copper passing to the copper precipitation circuit. This minimises concentrate, air and limestone inputs to downstream purification operations.

A small amount of the spent catholyte is fed to the anode chambers where Halex™ ($BrCl_2^-$) oxidant is generated for return to the leach via the following reaction:

$$Br^- + 2Cl^- \Rightarrow BrCl_2^- + 2e^- \quad (3)$$

2.4 COPPER PRECIPITATION AND LIQUOR PURIFICATION

Residual copper, in the spent catholyte from copper electrowinning, is precipitated by a metathesis reaction where copper exchanges with iron from the pyrrhotite in the concentrate according to the following reaction:

$$FeS + 2Cu^{+} \rightarrow Fe^{2+} + Cu_2S \quad (4)$$

The solids from the metathesis reaction are settled in a thickener with the underflow directed to the aeration leach. Due to the above reaction, the soluble iron in the thickener overflow is approximately 3g/l. This iron is subsequently rejected by air and limestone input, resulting in the following overall reaction:

$$2Fe^{2+} + 0.5O_2 + 2CaCO_3 \rightarrow Fe_2O_3 + 2Ca^{2+} + CO_2 \quad (5)$$

The precipitated iron is settled in a thickener with the underflow sent to the Halex Leach. Removal of the trace impurities can be performed through either precipitation or solvent extraction, both of which are practiced in the chloride hydrometallurgical treatment of nickel matte. Precipitation is effected by the addition of Halex™ and alkali, with the following participating reactions:

$$2M^{2+} + BrCl_2^- \rightarrow 2M^{3+} + Br^- + 2Cl^- \ (M=Co, Fe) \quad (6)$$
$$3NiO + 2M^{3+} \rightarrow M_2O_3 + 3Ni^{2+} \ (M=Co, Fe) \quad (7)$$

A drawback of this method is the co-precipitation of nickel with the cobalt and iron. However, the concentrate used for the development program contained only low levels of cobalt and this in conjunction with 1g/l residual iron in the clear liquor stream from copper precipitation results in minimal residue mass and thus minimal nickel loss. Residual copper and lead in solution also precipitate through the addition of Halex™ and alkali. For copper precipitation, Cu^+ is oxidised to Cu^{2+} by the Halex™, which in the presence of alkali is precipitated as $Cu_2(OH)_3Cl$, whilst lead removal is by precipitation as $Pb_2(OH)_3Cl$.

The solvent extraction option has not yet been thoroughly investigated and so is not currently included in the process flowsheet.

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2.5 NICKEL ELECTROWINNING

Nickel electrowinning differs from copper in that a cathode plate is produced as per current commercial practice. The cathodic reaction is:

$$Ni^{2+} + 2e^- \rightarrow Ni \qquad (8)$$

whilst the anodic reaction is the generation of Halex™ according to Equation 1.

3. RESULTS OF LABORATORY PROGRAM

3.1 LEACHING

Concentrate Characterisation

The particle size distribution of the concentrate was relatively coarse at a P_{80} of 105µ which has the potential to affect metal extractions and hence all subsequent testwork was carried out on concentrate reground to P_{80}=25µ. The metal composition of the concentrate (see Table 1) indicated it to be a low-grade nickel/copper concentrate, with economic levels of PMs.

Element	%	Element	ppm
Ni	3.23	Co	1230
Cu	2.03	Au	0.76
Fe	27.6	Pt	3.97
S	17.4	Pd	3.93

Table 1: Elemental Analysis of Concentrate

The mineralogical composition of the concentrate was not available, but chalcopyrite was assumed to be the predominant copper mineral and pentlandite the predominant nickel mineral. Based on these assumptions, the respective iron and sulfur not associated with these minerals inferred the presence of pyrrhotite (FeS) amounting to approximately 33% of the total concentrate composition.

The calcium and magnesium oxide content of the concentrate is important, as it must be compensated for by the addition of sulfuric acid to the leach. Rather than relying on the Ca/Mg elemental assay, a sample was titrated with H_2SO_4 to a pH of 2, indicating the need for 63kg per tonne of dry concentrate.

Aeration Leach

The majority (typically >95%) of the concentrate feed to the Intec Nickel Process is added to the aeration leach, where typically >95% of each of the base metals, nickel, copper and cobalt are extracted. The results from the laboratory leach test indicated slightly better extractions as shown in Table 2. Approximately, half of each of the gold, platinum and palladium was also leached in this stage.

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Element	% Metal Extraction
Au	54
Pt	47
Pd	45
Ni	97.9
Cu	97.3
Co	97.1

Table 2: Aeration leach metal extractions

An important aim of the aeration leach tests was to quantify equipment design parameters, which are summarised in Table 3.

Design Parameter	Test 2 (P_{80}=25□)
Oxygen uptake efficiency	28%
Reactor volume (m^3/kg-Fe[1])	0.39
Filtration rate (l/m^2/h)	170

Table 3: Aeration Leach Design Parameters

Air is used to supply oxygen for the formation of hematite from the leachable iron minerals in the concentrate. Oxygen uptake efficiency is the rate-determining step for reaction, thus setting the overall leach retention time. As the oxygen input is largely related to the iron content of the concentrate, the retention time and consequently reactor volume is also a function of the iron content. Hence, the total reactor volume for the aeration leach has been expressed in terms of iron content in the concentrate feed. The filtration rate determined is the average to form a 20mm cake (equivalent to a 40mm cake for a plate and frame pressure filter).

Halex Leach

The overall metal extractions achieved in the final Halex leach stage are presented in Table 4. Additional base metal leaching in this stage further improved already high extraction to 98-99%. The primary function of the Halex Leach is to optimise precious metal extraction, with respective gold and palladium concentrations in the residue of <0.1 and <0.5ppm respectively.

Element	% Metal Extraction
Au	87
Pt	54
Pd	86
Ni	98
Cu	99
Co	98

Table 4: Cumulative metal extractions -Halex leach

Overall platinum extraction remained unsatisfactory at 54%, in spite of significant improvement in the initial figure of 16% through optimisation in the areas of electrolyte

[1] Reactor volume is defined per kg of non-refractory iron in the concentrate.

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composition, temperature, solution oxidation potential and retention time during the course of testing. The program was constrained in terms of time and further work will be carried out in the future to gain an understanding of the platinum mineralogy that should lead to improvement in extraction efficiency.

3.2 PRECIOUS METAL RECOVERY

Recycled copper product and fresh concentrate were added as reductants to the clear liquor from the Aeration Leach. The copper product reduces cupric to cuprous, decreasing solution oxidation potential, whilst the concentrate provides sulfides for PM precipitation. With decreasing solution oxidation potential, PMs were precipitated to reach a total of approximately 180ppm in the solids recovered after filtration (see Table 5).

Final Eh (mV)	Gold (ppm)	Platinum (ppm)	Palladium (ppm)
336	58	59.5	57.4

Table 5: Precious metal content of Reduction Leach Product

Optimisation of the copper and concentrate inputs to the PM recovery circuit has not been carried out. Further work in this area and trial of precipitation by NASH can potentially improve the grade of the intermediate precious metal product.

3.3 COPPER ELECTROWINNING

The copper extracted during leaching is electrowon in a diaphragm cell as a copper dendrite product from the clear solution exiting the PM recovery step. During development, a two-stage electrowinning (EW) process was tested, based on the production of high purity copper from the first stage for sale and low grade copper from the second stage for use in the PM recovery step.

In the first stage, the spent catholyte is maintained at 10g/l with the spent catholyte passing to a stripping cell where the copper tenor is essentially zero. In both EW steps, about 10% of the spent catholyte is passed to the anode chambers to generate halex for return to the leach circuit.

The clear liquor was fed to the first stage EW at a tenor of 20-25g/l and at a rate to deliver a 10-15g/l strip, which is a compromise between maximising strip and minimising product contamination. A low current density of 250A/m^2 was employed to assist the copper product purity. Analysis of copper produced indicated nickel contamination at 97ppm and iron at 62ppm, exceeding LME Grade A purity for Group 5 elements of 10ppm combined. Consequently, the refining of copper produced either onsite or externally is required and so a single stage EW was substituted, operating at a spent catholyte tenor of 6g/l copper to minimize the residual copper passing to the copper precipitation step.

3.4 COPPER PRECIPITATION AND LIQUOR PURIFICATION

The spent catholyte from copper EW passes to copper precipitation where the remaining 6g/l is precipitated via a metathesis reaction at 100^{o}C by the addition of nickel concentrate according to reaction (4). The copper tenor was reduced from 6.8g/l to 0.36g/l in five hours with analysis of the recovered solid product, which is returned to the leach, confirming copper enrichment with an increase from 2.17% to 20.9%.

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The metathesis reaction liberates iron in a 1:2 mole ratio resulting in 3.3g/l of iron reporting to the liquor, essentially 100% in the ferrous oxidation state. Air is sparged to oxidize the ferrous iron to ferric with ferric iron precipitation by pH adjustment with limestone. By this method, iron was precipitated to 0.93g/l at 80^{o}C over a 5.5 hours period. However, oxygen uptake efficiency at these low copper and iron tenors is also low and so the precipitation of the residual iron would require the addition of a small amount of halex-bearing leach liquor. This could be carried out in conjunction with the subsequent cobalt recovery step, but would result in contamination of the cobalt by-product. Both cupric and ferric ion precipitate at a pH below that of cobaltic ion and so their inclusion with the bulk of the iron precipitate is both feasible and desirable. This will probably be incorporated into the flowsheet in future.

Cobalt is recovered in the final purification step prior to nickel electrowinning. Residual copper, iron and lead are co-precipitated with the cobalt at 100^{o}C unless these are removed first as described above. After 3 hours of reaction, 98.6% of the cobalt had been precipitated as shown in Table 6, leaving only Pb at a concentration sufficiently high to exceed the LME specification for 99.8% Ni purity in subsequent electrowinning.

Element	Co	Cu	Fe	Pb
Concentration (ppm) Before	320	230	20	10
Concentration (ppm) After	4.5	5.5	3.5	8.7

Table 6: Liquor assay before and after cobalt recovery

3.5 NICKEL ELECTROWINNING

Nickel is electrowon as a cathode plate in a conventional nickel cell. Purified pregnant liquor at 65g/l nickel was fed to the cell at a rate to maintain a 50g/l nickel background for a 15g/l strip. The strip is determined by the leach slurry density that was set at 25% w/w and kept constant during the development program. Further work is required to maximize the slurry density and consequently the nickel strip. Current density was set at 300A/m^2 and the temperature maintained at 55^{o}C. Cell potential was 2.3V and the current efficiency was 95%, which translates to relatively low energy consumption of 2250 kWh per tonne nickel plated.

The purity of the electrowon nickel is presented in Table 7. The only contaminants to exceed LME specification were lead (15 ppm over the 50ppm specification) and zinc (565ppm over the 50ppm specification). Solvent extraction or ion exchange are reported as viable options [5] for zinc and lead removal, but this route has not yet been investigated.

Element	Ag	Bi	C	Co	Cu	Fe	Mn	Pb	S	Sb	Si	Zn
LME Spec. (ppm)	N/A	50	300	1500	200	200	50	50	100	50	10	50
Nickel cathode assay (ppm)	1.2	0.3	<100	45	143	90	<1	65	25	<1	<10	615

Table 7: Nickel cathode assay

4. PRELIMINARY COST ESTIMATION

A mass balance and process flow diagrams (PFDs) have been developed from the program data for an Intec Nickel plant treating a low-grade nickel concentrate to generate saleable metal and intermediate products. The Intec Nickel Process shares similar unit operations to those used in the Intec Copper Process, for which extensive cost data has been amassed. Such cost data, combined with the mass balance and PFDs, are used to

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estimate operating and capital costs for the production of 45 000 tonnes per annum of nickel. The costs can vary considerably with the mineralogy of the concentrate and thus can only serve as a general guide.

4.1 OPERATING COSTS

A detailed breakdown of the estimated operating cost is presented in Table 8. For annual production of 45 000 tonnes of nickel, the operating cost of US$45.0 million equates to US45¢/lb-Ni before consideration of by-product credits.

The operating cost estimation has been divided into the five main categories of energy, labour, consumables, maintenance and miscellaneous. Energy has been calculated by comparing each unit operations with its equivalent in the Intec Copper Process. Labour uses North American rates, which has been the basis of previous calculations. Consumable costs were estimated using the same data as used in the Intec Copper Process. Over half of the consumables cost and a quarter of the total operating cost was due to sulfuric acid, for which a price of US$120/tonne is assumed. Such a price assumes importation from overseas and thus the ability to source this reagent locally can potentially reduce overall costs to less than US40¢/lb-Ni.

Maintenance costs associated with electrowinning are calculated separately, due to their higher intensity when compared to the remainder of the plant. For the latter, a factor of 6% of direct capital expenditure has been assumed. Miscellaneous costs are those associated with gangue disposal at US$1/t for return to the main tailings dam.

4.2 CAPITAL COSTS

The capital cost for an Intec Nickel plant with annual production capacity of 45 000 tonnes of nickel is summarised in Table 9. The plant cost is estimated at US$300 million, inclusive of a 20% contingency, which equates to approximately US$6500 per annual tonne of nickel capacity. This estimate is only accurate to ± 35%. The capital cost was calculated by estimating direct costs of equipment including installation. Factors were applied to the direct cost to estimate the indirect costs, which included owner's costs, consultant services, commissioning, construction management, capital spares and transportation.

The following are the major items in each of the direct cost categories:

- **General:** Piping, electrical, instrumentation and laboratory;
- **Building Civil Works:** Lighting, fire protection, ventilation, process buildings and site works;
- **Leach:** Agitated reactors and tanks, thickener, filters, PMS handling and conveyors;
- **Grinding/Material handling:** Grinding and concentrate handling;
- **Purification:** Copper precipitation, iron precipitation, cobalt recovery and magnesium bleed;
- **Product Recovery:** Nickel electrowinning, copper electrowinning, product washing, drying and packaging;
- **Utilities:** Electrolyte makeup, reagent inventory, leach scrubber, air compression, boiler and cooling towers.

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ITEM	Cost	Quantity	US¢/lb	% of Opex	US$(000/a)
Energy	3.5 US¢/kWh	8070 kWh/t-Ni	12.82	28.2%	12 908
Labour	35-75k US$/annum	151 persons	5.50	12.1%	5 540
Consumables	N/A	N/A	17.81	39.3%	17 937
Maintenance	N/A	N/A	7.26	16.0%	7 313
Miscellaneous	N/A	N/A	1.99	4.4%	2 000
TOTAL ANNUAL OPERATING COSTS			***45.37***		**45 698**

Table 8: Estimated operating cost of treatment of concentrate

Area	Cost US$
Direct costs	
General	28 570 000
Building/Civil Works	12 170 000
Leach	39 950 000
Grinding/Material Handling	12 850 000
Purification	9 330 000
Product Recovery	75 350 000
Utilities	25 020 000
Sub Total	**202 240 000**
Indirect costs	46 530 000
Project contingencies (20%)	49 760 000
Plant Cost	**298 530 000**

Table 9: Estimated Capital Cost of 45 000tpa Intec Nickel Plant

5. CONCLUSION

The INP development to date has demonstrated its potential to treat low grade and complex nickel concentrates at low capital and operating costs.

Further development is required to optimize several of the process unit operations prior to proceeding to a pilot operation. However, each of the unit operations has been successfully demonstrated at laboratory scale.

The cost structure of the INP is sufficiently low that many low-grade nickel deposits that are currently uneconomic may be transformed into viable projects.

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