Direct Nickel Process – Breakthrough Technology

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Abstract. Direct Nickel (DNi), in partnership with PT ANTAM, is commercializing its technology, which leaches the full nickel laterite profile in nitric acid. This breakthrough technology represents significant reductions in capital and operating costs compared to current technologies.

With the ability to make Ni, Co, Fe and Mg saleable products, the recycling of the nitric acid and the use of lower grade resources, the Process has high quality sustainability credentials. Following a successful pilot plant campaign during 2013 a feasibility study is underway to build the first commercial plant in Indonesia.

1 Introduction

The Direct Nickel Process can treat the full laterite profile, not just the saprolite material favoured by the smelting route or the limonite favoured by high pressure acid leaching plants

It can treat the lower grade resources remaining after smelters have taken their pick

It can produce Ni metal or nickel oxides to feed stainless steel plants or mixed nickel cobalt hydroxide to feed existing nickel refineries

The Process also makes commercial hematite and magnesium oxide products

Waste materials are benign and much smaller in proportion to the feed material than competing processes

The Process has excellent environmental credentials

The Process has very attractive economics

It provides a cost effective method of introducing down-stream processing for Indonesia's abundant nickel laterite resources and can support the Government's goal to supply nickel to a hungry world market with a significant degree of value adding done in Indonesia.

2 The DNi Story

Direct Nickel Limited (DNi) aims to become a leading global nickel producer, using the proprietary DNi Process to access and process nickel laterite resources, primarily focused on South East Asia, where there are abundant, known, high-grade resources.

DNi was incorporated in Australia on 11 November 2005 following the identification of Drinkard Metalox Inc (DMI) and their nitric acid technology that could be applied to the treatment of laterite nickel deposits. The founders of Direct Nickel, Julian Malnic and Russell Debney concluded a series of agreements in November 2006 to create Direct Nickel Pty Ltd as the platform for the commercialisation of the DNi Process.

In the period 2007 to 2009 DNi conducted bench scale test work at DMI's laboratories in Charlotte, North Carolina, USA on a range of laterite samples with particular focus on material from Wiluna, Western Australia and Araguaia in Brazil. In mid 2009 DNi established laboratory facilities at CSIRO Minerals Research Centre in Perth, Western Australia and executed a Consulting Services Agreement with CSIRO (Commonwealth Scientific and Industrial Research Organisation).

Two engineering studies were conducted with Aker Kvaerner (PFS1) and Aker Solutions (PFS2) (now Jacobs) in 2007 and 2009 respectively. These studies established base line capital and operating costs for the Process based on, current at the time, flow sheets and test results.

In the first nine months of 2010, in conjunction with DMI, an acid recycle pilot plant was assembled in Charlotte to verify the nitric acid recovery steps of the Process. This program concluded very successfully in August 2010.

In line with the PFS2 recommendations the design of a Test plant commenced in 2010 in partnership with Teck Resources' CESL team in Vancouver, Canada. The design was moved to Perth late in 2010 following CSIRO investing in DNi. The plant design was based on the treatment of Teck's Araguaia nickel deposit in Brazil.

By August 2011, construction of Stage 1 of the Perth Test Plant was completed, in preparation for commissioning, however the ore supply from Araguaia had become unavailable and the project was put on hold during 2012 whilst an alternative feed source was secured.

In April 2012 a Memorandum of Understanding was signed with PT ANTAM, which included the supply of 100 tonne of limonite and 100 tonne of saprolite from its Tanjung Buli mine on Halmahera Island, Indonesia. The material was received in December 2012. Laboratory test work on Buli samples was conducted during the second half of 2012 to confirm the suitability of the Test Plant design to this new ore supply.

In June 2012, CSIRO committed further funding to allow the construction of Stage 2 of the Test Plant achieving the full flow sheet. As discussed below commissioning and operation of the Stage 1 flow sheet took place in the first half of 2013 followed by Stage 2 operations in the second half of the year.

In conjunction with the technical program a series of patents are in place, which along with trade secrets and know how, give the key aspects of the technology global protection.

3 DNi Process Description and Flow Sheet

The DNi Process is an atmospheric hydrometallurgical processing route designed to treat all types of nickel laterite ores, in a single flow sheet to produce a number of final saleable products. The key to the Process is the use of nitric acid as the leaching agent and the subsequent use of a patented recovery and recycling process returning +95% of the nitric acid for re-use. See below in Figure 1.

The ore is mined and then fed to a comminution plant for crushing to <2mm. It is then mixed with the nitric acid to a solids percentage of between 20 and 30% and fed to the leaching tanks heated to 110°C. The residence time in the leaching tanks is between 2-6 hours with 4 hours being typical. During the leaching around half of the mass of the ore is dissolved into the nitric acid, leaving behind the acid insoluble minerals, usually consisting of silicates, and the pregnant leach solution (PLS). The solid-liquid separation of the post-leached slurry occurs in a series of counter current decantation thickeners (CCDs). After the acid insoluble residue has been separated from the leach solution it is washed and filtered prior to disposal in a residue storage facility.

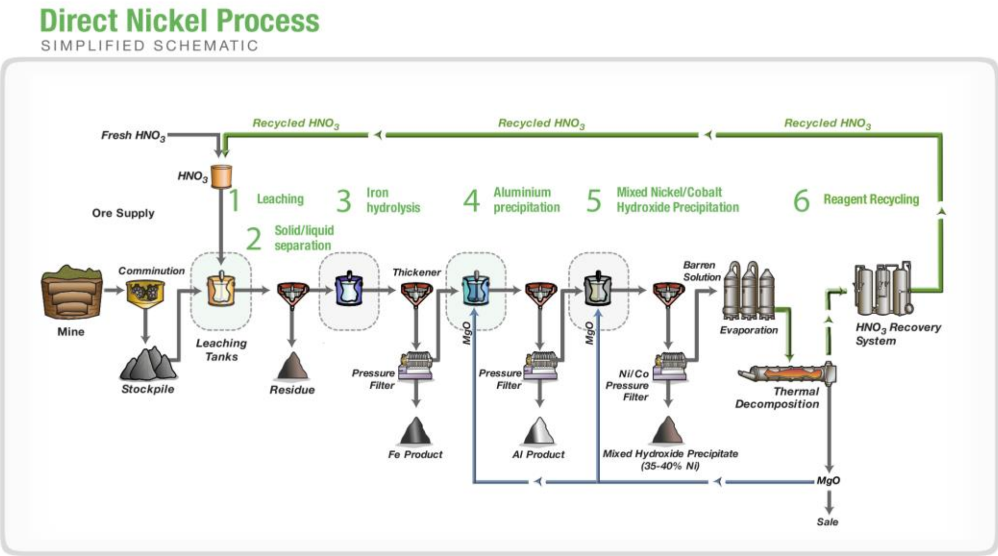

Figure 1 The Direct Nickel Process - Simplified Schematic

Following the CCD circuit the PLS is treated in the iron hydrolysis circuit where the PLS is heated in atmospheric tanks and nitric acid is distilled off for return into the nitric acid recovery system. During this distillation hematite, or Fe2O3, is formed as a solid in the tanks, effectively removing iron and chromium from the liquid. This slurry is filtered and the washed hematite filter cake is produced as a by-product for sale. The iron free solution is then treated with magnesia (MgO) slurry to increase pH and precipitate aluminium hydroxide, which is filtered to produce an Al product.

The low aluminium solution is now ready for mixed hydroxide precipitation where magnesia slurry is again used to raise pH, in a two-stage circuit, and precipitate out the mixed hydroxide product (MHP), containing most of the nickel and cobalt recovered from the feed. The slurry is then thickened to separate the solid MHP as thickener underflow from the barren solution in the thickener overflow. The MHP slurry is then filtered and washed producing MHP filter cake, the primary saleable product from the DNi Process. Alternative commercial products such as nickel and cobalt metal, nickel/cobalt oxide or nickel/cobalt sulphides can be produced with additional modules using existing proven technologies.

Subsequent to mixed hydroxide precipitation, the barren solution, containing primarily magnesium nitrate and water, is evaporated providing clean water for re-use in the Process. The concentrated magnesium nitrate, containing between 2 and 3 moles of water, is fed to a thermal decomposition unit where it is broken down into magnesium oxide (MgO) and nitrogen oxide gases. A small proportion of the MgO is reused in the Process; the remainder is available for sale. The nitrogen oxide gases are recovered through a series of absorption stages where nitric acid is formed, which is then fed back into the leach circuit.

A commercial plant will be supported by a nitric acid plant producing the small amount of make up acid required and a power plant which will produce both electricity and steam.

4 The Test Plant Program

4.1 Operations

The test plant was designed in 2 stages of construction, to fit within the availability of funding, whilst still aiming for successful operation of the full flow sheet. The first stage (Stage 1) consisted of the leach feed circuit, the leaching tanks, a 6-train CCD circuit, metal precipitation circuit, followed by the barren evaporation and nitric acid recycle. The second stage added solution purification with iron removal and aluminium removal. These can be seen below In Figure 2, with Stage 1 units coloured green and Stage 2 in blue. The test plant did not test the ore comminution step as it was outside the project brief.

The Stage 1 operation, commenced in March 2013 and demonstrated successful leaching and solid liquid separation. In addition the nitric acid recovery circuit, operating in closed loop with the leaching circuit was demonstrated, as were the barren evaporation and decomposition units.

The Stage 2 operation added three new circuits to the Stage 1 plant allowing for iron and aluminium removal before production of MHP as a final product. In other words the full flow sheet was demonstrated after the Stage 2 units were commissioned in July and August. Two lengthy continuous campaigns of 21 and 28 days were conducted during September and October 2013.

4.2 Results

The Test Plant delivered the desired outcomes, which can be summarised as follows:

- The Process proved to be simple and safe to operate on a continuous basis,
- High standards of occupational health, safety and environment were demonstrated,
- Metal recoveries for pay metals Ni, Co, Fe and Mg matched laboratory results with +90% for Ni,
- +95% recycling of nitric acid achieved,
- A working, accurate process model was developed and verified,
- 304 stainless steel was confirmed as the construction material of choice,
- Water and energy balances were confirmed,
- Process operating costs in the range US$2.00 to $3.00/lb (before credits) have been confirmed,
- MHP grades of +40%Ni were demonstrated,
- Good quality by-products were demonstrated.

4.3 Process Modeling

During the operation of the Test Plant, process data was collected to compare with earlier bench scale results and to assist in the preparation of a process model. The objective of process modelling was to establish a baseline model, grounded on chemical and thermodynamic fundamentals, to provide verification of the Test Plant

Process and its mass and energy balances. Specific objectives of the modelling process included the following:

- Simulate full closed loop process,
- Verify mass balances of key components,
- Generate water balance,
- Understand energy requirements,
- Identify information gaps,
- Provide a baseline for commercial scale-up,
- Provide a baseline for optimisation and sensitivity testing.

4.4 Materials of Construction

Stainless steel 304L and 316 are the main materials of construction used in the DNi Test Plant. Other materials used in the plant for piping and connections are Teflon and HDPE. Only two areas of the plant raised any questions about the suitability of 304L stainless steel as the chosen material of construction. These two areas were the leach tanks and iron hydrolysis vapour space. The coupon testing conducted during the program provided simple and cost effective answers to these two areas and appropriate materials can be further evaluated in future Test Plant programs.

4.5 Occupational Health and Safety (OHS)

Key OHS risks identified for the Test Plant operations were:

- Use of Nitric acid at strengths from 5% to 68%,
- Use of heat in several sections of the Process.
- The presence of molten salts and steam at temperatures from 160°C to 220°C,
- Oxides of nitrogen in gaseous forms, with temperatures up to 350-400°C,
- Potential for dust from ore and products,
- Vehicles (in the case of the Test Plant specifically fork lifts).

From an OHS perspective the Test Plant program demonstrated beyond doubt that the Process can be operated in a safe and healthy manner. Similar risks are satisfactorily managed elsewhere in the processing of nickel laterites and other base metal ores and concentrates.

4.6 Environment

The unique feature of the Process is that the leach reagent, nitric acid, is recovered and recycled from the waste streams. The efficient capture of nitrates from solutions and gases is an economic requirement of the Process, and this fact assists in reducing environmental impacts.

Furthermore, in addition to the main MHP product the DNi Process can make saleable by-products. These include an iron rich hematite product (60% Fe) and magnesia (+95% MgO). This feature results in the solids waste being limited to an acid insoluble residue and an aluminium product. Together these two solid waste streams will represent less than 50% of the original mass of feed material to the Process. This is a small amount compared to other hydrometallurgical processes for nickel laterites where the quantity of solid waste is well in excess of the initial mass of feed. The DNi Process waste solids will be inert in the environment.

Soluble nitrates are not welcome in the environment. Whilst the process water balance indicates a zero discharge operation, the issue of nitrate disposal has been addressed. If required, prior to discharge, effluent will be treated with alkaline Al rich solution, which will produce inert hydrotalcite that will fix nitrate anions. The effluent will be further polished by biological de-nitrification to meet regulatory standards. Both processes are used commercially.

Nitrogen oxide (NOX) gaseous discharges are very low in volume due to the +99% capture of NOx into useable nitric acid. The gas will assay <100ppm NOx and will meet regulatory requirements. It will contain no other contaminants such as dust, particulates or acid mists.

4.7 Products

The primary output from the DNi Process will be either a mixed hydroxide product (MHP) or a mixed oxide product (MOP). Table 1 shows product quality expected from a commercial plant treating ore from Tanjung Buli, Indonesia. Conventional technology can be used to convert MHP or MOP to Ni metal and cobalt products.

Table 1 DNi Nickel Products

Element	units	MHP	MOP
Ni	%	45.4	68.1
Co	%	1.2	1.8
Fe	%	0.5	0.7
Mg	%	2.0	3.0
Al	%	2.9	4.4
Mn	%	2.0	3.0
N	%	1.5	none

In addition to a nickel/cobalt product one of the unique features of the DNi Process is that it also yields saleable Fe and MgO products. The specifications of these products are shown in Table 2. Fine, chemically reactive MgO is sought after in the chemical and water treatment industries and lower qualities are used in agriculture. In general Fe products around 60% are readily marketable.

Table 2 DNi MgO and Fe Products

Element	Units	MgO Product	Fe Product
Mg	%	57.0	0.27
MgO	%	95.0	nr
Ca	%	1.0	<0.01
Fe	%	0.1	59.8
Fe2O3	%	nr	85.4
Ni	%	0.06	0.14
Mn	%	0.11	0.46
Cr	%	<0.02	0.35
Al	%	<0.02	0.78

5 Commercialisation and Scale Up

The DNi Process provides the step change the nickel industry needs to overcome high capital and operating costs in a low commodity price environment. The DNi Process delivers:

- "Elegant" process chemistry where reagents are regenerated and recycled within the Process,
- Ability to treat limonite and saprolite ores and blends without loss in nickel recovery,
- Low process intensity not requiring high temperatures or pressures,
- "Off-the-shelf" equipment with a well-known design and scale up methodology,
- No exotic materials of construction,
- Low volume, benign tailings that can be disposed as landfill, which are especially suited to tropical locations,
- Valuable by-products of MgO and hematite.

These features all combine to offer attractive operating and capital costs and simple operability benefits.

The Test Plant operation has demonstrated the operability of the Process as an integrated circuit. It has also confirmed the performance of a range of process control schemes across the plant, which can be further refined and built upon in a full-scale operation.

Overall, the DNi Process is significantly less arduous from an operability and maintenance viewpoint compared to a fully integrated High Pressure Acid Leaching (HPAL) Ni plant, Nickel Pig Iron or Ferro-Nickel smelters. The Process can be constructed and operated with commercially available off-the-shelf units using well-proven equipment, thus significantly reducing scale up risks.

Over the past eight years DNi has developed and implemented a plan that addresses the risks associated with commercializing a new approach to treating nickel laterites, taking into account pitfalls, traps and recommendations made by the many that have gone before.

There are two aspects of risk; first the process chemistry risk (i.e. does the chemistry work and is it understood sufficiently well to design the process) and second the equipment scale-up risk. The two aspects are often mixed together, and that creates confusion regarding scale-up.

The process chemistry risk has been addressed in the laboratory and in the Test Plant. Chemistry risk has been minimized by running a continuous Test Plant operation on the target ore, in a fully integrated circuit that contains all the process steps running in steady state for a sufficiently long time to obtain all process design data.

DNi has approached the equipment scale up risk with specific focus on people, process novelty and equipment scale-up.

5.1 People – maintaining continuity of knowledge

- The core technical team, which includes strong representation from DNi, CSIRO, Teck and RMDSTEM, has been consistent from process inception to date. This team, supported by independent consultants, has many years of experience in nitrate chemistry, designing, constructing and operating mineral and hydrometallurgical plants.

5.2 To address Process Novelty DNi has

- A corporate culture that supports thorough scientific confirmation of all process claims and design parameters,
- Completed a series of disciplined and well-designed development phases, with appropriate stage gate and risk review before and after the completion of each phase,
- Run a one-year, well-funded, Test Plant program to thoroughly test the Process on a continuous basis on a variety of ore blends,
- Processed material from the Buli resource, where the first commercial plant may well be based and
- Developed and verified a Process simulation model during the Test Plant program.

5.3 To address Equipment Scale-Up DNi has

- Implemented high quality data collection and interpretation by experienced process engineers,
- Developed a Process that is simple and elegant with the bulk of the equipment simple stirred tanks, thickeners and filters. Equipment vendors, who are skilled at scaling up equipment from small-scale test work results, have been engaged,
- Developed comprehensive design criteria that have been thoroughly researched and are supported by hard data from laboratory or Test Plant,
- Involved the thermal decomposition unit supplier, Therma-Flite, in the decomposition process testing with DNi since 2010, including the successful 2010 Acid Recycling Pilot work in Charlotte NC, USA, and the recent Test Plant program in Perth,
- Avoided the use of "first-of-a-kind" equipment,
- Confirmed, in the Test Plant, the use of 304 stainless steel as the major material of construction.,
- Re-confirmed in the Test plant the viability of the nitric acid recovery system.

DNi, by testing its new Process at pilot scale for a full year, with a highly experienced team has put in place the fundamentals to manage the inherent risks involved with commercialization of novel technology.

6 Commercial Plant Design

PT Nickel Halmahera Timur (NHT) is a 50:50 Joint Venture Company formed between PT International Mineral Capital (a subsidiary of ANTAM (Persero) Tbk ("ANTAM") and PT Direct Nickel (a subsidiary of Direct Nickel Pty Ltd), NHT is conducting a feasibility study for the first commercial plant to be established at ANTAM's Tanjung Buli operations on Halmahera Island. The key parameters for the feasibility study are shown below.

Table 3 Buli Project Key Parameters

Item	Unit	Value
Leach Feed Rate	tpa (dry basis)	1,500,000
Head Grade	% Ni	1.60
MHP Product	tpa	47,620
MHP Grade	% Ni	42
MHP Contained Ni	tpa	20,000
Nickel Recovery	%	90.4
Fe Product	tpa (dry basis)	306,000
Fe Product Grade	% Fe	60
Excess MgO Product	tpa (dry basis)	214,000
MgO Product Grade	% MgO	95
Internal Acid Recovery	%	97
Ammonia Consumption	tpa	16,700
Specific Energy Requirement	GJ/tNi produced	687
Coal Consumption (based on 17.6MJ/kg coal GAR)	tpa (wet basis)	780,000

Leaching has been conducted on a range of materials from the Buli mining district and the results built into the model. ANTAM's engineers have prepared a production schedule covering a 30 year operation. Process Design Criteria, mass, energy and water balances have been developed. A complete mechanical equipment list has been generated and initial discussions commenced with vendors.

The next step is to engage an engineering company to assist with some value engineering studies and the capital cost estimates. An additional three Test Plant runs will be conducted to confirm design parameters on more precise scheduled feed materials.

7 Conclusions

The Direct Nickel Process stands poised to move forward into commercial scale. Following 8 years of diligent development work the technology provides an elegant solution to the nickel laterites processing problems.

In parallel to the technical development strong commercial relationships have been established, particularly with PT ANTAM in Indonesia that will provide strong support during the feasibility and subsequent fund raising and construction phases.

As Indonesia looks to nurture quality nickel producing assets within its borders the DNi Process can comfortably take its place in the nations inspirational endeavours.

Acknowledgement

The authors acknowledge the strong support of their colleagues from a variety of organisations who have been so supportive over the last 8 years.

Figure 2 Test Plant-Staged Commissioning