ENGINEERING ASPECTS OF THE SELECTIVE ACID LEACHING PROCESS FOR REFINING MIXED NICKEL-COBALT HYDROXIDE

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The precipitation of mixed hydroxide is increasingly being considered as an intermediate step in the hydrometallurgical processing of nickel and cobalt. Producers currently receive roughly 75% of the value of the contained nickel and zero value for contained cobalt. In this paper, a new selective leach process for refining the mixed hydroxide is described that allows for recovery of the majority of the nickel as final metal product and realizes value for the cobalt. The features of the new process are compared with two other alternative routes (1) acid leaching followed by solvent extraction of the cobalt and (2) ammonia leaching followed by solvent extraction of the nickel. The outcomes of a process simulation for the selective acid leaching process are presented along with capital and operating cost estimates. The operating and capital costs of the process are estimated to $\pm 50\%$. For the processing of 50,000 t-Ni/y in the form of MHP, the operating cost is estimated to be \$93 million AUD (\$0.87 per lb of Ni contained in MHP) and the capital cost as defined for this study is estimated to be \$287 million AUD. A new 20 year plant processing MHP would have a payback period of less than 2 years, an IRR of over 60% and an NPV of greater than \$1.5 billion AUD. Over 94% of the total value (nickel and cobalt) contained in the MHP is extracted by the new process.

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1. INTRODUCTION AND BACKGROUND

1.1. PROCESSING OF NICKEL

Nickel reserves are largely in the form of laterite ore bodies. While laterite ore is easy to mine relative to sulfide deposits found deeper underground, efficient processing has proven to be a challenge. One of the problems is that for most nickel laterite ore bodies, the extent to which the nickel can be concentrated by physical means is limited, thus the majority of the ore must be chemically processed.

There are three main routes for chemical processing of nickel laterite ore, ferronickel smelting, reduction roast followed by ammonia leaching (the Caron process) and acid leaching (Figure 1). Ferronickel smelting is a high temperature process whereby the ore is melted and the dense iron-nickel rich component is separated from the slag by gravity. Due to the high energy requirement for heating, ferronickel smelting is limited to feed with nickel grades greater than 1.5%. Cobalt is not recovered in ferronickel smelting and the amount of cobalt in the feed that can be process is restricted to a Ni:Co ratio of greater than 30 (Oxley and Barcza, 2012). In the Caron process, nickel and cobalt are liberated from the host mineral by a reduction roast making them available for selective leaching in an ammonia / ammonium carbonate liquor (Caron 1924). While some Caron operations continue, no Caron plants have been built in recent times. In the past 20 years, acid leaching processes have been selected for treating the low grade nickel laterite ore. High temperature and pressure acid leaching is particularly suitable for the iron rich fraction of the laterite ore as the dissolved iron tends to precipitate as hematite in the autoclave making the leaching process somewhat selective for nickel and cobalt (Carlson 1961). Developments have been made in ambient pressure acid leaching (Panagiotopoulos and Kontopoulos 1988) using continuous stirred tank (Liu et al 2004) or heap leach configurations (Readett and Fox 2009). Processes that make use of acids that can be recycled such as hydrochloric acid (Harris et al 2006) and nitric acid (McCarthy and Brock 2011) are also currently being considered for the treatment of nickel laterites ores.

For the acid leaching processes, the solubilised nickel and cobalt can be concentrated and separated using solvent extraction as was practiced at Bulong in Western Australia and at Goro in New Caledonia. It is however more common to recover the nickel and cobalt from the leach solution together in a concentrated solid form by precipitation as a mixed sulphide or a mixed hydroxide.



Figure 1. Conventional nickel laterite processing routes.

1.2. PRECIPITATION OF MIXED NICKEL-COBALT SOLIDS

Precipitation of a mixed nickel-cobalt hydroxide intermediate product is a recent mainstream processing option compared with mixed sulphide precipitation. The movement away from sulphide precipitation is due to the complexity and toxicity of producing and using pressurised hydrogen sulphide gas as well as the requirement for high pressure oxidative leaching for refining the sulphide precipitate. Mixed sulphide preicipitation is more selective than mixed hydroxide precipitation for some key impurities, however, these metals can be effectively managed using the proposed refining method for feed compositions typical of mixed nickel-cobalt hydroxide precipitate.

Table 1. Comparison of nickel-cobalt mixed sulphide precipitation to mixed hydroxide precipitation

Process Features	Mixed Sulphide (Jha et al 1981) (Simons 1988) (Kyle 2010)	Mixed Hydroxide (White 1998) (Harvey et al 2011)		
Temperature /	90-120 °C	50 °C		
Pressure	200-1000 kPa H ₂ S	101 kPa Air (Atmospheric)		
Equipment	Pressurised Reactor, Gas Handling	Ambient Pressure Continuous Stirred Tank Reactor		
Precipitant	H ₂ S/NaSH, NaOH	MgO		
рН	2.5	7.5		
Selectivity for Ni,Co vs.	Fe, Al, Mn, Mg	Mg, Mn (partially)		
Reactor scaling	High	Low		
Reaction extent	>98%	~90 Stage 1, >98% Stage 2		
Single pass residence time	0.5-2 hour	~3 hours		
Seed recycle	100-400%	~100%		
Refining	Pressure oxidation leach	Atmospheric leach		

1.3. REFINING OF MIXED NICKEL-COBALT HYDROXIDE

Conventional refining of mixed nickel-cobalt hydroxide involves leaching nickel and cobalt together in ammonia ammonium carbonate solution as per the Cawse and Ravensthorpe-Queensland Nickel processes. The leached cobalt is oxidised and the nickel is then separated from kinetically inert cobalt(III) amine by selectively loading the nickel onto a cation exchange solvent extraction reagent (Mackenzie 2006).

At the ALTA conference in 2011 a new process for refining mixed nickel-cobalt hydroxide was proposed (Vaughan et al 2011). The new process is streamlined in that the separation of nickel from cobalt and manganese is carried out in the leaching stage by selectively leaching the nickel. The efficient separation is achieved by stabilising cobalt and manganese in the solid phase by oxidation. The oxidation of manganese and cobalt in the MHP matrix is fast and highly favourable with pH buffering provided by the excess nickel hydroxide (Williams et al 2013). The key technical aspects of the process have been demonstrated at the laboratory scale on a range of industrial samples.

Table 2 provides a comparison of the features of three different MHP refining routes: (1) Selective Acid Leaching as described in this paper, (2) Acid Leaching followed by solvent extraction using Cyanex 272 and (3) conventional ammonia leaching followed by solvent extraction using LIX 84I.

Feature	SELECTIVE ACID LEACH (Vaughan et al 2011) (this paper)	ACID LEACH - C272 (Dunn et al 2010) (Rishea et al 2012)	AMMONIA LEACH - LIX 84I (Mackenzie 2006) (Jones and Welham 2010)
Selectivity for nickel	Co, Mn, Cu, Fe, Al, Cr, Si	Cu, Fe, Al, Cr, Si	Mn, Fe, Al, Cr, Si
Overall Ni recovery	>98%	>98%	99%
Overall Co recovery	>98%	>98%	92-95%
Leaching robustness	High	High	Sensitive to MHP ageing, Fe, Al Oxidation Sulphate (loss of NH4 ⁺)
Leaching reagents	H ₂ SO ₄ Oxidant	H ₂ SO ₄	NH ₃ /(NH ₄) ₂ CO ₃ Reductant
Solvent extraction	n/a	Cobalt selective	Nickel selective

Table 2. Comparison of nickel-cobalt hydroxide refining processes

2. TECHNICAL-ECONOMIC ASPECTS OF THE SELECTIVE ACID LEACHING PROCESS

2.1. PROCESS FLOW DIAGRAM

The process flowsheet evaluated for an economic study of the selective acid leaching process is shown in Figure 2. In this process, nickel is selectively leached from the mixed hydroxide precipitate under weakly acidic and strongly oxidizing conditions (Williams et al 2013) and recovered as nickel metal by electrowinning (Tripathy et al 2001). If zinc is present in the MHP, an ion exchange process can be employed to remove zinc from the leach solution prior to electrowinning (Agnew et al 1988). Acid generated at the anode is recycled back to the leaching process.

The nickel and cobalt containing residue from the MHP leach stage could be sold as an intermediate product or refined further. In this scenario the residue is refined further to high purity cobalt-nickel sulphide and manganese carbonate products, similar to those proposed for the Mt. Thirsty project, as the relative concentration of manganese is high (Krebs et al 2010). The nickel and cobalt in the residue are solubilised under reductive conditions along with a portion of the manganese. The metal deportment for this type of process was recently described by Chong et al (2013).



Figure 2. Proposed refining process for mixed nickel-cobalt hydroxide (MHP). Notes: SAL Selective Acid Leach (for Nickel); RASCL Reductive Acid Selective Cobalt Leach

The input and output of a Metsim process simulation is as follows: Input:

- MHP Feed 13.8 t/h, 45 wt%Ni, 2.15 wt%Co (Dry Basis), 60 wt% solids density
- Plant Availability 92%
- Nickel in MHP Feed 50.0 kt/y
- Cobalt in MHP Feed 2,390 t/y

Output:

<u>Nickel</u>

- Ni Cathode 39.8 kt/y (79.6% of feed Ni)
- Ni in EW Bleed/Cooing Tower Vent 4.4 kt/y (8.8% of feed Ni)
- Ni in Ni/CoS 5.7 kt/y (11.4% of feed Ni)
- Ni in RASCL Residue 0.1 kt/y (0.2% of feed Ni)

Cobalt

- Co in Ni/CoS 2,354 t/y (98.5% of feed Co)
- Co in RASCL Residue 36 t/y (1.5% of feed Co)

The unit operations are described in more detail as follows with major reactions listed including reaction extents specified for the mass balance.

Selective Acid Leaching (SAL)

The majority of the nickel is selectively leached from the mixed hydroxide precipitate under oxidising and mildly acidic conditions at a temperature of 65°C. The elemental composition of the feed MHP is shown in Table 3. Oxidation with persulphate stabilises the manganese and cobalt in the solid phase, this reaction is very fast and and efficient in terms of the amount of oxidant required. The persulphate addition rate is 100% of the stoichiometric requirement to oxidise the manganese and cobalt by one oxidation state. Acid contained in the anolyte from electrowinning is used to dissolve the nickel, magnesium and some of the zinc. Calcium will tend to dissolve but is limited by its low solubility. Gypsum scaling could be managed by a combination of pre-washing of the MHP, controlled crystallisation and ion exchange. A small amount of chromium also leaches. If the pH is maintained above 4.5, leaching of copper is minimal. The processing equipment consists of three continuous stirred tank reactors in series with a total residence time of 1.25 hours. The pregnant leach solution is separated from the residual solids by high rate thickening with 40 wt.% solids in the underflow and filtered for a residue of 60 wt.% solids.

Table 3. MHP feed composition, wt. %

0	Ni	S	Со	Mn	Н	Si	Mg	Fe	Zn	Ca	Al	Cu	Cr
41.9	45	3.5	2.15	2.1	2.6	1.5	0.93	0.15	0.05	0.04	0.03	0.02	0.01

Key Reactions

$2Mn(OH)_{2}(s) + Na_{2}S_{2}O_{8}(s) \rightarrow 2MnOOH(s) + Na_{2}SO_{4}(aq) + H_{2}SO_{4}(aq)$	EXT: 100%
$2 \operatorname{Co(OH)}_{2}(s) + \operatorname{Na}_{2}S_{2}O_{8}(s) \rightarrow 2\operatorname{CoOOH}(s) + \operatorname{Na}_{2}SO_{4}(aq) + \operatorname{H}_{2}SO_{4}(aq)$	EXT: 100%
$Mg(OH)_2(s) + H_2SO_4(aq) \rightarrow MgSO_4(aq) + 2 H_2O(l)$	EXT: 100%
$Ni_{7}(OH)_{12}SO_{4}(s) + 6 H_{2}SO_{4}(aq) \rightarrow 7 NiSO_{4}(aq) + 12 H_{2}O(l)$	EXT: 90%
$Zn(OH)_2(s) + H_2SO_4(aq) \rightarrow ZnSO_4(aq) + 2 H_2O(l)$	EXT: 85%
$CaSO_4(s) \rightarrow CaSO_4(aq)$	EXT: 80%
$Cu(OH)_2(s) + H_2SO_4(aq) \rightarrow CuSO_4(aq) + 2 H_2O(l)$	EXT: 10%

Ion Exchange

Pregnant leach solution passes through polishing filtration and the pH is adjusted to 3.0 prior to the ion exchange process. In ion exchange, zinc is removed from the pregnant leach solution using DEHPA (Di-2-ethyl hexyl phosphoric acid) impregnated cation exchange resin which is highly selective for zinc over nickel Laboratory test work indicates that the zinc concentration in the catholyte can be maintained below 5 mg/L in the presence of 100 g/L nickel. The process configuration is two columns in series with a third column on standby or being regenerated. The nickel catholyte passes through a carbon filter prior to electrowinning to remove residual/minor amounts of organics (DEHPA) dissolved from the ion exchange resin.

 $ZnSO_4 + IX-H_2 \rightarrow IX-Zn + H_2SO_4$

 $CuSO_4 + IX-H_2 \rightarrow IX-Cu + H_2SO_4$

Nickel Electrowinning

The nickel electrowinning circuit is conventional with the only difference from current industrial practice being the elevated magnesium concentration in the electrolyte. It has been demonstrated that bright, smooth, coherent nickel electrodeposits can be produced with a catholyte feed of 60 g-Ni/L, 15 g-Mg/L at 60°C, pH 2.5 and current density of 200 A/m² (Tripathy 2001). These results were confirmed with an electrowinning study at the University of Queensland using a catholyte containing 85 g-Ni/L, 20 g-Mg/L, 20 g-Na/L at the same operating conditions. In the current flowsheet, evaporative cooling is employed to control the catholyte feed solution to 50°C.

Key reaction

NiSO₄ (aq) + 2H₂O \rightarrow Ni (s) + 0.5 O₂ (g) + H₂SO₄

Reductive Acid Selective Cobalt Leach (RASCL)

The remaining nickel along with the cobalt and manganese are extracted from the solid residue in the reductive leaching process. The process is carried out in two stages. In the first stage the pH is controlled to 2.5 to maximise the nickel and cobalt extraction. In the second stage the pH is adjusted to the range of 4-6 to precipitate dissolved impurity metals. The residue from this process could be recycled back to the start of the primary neutralisation stage (prior to MHP precipitation) to recover some of the small amount of nickel and cobalt in the residue. If the residue contains significant copper, a copper recovery process could also be considered.

Key reactions

 $2 \operatorname{CoO(OH)}(s) + \operatorname{SO}_2(g) + \operatorname{H}_2 \operatorname{SO}_4 \rightarrow 2 \operatorname{CoSO}_4(aq) + 2 \operatorname{H}_2 O$

 $2 \operatorname{MnO(OH)}(s) + \operatorname{SO}_2(g) + \operatorname{H}_2 \operatorname{SO}_4 \rightarrow 2 \operatorname{MnSO}_4(aq) + 2 \operatorname{H}_2 \operatorname{O}$

 $Ni_7(OH)_{12}SO_4(s) + 6 H_2SO_4 \rightarrow 7 NiSO_4(aq) + 12 H_2O$

 $Zn(OH)_2(s) + H_2SO_4 \rightarrow ZnSO_4(aq) + 2H_2O$

Mixed Sulphide Precipitation

Cobalt and nickel are recovered together as a high purity mixed sulphide by precipitation using sodium hydrosulphide. The mixed sulphide also contains a small amount of zinc sulphide. The reaction pH is controlled to 2.5 by the addition of sodium hydroxide.

Key reactions

 $2\text{CoSO}_4(\text{aq}) + 2\text{NaSH} \rightarrow 2\text{CoS}(\text{s}) + \text{H}_2\text{SO}_4 + \text{Na}_2\text{SO}_4 \text{ (aq)}$

 $2NiSO_4(aq) + 2NaSH \rightarrow 2NiS(s) + H_2SO_4 + Na_2SO_4(aq)$

 $H_2SO_4(aq) + 2NaOH \rightarrow 2H_2O + Na_2SO_4(aq)$

From discussions with various parties, high payable values can be obtained for the high purity mixed sulphide produced by this flowsheet. However, as an alternative nickel could be separated out of this stream using IX and fed back into the nickel EW to produce LME-grade nickel cathodes. It would then be possible to produce a pure cobalt product through electrowinning or precipation.

Manganese Carbonate Precipitation

Manganese is recovered as a high purity carbonate by precipitation using sodium carbonate.

 $MnSO_4$ (aq) + $Na_2CO_3 \rightarrow MnCO_3$ (s) + Na_2SO_4 (aq)

 $Na_2CO_3 + H_2SO_4 \rightarrow Na_2SO_4$ (aq) $+ H_2O + CO_2$ (g)

2.2. OPERATING COSTS

Using a basis of 50,000 t-Ni/y (2,300 t-Co/y) in the MHP feed, from the mass balance it was determined that approximately 80% of the nickel is electrowon as cathode product. Of the remaining nickel, 11% is recovered as the mixed nickel cobalt sulphide and 9% exits in the spent Ni EW (anolyte) bleed stream which can be recycled upstream. Greater than 98% of the cobalt is recovered in the nickel-cobalt sulphide. The operating costs are summarised in Figure 3 with reagent expense and power requirements being the highest. Approximately 93% of the power is used in nickel electrowinning, the cost of power used in the calculations was 14 cents/kWh. Labour costs are for a workforce of 47 full time staff. All costs are refered to in **Australian Dollars**. The total annual operating cost is estimated to be \$93,000,000/year.



Figure 3. Operating costs for refining MHP by the selective acid leaching process as described in Section 2.1.

Details of the reagents and consumables costs are shown in Figure 4. A large portion of the total reagent cost is for processing of the residue from selective acid leaching which requires sodium hydrosulphide (40 wt.% NaHS solution, \$1200/t), sodium hydroxide (50 wt.% NaOH solution, \$500/t), and sodium carbonate (solid Na₂CO₃, \$630/t). The main cost for the selective acid leaching process is the cost of the oxidant, sodium persulphate (\$1,800/t-Na₂S₂O₈). Other costs include an allowance for electrowinning consumables, assays, replacement costs for cathodes and anodes, sulphuric acid, sulphur dioxide, resin, residue/product filters, flocculants and anthracite for carbon filters.



Figure 4. Reagents and consumables cost breakdown for refining MHP by the selective acid leaching process as described in Section 2.1.

Operating costs can be reduced by:

- Remove residue leach process by selling MHP leach residue directly (>50% savings in reagents).
- On-site generation of hydrogen sulphide gas for sulphide precipitation.
- Minimise nickel to sulphide precipitation by a second stage MHP leach.
- Use a lower cost oxidant or combination of oxidants in the MHP leach.

2.3. CAPITAL COSTS

Capital costs have been estimated assuming this process is built near an existing MHP production operation in Australia. Services such as plant/instrument air, water and waste treatment have been excluded from the calculations. The direct installed capital cost is estimated to be \$155,000,000 with a breakdown by plant areas shown in Figure 5. Capital costs are clearly dominated by the electrowinning plant. Indirect costs are estimated to be \$50,000,000 for project management, common distributable and owner's costs. A contingency of \$82,000,000 was also included in the estimate for a total capital cost estimate of \$287,000,000.



Figure 5. Capital costs for refining MHP by the selective acid leaching process as described in Section 2.1.

3. ECONOMIC ANALYSIS

An economic analysis has been undertaken for a plant purchasing 50,000 tpa nickel and 2,390 tpa cobalt as MHP at a cost of 75% of the contained nickel value. The plant produces two main products, LME-grade nickel cathodes and a mixed nickel-cobalt sulphide. Manganese carbonate is also produced as a secondary product and is created to reduce the costs of disposal of manganese. In the economic analysis nickel in the bleed stream is precipitated as nickel hydroxide using magnesium oxide and is 'sold' at 75% of the contained nickel value. In a real situation this nickel hydroxide would be recycled into the front-end of the plant. The following payable amounts for the products have been used:

Product	Payable
LME-grade nickel cathode	100% Ni
Mixed sulphide	80% Ni, 80% Co
Nickel hydroxide	75% Ni

The following long-term metal and product prices have been assumed:

Product	Price (AUD)
Nickel	\$9.00 / lb
Cobalt	\$18.00 / lb
Manganese carbonate	\$0.27 / lb

Two scenarios have been considered. Scenario A includes the entire flowsheet as described in section 2.1. A reduced operation and capital cost scenario, Scenario B only includes the main nickel EW pathway and does not include the processing of the SAL residue to produce mixed sulphide and manganese carbonate. Instead, in scenario B, the SAL residue is sold at 75% of the contained nickel value and no value is obtained for the contained cobalt.

This results in the following estimate of profit:

	Scenario A	Scenario B
Revenue		
LME-grade nickel cathode	\$790M	\$790M
Mixed Ni-Co sulphide	\$165M	
Nickel hydroxide	\$66M	\$66M
Manganese carbonate	\$3M	
SAL residue		\$84M
Total revenue	\$1,024M	\$940M
Cost of sales		
Cost of MHP	\$744M	\$744M
Operating expenses	\$96M	\$62M
Total cost of sales	\$840M	\$806M
Gross profit (before tax)	\$184M	\$134M

Note: The operating expenses above includes operating expenses presented in section 2 plus \$2.6M of magnesium oxide reagent costs to precipitate the nickel bleed stream, other expenses regarding the bleed treatment were not considered.

For a 20 year plant life the following table summarises the financial overview of a project as a whole:

	Scenario A	Scenario B
Production rates		
LME-grade nickel cathode	39.8 ktpa	39.8 ktpa
Ni in mixed sulphide or SAL residue	5.7 ktpa	5.7 ktpa
Co in mixed sulphide or SAL residue	2.4 ktpa	2.4 ktpa
Ni in nickel hydroxide	4.4 ktpa	4.4 ktpa
Annual gross profit (before tax)	\$181M	\$134M
Capital cost	\$287M	\$244M
Payback	1.6 years	1.8 years
NPV (8% discount rate)	\$1,519M	\$1,071M
IRR	64%	55%

The following chart plots the sensitivity of the annual gross profit and IRR to nickel price for scenario A. Note that cobalt price is assumed to be equal to two times nickel price in this chart.



Figure 5. Annual gross profit and internal rate of return for Scenario A as a function of nickel price.

4. SUMMARY AND CONCLUSIONS

A new process for refining mixed hydroxide precipitate is described based on selectively leaching nickel at mildly acidic and strongly oxidising conditions. The new process is streamlined as the leaching of nickel and separation of nickel from cobalt, manganese and copper occurs in the same stage unlike alternative processes which require leaching followed by solvent extraction to achieve this outcome. Two scenarios were evaluated based on a feed of 50,000 t/y nickel in the form of mixed hydroxide, both of which are favourable, even at low nickel price. The impact of a smaller refinery and the location of the plant need to be carefully considered. The next stage in the process development is for a continuous integrated pilot plant campaign.

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