NICKEL AND COBALT RECOVERY FROM A BULK COPPER-NICKEL CONCENTRATE USING THE CESL PROCESS

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ABSTRACT

CESL has developed a novel hydrometallurgical process for treating Cu and Ni sulphide concentrates, including bulk concentrates. Both Cu and Ni are leached efficiently into solution, using a proprietary pressure oxidation process, along with other base metals, notably Co. A previous publication has described the Cu recovery from a low grade bulk concentrate, Mesaba, using this CESL Process, including an extensive piloting campaign. Ni and Co recovery from the same solution was investigated at the time, but not finalized. This paper will describe the latest work at CESL on Ni and Co recovery from the same concentrate.

INTRODUCTION

In 1992 CESL, a wholly owned subsidiary of Teck Resources, began developing a hydrometallurgical alternative to the smelting of Cu sulphide concentrates. Later the project was expanded to include process development on other base metals, as well as precious metals recovery from these concentrates when warranted.

The Cu process utilizes a unique technology for pressure oxidative leaching (PO), together with the well established SX/EW process, modified to suit concentrate leaching. Details of the process flowsheet have been presented previously [1].

The CESL Process has been extensively tested using a variety of concentrates at bench, pilot, and demonstration plant scales. Recently, Vale built a small Cu refinery in Brazil known as Usina Hidrometalúrgica Carajás plant (UHC), with a capacity of 10,000 tpy cathode which uses CESL technology. The UHC plant was commissioned in 2008, and is now in its optimization phase [2]. The applicability of the CESL process to Ni, and bulk Cu-Ni concentrates has also been explored. Extensive bench and pilot scale testwork has been undertaken periodically since 1995 [3-4].

Previous CESL Ni Process Leach Results

The CESL Ni Process uses similar pressure oxidation conditions to the Cu process. Ni sulphides all oxidize very efficiently under these conditions, but Ni behaves differently to Cu in that it mostly goes into solution in PO, and seldom requires a secondary atmospheric leach.

CESL has examined several Ni and bulk Cu-Ni concentrates at the bench and pilot scale. Table 1 presents metallurgical results from five different concentrates, labeled concentrate A through E. Despite the wide variation in Ni grade between these concentrates, extractions in excess of 97% were realized for all samples with the exception of the low-grade bulk concentrate. Note that invariably bench results give 0.5-1.0% better Ni extraction than pilot due to the exclusion of recycles, with this effect diminishing with higher Ni grade concentrates due to smaller aqueous recycles.

| Concentrate | Concentrate Grade | | | Metallurgical Results | | | |
|-------------|-------------------|------|------|-----------------------|---------|-----|--|
| | % Ni | % Cu | % Co | Ni Ext. | Co Ext. | SOx | |
| А | 20.4 | 1.8 | 0.85 | 97.2% | 98.9% | 11% | |
| В | 19.2 | 0.11 | 0.29 | 98.1% | 97.5% | 5% | |
| С | 15.6 | 0.05 | 0.53 | 97.7% | 97.0% | 9% | |
| D | 3.7 | 8.0 | 0.15 | 98.0% | 99.2% | 3% | |
| Е | 2.1 | 18 | 0.09 | 96.4% | >98.5% | 8% | |

Table 1 – CESL Ni Process Bench Metallurgical Results

Sulphur oxidation at CESL is defined as the percentage of sulphur in the concentrate that is oxidized to sulphate. The amount of sulphur oxidized affects numerous aspects of the flowsheet, and low sulphur oxidization is usually desirable, if not essential for an economic process. Most importantly, sulphur oxidation has a large impact on the autoclave heat balance, the resultant solids density in the vessel, and subsequently the autoclave size and all associated equipment including feed and discharge circuits. In addition to the effect on capital cost, the amount of sulphur oxidation affects the overall autoclave oxygen consumption, as well as the amount of acid and soluble Fe that must be neutralized with limestone. Thus the operating cost is also strongly dependent on the degree of sulphur oxidation. These effects are so strong that CESL believes low sulphur oxidation is essential for an economic process. For the concentrates tested with the CESL Nickel Process, sulphur oxidation numbers were generally low, under 10 %.

The concentrate denoted 'E' in Table 1 represents recent results achieved while processing the Mesaba concentrate. CESL has done pilot plant testwork, examining the application of the CESL Process to Mesaba, a low grade, bulk Cu-Ni-Co concentrate, which is the subject of this paper.

MESABA PROJECT

The Mesaba Cu-Ni deposit of Teck Resources lies within the Duluth Complex of Northern Minnesota, and contains an estimated historic (non 43-101 compliant) open pit resource of 1.2 billion tonnes grading 0.43% Cu and 0.09% Ni. This region contains several similar large Cu-Ni deposits, some of which have significant values in PGE. Access and infrastructure is excellent as the property is located immediately adjacent to Cliffs' Northshore Iron Ore Mine.



Figure 1 - Location of Teck's Mesaba Property

The mineralogy of the Mesaba ore makes it challenging to produce a marketable Cu concentrate at reasonable recoveries due to the fact that the Cu frequently occurs as the Cu-poor sulphide mineral, cubanite, $(CuFe_2S_3)$, which can yield a theoretical maximum grade of only 23% Cu. In addition the cubanite is often tightly interlocked with the main Ni mineral, pentlandite, thus further reducing the Cu grade and introducing a deleterious impurity for the Cu smelter. Ni recovery to a marketable concentrate is virtually impossible for similar reasons. Some of the Cu does occur as chalcopyrite and therefore Teck is investigating the limited production of a marketable Cu concentrate at modest Cu grade, as well as a low-grade bulk Cu-Ni concentrate from Cu-rich parts of the Mesaba deposit [5].

The processing of the Mesaba ore to a single bulk Cu-Ni concentrate has been achieved at reasonable Cu and Ni recoveries; such a product would be ideally suited to on-site processing rather than selling to a smelter. Teck Resources initiated studies to determine the amenability of the Mesaba bulk concentrate to the CESL Process, with the eventual aim of producing refined Cu and Ni onsite.

Previous CESL Mesaba Testing

In September 2001, CESL started pilot plant work on the bulk Mesaba concentrate that was produced earlier that year, with grades of 14% Cu, 1.7% Ni and 0.08% Co. The results from this campaign have been documented previously [6] and will only be briefly reviewed. Considerable work went into developing the Cu portion of the flowsheet, and while the leached Cu was recovered effectively using the SX/EW portion of the CESL technology, recovery of Ni and Co from the leach solution was carried out by

lime precipitation, resulting in a low grade MHP contaminated with gypsum. The decision to first produce a (potentially) saleable intermediate for Mesaba prior to the eventual (intended) refining to metal simplified the Ni flowsheet and thus the overall process for more immediate consideration. This flowsheet was operated successfully at pilot scale from February through July 2002. The design basis for the pilot plant was 5 kg/d Ni to a mixed hydroxide intermediate. Table 2 outlines the key results from the pilot testing program.

| 5 | | | υ |
|-------------------|--------|--------------------|----------|
| Parameters | Result | Parameters | Result |
| Cu Extraction | 95% | Gross Oxygen Ratio | 0.21 t/t |
| Ni Extraction | 91%* | Net Oxygen Ratio | 0.17 t/t |
| Co Extraction | 90% | Autoclave Solids | 235 g/L |
| Sulphur Oxidation | 5.2% | Autoclave RT | 60 min |

Table 2 - Major Process Parameters from 2001-02 CESL Pilot Test Program

* With recycle of 40 g/L Ni Cu SX raffinate to PO; once through bench results showed Ni ext. >95%

In the fall of 2002, the whole Mesaba Project was put on hold due to low metal prices.

MESABA CESL PROCESS FLOWSHEET DEVELOPMENT

Further development of the purification and recovery stages in the CESL Ni Process resumed in late 2006. This was driven by both a renewed interest in the Mesaba property as an economically viable Cu-Ni deposit, and also by some new ideas at CESL on producing a better intermediate product.

Study of the industrial production and marketing trends assisted in the definition of a Ni flowsheet that was developed from bench evaluations, progressing to small scale semi-continuous operations, and culminating in a fully continuous pilot campaign. The testing defined a conceptual Ni flowsheet for Mesaba, which consisted of the following unit operations: Neutralization, 4° Cu SX, Fe and Al Removal, Sulphide Precipitation for Zn, Cu and Cd, Mixed Hydroxide Precipitation (with magnesia), Ni Scavenging, and Mg Precipitation.

Integrated Pilot Plant Campaign

Based on the success of the semi-continuous testing, an integrated Mesaba Cu-Ni pilot plant operated in Q4-2008 through Q1-2009. The plant ran continuously on a 24-hour per day, 7-day per week schedule. The design basis for the plant was processing 144 kg/d concentrate through to 26 kg/d Cu cathode and 3.0 kg/d Ni contained in a mixed hydroxide. The major objectives of the operations were to:

- Confirm and improve upon process metallurgy;
- Confirm flexibility of the CESL flowsheet by processing Mesaba bulk concentrates at varying mineralogical composition and grade;
- Operate the plant in a fully integrated manner ensuring that all recycles were continuous;
- Produce Ni-Co intermediate samples for potential customers;
- Collect environmental samples for long-term residue stability testing; and
- Optimize and collect solid-liquid separation engineering data.

Table 3 summarizes the pilot plant production data for the Mesaba campaign, including the commissioning period which consisted of only leach plant operations (PO, SX, EW) in order to build the inventory of Ni bleed solution to a target Ni tenor of 24 g/L.

| 7572 | Sulphide Ppt. Cake | 31 |
|------|----------------------------|--|
| 6966 | Mg Removal Residue | 235 |
| 460 | Cu Cathode | 1245 |
| 296 | MHP Cake | 135 |
| | 7572 6966 460 296 | 7572 Sulphide Ppt. Cake 6966 Mg Removal Residue 460 Cu Cathode 296 MHP Cake |

Table 3 – Pilot Plant Production Data (kg)

There are four residues produced from the Mesaba CESL Process flowsheet. Pressure Oxidation (PO) residue, containing mainly hematite and elemental sulphur, is the principal residue. Gypsum, metal hydroxide and a sulphide precipitate were produced in much smaller quantities. During the campaign, each of the Mesaba residues were collected on a daily basis for environmental stability testing.

The process flowsheet and key findings from the 2008-09 Mesaba pilot plant campaign are discussed in the following section.

MESABA CESL REFINERY FLOW DIAGRAM AND PILOT PLANT RESULTS

The integrated Mesaba flow diagram is presented in Figure 2. The plant processes a bulk concentrate through to Cu cathode and a Ni-Co hydroxide intermediate.



Figure 2 - Simplified Mesaba CESL Process Flow Diagram

Feed Material

A bulk sample of Mesaba ore was gathered from the Mesaba property in the fall of 2008. From this sample, nine tonnes of concentrate were produced at the Coleraine Minerals Research Laboratories in Coleraine, Minnesota. Various grades of concentrates were produced; with the following table presenting the concentrate feed composition from three distinct periods of operation.

| Period | Cu (%) | Ni (%) | Co (%) | Fe (%) | Mg (%) | S (%) | Zn (%) | Pt (g/t) | Pd (g/t) | Au (g/t) | Ag (g/t) |
|--------|-----------|-----------|-----------|-----------|-----------|----------|-----------|-------------|-------------|-------------|-------------|
| А | 13.9 | 1.64 | 0.08 | 26.7 | 2.3 | 21.3 | 0.07 | 0.83 | 2.34 | 0.73 | 57 |
| В | 19.0 | 2.30 | 0.10 | 30.6 | 2.0 | 26.2 | 0.07 | 0.84 | 1.88 | 0.78 | 52 |
| С | 21.7 | 2.36 | 0.10 | 31.6 | 1.5 | 28.7 | 0.08 | 0.92 | 1.97 | 0.82 | 38 |

Table 4 – Composition of Bulk Concentrates (2008-09 Mesaba Campaign)

Note that there was no precious metal recovery during the CESL campaign, using these concentrates. Table 5 provides a brief analysis of the gross concentrate metal value during period C of operations, with results indicating that about 95% of the value is attributed to the base metals.

Table 5 – Mesaba Concentrate Contained Metal Value*

| | Cu | Ni | Со | Pt | Pd | Au | Ag |
|---------------------|-------|-------|------|------|------|------|------|
| Value (US\$/t conc) | \$861 | \$312 | \$33 | \$27 | \$13 | \$21 | \$13 |
| Distribution (%) | 67% | 24% | 2.6% | 2.1% | 1.0% | 1.6% | 1.1% |

* Based on the following metal values: Cu @ \$1.80/lb, Ni @ \$6/lb, Co @ \$15/lb, Pt @ \$900/oz, Pd @ \$200/oz, Au @ \$800/oz & Ag @ \$11/oz.

The mineralogy of the 2008 concentrate samples was considerably different to the cubanite-rich concentrate processed at CESL in 2001-02; in 2008 the average chalcopyrite to cubanite ratio was 1.8:1. The change in mineralogical composition was due to a different sampling location of the bulk ore, which enabled further examination of the leaching characteristics of concentrate produced from a different area of the deposit. Exploration work is ongoing to further define the chalcopyrite to cubanite ratio throughout the deposit.

The following table presents the mineralogical composition of the concentrate processed in 2001 and the high-grade concentrate processed during period C of the 2008-09 campaign.

| Concentrate | Chalcopyrite (%) | Cubanite (%) | Pentlandite (%) | Bornite (%) | Pyrrhotite (%) | Gangue (%) |
|-------------|---------------------|--------------|-----------------|-------------|-------------------|---------------|
| 2001 | 10 | 45 | 5 | Trace | 5 | 35 |
| 2008 | 50 | 28 | 7 | 0.3 | 2 | 12 |

Table 6 - Mesaba Concentrate Mineralogy

The 2008 Mesaba concentrate sample was shown by MLA to consist mainly of chalcopyrite and cubanite with minor amounts of pentlandite, pyrrhotite, phyllosilicates, Fe-Mg silicates, plagioclase and hematite. Chalcopyrite accounted for 71% of the total Cu, compared with cubanite at 28.2% and the remainder as bornite. Pentlandite hosted more than 99% of the Ni, while the proportion of silicate-hosted refractory Ni was negligible [5].

Pressure Oxidation

One of the key unit operations in the CESL Process is Pressure Oxidation (PO) in an autoclave, where the Cu and Ni sulphide minerals are oxidized to form acid-soluble forms of Cu and Ni. During Pressure Oxidation the primary objective is to maximize metal extraction while minimizing sulphur oxidation.

The Cu in the Mesaba concentrate is mainly contained in chalcopyrite and cubanite. The oxidation reactions of these minerals are shown below:

$$12 \text{ CuFeS}_2 + 15 \text{ O}_2 + 12 \text{ H}_2\text{SO}_4 \rightarrow 12 \text{ CuSO}_4 + 6 \text{ Fe}_2\text{O}_3 + 24 \text{ S}^\circ + 12 \text{ H}_2\text{O}_4$$

$$CuFe_2S_3 + 2O_2 + H_2SO_4 \rightarrow CuSO_4 + Fe_2O_3 + 3S^{\circ} + H_2O$$

The Ni in the Mesaba concentrate is mainly contained in pentlandite, with the oxidation reaction of pentlandite shown below:

4 (Ni,Fe)₉S₈ + 45/2 O₂ + 18 H₂SO₄
$$\rightarrow$$
 18 NiSO₄ + 9 Fe₂O₃ + 18 H₂O + 32 S⁶

Pyrrhotite in the concentrate is oxidized almost quantitatively to hematite and elemental sulphur, without any sulphate formation. This reaction is very fast, has only a minor effect on the overall processing cost, and generates an ideal "gangue" material or dilution mineral for the concentrate. The oxidation of pyrrhotite is shown below (for simplicity, it is assumed that the Fe to sulphur ratio is one):

$$4 \text{ FeS} + 3 \text{ O}_2 \rightarrow 2 \text{ Fe}_2 \text{ O}_3 + 4 \text{ S}^3$$

In addition to the sulphide minerals, 5-10% of the elemental sulphur oxidizes:

$$S^{\circ} + H_2O + 1.5 O_2 \rightarrow H_2SO_4$$

Fortuitously, there is only about 0.2% pyrite within the Mesaba concentrate: pyrite generally adversely affects the economics of the process as it leads to increased sulphur oxidation.

Chloride increases reaction kinetics in the autoclave and works as a catalyst to ensure complete oxidation of the sulphide minerals to form elemental sulphur, as shown above, whilst minimizing sulphate formation. Therefore, the entire plant process liquor inventory has a chloride concentration of approximately 8-12 g/L.

Operating conditions for the Mesaba Pressure Oxidation circuit were established during the 2001-02 process development campaign [6]. The autoclave retention time was 60 minutes at a total pressure of 1,380 kPag and a temperature of 150 $^{\circ}$ C.

A particular advantage of the CESL Process is that the Pressure Oxidation operates in a relatively low acid environment, where the typical product liquor has a pH of 1.5 - 2.5. This results in low [Fe] in autoclave discharge liquor, as soluble Fe hydrolyzes under these conditions to hematite, leaving only modest amounts of Fe in solution. Under the conditions employed on this campaign, typical product liquor contained about 1 - 2 g/l Fe, despite Fe being the predominant element in the concentrate. This feature results in reduced Fe removal costs during subsequent purification compared with what is required with more acidic processes, where much of the Fe is put into solution during the leach.

Metallurgical Results

Table 7 presents a summary of the major metallurgical results from the campaign. Overall metal extraction results were in line with those achieved during the previous pilot campaign, with improvements made to both Ni and Co extraction through optimization of the acid feed stream composition.

Achieving 6.9% sulphur oxidation in the Pressure Oxidation circuit assisted in minimizing oxygen and limestone consumption within the plant, and allowed for a concentrated slurry in PO (i.e. reduced volume of slurry and thus higher throughput for a given vessel size).

| Parameters | Result | Parameters | Result |
|---------------|--------|---------------------------------------|--------|
| Cu Extraction | 95.0% | Co Extraction | 97.4% |
| Cu in Residue | 1.17% | Mass Loss, Concentrate to Residue | 8.0% |
| Ni Extraction | 95.2% | O2 Ratio – Net (t O2 / t concentrate) | 0.23 |
| Ni in Residue | 0.12% | Sulphur Oxidation | 6.9% |

Table 7 - Major Metallurgical Results from 2008-09 CESL Mesaba Campaign

Cu SX/EW

The Solvent Extraction (SX) circuit is the purification step in the CESL Cu flowsheet. The SX circuit enables the production of high purity Cu cathode in electrowinning by selective transfer of Cu from a pregnant leach solution (PLS) to a Cu electrolyte. Mesaba operations required two extraction circuits, 3° and 4° . The 3° SX circuit extracted 96% of the Cu leached in the PO circuit. Of the resultant raffinate, containing about 65 g/l free acid, 80% was recycled to the PO circuit and 20% was sent to the Ni circuit. After Neutralization, the smaller 4° SX circuit processed the solution to remove almost all of the remaining Cu. The SX circuit targeted Cu tenors in the 3° and 4° Raffinates of 8 g/L and 0.5 g/L, respectively. Impurity transfer across SX to the electrolyte was low during the entire campaign, with no impurity issues in the EW circuit. During the campaign, 1245 kg of Cu cathode were produced at a current efficiency of 96.8%.

Neutralization

The purposes of the Neutralization stage are to remove sulphuric acid (H_2SO_4) from solution with limestone and to re-leach Ni and Co metal hydroxides recycled from the Ni Scavenging and Aluminum Removal circuits. The 3° Neutralization circuit processed 20% of the Cu SX raffinate stream, with the product stream (Table 8) sent to the Ni purification and recovery circuits. The composition of the gypsum produced in 3° Neutralization is shown in Table 9 below.

| Al | Cd | Co | Cu | Fe | Mg | Mn | Ni | Zn |
|-------|--------|-------|-------|-------|-------|-------|-------|-------|
| (g/L) | (mg/L) | (g/L) |
| 1.7 | 7 | 0.94 | 9.0 | 1.8 | 11 | 0.27 | 23 | 0.86 |

Table 8 – Average 3° Neutralization Product Solution Composition

| Cu (%) | Ni (%) | Fe (%) | Al (%) | Ca (%) | S (%) |
|--------|--------|--------|--------|--------|-------|
| < 0.02 | < 0.02 | 0.09 | 0.03 | 22.3 | 17.3 |

Table 9 – 3° Neutralization Solids Composition

The gypsum produced was virtually free of contaminants, indicating that all elements within the Ni Scavenging and Aluminum Removal recycles were re-leached into solution, thus minimizing Ni and Co losses to this byproduct.

Fe/Al Removal

Fe and aluminum were removed from solution to less than 5 mg/L in a two-stage precipitation circuit. The impurities were precipitated to minimize contamination of the MHP product and to reduce magnesia consumption. Each stage operated at 40° C and had a 2-hour residence time, with air sparging to promote ferrous to ferric oxidation. The pH was controlled using limestone to a pH of 3.7 in Stage 1 and 5.0 in Stage 2.

The residue solids from the first stage contained the bulk of the Fe and aluminum, with minimal Ni and Co values, and were a residue stream from the process. The second stage of Fe/Al Removal can tolerate some Ni loss to improve MHP product quality as the residue solids are recycled to 3° Neutralization. The recirculating load of Ni, Co and Cu in the second stage thickener U/F is included in Table 10. Note that recycling the Cu improves overall Cu recovery within the plant as the 3° Neutralization product feeds 4° SX.

| Table | 10 - | Recirculati | ng Load | l of Metals | from Fe/A | Removal | Circuit Stage | 2 |
|--------|----------|-------------|---------|-------------|-----------|---------|---------------|---|
| 1 4010 | . | | | · or meters | | 1 | en eare stage | - |

| Metal Deportment | Metal Precipitated |
|------------------|--------------------|
| Ni | 7% |
| Co | 3% |
| Cu | 79% |

As documented elsewhere [7], testing on the second stage of the circuit indicated that Cu in the feed solution aided in Fe oxidation and precipitation. Figure 3 shows the effect of feed Cu concentration on both ORP and Fe precipitation.



Figure 3 - Relationship between Cu in Feed Solution and Fe Precipitation

Sulphide Precipitation

Zn, Cu, and Cd are precipitated selectively with hydrogen sulphide prior to Mixed Hydroxide Precipitation and effectively removed these important impurities to target levels of <1ppm in solution. Ni precipitation was very low, averaging 0.15%, while Co precipitation was more significant at 1.1%. The solids in Sulphide Precipitation had a typical Zn grade in excess of 40% (see Table 11), which should enable the disposal of this byproduct to Zn plants.

Table 11 - Sulphide Precipitation Solid Composition

| Cd (%) | Co (%) | Cu (%) | Ni (%) | S (%) | Zn (%) |
|--------|--------|--------|--------|-------|--------|
| 0.53 | 0.58 | 10.0 | 1.4 | 33.0 | 43.4 |

Mixed Hydroxide Precipitation

Ni and Co were recovered from the process solution through the addition of calcined MgO obtained from Queensland Magnesia. The MHP circuit consisted of five cascading reactors that operated at 50° C with a residence time of 4 hours.

The composition of the MHP solids produced during the campaign is shown in Table 12. The high-quality MHP product will be sold to a third party refinery. As, Cd, and Cr tenors were all less than the detection limit. A discussion on the major constituents within the MHP solids is provided below.

| Ni | Co | Al | Cl | Cu | Fe | Mg | Mn | S | Zn |
|------|-----|-------|------|-------|------|------|------|------|-------|
| (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) | (%) |
| 46.0 | 2.0 | 0.044 | 0.11 | 0.020 | 0.10 | 0.78 | 0.68 | 4.05 | 0.018 |

Table 12 - Average MHP Composition from Mesaba Pilot Plant Campaign

• <u>Nickel</u>

The Ni grade in the product solids averaged 46.0%, compared to a theoretical 63.3% for pure Ni hydroxide. Dilution from Co, Mg and Mn partially accounts for the lower grade material. However, the association of sulphate with the mixed hydroxide further lowered the maximum Ni grade. Sulphur levels in the product averaged 4.05% due to the formation of sulphates.

Samples of the mixed hydroxide product were submitted for thermogravimetric analysis (TGA). Based on the weight loss from the TGA, and assays of samples dried over a range of temperatures, it was found that the Ni within the MHP solids are consistent with a formula of $Ni_5SO_4(OH)_8.5H_2O$, so that the product may be more accurately described as a Ni/Co hydroxysulphate.

• <u>Sulphur</u>

Bench tests were completed to determine if the MHP sulphur content could be reduced by lowering the MHP feed solution sulphate concentration. As shown in Table 13, three magnesia precipitation tests were performed with varying sulphate to chloride ratios in a feed liquor containing 10 g/L Ni. Feed solutions were prepared by dissolving reagent grade Ni hydroxide with sulphuric and hydrochloric acid to attain the target feed anions concentrations. No attempts were made to optimize magnesia dosage, as the same quantity was added to each of the tests. Table 13 provides product solid assays corresponding to varying feed anion ratios.

| Feed soluti | ion anions | Product solid assays | | | | | |
|-------------------|------------|----------------------|------|------|------|--|--|
| % SO ₄ | % Cl | % S | % Cl | % Ni | % Mg | | |
| 0 | 100 | 0.3 | 4.1 | 43.6 | 2.6 | | |
| 20 | 80 | 3.1 | 0.4 | 43.5 | 1.3 | | |
| 50 | 50 | 3.6 | 0.1 | 43.0 | 1.2 | | |

Table 13 - Effect of Feed Solution Sulphate Tenor on Solids Sulphur Content

Of interest is that the test without sulphate in the feed solution still indicated 0.3% sulphur in the product. An analysis of the water used for the synthetic solution make-up indicated 60 ppm sulphur present, of which all deported to the product solids, indicating the experimentally produced Ni hydroxide effectively scavenged the trace amounts of sulphate out of solution.

The testing proved unsuccessful as it showed that chloride would replace sulphur, to varying degrees, within the Ni hydroxide matrix. Thus, perhaps all MHP commercial products would in fact be a hydroxysulphates and/or a hydroxychlorides, placing a limit on the practical end use of the product.

• <u>Chloride</u>

As documented elsewhere [8], chloride forms a water insoluble complex within MHP solids. In an attempt to reduce the chloride content, the MHP solids were repulped with caustic.

During pilot plant operations, the primary MHP cake was repulped with wash filtrate to 50% solids. A dosage of 1 kg caustic per tonne of MHP product was added to the repulp tank and mixed at 25° C for 60 minutes. The resultant slurry was filtered to a secondary cake, and underwent a 3-stage wash at a wash ratio of 1.1 L/kg to displace the soluble chloride using process water.

The following table shows the pilot plant caustic wash results that indicate 71% of the insoluble chloride was removed from the MHP solids. The inclusion and degree of a caustic wash into the overall Mesaba flowsheet will depend on the off-take agreement chloride specification.

Table 14 - Chloride Associated with MHP Solids

| Insoluble Cl in 1° Cake | 0.21% |
|-------------------------|-------|
| Insoluble Cl in 2° Cake | 0.06% |
| Total Cl in 2° Cake | 0.11% |

<u>Magnesium</u>

Overall magnesia utilization was 96% with a residual Ni tenor of 0.5-1.0 g/L in the product stream. Mg utilization improved at the pilot plant scale compared to bench scale, with the Mg grade decreasing to 0.78% from 1.4%. It is suspected that the pilot plant MHP solid recycle ratio of 200% assisted in increasing magnesia utilization, and hence increasing the Ni grade.

Manganese

As the Ni to Mn ratio in the feed solution was high at 67, Mn rejection was considered secondary to maximizing magnesia utilization and minimizing the recirculating load of Ni. As a result, the majority (75%) of the Mn that fed the MHP circuit deported to the MHP solids.

Ni Scavenging

In order to maintain a quality MHP product (i.e. low Mg), a small fraction of the Ni (~ 1 g/l) and Co were deliberately left behind in the MHP product solution. The Ni Scavenging stage precipitated these metals with lime and recycled them back to 3° Neutralization. Overall recovery of Ni and Co within this circuit was greater than 97%, with a final Ni tenor of 20 mg/L.

Mg Removal

Mg is both leached from the concentrate in Pressure Oxidation and added via magnesia in the MHP circuit. The purpose of the Mg Removal circuit is to bleed Mg from the process yet leave enough Mg to support the chlorides to minimize the formation of calcium chloride. In the pilot plant, 65% of the Mg was precipitated per pass with the remainder recycled to PO.

METAL RECOVERY

Cu, Ni and Co leach extraction from the concentrate was 95%, 95%, and 97%, respectfully. Based on pilot plant metallurgical results, overall metal recoveries are presented in Table 15. Metal recovery from the leach liquor was excellent due to low Ni/Co losses in residue and byproducts, and complete recycle of all solutions. Also to be noted that metal losses were minimized by low sulphur oxidation leading to reduced gypsum production, hence decreased soluble metal losses to tailings.

| Tabl | le 1 | 5 – | Mesab | a C | Concentrate | Extraction | and | Recovery | Data |
|------|------|-----|-------|-----|-------------|------------|-----|----------|------|
| | | | | | | | | | |

| Lea | ch Extracti | on | Overall Recovery | | | |
|----------|-------------|------|------------------|------|------|--|
| % Cu | % Ni | % Co | % Cu | % Ni | % Co | |
| 95.0 | 95.2 | 97.4 | 94.6 | 93.8 | 95.5 | |

SUMMARY

The application of the CESL Process to the Mesaba property is being examined due to the difficulties involved in the production of saleable concentrate(s). Extensive pilot plant testing has shown the robustness of the CESL Process, processing a very low grade bulk Cu-Ni concentrate to attain extractions of 95% or higher for Cu, Ni and Co, followed by metal recoveries of >98 % from the leach liquor. Due to the closed circuit nature of the CESL Process (no effluents), essentially all process liquor streams were recycled, as well as any Ni bearing precipitates, leading to very high metal recoveries from solution.

The low sulphur oxidation in the CESL pressure oxidation process reduces operating and capital costs and thus allows for economic recovery of metals from such a low grade concentrate.

Cu was recovered to high grade cathode, whilst Ni and Co were recovered together (MgO pptn) as a mixed hydroxide product intended as an intermediate product for the project, prior to possible refining onsite to metals. The high-purity (46% Ni) intermediate will facilitate such refining in the future.

During the pilot campaign, a high-grade MHP was produced that exceeded the grades produced during bench testing. Levels of most impurities including Mg were satisfactorily low, but the MHP contained about 4% S, indicative of the presence of a mixed hydroxysulphate rather than a straight mixed hydroxide. In addition, the MHP contained 0.1% Cl, which could not easily be removed by washing.

Samples of the MHP intermediate were sent out for marketing purposes to various nickel refineries.

With the campaign complete, CESL continues to support the advancement of the Mesaba Project. A scoping study for the project is underway and is scheduled for completion later in 2009.

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