

Evaluation of a versatic 10 Acid/Nicksyn™ Synergistic system for the recovery of nickel and cobalt from a typical lateritic leach liquor

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Mintek has been involved in extensive test work since the early 1990s with respect to the recovery of nickel and cobalt from leach liquors, saturated in calcium, using synergistic solvent extraction systems. During this period the Nicksyn™ reagent was developed, optimized, commercially manufactured, and tested by Tati Nickel on a demonstration plant for more than 2800 operating hours. Efficient recovery of nickel without the co-extraction of calcium, thus avoiding gypsum formation in the extraction and stripping circuits, was illustrated. This synergistic system was recently evaluated on a laboratory scale for the recovery of nickel and cobalt from synthetic lateritic sulphate leach liquor containing about 3 g/L nickel, 0.5 g/L cobalt, 0.7 g/L manganese, 20 g/L magnesium, and with calcium at saturation. Extraction and stripping parameters were determined for this feed liquor and are discussed in this paper.

Introduction

The economic recovery of nickel from laterite ores has been pronounced for some years and will become more critical in the future, as lateritic ores constitute most of the world's known nickel and cobalt resources, with nickel production from sulphide deposits progressively decreasing. High-pressure acid leaching (HPAL) is being used for the recovery of nickel from nickel laterite ores, and increasingly atmospheric leaching is also being considered. There are almost twice as much laterite resources that are amenable to hydrometallurgical processing (limonite, nontronite/smectite) as that amenable to pyrometallurgical processing (saprolite, garnierite) (Bacon and Mihaylov, 2002).

Various hydrometallurgical flow sheets are being used for the recovery of nickel from laterite ores. Most plants use the Caron or HPAL processes, simple block flow diagrams for which are given in Figure 1. The Caron process is primarily considered for limonitic ores to avoid the acid consumption associated with the iron content in the ore. The ore is calcined reductively to reduce the ferric (associated with goethite) prior to ammoniacal leaching of the nickel and cobalt. Yabulu Nickel Refinery, Queensland, Australia implemented the Caron process.

The HPAL process can be used for limonitic as well as saprolitic ores (<4% Mg) and has been installed on numerous plants, including Moa Bay, Bulong, Murrin Murrin, and Goro (Bacon and Mihaylov, 2002). However, the actual recovery of nickel and cobalt and their separation primarily from calcium, magnesium, and manganese are done employing different flow sheets. In the Murrin Murrin flow sheet the pH of the HPAL pregnant solution is adjusted to pH 3.5-4 to neutralize excess acid and precipitate most of the ferric, aluminium, and chrome. This is followed by sulphide precipitation of nickel and cobalt, which is the primary technology employed to separate the nickel and cobalt from manganese, magnesium, and calcium.

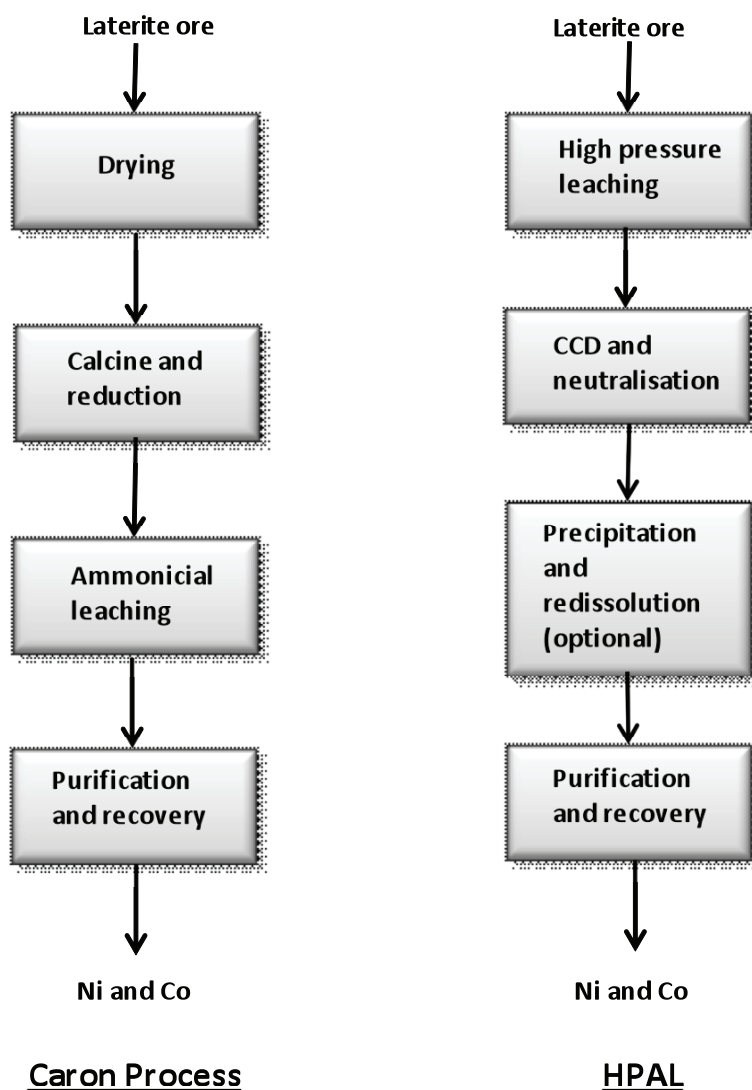


Figure 1. Primary hydrometallurgical processing options of lateritic nickel ores (after Dalvi, Bacon, and Osborne, 2004)

The Bulong flow sheet (Figure 2) also neutralized the free acid and precipitated ferric, aluminium, and chromium, but it employed direct solvent extraction (SX) for the recovery of nickel and cobalt (Flett, 2005). Cyanex 272 (2,4,4-trimethylpentyl phosphinic acid) was used to recover cobalt from the neutralized stream, followed by nickel SX using Versatic 10 acid (V10, a tertiary-branched carboxylic acid). One of the major drawbacks of the direct SX as operated at Bulong, was that the selectivity of the V10 extractant was inadequate to prevent calcium loading during extraction. This resulted in gypsum precipitation in the SX circuit, and hence major operational difficulties.

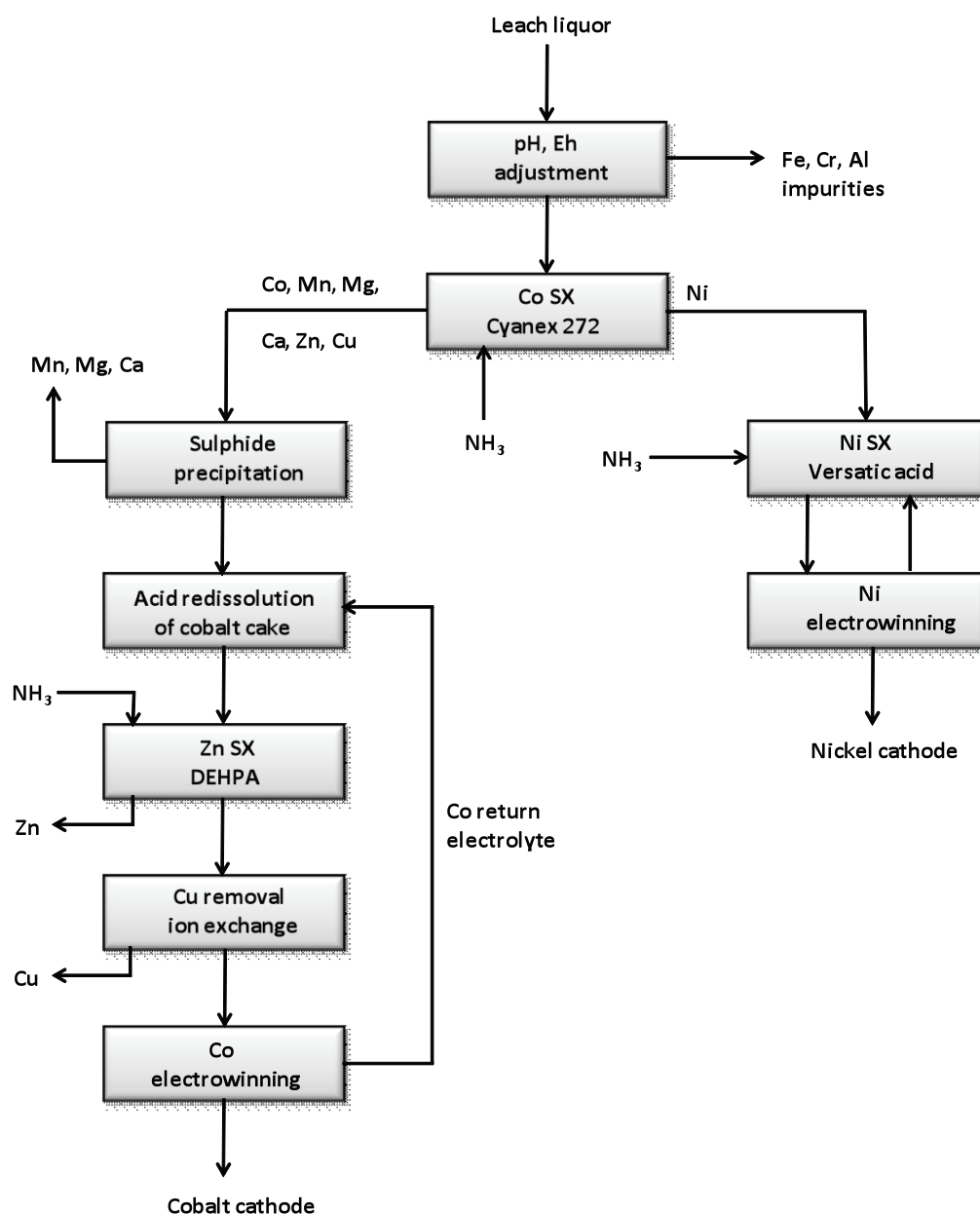


Figure 2. Simplified block flow diagram of the Bulong flow sheet

The most recent major nickel laterite project, namely Vale Inco's Goro nickel project, was commissioned during 2012. Nickel is also recovered via direct SX (no prior precipitation of nickel and cobalt) using Cyanex 301 [bis(2,4,4-trimethylpentyl) dithiophosphinic acid]. Metal distribution equilibria generated for pH vs. extraction (Figure 3) shows that nickel and cobalt could be extracted without neutralization during extraction (Mihaylov, Krause, Colton, and Okita, 2000). However, this reagent has a number of drawbacks:

- The very strong extraction of copper requires efficient removal of copper from the full flow of the pregnant leach solution via ion exchange (IX), which would be expensive. Furthermore, if any breakthrough from the IX circuit occurs, the copper would report to the Cyanex 301 circuit, which requires stripping with thiourea in sulphuric acid medium
- Strong nickel and cobalt extraction makes stripping difficult. The Goro process was designed to use hydrochloric acid stripping in four stripping stages, each with 5 minutes residence time in the mixer, at an operating temperature of 60°C and a residual hydrochloric acid concentration of 3M

- Due to the high residual acid concentration required for stripping, pyrohydrolysis is used for nickel recovery as NiO
- The introduction of chloride into the system requires more sophisticated materials of construction, and can have environmental concerns amongst other complications
- Cyanex 301 is chemically unstable, and in the presence of oxygen and metals such as ferric the reagent is oxidized to form a disulphide. Hence, air has to be excluded from the operating system, which Goro is achieving by employing the Bateman Pulsed Columns. Furthermore, the reagent can be regenerated using sulphuric acid and zinc powder.

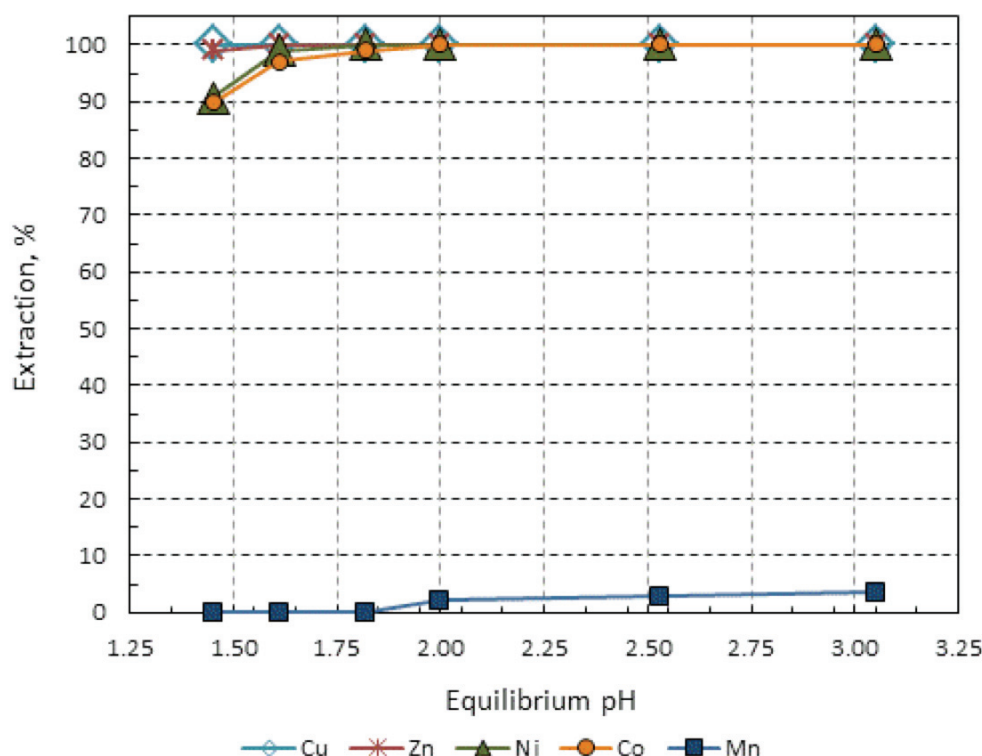


Figure 3. Metal extraction by 15 vol.% Cyanex 301 at an O:A phase ratio of 0.5 using NaOH for pH adjustment

Mintek developed the Nicksyn™ reagent during the 1990s, which, together with V10, provides an alternative and more cost-effective reagent for direct nickel and cobalt SX from laterite processing liquors. This synergistic system has been demonstrated over 2800 hours on the Tati Nickel Activox® demonstration plant in Botswana, and has since been commercialized (Du Preez *et al.*, 2007; Masiwa *et al.*, 2008). This paper describes the technical performance of the V10 and Nicksyn™ synergistic system for the recovery of nickel and cobalt from neutralized HPAL lateritic leach liquor.

Experimental procedures

Laboratory tests

Analytical methods

All metal analyses were done by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) with a detection limit of 2 mg/L for all metals. Organic samples were stripped with sulphuric acid (approx. 1M) at an organic-to-aqueous (O:A) phase ratio of 0.5, after which the strip liquors were submitted for analysis.

Reagents and solutions

Versatic 10 acid (V10, a tertiary-branched carboxylic acid) was obtained from Chemquest (produced by Resolution Performance Products Ltd.), while Nicksyn™ was prepared for Mintek by an international, reputable manufacturer. The chemical composition and technical information of Nicksyn™ remains the proprietary information of Mintek and can therefore not be disclosed. Appropriate dilutions of V10 alone and V10 mixtures with Nicksyn™ were done using an aliphatic hydrocarbon diluent, Shellsol D70, which was obtained from Shell Chemicals.

Metal distribution studies

Metal-distribution equilibria (pH vs. extraction profiles, extraction and stripping isotherms) were generated by contacting the required organic phase with the appropriate feed solution at various O:A phase ratios, using rapid magnetic stirring and controlling the temperature in a water-jacketed glass vessel at 25°C. An equilibrium time of 10 to 15 minutes was allowed to ensure steady state was reached.

The pH value of the aqueous phase (in the case of extraction isotherms) was adjusted or controlled by the addition of sodium hydroxide solution (approx. 1M to 10M), using a calibrated combined glass reference electrode. Samples of the organic phase were taken immediately after the aqueous samples to prevent possible re-equilibration after each pH adjustment. Aqueous samples were submitted for analyses. Organic samples were stripped with 1M H₂SO₄ (O:A phase ratio of 0.5), after which the strip liquors were analysed for the relevant elements by ICP-OES.

Organic phase (0.5M V10 plus 0.25M Nicksyn™) was batch-loaded for stripping purposes by contacting portions of fresh organic phase with synthetic laterite solution (at an O:A phase ratio of 0.45) at pH 6.0 and at 25°C. Samples of aqueous and organic phases were analysed by ICP-OES. This procedure was repeated two more times to simulate the three stages required according to the McCabe-Thiele construction (Figure 7). The batch-loaded organic phase obtained was then contacted with a synthetic nickel spent electrolyte (approx. 71 g/L nickel in 40 g/L H₂SO₄) at different O:A phase ratios at 25°C, measuring the final pH values of the loaded strip liquors. Samples of the loaded strip liquors and organic phases were analysed as previously described.

For the batch countercurrent extraction experiment, organic and aqueous phases were contacted (at 25°C), using magnetic stirring at an O:A phase ratio of 0.45. A sequence of batch contacts that simulates the conditions of a four-stage continuous-flow process was used as illustrated in Figure 4. Six full cycles (D to I, see Figure 4) were completed in this way to ensure steady-state conditions. Samples of the raffinates of the fourth stages (4D to 4I), and the aqueous phases of the first (1I), second (2I) and third stage (3I) of the last cycle (I) were submitted for analyses. Portions of the loaded organic phases of the first stages (1D to 1I) as well as the loaded organic phases of the second (2I), third (3I) and fourth (4I) stages of the last cycle were taken and stripped as described above, after which the strip liquors were analysed by ICP-OES.

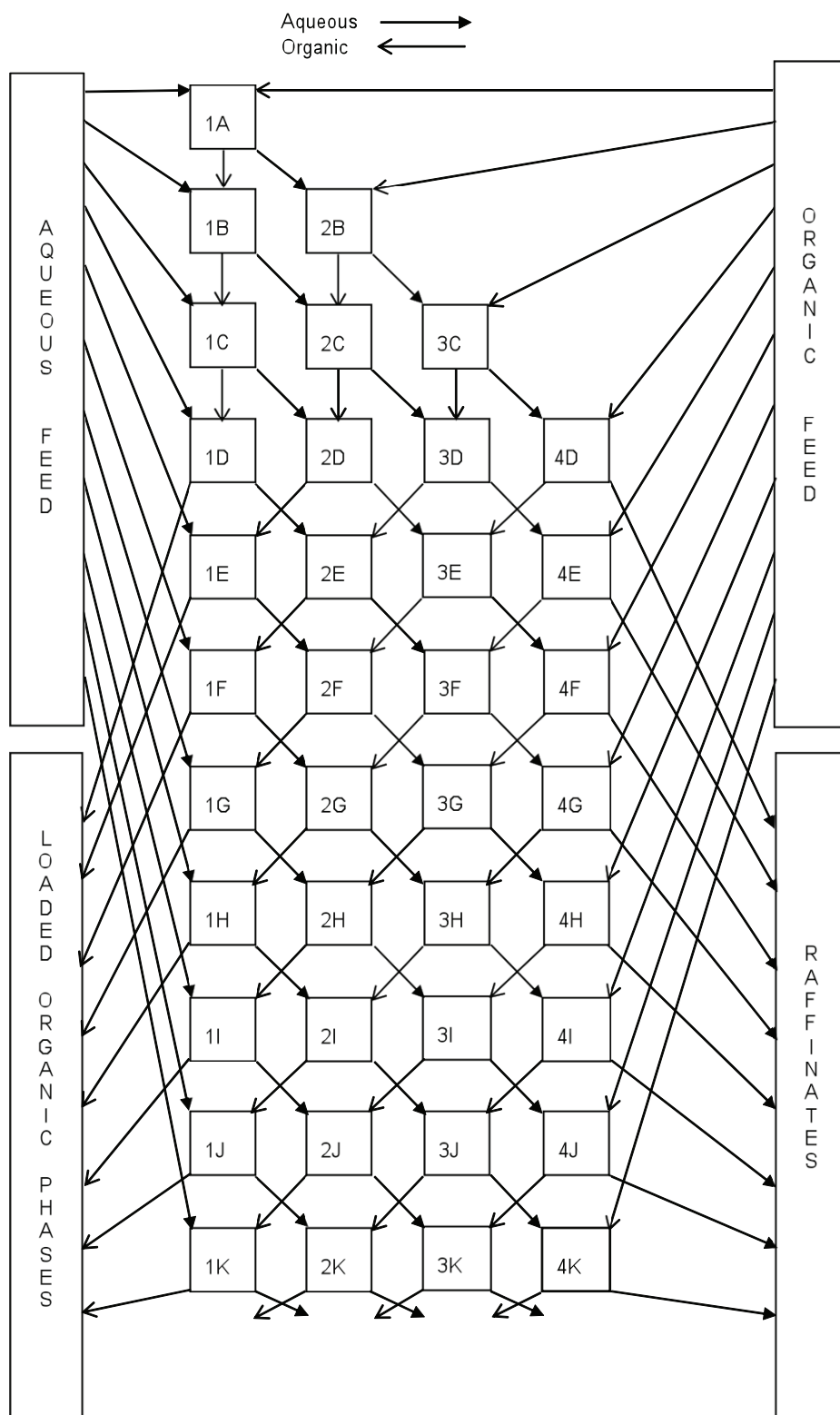


Figure 4. Scheme of contacts for batch counter current extraction experiment

Results and discussion

Direct recovery of nickel and cobalt from a synthetic solution representing a nickel HPAL laterite leach liquor after iron, aluminium, and chromium removal was tested using the V10/Nicksyn™ system. Nicksyn™ is now commercially available, and hence a very attractive option to be considered for HPAL leach liquor.

Feed solution

A synthetic feed solution was made up from metal sulphate salts to contain nickel, cobalt, manganese, calcium (at saturation) and magnesium. The average of various analyses of the synthetic laterite feed solution is given in Table I.

Table I. Average composition of synthetic laterite feed solution

Feed	Concentration, g/L				
	Ni	Co	Mn	Mg	Ca
Laterite leach liquor (synthetic)	3	0.5	0.66	20.2	0.46

Organic phase compositions

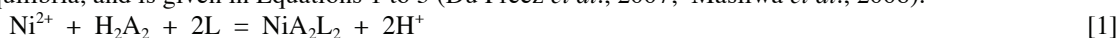
The different concentrations of V10 and the molar ratios of V10:Nicksyn™ diluted in Shellsol D70 are given in Table II.

Table II. V10 concentrations and molar ratios of V10:Nicksyn™

V10		Nicksyn™	V10:Nicksyn™
Vol. %	M	M	Molar ratio
9.6	0.50	-	-
9.6	0.50	0.125	4:1
9.6	0.50	0.25	2:1
9.6	0.50	0.50	1:1

Extraction metal-distribution equilibria (pH vs. extraction)

The origin of the synergistic effect for nickel by a carboxylic acid (such as V10, which exists in the form of dimers H_2A_2), with the addition of a synergistic compound (L) such as Nicksyn™, was discussed previously in terms of competing equilibria, and is given in Equations 1 to 3 (Du Preez *et al.*, 2007; Masiiwa *et al.*, 2008):



where H_2A_2 denotes the carboxylic acid dimer and L denotes the synergist.

Results for selected pH vs. extraction isotherms are shown in Figure 5 and Figure 6. The pH_{50} values (the pH at which 50% of the metal originally present in the aqueous phase is extracted under a given set of conditions) are summarized in Table III.

Table III. pH_{50} values for the extraction of metals from synthetic laterite leach solution using V10 alone and with Nicksyn™ in Shellsol D70 at 25°C

V10, M	Nicksyn™, M	pH_{50}						
		Mg	Ca	Mn	Co	Ni	Mn-Co	Ca-Co
0.5	-	>7.17	>7.17	>7.17	6.52	6.37	>0.65	>0.65
0.5	0.125	>7.19	>7.19	6.62	5.87	5.14	0.75	>1.32
0.5	0.25	>7.43	>7.43	6.47	5.53	4.97	0.94	>1.90
0.5	0.5	>7.30	>7.30	6.50	5.37	4.69	1.13	>1.93

* A > sign preceding a pH value indicates that 50% metal extraction was not reached yet at this pH value

Synergistic shifts in the pH_{50} values for the extraction of nickel increased from 1.23 to 1.68 units when Nicksyn™ addition was increased from 0.125M to 0.5M, respectively, while the shifts for cobalt increased from 0.65 to 1.15 units with the same Nicksyn™ additions. The extraction of manganese was largely unaffected, hence the separation ($\Delta\text{pH}_{50}^{\text{Mn-Co}}$) between cobalt and manganese increased from 0.75 to 1.13. Extraction of calcium and magnesium were negligible (<1%) under these conditions tested. This synergistic system therefore not only provides an option for the recovery and separation of nickel and cobalt efficiently from calcium and magnesium, but also gives the option of selection of degree of manganese removal.

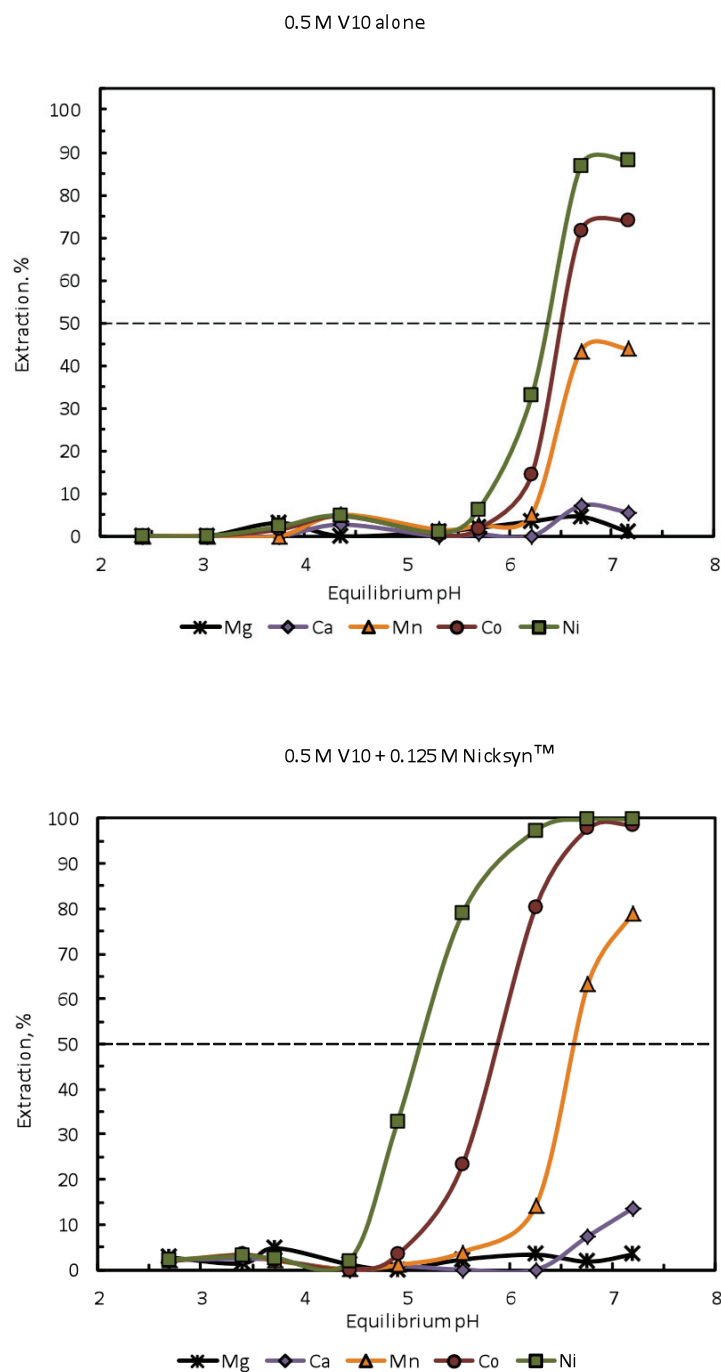


Figure 5. Extraction of metals from synthetic laterite leach solution by 0.5M V10 alone and 0.5M V10 plus 0.125M Nicksyn™ in Shellsol D70 at 25°C

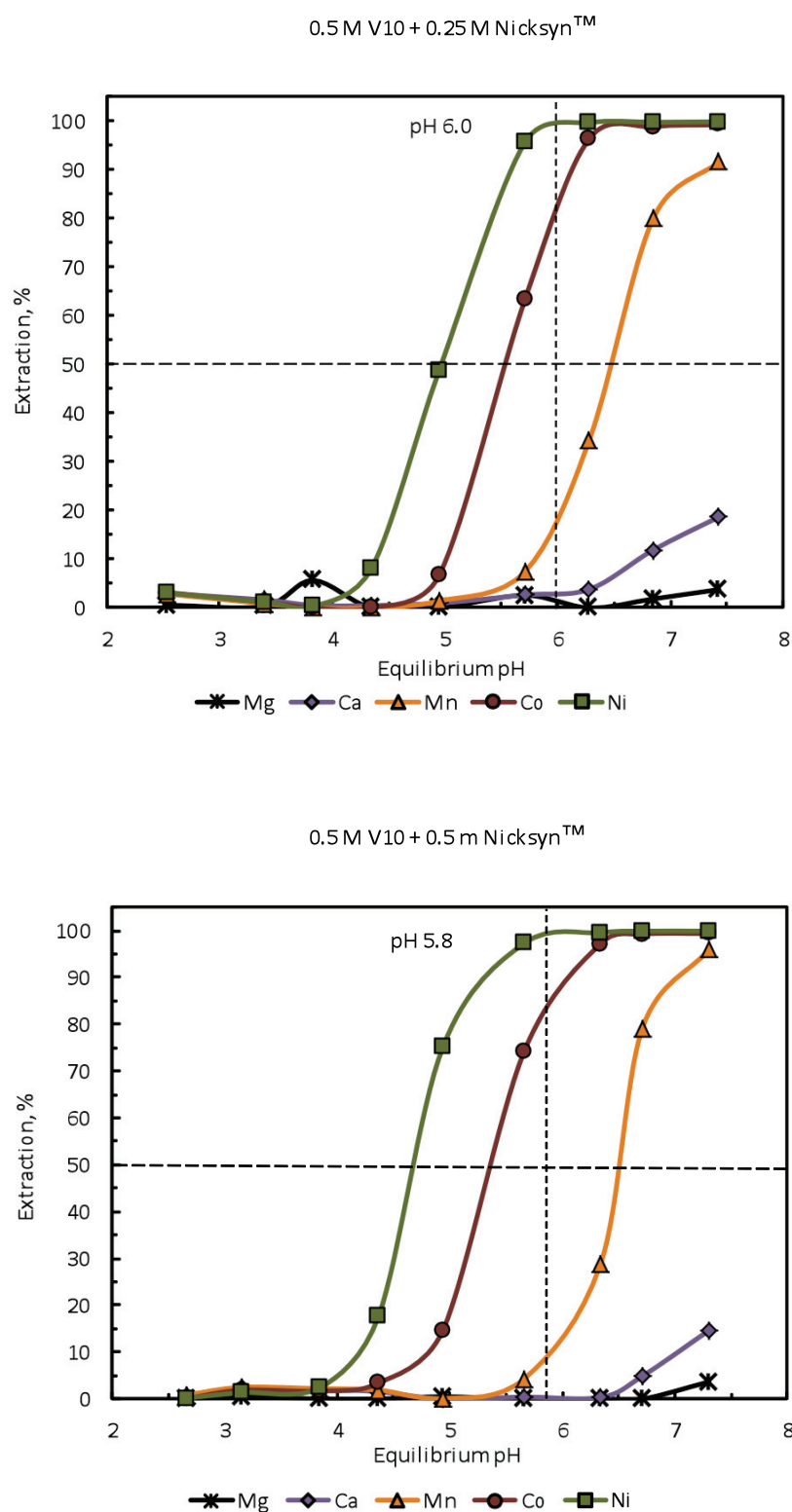


Figure 6. Extraction of metals from synthetic laterite leach solution by 0.5M V10 plus 0.25M and 0.5M Nicksyn™ in Shellsol D70 at 25°C

Extraction isotherms

The distribution isotherms and McCabe-Thiele constructions for the extraction of nickel and cobalt from synthetic laterite leach solution generated using 0.5M V10 plus 0.25M Nicksyn™ (at pH 6.0) and 0.5M V10 plus 0.5M Nicksyn™ (at pH 5.8) in Shellsol D70 at 25°C are shown in Figure 7 and Figure 8 respectively. The McCabe-Thiele construction was drawn for optimum recovery of nickel with the idea to gauge what the possible recovery for cobalt could be under the chosen conditions. In order to determine the effect of insufficient aluminium removal prior to the SX circuit, about 200 mg/L aluminium (as sulphate) was added to the leach solution used for the generation of the extraction isotherms.

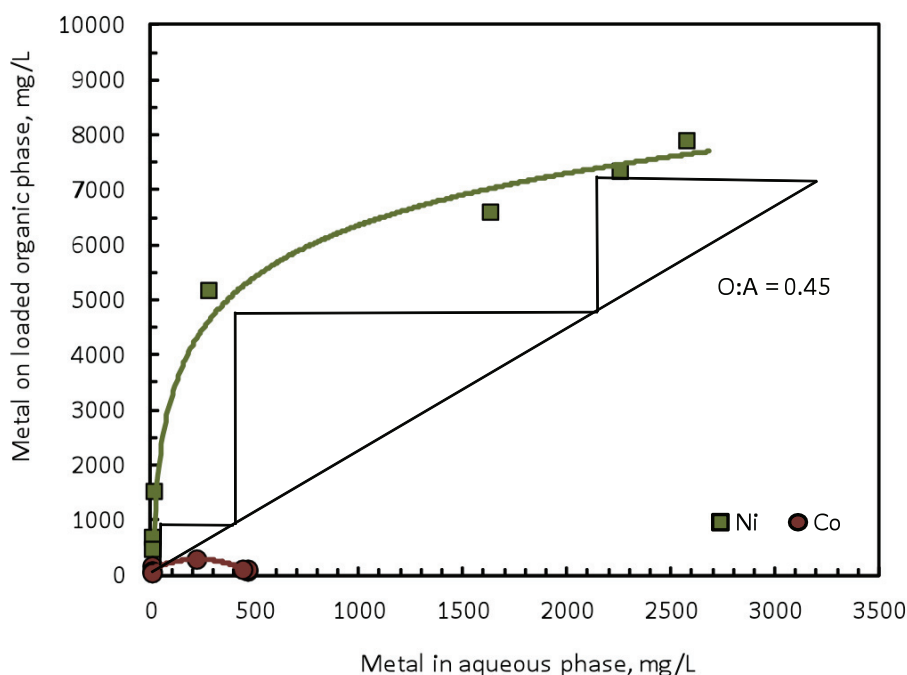


Figure 7. Distribution isotherm for the extraction of nickel and cobalt from synthetic laterite leach solution by 0.5M V10 plus 0.25M Nicksyn™ in Shellsol D70 at 25°C and pH 6.0

The McCabe-Thiele construction on the isotherm indicated that a loading of about 7.1 g/L nickel could be achieved in three countercurrent extraction stages at an O:A phase ratio of 0.45. The maximum loading of cobalt under these conditions was about 300 mg/L. Calcium and manganese co-extraction were about 6 and 34 mg/L, respectively.

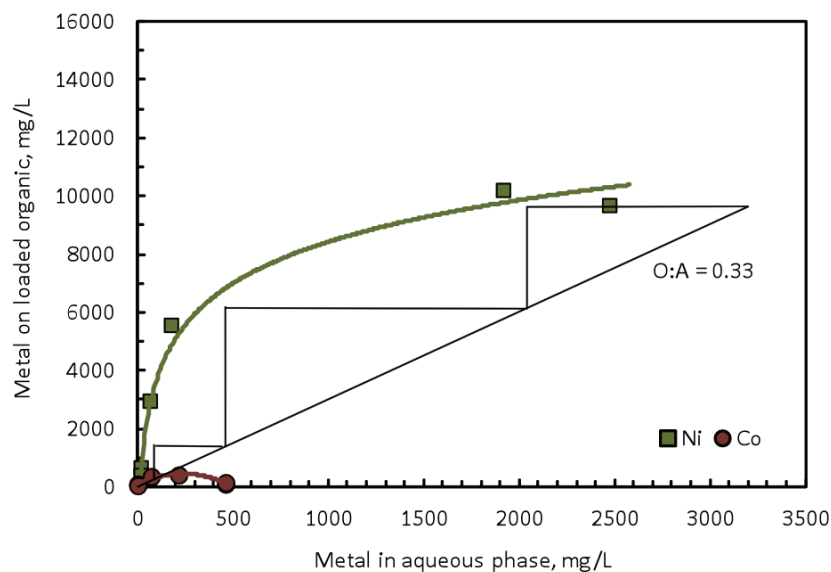


Figure 8. Distribution isotherm for the extraction of nickel and cobalt from synthetic laterite leach solution by 0.5M V10 plus 0.5M Nicksyn™ in Shellsol D70 at 25°C and pH 5.8

The results for the McCabe-Thiele construction shown in Figure 8 indicated that a slightly higher loading of about 9.6 g/L nickel could be achieved with the increased ratio of V10:Nicksyn™ of 1:1 in three countercurrent extraction stages at an O:A phase ratio of 0.33. Cobalt loading under these conditions was just slightly lower than 400 mg/L. The co-extraction of calcium and manganese under these conditions was about 4 mg/L each.

Although a slightly better separation (1.13 vs. 0.94 pH units) between cobalt and manganese, and higher loading of cobalt together with nickel (7.2 vs. 9.7 g/L) were achieved using a V10:Nicksyn™ ratio of 1:1 compared with the V10:Nicksyn™ ratio of 2:1, respectively, the additional costs of the increased Nicksyn™ concentration should be considered on an economic basis for each application.

In order to recover all cobalt together with nickel, the O:A phase ratio (and possibly the number of stages) would have to be adjusted to compensate for cobalt being ‘crowded off’ as illustrated by the unfavourable isotherms obtained for cobalt (Figure 9 and Figure 10).

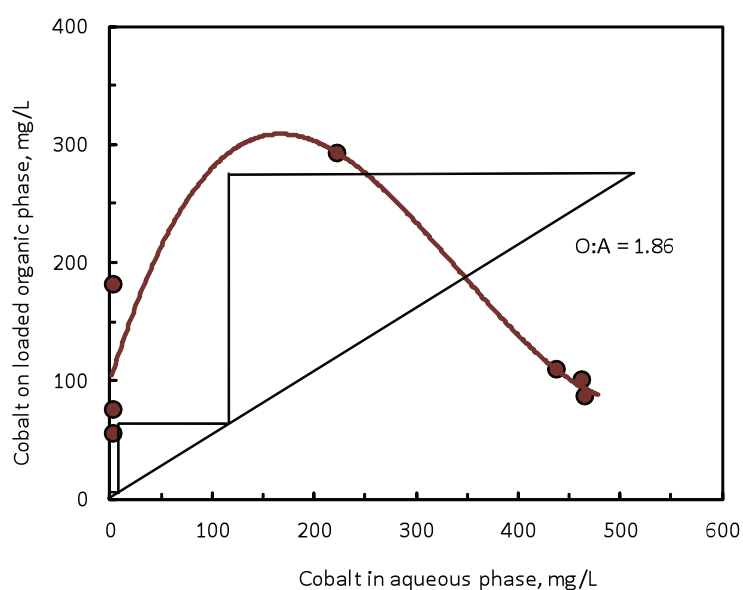


Figure 9. Distribution isotherm for the extraction of cobalt from synthetic laterite leach solution by 0.5M V10 plus 0.25M Nicksyn™ in Shellsol D70 at 25°C and pH 6.0

The McCabe-Thiele construction redrawn for optimum cobalt recovery under the conditions tested indicated that a higher O:A phase ratio (1.86 vs. 0.45 as previously drawn for nickel recovery) should be employed to ensure a loading of approximately 257 mg/L cobalt without being ‘crowded off’ by nickel, using two countercurrent extraction stages. Under these conditions, nickel would still be recovered (leaving <2 mg/L in the raffinate) with minimum co-loading of calcium (5 mg/L), magnesium (4 mg/L), and manganese (7 mg/L).

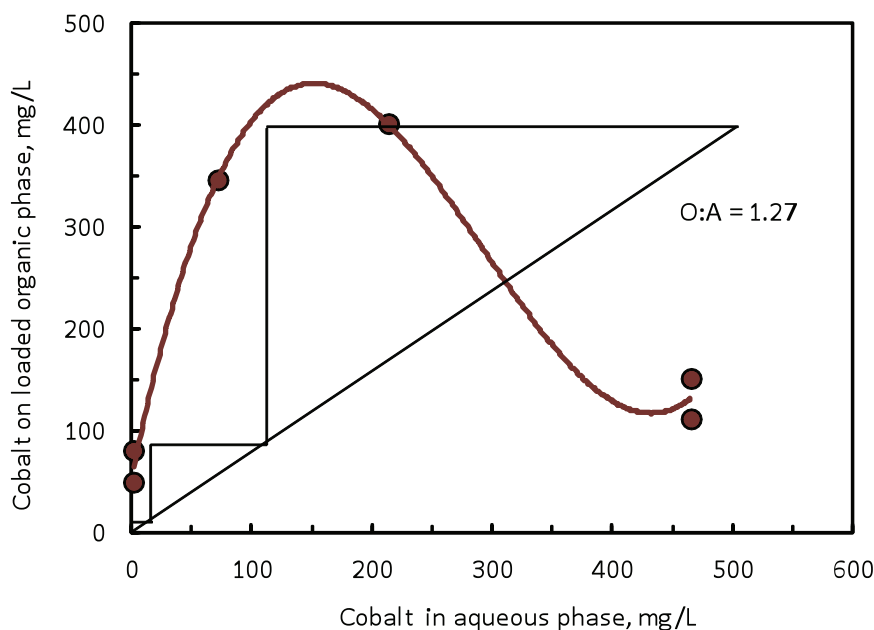


Figure 10. Distribution isotherm for the extraction of cobalt from synthetic laterite leach solution by 0.5M V10 plus 0.5M Nicksyn™ in Shellsol D70 at 25°C and pH 5.8

For optimum cobalt recovery under these conditions, the McCabe-Thiele construction indicated that an O:A phase ratio of 1.27 (instead of 0.33 previously drawn for nickel recovery) would be required to achieve a loading of about 400 mg/L cobalt without ‘crowding off’ by nickel in two to three countercurrent extraction stages. Under these conditions, similar nickel recovery would still be expected (<2 mg/L in the raffinate) with minimum co-loading of calcium (4 mg/L), magnesium (4 mg/L), and manganese (14 mg/L). In both these cases optimum design should include economic cobalt recovery without calcium co-extraction.

Batch countercurrent test work

A batch countercurrent test was performed for the extraction of cobalt and nickel with 0.5M V10 plus 0.25M Nicksyn™ in Shellsol D70 at 25°C. Four extraction stages were used with an O:A phase ratio of 0.45 and an equilibrium pH value of 6.0 in each stage (i.e. flat pH profile) over a total of six full cycles (see Figure 4). The results are illustrated in Figure 11.

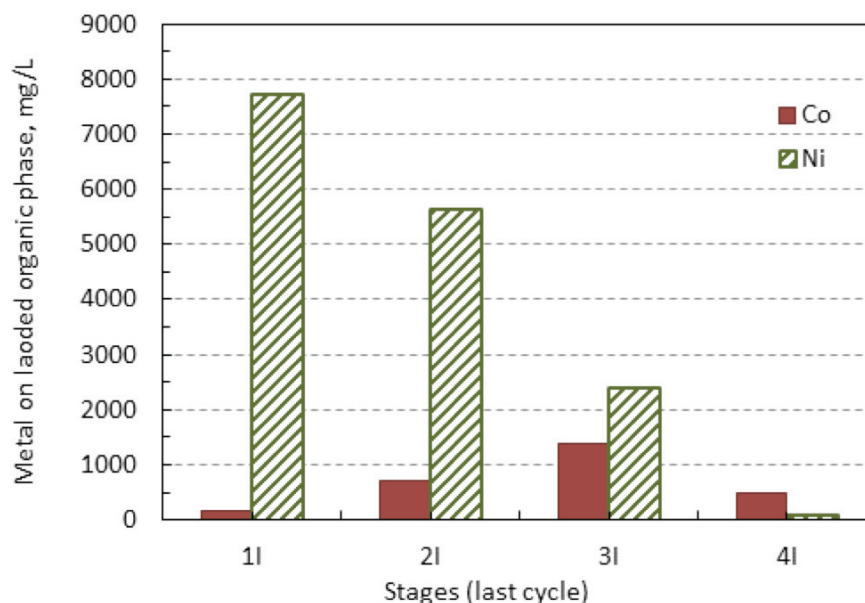


Figure 11. Batch counter current extraction of nickel and cobalt from simulated laterite solution by 0.5M V10 plus 0.25M Nicksyn™ in Shellsol D70 in 4 stages and an O:A phase ratio of 0.45 and at 25°C

A loaded organic phase (Stage 1I) containing about 7.7 g/L nickel was obtained at steady state (Cycle I, Figure 4), leaving about 5 mg/L nickel in the raffinate, which related to >99% extraction. Cobalt was loaded up to approximately 1.4 g/L in stage 3I, after which it was ‘crowded off’ by nickel to only approximately 176 mg/L on the loaded organic phase (Stage 1I) under these conditions tested. In order to recover both cobalt and nickel, a higher O:A phase ratio and probably more stages would be required, as shown in Figure 9 and Figure 10. A pH profile (and not a flat profile as was employed here) over all the stages could also assist to enhance cobalt recovery, providing no calcium is co-extracted.

The co-loading of some impurities at steady state of the batch countercurrent experiment is shown in Figure 12.

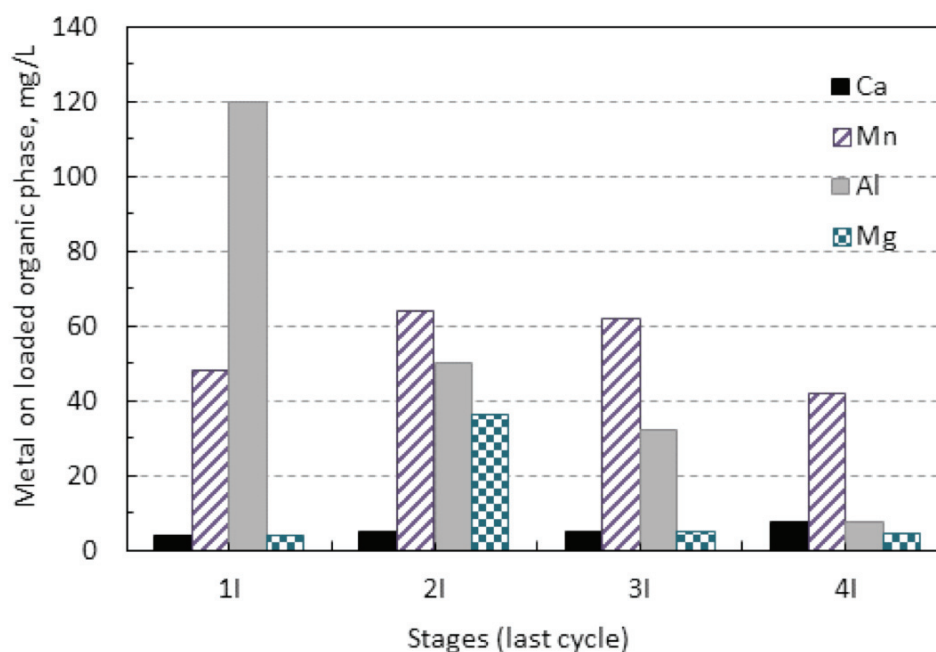


Figure 12. Batch countercurrent extraction of impurities from simulated laterite solution by 0.5M V10 plus 0.25M Nicksyn™ in Shellsol D70 in 4 stages and an O:A phase ratio of 0.45 and at 25°C

The efficient removal of aluminium prior to nickel and cobalt recovery was strongly indicated as aluminium was strongly extracted by the synergistic mixture (from the feed solution containing approximately 213 mg/L only <2 mg/L was left in the raffinate). Magnesium extraction was low (<4 mg/L on the loaded organic for stages 1I, 2I and 4I) and the anomaly observed in the higher loading in stage 2I (36 mg/L on the loaded organic phase) most probably was due to analytical error. Manganese was co-loaded (between 40 and 62 mg/L), with calcium co-loading minimal (approx. 7 mg/L) based on loaded organic phase analyses. Phase separations were clear in all stages and no crud formation was observed.

Further optimization of the O:A phase ratio, number of stages, and pH profile across the extraction circuit would have to be done to optimize cobalt recovery (including nickel) as well as limiting the co-loading of unwanted impurities such as manganese. This test work has to be performed for each individual lateritic feed solution.

Stripping test work

In order to conduct stripping test work, a batch of fresh organic phase (0.5M V10 plus 0.25M Nicksyn™ in Shellsol D70) was preloaded to reasonably represent a loaded organic phase that would be expected from an extraction circuit. Synthetic spent nickel electrolyte was prepared to contain approximately 71 g/L nickel and 40 g/L H₂SO₄ (which should relate to a delta of approx. 24 g/L nickel). A stripping isotherm was generated, and the results are shown in Figure 13.

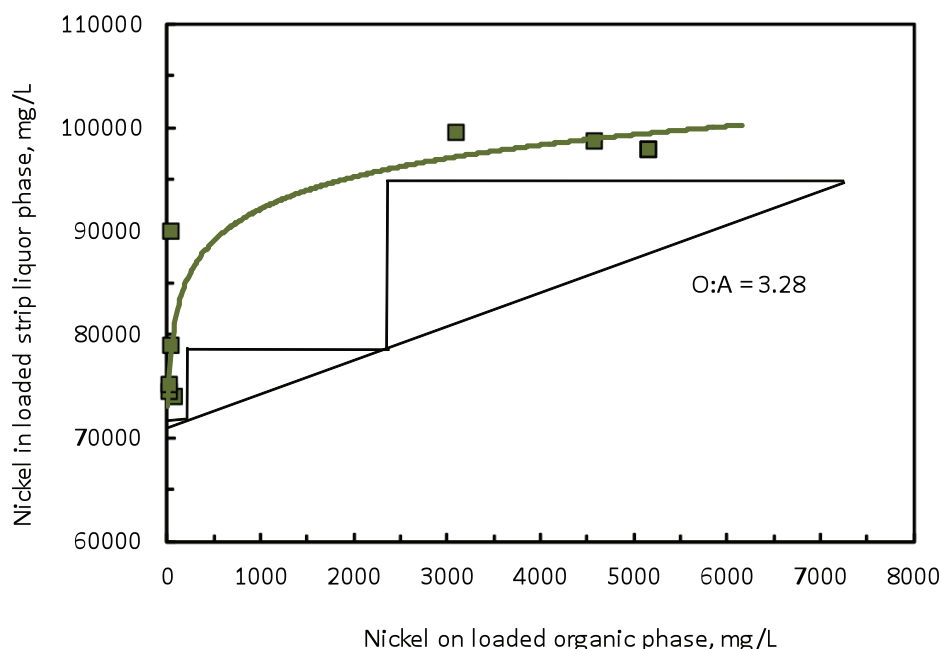


Figure 13. Distribution isotherm for the stripping of nickel from batch-loaded 0.5M V10 plus 0.25M Nicksyn™ in Shellsol D70 with synthetic spent electrolyte at 25°C

Loaded strip liquor containing about 95 g/L nickel (at a pH value between 1.1 and 4.2) could be generated. The McCabe-Thiele construction indicated that two to three stages and an O:A phase ratio of 3.3 would be required to achieve this. Strip liquor pH values measured for stripping done at O:A phase ratios of 0.1 to 2.0 varied between 0.5 and 1.1, which indicates that adequate sulphuric acid was available for complete stripping of nickel. Stripping done at O:A phase ratios of 5.0, 8.0, and 10.0 resulted in strip liquors exhibiting pH values of 4.2, 4.6, and 4.8 respectively, which indicated unfavourable stripping conditions for nickel. If plant operation required these operating conditions, it would be advisable to employ pH control (at about 3) in order to facilitate efficient stripping of nickel in a minimum number of stages and to provide an advanced electrolyte suitable for electrowinning.

Any co-loaded manganese could be removed by scrubbing with pH-adjusted water or a portion of the loaded strip liquor, depending on downstream requirements. The addition of a washing stage is recommended for removal of entrained aqueous phase from the loaded organic phase.

Conclusions

- The V10 plus Nicksyn™ synergistic mixture was evaluated for the use of direct solvent extraction of nickel and cobalt from lateritic leach liquor
- Nicksyn™ is now commercially available via a secured, reputable international supplier. It is a very attractive option and should be included in the evaluation for all HPAL hydrometallurgical projects
- For a solution containing about 3 g/L nickel, 0.5 g/L cobalt, 0.7 g/L manganese, 20 g/L magnesium, and with calcium at saturation, the following parameters were found:
 - ◆ Three to four extraction stages would be required for optimum nickel and cobalt recovery. A pH profile (instead of the same pH value in every stage) could be employed to assist in the recovery of cobalt. The O:A phase ratio could vary and would be determined by optimum cobalt recovery excluding co-extraction of calcium
 - ◆ If required, a scrub stage could be included for removal of co-extracted impurities such as manganese and calcium. Typical scrub solutions would be pH-adjusted water, or a portion of the loaded strip liquor
 - ◆ One wash stage is recommended for removal of any entrained aqueous phase from the loaded organic phase
 - ◆ Two to three stripping stages would be required using spent electrolyte at an O:A phase ratio of about 3. The pH of stripping should be maintained at about 3 to facilitate a suitable advanced electrolyte for subsequent electrowinning
- Nicksyn™ has been found to be stable over a testing period of 100 days (representing stripping conditions) in the laboratory. The solubility was found to be between 1 and 3 mg/L under specific conditions tested (Du Preez and Preston, 2004)
- Mintek is currently involved in an international project with a commercial client where Nicksyn™ will be included in the definitive feasibility study for nickel and cobalt recovery from laterite solutions.

Acknowledgements

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I have been involved in research and development, as well as commercial projects, at Mintek since 1984, with focus on the recovery, upgrading, and purification of uranium, rare earths, and base and platinum group metals using existing and/or new solvent extractants.