

LABORATORY AND PILOT-PLANT INVESTIGATIONS ON THE PERFORMANCE OF A SYNERGISTIC VERSATIC 10 ACID SOLVENT EXTRACTION SYSTEM FOR THE RECOVERY OF NICKEL FROM CALCIUM-SATURATED STREAMS

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Abstract

Laboratory and continuous countercurrent evaluations were conducted to investigate the effect of the use of the Mintek synergist (Nicksyn™) in combination with Versatic 10 acid (V10) on the selective solvent extraction (SX) of nickel from different nickel sulphate solutions.

V10 can be used for the SX of nickel from a sulphate solution close to saturation with calcium. Any upgrading of the calcium concentration in the aqueous media during the SX operation can potentially lead to gypsum precipitation. Careful control of the pH at which nickel extraction is performed must be exercised to prevent calcium co-extraction and subsequent precipitation when calcium is displaced off the organic phase. With the addition of certain synergists the differences in pH_{50} values between nickel and calcium can be increased from about 0.5 - 1.5 pH units normally achieved with V10 alone, to 3 - 4 pH units.

Mintek has been involved in different projects to separate nickel (and in some cases cobalt) from calcium, manganese and magnesium to produce pure nickel salts or cathodes, such as the Tati Activox® Project (TAP) in Botswana and the BioNIC™ project at the Queensland Nickel's Yabulu Refinery in Australia. Test work has also been conducted on synthetic solutions simulating typical laterite leach liquors containing magnesium concentrations of about 20 g/L.

During laboratory tests the effects of Nicksyn™ concentration in the organic phase, extraction temperature and pH on the separation achievable between both nickel and calcium, and cobalt and manganese were investigated. Results indicated that the ΔpH_{50} values between nickel and calcium varied between 2.0 - 3.2 pH units, compared to V10 alone, which was previously found to be about 1.1 pH units¹. Cobalt/manganese separations ranged between about 0.5 - 1.1 pH units, while it was about 0.2 pH units or less for V10 alone.

Operating conditions for optimum nickel extraction with minimum calcium co-extraction was determined during continuous countercurrent campaigns conducted at ambient temperature. Under optimised conditions nickel extraction greater than 99% was achieved with limited or no calcium extraction, which reduces the risk around gypsum precipitation in the extraction or stripping circuits significantly.

1. INTRODUCTION

Mintek recently optimised on laboratory scale the use of V10 and Nicksyn™ mixtures for nickel extraction from leach liquors obtained in the refining of sulphide concentrates, which was then demonstrated at the TAP in Botswana over a period of more than 2500 operating hours³. The system proved to be robust and nickel recoveries of >99.8% was achieved without co-extracting calcium. Although this V10/Nicksyn™ circuit employed four extraction stages yielding a 0.5% increase in nickel recovery, results showed that it was possible to drop one extraction stage and still maintain target recovery. Replacing V10 with V10/Nicksyn™ system increased the nickel SX capital and reagent costs by 35% and 4%, respectively, but improved nickel recoveries by 0.5%. The payback on the additional capital for the commercial plant would be less than 18 months at that time, but the major advantage is the reduction in gypsum precipitation risk.

The advantages afforded by the addition of the Mintek-developed synergist (Nicksyn™) to V10 on the separation achievable between nickel and calcium during SX from various simulated leach liquors were investigated.

Laboratory experiments at both ambient temperature and 45°C were performed to determine extraction pH versus the percent of extraction curves for various metals and distribution isotherms for the extraction of nickel. Continuous countercurrent campaigns investigated the operating conditions required for optimum nickel extraction from simulated bioleach liquor. The campaigns were aimed at maximum nickel extraction with minimum calcium and other impurity co-extraction. Nickel extraction was accomplished in a 4-stage extraction, 3-stage stripping SX circuit. The circuits employed a 5 or 10 vol.% V10 extractant in a commercial aliphatic C₁₂-C₁₃ hydrocarbon diluent as the organic phase, with the addition of Nicksyn™ in a 2:1 V10-to-Nicksyn™ molar ratio, to affect efficient nickel extraction at ambient temperature.

2. EXPERIMENTAL

2.1. Laboratory tests

2.1.1. Analytical methods

All metal analyses were done by Mintek's Analytical Services Division. Aqueous samples were analysed for Ni, Co, Ca, Cu, Zn, Mn, Fe and Mg (detection limit 2 mg/L for all metals) using Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES). Organic samples were stripped with sulphuric acid (1 M) at an organic-to-aqueous (O:A) phase ratio of 0.5, after which the strip liquors were submitted for analyses.

The concentration of V10 in the organic phase was determined by potentiometric titration of a sample (4 mL) dissolved in ethanol (25 mL) and water (10 mL) against a standard sodium hydroxide solution (0.10 M, made up from Merck ampoule), using a calibrated combined glass-reference electrode.

The Nicksyn™ content of the organic phase was determined by a method developed by Mintek to ensure accurate make-up of the required organic phases.

2.1.2. Organic phases

V10 (a tertiary-branched carboxylic acid) was obtained from Chemquest (produced by Resolution Performance Products Ltd.), whilst Nicksyn™ was prepared for Mintek by an independent 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, SSX 210, which was obtained from Sasol Wax. Organic phases were used as supplied without any further purification. Extensive tests have been done previously to prove the stability of Nicksyn™^{1,2}. It was found to be stable over testing periods of up to 100 days, representing extraction and stripping conditions. Lower reagent losses were experienced with the V10/Nicksyn™ trial on the TAP plant and the reagent make-up ratio closely matched the ratio maintained in the organic phase which indicated that no preferential loss of either component occurred³.

2.1.3. Metal-distribution studies

Metal-distribution equilibria (pH vs. extraction profiles and extraction isotherms) were determined by contacting the required organic phase with the appropriate feed solution at required O:A phase ratios, using rapid magnetic stirring and controlling the temperature in a water-jacketed glass vessel. The pH value of the aqueous phase was adjusted or controlled by the addition of sodium hydroxide solution (~10 M), using a calibrated combined glass-reference electrode. Equilibrium was established between 10 to 15 minutes after alkali additions. 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 sulphuric acid (~1 M) after which strip liquors were submitted for analyses.

2.2. Continuous countercurrent evaluation campaigns

2.2.1. Analytical methods

Samples were analysed as described in Section 2.1.1.

2.2.2. Plant equipment and operating conditions

The separation factor achievable between nickel and calcium is dependent on the Nicksyn™ concentration in the organic phase, the extraction pH and the temperature at which extraction is performed. Organic phases containing V10 and Nicksyn™ in a 2:1 molar ratio were used. An aliphatic hydrocarbon, C₁₂-C₁₃, (SSX 210) was used as diluent. The plant comprised of a 4-stage extraction, 3-stage stripping circuit for the exclusive extraction of nickel. No scrubbing to remove calcium from the loaded organic phase was

required. The operation was conducted in conventional box-type mixer-settler units with 500 mL mixer and 2000 mL settler capacities. Aqueous and organic phase feed solutions were pumped to the relevant stages by variable speed peristaltic pumps and interstage solution transfer was accomplished by pumping impellers. The pH in each of the extraction stages was automatically controlled using a PID controller linked to a peristaltic pump for the addition of sodium hydroxide.

3. RESULTS AND DISCUSSION

3.1. Laboratory tests

3.1.1. Feed solutions

The average compositions of the different simulated feed solutions used are shown in Table 1, which were prepared from the metal sulphate salts.

Table 1: Composition of different synthetic feed solutions used during laboratory tests

Feed	Concentration, g/L						
	Ni	Co	Mn	Mg	Zn	Cu	Ca
1 (Bioleach liquor)	2.5	0.25	1	1	0.1	0.1	0.5-0.6
2 (Bioleach liquor)	5	0.5	1	1	0.1	0.1	0.5-0.6
3 (TAP leach liquor)	30	3	6	1	0.1	0.1	0.5-0.6
4 (Laterite leach liquor)	3	0.5	0.7	20	-	-	0.5-0.6

Feed 1 and 2 represent some bioleach solutions, while Feed 3 was made up to simulate a typical feed solution to the nickel solvent extraction circuit produced by the TAP. Feed 4 represented a feed solution obtained from laterite leaching, containing about 20 g/L magnesium.

3.1.2. Organic phase compositions

The different concentrations of V10 and the molar ratios of V10:Nicksyn™ diluted in SSX 210 are given in Table 2.

Table 2: V10 concentrations and molar ratios of V10:Nicksyn™ used during test work

V10		Nicksyn™	V10:Nicksyn™
Vol.%	M	M	Molar ratio
3	0.157	0	-
3	0.157	0.078	2:1
4.8	0.25	0.25	1:1
5	0.26	0.13	2:1
5	0.26	0.26	1:1
9.6	0.50	0	-
9.6	0.50	0.50	1:1
10	0.52	0.26	2:1
30	1.56	0.78	2:1
30	1.56	1.56	1:1

3.1.3. Start-up conditions for continuous countercurrent campaigns

Start-up operating conditions for Campaign 1 and 2 are shown in Table 3.

Table 3: Continuous countercurrent operating conditions

Parameter	Campaign 1	Campaign 2
Feed	2.5 g/L Ni (simulated bioleach liquor)	5 g/L Ni (simulated bioleach liquor)
Organic phase	5 vol.% V10 V10:Nicksyn™ ratio of 2:1 Varied O:A phase ratios	10 vol.% V10 V10:Nicksyn™ ratio of 2:1 Varied O:A phase ratios
Strip solution	60 g/L Ni, 50 g/L H ₂ SO ₄ , 4 g/L H ₃ BO ₄ O:A phase ratio = 10	60 g/L Ni, 50 g/L H ₂ SO ₄ , 4 g/L H ₃ BO ₄ O:A phase ratio = 10
Temperature	Ambient	Ambient
Number of stages	4 extraction 3 stripping No scrubbing	4 extraction 3 stripping No scrubbing
Residence time	3-6 minutes	3-6 minutes
pH	5.8-6.0	5.7-6.0

3.1.4. Extraction metal distribution equilibria (pH vs. extraction)

The results for the pH vs. extraction curves generated using the different feed solutions in Table 1 are summarised in Table 4. The pH₅₀ value (the pH at which 50% of the metal originally present in the aqueous phase is extracted under a given set of conditions) and

the separations between the extraction profiles (ΔpH_{50}) for nickel and calcium, and cobalt and manganese are given.

Table 4: pH_{50} values of some metals obtained during extraction with V10 and Nicksyn™ mixtures

Temperature, °C	V10, vol.%	V10:Nicksyn™, molar ratio	pH_{50}				ΔpH_{50}	
			Ni	Ca	Co	Mn	Ca-Ni	Mn-Co
Biobleach liquor (2.5-5 g/L Ni; Table 2)								
45	5	2:1	5.3	7.4	6.1	6.6	2.1	0.5
45	5	1:1	5.0	7.5	5.7	6.5	2.5	0.8
TAP leach liquor (30 g/L Ni; Table 2)								
25	30	2:1	4.4	7.4	5.6	6.6	3.0	1.0
25	30	1:1	4.4	7.6	5.5	6.6	3.2	1.1
Laterite leach liquor (3 g/L Ni; 20 g/L Mg)								
25	3	V10 alone	7.3	-*	7.4	7.6	-	0.2
25	3	2:1	5.8	-*	6.5	-**	-	-
50	9.6	V10 alone	5.8	-*	6.0	5.6	-	-0.4 [#]
50	9.6	1:1	<5.0	-*	5.2	6.1	-	0.9
50	4.8	1:1	5.1	-*	5.6	6.3	-	0.7

* Calcium extraction was too low to determine pH_{50} value

** Manganese extraction only ~20% at pH 7

Manganese was extracted before cobalt

As previously discussed^{1,2}, the pH_{50} value for the extraction of nickel by V10 alone can vary between pH 6 and 7, depending on the concentration of nickel in the feed solution and the concentration of V10 used. Tight pH control is required to minimize the co-extraction of calcium in order to prevent gypsum precipitation during the recovery of nickel. The addition of Nicksyn™ can cause a substantial synergistic shift for nickel (up to 3 pH units), without affecting calcium extraction, enabling operation at lower pH values and improved nickel recoveries (employing the same number of stages). The cost (employing the same number of stages) implication of the addition of Nicksyn™ justifies the optimization of the molar ratio of V10:Nicksyn™ to be used for the specific feed solution, which is variable².

Tests conducted using a simulated biobleach liquor and 5 vol.% V10 indicated that the separation between calcium and nickel increased from 2.0 to 2.5 pH units with decreasing V10:Nicksyn™ ratios from 2:1 to 1:1, respectively, at 45°C, while the separation between manganese and cobalt increased from 0.5 to 0.8 pH units.

In the case of the simulated TAP leach liquor, the separation between calcium and nickel increased from 2.98 to 3.19 pH units using V10:Nicksyn ratios of 2:1 and 1:1, respectively, at 25°C, with no significant difference in the separation between manganese and cobalt for the two different ratios of V10:Nicksyn™ at the same temperature.

Extracting nickel from a simulated laterite leach liquor containing high magnesium (~20 g/L) concentrations, resulted in a synergistic shift in pH₅₀ values for nickel from 7.3 (using 3 vol.% V10 alone) to 5.8 (using V10:Nicksyn™ ratio of 2:1) at 25°C. In both cases <4% calcium extraction occurred only at pH 7, hence the pH₅₀ value for calcium extraction could not be determined. The ΔpH₅₀(Mn-Co) was 0.20 units when using V10 alone, but could not be determined when using V10 plus Nicksyn™ due to the low (~16 %) manganese extraction up to pH 7. Increasing the V10 concentration to 9.6 vol.% resulted in a further decrease in the pH₅₀ value for nickel from 5.8 (with V10 alone) to <5.0 (using a ratio of V10:Nicksyn™ of 1) at 50°C, with less than 18% extraction of calcium up to pH 7. It is interesting to note that when V10 (9.6 vol.%) alone was used, manganese was extracted before nickel and cobalt, resulting in a ΔpH₅₀(Mn-Co) value of -0.4. When Nicksyn™ was added, the ΔpH₅₀(Mn-Co) increased to 0.8. The pH₅₀ value for nickel using 4.8 vol.% V10 and V10:Nicksyn™ ratio of 1 (and at 50°C) was 5.1, with <4% calcium extraction occurring up to pH 7 and a ΔpH₅₀(Mn-Co) of 0.7 units.

These results show that optimising the V10/Nicksyn™ ratio for specific requirements can result in almost complete rejection of calcium and magnesium, while some manganese extraction might have to be tolerated if cobalt recovery is targeted to minimize the number of extraction stages. This manganese could be scrubbed off the loaded organic phase to a large extent.

3.1.5. Distribution isotherms for nickel extraction

Conditions for the generation of distribution isotherms for the extraction of nickel are shown in Table 5.

Table 5: Conditions for the generation of some nickel extraction isotherms

Test	Ni in feed solution, g/L	Temperature, °C	pH*	V10, vol.%	V10:Nicksyn™, molar ratio
1	2.5	Ambient	6.0	5	2:1
2	5	45	6.0	5	2:1
3	5	45	6.0	10	2:1
4	30	45	6.0	30	2:1

* pH was controlled at 6 using ~10 M NaOH solution

The distribution isotherms and McCabe-Thiele constructions for the extraction of nickel from different leach liquors by different V10 concentrations (but in the same V10:Nicksyn™ ratio of 2:1) at pH 6 and different temperatures are shown in Figure 1.

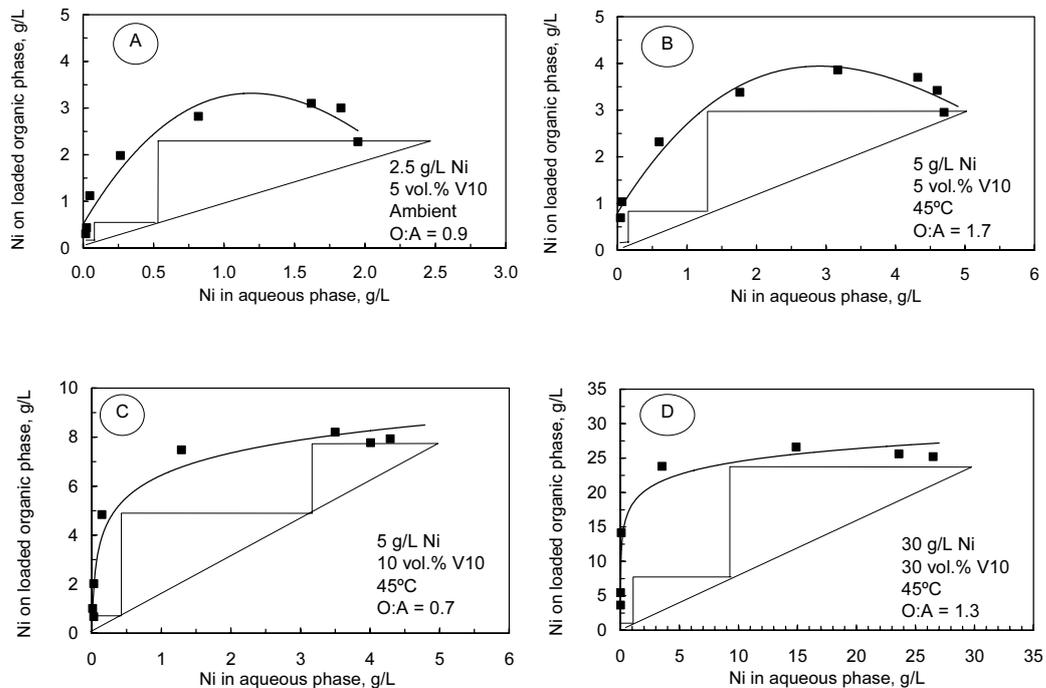


Figure 1: Distribution isotherms and McCabe-Thiele constructions for the extraction of nickel from different simulated leach liquors using different V10 concentrations in a V10:Nicksyn™ ratio of 2:1 and at different temperatures

The McCabe-Thiele constructions indicate that 3 countercurrent extraction stages are required in all cases shown in Figure 1. A loading of about 2.3 g/L nickel could be achieved at an O:A phase ratio of 0.9 using the simulated bioleach liquor containing 2.5 g/L nickel, leaving a raffinate of ~24 mg/L at ambient temperature (Case A). A higher O:A (1.7) phase ratio would be required to extract nickel at 45°C from a simulated bioleach liquor containing 5 g/L nickel, producing a raffinate of ~42 mg/L (Case B). When the concentration of V10 is increased to 10 vol.% for the same leach liquor, a loading of ~7.3 g/L can be achieved at an O:A phase ratio of 0.7 with a raffinate containing ~30 mg/L at 45°C (Case C). At 30 g/L nickel in the leach liquor, 30 vol.% V10 in the synergistic mixture resulted in a loaded organic phase of ~23 g/L nickel at an O:A phase ratio of 1.3, with a raffinate of ~14 mg/L at 45°C (Case D).

3.1.6. Summary of laboratory results

It is clear from these results that the operating conditions for a specific leach liquor to achieve the required nickel loading and raffinate can be optimized by varying the

V10:Nicksyn™ ratio, or the O:A phase ratio depending on the optimized economic outcome. Temperature improved the separation of nickel and cobalt from calcium, magnesium and manganese. Three extraction stages were adequate in most cases, as was demonstrated on the TAP plant².

3.2. Continuous countercurrent evaluations

3.2.1. Campaign 1

A continuous countercurrent evaluation (Campaign 1) was done using the operating conditions in Table 3 to determine the effect of the O:A phase ratio, pH and residence time on the extraction of nickel from a simulated bioleach liquor containing about 2.5 g/L Ni, 0.25 g/L Co, 1 g/L Mn, 1 g/L Mg, 100 mg/L Cu, 100 mg/L Zn and 0.5-0.6 g/L Ca using 5 vol.% V10 in a V10:Nicksyn™ ratio of 2:1 and at ambient temperature. The conditions applied during this campaign were:

- Profile 1: O:A of 1; residence time 3 min; E1, E2, E3 and E4 at pH 6;
- Profile 2: O:A of 1; residence time 3 min; E1, E2, E3 and E4 at pH 5.8;
- Profile 3: O:A of 1; residence time 6 min; E1, E2, E3 and E4 at pH 5.8;
- Profile 4: O:A of 2; residence time 3 min; E1 and E2 at pH 5.8; E3 and E4 at pH 6

3.2.1.1. Extraction

The aqueous and organic phase equilibrium nickel concentrations in each of the profiles taken over the different extraction stages are shown in Figure 2. As a basis, the extraction equilibrium isotherm generated during laboratory experiments using an organic phase with the same composition of V10 and Nicksyn™ as that used for Campaign 1 and at pH 6 is shown, as well as the profile number for each isotherm together with the O:A phase ratio, pH profile (E1-E4 pH) and residence time employed.

It is evident that higher extraction pH, O:A phase ratio and residence time positively influenced the extraction efficiency of nickel in these systems. The following observations were made:

- The two profiles generated at an O:A phase ratio of 1 and 3 minute residence time (Profiles 1 and 2) appeared similar in Figure 2, but in Figure 3 the profile generated at a higher pH (pH 6 compared to pH 5.8) indicated the advantage of higher pH for nickel extraction.
- Of all the conditions tested the profile generated employing a longer residence time (6 minutes) was closest to the actual equilibrium profile (Profile 3).
- Profile 4 was flat due to the phase ratio increase to 2, which resulted in half the nickel loading.

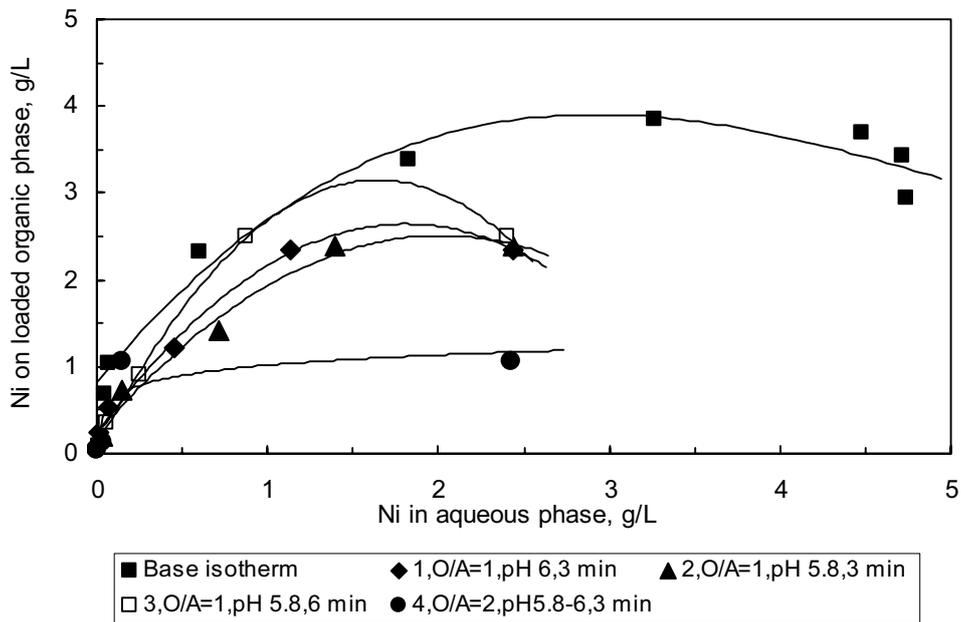


Figure 2: Extraction isotherms generated during Campaign 1 for nickel (2.5 g/L) at different profiles using 5 vol.% V10 in a 2:1 molar ratio with Nicksyn™ at pH 6 and at ambient temperature

The nickel aqueous concentration profile across the extraction bank for the conditions tested is shown in Figure 3.

Although the nickel concentration profiles illustrate the advantageous effect of increased O:A phase ratio (2 compared to 1), extraction pH (6 compared to 5.8) and residence time (6 min compared to 3 min) on the extent of extraction, nickel concentrations in the raffinate (E4) are similar for all the profiles. It may be advisable to operate at the lower O:A phase ratio and pH to limit the co-extraction of impurities. An increased residence time has implications on the size of the mixer and overall SX circuit. The removal of one extraction stage could also be considered, especially for the longer residence time. All profiles yielded nickel extraction of greater than 99% with raffinates less than 15 mg/L, except Profile 2, in which case the nickel extraction was 98.8% and the raffinate ~30 mg/L.

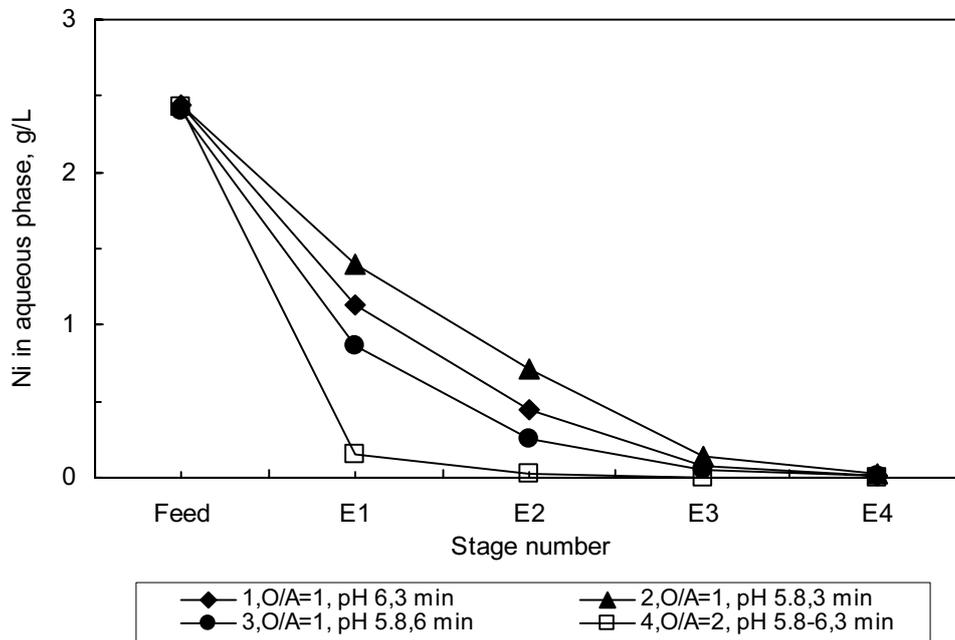


Figure 3: Nickel aqueous concentration profile across extraction bank for Campaign 1 using 5 vol.% V10 in a 2:1 molar ratio with Nicksyn™ and at ambient temperature

Although the nickel concentration profiles illustrate the advantageous effect of increased O:A phase ratio (2 compared to 1), extraction pH (6 compared to 5.8) and residence time (6 min compared to 3 min) on the extent of extraction, nickel concentrations in the raffinate (E4) are similar for all the profiles. It may be advisable to operate at the lower O:A phase ratio and pH to limit the co-extraction of impurities. An increased residence time has implications on the size of the mixer and overall SX circuit. The removal of one extraction stage could also be considered, especially for the longer residence time. All profiles yielded nickel extraction of greater than 99% with raffinates less than 15 mg/L, except Profile 2, in which case the nickel extraction was 98.8% and the raffinate ~30 mg/L.

Extraction impurity organic profiles (Profile 2 and 4) for the conditions tested are shown in Figure 4.

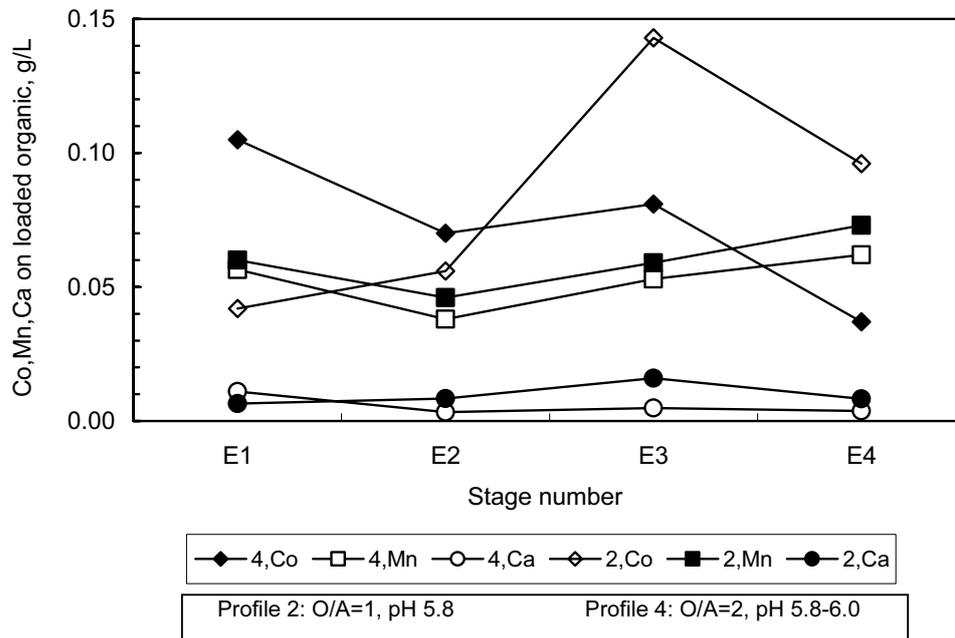


Figure 4: Cobalt, manganese and calcium organic concentration profiles across extraction bank for Campaign 1 using 5 vol.% V10 in a 2:1 molar ratio with Nicksyn™ and at ambient temperature

The ‘scrubbing’ of co-extracted cobalt is clearly illustrated during Profile 2 (O/A = 1, pH 5.8) where the concentration of cobalt on the organic decreased from about 140 mg/L in E3 to about 40 mg/L in E1. The increase in the O:A phase ratio to 2 (Profile 4) and therefore increasing the organic capacity, resulted in co-loading of cobalt of about 105 mg/L in E1 (Profile 4) and no ‘scrubbing’ across the circuit. Co-extracted manganese was ‘squeezed off’ to a lesser extent only from about 70 to 60 mg/L for both profiles, while the co-extraction of calcium was limited to about 10 mg/L on the loaded organic across the entire circuit.

3.2.1.2. Stripping

Stripping was done using a simulated strip solution (see Table 1) and an average O:A phase ratio of 10. No optimisation was targeted. The performance of the stripping circuit is summarised in Table 6.

Table 6: Stripping of metals from loaded 5 vol.% V10 in a 2:1 molar ratio with Nicksyn™ with a synthetic strip solution (~60 g/L Ni) at ambient temperature

Profile no.	Stripping, %					
	Ni	Co	Mn	Zn	Cu	Ca
1	97.8	97.2	60.0	98.7	98.1	85.5
2	98.3	96.8	46.7	98.8	98.2	84.5
3	98.3	98.5	63.5	98.9	98.2	85.2
4	96.1	98.4	54.0	97.2	95.4	88.2

Efficient nickel stripping (>96%) was achieved during all profiles employing an O:A extraction phase ratio of 1 under the chosen conditions. Profile 4 (O:A extraction phase ratio of 2) indicated slightly less efficient stripping.

3.2.2. Campaign 2

Campaign 2 was run using the operating conditions in Table 3 to determine the effect of O:A phase ratio and pH (using a 3 minute residence time) on the extraction of nickel from a simulated bioleach liquor containing about 5 g/L Ni, 0.5 g/L Co, 1 g/L Mn, 1 g/L Mg, 100 mg/L Cu, 100 mg/L Zn and 0.5-0.6 g/L Ca using 10 vol.% V10 in a V10:Nicksyn™ ratio of 2:1 at ambient temperature. The intended conditions aimed for were:

- Profile 1: O:A of 2; residence time 3 min; E1, E2, E3 and E4 at pH 5.8;
- Profile 2: O:A of 1; residence time 3 min; E1, E2, E3 and E4 at pH 5.8;
- Profile 3: O:A of 1; residence time 3 min; E1 at pH 5.7, E2, E3 and E4 at pH 5.8;
- Profile 4: as Profile 3

3.2.2.1. Extraction

The aqueous and organic phase equilibrium nickel concentrations in each of the extraction stages are shown in Figure 5. As a basis, the extraction equilibrium isotherm generated during laboratory experiments using an organic phase with the same composition of V10 and Nicksyn™ as that used for piloting and at pH 6 is also shown, as well as the profile number for each isotherm together with the O:A phase ratio and pH profile (E1-E4 pH) employed.

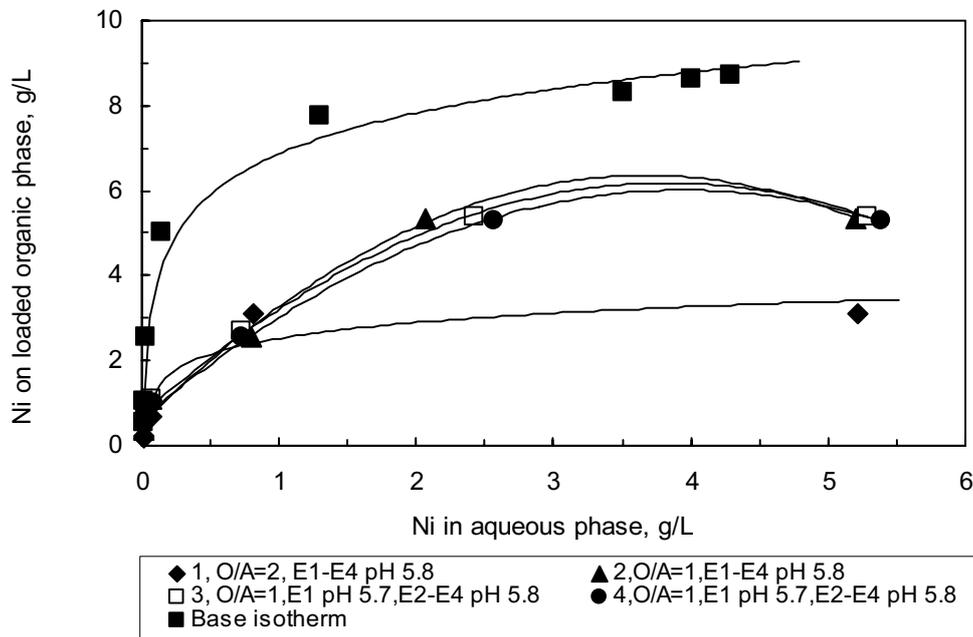


Figure 5: Extraction isotherms generated during Campaign 2 for nickel (5 g/L) at different profiles using 10 vol.% V10 in a 2:1 molar ratio with Nicksyn™ and at ambient temperature

The isotherms generated during Campaign 2 (Profile 2 to 4) indicated organic loadings of nickel of ~5 g/L, which was lower than that predicted by the McCabe-Thiele constructions (7.3 g/L, see Figure 1C). A number of factors contributed to the poorer performance, including a somewhat lower extraction pH and an inadequate residence time.

The nickel aqueous concentration profile across the extraction bank for each of the isotherms illustrated is shown in Figure 6.

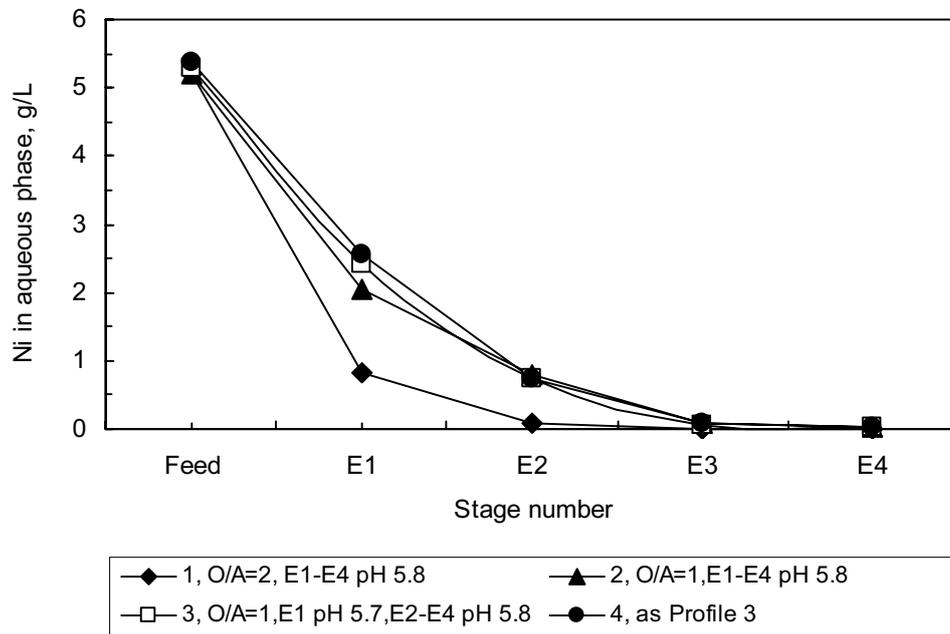


Figure 6: Nickel aqueous concentration profile across extraction bank for Campaign 2 using 10 vol.% V10 in a 2:1 molar ratio with Nicksyn™ and at ambient temperature

The advantageous effect of increased O:A phase ratio (2 compared to 1) on the extent of nickel extraction was again illustrated in the nickel concentration profiles. Nickel concentrations in the raffinate (E4) were similar for all the profiles in stages E3 and E4, which indicated the possibility of removal of one extraction stage. The slightly lower pH in E1 had no significant effect on nickel extraction, comparing Profiles 2 and 3. All profiles yielded nickel extraction of greater than 99% and raffinates of less than 20 mg/L nickel.

Extraction impurity organic profiles (Profile 1 and 2) for the conditions tested are shown in Figure 7.

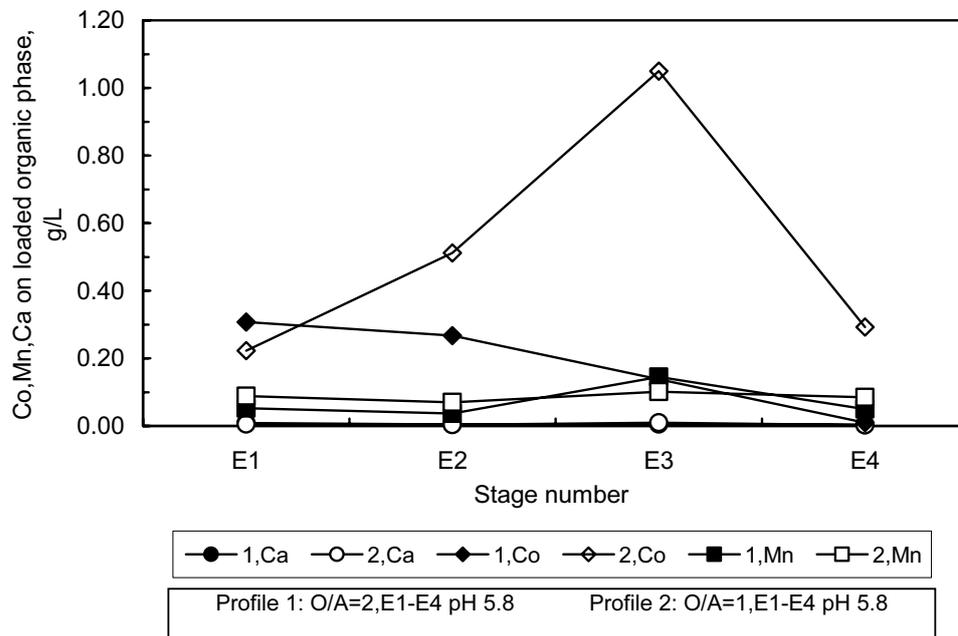


Figure 7: Cobalt, manganese and calcium organic concentration profiles across extraction bank for Campaign 2 using 10 vol.% V10 in a 2:1 molar ratio with Nicksyn™ and at ambient temperature

As seen previously, cobalt extraction is highest (~300 mg/L in E1) at an O:A phase ratio of 2 (Profile 1). When the O:A phase ratio is reduced to 1, cobalt is initially extracted to up to 1000 mg/L (in E3), but then ‘squeezed off’ to about 220 mg/L in E1 (Profile 2). Manganese was co-extracted up to about 150 mg/L (E3, Profile 1), but ‘scrubbed’ to about 50 mg/L in E1 (Profile 1). The concentration of calcium on the loaded organic phase for E1 (Profile 1, O:A phase ratio of 2) was about 80 mg/L. This calcium loading would cause gypsum precipitation problems in the scrub and strip circuits. Decreasing the O:A phase ratio to 1 (Profile 2), decreased the co-loading of calcium on the organic phase in E1 to about 5 mg/L.

4. CONCLUSIONS

- Laboratory tests were conducted to investigate the effect of the addition of Nicksyn™ to V10 for nickel recovery from synthetic feed solutions made up to represent typical bioleach, TAP and laterite leach solutions.
- Separation (ΔpH_{50} values) between nickel and calcium varied between 2.0 and 3.2 pH units, while cobalt/manganese separations ranged between 0.51 and 1.10 pH units, depending on the conditions used.

- Distribution isotherms and McCabe-Thiele constructions for the extraction of nickel indicated that three extraction stages should be adequate for efficient nickel recovery from a wide range of streams. Operating conditions for a specific leach liquor to achieve the required nickel loading and raffinate can be optimised by varying the V10:Nicksyn™ ratio. Temperature improved the separation of nickel and cobalt from calcium, manganese and magnesium.
- Two continuous countercurrent campaigns were run to determine optimum conditions for nickel recovery with minimum calcium co-extraction, using simulated bioleach liquors containing 2.5 and 5 g/L, respectively, and at ambient temperature. The organic phases employed were 5 and 10 vol.% V10 (for 2.5 and 5 g/L nickel feed solution, respectively) with the addition of Nicksyn™ in a 2:1 ratio.
- Efficient extraction (>99%) of nickel was achieved in all cases with raffinates containing <20 mg/L nickel.
- O:A extraction phase ratios of 2 resulted in higher extraction of impurities such as calcium, cobalt and manganese than with O:A phase ratios of 1. Calcium co-extraction varied between 5 and 20 mg/L. In one case where an O:A phase ratio was employed it was ~80 mg/L. Co-extracted cobalt and manganese could be ‘scrubbed off’ during extraction to a large extent. Co-extracted cobalt concentrations on the loaded organic phases varied between 100 and 220 mg/L, with manganese varying between 50 and 60 mg/L.
- Cobalt can be recovered by increasing the O:A phase ratio and/or pH of extraction, optimised for limited or no calcium co-extraction.
- These results confirm the importance of optimization of operating conditions for a specific leach liquor to achieve the required nickel loading and raffinate with regards to V10:Nicksyn™ ratio and concentration, O:A phase ratio, residence time, pH and temperature.

5. REFERENCES

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