Separation of nickel and cobalt from calcium, magnesium and manganese by solvent extraction with synergistic mixtures of carboxylic acids

by A.C. du Preez* and J.S. Preston*

Synopsis

The separation of nickel and cobalt from calcium, magnesium and manganese by solvent extraction using synergistic mixtures of carboxylic acids was studied. Addition of certain viable synergists to commercially available tertiary branched carboxylic acids, such as Versatic 10 (Shell) or Neodecanoic acid (Exxon), produced large synergistic shifts (ΔpH_{50}) in the pH_{50} values for nickel, and small antagonistic shifts (negative ΔpH_{50} values) in the extraction of calcium. Under the initial test conditions used (0.50 M each of Neodecanoic acid and the Mintek synergist, in xylene) a large separation ($pH_{50}^{Ca} - pH_{50}^{Ni} = 3.09$ pH units) between nickel and calcium in their extraction from 1.00 M sodium nitrate solutions was obtained, compared with only 1.11 pH units in the absence of the synergist.

The selectivity series for the extraction of divalent base metals from 0.333 M sodium sulphate solutions by 0.50 M Neodecanoic acid and 0.25 M of the Mintek synergist in C_{12}-C_{13} aliphatic diluent was found to lie in the order (with pH_{50} values in parentheses): Cu (3.42) > Ni (5.08) > Zn (5.58) > Co (5.73) > Mn (6.62) > Ca (7.48) > Mg (8.25). In batch countercurrent experiments, a simulated leach liquor containing (in gL^{-1}): Ni 5, Mg 5, Mn 2, Co 0.5, Ca 0.5, Cu 0.1 and Zn 0.1 was contacted with the mixed extractant in three stages at unit phase ratio and an equilibrium pH of 5.9. The recovery of nickel was 99.9 per cent and that of cobalt was 84.7 per cent, whilst the co-extractions of manganese, calcium and magnesium were 2.7, 1.5 and 0.04 per cent, respectively. The recovery of cobalt can be enhanced by employing a slightly higher pH, although this will also increase the extraction of manganese, calcium and magnesium somewhat.

Nearly quantitative stripping (99 per cent) of nickel from the loaded organic phase was achieved in a single contact at an organic-to-aqueous phase ratio of 4 using a simulated spent electrolyte containing 39 gL^{-1} sulphuric acid and 33 gL^{-1} nickel, thereby producing an 'advance electrolyte' containing 57 gL^{-1} nickel at pH 3.5. Loading of acid into the organic phase was very slight, amounting to only 0.002 M H$^+$ in contact with the above-mentioned spent electrolyte.

Keywords: solvent extraction; synergism; nickel; calcium; carboxylic acids.

Introduction

The potential use of synergistic mixtures of reagents for the solvent extraction of nickel (and cobalt) from lateritic leach liquors was pointed out in the early 1980s by Grinstead, who used dinonylnaphthalene sulphonic acid in conjunction with some 2-pyridyl compounds for the recovery of these metals from slightly acidic solutions[1,2]. Shortly thereafter, studies at Mintek revealed marked synergistic effects in the extraction of nickel by organophosphoric acids in the presence of certain non-chelating oximes[3,4].

Subsequently, we extended our work to include mixtures of carboxylic acids and non-chelating oximes, and it was shown that several potentially useful separations between valuable and gangue metals could be obtained, for example $pH_{50}^{Ca} - pH_{50}^{Ni} = 3.5$ pH units, and $pH_{50}^{Mn} - pH_{50}^{Co} = 1.5$ pH units[5]. This work was patented in several countries during the early 1980s[6], but the depressed state of the nickel market at that time discouraged any further development of these extractants.

Some time later, however, when renewed interest began to be expressed in the recovery of nickel from laterite sources[7], we resumed our studies on mixed extractant systems, and chose to use synergists different from the less acid-resistant oximes used previously[8]. In continuing studies[9–11], particular attention was given to the separation of nickel from calcium since, in commercial operations, the low solubility of the sulphate salt of the latter metal can result in the troublesome precipitation of gypsum ($CaSO_4.2H_2O$), not only in the stripping and scrubbing circuits (where high organic-to-aqueous phase ratios are desirable in order to upgrade the concentration of nickel from its value in the leach liquor), but sometimes in the extraction circuit as well[12]. The separation of cobalt and manganese is also of consideration, as this would allow recovery of nickel and cobalt from sulphate leach liquors, whilst effectively rejecting the manganese, calcium and magnesium in the

These extraction systems are characterized by substantial synergistic shifts (ΔpH_{50}) in the

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pH_{50} values for the extraction of nickel that occur when the synergist is added to the system. In many cases, the synergistic effect for nickel is accompanied by an antagonistic effect in the extraction of calcium (i.e. ΔpH_{50}^{Ca} is negative), with the overall result that the separation between the two metals ($pH_{50}^{Ca} - pH_{50}^{Ni}$) can be in the range of 3 to 4 pH units, in comparison with 0.5 to 1 pH unit for the carboxylic acid alone. Mixtures based on alkylsalicylic acids were found to give rise to particularly large synergistic shifts in the pH_{50} values for the extraction of nickel[10,11], although the nickel-calcium separations attainable in systems based on tertiary-branched carboxylic acids were also found to be adequate[8,11]. In addition, alkylpyridines (such as 4-(5-nonyl)pyridine) were found to produce the largest synergistic shifts for nickel, as well as the largest antagonistic effects for calcium.

In so far as potential commercial applications are concerned, however, the choice of the extractant mixture is strongly influenced by additional factors such as the availability, the stability and the cost of the components. Taking this into consideration, it seemed a reasonable compromise to base the extractant on a commercially available tertiary-branched carboxylic acid, such as Versatic 10 (Shell Chemicals) or Neodecanoic acid (Exxon), together with a commercially available or readily synthesized synergist. The potential use of the erstwhile copper extractant Avecia CLX50 (formerly Acorga DS5443A) as synergist was demonstrated at an early stage[8] and was later tested briefly elsewhere[13]. However, this compound (diisodecyl 3,5-pyridinedicarboxylate) is fairly expensive to manufacture, and has been reported to be no longer commercially available[14].

In this work, we present data on the extraction properties of mixtures of carboxylic acids and different viable synergists, with a view to designing a hydrometallurgical process for the recovery of nickel from dilute acidic leach liquors. For the preferred system (Neodecanoic acid plus Mintek synergist), extraction and stripping isotherms are presented, together with the results of batch countercurrent tests and measurements of the uptake of acid into the organic phase during stripping.

Experimental

Solvent extraction reagents

Versatic 10 and Neodecanoic acid were obtained from Shell Chemicals and Exxon, respectively, whilst CLX50 was a sample that had been provided under the designation DS5443A by the former Zeneca Specialties. The Mintek synergist was initially prepared on a laboratory scale at Mintek.

Metal-distribution studies

Distribution equilibria for nickel, calcium and other metals were determined at $20 \pm 0.2°C$ (unless stated otherwise) by means of the technique described previously[8].

To permit comparison with our earlier work, distribution data were obtained in the first instance using xylene as the organic diluent, and sodium nitrate (1.00 M) as the 'background electrolyte' in the aqueous phase. However, in the experiments related to the design of a practical process for the recovery of nickel and cobalt (extraction and stripping isotherms, batch countercurrent tests, etc.), a commercial aliphatic C_{12}-C_{13} hydrocarbon diluent (obtained as SSX 210 from Sasol Wax) was employed, and aqueous phases were prepared from sulphate salts.

Acid extraction by organic phase

Aliquots of an aqueous phase (15 ml) consisting of 0.25 M sulphuric acid and 0.25 M sodium sulphate, were adjusted to the required pH using a 10 M sodium hydroxide solution, and contacted with the appropriate organic phase (15 ml). A sample of the organic phase (5 ml) was stripped with water (50 ml) and the resulting aqueous phase was separated and titrated against a 0.10 M sodium hydroxide solution to a bromothymol blue endpoint. The pH of the raffinate was measured.

Results and discussion

Extraction of nickel and calcium

Figure 1 shows the extraction of nickel and calcium (separately, as 0.05 M metal nitrates in 1.00 M sodium nitrate) by 0.50 M Neodecanoic acid and its mixture with 0.50 M of the Mintek synergist in xylene. Addition of the synergist causes a substantial synergistic shift for nickel ($\Delta pH_{50}^{Ni} = 1.72$ pH units), as well as a small antagonistic shift for calcium ($\Delta pH_{50}^{Ca} = -0.26$ pH units). Consequently, the separation between the two metals (expressed as the difference between their pH_{50} values, $pH_{50}^{Ca-Ni} = pH_{50}^{Ca} - pH_{50}^{Ni}$) increases from 1.11 pH units for Neodecanoic acid alone to 3.09 pH units for the mixed extractant.

The origins of the synergistic and antagonistic effects were discussed previously[11] in terms of the competing equilibria:

$$Ni^{2+} + H_2A_2 + 2L = NiA_2L_2 + 2H^+ \qquad [1]$$

$$Ca^{2+} + 3H_2A_2 = CaA_2(HA)_4 + 2H^+ \qquad [2]$$

$$H_2A_2 = 2L = 2HA.L \qquad [3]$$

where H_2A_2 denotes the carboxylic acid dimer and L denotes the synergist. It was shown previously that the selectivity towards nickel is enhanced by the use of a sterically hindered carboxylic acid in conjunction with a synergist of low steric bulk[9,11].

Effect of structure of extractant on separation of nickel and calcium

Table I shows the effect of the addition of different concentrations of the Mintek synergist and CLX50 on the extraction of nickel and calcium from 1.00 M $NaNO_3$ solutions by 0.50 M Neodecanoic acid in xylene. The synergistic effect for equimolar amounts of these extractants is somewhat greater for the Mintek synergist than for the CLX50. A good separation between nickel and calcium can be effected in the pH range of about 5.75–6.75 using Neodecanoic acid with either 0.25 M Mintek synergist or CLX50 as shown in Figure 2.
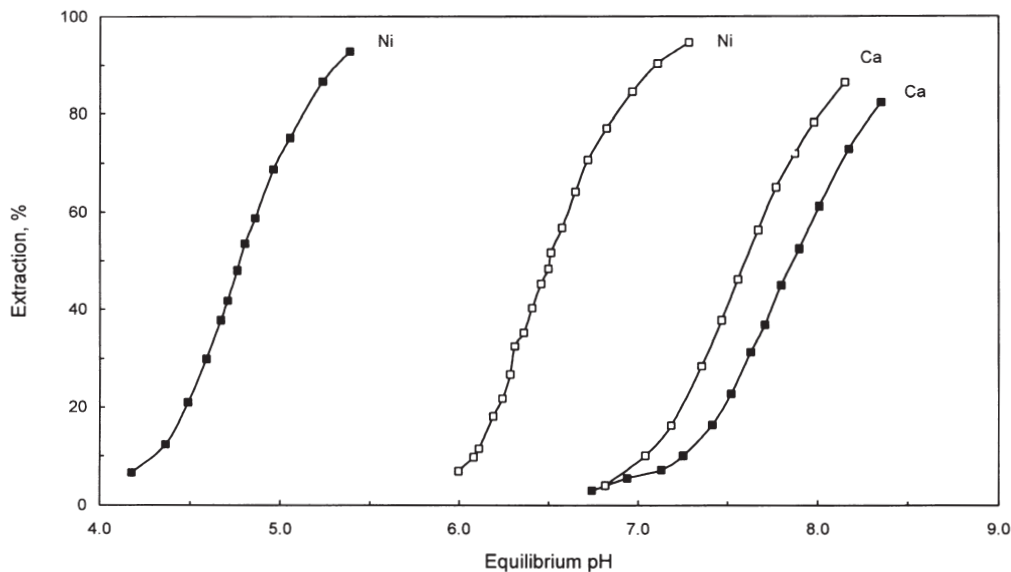
Figure 1—Extraction of nickel and calcium (0.05 M, separately) from 1.00 M NaNO₃ by 0.50 M Neodecanoic acid (white symbols) and its mixture with 0.50 M Mintek synergist in xylene (black symbols)

Table I

Values of pH_{50} and ΔpH_{50} in the extraction of nickel and calcium (0.05 M as nitrates) from solutions of sodium nitrate (1.00 M) by mixtures of Neodecanoic acid (0.50 M) and Mintek synergist or CLX50 (0.125–0.5 M) in xylene at 20°C

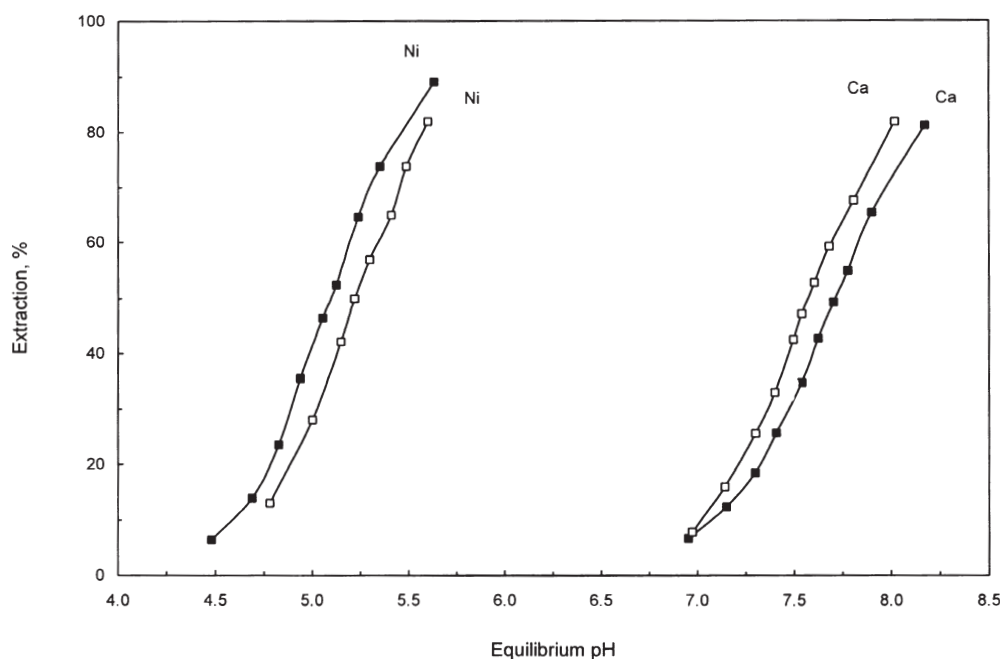| Synergist	Conc (M)	pH_{50}			ΔpH_{50}	
		Ni	Ca	Ca-Ni	Ni	Ca
Mintek synergist	0.50	4.78	7.87	3.09	1.72	-0.26
	0.25	5.10	7.71	2.62	1.40	-0.10
	0.125	5.38	7.60	2.22	1.12	0.01
CLX50	0.25	5.22	7.58	2.36	1.28	0.03
	0.125	5.50	7.55	2.05	1.00	0.06

Figure 2—Extraction of nickel and calcium (0.05 M, separately) from 1.00 M NaNO₃ by 0.50 M Neodecanoic acid plus 0.25 M Mintek synergist (black symbols) and by 0.50 M Neodecanoic acid plus 0.25 M CLX50 in xylene (white symbols)

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It is important to note that in comparing the efficacy of these synergists, the Mintek synergist as prepared in this work (76 wt. per cent in unreacted reagent) has the added advantage over CLX50 (54 wt. per cent in hydrocarbon diluent) in its appreciably lower molecular weight. Thus, a 0.25 M solution of the Mintek synergist corresponds to a 9.3 vol. per cent solution, whereas a solution of the CLX50 of the same molarity corresponds to a 23.5 vol. per cent solution as supplied. In subsequent experiments, the concentration of carboxylic acid was set at 0.50 M, while that of the synergist was decreased to 0.25 M for reasons of economy, especially in cases where comparisons with CLX50 were required.

The effect of temperature on the separation of nickel and calcium

In the investigation of the effect of temperature, a mixed extractant containing Neodecanoic acid (0.50 M) and Mintek synergist (0.25 M) in C_{12}-C_{13} paraffin diluent was used. Aqueous phases containing 0.333 M sodium sulphate and 0.05 M nickel sulphate (or 0.02 M calcium sulphate) were used to relate more directly to practical extraction conditions.

Temperature was found to have little effect on the pH_{50} values for the extraction of nickel, these being 5.05, 5.07 and 5.01 respectively at 40, 20 and 5°C. The effect on the pH_{50} values for extraction of calcium was more marked, however, these being 7.20, 7.47 and 7.57, respectively. As a result, the separation between the two metals (pH_{50}^{Ca-Ni}) increases with decreasing temperature, the values being 2.15, 2.40 and 2.56 pH units at 40, 20 and 5°C, respectively.

Selectivity series for divalent base metals

Figure 3 shows the extraction vs. pH curves for the principal divalent base metals (0.05 M sulphates in 0.333 M sodium sulphate) that might be expected to occur in liquors prepared by acid leaching of nickel laterites. It is assumed that trivalent impurities (Fe^{3+}, Al^{3+} and Cr^{3+}) would be removed by co-precipitation of their hydroxides before the leach liquor entered the solvent extraction circuit. The order of extraction

for this system (with pH_{50} values shown in parentheses) is:
Cu (3.42) > Ni (5.08) > Zn (5.58) > Co (5.73) > Mn (6.62) > Ca (7.48) > Mg (8.25).

This order is the same as that reported for mixtures of Versatic 10 and 4-nonylpyridine[11], and suggests that these metals can be separated by pH-control into two groups comprising the valuable metals (Cu, Ni, Co and Zn) and the gangue metals (Mn, Ca, Mg). Since cobalt is the least strongly extracted of the valuable metals, and manganese is the most strongly extracted of the gangue metals, the Co-Mn separation accordingly assumes particular importance.

Distribution isotherms for extraction of nickel and cobalt

Distribution isotherms for the extraction of nickel and cobalt from 0.333 M sodium sulphate solutions by 0.50 M Neodecanoic acid and 0.25 M Mintek synergist in C_{12}-C_{13} paraffin diluent are shown in Figure 4. At an equilibrium pH of 5.50, there exists an appreciable selectivity for nickel over cobalt (Figure 4a), whereas at pH 6.0 both metals are strongly extracted (Figure 4b), with 2 countercurrent stages at unit phase ratio being sufficient for 99 per cent recovery of nickel, and 3 stages for 96 per cent recovery of cobalt. However, in a commercial process it is probable that the co-extraction of these metals would be more convenient, rather than attempting their separation at this point.

Batch countercurrent extraction experiments

The first batch countercurrent extraction experiment was conducted using an aqueous feed solution containing (gL^{-1}): Ni 5.00, Co 0.51, Cu 0.09, Zn 0.10, Mn 1.93, Ca 0.48 and Mg 4.96 and an organic phase containing 0.50 M Neodecanoic acid plus 0.25 M Mintek synergist in C_{12}-C_{13} diluent. Three extraction stages were used with an organic-to-aqueous phase ratio of unity and an equilibrium pH value of 5.9 in each stage over a total of six full cycles. The results given in Table II show that high extractions are obtained for the valuable metals (Ni 99.9, Co 84.7, Cu 100 and Zn 100 per
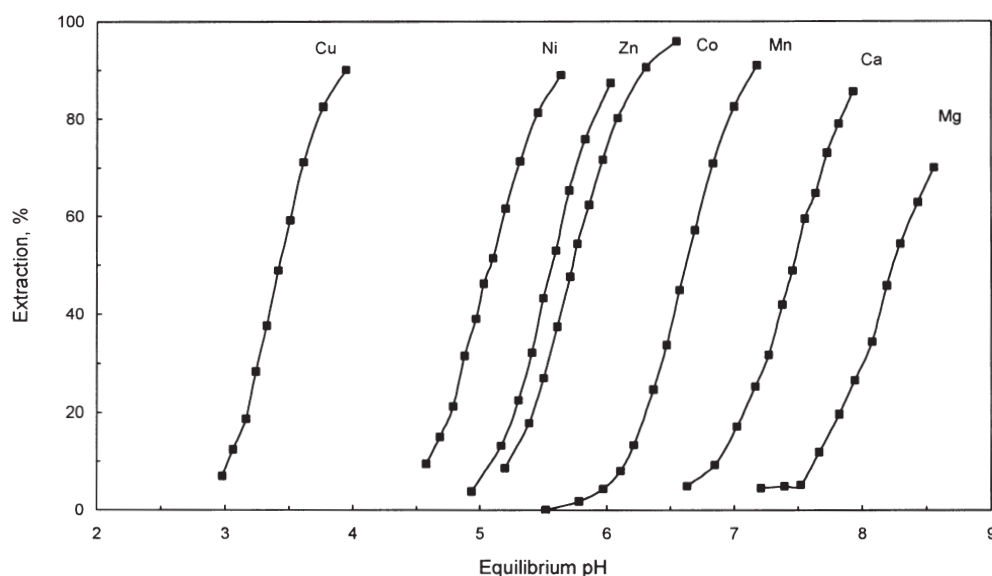
Figure 3—Extraction of divalent base metals (0.05 M separately as sulphates, calcium 0.02 M) from 0.333 M Na₂SO₄ by 0.50 M Neodecanoic acid plus 0.25 M Mintek synergist in C_{12}-C_{13} diluent at 20°C

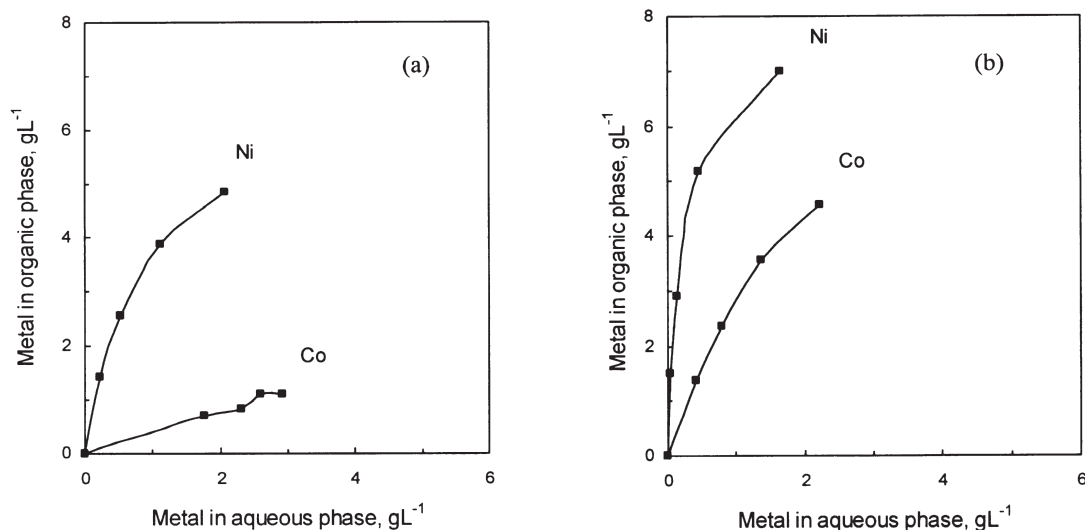# Separation of nickel and cobalt from calcium, magnesium and manganese

Figure 4—Distribution isotherms for the extraction of nickel and cobalt from 0.333 M Na$_2$SO$_4$ by 0.50 M Neodecanoic acid plus 0.25 M Mintek synergist in C$_{12}$-C$_{13}$ diluent at an equilibrium pH of 5.5 (a), and pH 6.0 (b)

Table II

Extraction of individual metals and compositions of aqueous feed, loaded organic phase and raffinate in batch countercurrent extractions with 0.50 M Neodecanoic acid and 0.25 M Mintek synergist in C$_{12}$-C$_{13}$ aliphatic diluent.^{a}

Metal	Extraction (%)		Composition (%)^b					
			Feed	Loaded organic		Raffinate	
Ni	99.9	(99.2)	38.2	89.0	(92.3)	<0.07	(0.51)
Co	84.7	(76.7)	3.9	6.6	(3.9)	1.1	(1.60)
Cu	100	(98.4)	0.7	1.5	(1.3)	<0.03	(<0.03)
Zn	100	(100)	0.8	2.0	(1.6)	<0.03	(<0.03)
Mn	2.7	(2.6)	14.8	0.8	(0.7)	25.4	(25.0)
Ca	1.5	(1.1)	3.7	0.12	(0.07)	6.5	(5.9)
Mg	<0.04	(<0.04)	37.9	0.03	(0.03)	66.9	(67.0)

^{a} Values in parentheses are for a similar experiment using 0.50 M Neodecanoic acid with 0.25 M CLX50
^{b} Amount of each metal expressed as a percentage of total metal content on mass basis

cent) while the extraction of the unwanted gangue metals (Mn 2.7, Ca 1.5 and Mg <0.04 per cent) is low. The overall result is that the aqueous feed containing a mixture of the valuable metals (44 per cent) and the gangue metals (56 per cent) is split into an organic phase in which only 0.9 per cent of the loaded metal consists of unwanted impurities, and a raffinate in which only 1.2 per cent of the total metal content represents losses of valuable metals. In actual practice this metal would not be lost, owing to the recycle of the raffinate as a wash liquor elsewhere in the process[12].

In a similar batch countercurrent extraction using 0.25 M CLX50 as the synergist at pH 6.1 in each stage, the loaded organic phase composition was almost identical to that in the previous experiment (Table II), while the raffinate contained a slightly larger amount (2.1 per cent) of valuable metals. Phase separations were slow and some interfacial precipitate was noted in this experiment, having the appearance of manganese dioxide.

Stripping of nickel and uptake of acid into organic phase

It was shown previously[11] that the metals following nickel in the process (Zn, Cu, Co and small amounts of Mn) could be removed from the (loaded) nickel strip liquor by contacting it in 1 or 2 stages with a 7.5 vol. per cent solution of Cyanex 272 in C$_{12}$-C$_{13}$ diluent. In this way, the metals other than nickel enter the electrowinning circuit only in trace amounts.

Stripping tests were carried out using 0.50 M Neodecanoic acid plus 0.25 M Mintek synergist in C$_{12}$-C$_{13}$ diluent containing 6.4 gL^{-1} of nickel as the loaded organic phase, and a simulated stripping solution (spent electrolyte) containing 39 gL^{-1} sulphuric acid with 33 gL^{-1} of nickel. Essentially complete stripping of the loaded organic phase was obtained in one stage at an organic-to-aqueous phase ratio of 4:1, yielding an 'advance electrolyte' containing 57 gL^{-1} of nickel at a pH of 3.5. The stripped organic phase contained only 0.05 gL^{-1} of nickel, representing less than 1 per cent of the initial value, while the uptake of acid into the organic phase was almost negligible (0.0014 M as H$^+$).

In the event that two stripping stages might be required in a continuous countercurrent process in order to compensate for non-equilibrium conditions, the second stage organic phase would come into contact with fresh stripping solution containing, in this instance, 33 gL^{-1} of nickel and 39

gL^{-1} sulphuric acid (pH 0.37). Simulation of this situation at an organic-to-aqueous phase ratio of 3.5:1 resulted in an acid uptake of only 0.0021 M into the organic phase. This behaviour differs markedly from that of systems containing an alkylpyridine synergist, which display a marked tendency to extract acid from aqueous phases at pH values lower than about 2.5[11]. The advantageous behaviour of systems that contain the Mintek synergist rather than alkylpyridines is clearly demonstrated in Figure 5, which shows the uptake of acid as a function of the equilibrium pH of the aqueous phase (0.50 M (Na,H)$_2$SO$_4$) using organic phases containing 0.50 M Neodecanoic acid together with 0.25 M Mintek synergist or 0.25 M 4-(5-nonyl)pyridine.

Stability and solubilty of the Mintek synergist

Tests have been done to determine the stability and the solubility of the Mintek synergist. It was found to be stable over a period of 100 days, representing stripping conditions. The solubility was found to be between 1 and 3 mgL^{-1} under specific conditions.

Conclusions

The Mintek synergist (0.25 M), in combination with a branched carboxylic acid, specifically Neodecanoic acid (0.50 M in Sasol C$_{12}$-C$_{13}$ aliphatic diluent) gives a good separation between the extraction vs. pH curves for nickel and calcium

(2.40 pH units) and a reasonable separation between cobalt and manganese (0.9 pH units). In a batch countercurrent extraction experiment with three stages at unit phase ratio and an equilibrium pH of 5.9, high recoveries of nickel (99.9 per cent) and cobalt (84.7 per cent) were obtained, with only small co-extractions of calcium (1.5 per cent), manganese (2.7 per cent) and magnesium (0.04 per cent). To increase the recovery of cobalt, the pH in extraction would have to be increased slightly.

Nickel can be stripped from the loaded organic phase by a synthetic 'spent electrolyte' containing 39 gL^{-1} sulphuric acid plus 33 gL^{-1} nickel in a single stage at an organic-to-aqueous phase ratio of 4 to give an 'advance electrolyte' containing 57 gL^{-1} of nickel at pH 3.5. The Mintek synergist has the added advantage of negligible extraction of acid from aqueous solutions at a pH as low as 0.37.

Finally, CLX50 can be employed to give similar results, but needs to be used at a much higher volumetric concentration (23.5 per cent compared with 9.3 per cent for the Mintek synergist) and seems to be more prone to crud formation and slow phase separations. Moreover, CLX50 is almost certainly no longer commercially available.

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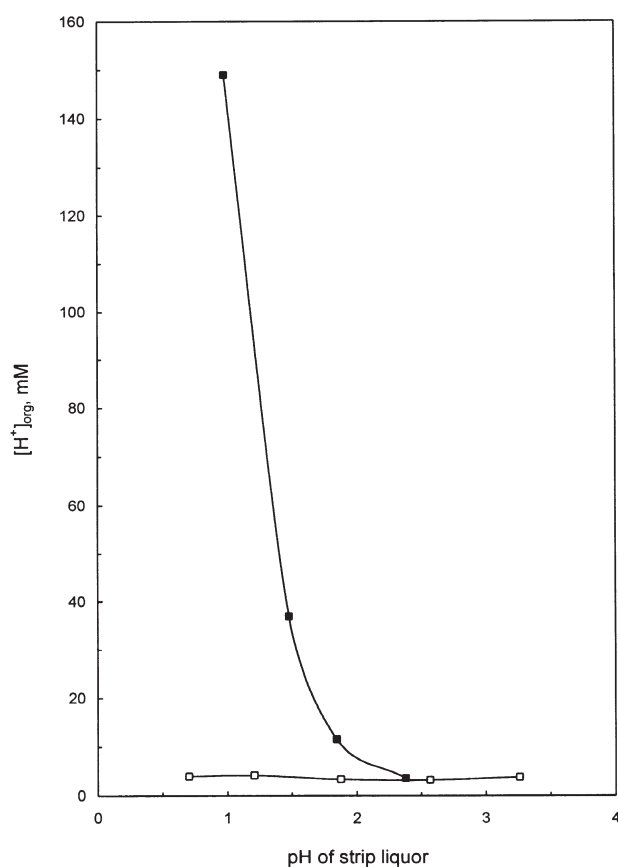
Figure 5—Extraction of acid (as H+) from 0.50 M (Na,H)$_2$SO$_4$ by 0.50 M Neodecanoic acid plus 0.25 M 4-(5-nonyl)pyridine (black symbols) and by 0.50 M Neodecanoic acid plus 0.25 M Mintek synergist in C$_{12}$-C$_{13}$ diluent (white symbols)