Hydrometallurgy 169 (2017) 135–141

Contents lists available at ScienceDirect

Hydrometallurgy

journal homepage: www.elsevier.com/locate/hydromet

Solvent extraction process for the recovery of nickel and cobalt from Caldag laterite leach solution: The first bench scale study

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ARTICLE INFO

Article history:
Received 3 August 2016
Received in revised form 31 October 2016
Accepted 15 January 2017
Available online 18 January 2017

Keywords:
Nickel
Cobalt
Caldag
Versatic 10
Cyanex 272
TBP
ShellSol 2046
Solvent extraction

ABSTRACT

A solvent extraction application consisting of two-sequential solvent extraction circuits to separate the nickel and cobalt from a synthetic sulphate leach solution which simulates a typical Caldag lateritic leach solution was conceived and experimentally explored. The first circuit allowed the simultaneous extraction of most of the nickel (98%), cobalt (98%) and manganese (94%) with 20% neodecanoic acid (Versatic 10) and 5% tri-n-butyl phosphate (TBP) in ShellSol 2046 at pH 7.2 together with substantial amounts of calcium (65%) and magnesium (12%). Three stages of scrubbing at pH 5.6 using diluted sulphuric acid solution allowed the removal of most of the magnesium (90%) and substantial amount of calcium (16%). Complete stripping of nickel, cobalt, manganese, magnesium and calcium was achieved at pH 0.75. This loaded strip solution was the feed for the second circuit. The use 15% bis(2,4,4-trimethylpentyl) phosphinic acid (Cyanex 272) and 5% TBP in ShellSol 2046 allowed the extraction of most of the cobalt (94%) and manganese (98%) at pH 5.0 but with substantial co-extraction of magnesium (41%) and calcium (40%) and a minor amount of nickel (3%). A two-stage scrubbing of this loaded organic with cobalt (20 g L^{-1}) solution was performed. Magnesium, calcium and nickel were completely displaced with cobalt. Most of the manganese (93%) was removed from the organic. Complete stripping of the cobalt and manganese in the scrubbed organic phase was achieved at pH 1.0. Therefore, nickel (94%) and cobalt (91%) were totally separated from the feed solution. In addition, slope analyses were carried out to determine the nature of the extracted complexes of the nickel and cobalt with each extractant. Based on the experimental results, a flowsheet for the separation process is presented.

1. Introduction

In hydrometallurgy of laterite nickel ores, solvent extraction (SX) has been an important separation technique to enrich the nickel and its primary by-product, cobalt. This is because SX is currently the only proven commercial technique to separate the two chemically-similar metals from the pregnant leach solution (PLS). Therefore, SX operation is preferred if separate high purity products of the nickel and cobalt are targeted.

Currently, commercial methods for hydrometallurgy of laterite nickel ores, depending on the downstream processing, is categorised as mixed hydroxide precipitation (MHP), mixed sulphide precipitation (MSP) and direct solvent extraction (DSX). The MHP and MSP processes involve intermediate precipitation of the nickel and cobalt and thus require transport and further processing steps involving re-leaching of the precipitate. The nickel and cobalt in the leach solution are then separated via SX to eventually obtain the maximum value of each metal. In the DSX process, on the other hand, the nickel and cobalt are separated directly from the PLS. This process, therefore, has the potential to lower the capital and processing costs for the production of each metal as a separate high purity product since it circumvents four separate processing units: precipitation, solid-liquid separation, re-leaching and another solid-liquid separation.

The first commercial DSX process was used in the now decommissioned Bulong Nickel Operation (Bulong) in Western Australia. The Bulong process consists of two SX circuits wherein cobalt, zinc, copper and manganese were separated from nickel, magnesium and calcium using bis(2,4,4-trimethylpentyl) phosphinic acid, Cyanex 272, in the first circuit and nickel was separated from magnesium and calcium using neodecanoic acid, Versatic 10, in the second circuit. Bulong encountered a number of technical issues in their early operation which have been reviewed in details by O'Callaghan (2003) and Donegan (2006). Notably, gypsum formation in the SX circuits was regarded as the most difficult challenge. Most of these issues, however, were able to be solved and by the time of its closure, the DSX process was able to meet most of its design parameter (Donegan, 2006).

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http://dx.doi.org/10.1016/j.hydromet.2017.01.001
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Table 1
Typical composition of synthetic feed solution.

Element	Ni	Co	Mn	Ca	Mg
Concentration (mg L^{-1})	4100	240	1600	530	8750

The second commercial DSX process is used in the Goro Nickel Project (Goro) in New Caledonia which is the only operating plant with DSX to date. The Goro process relies on the use of bis(2,4,4-trimethylpentyl) dithiophosphinic acid, Cyanex 301, to separate both the nickel and cobalt from manganese, magnesium and calcium. The nickel and cobalt are then separated by the use of an amine extractant. Cyanex 301 is currently the only commercially-available extractant that allows complete separation of nickel and cobalt from the three impurities but the extractant is prone to metal-catalysed degradation by air and extracts the nickel and cobalt too strongly (Mihaylov et al., 2000; Bacon and Mihaylov, 2002; Tsakiridis and Agatzini-Leonardou, 2004). Hence, the Goro process requires rigorous elimination of both iron(III) and copper prior to the SX step, operation under inert atmosphere, periodic extractant regeneration and very high acid concentration (6 mol L^{-1} HCl) in the stripping stage.

The use of synergistic solvent extraction systems (SSX) has been gaining considerable interest. Many studies have been carried out by various investigators using a mixture of two or more commercially-available extractants and reported synergistic effect toward the nickel and cobalt (Preston, 1983a; Preston, 1983b; Devi et al., 1994; Du Preez and Preston, 2004; Cheng, 2006; Cheng et al., 2010a; Cheng et al., 2010b; Cheng et al., 2011; Mayhew et al., 2011; Zhang et al., 2012; Guimaraes et al., 2014; Cheng et al., 2015). This results in better selectivity against major impurities such as manganese, magnesium and calcium. Notably, the systems of 5,8-diethyl-7-hydroxydodecan-6-oxime (LIX 63)/Versatic 10/tri-n-butyl phosphate (TBP) (Cheng, 2006) have shown remarkable improvement in the selectivity. Attempts to develop new extractants for the separation has also been carried out such as *N*-[*N*,*N*-di(2-ethylhexyl)aminocarbonylmethyl]glycine (D2EHAG) and *N*-[*N*,*N*-di(2-ethylhexyl)aminocarbonylmethyl]sarcosine (D2EHAS) (Baba et al., 2014) but none is likely to be commercialised in the near future (Cheng et al., 2016).

In the present study, the use of two SX steps using Versatic 10 and Cyanex 272 in the first and second step, respectively, to carry out SX of the nickel and cobalt from a partially neutralised atmospheric leach solution of Caldag laterite nickel ores, i.e. after iron (<1 mg L^{-1}), aluminium (<16 mg L^{-1}) and chromium (<0.5 mg L^{-1}) removal, was investigated. The use of this configuration instead of the reverse as was practiced in Bulong has been suggested by Donegan (2006) as it has the potential to reduce reagent costs by reducing ammonia and acid consumptions and reducing Cyanex 272 inventory, which is much more expensive than Versatic 10. Although this configuration has been investigated by Tsakiridis and Agatzini-Leonardou (2004) to separate nickel and cobalt from magnesium and calcium, the behaviour of manganese which is the most difficult impurity to be removed in this system, has not been reported. In the present study, therefore, the possibility for nickel and cobalt separation from manganese, magnesium and calcium by the use of Versatic 10 and Cyanex 272 was explored through series of extraction, scrubbing and stripping tests. The aim is to present a flowsheet to generate two separate streams of nickel and cobalt electrolytes that are sufficiently pure for electrowinning of each metal.

2. Experimental

2.1. Aqueous and organic solutions

Synthetic sulphate solutions that simulated those produced in Caldag, following the partial neutralisation step for the removal of iron, aluminium and chromium from the PLS by limestone, were prepared by dissolving analytical grade salts of $NiSO_4 \cdot 6H_2O$ (99% purity, Chem-Supply), $CoSO_4 \cdot 7H_2O$ (99% purity, Chem-Supply), $MnSO_4 \cdot H_2O$ (98.5% purity, Chem-Supply), $MgSO_4 \cdot 7H_2O$ (98% purity, Chem-Supply) and $CaCl_2 \cdot 2H_2O$ (98% purity, Rowe Scientific) in deionised water. Analytical grade sodium hydroxide pellets and sulphuric acid diluted in deionised water were used for pH adjustment. All reagents were used as supplied. The typical composition of the synthetic feed solution is shown in Table 1.

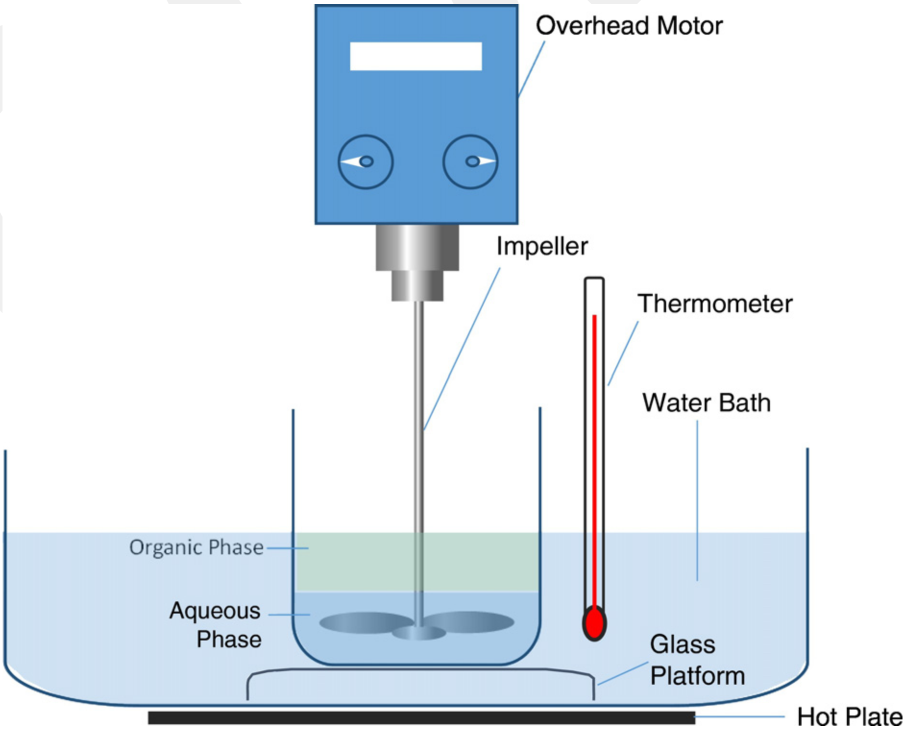

Fig. 1. Experimental set-up.

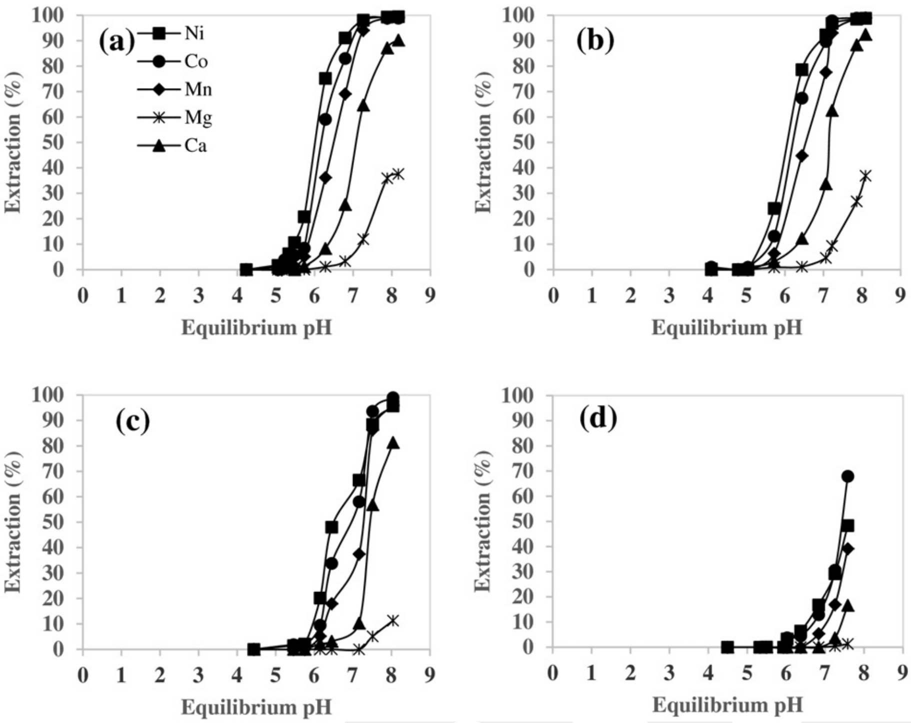

Fig. 2. Extraction of metals with Versatic 10 a) 20% Versatic 10 + 5% TBP; b) 15% Versatic 10 + 5% TBP; c) 10% Versatic 10 + 5% TBP; d) 5% Versatic 10 + 5% TBP; $T = 40$ °C; A/O = 1.

The extractants Versatic 10 (Industrial Grade) was kindly provided by Momentive the Netherlands and Cyanex 272 (Industrial Grade) was purchased from Cytec Australia. TBP (≥99% purity) was supplied by Merck Australia and was used as a phase modifier to prevent emulsions or third phase formation during the extraction process. All organic solutions dissolved in ShellSol 2046 (Industrial Grade, Shell Chemicals Australia) and were pre-treated by manually shaking them with an equal volume of deionised water in a separation funnel to remove any water-soluble constituent. The organic phase was filtered through Whatman 1PS phase separation paper to prevent any water entrainment.

2.2. Experimental procedure

All extraction, scrubbing and stripping experiments were carried out in a 200-mL glass reactor immersed in a temperature-controlled water bath. The temperature of the solution was maintained at 40 ± 1 °C. For each experiment, the aqueous solution was contacted with equal volume of the organic solution (A/O = 1) using a three-blade stainless-steel impeller (35 mm diameter) driven by a digital overhead motor (Heidolph RZR 2020) at 400 rpm for 10 min. The experimental set-up is shown in Fig. 1.

The aqueous solution pH was controlled by small additions of NaOH (5 mol L^{-1}). After mixing, the mixture was allowed to stand for 5 min to disengage sufficiently and the aqueous solution (10 mL) was taken by a syringe for pH measurement using a pH meter equipped with an Ionode IJ44-C pH probe. The sample was filtered using a 0.45-μm syringe membrane filter and then, the filtrate was diluted with acidified deionised water to prevent precipitation. The concentration of metal ions in the aqueous solution was analysed by Bureau Veritas Perth/Australia using inductively coupled plasma atomic emission spectroscopy (ICP-AES, Thermo Fisher iCAP 6000). The concentration of metal ions in the organic phase was calculated by mass balance. The extraction percentage (E) of metal ions was calculated according to the following equations:

$$E = \frac{[M]_{org}}{[M]_{aq}} \times 100 \tag{1}$$

where $[M]_{org}$ and $[M]_{aq}$ denote the concentration of metal ions in the organic and aqueous phase (mg L^{-1}), respectively.

3. Results and discussion

3.1. Metal extraction with Versatic 10

3.1.1. Extraction pH isotherms

A series of solvent extraction tests were carried out with Versatic 10 concentrations in the range of 5–20% (v/v) and 5% TBP (v/v) in ShellSol 2046. Fig. 2 shows the effect of equilibrium pH and the concentration of Versatic 10 on the extraction of various metals from aqueous solution. The increase in pH and concentration of Versatic 10 resulted in higher metal extraction. Based on the extraction of nickel and cobalt, and the consideration of third phase formation and local precipitation, the organic system containing 20% Versatic 10 and 5% TBP was selected as the best composition at an A/O of unity. By the use of this system, most of the nickel (98%), cobalt (98%) and manganese (94%) were extracted at pH 7.26 in a single contact whereas substantial amounts of calcium (65%) and magnesium (12%) were co-extracted. The following metal extraction order was obtained: $Ni^{2+} > Co^{2+} > Mn^{2+} > Ca^{2+} > Mg^{2+}$, which agrees with the findings of Preston (1985) and Cheng (2006).

3.1.2. Scrubbing of loaded organic phase

To remove magnesium and calcium from the loaded organic solutions, scrubbing tests were carried out using the loaded organic solutions generated from the extraction tests at previously determined

Table 2
Effect of pH on the three stages scrubbing of metals using diluted sulphuric acid solution at 40 °C and A/O:1.

pH	Scrubbing									
	Metal in aqueous phase, %					Metal in organic phase, %				
	Ni	Co	Mn	Ca	Mg	Ni	Co	Mn	Ca	Mg
5.6	0.3	1.9	2.1	15.7	90.1	99.7	98.1	97.9	84.3	9.9
5.8	0.4	1.0	2.2	13.9	82.1	99.6	99.0	97.8	86.1	17.9

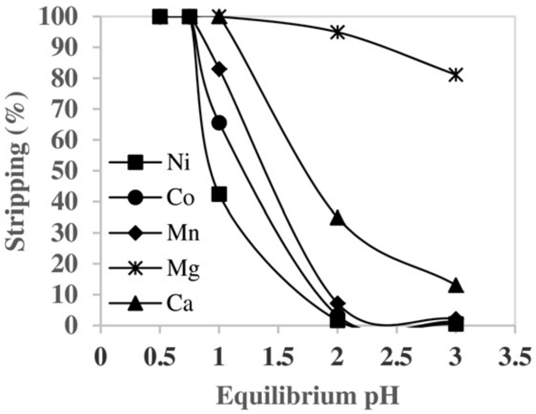

Fig. 3. Stripping pH isotherms using diluted sulphuric acid solutions.

optimum conditions. The loaded organic solutions were subjected to scrubbing with diluted sulphuric acid solution at pH 5.6 and 5.8, and A/O of unity. pH of the deionised water was adjusted with sulphuric acid. >90% of the magnesium was scrubbed out after three stage scrubbing tests at pH 5.6 whereas only 16% of the calcium was removed from the loaded organic solution. It was found that nickel, cobalt and manganese formed relatively stable complexes with Versatic 10 as <3% of the nickel, cobalt and manganese were scrubbed out. The results of the three stages scrubbing are given in Table 2.

3.1.3. Stripping of the loaded organic phase

Stripping of the loaded organic solutions obtained from the scrubbing tests at the determined optimum conditions (pH = 5.6, T = 40 °C, t = 10 min, A/O = 1) was performed by using diluted sulphuric acid solutions at pH 0.5, 0.75, 1.0, 2.0 and 3.0, and A/O of unity. Fig. 3 shows the stripping percentage of the metals from the loaded organic solution after three stage of scrubbing experiments. From Fig. 3, it can be seen that at a pH of 0.75, nickel, cobalt, manganese, magnesium and calcium can be stripped completely from the loaded scrub solution. The stripping percentage of nickel, cobalt, manganese and calcium significantly decreased with increasing equilibrium pH whereas magnesium decreased only slightly.

3.2. Metal extraction with Cyanex 272

3.2.1. Extraction pH isotherms

The loaded strip solution was subjected to solvent extraction tests using Cyanex 272 concentration in the range of 5–20% (v/v) and 5% TBP (v/v) in ShellSol 2046. Fig. 4 shows the effect of equilibrium pH and the concentration of Cyanex 272 on the extraction of various metal ions from the loaded strip solution. Based on the data shown in Fig. 4, the organic system containing 15% Cyanex 272 and 5% TBP was chosen for selective extraction of cobalt and manganese, leaving most of the nickel, magnesium and calcium in the raffinate. The pH isotherms of manganese and cobalt shifted toward lower pH when the concentration of Cyanex 272 increased from 5% to 15% Cyanex 272 and then remained relatively constant. The extraction of nickel was not significantly affected by the increase in the concentration of Cyanex 272. Cobalt (94%) and manganese (98%) were extracted by 15% Cyanex 272 and 5% TBP at pH 4.83 in a single contact, while approximately 2% of the nickel was co-extracted from the aqueous phase.

3.2.2. Scrubbing of the loaded organic phase with cobalt salt solution

Although nickel extraction was very low with 15% Cyanex 272 and 5% TBP system under the determined optimum conditions, a substantial amount of manganese (98%), calcium (21%) and magnesium (30%) were co-extracted with cobalt. Consequently, scrubbing is required to ensure the high purity of the final cobalt product. The loaded organic phase for the scrubbing test was prepared by loading the organic solution (15% Cyanex 272 and 5% TBP in ShellSol 2046) at an A/O ratio of unity and pH 5.0 by which most of the nickel (96%) and manganese (98%) were extracted with substantial amounts of magnesium (41%) and calcium (40%) were co-extracted. The amount of nickel co-extraction was low (3%).

The scrubbing tests were carried out at an A/O ratio of unity as well. The co-extracted nickel, manganese, calcium and magnesium were

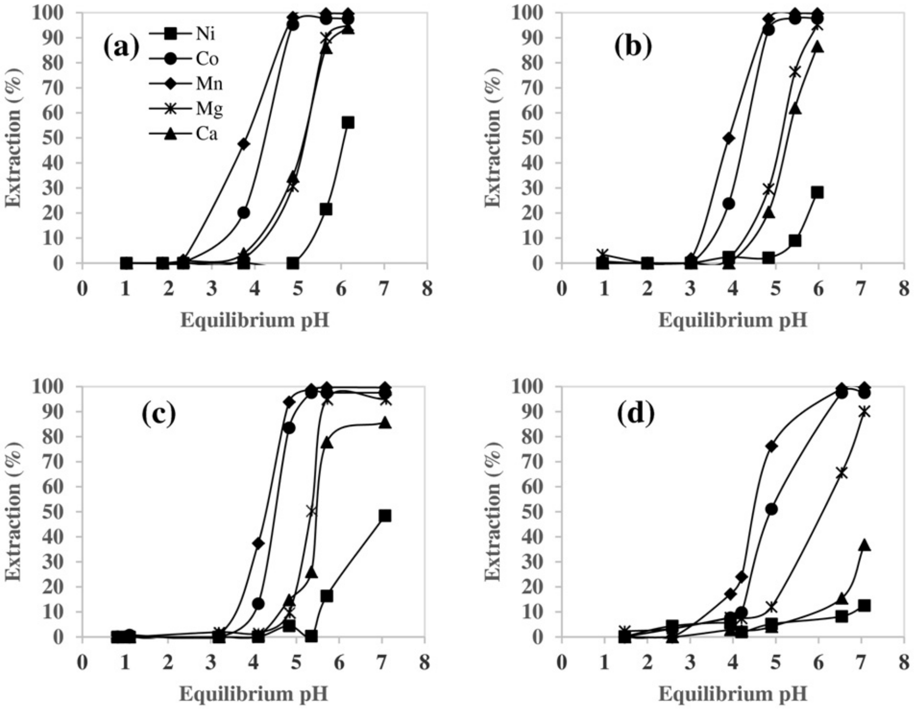

Fig. 4. Extraction of metals with Cyanex 272 a) 20% Cyanex 272 10 + 5% TBP; b) 15% Cyanex 272 + 5% TBP; c) 10% Cyanex 272 + 5% TBP; d) 5% Cyanex 272 + 5% TBP; T = 40 °C; A/O = 1.

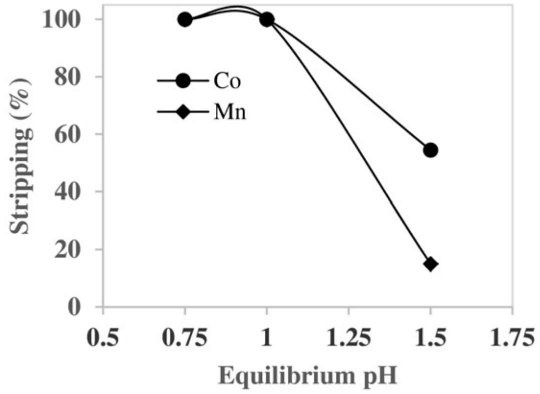

Fig. 5. Stripping pH isotherms using diluted sulphuric acid solutions.

contacted with a cobalt salt solution containing 20 g L^{-1} of cobalt (Ritcey, 2006). The cobalt in the aqueous solution displaced nickel, manganese, calcium and magnesium from the loaded organic. The displacement mechanism is illustrated by Eq. (2). Nickel, magnesium and calcium were completely scrubbed out from the loaded organic solution whereas scrubbing efficiencies were 93% for manganese.

$$(C_{16}H_{35}O_2P)(\bar{N}i, Mn, Ca, Mg)\ + +Co\bar{S}O_4 \leftrightharpoons (C_{16}H_{3\bar{5}}O_2P)Co + (Ni, Mn, Ca, Mg)SO_4 \tag{2}$$

3.2.3. Stripping of the loaded organic phase

The loaded scrub organic was subjected to stripping using diluted sulphuric acid solutions at pH 0.75, 1 and 1.5, and A/O of unity. Fig. 5 shows the stripping percentages of cobalt and manganese from loaded organic solution. As can be seen from the Fig. 5, the stripping of cobalt and manganese were completely achieved at pH of 1.0. Stripping of cobalt and manganese significantly decreased with increasing equilibrium pH. According to the stripping results, the loaded strip liquor contains 2005 mg L^{-1} Co and 87 mg L^{-1} Mn.

3.2.4. Nature of the extracted species

The extraction of reaction for a divalent cation, such as Ni^{2+} or Co^{2+}, is written as:

$$\overline{2HA_{(org)}} + M^{2+}_{(aq)} \leftrightarrow \overline{MA_{2(org)}} + 2H^{+}_{(aq)} \tag{3}$$

where M^{2+} is the divalent metal ion and HA is the cationic extractant. It is known that cationic extractants are dimerised or self-associated in organic diluents. The general reaction for nickel and cobalt extraction can be written (Preston, 1982; Tait, 1993) as:

$$\overline{n(HA)_{2(org)}} + M^{2+}_{(aq)} \leftrightarrow \overline{(MA_{2n}H_{2n-2})_{(org)}} + 2H^{+}_{(aq)} \tag{4}$$

The equilibrium constant, K, of the reaction is written as:

$$K = \frac{[MA_{2n}H_{2n-2}]_{(org)}[H^+]^2_{(aq)}}{[HA_2]^n_{(org)}\left[M^{2+}\right]_{(aq)}} \tag{5}$$

or,

$$K = D\frac{[H^+]^2_{(aq)}}{[HA_2]^n_{(org)}} \tag{6}$$

where the distribution coefficient of the metal $D = \frac{[MA_{2n}H_{2n-2}]_{(org)}}{[M^{2+}]_{(aq).}}$

Taking logarithm of Eq. 6 and on rearranging, results:

$$\log D - 2pH = \log K + n \log[(HA)_2] \tag{7}$$

The plots of log$D$ – 2pH versus log$[(HA)_2]$ should be a straight line with a slope of n. In the case of Versatic 10, the values of n for nickel and cobalt were about 3.1 and 2.8, respectively, which was very close to 3.0 (Fig. 6). These indicate the formation of $CoA_2\cdot 4HA$ and $NiA_2\cdot 4HA$ which are in agreement with the findings of Shibata and Nishimura (1979) and Tsakiridis and Agatzini-Leonardou (2004). The values of the slopes for log$D$ – 2pH versus log [HA] for Cyanex 272 were 2.3 (Fig. 7). This indicate the formation of $Co(HA_2)_2$ which is in agreement with the findings of Tait (1993).

3.2.5. Proposed solvent extraction flow sheet

Based on the experimental results obtained in the experimental campaign, a flow sheet was proposed for the extraction and separation of nickel and cobalt from Caldag laterite leach solution (Fig. 8) (Kursunoglu, 2016). The main features of this flowsheet are that two-sequential solvent extraction circuits were used to separate both nickel and cobalt from bulk of magnesium and calcium in the first circuit and to separate the nickel and cobalt in the second circuit. In the second extraction circuit, 3890 mg L^{-1} of nickel was remained in the nickel rich raffinate while 220 mg L^{-1} of cobalt was transferred into the organic phase. Therefore, 94% of nickel and 91% of cobalt were separated from the feed solution. The co-extracted manganese in the second circuit, which contaminate the cobalt stream, was scrubbed out of the loaded organic with the use of cobalt-containing scrub solution.

It has to be noted that the amounts of calcium co-extraction in each extraction circuit was relatively high but the amounts of calcium ions in the final streams of nickel and cobalt were still sufficiently low for electrowinning of each metal. For example, the designed calcium content in the Bulong's nickel electrolyte solution was 150 mg L^{-1} (Donegan,

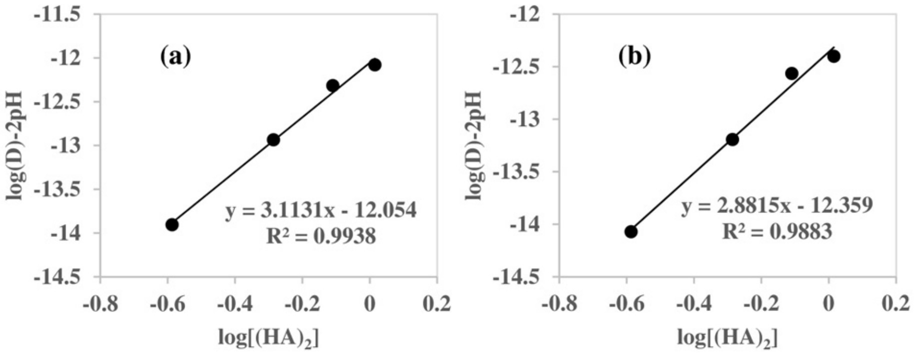

Fig. 6. Plot of $logD-2pH$ versus $\log[(HA)_2]$ for the extraction of (a) nickel and (b) cobalt by Versatic 10 at pH of 6.4.

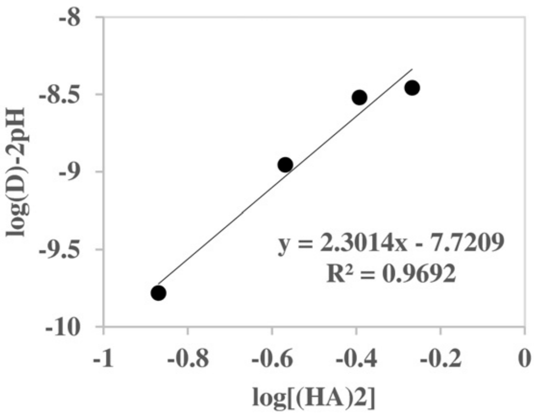

Fig. 7. Plot of $logD - 2pH$ versus $\log[(HA)_2]$ for the extraction of cobalt by Cyanex 272 at pH of 4.9.

2006). Also, the risk of gypsum precipitation can be minimised by the use of an antiscalent reagent as evident in the Bulong (O'Callaghan, 2003; Donegan, 2006).

4. Conclusions

A two-sequential solvent extraction circuits to extract and separate the nickel and cobalt from a synthetic sulphate solution which simulates a typical Caldag lateritic leach solution was carried out. The nickel (98%) and manganese (94%) were simultaneously extracted using 20% Versatic 10 and 5% TBP dissolved in ShellSol 2046 at pH 7.2 in the first extraction circuit. The loaded organic was subjected to three-stage scrubbing with diluted sulphuric acid solution to remove co-extracted impurities. Most of magnesium (90%) and small amounts of calcium (16%) were scrubbed out at pH 5.6. The remaining metal ions in the scrubbed organic was completely stripped at pH 0.75. The cobalt (96%) and manganese (98%) were extracted using 15% Cyanex 272 and 5% TBP in ShellSol 2046 at pH 5.0 in the second circuit. The loaded organic was contacted with a cobalt salt solution containing 20 g L^{-1} of cobalt. A two-stage scrubbing of this loaded organic with the cobalt-containing solution displaced the co-extracted manganese, magnesium, calcium and nickel with cobalt. The manganese (93%) was scrubbed out from the loaded organic along with all other impurities. The cobalt in the scrubbed organic solution was completely stripped at pH 1.0. Overall, 94% of the nickel and 91% of the cobalt were totally separated from the feed solution. Based on these experimental results, the flow sheet for the separation process is proposed.

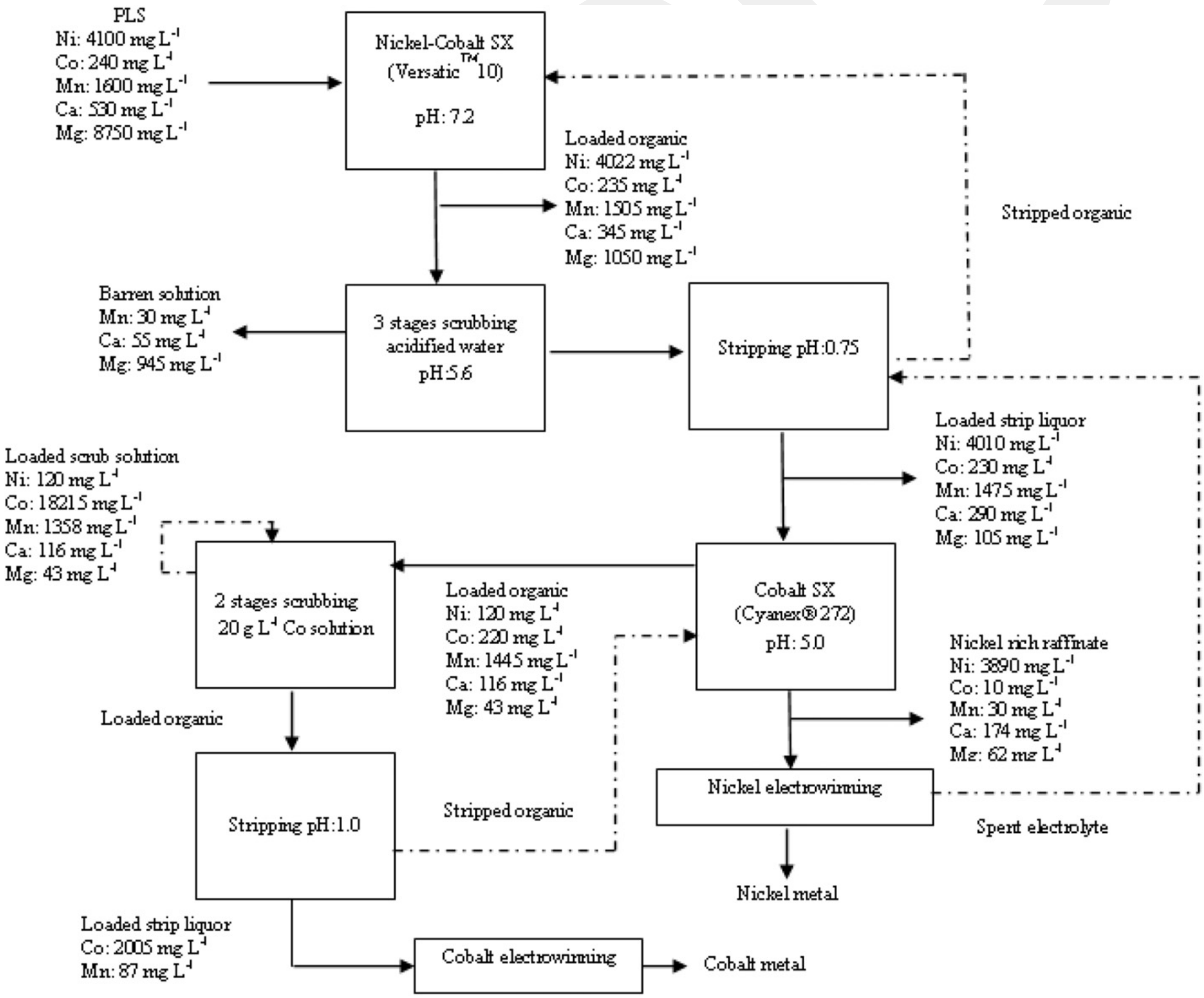

Fig. 8. Proposed SX flow sheet for the extraction of nickel and cobalt from Caldag laterite leach solutions.

Acknowledgements

Sait acknowledges the Scientific and Technological Research Council of Turkey (TUBITAK) for the financial support provided through the 2214-A International Doctoral Research Fellowship Programme and A/Prof Don Ibana from Curtin University's Western Australian School of Mines for allowing the use of their facilities and collaboration. We thank Dr. Dincer Erer from Perth, Australia for proofreading the manuscript. The authors are also grateful to Caldag Nickel for providing the data of the partially neutralised leach solution.

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