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

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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.

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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, releaching and another solid-liquid separation. The first commercial DSX process was used in the now

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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Table 1

Typical composition of synthetic feed solution.

Element	Ni	Со	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(2,4,4trimethylpentyl) 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 commerciallyavailable 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 ($<1 \text{ mg L}^{-1}$), 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 NiSO₄·6H₂O (99% purity, Chem-Supply), CoSO₄·7H₂O (99% purity, Chem-Supply), CoSO₄·7H₂O (99% purity, Chem-Supply), MgSO₄·7H₂O (98% purity, Chem-Supply) and CaCl₂·2H₂O (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.

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