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# Iron, aluminium and chromium co-removal from atmospheric nickel laterite leach solutions

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#### ABSTRACT

Atmospheric leaching (AL) of low-grade nickel laterite ores often produces leach liquor containing significant amounts of trivalent iron, aluminium and chromium ions. These impurities are normally removed by increasing the leach liquor pH to precipitate these metals before the recovery of nickel, cobalt and other metal values. This paper documents an investigation of the removal of iron, aluminium and chromium from both synthetic and real nickel laterite AL leach solutions using single- and multi-stage precipitation methods. Single-stage precipitation  $experiments \ performed \ using \ synthetic \ leach \ solutions \ containing \ Fe(III) \ + \ Ni(II) \ + \ Al(III), \ Fe(III) \ + \ Ni(II) \ + \ Ni$ (III), and Fe(III) + Ni(II) + Al(III) + Cr(III) showed that greater losses of nickel to solids occurred in the presence of aluminium and chromium. Increasing the pH value of the solution and the precipitation temperature favoured the removal of iron, aluminium and chromium, but at a cost of greater nickel losses. However, by carefully controlling pH and temperature using a multi-stage precipitation process, the iron, aluminium and chromium can be rejected effectively by precipitation with minimal nickel loss and desirable sludge properties. The optimum conditions for a multi-stage precipitation process were found to be pH 3.0 and 55 °C in the first stage and pH 3.0 and 85 °C in the second stage. Using this approach, as much as 95% iron and chromium together with more than 80% aluminium can be removed; the level of nickel loss to the solid can be reduced to below 1%. The sludge showed a fast settling rate of 5.05 m/h following the addition of a cationic flocculant. Similar satisfactory results were also obtained when performing this multi-stage precipitation procedure with real leach solutions.

#### 1. Introduction

The use of atmospheric acid leaching (AL) as an alternative to high pressure acid leaching (HPAL) for the processing of low-grade nickel laterite ores has been examined extensively in recent years (McDonald and Whittington, 2008a,b; Büyükakinci and Topkaya, 2009; Liu et al., 2009, 2010; Luo et al., 2009, 2010, 2015; Das and de Lange, 2011; Panda et al., 2014; Senanayake et al., 2015; Basturkcu and Acarkan, 2016; MacCarthy et al., 2014, 2015, 2016; Basturkcu et al., 2017). In contrast to HPAL, AL often produces leach liquor containing significant amounts of trivalent iron, aluminium and chromium ions. For example, typical concentration values for these impurities in the liquor from the heap leaching of Greek nickeliferous laterite with sulphuric acid have been reported as 23 g/L Fe<sup>3+</sup>, 6.0 g/L Al<sup>3+</sup> and 1.0 g/L Cr<sup>3+</sup> (Agatzini-Leonardou et al., 2009). Inefficient removal of these impurities represents a significant impediment to producing pure cobalt and nickel compounds and/or metals in the subsequent downstream hydrometallurgical processing to recover valuable metals. This is particularly

important when the value metals are recovered and separated using solvent extraction (e.g. Donegan, 2006) or when intermediate products such as mixed hydroxides are generated (e.g. White, 2009). In comparison the removal of iron(III) and chromium(VI) are most critical when sulphide precipitation is employed to recover the metal values (e.g. Collins et al., 2016), while ion exchange is currently employed in laterite processing for the removal of residual copper in the Goro HPAL circuit (Mihaylov et al., 2000).

Effective purification methods are required to remove Fe(II), Cr(III) and Al(III) ions selectively from leach liquors for treatment by solvent extraction or to produce mixed hydroxides. This is often achieved by precipitation but usually involves co-precipitation and/or incorporation of nickel and cobalt. In our earlier work, Wang et al. (2011), the effects of the factors governing nickel loss during iron removal from synthetic AL leach solutions containing just nickel and iron were examined using statistical methods. The characterisation of the iron-rich precipitates obtained from these solutions was detailed more recently (Wang et al., 2013).

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During leach liquor purification, the pH value is the main variable that governs the impurity rejection efficiency. The precipitation curves of metallic cations reported by España (2007) and Zinck (1993) indicated that ferric iron precipitation occurred at a lower pH range than aluminium and chromium. Hence, in order to achieve maximum removal of aluminium and chromium, higher pH values for precipitation are often used. In the study by Agatzini-Leonardou et al. (2009) on the separation and recovery of nickel from heap leach liquor of nickeliferous laterite ore it was shown that more than 92% of the iron in solution could be easily removed by raising the pH value of the solution to approximately 2.8 at 95 °C, whereas the removal of aluminium and chromium under the same conditions was lower, reported as 73% and 76%, respectively. By increasing the solution pH to 4.0, 98.7% iron. 97.7% aluminium and 95.8% chromium could be removed. A conventional single-stage precipitation procedure was used in the work of Agatzini-Leonardou et al. (2009), similar to the experiments reported by Wang et al. (2011). However, a multi-stage precipitation process for impurity removal has attracted more attention in industrial operations to obtain better impurity removal efficiency, easier handling of waste products, and to minimise losses of valuable metals (e.g. White, 2009).

Boliden Mineral AB (Bolin and Sundkvist, 2008; Sundkvist, 2002) patented a two-stage precipitation process for the efficient separation of iron and arsenic from acidic leach solution that produced a very clean iron-arsenic precipitate with minimum co-precipitation of base metals. The pH of the acid leach solution was raised and controlled in the range of 2.2–2.8 at 35 °C to remove more than 90% ferric iron in the first stage. The resulting slurry was discharged into a thickener. The overflow from the thickener was then diverted to the second stage precipitation, where pH was increased to a value in the range of 3.0–4.5 and air was injected simultaneously to remove ferrous iron as well. More than 99% zinc and copper recoveries to solution were reported by using this two-stage precipitation process while the overall removal of iron was also greater than 99%.

The study on iron, aluminium and chromium removal from laterite heap leach solutions by Guise and Castro (1996) showed that 41.3% iron, 21.8% aluminium and 29.5% chromium were rejected at pH 1.8 and 90 °C by the addition of 50 g/L MgO slurry in the first stage of the precipitation process. In the second stage of the precipitation process, operated at pH 2.5 and 90 °C, 99.9% iron, 53% aluminium and 93.9% chromium of that remaining after the first stage were removed, but at a cost of 26.5% nickel and 37.1% cobalt losses to the solids. Köse and Topkaya (2011) recently studied the Mixed Hydroxide Precipitation (MHP) process to remove impurities from the leach liquors of nontronite type lateritic ores, and showed that the concentrations of  $Fe^{3+}$ ,  $\mathrm{Al}^{3+}$  and  $\mathrm{Cr}^{3+}$  were decreased from 38,600, 3950, and 1060 ppm respectively to 290, 1260 and 202 ppm by a first removal stage conducted at pH 2.5 and 90  $^\circ C$  with minimal nickel loss, and these values further dropped to 1.38, 3.6 and 0.77 ppm by a second removal stage conducted at pH 4.25 and 70 °C. However, nickel loss at the second removal stage was still substantial at 17.2% of the initial nickel concentration of 4800 ppm. The solids from this step would therefore need to be treated (recycled) to recover the nickel (and cobalt).

The very high levels of nickel losses to the solids in the impurityremoval processes of Guise and Castro (1996) and Köse and Topkaya (2011) highlighted the need for a more detailed study on the precipitation process for impurity removal to achieve acceptable levels of nickel loss. The present study, which is an extension of our earlier work (Wang et al., 2011), aims to study the effects of iron, aluminium and chromium co-elimination upon nickel losses, develop a multi-stage precipitation process to remove impurities efficiently without causing significant losses of nickel, and produce precipitation sludge with satisfactory physical properties such as a fast settling rate. In order to achieve these goals, both single- and multi-stage precipitation experiments were carried out with synthetic leach solution and real leach liquor obtained by column leaching of low-grade Western Australia nickel laterite ores with sulphuric acid at ambient temperature. It is also proposed that the (batch) experimental approach employed for this study provides better control of metal supersaturation and pH than those typically employed for this type of test work. The precipitation experiments for the synthetic leach solution were conducted using a solution containing various combinations of metal ions: Fe(III) + Ni(II), Fe(III) + Ni(II) + Al(III), Fe(III) + Ni(II) + Cr(III) and Fe(III) + Ni(II) + Al(III), respectively. An experimental statistical design with analysis of variance approach was applied to determine if chromium(III) and/or aluminium(III), in addition to iron, have a significant impact upon nickel loss to the precipitates under the conditions examined.

#### 2. Experimental

#### 2.1. Single-stage precipitation

All precipitation experiments were conducted using a semi-batch reaction vessel. The experimental set-up and procedure was described by Wang et al. (2011). For each test a volume of 500 mL of synthetic leach solution containing 30 g/L Fe<sup>3+</sup> [as Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·xH<sub>2</sub>O], 5 g/L Ni<sup>2+</sup> [as NiSO<sub>4</sub>·6H<sub>2</sub>O], 0–10 g/L Al<sup>3+</sup> [as Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·18H<sub>2</sub>O], and 0–2.5 g/L  $Cr^{3+}$  [as  $Cr_2(SO_4)_3$ :xH<sub>2</sub>O] was pumped into a baffled reaction vessel containing a pre-determined amount of de-ionized water, typically around 500 mL, over a period of 2.5 h. A CaCO<sub>3</sub> (25% w/w) suspension in de-ionized water was simultaneously pumped into the reaction vessel to achieve the target pH. The temperature was controlled using an oil bath and the precipitation reactions conducted at 25, 55 and 85 °C. At the completion of the reaction, the resulting slurry was vacuum filtered through a 0.45 µm Gelman Supor® membrane. The metal concentrations in the filtrate were analysed by ICP-OES. The filter cake was carefully washed with large amounts of de-ionized water, and then dried overnight at 60 °C. As the minimum nickel content in the precipitates prepared in this study was 0.25% (see Section 3), the potential for precipitation from filtrate entrained in the filter cake through the use of de-ionized water was considered to be low. The chemical composition of the dried solid products was determined by ICP-OES after aqua regia digestion. The accuracy of the data obtained by this method is considered to be within  $\pm$  5% of the actual values.

#### 2.2. Multi-stage precipitation

Multi-stage precipitation experiments with a combination of pH and temperature control were conducted using both synthetic and real leach solutions. The multi-stage precipitation experiments consisted of two consecutive precipitation processes. Similar to the single-stage precipitation described in Section 2.1, the first stage precipitation process involved pumping 500 mL of leach solution into a reaction vessel within 2.5 h. A CaCO<sub>3</sub> (25% w/w) suspension in de-ionized water was pumped simultaneously to control the pH value. Temperature was controlled using an oil bath. Instead of stopping the reaction after 2.5 h, a second stage of precipitation was conducted by ageing the resultant slurry for one hour. After leach solution pumping was completed, the pH was adjusted to the desired value by pumping CaCO<sub>3</sub> (25% w/w) suspension in de-ionized water or adding concentrated H<sub>2</sub>SO<sub>4</sub>, and the temperature was increased as required by heating. Once the new equilibrium pH and/or temperature were reached, the reaction was maintained for 1 h. It should be noted that, while the second stage is described as a precipitation process, it may involve a combination of precipitation and/or dissolution of existing precipitate, depending on the final pH and temperature compared to the first stage.

#### 2.3. Flocculant preparation and settling rate test

Two commercial polyacrylamide polymers were used as flocculants to improve the settling rate of sludge produced from the precipitation: a non-ionic polymer, FA 920 VHM, and a cationic polymer, FO 4190 Download English Version:

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