Behaviour of Cu, Fe, Ni, and PGMs during leaching of Ni-Fe-Cu-S converter matte

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Synopsis

In the mineral processing industry, Ni-Cu-Fe-S converter matte is leached to remove base metals from a concentrate containing platinum group metals (PGMs). We investigated the atmospheric leaching process to develop a better understanding of the leaching behaviour of the base metals (Cu, Fe, and Ni) and the PGMs, in particular Ru, Ir, and Rh with respect to key process variables, acid concentration, and Cu concentration under oxidative and non-oxidative conditions.

With oxidative leaching, a higher initial acid concentration resulted in higher Cu, Ni, and Fe extractions, as well as faster leaching reaction rates. A higher initial acid concentration also resulted in faster precipitation of Cu, Ru, and Ir under oxidizing conditions.

Under non-oxidative conditions, Ni and Fe extraction was much slower, and the effect of initial acid concentration on Cu precipitation was less pronounced. The initial Cu concentration had a slightly smaller effect on the leaching behaviour of Fe and Ni, as well as precipitation of Ru, Ir and Rh. Moreover, a higher initial Cu concentration suppressed both Ni and Fe leaching and had a slight inhibiting effect on the precipitation behaviour of Cu, Ru, and Ir.

Keywords
platinum group metals, converter matte, leaching, precipitation.

Introduction

The largest platinum group metal (PGM) deposit in the world, located in the Bushveld Complex in South Africa, holds half of the world's platinum group elements (PGEs) and chromium. Smelting and processing of the PGM concentrates in this region is carried out by four major companies, namely Anglo American Platinum, Impala Platinum, Sibanye-Stillwater, and Northam Platinum (Jones, 2005).

Due to their unique physical and chemical properties such as good corrosion and oxidation resistance, high melting temperatures, good conductivity, and electronic and catalytic properties, the PGMs are used for their superior performance in certain industries and markets. These applications include (i) platinum or rhodium as autocatalysts to reduce greenhouse gas emissions produced by the combustion of fossil fuels, (ii) platinum and palladium jewellery, (iii) catalysts in the chemical sector, (iv) electrical equipment and electronic devices, (v) platinum used in the manufacturing of glass, and the (vi) production of drugs in the medical sector (Creamer, 2006). Moreover, platinum-catalysed hydrogen-powered fuel cells for electric vehicles (EVs) offer the most natural solution for emission-free vehicles, discharging only water and requiring negligible changes to current driving and refuelling habits (Theron-Ord, 2017).

The Sibanye-Stillwater (formerly Western Platinum) process involves milling, flotation, and smelting followed by Peirce-Smith converting to produce a Ni-Cu-Fe-S converter matte containing PGMs. The base metals and sulphur contained in the converter matte are removed through a multi-stage leaching process. The first stage leach, also known as the atmospheric leach, serves to remove Ni from the matte, while at the same time precipitating Cu and the PGMs from the leaching solution. Due to very little insight into the mechanisms of the atmospheric leaching process, studies conducted by Hofirek and Kerfoot (1992), Lamya and Lorenzen (2006), van Schalkwyk *et al.* (2011) and Snyders *et al.* (2018) were aimed at investigating the chemistry and mechanism of the process, as well as determining the effects of Fe endpoints, initial acid and Cu concentrations of the spent electrolyte solutions, and oxidative/non-oxidative conditions on the leaching behaviour of Ni-Cu-Fe-S converter mattes.

The purpose of atmospheric leaching is to firstly leach the Ni from the matte, while at the same time rejecting the Cu from the solution, through metathesis reactions. Leaching takes place in five continuously stirred tank reactors (CSTRs) in series, with oxygen fed to the first three tanks only. The acid supplied to the first tank is spent electrolyte solution recycled from the copper electrowinning tankhouse and which contains 20–30 g/L Cu and 80–90 g/L sulphuric acid. Sulphuric acid make-up is only added during upset conditions on the plant. A Ni crystallizer bleed stream is added to the first stage leach in order to recovery any Cu that may have slipped through to the Ni crystallizer unit. The operating temperature of the first stage circuit is 85°C (Crundwell *et al.*, 2011). The metathesis reactions serve to exchange Cu from the solution with Ni from the alloy and nickel sulphide phases in the matte. The PGMs present in the matte, along with the Cu, report to the leach residue (van Schalkwyk *et al.*, 2011.)

In this project we investigated the first stage atmospheric leaching process of the converter matte, using base metal refinery (BMR) spent electrolyte from Sibanye-Stillwater, in order to develop a better understanding of the effect of the initial acid and Cu concentrations on the leaching behaviour of the base metals, in particular Fe and Ni, as well as Rh, Ir, and Ru, under oxidative and non-oxidative conditions. A better understanding of the process will assist in improving the process efficiency.

Experimental

Equipment

Atmospheric leaching tests were carried out in a 6 L stainless steel batch reactor with an active volume of 4 L. The reactor set-up was geometrically scaled down from the atmospheric leaching reactors used at the Sibanye-Stillwater BMR. The temperature during the experiments was kept constant at 85°C by using a PID controller. Cooling coils which provided a constant flow of cooling water were mounted inside the vessel to remove the excess heat generated by the exothermic leaching reactions. To monitor the temperature, a thermocouple was placed inside the reactor. The vessel was fitted with a liquid sampling port to draw samples at set intervals. To ensure perfect mixing of the reactor contents a stirrer fitted with two agitation blades was used, and the reactor was fitted with four baffles to promote turbulent mixing. During oxidative leaching tests, oxygen was supplied from an oxygen cylinder and delivered to the reactor contents through a stainless-steel sparger. The flow rate of oxygen was regulated by a manual flow control valve and measured by a flow meter.

Table I

Quantitative XRD analysis of the converter matte

Phase	Formula	Abundance (wt%)
Heazlewoodite	Ni_3S_2	65.26
Chalcocite	Cu_2S	20.09
Cu-Ni alloy	CuNi	13.27
Magnetite	Fe_3O_4	0.57

Table II

Bulk chemical composition of matte sample (wt%)

	Sample 1	Sample 2	Sample 3	Mean	Std. dev.
Cu	29.36	28.81	29.79	29.32	0.49
Ni	49.31	52.37	51.79	51.16	1.63
Fe	0.16	0.19	0.17	0.18	0.02
S (balance)	21.18	18.63	18.24	19.35	0.02

The sparger was designed in an 'L' shape, with small equally-spaced holes in the bottom to disperse small oxygen bubbles to the reactor contents. The oxygen entered the reactor vessel through a stainless steel tube with holes at the bottom to ensure that sufficient oxygen bubbles were provided to the reactor contents. A Liebig condenser was fitted to the reactor set-up to reduce the evaporation rate of the contents. Along with the Liebig condenser, a rubber O-ring was placed between the reactor vessel and reactor lid to ensure that vapours formed did not escape.

Materials

Granulated converter matte provided by Sibanye-Stillwater was analysed by quantitative XRD analysis to determine the mineral phases present. The results are shown in Table I and Figure 1. The bulk chemical composition is given in Table II.

The converter matte was milled using a ball mill to a particle size distribution similar to that used by Sibanye-Stillwater so as to be in line with industrial practice. The size distributions are compared in Table III.

Spent electrolyte solution, which is the return anolyte from the copper electrowinning cells, was received from the plant, and analysed by ICP-OES for base metals and PGMs. The concentrations are shown in Table IV.

The acid level was determined by precipitating out all the metals with a Na_2CO_3/$NaHCO_3$ buffer and analysing the solution

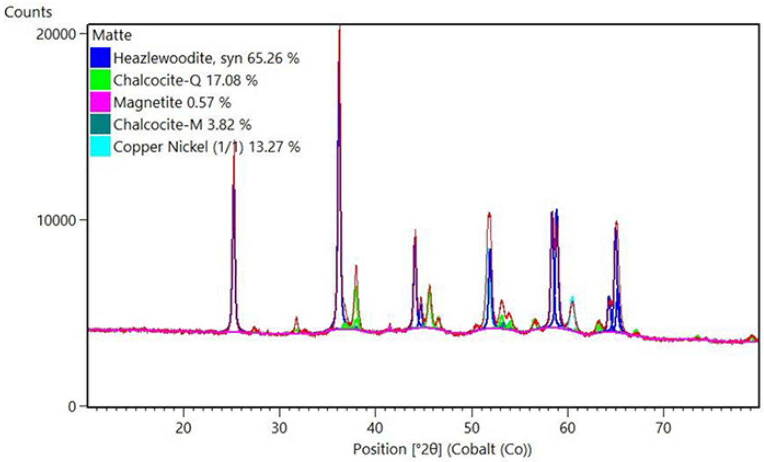

Figure 1—XRD spectrum of the matte sample

Table III

Particle size distributions of milled converter mattes (wt%)

Fraction (µm)	Industry	This study
–200+100	10.5	10.1
–100+80	6.5	6.3
–80+60	10.6	16.9
–60+40	16.1	14.8
–40	56.3	50.9

using high-performance liquid chromatography to determine the sulphate concentration. Four leaching solutions were prepared by diluting the concentrated spent electrolyte solution with an equal volume of distilled water. These four leaching solutions were used to investigate the effects of initial acid concentration or initial Cu concentration on the leaching kinetics of the base metals (Fe and Ni) and PGMs. For tests where a higher acid or Cu concentration was required, the desired concentrations were obtained by adding 98% sulphuric acid or copper sulphate pentahydrate crystals.

Experimental methods

The clean reactor vessel was weighed, 4 L of leaching solution added, and the vessel was weighed again to obtain the weight of the solution. The stirrer was started and set to a speed of 1100 r/min; simultaneously, the heating element was attached to the reactor vessel and the temperature on the PID controller set to 85°C. For oxidative leaching tests, the oxygen flow was turned on and the manual flow control valve set to a flow rate of 0.2 L/min. While the reactor contents were being heated to the set-point value of 85°C, the pH and Eh probes were calibrated using pH standards of 1.69 and 4.0. The Eh probe was a platinum pin double junction Ag/AgCl electrode calibrated with ORP Quinhydrone solutions. Eh-pH stability diagrams were also depicted in Ag/AgCl (mV) used in simulations. The milled converter matte was added to the reactor once the leach solution reached the set temperature. Liquid samples were continually taken throughout the leaching tests at the 15, 30, 60, 120, 150, and 180 minute marks using syringes and 0.45 µm syringe filters.

The four leaching solutions prepared were used to investigate the effect of the initial acid concentration and initial Cu concentration on leaching behaviour, under oxidative and non-oxidative conditions. Table V summarizes the initial Cu and acid concentrations of each of the four leaching solutions used. The experiments carried out are summarized in Table VI. Three repeat experiments were conducted for each set of conditions in order to establish confidence in the results produced. The standard deviations were less than 10.

The oxygen flow rate used in this study was geometrically scaled down from the flow rate used at the BMR. Since the active volume of the laboratory-scale reactor was 4 L, the required oxygen flow rate for the oxidative leaching tests was 0.2 L/min. Van Schalkwyk *et al.* (2011) investigated three different solids to liquid ratios, namely 80 g/L, 150 g/L, and 540 g/L. It was found that at a solid to liquid ratio of 80 g/L complete Cu removal was not possible and hence in this study a solids to liquid ratio of 150 g/L was used to investigate the possibility of maximum Cu and PGE precipitation with reasonable Ni extraction using solutions with different chemical compositions.

Table IV

Composition of the spent electrolyte solution

Species	Ni^{2+} (g/L)	Cu^{2+} (g/L)	Fe^{3+} (mg/L)	Co^{2+} (mg/L)	Ir (mg/L)	Pd (mg/L)	Rh (mg/L)	Ru (mg/L)	Pt (mg/L)	H_2SO_4 (g/L)
Concentration	42.36	26.77	337.77	297.8	31.49	0.0101	10.13	122.2	0.0151	100.04

Table V

Initial Cu and acid concentrations used in the experiments

Species	Solution 1 Low Cu-low acid (LCu-LA)	Solution 2 High Cu-low acid (HCu-LA)	Solution 3 Low Cu-high acid (LCu-HA)	Solution 4 High Cu-high acid (HCu-HA)
Cu (g/L)	13.385	26.77	13.385	26.77
H_2SO_4 (g/L)	50	50	100	100

* Low (L), High (H) and Acid (A)

Table VI

Summary of experimental conditions

Experiment no.	Initial Cu conc. (g/L)	Initial acid conc. (g/L)	Temperature (°C)	Oxygen flow rate (L/min)
1	13.385	50	85	0
2	13.385	50	85	0.2
3	13.385	100	85	0
4	13.385	100	85	0.2
5	26.77	50	85	0
6	26.77	50	85	0.2
7	26.77	100	85	0
8	26.77	100	85	0.2
9 (repeat 1)	13.385	50	85	0
10 (repeat 6)	26.77	50	85	0.2
11 (repeat 3)	13.385	100	85	0

Sample analysis

Liquid samples taken during batch leaching tests were analysed immediately for pH and Eh to obtain data that was as close as possible to the conditions within the reactor. The instruments used were a HI 9321 microprocessor pH meter, capable of measuring pH at elevated temperatures, and a Eutech pH700 instrument with a platinum pin double junction Ag/AgCl electrode for Eh. Dissolved Cu, Ni, and Fe concentrations in the liquid samples from the leaching experiments, as well as in the original spent electrolyte solution, were analysed by means of atomic absorption spectroscopy (AAS) (Varian SpectrAA-250 Plus).

The dissolved PGE (Ir, Rh and Ru) concentrations in the liquid samples obtained from leaching experiments and in the original spent electrolyte solution were determined by ICP-MS. Pt and Pd were present at low concentration levels and were not considered in the study. For solid sample analysis, matte samples were digested in *aqua regia* for 24 hours to dissolve all the metals, and the resulting solutions analysed for Cu, Ni, and Fe by AAS.

Results and discussion

Previous research on the leaching behaviour of Ni-Cu-Fe-S mattes in sulphuric acid was conducted by Lamya and Lorenzen (2006), Fügleberg *et al.* (1995), Hofirek and Kerfoot (1992), Symens *et al.* (1979), and Llanos, Queneau, and Rickard (1974). Although these authors established the major features of the process, the results pertaining to the effects of different initial acid and Cu concentrations are sometimes contradictory or limited.

The Cu concentration is regarded as a leading indicator of PGE behaviour in plant operations, but this relationship has not been conclusively established. Van Schalkwyk, Eksteen, and Akdogan (2013) showed that the behaviour of Cu during oxidative leaching may possibly be used as an indicator of whether PGEs will precipitate, but during non-oxidative tests Cu precipitation was found to be a poor indicator. Batch oxidative and non-oxidative experiments similar to those by van Schalkwyk, Eksteen, and Akdogan (2013) were therefore performed with the main aim of investigating the link between PGE and Cu behaviour, as well as establishing the conditions that promote PGE precipitation in the first-stage atmospheric leach. Here, we report the results of laboratory-scale experiments on the leaching of converter matte at atmospheric conditions to elucidate the effects of oxygen, Cu, and acid concentration on the precipitation behaviour of Cu, PGEs, and Ni extraction.

Effect of oxygen, copper, and acid concentration on PGE behaviour

The effect of oxygen is shown in Figure 2, which indicates that oxidative conditions are conducive to the precipitation of Cu, Ru, Ir and Rh. This is similar to the findings of van Schalkwyk, Eksteen, and Akdogan (2013) with respect to low-Fe mattes. Pt and Pd concentrations in the spent liquor were very low, and although some Pt and Pd precipitated out of solution, the amounts were negligible compared to those for other PGEs and these elements are therefore not included in further discussions.

Figure 3 compares Cu and Ru precipitation out of solution under oxidative and non-oxidative conditions. As seen, both Cu and PGE precipitation exhibit faster kinetics at low Cu and high acid concentrations under oxidizing conditions.

Van Schalkwyk, Eksteen, and Akdogan (2013) argued that Rh, Ru, and Ir are cemented, similarly to Cu. Dorfling (2012) agreed that the precipitation of Rh, Ir, and Ru proceeds primarily via reactions similar to the cementation and metathesis reactions of Cu precipitates (Equation [1]).

$$(\mathrm{Ni,\ Cu}) + (\mathrm{Ru,Ir,Rh})\mathrm{SO_4} \rightarrow (\mathrm{Ni,Cu})\mathrm{SO_4} + (\mathrm{Ru,Ir,Rh})^{\circ} \quad [1]$$

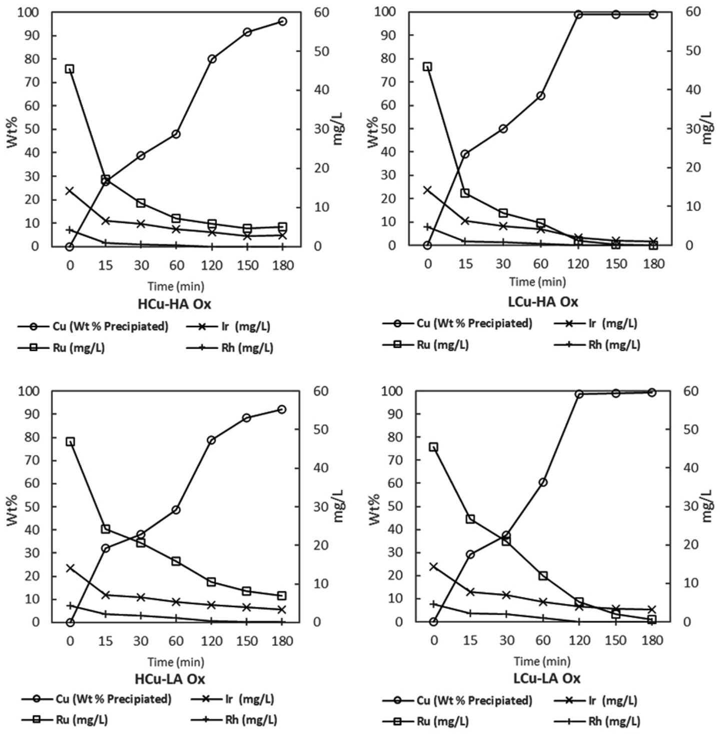

Figure 2—Precipitation kinetics of Cu, Ru, Ir, Rh, and Pt under oxidative leaching with LCu-(LA,HA) and HCu-(LA,HA) (Cu: 13.385 and 26.77 g/L, H_2SO_4: 50 and 100 g/L)

However, various studies propose different formulations for the precipitate, including $Ru(OH)_4$, $RuO_2{\cdot}mH_2O$ and $RuO(OH)_2{\cdot}H_2O$ (Baes and Mesmer, 1976).

Figures 4 and 5 depict Eh-pH measurements, monitored for a duration of 180 minutes, from each group of tests under different conditions, superimposed onto stability diagrams for Cu and Ru, Ir, and Rh.

As can be seen from Figure 4, Cu precipitated as Cu_2S in both oxidizing and non-oxidizing conditions. It is also observed that a low Cu concentration provided slightly better conditions for precipitation. Non-oxidizing conditions obviously shifted the ORP readings towards more reducing conditions closer to the Cu predominance field.

In Figure 5 one can clearly see that oxidizing conditions lead to the production of RuO_2 rather than Ru metal, perhaps closer to the postulation by Baes and Mesmer (1976). This is in contrast to non-oxidizing conditions, in which the precipitation product was Ru metal due to the prevailing reducing conditions. Figure 5 also indicates that under all conditions, Ir and Rh should precipitate as the metals, which is in line with Dorfling's proposal (2012) summarized by Equation [1].

Effect of oxygen, copper, and acid concentration on Ni and Fe behaviour

It is generally accepted that in the presence of oxygen, leaching of the nickel sulphide phase proceeds according to Equation [2] (Llanos, Queneau, and Rickard, 1974; Plasket and Romanchuk, 1978; Hofirek and Kerfoot, 1992; Fugleberg *et al.*, 1995).

$$Ni_3S_2 + H_2SO_4 + \tfrac{1}{2}\,O_2 \rightarrow NiSO_4 + 2NiS + H_2O \qquad [2]$$

In the absence of oxygen, dissolution of the Ni from the sulphide phase takes place according to Equation [3] (Lamya, 2007);

$$NiS + H_2SO_4 \rightarrow NiSO_4 + H_2S \qquad [3]$$

Chalcocite (Cu_2S) leaching under atmospheric oxidative conditions proceeds by Equation [4] (Plasket and Romanchuk, 1978):

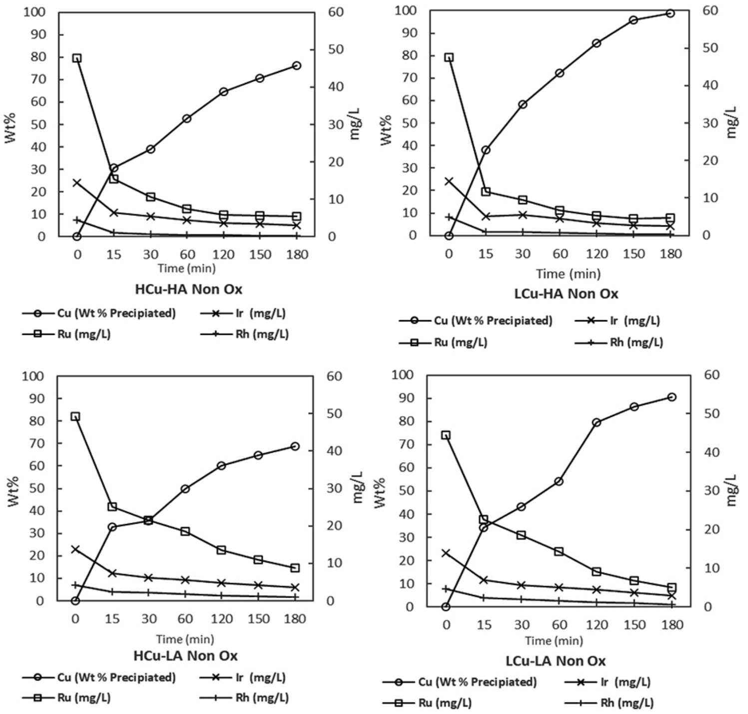

Figure 3—Precipitation kinetics of Cu and Ru under non-oxidative leaching with LCu-(LA,HA) and HCu-(LA,HA) (Cu: 13.385 and 26.77 g/L, H_2SO_4: 50 and 100 g/L

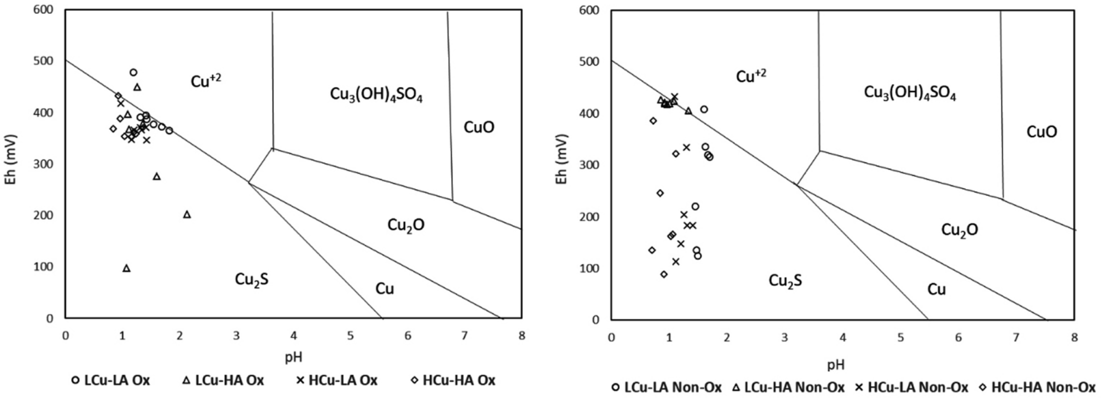

Figure 4—Stability diagrams for Cu precipitation under oxidative and non-oxidative leaching with (L,H)Cu-(L,H)A (Cu: 13.385 and 26.77 g/L, H_2SO_4: 50 and 100 g/L) at 80°C (after Lamya, 2007)

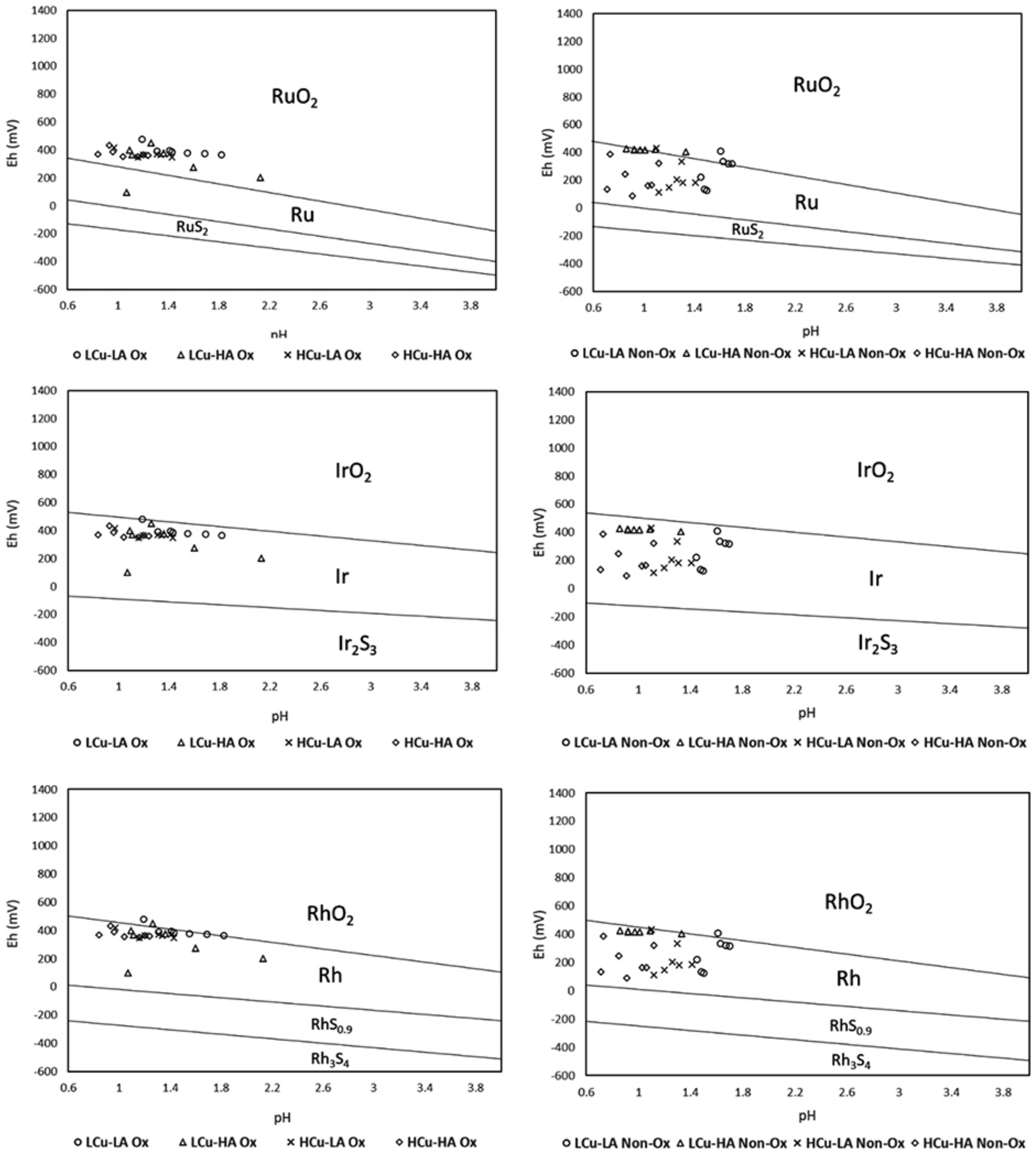

Figure 5—Stability diagrams for Ru, Ir, and Rh precipitation under oxidative and non-oxidative leaching with (L,H)Cu-(L,H)A (Cu: 13.385 and 26.77 g/L, H_2SO_4: 50 and 100 g/L), at 85°C

$$Cu_2S + H_2SO_4 + \frac{1}{2} O_2 \rightarrow CuS + CuSO_4 + H_2O \qquad [4]$$

Being more noble than Ni, Cu from solution can exchange with Ni in the nickel sulphide matrix by metathesis (Equations [5], [6]) or with Ni from the alloy by cementation (Equation [7].

$$Ni_3S_2 + 2CuSO_4 \rightarrow Cu_2S + NiS + 2NiSO_4 \qquad [5]$$

$$NiS + CuSO_4 \rightarrow CuS + NiSO_4 \qquad [6]$$

$$Ni^{o} + CuSO_4 \rightarrow NiSO_4 + Cu^{o} \qquad [7]$$

Although present in relatively small concentrations, Fe has been reported to play an important role as a catalyst and oxygen carrier to enhance the rates of the leaching reactions (Burkin, 2001; Mulak, 1987).

Figures 6 and 7 illustrate the leaching behaviour of Fe and Ni in oxidative and non-oxidative conditions in the presence of Cu, and PGEs. It is clear from these figures that both Fe and Ni undergo greater dissolution from the matrix in the presence of high acid concentrations in an oxidative environment than in the absence of oxygen. Fe leaching proceeds much faster than Ni under both oxidative and non-oxidative conditions in low Cu-high acid conditions. However, the increase in Fe extraction and Cu precipitation coincides with diminishing Ru concentration in solution towards 20 mg/L.

(Hofirek and Kerfoot, 1992). At a low pH, ferrous ions are oxidized to ferric according to Equation [8].

$$2Fe^{2+} + 2H^{+} + \frac{1}{2} O_2 \rightarrow 2Fe^{3+} + H_2O \qquad [8]$$

Ferric ions can act as an oxidant in the leaching of heazlewoodite, which leads to ferrous ions being continuously regenerated (Equation [9]).

$$Ni_3S_2 + 2Fe^{3+} \rightarrow 2Fe^{2+} + 2NiS + Ni^{2+} \qquad [9]$$

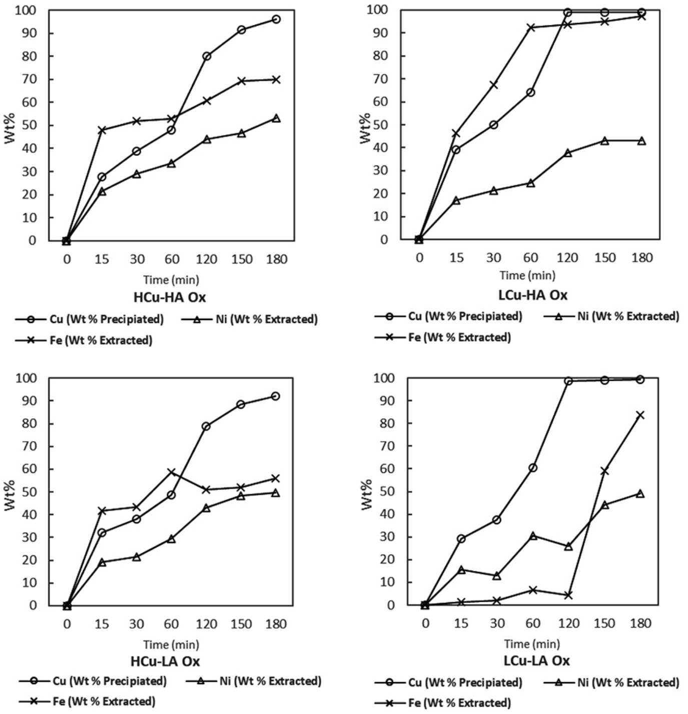

Figure 6—Leaching of Fe and Ni together with precipitation of PGEs under oxidative leaching with LCu-(LA,HA) and HCu-(LA,HA) (Cu: 13.385-26.77 g/L, H_2SO_4: 50-100 g/L)

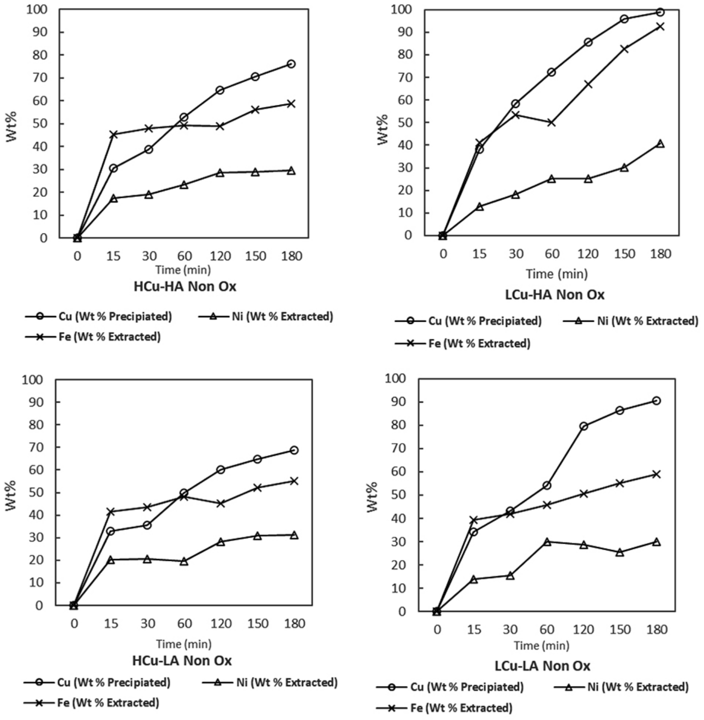

Figure 7—Leaching of Fe and Ni together with precipitation of PGEs under non-oxidative leaching with LCu-(LA,HA) and HCu-(LA,HA) (Cu: 13.385-26.77 g/L, H_2SO_4: 50-100 g/L)

The pH and Eh diagrams for Ni and Fe under both the oxidative and non-oxidative tests are shown on the Pourbaix diagrams in Figures 8 and 9. Ni and Fe are dissolved as Ni^{2+} and Fe^{2+}, while Cu is precipitated as Cu_2S (refer to Figure 4).

Conclusions

The relationship between Cu, Ni, Fe, and PGE behaviour was investigated through a series of batch leaching tests replicating the first-stage atmospheric leach in a base metal refinery. A low-Fe converter matte consisting mainly of heazlewoodite, chalcocite, Cu-Ni alloy, and minor magnetite was leached in a batch reactor under both oxidative and non-oxidative conditions at various acid and Cu concentrations.

The results revealed a higher degree of precipitation for Cu, Ru, Ir, and Rh under oxidative conditions, which agrees with the findings of van Schalkwyk, Eksteen, and Akdogan (2013)

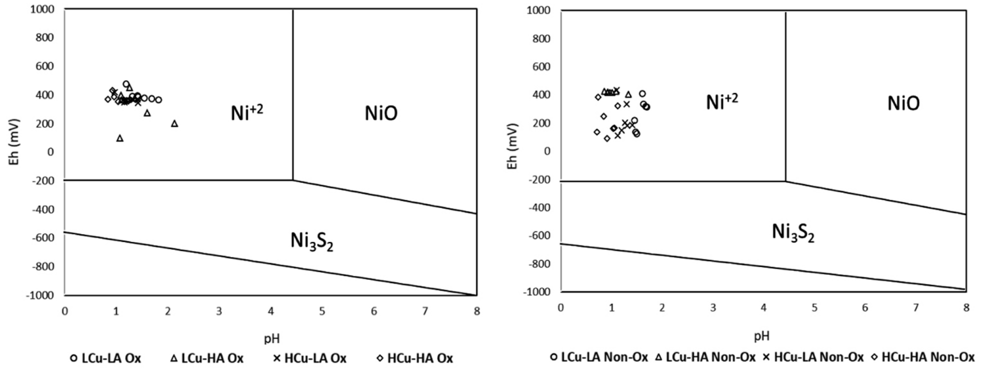

Figure 8—Stability diagrams for the Ni-S-H_2O system at 80°C (after Lamya, 2007) with the superimposed points indicating oxidative and non-oxidative leaching test results

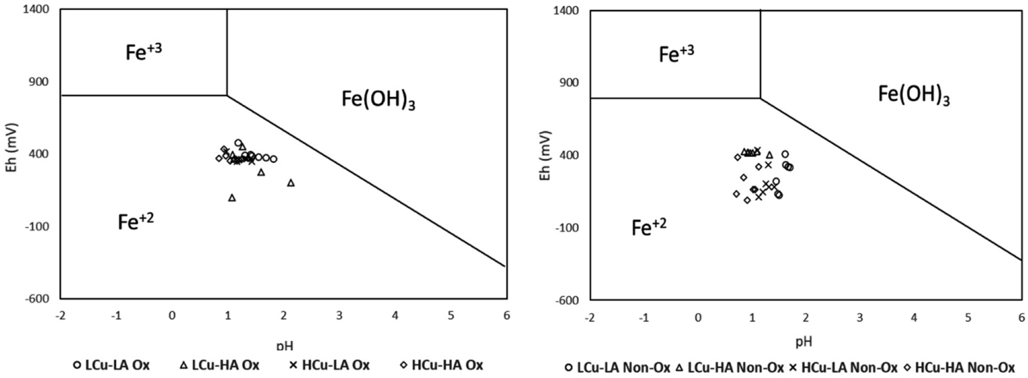

Figure 9—Stability diagrams for the Fe-S-H_2O system at 80°C (after Lamya, 2007) with the superimposed points indicating oxidative and non-oxidative leaching test results

and Snyders *et al.* (2018).The precipitation behaviour of Ru and Ir closely followed that of Cu. A high acid concentration had a positive effect on Cu, Ru and Ir precipitation, as well as Fe and Ni dissolution.

For oxidative leaching experiments it was found that a higher initial acid concentration resulted in higher Ni and Fe extractions, as well as faster leaching rates. A high initial acid concentration also resulted in faster precipitation of Cu, Ru, and Ir.

The effect of initial acid concentration on the leaching behaviour of Ni and Fe, as well as on the extent of Cu precipitation, was much less marked under non-oxidative conditions. A high initial Cu concentration had a negative effect on the leaching of Ni and Fe, as well as the precipitation of Cu and PGEs, under non-oxidative conditions.

Acknowledgements

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