

Leaching of Nickel from a Jarosite Precipitate with Hydrochloric Acid

Willie. Nheta and Mamookho E. Makhatha

Abstract— Jarosite precipitates produced at Impala's Base Metal Refineries contain substantial amounts of nickel. Laboratory-scale experiments were carried out in an attempt to recover nickel by leaching with hydrochloric acid. The leaching efficiency of nickel was 60% at 25°C. The acid concentration of 0.5 – 1 molar, pulp density of 10% and leaching time of 90 min was found to be optimum under the present conditions. High pH could break the jarosite structure and release more iron into the solution which will adversely affect the following precipitation process to recover nickel. 27% of the nickel lost is by entrainment. The reaction is diffusion independent.

Keywords— Hydrochloric acid, Jarosite precipitate, Leaching, Nickel,

I. INTRODUCTION

IN base metal refinery plants, thousands of tonnes of synthetic jarosite precipitates containing 25-36 wt% Fe are produced annually [1] in order to prevent iron from entering the electrowinning process. However, losses of nickel and other valuable base metals may occur either by entrainment or co-precipitation with jarosite because the precipitate is generally discarded. Such losses could be reduced either at the precipitation stage or by subsequent recovery of contained values by leaching.

Generally jarosite has the following formula $(\text{MFe}^{3+}_3(\text{OH})_6(\text{SO}_4)_2)$. Where M represents any of the ions Na^+ , NH_4^+ , H_3O^+ , Li^+ , K^+ , 0.5Pb^{2+} and $\frac{1}{2}\text{Hg}^{2+}$ [2], [3], [4], [5]. The jarosite group minerals are one of the most prevalent naturally occurring basic iron-sulfates. They usually occur as yellow crusts and coatings within the saturated zones of mine tailings and acid sulfate soils [6]. The jarosite group minerals is part of the alunite supergroup, which consists of more than 40 different mineral species that have the general formula $\text{AB}_3(\text{TO}_4)_2(\text{OH}, \text{H}_2\text{O})_6$. Minerals of the alunite-jarosite group can have Na^+ , K^+ , H_3O^+ , NH_4^+ , Ag^+ and $\frac{1}{2}\text{Pb}^{2+}$ forming the A site, and Fe^{3+} (jarosite group). As the jarosite structure can incorporate a large number of elements, its chemical

composition reflects the chemical compositions that of the fluids from which it formed. For example, in the jarosite-alunite series Al may substitute for Fe and a complete solid solution series exists between jarosite and alunite, $\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$. [7]

Dutrillac et al. [8] studied the behaviour of impurities during jarosite precipitation and as far as divalent metals such as Zn^{2+} , Cu^{2+} , and Ni^{2+} are concerned, they are incorporated in the alkali and ammonium jarosites only to a small extent. The order by which the metals are incorporated in the jarosite is as follows: $\text{Fe}^{3+} > \text{Cu}^{2+} > \text{Zn}^{2+} > \text{Co}^{2+} > \text{Ni}^{2+}$.

Laboratory studies have shown that minor amounts of cobalt and nickel are structurally incorporated in sodium jarosite, potassium jarosite and ammonium jarosite and that the extent of incorporation increases as the concentration of dissolved cobalt or nickel increases. Potassium jarosite incorporates more cobalt or nickel (~1% Co or Ni) than does sodium jarosite or ammonium jarosite (~0.4% Co or Ni)[9].

Jarosite forms at low value pH of <1.5 [2]. At high pH values it will dissociate and release Fe back into the solution. Hence when leaching, it must be done at low values (< 1.5) to avoid destruction of the jarosite structure.

A lot of work was done on the study of jarosite structure and its formation but there is limited information on the extraction of base metals lost to the jarosite precipitates. So in this study, the extraction of nickel using hydrochloric acid was investigated. The effects of the test parameters such as reagent concentration, time and pulp density on extraction process was also investigated and compared to earlier results.

II. EXPERIMENTAL

2.1 Material

The jarosite sample used in this study was produced at Implats Base Metal Refineries at Springs, Gauteng. It is an ammonium jarosite precipitate. The sample contains 4.9 % Ni, 38.6 % Fe, 0.02% Cu and other minor elements. The bulk size of the material was 93% - 45µm.

2.2. Experimental Methods

2.2.1 Water Wash

10 g of the jarosite sample was washed under stirring in 200 ml of distilled water to determine the amount of nickel that may have been entrained during solid liquid separation at the plant.

Willie. Nheta is with the University of Johannesburg, South Africa, P.O. BOX 17011, Doornfontein, 2028 (phone: +27 83 737 9427 ; fax: +27 11 559 616 ; e-mail: wnheta@uj.ac.za).

Mamooko. E. Makhatha, is with the University of Johannesburg, South Africa, P.O. BOX 17011, Doornfontein, (e-mail: emakhatha@uj.ac.za).

2.2.2 Leaching

The leaching experiments were carried out by taking required amount of hydrochloric acid in a glass beaker of 500ml capacity placed under an overhead stirrer. The concentration of HCl was varied from 0.01 to 1.0 molar. The solid/liquid ratio was varied from 5% to 30%. The temperature of all the experiments was maintained at 25°C. Time of leaching was varied from 30 to 135 min and the rate of stirring was varied from 50 to 250 rev/min. The leaching efficiency was calculated as- $\text{Leaching efficiency} = \frac{[\text{Ni}]_t}{[\text{Ni}]_0} \times 100$ Where $[\text{Ni}]_t$ mass of nickel in solution after a certain period of time t and $[\text{Ni}]_0$ initial mass of nickel in sample

III. CHARACTERISATION

The chemical composition of the jarosite precipitate was carried out using Rigaku SX Primus ii X-ray fluorescence and atomic absorption spectrometer (Thermo Scientific ICE 3000 Series) for confirmation. The mineral phases present in the jarosite precipitate mineralogical phases of the above mentioned jarosite precipitates were determined by X-ray diffraction XRD analysis, using a Rigaku Ultima IV X-ray diffractometer with Cu $K\alpha$ radiation. The particle size distribution of the jarosite precipitates was measured by the Microtrac M 3551 – XW laser diffraction particle size analyser.

IV. RESULTS AND DISCUSSION

4.1 Mineralogy

The XRD of the jarosite precipitate is given in Fig 1. The peaks above 100 % refers to ammonium jarosite ($\text{NH}_4(\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6)$) and hematite (Fe_2O_3), indicating the major phases. There are other minor phases present which are represented by smaller peaks.

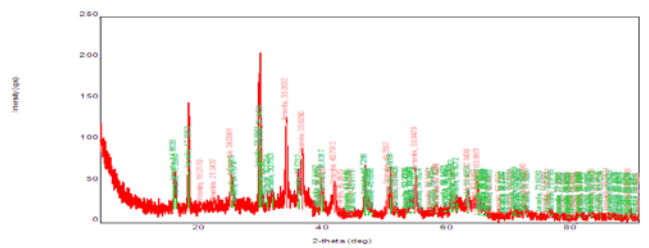


Fig 1 XRD of the sample

4.2 Waterwash

Washing the jarosite precipitate with water resulted in the removal of 27% of the contained nickel. Fig 2 demonstrates that this was readily achieved and just reflects nickel sulfate entrained from the mother liquor. Improved washing of the filter cake is indicated. However, since prolonged washing did not remove further nickel, the remainder might have either been co-precipitated with the bulk of the jarosite [8,9] or otherwise being present as a distinct compound.

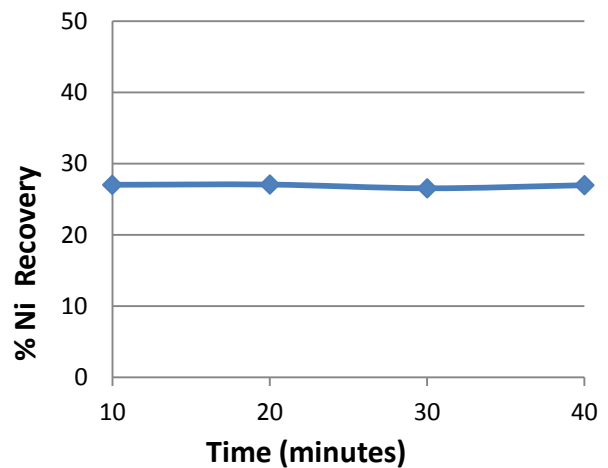


Fig. 2 Water washes at different times, 25°C

4.3 Leaching

4.3.1 Effect of leaching time.

The effect of time on the solubilisation of nickel was only investigated at one acid concentration, viz. 1-molar HCl. The obtained result shown in Fig 3 is rather interesting: During the first 90 minutes only about 45-40% of the nickel contained went into solution; this increased substantially in the following 25 minutes (maximum leaching time 135 min) to reach some 85%.

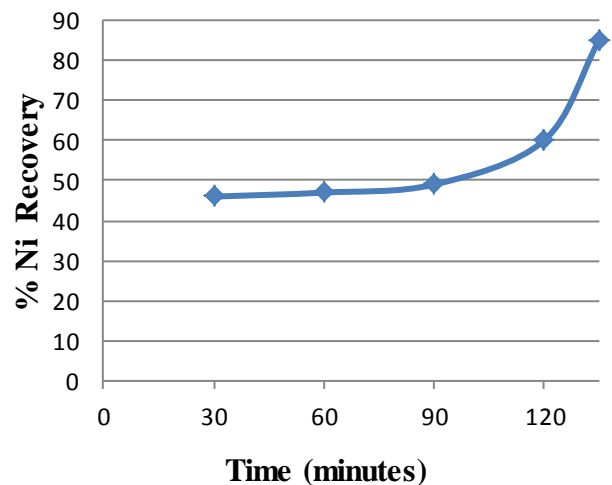


Fig 3. Effect of leaching time, 1M HCl, 10% solid/liquid, 25°C

From the SEM micrographs Fig 4 and results shown in Table I demonstrate that there might be jarosite without nickel (spectrum 8) and nickel in particles low in iron (Spectrum 5). This may explain the trend in the recovery of nickel.

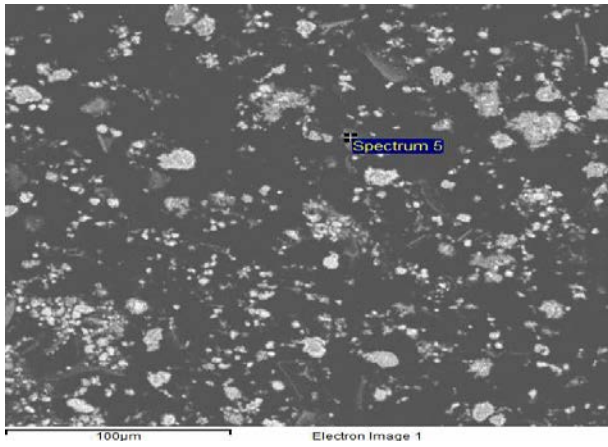
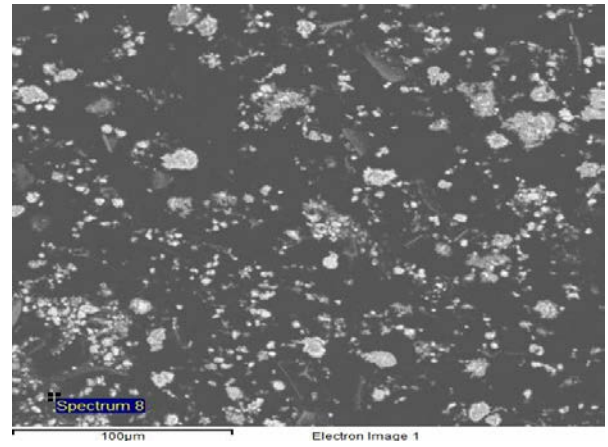


Fig 4. SEM micrographs of the jarosite precipitate



Of interest is the low level of iron that went into solution: it points to either a selective nickel removal from the jarosite lattice – which is difficult to explain – or rather to the presence of a distinct nickel compound

TABLE I
RESULTS FOR SEM ANALYSIS

Elements	Spectrum 5	Spectrum 8
Iron	4.36	29.62
Aluminium	10.58	6.25
Silicon	69.25	33.88
Nickel	1.99	-
Potassium	9.37	2.95
Sulphur	-	-
Sodium	1.93	-
Arsenic	-	-
Chlorine	2.51	27.30
Total	100%	100%

4.3.2 Effect of acid concentration.

In the present study, jarosite precipitate was leached at different concentrations at a constant time of 90 min, 10% solid/liquid, 25°C. As shown in Figure 5, 0.2-molar (7 g/l) HCl is sufficient to dissolve about 60% of the contained nickel; further increase of acid strength has little effect.

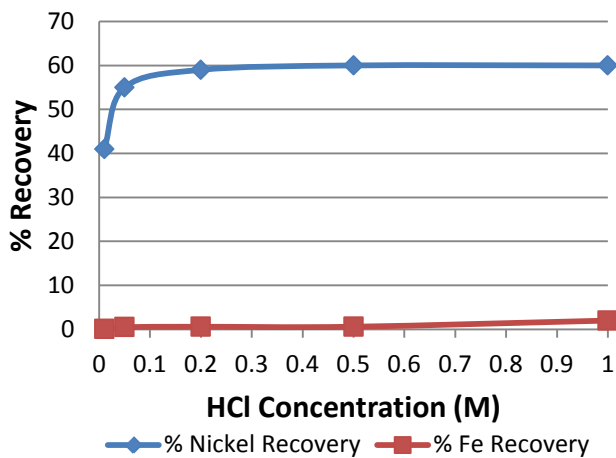


Fig. 5 Effect of HCl Concentration, 90min, 10% solids, 25°C

4.3.3 Effect of stirring speed

The effect of stirring speed on the leaching of the jarosite precipitate using HCl was investigated and the results are shown in Fig 6. Fig 6 shows that the recovery of nickel ranges from 51 to 56 % with the increase of speed from 50 to 250rpm which very insignificant and thus it can be concluded that the leaching reaction is diffusion independent.

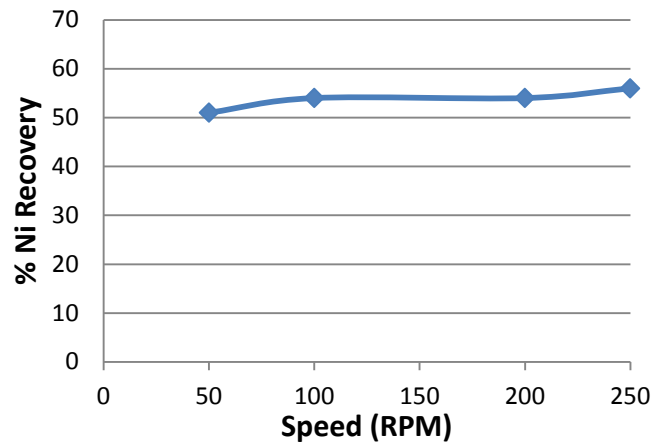


Fig. 6 Effect of stirring speed, 1M HCl, 10% solids, 90 min, 25°C

4.4.5 Effect of Pulp Density

The solid to liquid ratio (w/v) is termed the pulp density. The effect of pulp density on nickel extraction is given in Fig 7. It is clear that from 10% to 30 % pulp density, 58% extraction was achieved.

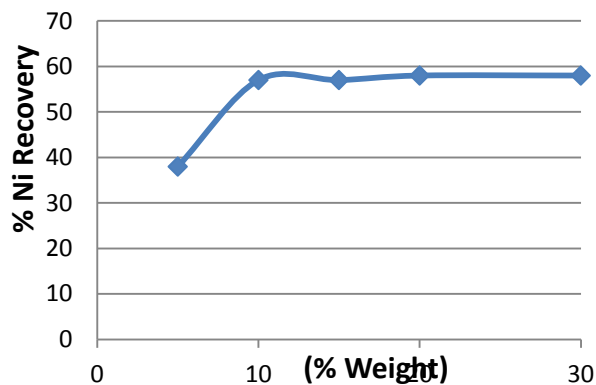


Fig 7. Effect of Pulp Density, 1M HCl, 90min, 25°C

IV. CONCLUSIONS

Apart from leaching nickel from jarosite precipitate, Impala Refineries should: 1. Wash properly the precipitate after filtration 2. Modify the precipitation process to reduce nickel co-precipitation and this would require more experimental work.

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