

Quantitative Leaching of Nickel From Jarosite Using Sulphuric Acid

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ABSTRACT This paper deals with the extraction of nickel from jarosite precipitates. Nickel loss to these precipitates is mainly by entrainment and coprecipitation. Nickel is hazardous to the environment. In this study, an attempt has been made to extract nickel entrained in jarosite precipitates produced at Implats Base Metal Refineries. The leaching efficiency of nickel was 59% at 30°C. The acid concentration of 0.5 – 1M, pulp density of 10%, leaching time of 21/2 hours was found to be optimum. High concentrations of acid and high temperatures could break the jarosite structure and release more Fe into the solution which will adversely affect the following precipitation process. The reaction is diffusion independent.

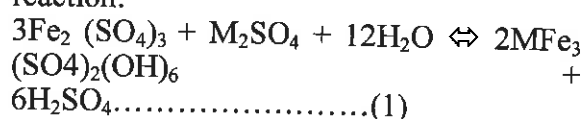
Key words: Jarosite precipitates, Leaching, Nickel, Waste and Effluent

1 INTRODUCTION

Increase in environmental alarm due to unacceptable management of both hazardous and non-hazardous wastes on the rampage from different industrial processes prioritize the need for the improved research. Nowadays, with the environmental regulations becoming more and more severe and growing pressure on the conservation of mineral resources, economically-viable refining technology in recycling has gained interest. The most serious environmental issue faced by the processing industry is the process of effluents. Mineral processing industries may contain heavy metals, organic waste, and oils, including the waste liquids from wastewater streams. Toxic metals existing in high concentrations must almost certainly be well treated (Dutrillac & Chen, 2000). On the other hand, the disposal process can bring about some complexity. Companies are also suffering from an increased disposal cost besides the environmental sensitivity.

The focus is lately on the ability to clean up waste and return or recycle a significant proportion (Kurama, 2009; Kurama, 2007). The waste which is generally regarded as hazardous could be further considered as a rich secondary source of metal such as nickel from jarosite precipitates.

Jarosite ($KFe^{3+}_3(OH)_6(SO_4)_2$) is a basic hydrous sulphate of potassium and iron, its formation is represented by the following reaction:



Where M represents any of the ions Na^+ , NH_4^+ , H_3O^+ , Li^+ , K^+ , $0.5Pb^{2+}$ and $\frac{1}{2}Hg^{2+}$ (Long, et al., 1992).

Nickel is not on the above list of ions that can be incorporated in a jarosite structure neither can it substitute Fe^{3+} . Hence loss of nickel to the jarosite precipitate can either be by entrainment or co-precipitation. However with formation of Beaverite jarosite $Pb(Fe,Cu)_3(SO_4)_2(OH)_6$, Fe^{3+} can be replaced by either Cu^{2+} or Zn^{2+} or both in

the structure (Dutrizac & Chen, 2000; Dutrizac, et al., 1980).

Jarosite structure have been intensely researched and it is best described in the space group R-3m and has lattice parameters $a \sim 7.3 \text{ \AA}$, $c \sim 17 \text{ \AA}$. The kagome plane is made up of iron coordination octahedra, and the Fe octahedra are capped above and below by sulphate tetrahedral (Wills, et al., 2006).

The jarosite group of minerals has been extensively studied as a result of its importance as a by-product of the metal-processing industry as well as being very common in acid-mine waste. Minerals within the jarosite group are commonly found in acidic, high-sulfate environments associated with mine tailings (Basciano & Peterson, 2007).

Annually thousand tonnes of synthetic jarosite is produced and contains 25-36 wt% Fe (Dutrizac & Chen, 2000). Group minerals of jarosite are one of the most commonly natural occurring iron-sulfates. They usually occur as yellow crusts and coatings within the saturated zones of mine tailings and acid sulfate soils. These group of minerals consists of more than 40 different mineral species that have the general formula $AB_3(TO_4)_2(OH, H_2O)_6$ and is part of the alunite supergroup (Bigham & Nordstrom, n.d.). Minerals of the alunite-jarosite group can have Na^+ , K^+ , H_3O^+ , NH_4^+ , Ag^+ and $\frac{1}{2}Pb^{+2}$ forming the A site, Fe^{3+} (jarosite group). As the jarosite structure can incorporate a large number of elements, its chemical composition reflects the chemical compositions of the fluids from which it formed. For example: Oxygen in the SO_4 site reflects the source of oxygen during oxidation of the sulfide, this value will depend on whether water or air provides the oxygen and if any biogeochemical (microbial) processes are involved. Oxygen in the OH site is more complex and reflects the character of the parent fluid, equilibrium exchange processes, and temperature. Natural and synthetic jarosite group minerals commonly

have significant quantities of hydronium in the alkali site and minor to major deficiencies in the iron site. Jarosite is also important in the base metal industry as a sink for iron; it is precipitated as a means of removing the iron that is commonly present in base metal concentrates.

Sulfuric acid is most widely used acid for leaching due to the following advantages: (i) high solubility of base metals, (ii) Low price, (iii) well established technology for solvent/electrowinning in sulphate media and (iv) regeneration of acid after solvent extraction.

2. EXPERIMENTAL

2.1 Material

The jarosite precipitate used in this study was produced at Impala Base Metal Refineries. The bulk size of the material was 93% - 45 μ m.

2.2 Method

2.2.1 Water Wash

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

2.2.2 Leaching

The leaching experiments were carried out by taking required amount of sulfuric acid in a glass beaker of 500ml capacity placed under an overhead stirrer in a water bath. The concentration of sulphuric acid was varied from 0.01 M to 3M. The pulp density was varied from 5% to 30%. The temperature of all the experiments was maintained at 25 °C except where it was varied between 25 °C to 60 °C. Time of leaching was varied from ½ hr to 8hrs and the rate of stirring was varied from 50 to 250revs/minute. The rate of leaching was monitored in terms of leaching efficiency calculated as-

$([W]t/[W]c) \times 100 = \text{leaching efficiency in \%}$
(2)

Where, [W]t=Wt. of the metal dissolved in solution after a particular time period

[W]c=Total wt. of the metal in the sample taken.

3. 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 mineralogical phases of the above mentioned jarosite precipitates were determined by XRD analysis, using a Rigaku Ultima IV X-ray diffractometer. The morphology of the jarosite precipitate was studied by Scanning Electron Microscope (Tescan model). The particle size distribution of the jarosite precipitates was measured by Microtrac particle size distribution analyser.

4. RESULTS AND DISCUSSION

4.1 Mineralogy

The XRF of the jarosite precipitate results are shown in Table 1. From The table, it is

seen that it contains about 6.16% nickel and this is too high to be lost to the jarosite precipitate. Major component of the jarosite precipitate is iron as expected.

Further quantification of the elements was determined using an atomic absorption spectrometer and the results are shown in Table 2. Only nickel, iron and copper were analysed as they were going to be investigated in the leaching experiments. There is a huge variation with the XRF results obtained in Table 1. According to AAS results, nickel content is on average of 4.87% while iron at an average of 38.63% and copper was found to be at the lowest of 0.0197%. Being more precise AAS was used for all the chemical analysis of all the following experiments.

The XRD of the jarosite precipitate is given in Fig 1. The peaks above 100 % refers to jarosite ($K(Fe_3(SO_4)_2(OH)_6)$) and Hematite (Fe_2O_3), indicating the major phases. There are other minor phases present which are represented by smaller peaks.

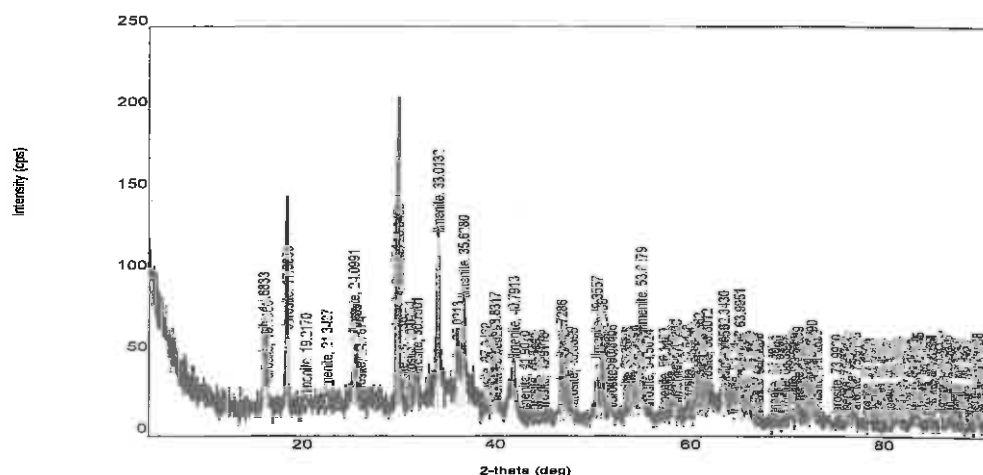


Figure 1: XRD of the jarosite precipitate.

Table 1: XRF results

Element	Mass %	Element	Mass %	Element	Mass %
Ni	6.16	Pb	1.84	K	1.00
Fe	65.98	Na	0.39	Si	10.97
Al	2.90	Cr	0.28	S	7.76
Ca	0.14	As	2.38		

Table 2: AAS results

Element	Sample 1	Sample 2	Sample 3	Average
Nickel	4.65%	4.91%	5.04%	4.867%
Iron	37.59%	39.86%	38.43%	38.63%
Copper	0.0273%	0.0197%	0.0120%	0.0197%

The morphology of the jarosite precipitate was studied and the micrographs and results

are shown in Figure 2 and Table 3 respectively.

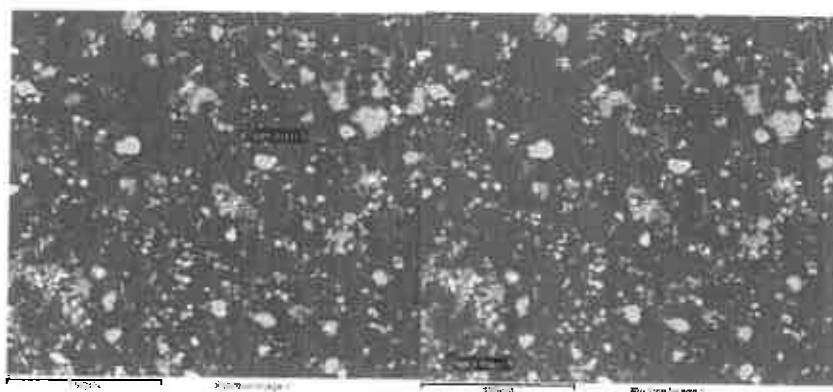


Figure 2: SEM micrographs of the Jarosite precipitate

Table 3: SEM results

Element	Fe	Al	Si	Ni	K	S	Na	As	Cl
Spectrum 5	4.36	10.58	69.25	1.99	9.37	-	1.93	-	2.51
Spectrum 8	29.62	6.25	33.88	-	2.95	-	-	-	27.30

It can be seen that a particle on spectrum 5 is rich in silicon at 69.25%, while iron and nickel are 4.36% and 1.99 % respectively. A particle on spectrum 8 has 29.62% iron and no nickel. These two results gives an indication that a particle on spectrum 8 might be a jarosite crystal and the one on spectrum 5 might be another form of precipitate which contains nickel.

4.2 Water Wash.

A water wash was carried on the jarosite precipitates at different times and the results are shown in figure 3. Figure 3 shows that the nickel recovery remained constant around 27% nickel recovery regardless of the leaching time. It can thus be deduced that 27% of nickel losses in the plant to jarosite precipitates are a result of poor washing techniques employed at Implats plant. The remaining 73% either co-precipitates with the jarosite precipitates or forms part of the jarosite crystal structure. There is evidence form literature that base metals can be incorporated in a jarosite crystal eg Beaverite jarosite (

$Pb(Fe,Cu)_3(SO_4)_2(OH)_6$) where Fe^{3+} is replaced by Cu^{2+} or Zn^{2+} or both.

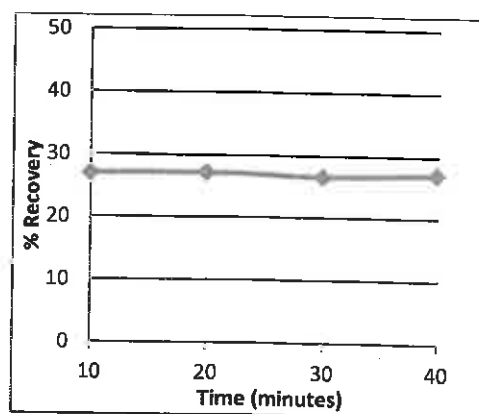


Figure 3: Water washes at different times, 25°C

4.3 Leaching

Leaching of the jarosite precipitate was carried out to extract nickel from the precipitates. This material is not a naturally occurring geological material but an industrial deposition. Therefore, its

mineralogy and leaching behaviour are quite different from the normal ores and minerals. Variation of parameters were studied and optimised. The results are given in the following sections.

4.3.1 Effect of leaching time.

The jarosite precipitate was leached at different times using 1Molar H_2SO_4 and 10% pulp density at room temperature and the results are shown in figure 4. From figure 4, it follows that with the increase of leaching time from 30 minutes to 150 minutes, recovery of nickel increases from 38.9 to 51.3 %. Further increase in leaching time from 150 to 480min recovery of nickel does not significantly increase (2.4%).

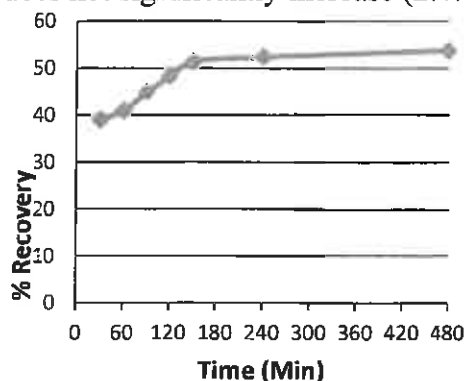


Figure 4: Effect of time on leaching of jarosite precipitate, 1M H_2SO_4 , 10% PD, 25°C

4.3.2 Effect of acid concentration.

The concentration of the acid is one of the major parameters for recovery of nickel from jarosite precipitates. Acid breaks the complex to release the metal values. In the present study, jarosite precipitate was leached at different concentrations at a constant time of 2 1/2 hrs, 10% pulp density, 25 °C and the results are shown in figure 5. Figure 5 shows that the optimum leaching concentration of H_2SO_4 is between 0.5 and 1M H_2SO_4 where there is a recovery of around 48% Nickel and less than 5% Fe Recovery. With the increase in the acidity

from 0.05 to 1M H_2SO_4 , recovery of nickel increases from 50 to 58% and recovery of Fe from 0.4 to 1.5%. There is need to keep the concentration of Fe as minimum as possible (5%) as this will adversely affect in the following precipitation process. Further increasing H_2SO_4 concentration will only increase recovery of Ni by 10% and that of Fe will go up to 3.5%.

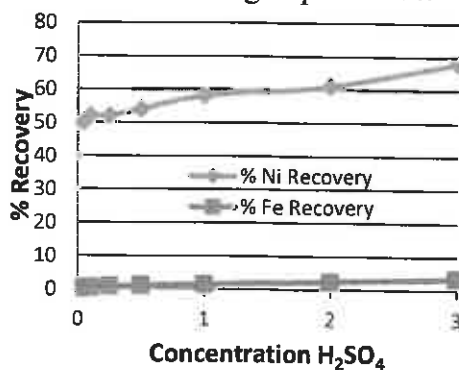


Figure 5: Effect of acid concentration on leaching of jarosite precipitate, 2 1/2 hrs, 10% PD, 25°C

4.3.3 Effect of stirring speed

The effect of stirring speed on the leaching of the jarosite precipitate was investigated and the results are shown in figure 6. Figure 6 shows that the recovery of nickel does not change much with the change in stirring speed and thus it can be concluded that the leaching reaction is diffusion independent.

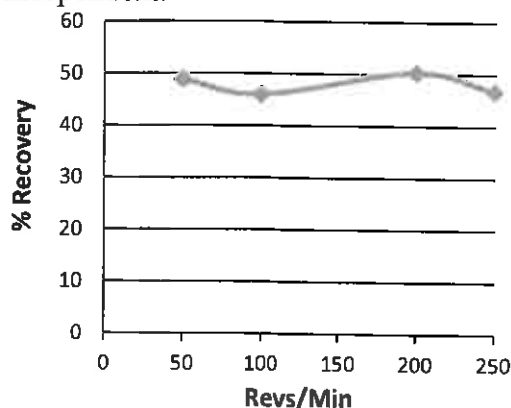


Figure 6: Effect of stirring speed on leaching of jarosite precipitate, 1M H_2SO_4 , 2 1/2 hrs, 10% PD, 25°C.

4.3.4 Effect of temperature

The effect of temperature was studied for the liberation of nickel and Iron. Jarosite precipitate was leached at different temperatures and the results are shown in figure 7. It was found that increase in temperature increases the recovery of Ni and Fe. Increasing leaching temperature from 25 to 60 °C, recovery of Ni increases from 52 to 75% and Fe from 1.7 to 14%. Thus at high recovery of Ni (60 °C) there is also a high recovery of Fe and this will adversely affect the following precipitation process. Optimum leaching temperature will be 30°C where recovery of Ni is 59% and Fe 5.5%.

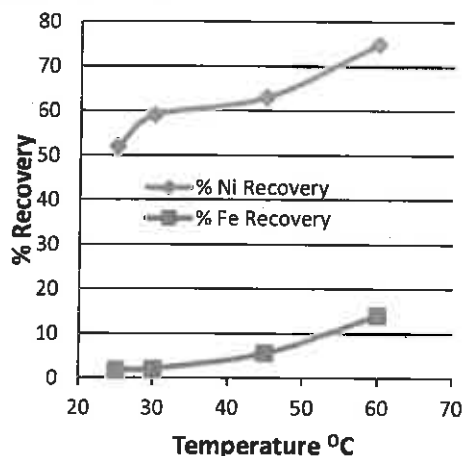


Figure 7: Effect of leaching temperature, 1M H₂SO₄, 2 ½ hrs, 10% PD.

4.3.5 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 figure 8. It is clear that in both 5% and 10% pulp density, 48% extraction was achieved. But when the pulp density was increased to 30%, the recovery was reduced to around 43%. Hence in this case 10% may be taken as the optimum pulp density for optimal recovery of nickel.

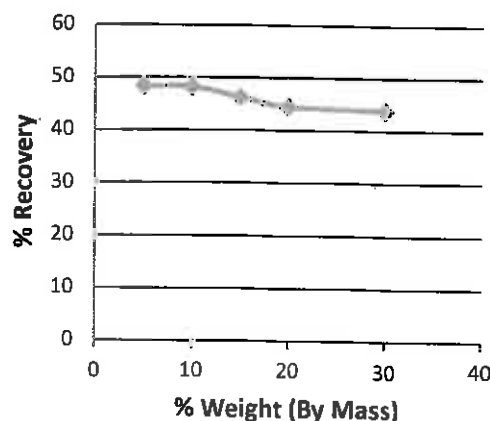


Figure 8: Effect of pulp density, 1M H₂SO₄, 2 ½ hrs, 25°C

5. CONCLUSION

The jarosite precipitate containing nickel was leached in sulphuric acid medium at different temperature and acid concentrations. The leaching efficiency of nickel was 59% at 30°C. The acid concentration of 0.5 – 1M, pulp density of 10%, leaching time of 2 ½ hours was found to be optimum under the present conditions. High concentrations of acid and high temperatures could break the jarosite structure and release more Fe into the solution which will adversely affect the following precipitation process. The reaction is diffusion independent.

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