Potentiodynamic Polarisation and Chronopotentiometric Studies of Cobalt-Copper Oxidised Ore in Sulphuric Acid

Kgadi A. Makgamatha, Antoine F. Mulaba-Bafubiandi, and Feyisayo V. Adams

***Abstract*—** The electrochemistry of leaching low grade Co-Cu oxidized ore containing 3.32% Co and 0.403% Cu in sulphuric acid was studied. Potentiodynamic polarization and potentiometric method were used. The potentiodynamic polarization curve (potential vs. current density) at different pH of 1.5, 2.5, 3.5 and 6.5 were measured and obtained results revealed that at high current density ($1x10^{-3}$ to $1x10^{-4}$ A/cm^2) the amount leached in acidic solution was higher, whereas at constant current density ($1x10^{-4}$ A/cm^2) passivation occurs and at zero current density little leaching occurs. Consequently pH of 2.5 gave percentage dissolution of 3.34% Co and 4.22% Cu at a current density starting from $1x10^{-3}$ to $1x10^{-4}$ A/cm^2. The small Co^{3+} recovery was mainly due to the absence of a reducing agent in solution. During potentiometric method readings were taken for every 2 minutes interval for 30 minutes prior changing the samples at different concentration of 0.15M, 0.25M and 0.35M. The results showed pH trends of 0.15M – 0.35M decreased respectively as leaching proceeded with time. The plot of potential vs. time at 0.15M and 0.35M system yielded fluctuated trends which suggested the possibility of passivation occurring, however 0.25M trend showed potential being maintained constantly at 0 V with increasing time. The AAS results of the potentiometric at 0.25M for Co-Cu in percentage dissolution were 24.64% and 14.28% as most leached compared to 0.15 and 0.35M. Consequently in 0.25M concentration, less passivation occurred with time. The pourbaix diagrams (Eh vs pH) of Co-Cu for this study was constructed each at 0.15M, 0.25M and 0.35M, where it was found that to obtain maximum percentage dissolution for both cobalt-copper in sulphuric acid, pH should be maintained at pH<1.

***Keywords*—** Dissolution, electrochemistry, leaching, ore.

I. Introduction

THE DRC and Zambian copper belts have been reported to be the second largest global reserve of copper, about 1/3 the size of Chilean reserve [1]. Several oxidized Cobalt-Copper (Co-Cu) mining industries are situated in the Katanga province of the DRC. There are about seventy two economic deposits of Co-Cu and four large mining centers all together. The extraction of base metals from oxidized Co-Cu ore have been conducted by several researchers and to date, studies have shown the effectiveness of leaching in this mineral processing [2]. There are different types of leaching techniques used in the industries to leach different types of minerals in their body of ore. These techniques are known as in-situ leaching, heap leaching, dump leaching and agitator leaching [2].

The oxidized Co-Cu ores are normally leached through an acidic or alkaline medium such as H_2SO_4, HCl, HNO_3, basic NaOH, NH_4OH or salts $CuCl_2$ and $FeCl_2$[2]. The leaching of four different oxidized Co-Cu ore from DRC in hydrochloric acid and $FeCl_2$ solution were recently carried out and the test has proven to have yielded expected results and answered the study questions of utilizing acid as the recommended leaching media in oxidized ore [5]. It was found that Co leaching in the absence of $FeCl_2$ was < 36% but copper leaching was > 70%. Addition of $FeCl_2$ resulted in the reduction of Co^{3+} to soluble Co^{2+}. It was also reported that the leaching of cobalt was very rapid with only a rate of dissolution evident beyond 30min [3].

The effectiveness of HCl leaching of Co and Cu from four oxidized cobalt bearing ores in the presence of $FeCl_2$ to produce in-situ Fe^{2+} capable of reducing Co^{3+} to Co^{2+} has also been investigated [4]. The effects of the concentrations of HCl, $FeCl_2$, leaching time, particle size and the dissolution kinetics were considered. In this studies, Cu recoveries of 88%, 77%, 75% and 86% were obtained for high Cu ore, high Co, low Co and high Co with mica respectively. It was reported that the increase in $FeCl_2$ concentration did not have important effect on the Cu leaching. The optimum leaching conditions were found to be leaching time; 30min, reagent concentrations; mixture 1M HCl + 105.18g $FeCl_2$, stirring speed avoiding the vortex, room temperature, and pH values from 0.402 to 0.322 [4].

Ntengwe [5] reported that leaching of dolomitic-copper using sulphuric acid under controlled conditions yields effective recovery of copper from chalcopyrite. Efficiencies of 72 to 93% for 75µm particle size and 65 to 85% for the 212 micron sieve at different pulverizing time were achieved [5]. Although, the acid leaching of oxides, carbonated and sulphides ore have mostly been investigated with positive

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outcomes, little or no work have been done on the acid leaching of Co-Cu oxidized ore and its electrochemistry. It is therefore necessary and of great importance to study the electrochemistry of Co-Cu oxidized ore in acidic solution in the absence of a reducing agent. This research will be focused on the potentiometric and polarization behavior of oxidized Co-Cu ore in acidic medium.

II. Methodology

A. Materials

The oxidized Co-Cu ore for this study was collected from the Katanga province in the Democratic Republic of Congo. Its mineralogy was later shown after all the necessary analysis had been conducted. Equipment which were used for this project included: Crushers, X-Ray Diffractometer (XRD), Scanning Electron Microscope (SEM), Atomic absorption spectroscopy (AAS), and X-ray fluorescence (XRF). The compositions of the ore were determined using XRF, morphology was obtained from SEM, while XRD was used to study the mineralogy of the ore. The electrochemical tests were done using Autolab Potentiostat (PGSTAT30 computer controlled) with the General Purpose Electrochemical Software (GPES) version 4.9.

B. Reagent

Sulphuric acid (98 vol% H_2SO_4), was utilized as the leaching agent. The leaching concentration was ranged between 0.04 - 0.6 M and pH of 1.4 - 6.6.

C. Working electrode preparation

The working electrodes were prepared from the bulk ore of -75µm particle size (2g each) compressed under pressure using sucrose as binder into a pellet of 14 mm diameter and 5 mm thickness. The electrodes were mounted, with one surface exposed, in an epoxy resin and the other surface sealed with copper wire using aluminum (foil) conductive tape, to enable electrical contact of the ore with the copper wire. The exposed surface of the electrodes was further polished with 220 grade silicon carbide paper to remove any resin that might have remained on the electrode surfaces after molding. Scanning electron microscopy (SEM) technique was used to study the morphology of the working electrode.

D. Electrochemical methods

All electrochemical tests were carried out at room temperature and fixed particle size (-75µm). the electrochemical measurements were done at three different concentrations (0.15M, 0.25M and 0.35M) and four pH (1.5, 2.5, 3.5, 4.5, 5.5 and 6.5). The General Purpose Electrochemical Software (GPES) version 4.9 in an Auto-lab potentiostat (PGSTAT30 computer controlled) was used for all the electrochemical tests. Electrochemical measurements which were conducted on the electrodes included the potentiometric and linear potentiodynamic polarization techniques. Polarization curves were obtained at varied applied voltages between -0.6V to 1.2V at constant scan rate 1.6 mV/s. The potentiometric tests were carried out at constant current for 30 mins. Atomic absorption spectroscopy (AAS) was used to determine the concentration of metals in the electrolyte after electrochemical tests.

III. Results And Discussions

A. Mineralogical studies

The mineralogical studies of the crushed oxidized Co-Cu ore were classified by the XRD. Fig. 1 shows the main minerals found in the ore as clinochlore (Mg, Al), chromian (Cr, Al), copper cobalt oxide (CuCoO) and coesite (SiO_2). The attribution of the mineral phases was achieved by comparing the principal peak intensities to reference XRD cards, whereby the coesite peak as shown in Fig. 1 was the highest as compared to clinochlore, chromian and copper cobalt oxides. Practically the ore is more concentrated in silica oxide (SiO_2) than copper and cobalt [6].

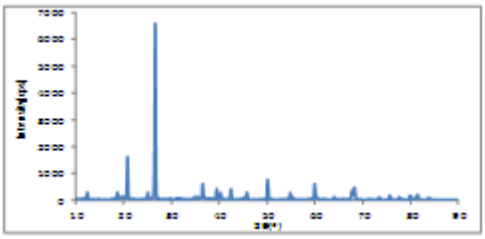

Fig. 1 XRD of oxidized Cu-Co Ore from Katanga Province in the DRC

B. Chemical compositions of Copper-Cobalt ore

The chemical compositions of the ore were determined by the x-ray fraction. Table I illustrates the compounds which were present within the ore. As shown SiO_2 was the most abundant compund found in the ore with 64.9 mass % and that of the oxides of Co and Cu were 3.32 and 0.403 mass%, respectively.

Table I
Quantitative Analysis Of Co-Cu Oxidised Ores Using Xrf

Components	Composition (mass %)
F	0.28
Al_2O_3	8.82
SiO_2	64.9
P_2O_5	0.26
SO_3	2.22
Cl	0.045
K_2O	0.66
CaO	1.48
Cr_2O_3	0.041

MnO	1.26
F_2O_3	3.35
Co_2O_3	3.32
NiO	1.61
CuO	0.403
ZnO	0.009
Ga_2O_3	0.004
Rb_2O	0.0023
Nb_2O_5	0.004
MoO_3	0.0075
PbO	0.146
U_3O_8	0.0722

C. SEM analysis of Cu-Co ore

The morphological characteristic of the ore surface was determined by SEM. As shown by SEM micrograph, the total morphological surface is more dominated by the greyish which is noted to be silica oxide as it contributes 64.98% of the total oxidized Cu-Co ore. The dark black spots were identified as cobalt copper oxide which contributes 3.5% and 0.5% of the ore, respectively. The clinochlore mineral was identified as whitish spot [7].

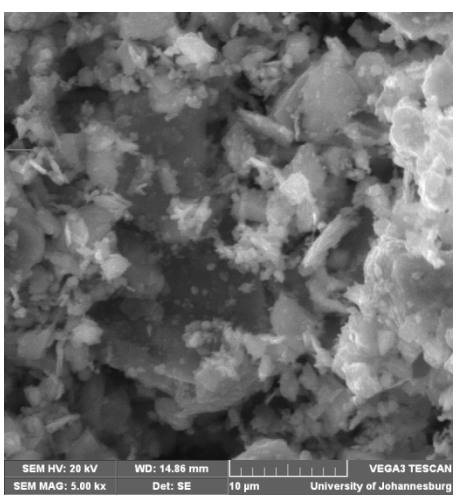

Fig. 2 SEM micrograph of Cu-Co ore

D. Potentiodynamic polarization tests

The linear potentiodynamic polarization was measured at different pH of 1.5, 2.5, 3.5 and 6.5. The polarization behavior of the oxidize ore in sulphuric acid at varied pH are shown in Fig. 3. The measurements were based on determining the polarization behavior of the ore. The pH of 2.5 displayed the most active dissolution rate through experiencing a small cathodic region as shown by a slight passivity at $1x10^{-5}$ (A/cm^2) and applied potential of -1 V. As observed in Fig. 3, the passivity of the ore at pH 2.5 broke and repassivation occurred as the ore leached constantly at $1.5x10^{-5}$ (A/cm^2) and applied potential of -0.9V in the cathodic region to 0.5 V in the active anodic region throughout the experiment [8]. A similar behavior was observed at pH of 6.5. However, an initial passivity was shown at 0.82 V compared to pH of 2.5. A rapid passivity was observed at pH of 1.5 and 3.5 with little leaching. It can therefore in this case be generalized that at more negative potentials, more leaching is expected, consequently pH of 2.5 showed the best polarization behavior as compared to pH of 1.5, 3.5 and 6.5.

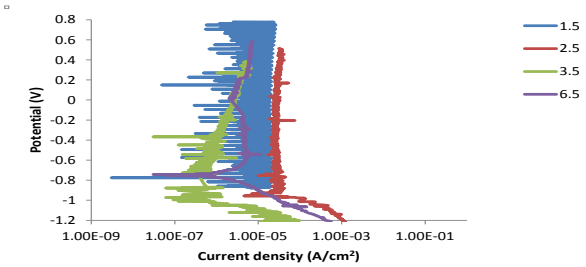

Fig. 3 Potentiodynamic polarization curve for 1.5, 2.5, 3.5, and 6.5 pH

E. Atomic absorption spectroscopy results of leached Co and Cu after potentiodynamic polarization tests

After potentiodynamic polarization tests, the electrolytes were taken for AAS analyses to determine the percentage amount of Co and Cu leached in the solution. As shown on Table II, the highest Cu leached was 4.22% at a pH of 2.5, followed by a pH of 1.5 at which 0.65% was leached. The pH of 3.5 and 6.5 had the lowest dissolutions rate that yielded 0.16% and 0.12% respectively. Similar behavior was discovered by Ntengwe in 2010 during a study on the leaching of dolomitic-copper ore of 75 micron particle size using sulphuric acid for 0-6 hours under controlled conditions. The author reported that leaching was favored at low pH values (1.8<pH<3) than at high ones (pH>3) moreover profit levels for leaching at low to high pH were higher than that of leaching at high to low pH particularly for acid sales [9]. The same was true for Co where a pH of 2.5 extracted 3.34% of Co from the ore followed by pH of 1.5 with 0.37% leaching. The pH of 3.5 and 6.5 were the least respectively as with Cu leached.

TABLE II
QUANTITATIVE ANALYSIS OF CU-CO OXIDIZED ORES BY FLAME AAS

pH	Concentration (ppm)	
	Cu	Co
1.5	1.06	5.58
2.5	6.91	50.5
3.5	0.27	2.65
6.5	0.2	0.42

TABLE III
PERCENTAGE LEACHED OF CU-CO AT VARIED PH FROM POLARIZATION TESTS

pH	% leached	
	Cu	Co
1.5	0.65	0.37
2.5	4.22	3.34
3.5	0.16	0.18
6.5	0.12	0.03

F. Potentiometry tests in sulphuric acid

Potentiometric test at varying concentration was conducted to observe the applied potential trends as pH changes with time. Concentration of 0.15M, 0.25M and 0.35M were prepared to perform the potentiometric study. The pH of the solutions was recorded as it changes with time and simultaneously the potential was recorded as it was affected by pH per time change. The readings were taken for every 2 min interval for 30 min. Fig. 4 and 5 show the different electrochemical behavior as observed during the run of the experiments.

To establish a reasonable concept of what happened to potential with time and the stability of the ore in the test solutions, a plot of potentials against time was carried out. The concentration of 0.15M and 0.35M showed almost similar trends. The solution of 0.15M showed a higher potential decrease from 2V to less than 0V, where it constantly increased from 0V to 1.55V and again decreased constantly from 1.55 V to less than 0V and was inconsistently fluctuated with time. Similar behavior was observed for 0.35M concentration as potential decreased from 1.35 V to -0.2V, increased to 0.1 V and further decreased to -0.2V and started leaching there at less than zero potential with time. This behavior suggested that there was a breakdown in the passive films on the surface of the ore pellet before the ore leached and break passivity trend at a less than zero voltage. As reported by Olubambi [11] it took zero fewer volts to break a film on the ore surface for 0.25M where it passivated at exactly 0 V and remained stabilized throughout the test without undergoing further leaching. The fluctuating trends shown by the three slopes suggest the possibility of potential increase with time to overcome film passivity. However, the ore become a bit stable as it leached and repassivated.

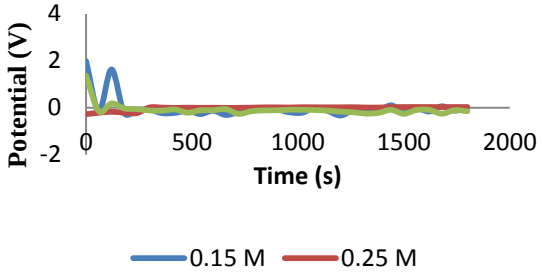

Fig. 4 Potential against time for potentiometric method

Fig. 5 shows the change in solution pH with time during the potentiometric tests for different concentrations. A decreasing pH trend with time was observed as the concentration of the lixiviate decreases. The highest concentration (0.35M) showed the highest pH decrease with time followed by 0.25M and 0.15M. From the literature, the progressive decrease in solution pH could have resulted from the iron hydrolysis, the consequent precipitation of ferric compounds, and the oxidation of sulphur [9,10]. Similar pH was observed for all the concentrations at time 120 seconds. This reason for this behavior is yet to be known.

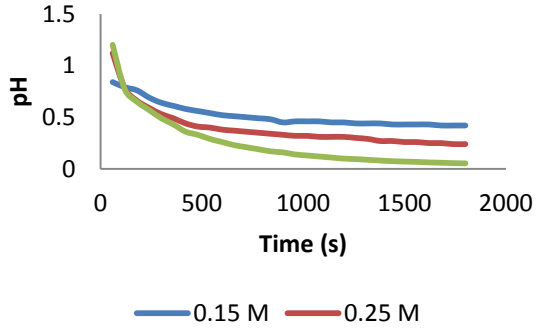

Fig. 5 pH against Time for potentiometric method

G. AAS results of leached Co and Cu after potentiometric

Table IV shows the results of the percentage Cu and Co leached during the potentiometric tests. A concentration of 0.25M gave the highest copper leached (24.64%) followed by 0.35M with 22.37% and lastly 0.15M with 10.74%. For Co, a concentration of 0.25M and 0.35M almost gave the same percentage leached as 14.28% and 13.88% were leached, respectively. Concentration of 0.15M gave the least percentage leached, while concentration of 0.25M gave the highest concentration leached for both Cu and Co leaching. These suggested that a concentration of 0.25M was optimum in this case.

The AAS results obtained from the potentiometric test were compared yet with another study of Cu-Co ore containing 1.5% Co and 1.6% Cu from Katanga Province; the Republic of Congo leached using sulphuric acid [12]. The results obtained from the study revealed that without hydrogen peroxide, the maximum extraction of Cu and Co were found to be ~80% and ~15%, respectively when the acid concentration was varied between 0.36 – 1.1M [11]. However, by analyzing and comparing the leaching results of the above mentioned study to the current study it was found that from 1.5% - ~80% Co and 1.6% - ~15% Cu were leached. Contrary to the above mentioned authors, 3.32% - ~14.28% Co and 0.403% - ~24.64% Cu were leached in this current study. This different in the leaching results could be due to different concentration composition of individual mineral in different ores bodies or due to the degree of film passivity behavior of individual minerals during leaching.

TABLE IV
PERCENTAGE LEACHED OF CU-CO AT VARIED PH FROM POTENTIOMETRIC TESTS

pH	% leached	
	Cu	Co
0.15	10.74	6.06
0.25	24.64	14.28
0.35	22.37	13.88

H. Pourbaix representation of the electrochemical behavior of Copper-Cobalt ore in sulphuric acid

Pourbaix diagrams represent the stability of a metal as a function of potential and pH. These diagrams are constructed from calculations based on Nernst equations and solubility data for metal and its species. At a particular combination of pH and potential, a stable phase can be determined from the Pourbaix diagram. In such diagrams, the redox potential of the leached system is plotted on a vertical axis and the pH on a horizontal axis. Three regions are identified on the pourbaix diagram that is the immunity (precipitation) zone where leaching does not occur at all, leaching zone and passivity zone. By altering the pH and potential to the regions of immunity and passivation, leaching can be controlled. Each line of a Pourbaix diagram represents conditions of thermodynamic equilibrium for some reaction. Fig. 6-11 presents Pourbaix diagrams for Co and Cu at different sulphuric acid concentrations of 0.15M, 0.25M and 0.35M relative to their change in pH and potential effects. All the diagrams were presented at normal thermodynamical conditions i.e. 25°C, and 1atms.

In general all six Pourbaix diagram are divided into three sections the lower part as the immunity zone, middle section as the leaching zone and the upper part the passivity zone. The green dotted lines in the leaching zone represent the stability of water in relation with the Co^{2+} and $CoSO_4$ in aqueous solution (Fig, 6-8). Concentrations of Co^{2+} in solution were different in each sulphuric acid molarity (0.15-0.35M). At 0.15M, Co^{2+} was stable in two parts of the leaching zone area. First stability can be seen between pH of 0.42 and 0.49 with a corresponding potentials of -0.5-1.99 V followed by the second stability at pH of 0.47-0.84 with corresponding potentials of -0.5-0.4 V. $CuSO_4$ is leachable from a pH of 0.47 with a corresponding potentials of 0.4-1.99 mV where it started to passivate from a potential of 2V upwards at a pH of 0.47 in CoO_2(c) form. Consequently if applied potential of less than -0.5 is induced, $CuSO_4$ will precipitate to give Co(c). Similarly the same behaviors were true for 0.25M and 0.35M.

At 0.25M the first part of stability was between pH of 0.24-0.1 which correspond to potential of -0.5 to1.99 V followed by a 0.24-1.12 pH and at -0.5to 0.47 V. $CuSO_4$ was also present at the range of 0.47-1.99 V where it started to passivity and similarly precipitating at less than -0.5 V. Similar remarks could be made for 0.35M.

Fig. 6 Eh-pH Co diagram (25◦c) at 0.15M Sulphuric acid

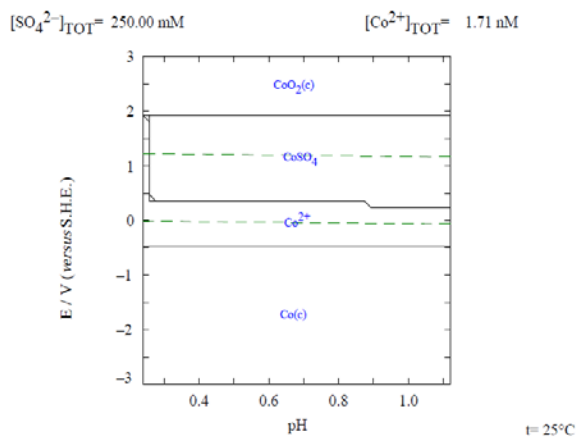

Fig. 7 Eh-pH Co diagram (25◦c) at 0.25M Sulphuric acid

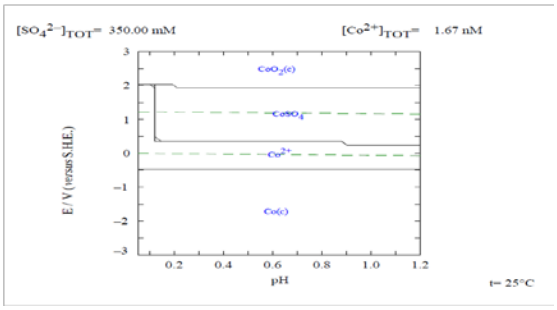

Fig. 8 Eh-pH Co diagram (25°C) at 0.35M Sulphuric acid

The CuHS is stable in 0.25 M solution from a pH of 0.42 to 1 and potential of -0.51 to 0.3 V where it drastically undergoes passivity at 0.4 V as CuS(c). $CuSO_4$ was present in solution at pH of 0.51 and potential of 2.2V where it then dissolute as Cu^{2+} above a 2.2V. The Cu^{2+} was stable in two part of the leaching zone area. Firstly stability can be seen between pH of 0.4-0.52 with corresponding potential of 0.4-2 V, followed by second stability at a pH 0.52-1 with corresponding potential of 2.2-3 V. Again if the applied potential of less than -0.4 is induced on $CuSO_4$, Cu(c) will be formed.

At 0.25M a more similarly behaviour is noticed where CuHS was stable in solution with a pH of 0.2-2.2 and corresponds with potential of -0.4-0.3 V where it started passivating at 0.3 to 0.4 V as CuS(c). $CuSo_4$ was leacheable at

0.31-2.2 pH and 2.2V where it further dissolved as Cu^{2+}.The Cu^{2+} was stable in two part of the leaching zone area. Firstly stability was experiences between pH of 0.2-0.31 with corresponding potential of 0.4-2.2V and then followed by second stability at a pH 0.31-2.2 with corresponding potential of 2.2-3V from 2.2V upwards. Inducing the applied potential of less than -4V on $CuSO_4$ resulted toCu(c).

In 0.35M, Cu(c) precipitated at less than -1.4V. CuHs was stable at 0.2-2.2 pH and precipitated as CuS(c) at 0.3-0.4V with corresponding pH of 0.2-2.2. $CuSO_4$ was leachable at 0.2-2.2 pH and 0.4-2.2 V and dissolved as Cu^{2+} from 2.2-3V upwards.

Fig. 9 Eh-pH Cu diagram (25°C) at 0.15M Sulphuric acid

Fig. 10 Eh-pH Cu diagram (25°C) at 0.25M Sulphuric acid

Fig. 11: Eh-pH Cu diagram (25°C) at 0.15M Sulphuric acid

The comparison of Eh vs pH for this current study was compared with the work done by Seo et al [11] and the dissolution of Co and Cu was at a pH between 0.054 and 1.2 for all concentration at which the tests were carried out.

The current study shows reasonable lower Co^{2+}, $CoSO_4$ in solution as there was no reductant to stimulate the dissolution. This also can be said for Cu^{2+} and $CuSO_4$.

IV. Conclusions

The following conclusions were drawn based on the results obtained: XRF results showed that the ore had 3.32% Co oxide and 0.403% Cu oxides, however; it was highly concentrated by silica oxide. The linear potentiodynamic polarization measurement at pH of 2.5 resulted in high percentage of Co and Cu leached at lower current density and lower applied potential as compared to other respective pH. The effectiveness of leaching at lower sulphuric acidic pH of between 1.8 and 3 was also confirmed by other studies that have been done before. At 2.5 pH polarization behaviour of the ore suggested stronger ability to overcome film passivity during leaching.

As pH decreased with time, concentration of the solution decreased. The AAS results showed that at 0.25M concentration highest leaching was achieved for both Co and Cu followed by a concentration of 0.35M and 0.15M, respectively. Pourbaix diagrams for Cu/Co at different sulphuric acid concentrations of 0.15M, 0.25M and 0.35M relative to their change in pH and potential effects were constructed to show different regional leaching predictions zone. The comparison of Eh vs pH for this current study showed that to obtain maximum recovery during leaching of u-Co using sulphuric acid, pH should be maintained at pH<1.

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