Investigation of Affecting Parameters on Heap Leaching Performance and Reducing Acid Consumption of Low Grade Oxide-Sulfide Copper Ore

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Abstract: Heap leaching method is known as cheap method for copper extraction. Presence of clay and acid consumer waste minerals are the most critical issues because they may bring out the process from economic mode. For reduce acid consumption and increasing copper extraction in heap leaching method, about 3 tons sample was taken randomly from different low grade zones. After homogenization and spelling into small parts, bottle roll tests were carried out to achieve maximum copper recovery and acid consumption in a short time at pH= 1, 1.1 and 1.2. Maximum copper recovery were 60.8, 60.2 and 60.1%, respectively. Also acid consumption were 107.30, 99.90 and 93.23 kg/t of ore, respectively. After diagnostic tests, nine columns with dimension 2m height and 15cm diameter were filled by 0.5, 1 and 1.5 inches ore. Columns were irrigated with 7, 9 and 11 g/l as acid solution concentration at flow rates of 7, 11 and 15 l/m²h for 63 days. Results showed that there are high correlation between copper and iron recoveries. Copper recovery changed from 25.5% to 58.9% while iron recovery changed from 4.7% to 15.2%. Acid consumption were achieved between 18 to 54 kg per ton of ore. Acid solution concentration and irrigation flow rate are the most important parameters on the copper and iron recoveries. By increasing of leaching period Copper extraction will increase, but acid will be waste more. Copper and iron recoveries were increased with increasing the acid solution concentration and irrigation flow rate but increasing in amount of acid consumption does not increase copper extraction inevitably. Irrigation flow rate of acid solution has significant effect on acid consumption. With decreasing the ore particle size, both of copper recovery and acid consumption were increased.

Keywords: Copper Oxide Heap Leaching, Bottle Rolling Test, Column Tests, Copper and Iron Recoveries, Acid Sulfuric Consumption

1. Introduction

Sarcheshmeh copper complex is located in 160 kilometers south-west of Kerman and 50 kilometers south of Rafsanjan and the central area of the Zagros Mountains [1]. Copper production at this complex is done by pyro-metallurgy and hydrometallurgy methods [2]. Following decline of high-grade copper reserves and increasing global demand of copper metal caused that hydrometallurgical and heap leaching method has been designed and used. These method, in addition to being less pollution to the environment is included, provides the extraction of copper from low grade oxide and sulfide ores [3]. Heap leaching has been demonstrated as a viable and low cost approach to open-pit mining operations with low-grade and complex run of mine (ROM) ores, agglomerated flotation tailings and also for the treatment of coarse rejects from semi-autogenous grinding (SAG) circuits [4]. Modern heap leaching might be defined as a controlled process whereby ore is stacked in thin lifts (under 10m in most cases), usually crushed and often agglomerated, on a carefully prepared containment system and irrigated in a controlled manner with a solvent to extract the optimum amount of the target mineral [5]. Low capital
and operating costs and simple atmospheric leach process make heap leaching suitable for low grade ores and small deposits. Disadvantages include low recoveries, long ramp-up times, large footprint and acid-mine drainage of wastes [6]. Ore and gangue mineralogy is probably the most important parameter affecting the operating costs and recoveries [7]. Heap leaching issues are classified in three categories: 1) Mineralogy handling include: crushing, agglomeration, heaping and heap configuration, 2) Solution handling include: irrigation configuration, solution application, irrigation media and rates, forms of solution collection, water consumption, 3) Operating aspects such as drag of fines, flooding and solution channeling, heap erosion, air injection and others [8]. Factors that may make ores unsuitable for heap leaching are poor percolation due to the presence of swelling clays, and high gangue acid consumption [6]. Percolation can be improved by agglomerating with acid binder, and by minimizing ore compaction during stacking heap lifts [9]. Silicate and iron oxide gangue minerals such as limonite consume sulfuric acid during initial reaction with acid [10]. Increasing acid concentration in solution caused increasing the acid consumption and consequently dissolve gangue minerals without significant effect on copper extraction [11]. The economics of heap leaching are strongly governed by the trade-off between the slow rate and limited extent of leaching from large particles and the cost of crushing finer [12].

Most affecting parameters on copper extraction in heap leaching method are ore and gangue mineralogy, size distribution, leaching time, percolation, irrigation flow rate, temperature and concentration that some of them are uncontrollable [13]. In this study effect of ore size distribution, acid concentration in solution and irrigation flow rate of solution has been investigated on copper extraction and acid consumption in column and bottle roll tests for Sarcheshmeh copper mine low grade oxide-sulfide ore.

2. Material and Methods

2.1. Sample Preparation and Analyses

For metallurgical test works, approximately 3 tons ore was taken from low grade zones of Sarcheshmeh copper mine for preparing representative sample. After homogenizing and splitting into smaller parts, 3 parts about 300 kg were selected randomly and crushed below than 1.5, 1 and 0.5 inches. Then some samples from 0.5 inch fraction were taken and sent to XRF, XRD and mineralogical analyses. Table 1 shows result of XRF analyses that analyzed by XRF Philips PW 1480 instrument.

Table 1. XRF analysis.

| Element/compound | Cu | Cu₂⁺ | Fe | S | Mo | Al₂O₃ | SiO₂ |
|------------------|----|------|----|---|----|-------|------|
| Content (%)      | 0.83 | 0.51 | 6.25 | 3.90 | 0.002 | 16.90 | 55.36 |

Mineralogy analysis showed that chalcopyrite is the major phase of the copper sulfide mineral and chalcocite is the minor phase. Also, other minerals such as pyrite, limonite, hematite and magnetite were distinguished in the representative sample. It was determined that 0.51 percent of copper is presented in oxide minerals. XRD analysis is presented in Table 2.

Table 2. Quantitative XRD analysis.

| Phases          | Clinochlore | Muscovite | Quartz | Pyrite | Albite |
|-----------------|-------------|-----------|--------|--------|--------|
| Content (%)     | 17          | 40        | 24     | 8      | 12     |

Chalcopyrite, chalcocite and pyrite were identified in polished sections images. The polished section of sample are shown in Figure 1.

Figure 1. Polished section of ore sample, blue: chalcocite, golden: chalcopyrite, yellow: pyrite.

2.2. Bottle Roll Tests

Bottle roll tests were conducted on a bottle roller apparatus under procedure that sowed in table 2. These experiments provide initial information for copper extraction and acid consumption in a short time [14]. Acid consumption are normally overestimated in rolling bottles, hence the acid consumption results should be considered as semi-quantitative [6]. Bottle roll tests were carried out at 7 days in 3 various pH (pH= 1.2, 1.1 and 1). One of tests was repeated.
to ensure the accuracy. The parameters and conditions for bottle roll tests are presented in Table 3.

Table 3. Parameters and conditions for bottle roll tests.

| Test number | Solid weight (gr) | Solution volume (cc) | Total volume (cc) | Rotational speed (RPM) | pH  |
|-------------|------------------|----------------------|-------------------|------------------------|-----|
| 1           | 105              | 700                  | 735               | 60                     | 1.2 |
| 2           | 105              | 700                  | 738               | 60                     | 1.1 |
| 3           | 105              | 700                  | 737               | 60                     | 1   |
| 4           | 105              | 700                  | 736               | 60                     | 1.2 |

Bottles’ pH were measured every day and were fixed at the specified value by adding acid. Addition of acid were continued until the pH was fixed at the specified value. Because pH was remained constant during the test, the amount of acid consumed is equivalent to acid added. Copper and iron recoveries were calculated by PLS analysis that has been done by Atomic Absorption Spectroscopy.

2.3. Column Tests

Figure 2. Ore size distribution in columns.

Table 4. The conditions of nine column tests.

| Column Number | Ore weight (kg) | Max particle size (inch) | Acid concentration (g/l) | Irrigation rate l/(h·m²) | mm/min |
|---------------|-----------------|----------------------------|--------------------------|--------------------------|--------|
| A1            | 56              | 0.5                        | 7                        | 7                        | 1.75   |
| A2            | 53              | 0.5                        | 9                        | 11                       | 2.75   |
| A3            | 54              | 0.5                        | 11                       | 15                       | 3.75   |
| A4            | 54              | 1                          | 7                        | 11                       | 2.75   |
| A5            | 57              | 1                          | 9                        | 15                       | 3.75   |
| A6            | 57.5            | 1                          | 7                        | 7                        | 1.75   |
| A7            | 62              | 1.5                        | 7                        | 15                       | 3.75   |
| A8            | 60              | 1.5                        | 9                        | 7                        | 1.75   |
| A9            | 60              | 1.5                        | 11                       | 11                       | 2.75   |

Column leach tests are the most important part of the metallurgical test work program. The two key results can be obtained from the column tests which are the maximum copper extraction and acid consumption under percolation-contact mode [15]. Leaching tests were carried out using nine columns with dimension 2m height and 15cm diameter in open-cycle. Column height must be bigger than 10 times of the column diameter and it must be bigger than five times of largest particle diameter. These ratios are considered for remove channeling flow in column [16]. Literature review showed that ore size distribution, acid concentration and irrigation flow rate are the most controllable parameters on copper heap leaching. After diagnostic tests, nine columns were filled by 0.5, 1 and 1.5 inches ore. Figure 2 shows ore size distribution in columns.

Columns were irrigated with 7, 9 and 11 g/L as acid solution concentration at flow rates of 7, 11 and 15 L/m²·h for 63 days. The conditions of nine column tests are presented in Table 4 and figure 3 shows schematic column heap leaching test.

3. Results and Discussion

3.1. Bottle Roll Tests

Results showed acid consumption was changed from 92.23
to 107.3 kg per ton of ore and maximum copper recovery was 60.8% for total copper. The residuals for bottle roll tests and their results are presented in Table 5.

| Number | pH  | Solution Volume (cc) | Solid Weight (gr) | Cu Recovery (%) | Fe Recovery (%) | Acid consumption (cc) | Acid consumption (kg/t) |
|--------|-----|----------------------|-------------------|-----------------|-----------------|-----------------------|------------------------|
| 1      | 1.2 | 716                  | 101.0             | 60.10           | 6.95            | 7.6                   | 107.30                 |
| 2      | 1.1 | 728                  | 99.4              | 60.20           | 7.63            | 8.1                   | 99.90                  |
| 3      | 1   | 725                  | 100.0             | 60.80           | 8.85            | 8.7                   | 93.23                  |
| 4      | 1.2 | 728                  | 100.6             | 60.15           | 7.15            | 7.5                   | 92.50                  |

The acid consumption was reduced over time. By advancement of minerals dissolution, the less acid is required to dissolve the remaining minerals and some acid is produced during the reactions. Reactions 1 and 2 shows acid production in dissolution copper minerals [17].

\[ \text{Cu}_2\text{S} + 2\text{Fe}(\text{SO}_4)_3 \rightarrow 2\text{CuSO}_4 + 4\text{FeSO}_4 + \text{S}^+ \]  

\[ \text{S}^+ + 1.5\text{O}_2 + \text{H}_2\text{O} \rightarrow \text{H}_2\text{SO}_4 \]  

The acid consumption was reduced over time. By advancement of minerals dissolution, the less acid is required to dissolve the remaining minerals and some acid is produced during the reactions. Reactions 1 and 2 shows acid production in dissolution copper minerals [17].

The generation of acid in bottles and the reduction of soluble minerals by reaction advancement caused to reduce acid consumption over time. Figure 4 shows the consumption of acid for each bottle.

**Figure 4.** Acid consumption in bottle roll tests.

3.2. Column Tests

3.2.1. Copper Recovery

Column tests results showed copper recovery changed from 25.5% to 58.9%. Monitoring the daily copper concentration in PLS showed column number A6 with \( 7\text{L}/\text{m}^2 \times \text{h} \) irrigation flow rate and \( 11 \text{g}/\text{L} \) acid concentration in solution has maximum copper concentration in PLS and column A7 has minimum concentration of copper by \( 15 \text{L}/\text{m}^2 \times \text{h} \) irrigation flow rate and \( 7 \text{g}/\text{L} \) acid concentration in solution. Maximum Copper recovery was obtained at \( 11 \text{g}/\text{L} \) acid concentration, \( 15\text{L}/\text{m}^2 \times \text{h} \) irrigation flow rate and ore particle size below than 0.5 inch. Cumulative copper recovery in columns are shown in Figure 5.

Study of table 5 and compare it with table 3 shows that increasing of irrigation flow rate and acid concentration in solution, caused increasing copper and iron recovery. Also reducing of ore size distribution has same effect on copper and iron recovery that caused for increasing contact surface between ore and solution. There is no significant correlation between copper recovery and volume of irrigation solution. For example columns number A3 and A5 by irrigation same volume of solution (approximately 282 Liters) has 58.9% and 48.8% copper recovery respectively and columns number A2 and A4 by irrigation same volume of solution (approximately 195 liters) has 45.8% and 30.9% copper recovery respectively.

**Figure 5.** Cumulative total copper recovery in columns.

3.2.2. Iron Recovery

Presence of iron sulfate in solution increased copper recovery in heap leaching of copper ores [18]. The dissolution reactions under atmospheric conditions can be written as equation 3 and 4 for Chalcocite and Covellite, respectively [19]. Furthermore, increase of iron dissolution waste acid in the process.

**Figure 6.** Cumulative iron recovery in columns.
\[ Cu_2S + 2Fe(SO_4)_3 \rightarrow 2CuSO_4 + 4FeSO_4 + S^\ell (3) \]
\[ CuS + Fe(SO_4)_3 \rightarrow CuSO_4 + 2FeSO_4 + S^\ell (4) \]

Results showed there are high correlation between copper and iron recoveries. Iron concentration was increased in PLS after 35 days treatment whereas iron recovery changed from 4.72% to 15.64%. Cumulative iron recovery in columns are demonstrated in Figure 6.

### 3.2.3. Acid Consumption

Acid consumption in copper heap leaching is related to gunge mineralogy more than other factors. Silicate and iron oxide gangue minerals such as limonite consume sulfuric acid during initial reactions. Limonite consumes acid by breakdown to ferric ions in solution [20]. Silicate minerals consume acid by breakdown to a wide range of soluble solution products. K-feldspar, Na-feldspar, Ca-plagioclase and Biotite breakdown to Ortho-silicic acid, \(H_4SiO_4\), and various metal cations such as \(Na^+,\ K^+,\ Na^{2+},\ Ca^{2+},\ Fe^{2+},\ Fe^{3+}\) and \(Mg^{2+}\) [21].

Acid consumption in bottle roll test is considered as semi quantitative. To achieve real acid consumption, column leaching tests are conducted. Maximum acid consumption in column leaching tests is about 40% of acid consumption in bottle roll tests [16]. The results of column test presented in Table 6.

Also it was defined there is no significant relation between acid consumption and copper recovery. Column number A1 by consumption 18.53 kilograms acid has 34% copper recovery whereas column number A8 by consumption 20.47 kilograms acid has 25.5% copper recovery.

The amount of unused acid in columns is calculated and illustrated in Figure 7. After 35 days acid concentration was increased in PLS and also returned acid has grown at this time.

### Table 6. The results about irrigation and acid consumption in column tests.

| Column number | copper recovery | Iron recovery | Volume of irrigated solution (L) | Acid consumption (kg/t) | Acid consumption (ton/ton copper) |
|---------------|-----------------|--------------|----------------------------------|------------------------|----------------------------------|
| A1            | 34              | 8.03         | 149                              | 18.62                  | 2.94                             |
| A2            | 45.8            | 8.49         | 191.4                            | 31.9                   | 4.40                             |
| A3            | 58.9            | 8.25         | 282.9                            | 54.56                  | 8.47                             |
| A4            | 30.9            | 6.17         | 195.5                            | 25.85                  | 4.81                             |
| A5            | 48.8            | 4.72         | 282.3                            | 47.05                  | 9.63                             |
| A6            | 41.7            | 15.64        | 148.7                            | 28.44                  | 3.72                             |
| A7            | 34.0            | 4.97         | 265.4                            | 29.96                  | 9.03                             |
| A8            | 25.5            | 6.83         | 136.5                            | 20.47                  | 3.36                             |
| A9            | 45.5            | 8.19         | 198.7                            | 36.42                  | 5.45                             |

Monitoring the daily copper concentration in PLS showed copper concentration is low in columns with high irrigation flow rates. In contrast, high copper concentration in PLS obtained by columns with low irrigation flow rates. It can be concluded that at low irrigation flow rate, contact time is sufficient between minerals and sulfuric acid, then reaction is completed. So, increasing the irrigation flow rate reduced copper concentration in PLS. But high irrigation flow rate increase PLS volume and copper recovery. Reducing the particle size increases the contact surface between minerals and sulfuric acid which results the increase of copper recovery.

Comparing the graphs of iron recovery and returned acid showed that after 35 days, reaction between sulfuric acid and copper minerals was decreased and sulfuric acid wasted. For control iron concentration in PLS and prevent acid wasting, irrigation algorithm should be optimized, acid concentration in solution should be decreases and irrigation flow rate must be increase.

The correlation between copper and iron recoveries are shown in Figure 8. There are direct relationship between copper and iron recoveries. One of reasons could be probability dissolution of secondary copper minerals which have copper and iron. Also, there is no meaningful relation between copper and iron recoveries and acid consumption.
4. Conclusion

1. The results of bottle roll and column leaching tests showed that acid consumption was high initially, then reduced and finally fixed at constant value.
2. Column test results conducted under various condition, there was high correlation between copper and iron recoveries.
3. Detailed study of copper, iron and acid concentration in PLS showed copper recovery was high initially and reduced over time. But iron recovery and returned acid were contrast. That means, their concentrations were low initially and then increased. Therefore, optimizing the irrigation algorithm resulted substantially reduction of iron concentration in PLS and prevented acid waste.
4. Increasing the concentration and irrigation flow rate had a positive effect on the recovery of copper. Reduction of size distribution increased copper and iron recoveries. In high irrigation flow rate, copper concentration in PLS decreased, but recovery increased for high volume of PLS.
5. Irrigation flow rate and acid concentration in solution had the most positive effects on acid consumption, respectively. Acid consumption reduced substantially by optimizing the acid irrigation algorithm.
6. Increasing of leaching period increases copper extraction, but acid being waste more.
7. Diagram of copper and iron recoveries and acid consumption did not show a significant relationship between them. This means that increase in acid consumption does not increase copper recovery.

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