Chapter 5
Extraction of Copper Oxide (I): Purified $CuSO_4$ Solution

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5.1 Introduction

Heating and melting are frequently used to extract copper (Cu) from its parent ores [1–4]. Roasting, smelting, and conversion are the three phases of the heating and melting process for primary copper ores [5–11]. Significant amounts of copper are lost as dust in the first and second phases; this dust is referred to as waste copper dust (WCD). According to the research done by Gorai, Jana, and Khan [12], for every 1000 tons of Cu concentrate treated, over 70–100 tons are lost as WCD.

According to reports on the mineralogy of WCD by Balladares et al. [13], Wang et al. [14], Jaroková et al. [15], and Okanigbe et al. [16], the considerable Cu content of WCD was deduced by Gorai et al. [12]. Additionally, according to these mineralogical investigations, WCD is a complex low-grade copper ore in which the copper content frequently occurs in conjunction with other metals including iron (Fe), zinc (Zn), lead (Pb), silicon (Si), aluminum (Al), and others. Lacking a reliable waste management method, disposing of WCD in landfills defies the purpose of protecting the planet's mineral resources [17].

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According to Morales et al. [18], a hydrometallurgical process that operates at room temperature, atmospheric pressure, dilute reagent, few unit operations, and low energy cost should be the best waste management technique for the treatment of WCD. However, using hydrometallurgy alone presents difficulties due to an extremely small pH window, leading to the following:

1. Side reactions by contaminants that reduce leaching efficiency.
2. Contaminants co-exist in pregnant leach solution.
3. High chances of contaminants carry over to the extraction stage.
4. Increase in the number of cycles required to achieve 99.9% pure copper electrolyte.
5. Increase in the cost of recovery.
6. Increase in the use of toxic extractants deleterious to the environment.

Three steps make up hydrometallurgy: leaching, solvent extraction, and electrowinning. Sulfuric acid (H_2SO_4) is frequently utilized as the leaching agent during the leaching stage. In comparison to hydrochloric and nitric acid, Teir et al. [19] and Habashi [20] indicated that (H_2SO_4) is the most effective and often used acid for leaching oxides. H_2SO_4 is a low-cost leachant that is also frequently used to treat metallurgical wastes that contain copper [21]. The choice of H_2SO_4 as reagent for the hydrometallurgical treatment of WCD is established by its usage as a reagent for the conversion step, i.e., electrolysis to create copper cathode [22].

The inability of H_2SO_4 to prevent iron from dissolving from copper ore, however, poses a selectivity difficulty that reduces the likelihood of achieving pure Cu electrolyte at a low cost. As a result, the PLS's solvent extraction (SX) stage was added to decrease and eliminate iron [23, 24].

Emulsification problems exist for SX because they lead to extractant loss and electrolyte contamination. Additionally, iron impurity induces entrainment and crud development in the SX process, according to Chen et al. [25]. In addition, SX is extremely sensitive to low temperatures and low-grade solutions. Expensive organic reagents that are hazardous to the environment further complicate the process. Because iron is involved in a parasitic reaction that causes electricity to be diverted from plating of copper as it is oxidized from ferrous to ferric at the anode and reduced from ferric to ferrous at the cathode, a high concentration of iron in SX-treated PLS affects current efficiency during electrowinning [26].

Even though the dosage of H_2SO_4 is frequently the key economic component in the process of leaching copper in its oxide form from this ore, copper oxide ore dissolves in H_2SO_4 solution at room temperature. Researchers have also identified other factors, such as acid content, leaching period, temperature, pulp stirring, solid-to-solution ratio, etc., that can affect leach rates during recovery [27, 28]. Pulp stirring has been shown as a crucial process parameter for efficient leaching, according to Ghosh and Ray [29].

The results of comparing digital hotplate leaching with oven leaching will be presented in this chapter first, based on the discussion that has led up to this point. Second, it will be discussed how altering the compositional proportion of H_2SO_4:$FeSO_4.7H_2O$ can prevent iron from dissolving into pregnant leach solution (PLS).

5.2 Materials and Methods

5.2.1 Material

The study's primary source of material is the WCD from South Africa.

5.2.2 Methods

5.2.2.1 Sampling Using Rotary Splitter

Since it creates the least variance between samples and can produce a greater number of samples in one operation, the rotary splitter was employed to separate WCD into representative samples. A vibratory feeder was fed 4000 g of the WCD from a feed hopper (Fig. 5.1). As depicted in Fig. 5.1, the feeder evenly disperses the material into a succession of collectors on a spinning table.

Before the feed hopper is empty, the turntable speed was adjusted so that each sample collector would pass several times beneath the feeder. The revolving speed was made slow enough to prevent the edge of the sample collectors from touching the falling particles, which would cause them to leap into a different container or fling them out of the machine entirely. This was done in order to obtain consistent and dependable findings. To attain a WCD mass of 250 g in each sample collector, the speed was optimized, and the rotation switch was set at 17 rpm.

5.2.2.2 Sampling Using the Coning and Quartering Method

WCD was first poured over a level surface until it assumed a cone-like shape. The coned WCD was then flattened into a cake and cut into four equal pieces (A, B, C, and D), as shown in Fig. 5.2. The two pieces that are directly across from one

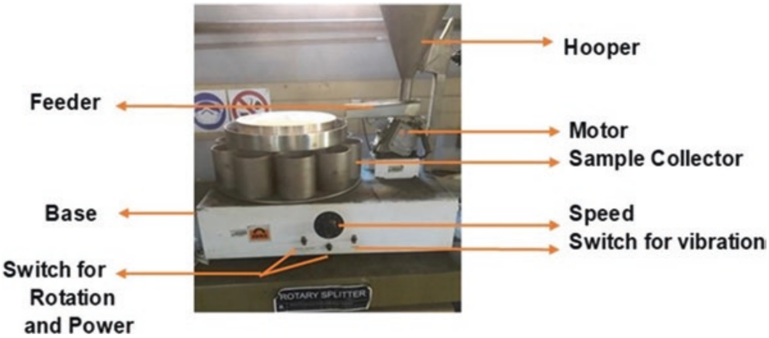


Fig. 5.1 Rotary splitter sampling

Fig. 5.2 WCD coning and quartering sampling

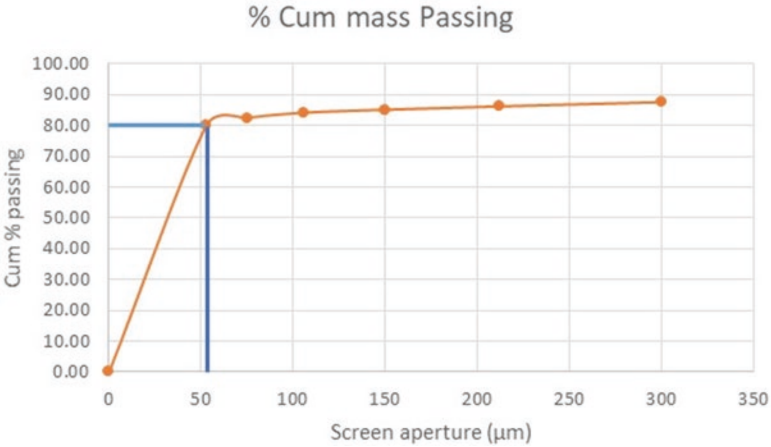


Fig. 5.3 Graphical representation of WCD's PSD

another are removed, for example, A and C (Fig. 5.2), while the remaining two (B and D) are joined to create the reduced sample. The same procedure was carried out four times until a suitable sample size of 4 g of 60 WCD remained.

These aliquot WCD samples of 4 g each were used for subsequent test works like characterization and leaching exercises.

5.2.2.3 Particle Size Distribution (PSD) of As-Received WCD

Following screening tests on the WCD sample, Fig. 5.3 displays the intended 80% mass passage of WCD at a −53 m screen size aperture.

5.2.2.4 Calculations for Preparing Sulfuric Acid Solution

The calculations were made using the following concentrations of 2 M, 4 M, 6 M, 8 M, and 10 M:

Molarity (M) = mol/L
Density (D) = 1.84 g/mL
Molar mass (MM) = 98.079 g/mol

For 2 M sulfuric solution Molarity was calculated using Eq. (5.1)

$$\begin{aligned} M &= D \times MM \\ &= \frac{1.84\text{g}}{\text{mL}} \times \frac{1000\text{mL}}{1\text{L}} \times \frac{1\text{mol}}{98.1\text{g}} \\ &= \frac{(1.84 \times 1000)\text{mol}}{98.1\text{L}} \\ &= \frac{1840\text{mol}}{98.1\text{L}} \\ &= 18.75\text{mol}/\text{L} \end{aligned} \tag{5.1}$$

We shall start with 2.5 L of roughly 18.8 M H_2SO_4 in this experiment to recover copper from the WCD; hence, the amount of water needed to make the 2 M diluted H_2SO_4 acid solution will be determined from the dilution factor in Eq. (5.2) as follows:

$$\begin{aligned} M1V1 &= M2V2 \\ 18.8\text{M} \times 2.5\text{L} &= 2\text{M} \times V2 \\ V2 &= \frac{18.8\text{M} \times 2.5\text{L}}{2\text{M}} \\ V2 &= \frac{47\text{L}}{2} \end{aligned} \tag{5.2}$$

This assumes that in order to prepare the 2 M diluted H_2SO_4 acid solution, we require 23.5 times as much water as 18.8 M. Therefore, we would apply the algebraic expression given below (Eq. 5.3) to generate a 40 mL, 2 M diluted H_2SO_4 solution:

$$x + 23.5x = 40\,\text{mL} \tag{5.3}$$

where $23.5x$ = volume of water.

From Eq. (5.3)

$$\begin{aligned} 24.5x &= 40\text{mL} \\ x &= 1.63\text{mL} \end{aligned}$$

As a result, 1.63 mL of concentrated acid is needed, whereas 40 mL of water is needed to dilute it.

$$\begin{aligned} &= 1.63 \times 23.5 \\ &= 38.37\text{mL} \end{aligned}$$

Total volume according to Eq. (5.3)

$$1.63 + 38.37 = 40\,\text{mL}.$$

These calculations were repeated for 4 M, 6 M, 8 M, and 10 M.

5.2.2.5 Leaching Parameters

The experiment was conducted under certain circumstances, with the following points being stressed:

- The temperature was kept constant at 45 °C.
- The leaching ratio of 1:10.
- Time: 30, 60, and 90 minutes.
- Speed: 300 Rev/min.

5.2.2.6 Experimental Procedures for Sulfuric Acid Leaching

The subsequent experimental procedures were broken down into two parts. The leaching of WCD from ovens is one topic, while the leaching of WCD from hotplates is another. The following steps are detailed.

Experimental Procedure for Leaching of WCD Using Hotplate with Stirrer

1. Fill a 250 mL conical flask with 4 g of WCD.
2. Prepare a 2 M diluted H_2SO_4 acid solution by adding 1.63 mL of H_2SO_4 acid to a beaker using a pipette.
3. Fill the beaker with concentrated H_2SO_4 acid solution with 38.37 mL of pure water (i.e., 2 M dilute H_2SO_4 solution).
4. Place a stirring bar inside the conical flask containing the 4 g of WCD and this 2 M diluted H_2SO_4 solution.
5. Place a piece of aluminum foil over the opening of the conical flask containing the acid and WCD.
6. Repeat steps 1, 2, 3, and 4 in reverse order.
7. Place the two conical flasks holding acid and WCD that have been coated in aluminum foil on the magnetic stirrer that has a heater set to 45 °C and an agitation speed of 340 rpm.
8. Wait 30 minutes after using the conical flask and its contents.
9. Repeat steps 1 through 7 for 30, 60, and 90 minutes.
10. After these leaching periods, take the conical flask and its contents out of the oven.
11. Use filter paper to separate the leach solution from the waste.

12. Fill a sample with the leach solution, and then label it for quick identification.
13. Keep the contents of the sample bottle at room temperature.

These steps were repeated for 4 M, 6 M, 8 M, and 10 M.

Experimental Procedures for Leaching of WCD Using a Laboratory Oven

1. Fill a 250 mL conical flask with 4 g of WCD.
2. Prepare a 2 M diluted sulfuric acid solution by adding 1.63 mL of sulfuric acid to a beaker using a pipette.
3. Fill the beaker with concentrated H_2SO_4 acid solution with 38.37 mL of pure water (i.e., 2 M dilute H_2SO_4 solution).
4. Fill the conical flask containing the 4 g WCD with this 2 M diluted H_2SO_4 solution.
5. Place a piece of aluminum foil over the opening of the conical flask containing the acid and WCD.
6. Repeat steps 1, 2, 3, 4, and 5 twice.
7. Preheat the oven to 45 °C, and place the two aluminum-foil-covered conical flasks containing acid and WCD inside.
8. Wait 30 minutes after using the conical flask and its contents.
9. Repeat Steps 1 through 7 for 30, 60, and 90 minutes.
10. After these leaching durations, remove the conical flask and its contents from the oven.
11. Using filter paper, separate the leach solution from the waste.
12. Fill a sample container with the leach solution, and then label it for quick identification.
13. Keep the contents of the sample bottle at room temperature.

These steps were for 4 M, 6 M, 8 M, and 10 M.

5.2.2.7 Sample Filtration

In this investigation, gravity-driven filtration was used. As seen in Fig. 5.4, the PLS was allowed to fall through gravity from the cylinder into the conical flask.

5.2.2.8 Proposed Process Flow Diagram

An outline of the hydrometallurgical group projects at Tshwane University of Technology in Pretoria, South Africa, can be found in Fig. 5.5. The topic of this study is sulfuric acid leaching, with red arrows indicating the process flow (Fig. 5.5).

Fig. 5.4 Gravity-driven filtration process of copper recovery from WCD

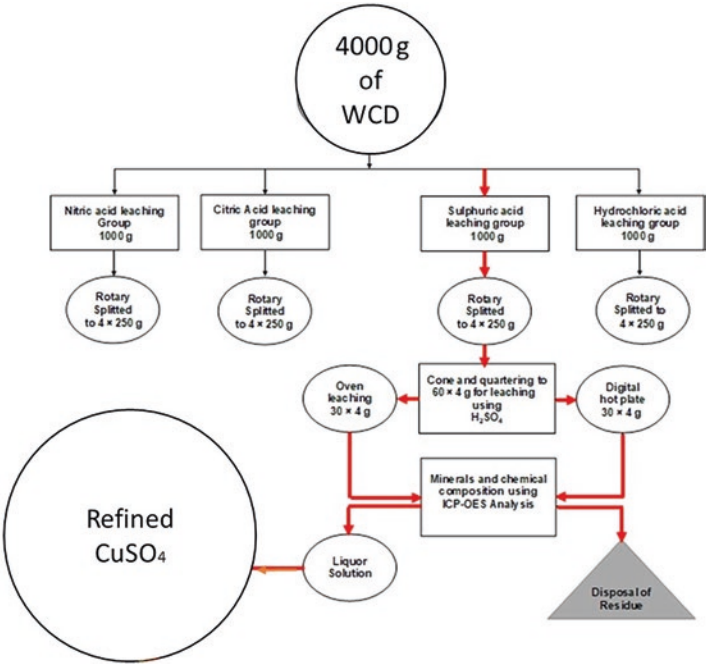


Fig. 5.5 Initial process flowsheet for Cu leaching from WCD

5.3 Results and Discussion

The recovery method for the copper value that was present in WCD was determined by its distinctive characteristics [16]. Consequently, hydrometallurgy was selected as an acceptable technique for its treatment. This investigation is restricted to the hydrometallurgical dissolution of copper from the WCD. The experiment will contrast agitation leaching with oven leaching techniques. This report offers commentary on accomplishments accomplished during the B-Tech period, with a particular emphasis on project test work carried out at Tshwane University of Technology in Pretoria, South Africa. The findings and discussion related to the following sub-heading are reported in this chapter:

1. Visual observation of as-received WCD
2. Visual observation of residue after leaching
3. Visual analysis of digital hotplate leachate
4. Visual analysis of oven leachate
5. Results on mass balance

5.3.1 Visual Observation of As-Received WCD

Before the test work was done, the WCD by-product sample was scrutinized. As seen in Fig. 5.6, the WCD was discovered to be a dark gray color. According to the WCD by-product XRF data, the ore mostly comprises copper, iron, and gypsum as impurities, along with other minerals. These metals and the impurities significantly influenced the ore's color. The sample felt generally powdery; however, the feel was uneven and parts of the particle were not very fine, as shown in Fig. 5.6. The head assay examination for the WCD as received reveals that the ore contains very fine particle, mostly in the -53 µm size fraction.

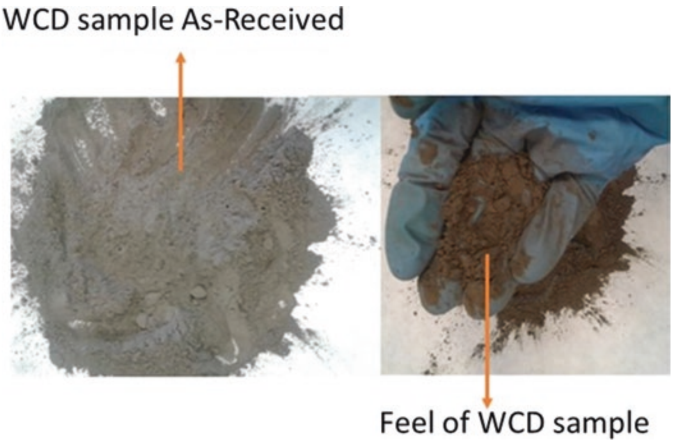
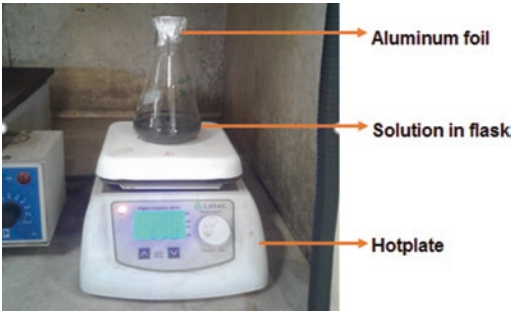


Fig. 5.6 Visual analysis for CSD by-product as received

Fig. 5.7 Digital hotplate leaching process



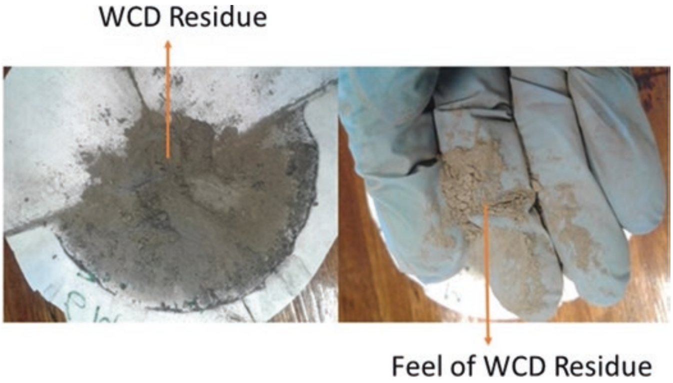


Fig. 5.8 Visual analysis of residue after leaching process

5.3.2 *Digital Hotplate Leaching Process of WCD*

The leaching process utilizing a digital hotplate is shown in Fig. 5.7. The leaching process may be impacted by the black mineral in the solution that was seen to be separating from and sticking to the conical flask above the solution.

5.3.3 *Visual Analysis of Residue After Leaching Process*

Following the leaching procedure, the solution was filtered to remove the solid particles from the liquid, and the leftovers were dried. The observation was successfully performed. The WCD residue had a more uniform texture and felt more powdery as depicted in Fig. 5.8. The light gray color of the WCD residue indicates that the majority of the metals responsible for the dark color of the WCD as received have been taken into the leach solution.

5.3.4 Visual Analysis Digital Hotplate Leachate

The difference in leachate color after the filtration process was clearly seen on digital hotplate leachate, as illustrated in Fig. 5.9. The solution was seen to be a dark blue color. Clearly, the majority of the copper metal was taken.

5.3.5 Visual Analysis on Oven Leachate

In addition, oven leachate was observed, as can be shown in the following Fig. 5.10. The leachate's pale blue color is evident in the results. The results almost certainly point to a sizeable number of copper metals that are still trapped in gangue particles.

5.3.6 Results on Mass Balance: Digital Hotplate

The comprehensive test work for digital hotplate leaching is detailed as follows.

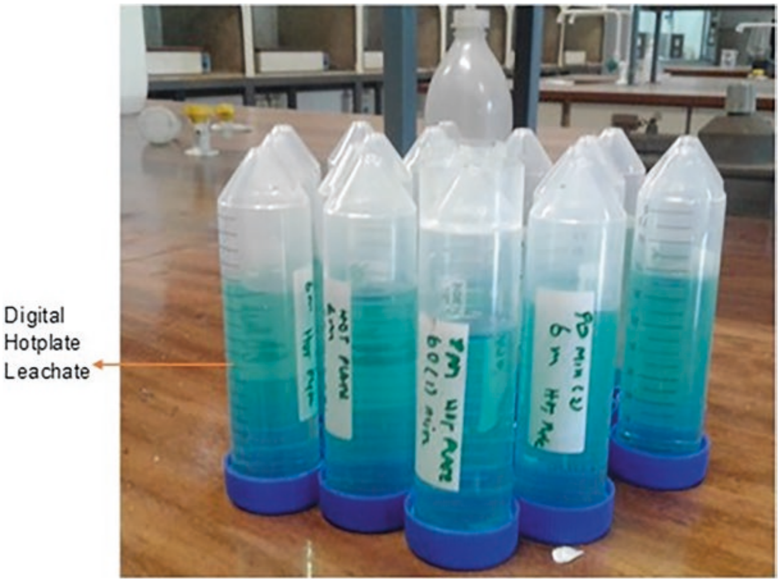


Fig. 5.9 Leachate from hotplate leaching

Fig. 5.10 Leachate from oven leaching

Table 5.1 Test 1 hotplate leaching results for 2 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.0140	1.6347	3.0590	38.3700
	60	4.0230	1.6329	2.7890	
	90	4.0330	1.6317	2.9305	
Total		12.0700		8.7785	

5.3.6.1 Test 1 Hotplate Leaching Results for 2 M H_2SO_4

According to the data in Table 5.1, leaching that took place in 60 minutes produced the least amount of residue when compared to leaching that took place in 30 and 90 minutes, whereas leaching that took place in 30 minutes produced the most mass. The total mass percentage of residues was 73%.

5.3.6.2 Test 2 Hotplate Leaching Results for 2 M H_2SO_4

According to the data in Table 5.2, leaching that was completed in 60 minutes produced the least amount of residue when compared to when the solution was leached for 30 and 90 minutes, while leaching that was completed in 90 minutes produced the most mass.

Overall, a mass percentage of 90% residue was obtained.

Table 5.2 Test 2 hotplate leaching results for 2 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0080	1.6332	3.5279	38.3700
	60	4.0090	1.6320	3.4492	
	90	4.0010	1.6330	3.8754	
Total		12.0180		10.853	

Table 5.3 Test 1 hotplate leaching results for 4 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.0100	3.1380	3.5600	36.8700
	60	4.0800	3.1400	3.7900	
	90	4.0600	3.1390	3.4500	
Total		12.1500		10.8000	

5.3.6.3 Test 1 Hotplate Leaching Results for 4 M H_2SO_4

According to the findings in Table 5.3, the leaching process that was carried out in 90 minutes produced the least quantity of residue as compared to when the solution was leached for 30 and 90 minutes. In comparison to 90 and 30 minutes, the leaching operation carried out in 60 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 89%.

5.3.6.4 Test 2 Hotplate Leaching Results for 4 M H_2SO_4

Compared to when the solution was leached for 30 and 90 minutes, the results in Table 5.4 demonstrate that the leaching process done in 30 minutes obtained the least quantity of residue after leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 90 minutes produced the highest mass. After leaching, residual mass percentage reached 90% overall.

5.3.6.5 Test 1 Hotplate Leaching Results for 6 M H_2SO_4

Compared to when the solution was leached for 30 and 60 minutes, the results in Table 5.5 demonstrate that the leaching process done in 90 minutes yielded the least quantity of residue after leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 60 minutes produced the highest mass. After leaching, a residual mass percentage of 91% was achieved.

Table 5.4 Test 2 hotplate leaching results for 4 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0200	3.1370	3.2900	36.8700
	60	4.0000	3.1400	3.6500	
	90	4.0100	3.1390	3.9200	
Total		12.0300		10.8600	

Table 5.5 Test 1 hotplate leaching results for 6 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.0100	4.5360	3.6000	35.4700
	60	4.0100	4.5300	3.8100	
	90	4.0100	4.5400	3.5500	
Total		12.0300		10.9600	

5.3.6.6 Test 2 Hotplate Leaching Results for 6 M H_2SO_4

The results in Table 5.6 obtained show that leaching process executed in 30 minutes obtained the least amount of residue after leaching compared when the solution was leached for 30 and 60 minutes. The leaching process conducted in 90 minutes achieved the highest mass compared to 60 and 30 minutes. The overall 92% mass percentage was obtained of residue after leaching.

5.3.7 *Results on Mass Balance: Oven*

5.3.7.1 Test 1 Oven Leaching Results for 2 M H_2SO_4

According to the results in Table 5.7, the solution was leached for 30 and 60 minutes, and the 60-minute method yielded the least quantity of residue after oven leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 30 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 87%.

5.3.7.2 Test 2 Oven Leaching Results for 2 M H_2SO_4

In comparison to leaching the solution for 30 and 60 minutes, the results in Table 5.8 reveal that leaching processes completed in 30 minutes yielded the least amount of residue after oven leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 90 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 86%.

Table 5.6 Test 2 hotplate leaching results for 6 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0000	4.5320	3.4600	35.4700
	60	4.0000	4.5430	3.6500	
	90	4.0200	4.5320	3.9200	
Total		12.0200		11.0300	

Table 5.7 Test 1 oven leaching results for 2 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.0061	1.6343	3.5649	38.3700
	60	4.0020	1.6305	3.3438	
	90	4.0041	1.6345	3.5164	
Total		12.0120		10.4250	

Table 5.8 Test 2 oven leaching results for 2 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0212	1.6308	3.2393	38.3700
	60	4.0027	1.6316	3.4590	
	90	4.0020	1.6349	3.6144	
Total		12.0260		10.3130	

5.3.7.3 Test 1 Oven Leaching Results for 4 M H_2SO_4

In comparison to leaching the solution for 30 and 60 minutes, the results in Table 5.9 reveal that leaching processes completed in 30 minutes yielded the least amount of residue after oven leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 90 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 89%.

5.3.7.4 Test 2 Oven Leaching Results for 4 M H_2SO_4

In comparison to leaching the solution for 30 and 60 minutes, the results in Table 5.10 reveal that leaching processes completed in 30 minutes yielded the least quantity of residue after oven leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 90 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 87%.

Table 5.9 Test 1 oven leaching results for 4 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.0100	3.1380	3.1300	36.8600
	60	4.0000	3.1360	3.6200	
	90	4.0000	3.1380	3.9300	
Total		12.0100		10.6800	

Table 5.10 Test 2 oven leaching results for 4 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.0000	3.3170	2.7500	36.8600
	60	4.0000	3.1380	3.7400	
	90	4.0000	3.1370	3.9600	
Total		12.0000		10.4500	

5.3.7.5 Test 1 Oven Leaching Results for 6 M H_2SO_4

According to the results in Table 5.11, the solution was leached for 30 and 60 minutes, and the 60-minute method yielded the least quantity of residue after oven leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 90 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 84%.

5.3.7.6 Test 2 Oven Leaching Results for 6 M H_2SO_4

The data shown in Table 5.12 indicate that when the solution was leached for 90 minutes as opposed to 30 or 60 minutes, there was the least quantity of residue left over.

In comparison to 60 and 30 minutes, the leaching operation carried out in 60 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 87%.

5.3.7.7 Test 1 Oven Leaching Results for 8 M H_2SO_4

The results in Table 5.13 demonstrate that, when compared to leaching the solution for 30 and 60 minutes, leaching the solution for 30 minutes yielded the least quantity of residue following oven leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 90 minutes produced the highest mass. After leaching, a residual mass percentage of 85% was achieved.

Table 5.11 Test 1 oven leaching results for 6 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.0100	4.5320	3.4640	36.8600
	60	4.0100	4.5350	2.8200	
	90	4.0000	4.5320	3.8620	
Total		12.0200		10.1460	

Table 5.12 Test 2 oven leaching results for 6 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0000	4.5360	3.8500	35.0000
	60	4.0100	4.5340	3.4400	
	90	4.0100	4.5350	3.3400	
Total		12.0200		10.6300	

Table 5.13 Test 1 oven leaching results for 8 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
1	30	4.010	5.8150	3.2900	34.1800
	60	4.000	5.8130	3.5500	
	90	4.010	5.8150	3.4100	
Total		12.0200		10.2500	

5.3.7.8 Test 2 Oven Leaching Results for 8 M H_2SO_4

When compared to leaching the solution for 30 and 60 minutes, the results in Table 5.14 reveal that a 90-minute leaching method yielded the least quantity of residue after oven leaching. In comparison to 60 and 30 minutes, the leaching operation carried out in 60 minutes produced the highest mass. After leaching, residue was collected in an overall mass percentage of 95%.

5.3.7.9 Test 1 Oven Leaching Results for 10 M H_2SO_4

According to the data in Table 5.15, the leaching process that was carried out in 90 minutes yielded the least quantity of residue following oven leaching as compared to when the solution was leached for 60 and 90 minutes. Comparing 60- and 30-minute leaching processes, the 60-minute method produced the highest mass. After leaching, residue had a mass percentage of 91% overall.

Table 5.14 Test 2 oven leaching results for 8 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0100	5.8130	3.9000	34.1800
	60	4.0000	5.8140	3.9100	
	90	4.0200	5.8130	3.6400	
Total		12.0300		11.4500	

Table 5.15 Test 1 oven leaching results for 10 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0100	7.0240	3.6000	32.9900
	60	4.0100	7.0510	3.8100	
	90	4.0100	7.0340	3.5500	
Total		12.0300		11.4500	

5.3.7.10 Test 2 Oven Leaching Results for 10 M H_2SO_4

Based on the data from Table 5.16, it can be seen that when the solution was leached for 90 minutes as opposed to 60 or 90 minutes, there was the least amount of residue left behind after leaching in the oven. Comparing 60 and 30 minutes, the leaching process conducted in 60 achieved the highest mass. Leaching produced residue with a mass percentage of 91% overall.

5.3.8 Graphical Leaching Results for Digital Hotplate

Cu was leached using a digital hotplate, and the findings were tabulated and graphically displayed.

5.3.8.1 Digital Hotplate Leaching for 2 M H_2SO_4 Test Work

The following may be seen from the results of the digital hotplate leaching for 2 M H_2SO_4 in Fig. 5.11:

(i) Test 1 residue leftovers' average mass was determined to have the least residue, with residue levels averaging 73% and metal recovery rates of 27% in 60 minutes for average mass ranging from 2.7 to 3.1 g.
(ii) Test 2 average residue weight ranged between 3.4 and 3.8 g, and after 60 minutes, 13% of the metals and 87% of the average residue were recovered.

Table 5.16 Test 2 oven leaching results for 10 M H_2SO_4

Test no.	Time (minutes)	Initial mass (g)	Volume of H_2SO_4 (mL)	Mass of residue (g)	Volume of deionized water (mL)
2	30	4.0100	7.0280	3.4600	32.9900
	60	4.0100	7.0250	3.6500	
	90	4.0200	7.0320	3.9200	
Total		12.0200		11.0300	

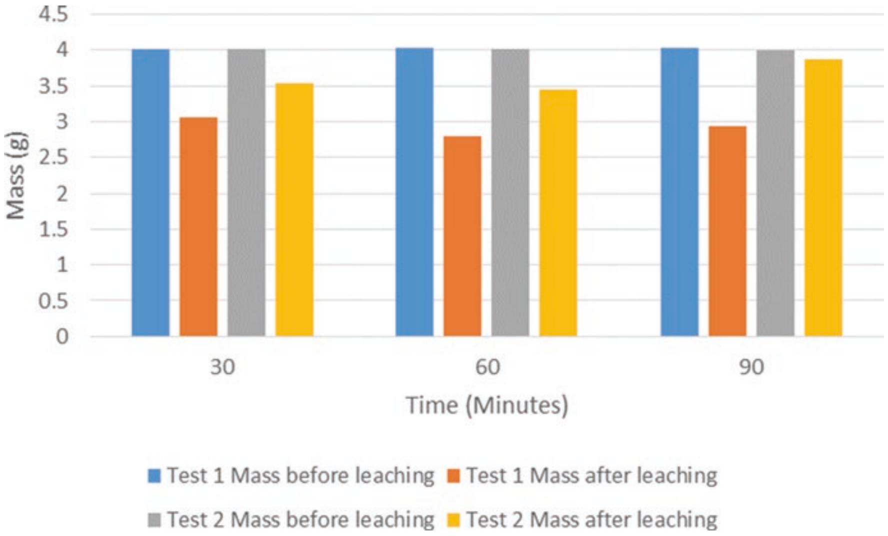


Fig. 5.11 Graphical results for 2 M H_2SO_4 hotplate leaching

5.3.8.2 Digital Hotplate Leaching for 4 M H_2SO_4 Test Work

The following may be seen from the results of the digital hotplate leaching for 4 M H_2SO_4 in Fig. 5.12:

(i) The residue of Test 1 average mass in the intervals of 30 and 60 minutes obtained the least quantity of residue.
(ii) The average mass of Test 1 varied between 3.4 and 3.5 g, with an average residue content of 86.03% and a 60-minute metal recovery rate of 13.97%.
(iii) Test 2 average residue weight ranged between 3.2 and 3.6 g, and after 60 minutes, 13% of the metals and 87% of the average residue were recovered.

5.3.8.3 Digital Hotplate Leaching for 6 M H_2SO_4 Test Work

The results of the digital hotplate leaching for 6 M H_2SO_4 are shown in Fig. 5.13.

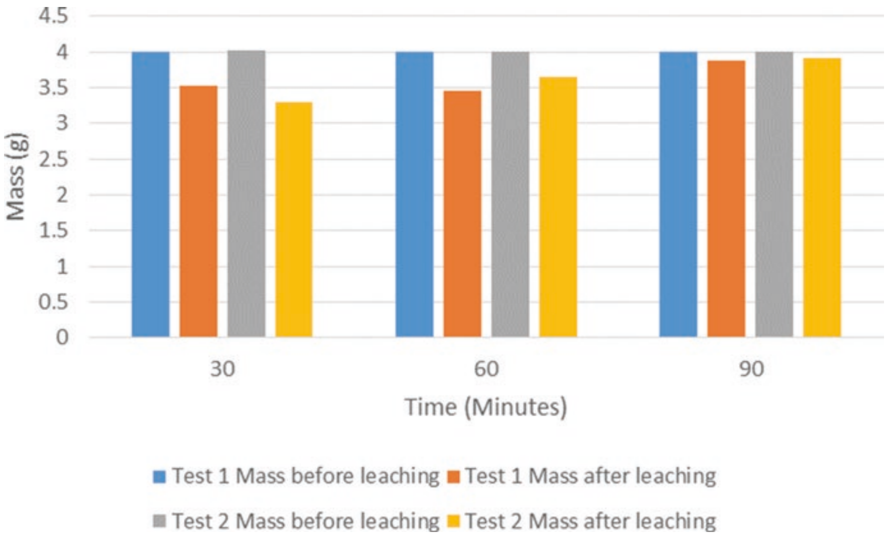


Fig. 5.12 Graphical results for 4 M H_2SO_4 hotplate leaching

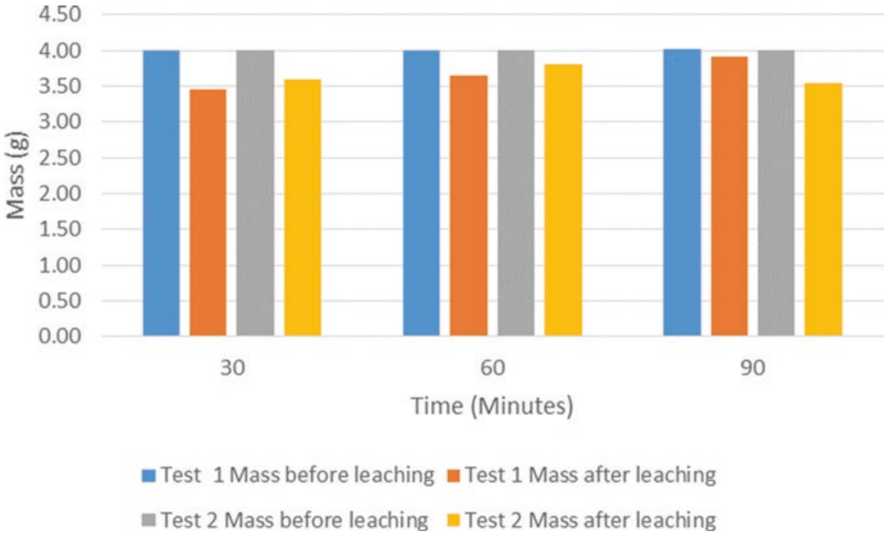


Fig. 5.13 Graphical results for 6 M H_2SO_4 hotplate leaching

(i) The residue of Test 1 average mass was produced with the least quantity of residue in the intervals of 30 and 60 minutes.
(ii) The average mass of Test 1 ranged between 3.46 and 3.65 g, with an average residue recovery rate of 86.5% and a metal recovery rate of 13.5% in just 30 minutes.

(iii) Test 2 average residue weight ranged between 3.5 and 3.8 g, and in 90 minutes, an average of 88.3% of the residue and 11.7% of the metals were recovered.
(iv) The process was found to be dormant after 90 minutes.

5.3.8.4 Oven Leaching for 2 M H_2SO_4 Test Work

The results obtained on oven leaching for 2 M H_2SO_4 in Fig. 5.14 show the following:

(i) The residue of Test 1 average mass in 60 and 90 minutes interval achieved least amount of residue.
(ii) The average mass of Test 1 varied from 3.34 to 3.56 g, and about 83.5% average of residue and 16.5% of metal were recovered in 90 minutes.
(iii) Test 2 average residue varied between 3.24–3.6 g , and about 80.8% average residue and 19.20% of metals were recovered in 90 minutes.

5.3.8.5 Results for Oven Leaching of 4 M H_2SO_4

The following can be seen from the oven leaching results for 4 M H_2SO_4 in Fig. 5.15:

(i) The residue of Test 1 average mass in the intervals of 60 and 90 minutes obtained the least quantity of residue.
(ii) The average mass of Test 1 ranged from 3.34 to 3.56 g, and in 90 minutes, an average of 83.5% residue and 16.5% metal were recovered.
(iii) Test 2average residue weight ranged between 3.24 and 3.6 g, and in 90 minutes, an average of 80.8% of the residue and 19.20% of the metals were recovered.

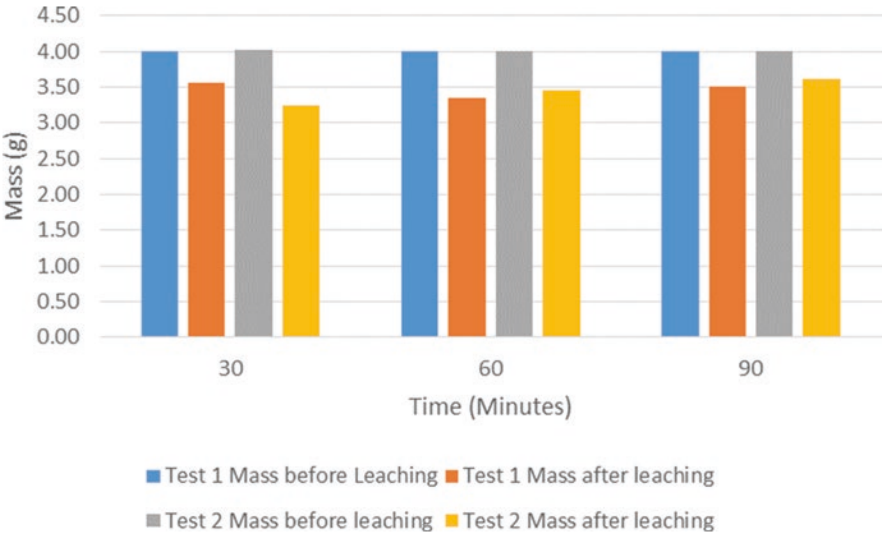


Fig. 5.14 Graphical results for oven leaching of 2 M H_2SO_4

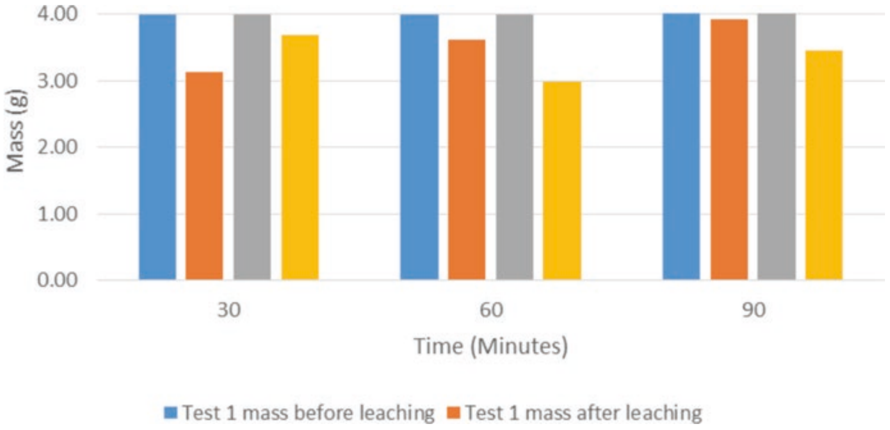


Fig. 5.15 Graphical results for oven leaching of 4 M H_2SO_4

5.3.8.6 Results for Oven Leaching of 6 M H_2SO_4

The following can be seen from the oven leaching findings for 6 M H_2SO_4 in Fig. 5.16:

(i) The residue of Test 1 average mass in the intervals of 30 and 60 minutes obtained the least quantity of residue.
(ii) The average mass of Test 1 ranged from 2.82 to 3.86 g, and in 60 minutes, an average of 70.5% residue and 25.5% metal were recovered.
(iii) Test 2 average residue weight ranged between 3.34 and 3.86 g, and in 90 minutes, an average of 83.5% of the residue and 16.5% of the metals were recovered.

5.3.8.7 Results for Oven Leaching of 8 M H_2SO_4

The following can be seen from the oven leaching findings for 8 M H_2SO_4 in Fig. 5.17:

(i) The residue of Test 1 average mass was produced with the least quantity of residue after 30, 60, and 90 minutes.
(ii) The average mass of Test 1 varied between 3.29 and 3.55 g, with an average residue content of 82.04% and a 30-minute metal recovery rate of 17.94%.
(iii) Test 2 average residue weight ranged from 3.64 to 3.91 g, and in 90 minutes, an average amount of 90.77% of the residue and 9.23% of the metals were recovered.

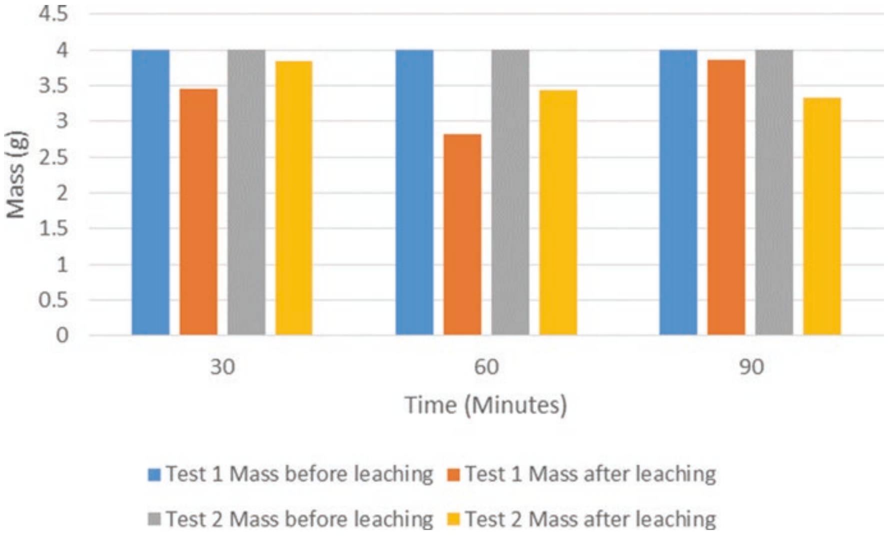


Fig. 5.16 Graphical results for oven leaching of 6 M H_2SO_4

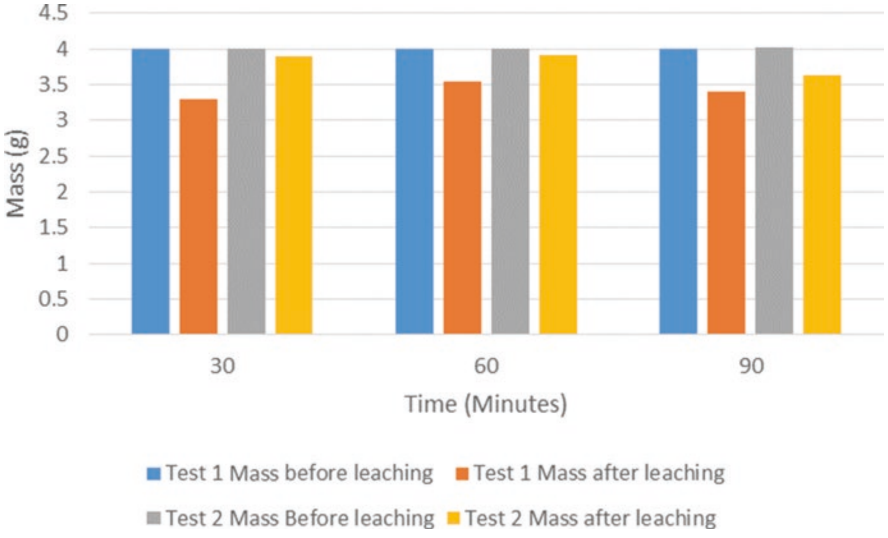


Fig. 5.17 Graphical results for oven leaching of 8 M H_2SO_4

5.3.8.8 Results for Oven Leaching of 10 M H_2SO_4

The following can be seen from the oven leaching findings for 10 M H_2SO_4 in

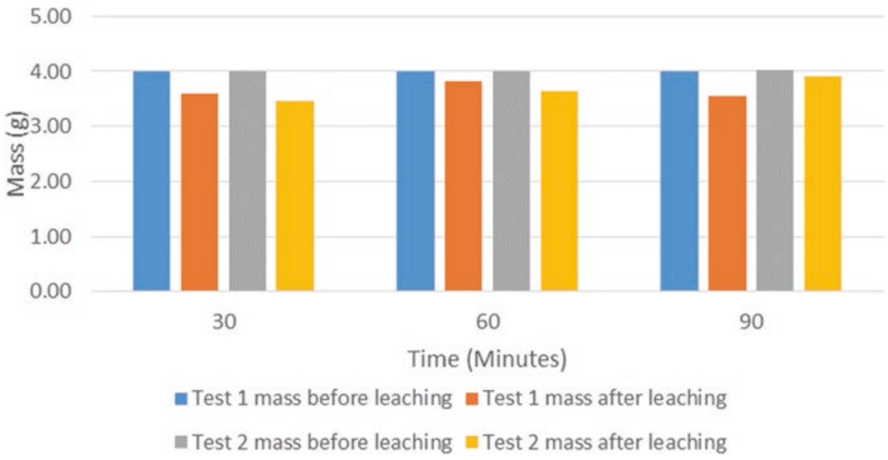


Fig. 5.18 Graphical results for oven leaching of 10 M H_2SO_4

Fig. 5.18:

(i) The residue of Test 1 average mass was produced with the least quantity of residue after 30, 60, and 90 minutes.
(ii) The average mass of Test 1 ranged from 3.55 to 3.81 g, and in 90 minutes, an average of 88.5% residue and 11.5% metal was recovered.
(iii) Test 2 average residue ranged between 3.46 and 3.92 g, and in 90 minutes, 13.72% of the average residue and 86.28% of the average residue were recovered.

5.3.9 Production of Purified Pregnant Leach Solution from Leachate

In the leach solution, the copper to iron ratio is typically 1 to 0.73. This proportion is rather high; hence, the following strategy is used.

By adding a little amount of $FeSO_4.7H_2O$ to the leaching system to slow down the dissolving of iron, the iron in the $CuSO_4$ solution was reduced. By doing this, iron was precipitated from the $CuSO_4$ solution by the hydrolysis of iron III (Fe^{3+}), which then produced complexed iron hydroxides and solid precipitates. Stumm and Lee [30], Lawson [31], and Dutrizac [32], respectively, explain the chemical reactions occurring in Eqs. (5.4), (5.5), and (5.6).

$$4Fe^{2+} + O_{2(aq)} + 4H^{+} \rightarrow 4Fe^{3+} + 2H_2O \tag{5.4}$$

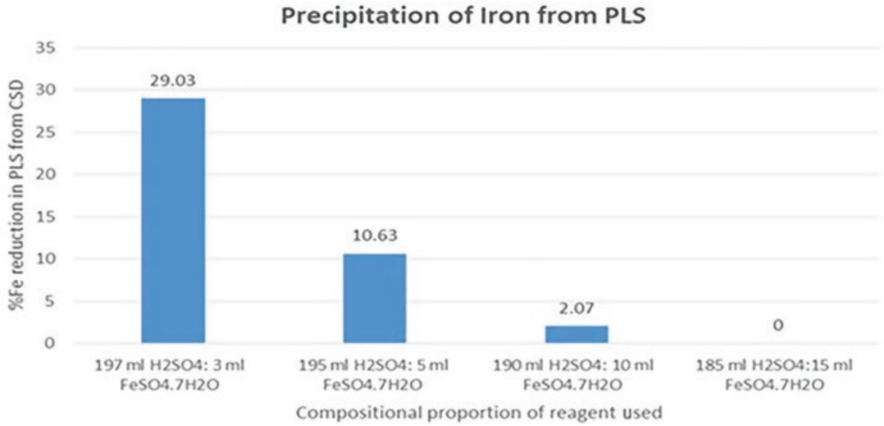


Fig. 5.19 Reduction of iron content in $CuSO_4$ solution

$$Fe(OH_2)_6^{3+} \rightarrow Fe(OH)n(OH_2)_{6-n}^{3-n} + n\mathrm{H}^+ \tag{5.5}$$

$$2Fe^{3+} + 2\mathrm{H_2O} \rightarrow Fe_2(OH)_2^{4+} + 2\mathrm{H}^+ \tag{5.6}$$

Iron was reduced by 29.03% using a mixture of 197 mL H_2SO_4: 3 mL $FeSO_4.7H_2O$ (Fig. 5.19). The enhanced reduction in dissolved iron is explained by the formation of insoluble but thermodynamically stable species such as goethite (α-FeOOH) and hematite (α-Fe_2O_3). As precipitation started at 75 °C, this reaction is percentage.

When the CSD was treated with 185 mL H_2SO_4: 15 mL $FeSO_4.7H_2O$, there was a noticeable decrease (0.5%) in the reduction of dissolved iron. This can be linked to the formation of soluble, thermodynamically stable iron sulfate precipitates like jarosite [33].

In ferrihydrite complexes, some of the sulfur may not be structurally combined but may instead be adsorbed to the surface of the iron species, according to Bigham et al. [34]. Sulfur is treated as an anionic contaminant in these situations. The iron sulfate complexes, as shown by Eqs. (5.7) and (5.8), have the potential to act as an intermediary in the formation of iron sulfate precipitates [35].

$$Fe^{3+} + \mathrm{SO_4}^{2-} \rightarrow Fe(\mathrm{SO_4})^+ \tag{5.7}$$

$$Fe(\mathrm{SO_4})^+ \, \mathrm{SO_4}^{3-} \rightarrow Fe(\mathrm{SO_4})_2^- \tag{5.8}$$

5.4 Conclusion

The leaching of copper from WCD by H_2SO_4 acid is the goal of this study. The outcomes of the digital hotplate leaching test work demonstrate that the best outcomes were obtained between 30 and 60 minutes. This demonstrates how processes attain equilibrium after a specific amount of time. Hotplate leaching has been significantly impacted by stirring rate, concentration, and temperature.

The majority of the results from oven leaching indicate that leaching time and concentration play a significant role in achieving high recovery of important metals in WCD ore. The conclusion is that if the process is conducted for a long time, the best leaching results can be attained.

Under the situation of a compositional ratio of 197 mL H_2SO_4: 3 mL $FeSO_4.7H_2O$, the inhibition of iron dissolution resulted in a reduction of iron by 29.03%.

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