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Development of a Combined Flotation and High Pressure Leaching Process for Copper and Nickel Recovery from Mine Tailing

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Abstract

The present study focused on the re-processing of copper and nickel from mine tailings. In this work, recovery of copper and nickel from mine tailing by combined process of flotation and high pressure oxidative leaching were considered. In the first stage, effects of flotation parameters including collector type, collector dosage, and pH and pulp density were examined. The results showed that over 80% copper recovery was achieved under the optimized flotation conditions while nickel recovery was lower than 30% due to its co-existence with gangue minerals of pyrrhotite, pyrite and other clay minerals. In the second stage, key parameters, particularly concentration of sulfuric acid, temperature, pressure and leaching time were investigated to test the leaching efficiency of copper and nickel from the flotation concentrate with high pressure oxidative leaching (HPOL). A comparison was made between the leaching efficiencies of copper and nickel from flotation concentrates and mine tailing.

Keywords

Mine Tailing, Flotation, High Pressure Leaching, Copper, Nickel

1. Introduction

Flotation is of interesting topic in recycling ions by using surficial reactors. Its efficiency is dependent on the hydrophobic and hydrophilic properties of mineral sample [1] [2] [3]. Improving recovery of minerals by studying their relationship between different parameters of flotation and floatability has been a long-standing goal within the mineral processing industry. However, some of the minerals are not fully recovered during this process and end up reporting to the ever increasing mine tailings that occupy a wide area in many mining countries. This increase in tailings presents an environmental problems in many regions [3]. In the case that the mine tailings are exposed to weathering, chemical percolation, some heavy metals become more soluble and mobile [4] [5] [6]. The metals that had been leached into the ground act as sources of contamination, which extends to the underground water reserves and soil. In order to address these problems, many researches have been made, aiming at metal recovery through improving separation methods, such as flotation, pyrometallurgy and hydrometallurgy [5] [7] [8] [9]. Despite that, it is difficult to achieve a comprehensive recovery. Minerals in the flotation concentrates from mine tailing have to be further processed by leaching. High pressure leaching presents a better option as it is operationally economic. In the leaching process, metals from the flotation concentrates are enriched into a pregnant leached solution with aid of acid reagent. It is therefore important that mineral interactions at flotation and leaching are quantified to understand effective recoveries of minerals and ways of rejecting iron gangue minerals [10] [11] [12] [13]. As a typical base metal mine in Botswana, BCL Limited mine has been storing its tailing from flotation since commencement in 1973 [14]. The reservoir contains large volumes of tailings that contain significant amount of copper and nickel minerals due to poor liberation or mechanical faults associated with mining operations. Therefore, a comprehensive reprocessing of these mine tailings to recover valuable metals by advanced mineral processing is very important to improve economic efficiency and lessen environmental destruction.

The main objective of this study was to assess an efficient approach for recovery of copper and nickel from the BCL Limited mine tailings of Botswana. For this purpose, influences of parameters in flotation and high pressure oxidative leaching processes on the recovery of copper and nickel from the mine tailings were discussed. Positive results of the research would help in ascertaining their feasibility for secondary resources.

2. Experimental Section

2.1. Mine Tailing Composition and Analysis

The mine tailings sample used in this study was taken from BCL Limited mine, Selebi Phikwe, Botswana. The mine tailing is represented as flotation tailings in the form of mud containing residual metal sulfides in the form of chalcopyrite, pyrite, pyrrhotite and pentlandite and reagents after flotation. The chemical content of the tailing sample was determined by coupled plasma-optical emission spectroscopy (ICP-OES, SPS 3000, Seiko instrument Inc., Japan) with a 5% error, after the sample was digested using two acid digestion (HCl and HNO_3) procedure in order to dissolve most metals and mineral. Results showed that contents of copper and nickel were 0.19 wt% and 0.23 wt%, while aluminum, magnesium, iron and silicon were 1.91 wt%, 1.32 wt%, 36.66 wt% and 37.37 wt% respectively (**Table 1**). Mineral composition of the tailing sample was determined using *X*–ray diffraction (XRD, Rigaku Co., Japan) and confirmed presence of pyrite (FeS_2), pyrrhotite ($Fe_{(1-x)}S$) muscovite and quartz (SiO_2) as shown in **Figure 1**.

Table 1. Chemical composition of mine tailing.

Element	Al	Cu	Mg	Ni	Fe	Si
Content (wt%)	1.91	0.19	1.32	0.23	36.66	37.37

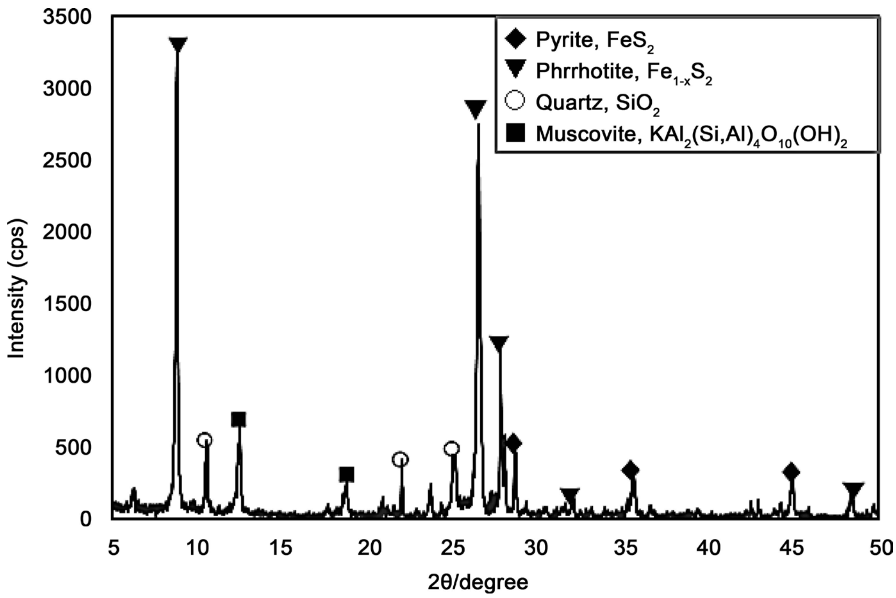

Figure 1. XRD pattern of mine tailing.

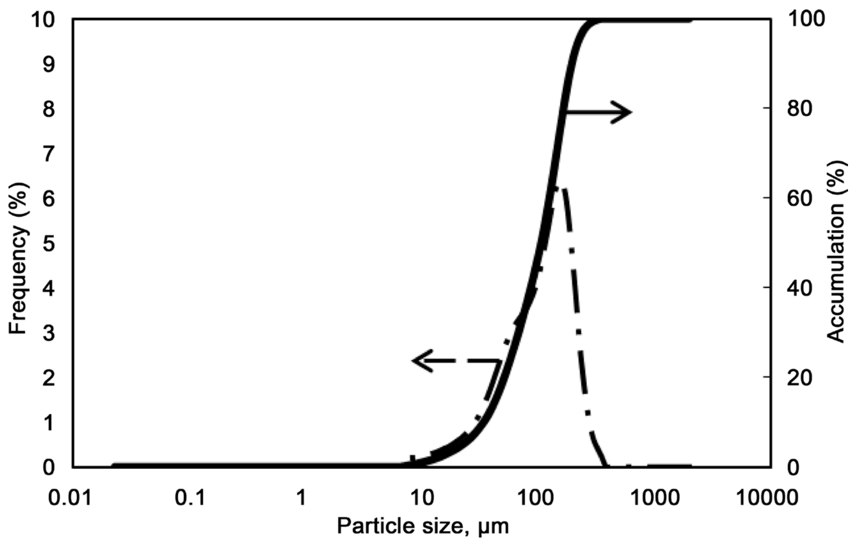

Figure 2. Particle size distribution of mine tailing.

The particle sizes of the tailing were from 35 to 100 μm with the average particle size (D_{50}) of 70 μm (Figure 2).

2.2. Flotation Circuit

The flotation experiments were performed in a 0.5 L mineral separator (MS) type laboratory flotation cell to upgrade recoveries of Cu and Ni (bulk flotation) from mine tailing. In flotation tests, an appropriate weight of the mine tailing sample was poured into 500 mL of water to prepare slurry/feed with different pulp densities from 10% to 25%. Prior to flotation, desliming was carried out for 10 minutes to filter out floating clay minerals. At each experiment, conditioning was kept at 3 minutes after addition of reagents and flotation carried out for 6 minutes.

The reagents used were collector; Potassium amyl xanthate (PAX, $C_5H_{11}OCSSK$), Mercaptan R247 (NaMBT, sodium 2-Mercaptobenzothiazole) and Aerofloat (AF208, sodium diethyl dithiophosphate); frother 200 g/t of methyl isobutyl carbinol (MIBC, $C_6H_{14}O$); sodium hydrosulfide (NaHS) as a sulfurizing agent, and calcium hydroxide ($Ca(OH)_2$) for pH adjustments. The collectors used in the experiments were supplied by Tokyo Chemical Industry, Japan. The flotation experiments were carried out at a stirring speed of 400 rpm and air injected to promote bubble production as optimum parameters. In order to investigate the effect of other parameters such as pulp density, pH, collector type and collector dosage, on the flotation efficiency, the stated parameters were varied individually within ranges. The concentrate and tailing were collected and dried in an oven for 6 - 8 hours and determined the contents of copper and nickel by ICP-OES.

2.3. High Pressure Leaching

Leaching of froth concentrate obtained from flotation was studied in an autoclave system using sulfuric acid solution. Schematic diagram of an autoclave system used in the study is shown in **Figure 3**. To obtain optimal conditions for leaching conditions, experiments were performed in a 200 mL Teflon vessel placed into an autoclave under different sulfuric acid concentrations (0 - 3 M), different temperatures (100˚C - 180˚C), different pressures (1 - 2 MPa) and different contact times (30 - 150 minutes) at a fixed solid/liquid ration of 1/10 and stirring speed of 400 rpm. Namely, a vessel with slurry was placed into the autoclave and stirring speed and temperature were set up. When a certain target temperature stabilized, oxygen gas was supplied into the autoclave until the internal pressure reached a desired value and then leaching was conducted for the

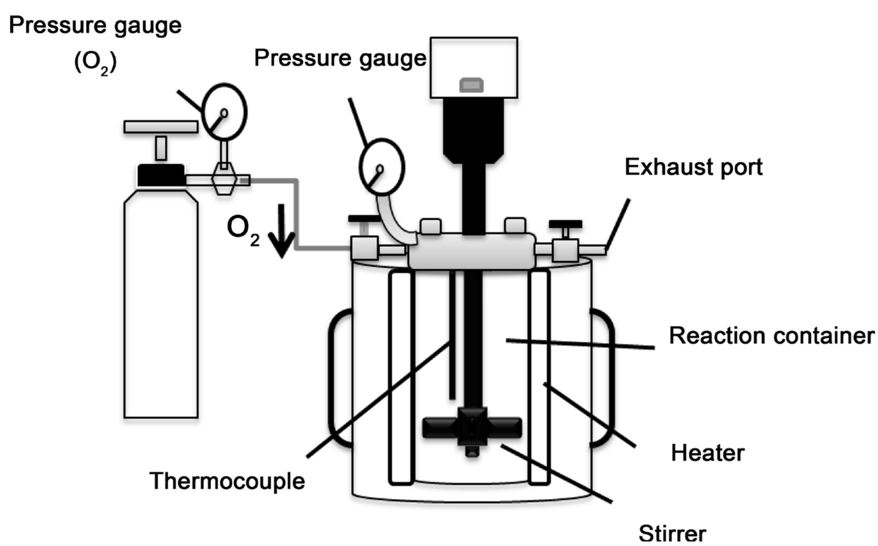

Figure 3. A schematic diagram of autoclave equipment used in the study.

time adjusted. After the leaching process, the slurry was cooled down and filtered to obtain pregnant leached solution and solid residue. Leaching behavior of mine tailing sample and froth concentrate was compared under high pressure leaching using the determined conditions.

3. Results and Discussion

3.1. Flotation

3.1.1 Effect of Different Collectors

The effect of collector type on the recoveries of copper and nickel from mine tailing was studied using various collectors such as Potassium amyl xanthate (PAX, C5H11OCSSK), Mercaptan R247 (NaMBT, sodium 2-Mercaptobenzothiazole) and Aerofloat (AF208, sodium diethyl dithiophosphate). These collectors are widely used in sulphide minerals. In this study, flotation time was set at 6 minutes while collector and MIBC additions were fixed at 50 g/t and 200 g/t respectively. Natural pH of the slurry was 5. The results for determining the effective reagent to use in the study are presented in **Table 2**. It can be seen that Cu and Ni grades were highest at 1.89 wt% and 0.61 wt% when NaMBT was added. The recoveries observed were 67.40% and 1.34% for Cu and Ni respectively therefore it shows no significant recovery to Ni due to poor floatability. Effect of PAX provided an overall effective bulk flotation as grades of 1.56 wt% and 0.75 wt%, and recoveries of 84.73% and 25% Cu and Ni were observed respectively. It has been observed that Ni recovery was substantially lower than copper recovery; this is particularly due to the presence of non-sulphide Ni in the ore and also lower floatability of pentlandite. Copper recoveries were favored in AF208 and Mercaptan R247 due to the shorter chain length of the collector's therefore increasing selectivity as discussed [15]. However, the mine tailings contained significant amount of sulphide minerals that associated with copper and nickel, PAX collector was suggested for further experiments due to its long chain and ability to promote the recoveries of copper and nickel which are associated with chalcopyrite, pyrrhotite and pentlandite.

3.1.2. Effect of Collector Dosage

Flotation behavior of copper and nickel from mine tailing was investigated under varying concentrations of PAX ranging from 0 g/t to 200 g/t when other flotation parameters were fixed at 200 g/t of MIBC concentration, 50 g/t of pulp density, slurry pH of 5400 rpm of stirring speed and 6 minutes flotation time. As shown in **Figure 4**, an increase in PAX concentration lead to drastic increases in the grade and recovery of copper, whereas further increases in PAX had moderate changes in the grade and recovery of nickel. The grade and recovery of nickel increased up to 0.41 wt% and 31.0% from 0.17 wt% and 10% with increasing PAX dosage from 0 to 75 g/t. A further increase in PAX took place a gradual decrease in the grade and recovery of nickel. Results showed that the maximum recoveries of Cu and Ni were achieved at 87.80% and 29.77% when PAX dosage was 100 g/t and 75 g/t, respectively. But a larger amount of collector would cause to float more iron sulfide minerals such as pyrite (FeS_2) and pyrrho-

Table 2. Chemical composition of mine tailing.

Collectors	Grade (wt%)			Recovery (%)		
	Cu	Fe	Ni	Cu	Fe	Ni
NaMBT	1.89	14.12	0.61	67.40	34.54	1.34
PAX + NaMBT	1.67	19.10	0.61	78.66	37.15	1.25
PAX	1.56	13.97	0.75	82.73	33.52	25.00
AEROFLOAT 208	1.12	15.93	0.37	80.60	29.51	17.82

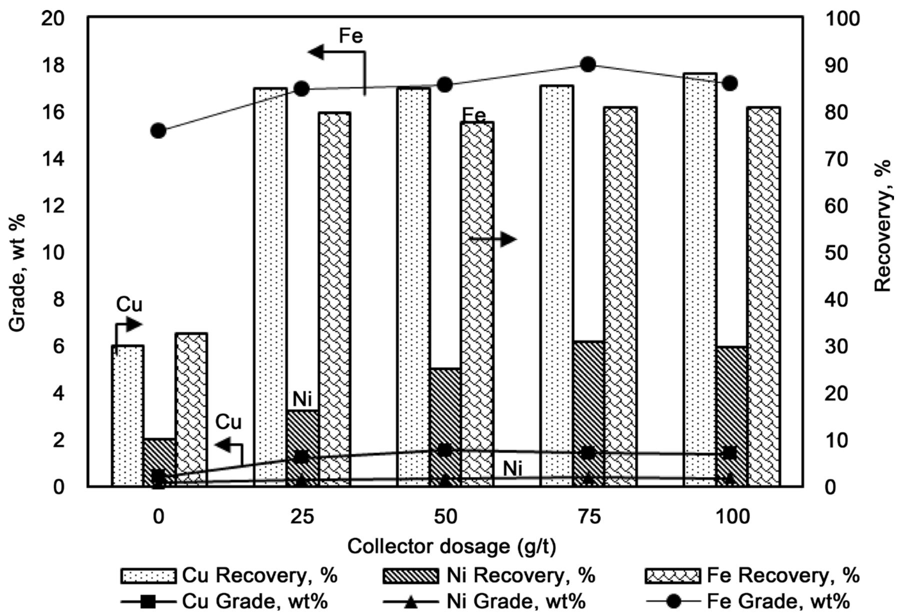

Figure 4. Flotation behavior of copper and nickel as a function of collector dosage (Flotation conditions: 10% pulp density, pH 5, and flotation time of 10 minutes).

tite ($Fe_{1-x}S$) because the grade and recovery of iron increased with increasing of PAX dosage [15] [16] [17]. Therefore, PAX dosage was selected as 50 g/t in further flotation studies.

3.1.3. Effect of pH

To study the effect of pH on the flotation of mine tailing, Ca $(OH)_2$ was used as a pH regulator through flotation experiments. The flotation tests were carried out at fixed conditions such as 50 g/t PAX, 200 g/t MIBC, 50 g/t pulp density for 6 minutes under different pH values ranging from 4 to 12. It can be seen that both recovery and grade of metals (Cu, Ni and Fe) declined with increase in pH. From Figure 5, it can be seen that 84.73% of copper and 30.89% of nickel were recovered as a flotation concentrate at pH 6. Consequently, Cu and Ni grade increased to 1.21 wt% and 0.99 wt% respectively. This observation could be attributed to the association of copper and nickel minerals with pyrrhotite and pyrite which are generally depressed in alkaline condition. On the other hand, under alkaline condition, iron sulfide minerals associated with copper and nickel could oxidized to form a layer of iron oxide or iron hydroxide resulting in an increase in the surface hydrophilicity of the mineral [17]. It was observed that the pH value

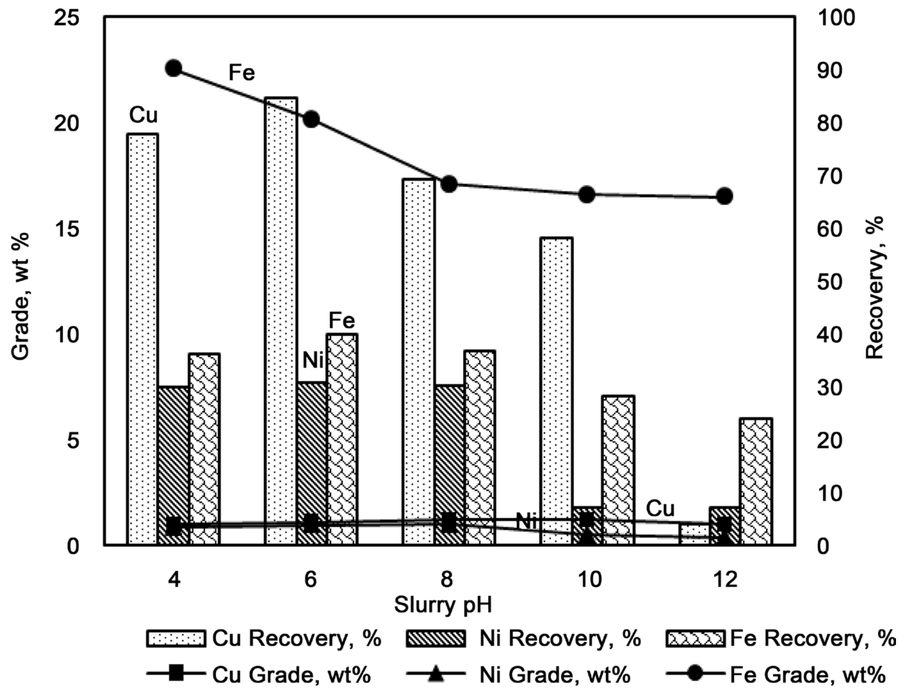

Figure 5. Effect of slurry pH on the recovery of copper and nickel. Flotation conditions: pulp density of 10%, flotation time of 10 min, PAX dosage (collector) of 50 g/t and frother dosage of 200 g/t.

has shown to have a significant impact on the grade and recovery of copper and nickel from mine tailing. Therefore, a better approach to maintain reasonable recovery and grade is to keep at pH 6.

3.1.4. Effect of Pulp Density

For recovery of copper and nickel from mine tailing, a number of flotation tests were carried out at fixed PAX dosage of 50 g/t, fixed MIBC dosage of 200 g/t, pH of 6 for 6 minutes when the pulp density was varied from 10% to 25%. Experimental results were summarized in Figure 6. The results revealed that with increasing the pulp density from 10% to 25%, recoveries of copper and nickel decreased up to 61.15% and 5.93% from 76.63% and 23.03%, respectively. Figure 6 showed that the copper grade increased from 2.09% to 2.89%, while Ni grade decreased from 0.95 wt% to 0.5 wt%. The maximum recovery of Cu was 76.63% at pulp density of 10%, but its grade was 2.09 wt%.

3.2. Optimized Conditions

Optimum flotation conditions for recovery of the Cu and Ni from mine tailing were selected as 50 g/t PAX, 200 g/t MIBC, 10% pulp density at pH 6 for 6 minutes. Under these conditions, the recoveries of Cu and Ni were 82.76% and 29.89%, when grades of Cu and Ni were 2.09 wt% and 0.99 wt% respectively. Figure 7 showed the XRD patterns of the tailing sample and concentrate obtained from the flotation under the optimized conditions. It is evident that Cu bearing minerals gave a positive response to flotation while Ni-bearing minerals confirmed a more complicated floatability due to coexistence with gangue minerals such as

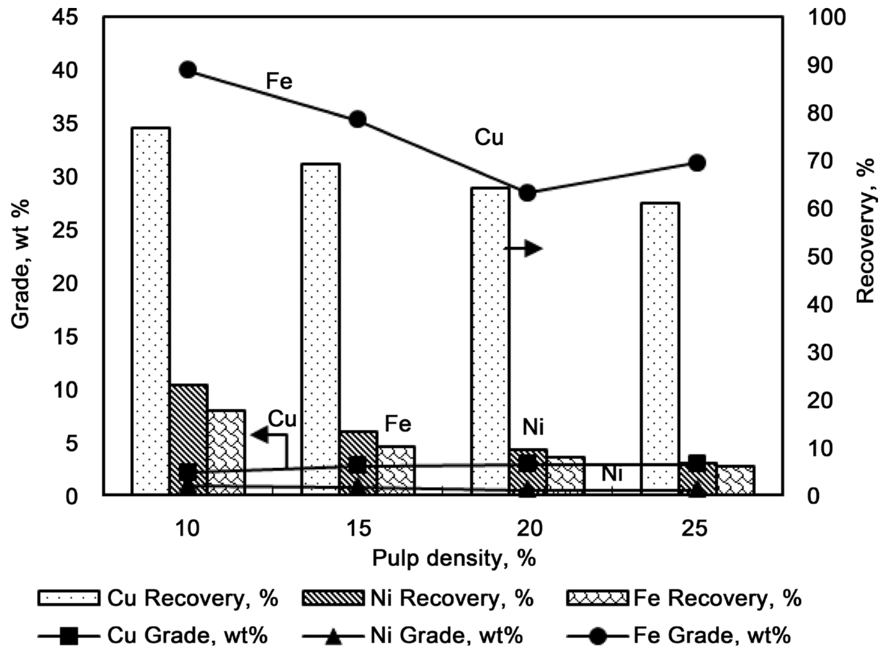

Figure 6. Effect of slurry pH on the recoveries of copper and nickel. Flotation conditions: pulp density of 10%, flotation time of 10 min, PAX dosage (collector) of 50 g/t and frother dosage of 200 g/t.

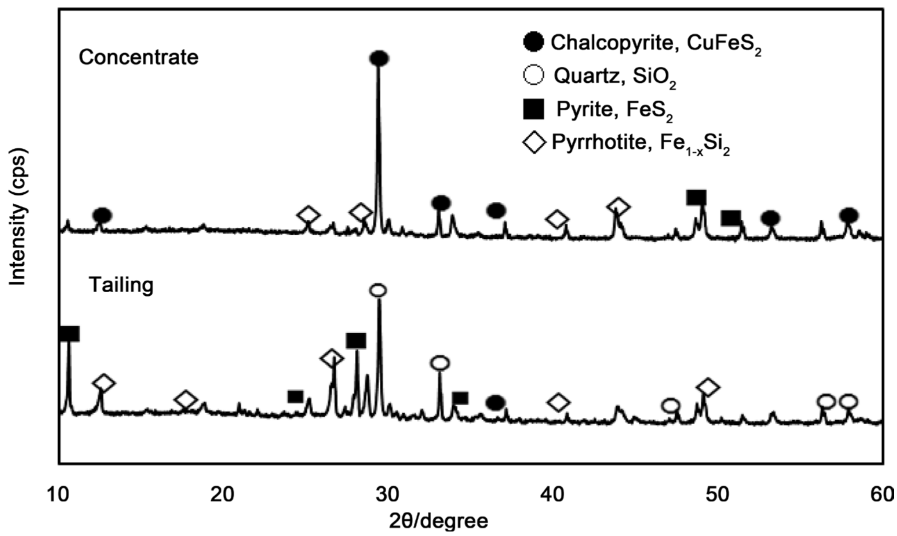

Figure 7. XRD patterns of the mine tailing sample and froth concentrate obtained from flotation under the optimum conditions determined (Pulp density: 10%, collector (PAX) dosage: 50 g/t, frother (MIBC) dosage: 200 g/t, at pH 6 for 6 minutes).

pyrite and pyrrhotite.

3.3. High Pressure Leaching

3.3.1. Effect of Sulfuric Acid on the Dissolution of Copper and Nickel

Dissolutions of copper and nickel from the froth concentrate and mine tailing were investigated under different sulfuric acid concentrations (0 - 3 M), when other parameters like leaching temperature, stirring speed, solid/liquid phase ratio, total pressure and leaching are fixed at 140˚C, 200 rpm, 1:10, 1.5 MPa and 30 minutes. Sulfuric acid was chosen as a leaching agent because it is cheap and easily available.

As presented in **Figure 8**, under the experimental conditions, the leaching efficiencies of Cu and Ni from mine tailing gradually increased with increasing the concentration of H_2SO_4. Whereas a reverse trend observed in case of Cu and Ni leaching from the froth concentrate with increase in the H_2SO_4 concentration. When increase the H_2SO_4 concentration from 1 M to 2 M, Cu leaching efficiency increased from 82.0% to 85.1%, and then decreased up to 80.4% while the H_2SO_4 concentration further increase. The efficiency of nickel leaching from the froth concentrate decreased with increasing the H_2SO_4 concentration (**Figure 8**). It was observed that an increase of the H_2SO_4 concentration can enhance the Fe leaching efficiency from the both froth concentrate and mine tailing in the same trend. Under the leaching condition with 1 M H_2SO_4 leaching conditions as an optimum H_2SO_4 conditions, the efficiencies of Co and Ni leaching were 85.09% and 87.29% from the flotation concentrate, whereas 58.77% and 61.07% from the mine tailing sample. Results showed the nearly complete dissolution of copper and nickel from flotation concentrate compared to direct leaching of mine tailing under same leaching conditions.

As a result of the above experiments flotation concentrate was used for further leaching study.

3.3.2. Effect of Temperature on the Dissolution of Copper and Nickel

The effect of temperature on the dissolution of copper and nickel from the froth concentrate in an autoclave was studied under the stirring speed of 200 rpm stirring, total pressure of 1.5 MPa, H_2SO_4 concentration of 1 M for 30 minutes at different temperatures ranging from 100˚C - 180˚C. A closely monitored temperature is important for controlling products that are produced. The results

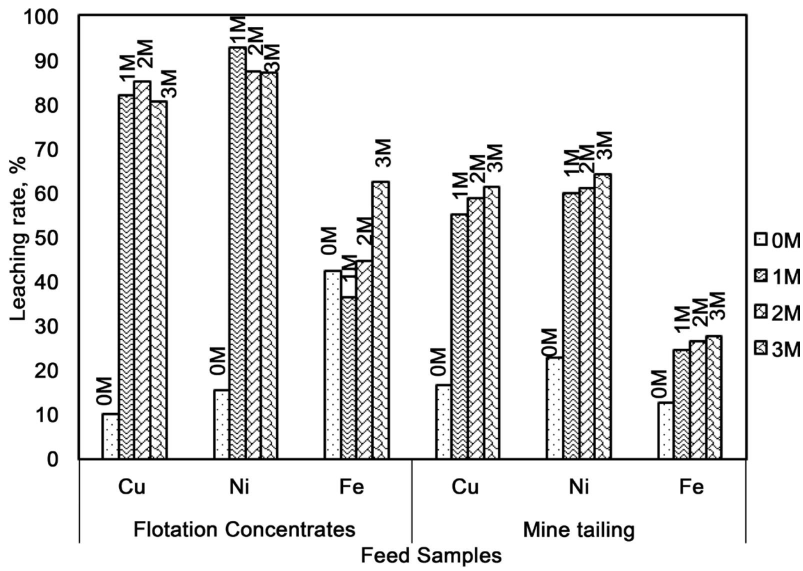

Figure 8. Dissolution of flotation concentrate and mine tailing with different concentrations of H_2SO_4 (H_2SO_4: 0 - 3 M, total pressure 1.5 MPa, S:L = 1:10 at 400 rpm, 140˚C temperature for 30 minutes).

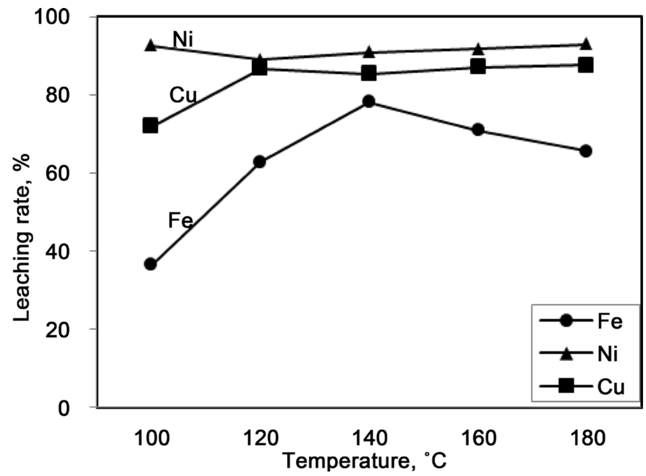

Figure 9. Dissolution of flotation concentrate as a function of temperature (1 M H_2SO_4, 1.5 MPa total pressure, S:L = 1:10 at 400 rpm for 30 min).

show that at a temperature of 140˚C leaching rate reached to 98% and 96% for Ni and Cu respectively, Figure 9.

Dissolution of Ni, Cu and Fe as a function of temperature can be made as indicated from preceding studies that have been reviewed. At an optimized temperature of 140˚C, complete dissolution of chalcopyrite, pentlandite, pyrite and pyrrhotite yield products of copper sulphate, nickel sulphate, ferric sulphate and ferrous sulphate. The dissolution of metals with H_2SO_4 solution under high pressure oxidative leaching conditions can be described according to the following Equations (1)-(3) [18]:

$$2CuFeS_2 + 5H_2SO_4 + 2.5O_2 \rightarrow 2CuSO_4 + Fe_2\left(SO_4\right)_3 + 5H_2O + 4S^v \quad (1)$$

$$2FeS_2 + H_2O + 11/2\,O_2 \rightarrow 2FeSO_4 + H_2SO_4 + S^v \quad (2)$$

$$\left(Ni_3Fe\right)_9 S_8 + 3H_2SO_4 + 31.5O_2 \rightarrow 9NiSO_4 + 9FeSO_4 + 3H_2O + S^v \quad (3)$$

The mechanism of hematite formation is explained as shown in below Equation (4):

$$Fe_2\left(SO_4\right)_3 + 3H_2O \rightarrow Fe_2SO_3 + 3H_2SO_4 \quad (4)$$

3.3.3. Effect of Pressure on the Dissolution of Copper and Nickel

The influence of total pressure ($P = P_{\text{vapor}} + P_{O_2}$) on the leaching efficiencies of copper, nickel and iron in 1 M H_2SO_4 solution was investigated under various pressures from 1 MPa to 2 MPa at stirring speed of 200 rpm, solid/liquid phase ratio of 1:10, temperature of 140˚C and leaching time of 30 minutes. Experimental results obtained are presented in Figure 10. The leaching efficiencies of Cu and Ni increase with increasing the pressure from 1 MPa to 2 MPa, and become almost constant when the pressure increases further up to 3 MPa. When increasing the pressure, Fe leaching efficiency decreases drastically from 78.2 to 65.6%. It is obvious that pressure leaching allows the simultaneous leaching of Cu, Ni and Fe from the froth concentrate under the conditions. At 1.5 MPa pressure, the maximum leaching efficiencies of Cu and Ni were achieved at

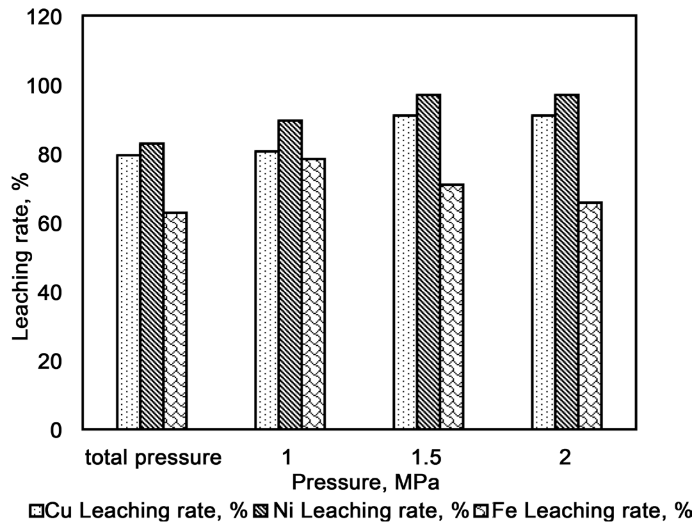

Figure 10. Dissolution of copper and nickel from flotation concentrate as a function of pressure (1 M H_2SO_4, 1.5 MPa total pressure, S:L = 1:10 at 400 rpm, 140˚C temperature for 30 min).

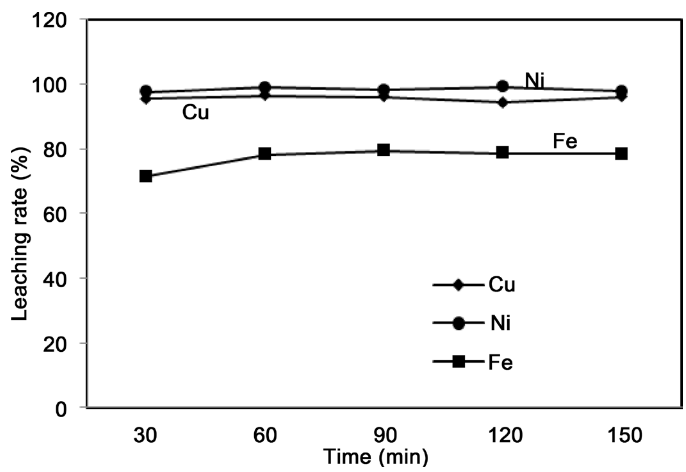

Figure 11. Dissolution of copper and nickel from flotation concentrate as a function of leaching time (30 - 150 min leaching, 1 M H_2SO_4, S:L = 1:10, 1.5 MPa pressure, 400 rpm stirring speed at 140˚C).

91.1% and 96.7%, while the Fe leaching efficiency was 70.8%. Therefore a 1.5 MPa pressure was chosen as an optimum pressure in the leaching process.

3.3.4. Effect of Time on the Dissolution of Copper and Nickel

The efficiencies of Cu, Ni and Fe leaching were studied under the conditions determined previously as the pressure of 1.5 MPa, H_2SO_4 concentration of 1 M, solid/liquid phase ratio of 1:10, temperature of 140˚C, while the leaching time varied from 30 min to 150 min. Results showed that the leaching time gave no obvious effect on the dissolution of copper and nickel from the concentrate under the conditions, because their leaching efficiencies were higher than 96% throughout the different time intervals (**Figure 11**). In the case of iron leaching, when leaching time increases from 30 min to 60 min, Fe leaching efficiency increased from 71.3% to 78.2%, but no further change was observed while the

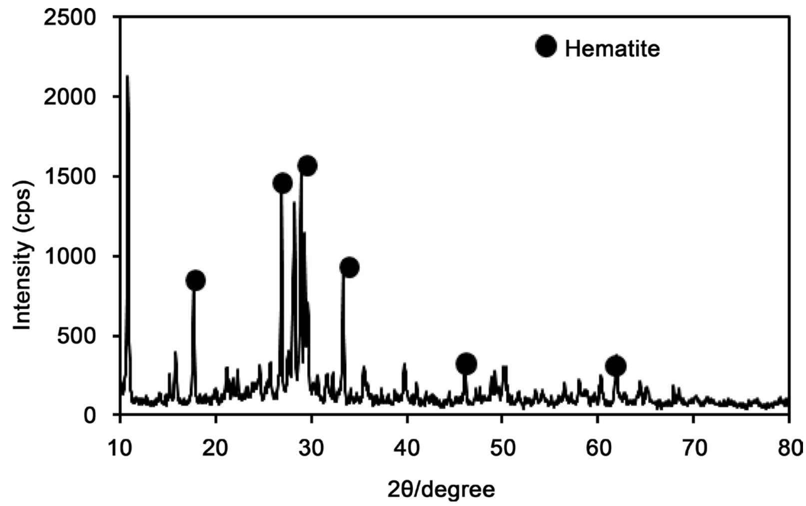

Figure 12. XRD patterns of the froth concentrate from flotation and solid residue from high pressure oxidative leaching.

leaching time increased until 150 min. As a result, 30 minutes leaching was selected due to the higher dissolution of Cu and Ni, and lower dissolution of Fe at the condition. Hence further leaching experiments would be carried out under the selected conditions as optimum leaching conditions. XRD measurement of the solid residue from the HPOL of the froth concentrate under the optimized conditions identified hematite as a main mineral constituent Figure 12. It is confirmed the formation of the hematite under the HPOL conditions according to Equation (4).

3.4. Proposed Flowsheet for Cu and Ni Recovery from Mine Tailing

Based on the above results, a general flowsheet for reprocessing of mine tailing can be proposed. The overall processes investigated in the current study are provided in Figure 13. Before flotation, desliming method was carried out for 30 minutes to allow gangue minerals that float to separate. In the case of flotation, nickel recovery was significantly restricted because of the finer particle size that occurred as solid inclusion with pyrrhotite. Other limiting factors to the upgrade and recovery of copper and nickel were the increased amount of floating gangue minerals. For the case of high pressure leaching, high dissolution values were achieved indicating the presence of readily leachable copper oxides, an observation that coincide with other researches [11].

4. Conclusion

In this study, recovery of copper and nickel from mine tailing was investigated by froth flotation and high pressure acid leaching processes. A maximum recovery of copper and nickel was reached at 84.73% and 30.9% while their grades were 2.09 wt% and 0.99 wt%, respectively under the optimized flotation conditions: 50 g/t PAX, 200 g/t MIBC, slurry pH 6, flotation time for 6 minutes. Over 96% of copper and 98% of nickel were extracted from the froth/flotation con

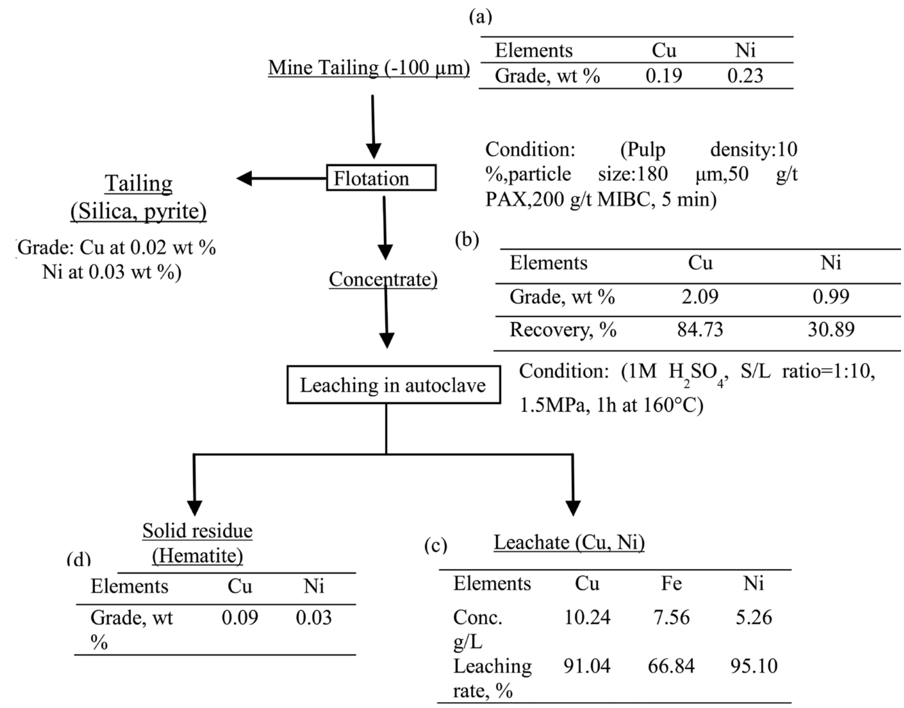

Figure 13. Schematic flowsheet for combined flotation and high pressure leaching for recovery of copper and nickel.

centrate, whereas about 55% of copper and 58% of nickel were dissolved from the mine tailing under the same leaching conditions. It can be concluded that the combined process of flotation and high pressure oxidative leaching (HPOL) is an efficient approach for recovery of copper and nickel from mine tailing. Throughout the flotation and HPOL processes, iron is simultaneously recovered from the mine tailing. It was revealed that the main iron minerals such as pyrite and pyrrhotite in mine tailing were converted to the hematite as results of the HPOL under the optimum conditions. According to this study, it is clear that other alternative methods such as magnetic separation need to be considered to increase nickel recover. The separation of iron bearing minerals from the final tailings is an important aspect to reduce possible acid mine drainage. As for the leachate solution, it is proposed that further separation method such as precipitation and electro winning need to be discussed.

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