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ORIGINAL ARTICLE

Chalcopyrite Leaching with Hydrogen Peroxide in Formic Acid Medium

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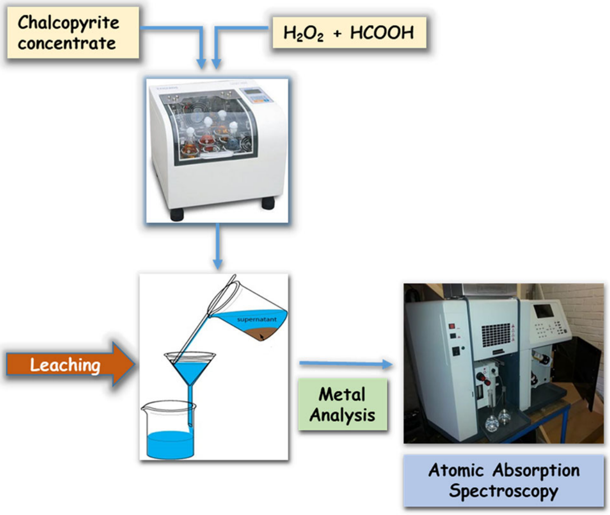
Abstract This study is concerned with the leaching process of copper with hydrogen peroxide (H_2O_2) and formic acid (HCOOH) from the Küre chalcopyrite concentrate. All experiments were carried out in a leaching system equipped with an adjustable temperature shaker under atmospheric pressure. The needed oxygen was provided by H_2O_2 decomposition to achieve the oxidizing leaching medium. The parameters affecting leaching process, H_2O_2 concentration, HCOOH concentration, sulfuric acid (H_2SO_4) concentration, leaching time, liquid/solid ratio and stirring speed, were studied. The optimum experimental conditions were determined as leaching temperature 40 °C, leaching time 120 min, H_2O_2 concentration 1.2 M, HCOOH concentration 3 M and stirring speed 500 rpm. It was observed that there was no positive effect of sulfuric acid (H_2SO_4) concentration on the metal extraction. In addition, the percentage of copper passing to solution was increased from 7.83 to 62.37% as a result of the liquid/solid ratio increasing from 2.0 to 100, respectively. Consequently, formic acid could be used as an alternative and organic leaching agent in the selective leaching of chalcopyrite and even for leaching processes of the other oxidized and sulfured ores containing precious metals.

Graphic Abstract



Keywords Leaching · Chalcopyrite concentrate · Formic acid · Hydrogen peroxide · Metal extraction

1 Introduction

The demand for copper has been increasing around the world day by day. The copper deposits are required to contain at least 0.5% and preferably 2% copper in terms of commercial mining operation [1]. It is known that copper is present in the form of oxidized and sulfide minerals in nature; chalcopyrite ($CuFeS_2$), chalcocite (Cu_2S), covellite (CuS), bornite (Cu_5FeS_4), malachite ($Cu_2(OH)_2CO_3$), azurite ($Cu_3(OH)_2(CO_3)_2$), etc. [2–4]. Of the above-

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mentioned minerals, sulfur minerals and their concentrates obtained by flotation techniques, especially chalcopyrite, are widely used in the copper production [5–7].

Chalcopyrite ($CuFeS_2$) is a mineral that meets a significant part of the world's copper needs, and about 70% of the existing copper ores are found in the form of chalcopyrite mineral [8]. Metal extraction from chalcopyrite and its concentrate is carried out by pyrometallurgical and hydrometallurgical methods. Due to the harmful influences of sulfur dioxide emission in the pyrometallurgical applications, the researchers have focused on the hydrometallurgical processes that can be used for the sulfide minerals in recent years. Therefore, considering that high-grade copper ore reserves are continuously decreasing, it is important to enhance the innovative and technological processes for copper production from such minerals [9–11]. Although the use of sulfuric acid with ferric ion as oxidant in the leaching processes of chalcopyrite is common [12, 13], the compact and refractory nature of chalcopyrite limits metal extraction efficiency [8, 14]. The reason why metal extraction from chalcopyrite is difficult is the formation of a passive film on the reactant surface due to by-products such as elemental sulfur, jarosites and metal sulfides [13]. In this sense, there are different options such as pressure leaching, pyrite addition and bioleaching to achieve high metal extraction efficiency from chalcopyrite [15–17]. On the other hand, the researchers have focused on examining the various leaching materials and methods for the extraction of copper from chalcopyrite via hydrometallurgical processes [12, 18, 19]. The leaching agents containing inorganic acids or ammonia compounds are prevalently utilized in leaching processes of the chalcopyrite, such as sulfuric acid [20], hydrochloric acid [21] and ammonia [22]. For instance, when sulfate solutions and chloride solutions are compared, it is seen in the presence of chloride, the copper and iron solubility is higher, iron ions are more easily oxidized, and leaching time is shorter [23]. Also, in the chalcopyrite leaching process using chlorite solutions, Fe(III) ions in the form of Fe(III) [24, 25] and Cu (II) ions in the form of Cu (II) [26] generally serve as oxidizing agents. On the other hand, without any pretreatment or other mineral additions, alternative chemicals have also been studied to improve the efficiency of metal extraction from chalcopyrite [27, 28]. Organic acids as leaching agents are generally more preferred than the aforesaid acids in hydrometallurgical processes owing to minimum acid overconsumption, to avoid the dissolution of undesirable impurities in the ore, and that as a result to reduce the problems in subsequent purification process [29, 30]. Therefore, we aimed to use the alternative chemicals suitable for copper leaching from chalcopyrite. And chalcopyrite leaching was investigated in the presence of formic acid, which is a chemical lixiviant.

In addition to the above-mentioned oxidants, strong oxidizing agents have also been studied in the chalcopyrite leaching systems; ozone [31, 32], Cr(VI) ions [33] and hydrogen peroxide [34, 35]. Among them, hydrogen peroxide shows acidic properties in aqueous solutions and may be decomposed by releasing oxygen or water depending on ambient conditions (Eqs. 1, 2). For this reason, it can be utilized as an oxidation agent in hydrometallurgical processes for the extraction of metals especially from sulfur ore, mineral ores and concentrates due to its strong oxidizing properties [36, 37].

$$H_2O_2 + 2H^+ + 2e^- \rightarrow 2H_2O \tag{1}$$

$$H_2O_2 \rightarrow 2H^+ + 2e^- + 2O_2. \tag{2}$$

According to dissolution mechanism (Eq. 4) between hydrogen peroxide and chalcopyrite concentrate in the sulfuric acid medium, sulfur is oxidized to sulfate ($SO_4{}^{2-}$) and releases elemental sulfur (S^o), but elemental sulfur occurs in very small amount (Eq. 5) [20].

$$CuFeS_2 \rightarrow Cu_2^+ + Fe_2^+ + 2S^o + 4e^- \tag{3}$$

$$2CuFeS_2 + 17H_2O_2 + 2H^+ \rightarrow 2Cu^{2+} + 2Fe^{3+} + SO_4^{2-} + 18H_2O \tag{4}$$

$$2CuFeS_2 + 5H_2O_2 + 10H^+ \rightarrow 2Cu^{2+} + 2Fe^{3+} + S^o + 10H_2O. \tag{5}$$

In the literature, most of the studies on the chalcopyrite extraction have been carried out in sulfuric acid solutions in the presence of hydrogen peroxide [20, 35, 38]. And, it has been found that the dissolution efficiency of sulfur minerals in sulfuric acid is high. For example, Adebayo et al. [38, 39] observed an increase in the amount of copper passing into solution by increasing concentration of both hydrogen peroxide and sulfuric acid. They calculated the reaction orders 0.77 and 1.45 depending on the concentrations of sulfuric acid and hydrogen peroxide, respectively. Also, as a result of the kinetic studies, it is proved that the leaching process fits to the shrinking core model and the dissolution reaction is controlled by surface chemical reaction. These findings overlap similar studies in the literature [20, 40–42]. On the other hand, hydrogen peroxide exhibits an unstable compound characteristic which can decompose rapidly during dissolution processes, particularly due to the presence of impurities. To overcome this problem, some stabilizers are used in the dissolution processes such as acetic acid, formic acid, phosphoric acid and polar organic solvents [36, 43–46].

While a great number of studies on the extraction of chalcopyrite using the various inorganic and organic acids were carried out in the literature, especially in the presence of hydrogen peroxide in the sulfuric acid medium, no studies were found to belong to oxidation of chalcopyrite with formic acid. Considering that there is no detailed study on dissolution of chalcopyrite using formic acid, it is a fact that the present study will fill the gap in the literature. The aim of this study is to examine the dissolution of chalcopyrite in the formic acid medium using hydrogen peroxide in detail and to determine the parameters affecting leaching kinetics.

2 Material and Methods

2.1 Preparation and Characterization of Material

The chalcopyrite ore extracted from the Küre region of Kastamonu was crushed, sieved and milled. Then, the smelting process was applied to the chalcopyrite concentrate obtained via floatation method. The chalcopyrite ore was supplied from Black Sea Copper Company. The chalcopyrite concentrate was dried at 80 °C for 12 h to remove moisture and then stored in closed containers for further experiments. The amounts of metallic components such as iron, copper and the major sulfur element in the chalcopyrite ore were determined by ICP and gravimetric analysis ($BaSO_4$ method), respectively [19]. The chalcopyrite sample to be analyzed was weighed to a precision of 0.100 g ± 0.001 and added to the Teflon bombs resistant to high pressures which were used for solubilization. Following that, 2 ml of concentrated HNO_3, 6 ml of concentrated HCl and 2 ml of concentrated HF were added to the sample. It was kept open for about 10 min in the case of gas outlet, and then, the nozzles were closed with sealed covers. After the prepared samples were placed in a microwave oven (Milestone Ethos D model), the connections of temperature and pressure measurement were established and then the solubilization procedure was performed by a microwave heating program via a computer.

To determine the ignition loss, the ceramic capsules were heated at 1000 °C for 6 h and cooled in a desiccator. Then, samples weighed with a precision of 5.0 g ± 0.001 g were heated at 900 °C for 4 h and the ignition loss was calculated from the difference between the initial and final weights of the samples. The chemical analysis of results in Table 1 presents that 20.94%, 27.52% and 28.01% of the sample consisted of Cu, Fe and S, while impurities like Si, K, Al and Mn were at negligible levels. The mineral structures, surface morphology and particle size distributions of the samples were characterized.

Analyses of the mineralogical structures, surface morphology and particle size distribution (Malvern, Mastersizer 3000) of chalcopyrite concentrate used in the experiments were carried out using X-ray diffractometer (RIGAKU, Smartlab) and scanning electron microscopy (TESCAN, MAIA3 XMU) in Bartın university central research laboratory.

As seen in Fig. 1, concentrate was mainly formed by chalcopyrite ($CuFeS_2$),), pyrite (FeS_2) and some alpha-sulfur (α-S). The sample also had a granular structure with different sizes and shapes (Fig. 2). According to the results of particle size distribution, it was determined that 50% and 10% of the particles had a particle size smaller than 23.9 μm and 6.80 μm, respectively (Fig. 3).

2.2 Preparation of Solutions

Formic acid (0–5 M), hydrogen peroxide (0–2 M) and sulfuric acid (0–3 M) solutions were prepared at different concentrations for the leaching process. The analytical grade stock solutions (35% H_2O_2, 100% CH_2O_2 and 96% H_2SO_4) were provided from Merck. Leaching solutions in the desired concentrations were prepared from the stock solutions using distilled water.

2.3 Experimental Study

All leaching tests were performed in 250-ml covered Erlenmeyer flasks in a shaker equipped with an adjustable agitation speed and temperature (Zhcheng ZHWY-200D). All studies were carried out in liquid/solid ratio of 20 ml/g. After the temperatures of the leaching solutions reached the desired temperature, the concentrated chalcopyrite powders were added and then mixtures were stirred at stipulated temperature and time. The mixtures were filtered (Double Rings-203), and supernatant was collected. Finally, the quantity of metal ions in the supernatant was calculated after pH measurements in order to determine the optimum conditions for the leaching of copper and iron from the chalcopyrite concentrate.

All experiments were performed in duplicate in terms of reproducibility of the results. Calculations were noted considering the mean values of the results in the variable range of ± 5%.

3 Results and Discussion

While the other process conditions (5 M HCOOH; L/S ratio 20 ml/g; time 240 min, stirring speed 400 rpm, temperature 40 °C) were constant, the experiments were

Table 1 Chemical analysis of chalcopyrite concentrate

Component	Percentage by weight
Al	0.38%
Co	752 mg/kg
Cu	20.94%
Fe	27.52%
K	2.01%
Mn	0.92%
Ni	145 mg/kg
Pb	1.18%
S	28.01%
Si	1.59%
Zn	4194 mg/kg
Loss ignition (900 °C)	16.95%

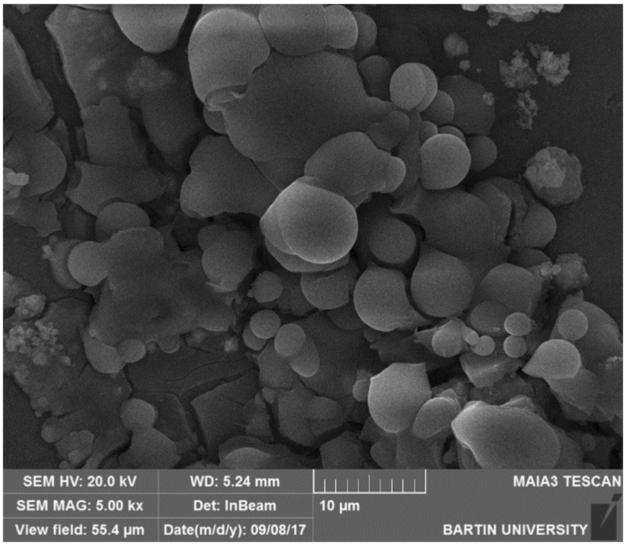


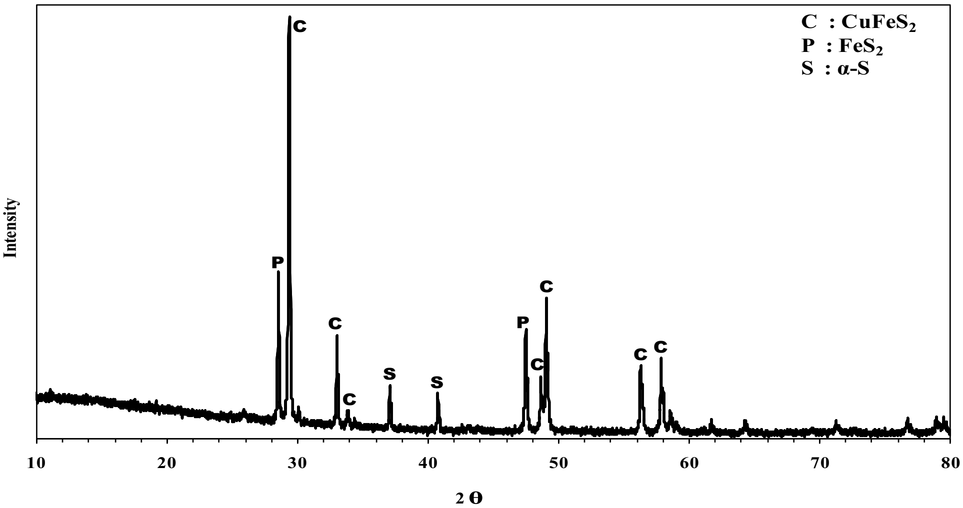
Fig. 2 SEM image of chalcopyrite concentrate

carried out owing to assess the effect of hydrogen peroxide on metal extraction in the range of 0–2 M. As seen in Fig. 4, the values of copper and iron extraction in the presence of 5 M HCOOH (no H_2O_2) appear to be 6.1% and 4.2%, respectively. On the other hand, it is observed that the hydrogen peroxide concentrations higher than 1.4 are not effective on copper extraction, while there is also a slight increase in the amount of iron passing through the solution. However, when the extraction percentages of metals are compared, it is seen that the percentage extraction of iron is lower than copper. The results clearly indicate that the hydrogen peroxide concentration (1.2 M) is adequate for maximum extraction of metals in the current experimental conditions. These findings are consistent with the previous studies that the rate of degradation of hydrogen peroxide is proportional to its concentration [38, 47].

Further experiments were carried out to examine the influence of formic acid concentration in the range of 0–5 M when the hydrogen peroxide concentration was kept constant at 1.2 M. The results in Fig. 5 clearly demonstrate that there is no a significant change in formic acid concentrations higher than 3 M on the extractions of both metals, while the percentage of iron and copper extraction increase from 4.9 to 8.6% and from 15.5 to 31.9% with an increase in the formic acid concentration, respectively. It may be attributed to the fact that formic acid inhibits rapid degradation of hydrogen peroxide and indirectly contributes to leaching efficiency.

As seen in Fig. 6, it is observed that the leaching time is a highly effective parameter on the extraction of metals in hydrometallurgy processes. Based on obtained data, it is seen that the leaching time (120 min) is sufficient and the amounts of copper and iron passing through the solution

Fig. 1 XRD pattern of chalcopyrite concentrate



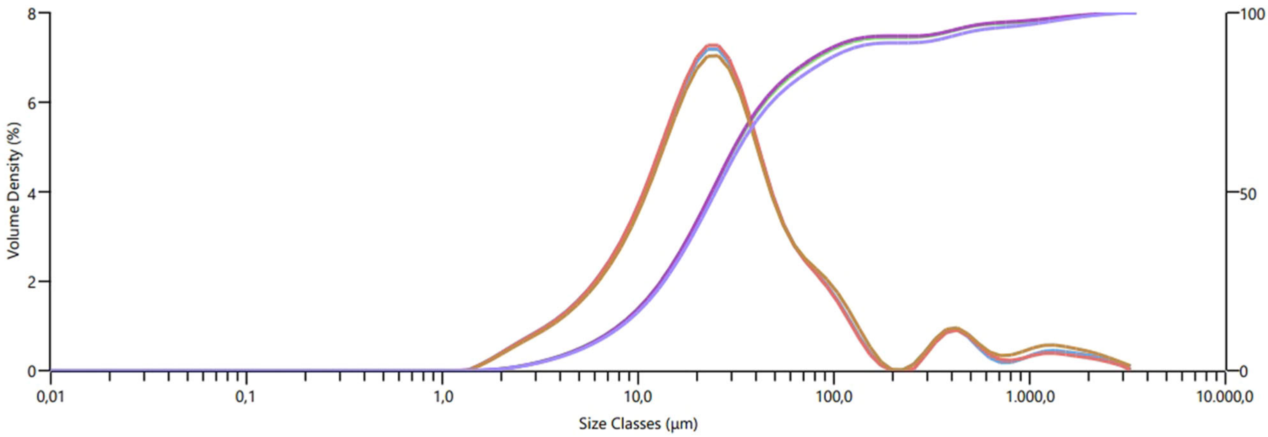


Fig. 3 Analysis of particle size distribution of chalcopyrite concentrate

are 30.2% and 15.3% at the end of this period, respectively. On the other hand, it is possible to indicate that the reactions occur rapidly as a result of the amount of copper (14.5%) passing through the solution in 30 min. Similarly, a study on the leaching kinetics of galena ore in the presence of acetic acid and hydrogen peroxide in the literature suggests that the maximum extraction yield is reached within the range of 40–60 min [20]. In addition, elevation of leaching time also causes an increase in the amount of iron passing through the solution as shown in Fig. 5. Therefore, the leaching time has been selected as 120 min for the subsequent experiments in order to minimize the amount of iron which is a contaminant in the leaching solutions.

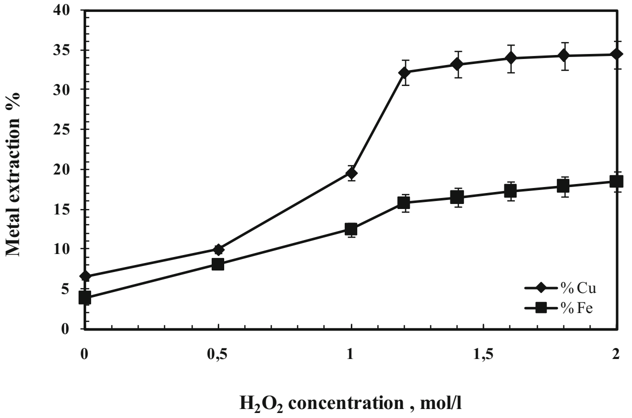


Fig. 4 Effect of hydrogen peroxide concentration (5 M HCOOH; L/S ratio 20 ml/g; leaching time 240 min; stirring speed 400 rpm; leaching temperature 40 °C)

It is extremely important to control the leaching temperature because the oxygen that is released as a result of rapid degradation of hydrogen peroxide in leaching processes at high temperature and atmospheric conditions does not enter into extraction.

The experiments were performed at various temperatures (10, 20, 30, 40, 50 and 60 °C) and atmospheric pressures to investigate the influence of temperature on the leaching yield of iron and copper metals when the other

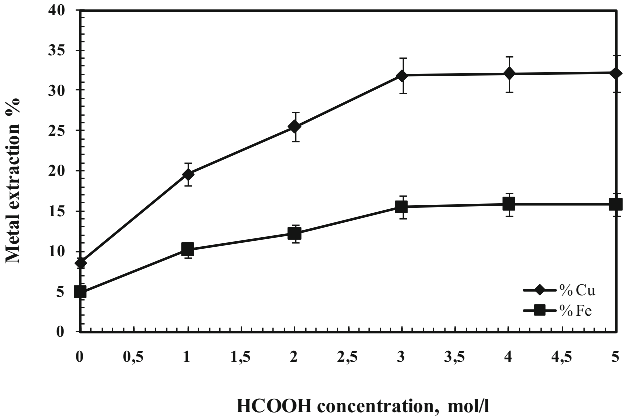


Fig. 5 Effect of formic acid concentration (1.2 M H_2O_2; L/S ratio 20 ml/g; leaching time 240 min; stirring speed 400 rpm; leaching temperature 40 °C)

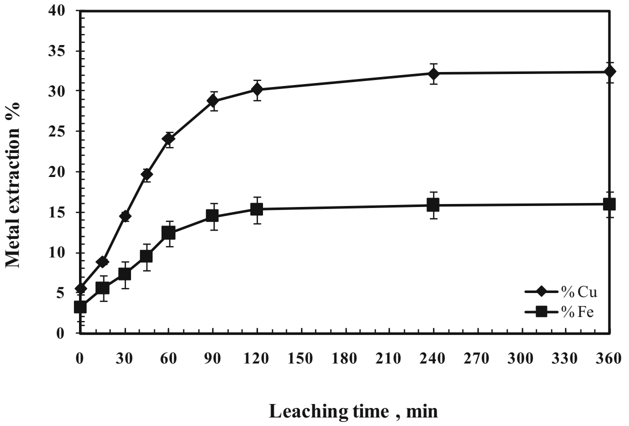


Fig. 6 Effect of leaching time (1.2 M H_2O_2; 3 M HCOOH; L/S ratio 20 ml/g; leaching time 240 min; stirring speed 400 rpm; leaching temperature 40 °C)

process conditions were constant (1.2 M H_2O_2; 3 M HCOOH; time 120 min; L/S ratio 20 ml/g; stirring speed 400 rpm). As shown in Fig. 7, the extraction percentages of both metals increase with an increase in temperature. The increased rate of copper extraction is achieved from 10.1 to 33.0%. This is due to an increase in the temperature from 10 to 60 °C. Similarly, a raise in the rate of iron extraction occurs from 6.7 to 16.4%. On the other hand, in spite of a raise in leaching efficiencies of both metals to 40 °C, there is no noticeable increase after 40 °C. As Sokić et al. [20] and Mahajan et al. [40] have reported, this decrease is due to the rapid decomposition of peroxide at high temperatures. Also, at temperatures above 50° C, Agacayak et al. [34] found a decrease in copper dissolution efficiency due to the high decomposition of hydrogen peroxide. In addition, they also reported that copper extraction yield increases with increasing temperature up to 90 min; after this leaching time, the copper leaching efficiency get affected at temperatures above 50 °C.

The results of the experiments carried out in different sulfuric acid concentrations in Fig. 8 demonstrate that there is no significant effect on metal extraction. The presence of formic acid in the leaching solutions is thought to provide sufficient acidity for leaching medium when the pH value of leaching solution without sulfuric acid is considered to be about 2.

The results in Fig. 9 illustrate that a raise in the extraction of metals from 10 to 20% is due to an increase in the stirring speed from 50 to 600 rpm. About 90% and 95% of the highest leaching yields of the copper and iron occur at 500 rpm, respectively. However, there is no remarkable change at the stirring speed higher than 500 rpm. It can be attributed to the more effective contact between the chalcopyrite particles and the leach solution at higher mixing speeds. For instance, Adebayo et al. reported that the increase in mixing speed accelerates the decomposition of hydrogen peroxide. Also, the increase in stirring speed during leaching of pyrite in sulfuric acid medium using hydrogen peroxide negatively affects pyrite oxidation. However, it is thought to be caused by the improvement in the interaction between the pyrite particles and the hydrogen peroxide, not the decomposition of hydrogen peroxide at high stirring speeds [38].

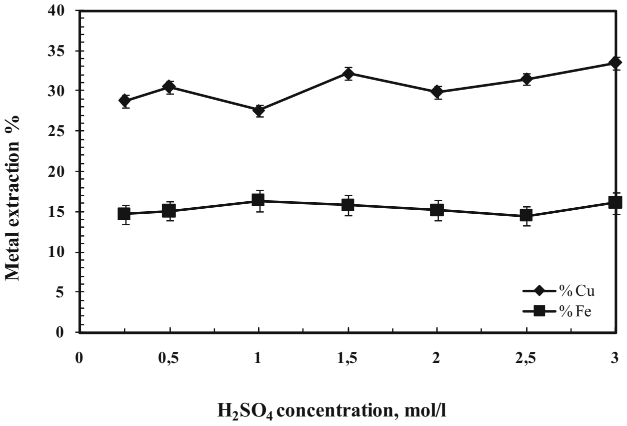


Fig. 8 Effect of sulfuric acid concentration (1.2 M H_2O_2; 3 M HCOOH; L/S ratio 20; leaching time 120 min; stirring speed 400 rpm)

With all other conditions kept constant (1.2 M H_2O_2; 3 M HCOOH; 120 min and 500 rpm), the experiments performed at different L/S ratios ranging from 2.5 to 100 ml/g determine their effect on the extraction of metals in the leaching process. It is seen that the ratio of L/S is generally high in most studies with hydrogen peroxide to elucidate the kinetic of leaching process. Moreover, it is not possible to leach chalcopyrite concentrate at low liquid/solid ratio in the presence of hydrogen peroxide (no formic acid) due to the formation of the leaching solution and non-isothermal conditions. Figure 10 clearly shows that the extraction values of the metals increases with an

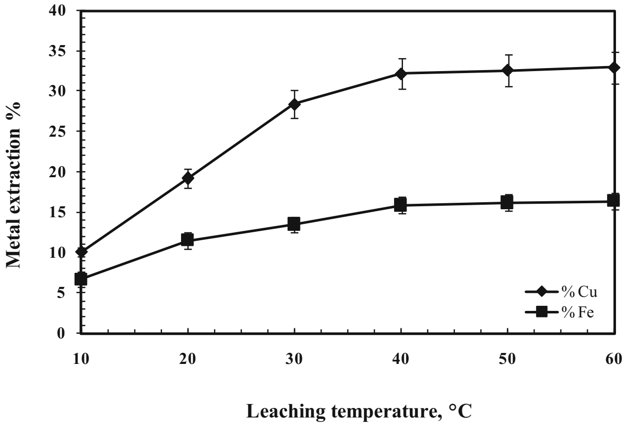


Fig. 7 Effect of leaching temperature (1.2 M H_2O_2; 3 M HCOOH; L/S ratio 20 ml/g; leaching time 120 min; stirring speed 400 rpm)

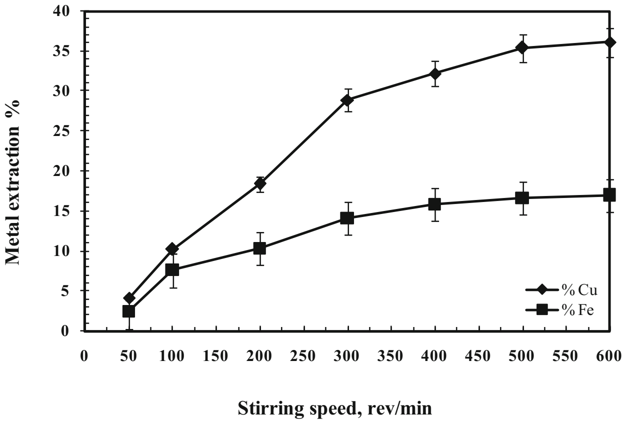


Fig. 9 Effect of stirring speed (1.2 M H_2O_2; 3 M HCOOH; L/S ratio 20 ml/g; leaching time 120 min)

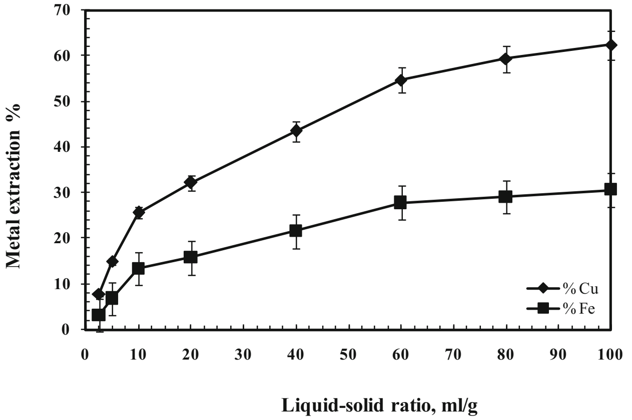


Fig. 10 Effect of liquid/solid ratio (1.2 M H_2O_2; 3 M HCOOH; leaching time 120 min; stirring speed 500 rpm)

increase in L/S ratio, and maximum leaching efficiencies of copper and iron are found to be 62.37% and 30.60% in 100 L/S ratio, respectively. It may be due to the fact that the amount of leach reactant per unit chalcopyrite concentrate is increased.

4 Conclusions

Due to the rapid degradation of hydrogen peroxide in leaching processes, the presence of formic acid in dissolution phenomenon is of great importance. Therefore, the utilization of formic acid with chalcopyrite concentrate decelerates the degradation rate of hydrogen peroxide in the solution and the leaching process is performed at lower liquid/solid ratio.

Based on the experimental results, it has been determined that the isothermal leaching conditions are maintained up to 2 M H_2O_2 concentration under the experimental conditions of L/S ratio of 20 and formic acid concentration of 5 M. Furthermore, the concentrations of formic acid higher than 3 M are not effective on the amount of copper and iron passing through the solution.

The maximum leaching yields of copper (30.2%) and iron (15.3%) are obtained under optimum experimental conditions; hydrogen peroxide concentration (1.2 M); formic acid concentration (3 M); temperature (40 °C) and time (120 min). On the other hand, it can be suggested that the reaction is quite fast in the presence of hydrogen peroxide taking into account that the maximum yield of copper is 8.9% after only 15 min under the same leaching conditions.

The experimental findings clearly demonstrate that the extraction yields of metals are not very effective on sulfuric acid concentration, leaching temperature and stirring speed. The optimum values of these parameters have been found to be 0 M H_2SO_4, 40 °C and 500 rpm, respectively. In addition, the results indicate that the leaching efficiency of copper highly depends on liquid/solid ratio and that the copper extraction value reaches from 7.83 to 62.37% for 2.5 and 100 L/S ratios, respectively. In conclusion, according to the experimental results, the formic acid may be utilized as an effective organic leaching agent in the leaching processes of other oxidized and sulfured ores containing precious metals such as copper, lead and zinc. For further studies, it may be advisable to leach chalcopyrite concentrate after mechanical activation for different periods owing to increased leaching efficiency.

Compliance with Ethical Standards

Conflict of interest The authors declare that they have no conflict of interest.

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