An Investigation on Electroleaching of Sar-Cheshmeh Copper Sulfide Concentrate

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ABSTRACT

The electroleaching of Sar-Cheshmeh copper sulfide concentrate in aqueous sulfuric media has been investigated under the influential parameters as; current density, leaching time, sulfuric acid concentration and temperature. The experiments were carried out in batch conditions and static bed status. Based on the illustrated results, concentration of copper in leaching solution increases strongly when electric current is applied. At the above-mentioned conditions the concentration of copper decreased while the electrical current is increased toward the maximum. On the other hand, the concentration of Fe²⁺ in leach liquor increases with increasing the current density. So, the irregular increasing of current density is not convenient to electroleaching of copper concentrate. The results showed that both anodic and cathodic reactions during the leaching process caused the copper concentration decreases at the long period of times, but iron concentration increases rapidly. It is also suggested that the deposition of copper on cathode surface during the leaching process "as a reduction reaction" causes the decreasing of the copper concentration in the solution. Decomposition of copper sulfides is an endothermic reaction, so the copper concentration of solution increases with increasing the temperature up to 80 °C. Increasing the temperature from 60 °C to 80 °C decreases the iron extraction during the process.

KEYWORDS

Electro-leaching, Sar-Cheshmeh, Thermodynamics, Kinetics

1 INTRODUCTION

About 90 percent of the world copper deposits are in the sulfide composition. The conventional extractive methods of copper are based pyrometallurgical processes. In all of pyrometallurgical methods, the sulfur content of the copper concentrate oxidizes to SO₂ and could cause air pollution. One of the most important advantages of hydrometallurgical processes is to reduce the air pollution.

Anodic treatment of copper matte was first patented in [1], but despite many attempts since then commercial operation was only achieved recently, in the Cymet process for the electroleaching of chalcopyrite concentrate [2-3].

A quick review of the proposed hydrometallurgical treatment methods for chalcopyrite and other copper sulfides [4-13] shows that they are generally either in sulfate or chloride media. Processes in sulfate media include acid pressure leaching, ferric sulfate leaching,

bacterial leaching, and others processes in chloride media include ferric chloride leaching, cupric chloride leaching, chlorine and hydrochloric acid leaching, and others Cyanide, ammoniac, and nitric acid leaching processes are also reported in the open literature.

In the previous works, leaching and electro-oxidation of the pure chalcopyrite, covellite and chalcocite under the chloride media conditions have been described. According to these investigations, electrode reactions of cuprous sulfide [14-17], white metal [18] and chalcopyrite [4, 14, 19-20] have been performed. Jones and Peters [20] have studied pure chalcopyrite leaching in 1 M FeC13, 0.1 M HC1 solution at 90 °C, observing that almost a hundred percent of chalcopyrite reacted following half-cell reaction (1) as:

$$CuFeS2 +4 FeCI2+ +3 CI \longrightarrow CuCI+ +5 FeCI2 (aq) +2 So$$
 (1)

The anodic oxidation of chalcopyrite in chloride medium was associated with a 0.26 V (SHE) potential by Ammon-Chokroum et al. [21-22], whom again noted two stages of



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electrooxidation. The first stage occurred in 90 minutes; the data adjust to the relationship $i_{\bullet} \times \sqrt{t} = \text{constant}$, which is characteristic of a diffusion-controlled process. On the other hand, the second stage "with little affecting" by the anodic potential level with the assumption of allowed chemically controlled process. The product of the second stage could be further electro-oxidized in the next. The final proposition for the mechanism consists of an acidbase reaction on the chalcopyrite-sulfur layer interface.

CuFeS₂ + 4 H⁺
$$\longrightarrow$$
 Cu²⁺ + Fe²⁺ + 2 H₂S (2)

followed by cupric sulfide precipitation

$$Cu^{2+} + H_2S \longrightarrow CuS + 2 H^+$$
 (3)

with the overall reaction corresponding to

$$CuFeS_2 + 2H^+ \longrightarrow CuS + Fe^{2+} + H_2S$$
 (4)

Reaction (4) is well known to be slow; hence it would control the second stage of electrooxidation reaction. The previously formed cupric sulfide diffuses through the pores of the elementary sulfur layer reaching the electrolyte, where it is finally oxidized anodically as in below reaction:

$$CuS \longrightarrow Cu^{2+} + S^{\circ} + 2 e^{-}$$
 (5)

Price and Chilton [14] investigated the electroleaching of bornite and chalcopyrite. They focused their research on effects of temperature, solution component and variation in the current density. They used both H₂SO₄ and HCl in electroleaching solutions. Járuegui and Reyes [19], studied the anodic electro oxidation of copper concentration in two electrolytic media, the first consisting of a solution of 0.5 M Fe3+, 0.1 M HCl and 2.0 M total Cl', the second, a solution of 0.5 M Cu²⁺, 0.1 M HCl and 2.0 M total Cl.

This work has focused on the electroleaching of Sar-Cheshmeh copper sulfur concentrate at sulfuric media, attempting to achieve a technical evaluation of its potential.

EXPERIMENTAL AND METHODS

2.1 Materials

The copper concentrate was prepared from Iranian national copper industries, Sar-Cheshmeh copper mine (Sarcheshmeh, Kerman, Iran). Chemical analysis of this concentration is given in Table 1. The sulfuric acid, hydrochloric acid and ammonium solution of analytical grade was used from Baran Company of Tehran, Iran. All of reagents and indicators same as stannous (II) chloride, mercury (II) chloride, potassium dichromate and diphenillamine-4-sulfonic acid barium salt were prepared from Merck chemical company.

2.2 Experimental procedure

Experiments were carried out in batch and static bed settings. In all of the experiments, 60 gr of copper concentrate were leached with 400 mL sulfuric acid solution at the same concentrations and temperatures. The

electroleaching apparatus is shown in Fig. 1. In this conditions, the concentrate fixed around anode in a bag

TABLE 1- CHEMICAL ANALYSIS OF SAR-CHESHMEH COPPER CONCENTE ATE

CONCENTRATE.						
	Cu	Fe	S	SiO_2	CaO	Al ₂ O ₃
(wt%)	30	26.5	31.3	5.4	0	4.7

filters. In order to controlling the temperature during the experiments, a circulating water bath was used at ± 1 °C. The effect of current density on leaching kinetics, was studied in fixed initial leaching solution concentration varied from 3 to 1 gr. of sulfuric acid /L at room temperature, respectively. The effect of acid concentration on leaching kinetics was studied at room temperature and optimum current density. The effect of temperature on leaching kinetics was studied at optimum acid concentration and current density. In all experiments, to avoid the solution vaporization during the experiments, a lid was used for cell especially in high temperatures.

Copper concentration of the solution was determined with spectrophotometery method. These measurements out with carried a Unicam UV/VIS spectrophotometer, 8700 series in visible wave light $\lambda_{\text{max}} = 655$ nm. Iron concentration of the solution was determined with titration method with potassium dichromate as titration agent in the presence of SnCl₂ and HgCl₂ as reduction agent and diphenillamine-4-sulfonic acid barium salt as indicator.

RESULT AND DISCUSSION

3.1 Effect of leaching time

The changes of the copper concentration in leach liquor for different current densities at constant sulfuric acid concentration 1 gr/L and temperature of 25 °C are illustrated in Fig. 2-a. As shown in Fig. 2-a, if the current density is high enough, the copper concentration of leaching solution both increases and decreases during the time. In lower the current densities, according to this figures, the increasing of current density from 160 up to 200 Amp/m² does not have important effect on copper concentration at the leaching solution. The both increasing and decreasing the copper concentration in leaching solution can be caused by the following reasons, simultaneously:

- 1- Copper concentration in leach liquor increased because of occurring the both acid - base reactions and oxidation condition of the system, especially at the anode surface.
- 2- Electrical current caused deposing the copper as a noble ion on cathode surface in the solution. According to this reducing reaction, copper concentration in solution

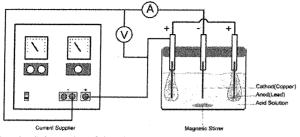
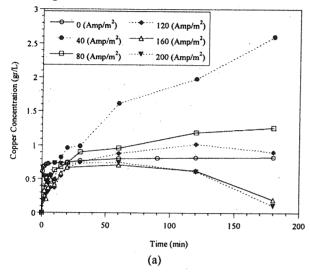


Fig. 1- Schematic of batch electroleaching apparatus used for this investigation.



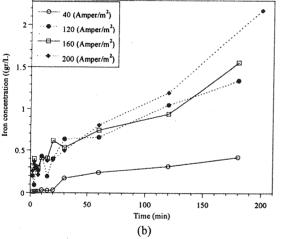


Fig. 2- Effect of time on extraction of metals at different current densities for static bed condition, (a) copper, (b) iron.

decreases consequently. According to Faraday law, the weight of deposed copper on cathode surface increases linearly by time at constant current density.

The external electrical potential causes many anodic reactions. Some of these reactions are illustrated in Table 2. Since, the external electrical potential causes the increasing of the copper concentration in leach liquor, however, the cathodic reactions decrease the copper concentration in leaching solution in the same way. So the external electrical current does not increase the copper concentration at leaching solution, effectively, but within tabulated reactions, the oxidation

 $(Fe^{+2} \longrightarrow Fe^{3+} + e^{-})$ could be very effective. In this case, the Fe³⁺ ions can decompose the chalcopyrite according to following reaction:

$$CuFeS_2 + Fe^{3+} \longrightarrow Cu^{2+} + FeS_2 + Fe^{2+} + e^{-} \quad \varepsilon^{\circ} = -1.43 \quad (6)$$

So, at the anode surface, according to reaction 6, one mol copper ion would be formed and one mol electron would be produced. However, at the cathode surface, to reduce one mol Cu⁺² ions to one mol metallic copper,

TABLE 2- ANODIC HALF-CELL REACTIONS.

Anodic half-cell reaction	ΔG° (J)	ε° (volts)
$Cu_2S \rightarrow 2 Cu^{2+} + S^{\circ} + 4 e^{-}$	216301	-0.560
$CuS \rightarrow Cu^{2+} + S^{\circ} + 2 e^{-}$	119150	-0.617
$CuFeS_2 \rightarrow Cu^{2+} + Fe^{2+} + 2S^{\circ} + 4e^{-}$	287653	-0.745
$CuFeS_2 \rightarrow Cu^{2+} + Fe^{3+} + 2S^{\circ} + 5e^{-}$	362142	-0.751
$Fe^{2+} \rightarrow Fe^{3+} + e^{-}$	74408	-0.771
$2\text{CuS+Fe}^{2+} \rightarrow 2\text{Cu}^{2+} + \text{FeS}_2 + 2\text{e}^{-}$	163134	-0.845
$CuFeS_2 \rightarrow Cu^{2+} + FeS_2 + 2 e^{-}$	212496	-1.101
$CuFeS_2 \rightarrow Cu^{2+} + FeS + S^{\circ} + 2e^{-}$	270606	-1.402
$CuFeS_2 + Fe^{3+} \rightarrow$ $Cu^{2+} + FeS_2 + Fe^{2+} + e^{-}$	138007	-1.430

two mol electrons is needed. Therefore, according to this mechanism, the rate of copper decomposition in leaching solution could be more than that of its deposition. At the mentioned condition, the copper ion diffusion into the solid state "chalcopyrite" could control the reaction rate. There could be similar reactions between Fe3+ and other copper sulfides. According to the following reaction, the dissolved oxygen in acidic media could oxidize the Fe2+ ions to the Fe³⁺ ions:

2 FeSO₄ + H₂SO₄ +
$$\frac{1}{2}$$
 O₂ \longrightarrow Fe₂(SO₄)₃ + H₂O (7)

and the Fe3+, again with oxygen leaches the copper sulfide as:

$$Fe_2(SO_4)_3 + Cu_2S + 2O_2 \longrightarrow 2FeSO_4 + 2CuSO_4$$
 (8)

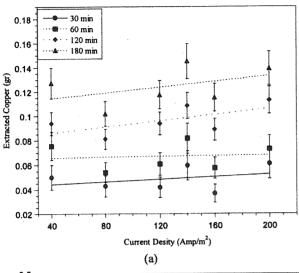
As the mixing increases, the quantity of dissolved oxygen in solution could be increased. On the other hand, the concentration of Fe⁺² is extremely more than Fe⁺³ ions, so the reactions 7 and then 8 lean to the right side.

The concentration of iron in leaching solution due to current density is shown in Fig. 2-b. The increasing of current density could also increase the iron concentration in leaching solution. The increasing iron concentration is very clear at long time periods. In the case of increasing the current density, the iron concentration would be higher respects to that of lower the current density.

Effect of current density

It should be mentioned that the main purpose of this research was electroleaching of copper (not its electrowining), so the reduction of copper on cathode

surface is not convenient. The amount of total extracted copper (i.e., the amount of copper in leach liquor and reduced copper on cathode surface) vs. current densities at constant sulfuric acid concentration 1 gr/L and temperature 25 °C were shown in Fig. 3-a. As illustrated in Fig. 3-a, increasing of current density (with error bar equal to 10% of measured value) has not important effect on the amount of total extracted copper.



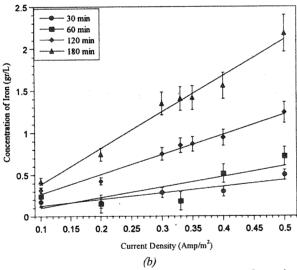


Fig. 3- Effect of current density on extraction of metals at different times for static bed condition, (a) copper, (b) iron.

The iron concentration changes in leaching solution vs.current densities at the same condition (Fig. 3-a) were shown in Fig. 3-b. It is clear that the concentration of iron in leached liquor increases as a function of current density.

Comparing Figs. 2-a and 3-a, it seems that during the electroleaching the increasing of current density at different times does not make essential difference on the amount of total extracted copper. Based on Figures 2-b and 3-b it seems that the increasing of current density raises the iron concentration in leach liquor. So, increasing the current density not only dose not affect electroleaching of copper but increases the concentration of iron in

leaching solution severely.

3.3 Energy consumption and cathodic efficiency

The consumption of electrical energy in an electrical process can be determined by:

$$Q = \int V I dt$$
 (9)

Since, during the electroleaching process the external current density was constant, so by measuring the electric potential of the system "within the time", the electrical energy consumption can be achieved. The energy consumption vs. time in different current densities at constant sulfuric acid concentration 1 gr/L and temperature 25 °C was shown in Fig. 4. As it is shown in this figure, the consumption of energy increases linearly vs. the time that it could be due to constant electric potential during the electroleaching. Also, the energy consumption increases extremely by increasing the current density. As in Fig. 3-a, the increasing of current density causes small change in copper electroleaching. In fact, excessive energy is wasted during the iron decomposition in anode that is not desired.

The cathodic efficiency changes based on current density after 180 minute is shown in Fig. 5. As shown in Figure 5, the cathodic efficiency decreases due to increasing the current density. It seems that in higher current densities, another cathodic reaction (like $Fe^{3+} + e \longrightarrow Fe^{2+}$) will be appeared at cathode surface. At higher current densities, especially in longer period of electroleaching process, the copper

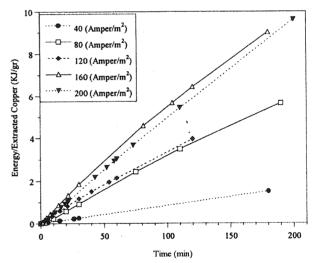


Fig. 4- Energy consumption for different current densities for static bed condition.

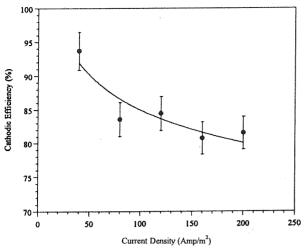
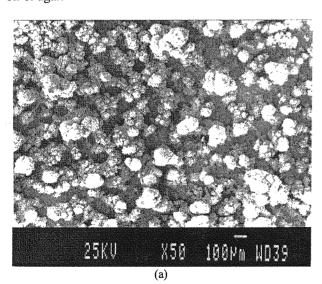


Fig. 5- Effect of current density on cathodic efficiency for static bed condition.

concentration in leaching solution would be decreased. Otherwise, in acidic solution, at the following conditions, the hydrogen ion concentration increases with respect to the copper, and based on thermodynamics, hydrogen would be reduced instead of copper. Based on reaction kinetics, it is clear that at the high current density, hydrogen over voltage on cathode surface would be supported and then it reduces at the surface of the cathode. So, increasing the current density has not any advantage in leaching of copper, so, the current density equal to 40 Amp/m² has been chosen and used for the next steps "as optimum current density".

Figure 6 is showing the SEM images of cathode surface. As it is shown in Fig. 6, the reduced copper is in dendrite structure. During the experiment, a small amount of concentrate particles pass through the concentrate sacks, these particles suspended in leach liquor and may trap among dendrite arms. So, to extract pure copper a secondary refining process is necessary. This problem could be solved by using the smoother materials, e.g., pine oil or agar.



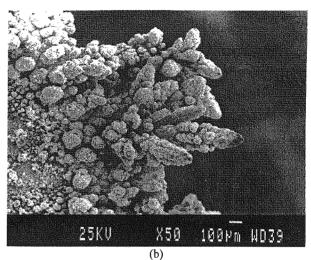
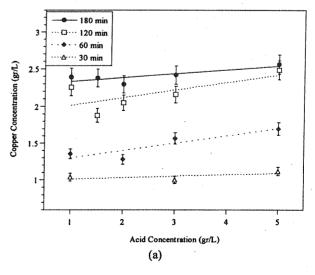


Fig. 6. SEM images of cathode surface (a) surface of cathode plate (b) at the edge of cathode plate.

3.4 Effect of sulfuric acid concentration

The changes of the copper concentration in leaching solution due to acid concentration for different period of electroleaching process (with error bar equal 5% of measured value) are shown in Fig. 7–a. As a result of this Figure, the increasing of acid concentration in leaching solution has not any important effect on copper concentration in leach liquor. Otherwise, the changes of iron concentration in leaching solution vs. acid concentration is shown in Fig. 7–b. It is clear that the iron concentration in leach liquor increases rapidly with the initial sulfuric acid concentration of more than 2 gr/L.

The change of consumption of energy to produce one gram of copper at different acid concentration is shown in Fig. 8. As it is shown in Fig. 8, the energy consumption at different leach liquor (with the initial acid concentration of one to two gr/L) are almost similar, but the energy consumption, decreases in solution with increasing the concentration of the acid. The electrical potential between anode and cathode decreases as a result of increasing the electric conductivity "due to the increasing ion



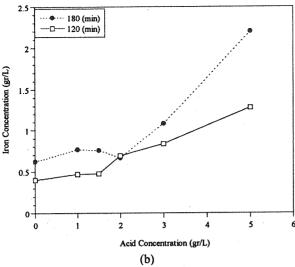


Fig. 7- Effect of acid concentration on extraction of metals at different times for static bed condition, (a) copper, (b) iron.

concentration in leaching solution". Thus, the energy consumption decreases with increasing concentration in the initial leaching solution.

Although, the energy consumption is reduced in the condition of high acid concentration, the iron concentration in leach liquor increased severely. In the high acid concentration, the iron concentration increases severely that makes some problems for the next stages of copper extraction. Thus, a solution with the concentration of 1.5 gr/L of sulfuric acid for the next stage was selescted.

3.5 Effect of temperature

Copper concentration changes in the leach liquor with time at different temperatures are shown in Fig. 9-a. As it is shown in Fig. 9-a, the copper concentration in the leaching solution increases with increasing temperature up to 80 °C. Also, the increasing temperature effect on copper concentration in the leach liquor decreases at high temperatures, especially at longer period of electroleaching. According to the Le Chatelier rule, it can be suggested that the copper sulfides decomposition during the electroleaching is an endothermic reaction.

The iron concentration change in the leaching liquor according to the temperature fluctuations is shown in Fig. 9-b. It is clear that, the iron concentration in leaching solution at different intervals of time increases with increasing of temperature up to 60 °C, but it decreases severely at 80 °C. There are two thermodynamic reasons to explain these changes: 1) sever decreasing the Fe⁺³ solubility and, 2) increasing the affinity of the reaction; $Fe^{2+} \rightarrow Fe^{3+} + e^{-}$. Based on this reaction, the iron concentration in leaching solution reduces after a maximum value.

Figure 10 shows the changes of total extracted copper during the electroleaching vs temperature fluctuations at different intervals of time. As it can be seen in this figure,

the temperature increasing from 60 °C to 80 °C causes the increasing of total amount of extracted copper. Also, the

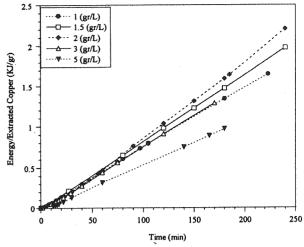
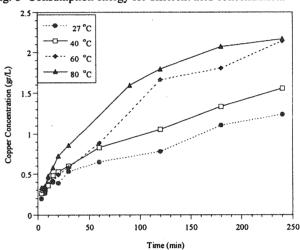


Fig. 8- Consumption energy for different acid concentration.



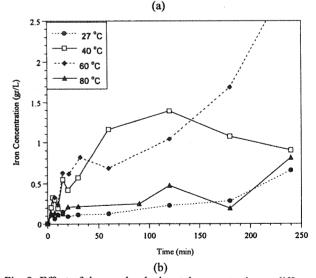


Fig. 9- Effect of time on leached metal concentration at different temperature static bed condition (b) Iron

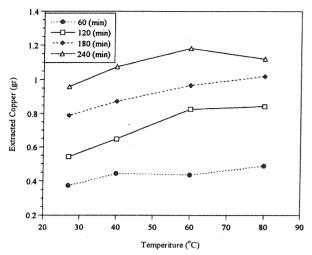


Fig. 10- Effect of temperature on leached the copper concentration at different times for static bed condition.

total amount of extracted copper after 240 minutes decreases with increasing the temperature from 60 $^{\circ}$ C to 80 $^{\circ}$ C.

By comparing Figs. 9 and 10, it is concluded that the of extraction mechanism of copper, electroleaching, is troubled when Fe⁺³ concentrations become less. So, in longer period of electroleaching at higher temperatures, the copper extraction almost will be prevented and the copper deposition on cathode surface will be continued (e.g., the concentration of copper in leaching solution would be decreased). The energy consumption to copper extraction at different temperatures is shown in Fig. 11. Based on this figure, although the increasing of temperature could cause the increasing of the ion mobility, it does not have any important effect on energy consumption for copper production. It is clear that at higher temperatures the external energy source is necessary to keep the temperature constant in the leaching system. Finally, the fixed temperature of 80 °C was chosen "as an optimum temperature for electroleaching".

4 CONCLUSION

The results shows that the copper concentration in solution increases in the presence of electrical currents. This increasing of copper concentration in leach liquor can be due to the increasing of oxidation intensity on the anode surface.

At this condition, if the current density is high enough, the copper concentration of leaching solution both increases and decreases during the time. In lower current densities, the copper concentration of leach liquor is more than those for fluidized bed condition. It also seems that the two simultaneous steps happen similar to that of fluidized bed condition. As long as, the first step is more effective than the second one, the copper concentration in leaching solution increases, otherwise it decreases. The increasing of current density could also increase the iron concentration in leaching solution. In the case of

increasing the current density, the iron concentration would be higher respects to that of lower the current density. At the static bed conditions, the increasing of current density dose not have an important effect on the

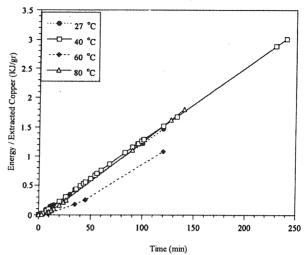


Fig. 11- Effect of time on consumption energy at different temperature for static bed condition.

amount of total extracted copper. It is clear that the concentration of iron in leach liquor increases as a function of current density.

The energy consumption increases extremely by increasing the current density. As mentioned above, the increasing of current density causes small changes in copper electroleaching. In fact, additional energy spent for iron decomposition in anode is not desirable. By increasing of current density, the cathodic efficiency changes after 180 minute. It seems that at higher current densities, another cathodic reaction (like $Fe^{3+} + e \rightarrow Fe^{2+}$) runs at cathode surface.

The increasing of acid concentration in leaching solution dose not has any important effect on concentration of copper in leach liquor. It is clear that the concentration of iron in leach liquor increases rapidly in leaching solution with initial sulfuric acid concentration more than 2 gr/L.

The energy consumption at different leach liquor (initial acid concentration between one to two gr/L) is almost the same, but consumption of energy decreases in solutions with higher acid concentration. Although the consumption of energy reduces in high acid concentration solutions, it increases the iron concentration in leach liquor severely. In higher acid concentration, the iron concentration increases severely that makes many problems for next stages of copper extraction.

The concentration of copper in leaching solution increases with rising of temperature up to 80 °C. Also, the effect of increasing of temperature on concentration of copper in leach liquor decreases on higher temperatures especially at longer period of electroleaching. The iron concentration in leaching solution increases with increasing of temperature up to 60 °C, but it decreases

severely at 80 °C. There are two thermodynamic reasons could cause these changes: 1) strong decreasing of solubility of Fe⁺³ and 2) increasing of affinity of reaction $Fe^{2+} \longrightarrow Fe^{3+} + e^{-}$. As a result of these two reasons, the concentration of iron in leaching solution reduces after a maximum. It is clear that, although the increasing of temperature increases the mobility of ions, the increasing of temperature dose not have any important effect on consumption of energy for production of copper.

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