

STUDY ON THE JAROSITE MEDIATED BY BIOLEACHING OF PYRRHOTITE USING *Acidithiobacillus FERROOXIDANS*

ESTUDO SOBRE A JAROSITA MEDIADA PELA BIOLIXIVIAÇÃO DA PIRROTITA USANDO *Acidithiobacillus FERROOXIDANS*

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ABSTRACT: Precipitation of jarosite is a very important phenomenon that is observed in the bioleaching of pyrrhotite by *Acidithiobacillus ferrooxidans* (*A. ferrooxidans*). Jarosite is a major secondary mineral formed in acid supergene environment by oxidation of metal sulphide. The formation of jarosite could decrease leached percentage. The Eh-pH diagram of $\text{FeS}_{1.12}\text{-H}_2\text{O}$ showed that the thermodynamic parameters of the jarosite were exists steadily on $\text{Eh}=360 \sim 800$, $\text{pH}=2.8 \sim 5$, and the results of pH condition test is consonant with the conclusions of thermodynamic analysis. By means of XRD and SEM, it could indicate that full propagation of *A. ferrooxidans* in the solution is beneficial to the formation of jarosite and jarosite mediated by bacterial has a better crystalline form than that synthesized by chemical method. This study indicates that pH value and ferrous/ferric iron concentration are key factors affecting the formation of jarosite. Leached percentage is higher when frequency was set more than 2.0. It is crucial to minimize jarosite formation in order to increase factory's efficiency.

KEYWORDS: Pyrrhotite. Bioleaching. *Acidithiobacillus ferrooxidans*. Jarosite.

INTRODUCTION

Jarosite is a major secondary mineral formed in acid supergene environment by oxidation of metal sulphide. *Acidithiobacillus ferrooxidans* (*A. ferrooxidans*) occurred extensively in metal sulphide ore district that has an important effect on oxidation of metal sulphide and formation of secondary minerals (ZHANG et al., 2013, ZHU et al., 2005). However, the depositing process of jarosite during biological metallurgy of sulfide ore has more harmful impact. Jarosite formation has negative effects on many applications that require the use of *A. ferrooxidans*, especially in the biological process of gas desulphurization and biological metallurgy of sulfide ore (JENSEN et al., 1995). Also, jarosite formation in coal desulfurization results in residual sulfur, which cannot be removed from coal (DAOUD et al., 2006). So it is crucial to minimize jarosite formation in order to increase efficiency and it has enlightenment function to practical industry production.

There are several factors that play a role in rate of oxidation of ferrous ions by *A. ferrooxidans*. These factors include ferrous/ferric iron concentration, cell and oxygen concentrations, pH, temperature and reactor type. Moreover, the pH has significant effects on the formation of jarosite which produced during bioleaching of pyrrhotite (DAOUD

et al., 2006, GU et al., 2013, POGLIANI et al., 2000, SONDI et al., 2001).

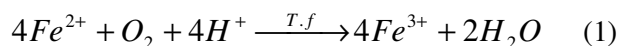
Several studies have been conducted in order to determine the importance of jarosite formation in bioreactors. But their same method is that studying on the jarosite during cultivation of bacteria (GAO et al., 2007, GU et al., 2013, POGLIANI et al., 2000, ZHENG et al., 2005, ZHU et al., 2005), such as *A. ferrooxidans* and *L. ferrooxidans*. Nevertheless, no experience on the production of jarosite by bio-metallurgy of sulfide ore that has been studied.

The main goal of this study was to investigate jarosite precipitation under different conditions and analyze the formation of thermodynamics condition to mediate precipitation of jarosite, and this results will be very important for determining the environment of ore leaching for minimize jarosite precipitation. Besides, for revealing the role of *A. ferrooxidans* in natural process of jarosite formation more reasonably, this paper was designed for leaching ore experiment, analysis of chemical and biogenic cause on the jarosite, combining with XRD and SEM, and had a further research on the effect of *A. ferrooxidans* on the jarosite formation in natural process. Study on the deposit production in leaching process was more according to practical geochemistry process, and it provides reference for practical production.

MATERIAL AND METHODS

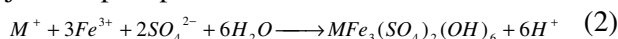
Jarosite formation

A. ferrooxidans can obtain energy from the oxidation of different inorganic substances, the most common substance is ferrous sulfate. The overall biochemical reaction of the oxidation of ferrous ions is



This bioreaction took a great part in practical process (DAOUD et al., 2006).

Since there is consumption of hydrogen ions, the pH of the liquid media initially increased. However, while this pH was increased, counteracted by the hydrolysis of ferric iron. Therefore, it is quite visible that the pH of the system has an effect on the extent of the oxidation and hydrolysis reactions. Furthermore, there is a reaction in competition with the hydrolysis reaction giving products of basic ferric hydroxysulphates with the formula $MFe_3(SO_4)_2(OH)_6$, where $M = K^+, Na^+, NH_4^+, Ag^+$, or $H_3O^{+5,6}$. These hydroxysulphate precipitates were known as jarosites. The following is the formula for jarosite precipitation:



Since the 9K medium or leaching solution contains a high concentration of NH_4^+ ions, the jarosites were produced in ammoniojarosites form with formula $NH_4Fe_3(SO_4)_2(OH)_6$ (WANG et al., 2005, WANG et al., 2006).

Thermodynamics analysis

Eh-pH diagram of $FeS_{1.12}$ - H_2O system and it's analysis

Thermodynamic analysis during the process of bioleaching sulfide minerals is one of the most important theoretical analysis. Through thermodynamic reaction analysis during the leaching process of sulfide minerals, this study is to discuss about the condition of thermodynamic during bioleaching sulfide minerals. The general thermodynamics laws of the process were analyzed by using the Eh-pH diagram of $FeS_{1.12}$ - H_2O system (QIN et al., 1999).

The chemical formula of pyrrhotite is $FeS_{1.12}$, all kinds of the iron compounds and sulfur compounds exist in bioleaching of pyrrhotite system. According to the Nernst equation, the possible equilibrium equations of reaction in the $FeS_{1.12}$ - H_2O system have been obtained which are showed in the Table 1.

Table 1. The possible chemical equations of pyrrhotite.

NO.	Reaction Equation	Potential Equations.
a	$2H^+ + e = H_2$	$E = -0.059pH$
b	$O_2 + 4H^+ + 4e = 2H_2O$	$E = 1.229 - 0.059pH$
1	$S + 2H^+ + 2e = H_2S$	$E = 0.142 - 0.059pH$
2	$HSO_4^- + 7H^+ + 6e = S + 4H_2O$	$E = 0.338 - 0.069pH$
3	$SO_4^{2-} + 8H^+ + 6e = S + 4H_2O$	$E = 0.357 - 0.079pH$
4	$SO_4^{2-} + H^+ = HSO_4^-$	$pH = 1.9$
5	$Fe^{3+} + e = Fe^{2+}$	$E = 0.770$
6	$Fe(OH)_3 + 3H^+ = Fe^{3+} + 3H_2O$	$pH = 1.72$
7	$Fe(OH)_3 + 3H^+ + e = Fe^{2+} + 3H_2O$	$E = 1.02 - 0.146pH$
8	$KFe_3(SO_4)_2(OH)_6 + 8H^+ = K^+ + 3Fe^{3+} + 2HSO_4^- + 6H_2O$	$pH = 2.80$
9	$KFe_3(SO_4)_2(OH)_6 + 8H^+ + 3e = K^+ + 3Fe^{2+} + 2HSO_4^- + 6H_2O$	$E = 0.672 - 0.157pH$
10	$KFe_3(SO_4)_2(OH)_6 + 6H^+ + 3e = K^+ + 3Fe^{2+} + 2SO_4^{2-} + 6H_2O$	$E = 0.597 - 0.118pH$
11	$2Fe^{2+} + 1.12SO_4^{2-} + 8.96H^+ + 7.72e = FeS_{1.12} + Fe^{3+} + 4.48H_2O$	$E = 0.208 - 0.068pH$
12	$2Fe^{2+} + 1.12HSO_4^- + 7.84H^+ + 7.72e = FeS_{1.12} + Fe^{3+} + 4.48H_2O$	$E = 0.191 - 0.060pH$
13	$Fe(OH)_3 + 1.12SO_4^{2-} + 11.96H^+ + 9.72e = FeS_{1.12} + 7.48H_2O$	$E = 0.366 - 0.037pH$
14	$Fe(OH)_3 + 1.12HSO_4^- + 10.84H^+ + 9.72e = FeS_{1.12} + 7.48H_2O$	$E = 0.456 - 0.084pH$

Calculation was using the parameter which is close to the leaching test system. Activities of aqueous sulphur species and iron species was $1.0\text{mol}\cdot\text{L}^{-1}$ and $0.01\text{mol}\cdot\text{L}^{-1}$, respectively. The reaction of bioleaching is usually in response on $\text{pH} = 1.5 \sim 4.5$, $E_h = 300 \sim 900\text{mv}$, so in the E_h - pH diagram, took the $\text{pH} = 0 \sim 5.0$, $E = -0.2 \sim 1.0\text{V}$. According to the parameters which we set in the test system based on the Portland's manual data, Fig. 1 showed relations among the equilibrium reaction (ZHANG, 2007).

The Figure 1 indicates that in the $\text{FeS}_{1.12}$ - H_2O system, the region which sulfur deposition exists steadily was composed by $\text{pH} = 0$, $\text{pH} = 5$, and 1, 2, 3 phase equilibrium lines. The sulfur mainly exist in the form of H_2S below the region and stable in HSO_4^- or SO_4^{2-} form above it. From the diagram, when the pH is in $1 \sim 5$ and the E_h is in $400 \sim 900\text{mV}$, the elemental sulfur can not exist steadily. But in the actual extraction process, redox reactions need to overcome a certain barrier, so sulfur maybe exists in this context.

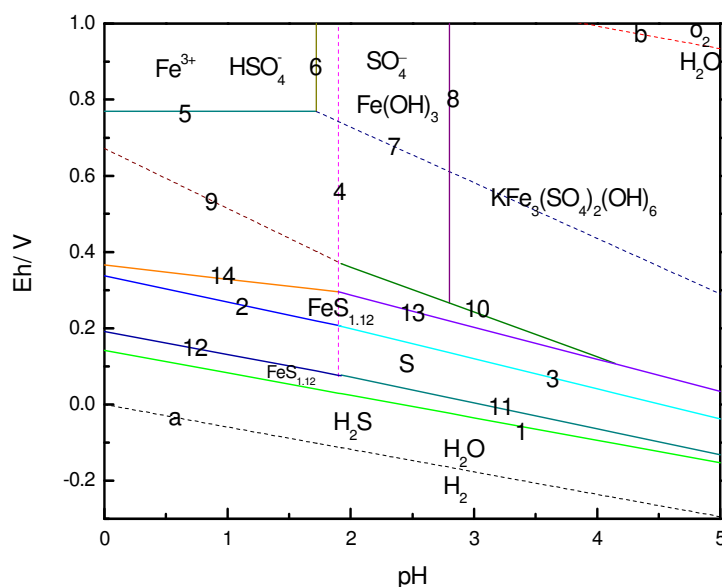


Figure 1. E_h - pH diagram for $\text{FeS}_{1.12}$ - H_2O system (298K, $1.013 \times 10^5 \text{Pa}$).

$$\{\alpha_{\text{Fe}^{2+}} = \alpha_{\text{Fe}^{3+}} = 0.01\text{mol} \cdot \text{L}^{-1}, \alpha_{\text{K}^+} = 0.001\text{mol} \cdot \text{L}^{-1}, \alpha_{\text{HSO}_4^-} = \alpha_{\text{SO}_4^{2-}} = \alpha_{\text{H}_2\text{S}} = 1.0\text{mol} \cdot \text{L}^{-1}\}$$

The region which Fe^{3+} exists steadily was enclosed by the lines of 5, 6 and the line of $\text{pH}=0$. So we can infer that Fe^{3+} exists steadily when the potential was above 770mv. When the Fe^{3+} reduced, the line 6 move to the right, and the region which Fe^{3+} exists steadily extend to the right. In practical leaching system, when pH is in $2.28 \sim 4.0$, E_h is in $360 \sim 800\text{mv}$, Fe^{3+} may be mainly exist in the form of precipitates¹¹.

The Fig. 1 shows that the region which $\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$ exists steadily was composed by 8, 10, 13 phase equilibrium lines and the line of $\text{pH}=5$. The stability region was above 360mv. $\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6$ generates and exists steadily when the pH is more than 2.80. Thus it is concluded that the thermodynamic parameters of the $\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6$ exists steadily were at $E_h=360 \sim 800$, $\text{pH}=2.8 \sim 5$. This results was also consonant with the the nether experiment of pH condition.

From the diagram, the regions $\text{FeS}_{1.12}$ that exists steadily have two parts, one part was composed by the phase equilibrium lines of 2, 3, 13, 14 and the lines of $\text{pH}=0$, $\text{pH}=5$, the other part was composed by the equilibrium lines of 1, 11, 12 and the lines $\text{pH}=0$, $\text{pH}=5$. The stability region was below 300mv and above the equilibrium line of $\text{H}_2\text{O}/\text{H}_2$, that can be said to be stable in aqueous solution. From the two-phase equilibrium lines, we can get that the products and the intermediate products were $\text{Fe}(\text{OH})_3$, Fe^{2+} , S , H_2S , HSO_4^- and SO_4^{2-} (OU et al., 2007).

Experimental analysis

Equipment

Based on procedure, the bacteria were grown in 11×250 ml Erlenmeyer flasks with the appropriate medium. The pH of each trial was adjusted using sulfuric acid and monitored using a pH meter (PH METER MODEL PHS-25). The

bacteria were allowed to grow in a Rotary flask shaker with speed and temperature adjustment (New Brunswick Scientific Model No. HZD-C). For the ferric and total iron analyses, we used a spectrophotometer (U-Varian 9200) with the appropriate procedure. In order to detect compounds produced during bioleaching of pyrrhotite, at the end of experiment, filter paper with pore size 45µm was used to separate the jarosite produced on the wall of each flask., and air dried for X-ray diffraction(XRD), scanning electron microscopy(SEM) and energy spectrum analysis(EDS).

Chemicals and metallic minerals

All the chemicals used in this study were of analytical grade, including 10% and 20% H_2SO_4 , the components of the 9K medium are given in Table 2. *A. ferrooxidans* which we used is supply by School of Minerals Processing & Bioengineering, Central South University. The grade of Fe in the pyrrhotite which used in these experiments is 57.32% and particle size is -0.043mm, the XRD of pyrrhotite is shown in Fig. 2 .It can be seen that except magnetic pyrite, it also has a few extraneous components, such as quartz, metasilicate and so on.

Table 2. Components of the 9K medium

Components	Contents
$(\text{NH}_4)_2\text{SO}_4$	3.0g
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	0.5g
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	44.2g
K_2HPO_4	0.5g
$\text{Ca}(\text{NO}_3)_2$	0.01g
KCl	0.1g
5 mol/l H_2SO_4	1.0ml to pH=1.8
Dist. H_2O	1000ml

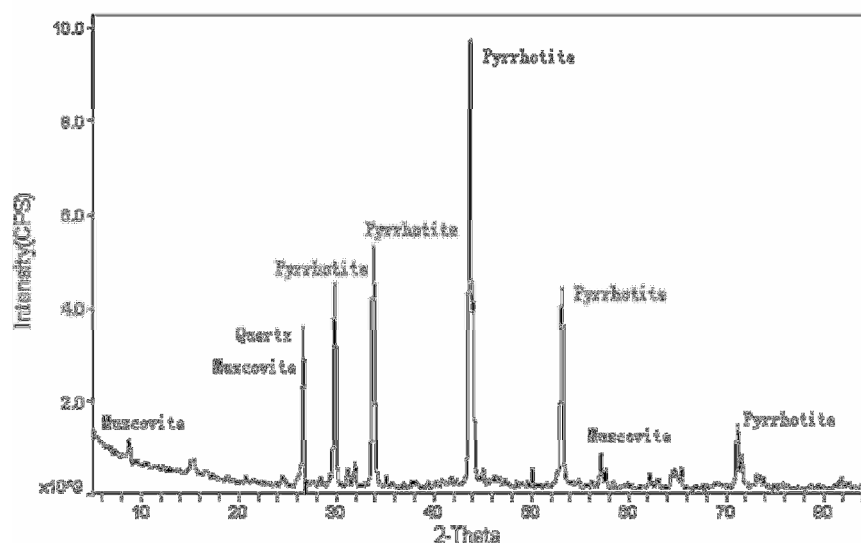


Figure 2. XRD of pyrrhotite which we used in this experimentation

Experimental method

This experiment deals with the effect of pH and the effect of *A. ferrooxidans* on the formation of jarosite. Therefore, the experiment was divided into two sections, dealing with varying pH and effect on the formation of *A. ferrooxidans*.

On this section, we discuss the effect of *A. ferrooxidans* on the formation of jarosite and analyze the different morphology of jarosite-group mediated from biologically and chemically (experiment I and II) condition and the effect of

varying pH on jarosite formation (experiment II and III). As shown in Table 3, three 250 ml Erlenmeyer flasks were used, the number for each flask is I, II and III. 150 ml of 9K medium and 5g of pyrrhotite were added into each of 3 flasks. pH adjustment in each flask was obtained by adding 20% H_2SO_4 dropwise, with continuous agitation and pH measurement. Furthermore, 15ml of *A. ferrooxidans* inoculum was added to II and III flasks. The inoculum contained an average of 8×10^7 cells per milliliter. The flasks were then covered with pieces

of aluminum foil. Finally, the flasks were placed in the rotary shaker at ambient temperature with

170rpm rotation speed.

Table 3. Experimental conditions in leaching of pyrrhotite

Number	Reactants	T/°C	Original pH	Experimental method
I	9K, pyrrhotite	30	2.0	Set solution pH value to 2.0 every two days
II	9K, 10ml <i>A. ferrooxidans</i> , 5g pyrrhotite	30	2.0	Set solution pH value to 2.0 every two days
III	9K, 10ml <i>A. ferrooxidans</i> , 5g pyrrhotite	30	2.0	Set solution pH value to 2.0 every three days

RESULTS AND DISCUSSION

This experiment consisted of two parts of analysis, which are the effect of pH and the difference of the jarosite-group precipitates between biologically and chemically jarosite precipitation. For each parameter, we will discuss the data and observations.

Table 2 shows that Experiment I and Experiment II is a group of comparable parallel experiment. Corresponding to the course of ore leaching, the former is the chemosynthesis producing jarosite, the latter is ore leaching producing jarosite using *A. ferrooxidans* in the same condition (SASAKI et al., 2000, ZHOU et al., 2004). In this experiment, after adding to reaction system the pyrrhotite appeared opaque deposit in the fifth day, and in the tenth day its color became deeper and turned into brownish yellow. They commonly exist in the experiment. The solid substance, that were collected from Experiment I and Experiment II at last, is mainly jarosite deposit

showed by X-ray diffractive and EDS analysis (Fig. 3). By observing the deposit of Experiment I and II separately with SEM and EDS (Fig. 4) (DING et al., 2007, XIA et al., 2008), we can see that jarosite crystal produced in the course of acid-leaching (Experiment I) developed incompletely, had hollow holes on its surface, and its deposit was irregularly round. It may be amorphous substance; however producing deposit of bioleaching process (Experiment II) was comparatively pure jarosite, and its crystal was regular rhombohedron and leveling the crystal face. Therefore, it is different between producing deposit and crystal degree in biological process and chemical process. Compared to the nonsterile with sterile result, we can infer that the Fe^{2+} fast transform by *A. ferrooxidans* and can cause the supply rate of Fe^{3+} increase in mineral surface micro area, and resulted in faster formation of jarosite crystals (GAO et al., 2007). Therefore, we got a further understanding effect of *A. ferrooxidans* on the jarosite formation in natural process.

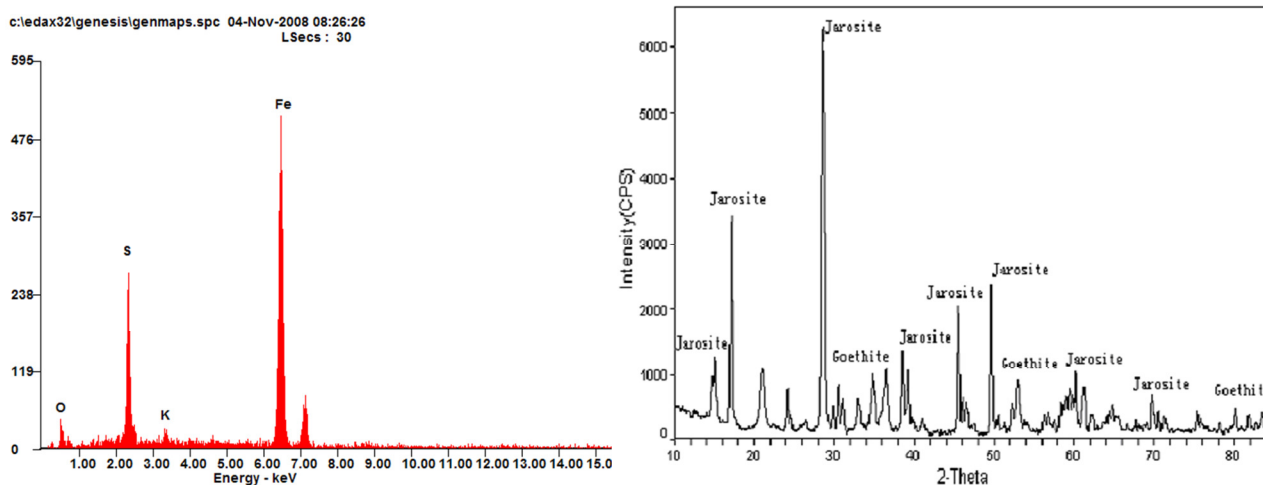


Figure 3. SEM and EDS of the jarosites from experiments I and II

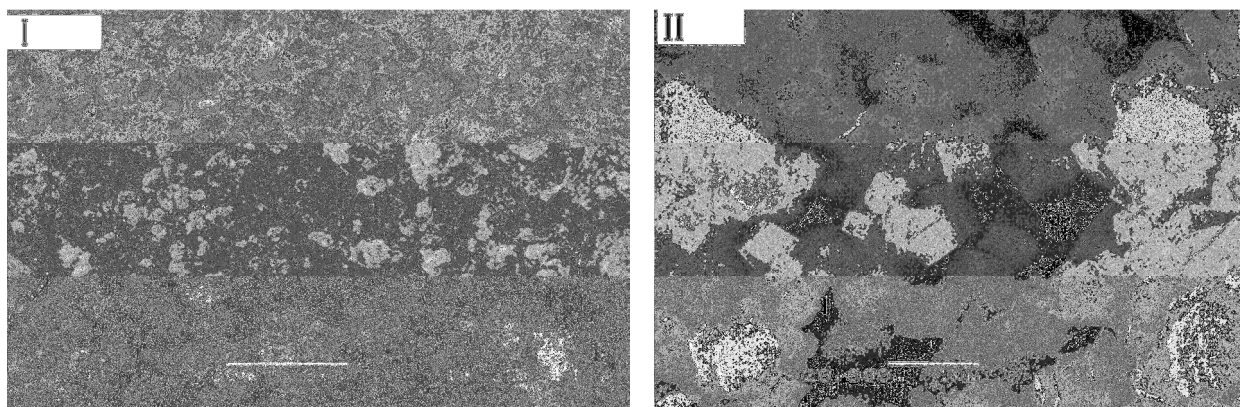


Figure 4. SEM of the jarosites from experiments I and II

Experiment II and Experiment III were also group of comparable experiment. The differences is only in regulating frequency of pH condition. Because pH value determines Fe^{3+} hydrolysis process directly, that it is decides whether the precursor of forming jarosite is formed, pH has a significant effect on the deposit forming rate. The research shows that the oxidation of pyrrhotite oxidation was fondness for acidic process, which pH value range from 1.8 to 2.8 the pyrrhotite produce less deposit during the process of leaching. So, when the adjustable frequency of pH value was higher, deposit production will be reduced. Therefore, the deposit produced in the Experiment

II was less than Experiment III, and because of jarosite deposit produced in the oxidation process from Fe^{2+} to Fe^{3+} by *A. ferrooxidans*. Formation of jarosite lost part of Fe^{3+} , because Fe^{3+} involved in jarosite deposit, in the biological oxidation of pyrrhotite, total concentration of Fe^{3+} solution is less than real concentration, seriously affected the leaching rate (JIANG et al., 2007). The total concentration of Fe^{3+} leaching solution from Experiment II and Experiment III was assayed separately every fourth days, we obtained the change curve of leaching rate, shown on Figure 5 as follow.

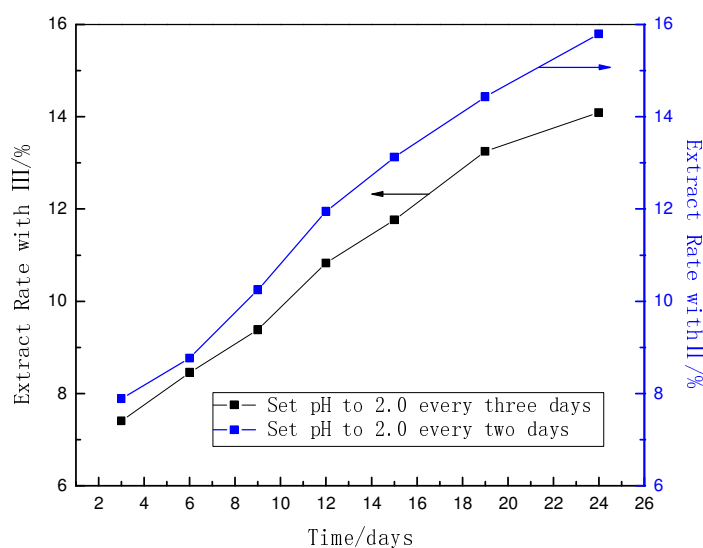


Figure 5. Change of Extract Rate with time in *A. ferrooxidans* leaching pyrrhotite systems

The Figure 5 shows that the leaching rate of the both rised gradually, and the leaching rate of Experiment II always larger than Experiment III. In the process of leaching pyrrhotite system, lot of jarosite precipitation were produced because of exsiting inorganic nutrient that needed for bacterial growth (such as K^+ , Na^+ , NH_4^+), as the leaching

process was going, the pH and potential rase, due to Fe^{3+} took part in the reaction and they covered on the surface of mineral, which unfavorable to this leaching process. Therefore, controlling pH value appropriately can reduce the quantity of deposit forming, so as to increase the leaching rate relatively, it has enlightenment function to practical

industry production (controlling pH value appropriately), and also to improve the leaching rate and production efficiency.

CONCLUSIONS

From the Eh-pH diagram of $\text{FeS}_{1.12}\cdot\text{H}_2\text{O}$, we can concluded that the conditions of jarosite that given at least $\text{Eh} < 360\text{mv}$, $\text{pH} < 2.8$, and the results of pH condition test was consonant with the conclusions of thermodynamic analysis. So when some factory acts as biological metallurgy of sulfide ore, the frequency is set to pH 1.8, and the leached percentage was higher.

Study on the deposit production in leaching process was more in line to the practical

geochemical process, it provides reference for practical production.

This paper also indicates that jarosite mediated by bacteria has a better crystalline form than that synthesized by chemical method. So we got a further understanding on the effect of *A. ferrooxidans* on the jarosite formation in natural process.

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RESUMO: A precipitação de jarosite é um fenômeno muito importante que é observado na biolixiviação da pirrotita por *Acidithiobacillus ferrooxidans* (*A. ferrooxidans*). A jarosita é um mineral secundário principal formado no ambiente supergênico ácido pela oxidação do sulfureto do metal. A formação de jarosite pode diminuir a porcentagem de lixiviação. O diagrama de Eh-pH de $\text{FeS}_{1.12}\cdot\text{H}_2\text{O}$ mostrou que os parâmetros termodinâmicos da jarosite estavam firmemente presentes em $\text{Eh} = 360\sim 800$, $\text{pH} = 2.8\sim 5$, e os resultados do teste de condição de pH estão em consonância com as conclusões da análise termodinâmica. Por meio de XRD e SEM, pode ser indicado que a propagação completa de *A. ferrooxidans* na solução é benéfica para a formação de jarosite e jarosite mediada por bactérias tem uma forma cristalina melhor do que a sintetizada por método químico. Este estudo indica que o valor do pH e a concentração de ferro ferroso/férrico são fatores chave que afetam a formação de jarosite. A porcentagem de lixiviação é maior quando a frequência foi ajustada a mais de 2,0. É crucial para minimizar a formação de jarosite, a fim de aumentar a eficiência da fábrica.

PALAVRAS-CHAVE: Pirrotita. Biolixiviação. *Acidithiobacillus ferrooxidans*. Jarosite.

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