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Transformation behavior of ferrous sulfate during hematite precipitation for iron removal

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Abstract: The transformation behavior of ferrous sulfate was examined during hematite precipitation for iron removal in hydrometallurgical zinc. Specifically, the effects of the method used for oxygen supply (pre-crystallization or pre-oxidation of ferrous sulfate) and temperature (170–190 °C) on the redissolution and oxidation–hydrolysis of ferrous sulfate were studied. The precipitation characteristics and phase characterization of the hematite product were investigated. The results showed that the solubility of ferrous sulfate was considerably lower at elevated temperatures. The dissolution behavior of ferrous sulfate crystals was influenced by both the concentrations of free acid and zinc sulfate and the oxydrolysis of ferrous ions. Rapid oxydrolysis of ferrous ions may serve as the dissolution driving force. Hematite precipitation proceeded via the following sequential steps: crystallization, redissolution, oxidation, and precipitation of ferrous sulfate. The dissolution of ferrous sulfate was slow, which helped to maintain a low supersaturation environment, thereby affording the production of high-grade hematite.

Key words: hydrometallurgical zinc; crystallization of ferrous sulfate; hematite precipitation for iron removal

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1 Introduction

Iron precipitation plays a vital role in the hydrometallurgical zinc industry. The three main processes that have been used, since the 1960s, for the removal of excess iron from leaching circuits are jarosite, goethite, and hematite processes [1,2]. Hematite precipitation has advantages over the jarosite and goethite precipitation processes from an environmental point of view; in addition, this precipitate has potential commercial value [3,4].

Hematite precipitation via the oxidation–hydrolysis of ferrous sulfate at elevated temperatures consists of two steps [5,6]: oxidation of ferrous to ferric ions by gaseous oxygen and hydrolysis of the ferric ions.

To achieve hematite precipitation of iron, process conditions must be controlled so that the operating point resides within the appropriate phase stability region. According to the equilibrium phase diagram for the $Fe_2O_3-SO_3-H_2O$ system, hematite is stable at 200 °C in highly acidic solutions (equivalent to 59.6 g/L H_2SO_4) [7,8]. At this initial acid concentration, basic iron sulfate ($Fe(SO_4)(OH)$) is produced; however, the sulfate content will gradually increase and the iron content will decrease by the same amount. Research results reported by DUTRIZAC and CHEN [9] indicated that the presence of hematite seed could suppress

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the formation of basic iron sulfate, thereby allowing hematite precipitation to proceed in a highly acid media. In terms of free acid concentration, the stability regions of hematite and basic ferric sulfate were divided by a vertical line in the temperature range of 150−220 °C, with the stability region of hematite increasing with a rise in temperature. Similar results were reported by SASAKI et al [10] and ISMAEL and CARVALHO [11].

The oxidation of ferrous sulfate with molecular oxygen in sulfuric acid solutions [12], hydrolysis of ferric sulfate to hematite [13,14], and nucleation and crystal growth of hematite [15] have been studied extensively. Considering the solubility of ferrous and zinc sulfates during hydrothermal iron precipitation is important particularly when high temperatures (>160 °C) or solutions containing high iron concentrations are involved. According to HASEGAWA et al [16], ferrous sulfate is insoluble at the temperature at which the hematite process operates. Thus, subsequent redissolution of ferrous sulfate crystals cannot be ignored. Some ferrous sulfate crystals that formed during the heating period remained undissolved at the end of the test—these crystals dissolved on cooling, resulting in a higher total iron concentration in the cooled final filtrates than those collected at the oxyhydrolysis temperature [17]. Therefore, studying the relationship between redissolution and the oxidation−hydrolysis of ferrous sulfate is important.

Herein, we investigate the transformation behavior of ferrous sulfate during the hematite precipitation process. Particularly, we examine the effects of operating conditions (method of oxygen supply and temperature) on the redissolution and oxidation−hydrolysis of ferrous sulfate crystals and investigate the precipitation characteristics and phase characterization of the hematite product in a sulfate system. In addition, the relationship between redissolution and oxidation–hydrolysis of ferrous sulfate is examined.

2 Experimental

2.1 Apparatus

The precipitation tests were conducted in a 2 L polyclave autoclave (BüchiGlasUster, Switzerland). The autoclave was made of CrNiMoTi and equipped with a sampling system, a proportional–integral–differential temperature controller, a heating circulation bath, data logging software for a personal computer, and a stirrer with a rotation speed range of 0−1500 r/min. The working temperature of the autoclave was set in the range of 0−300 °C to meet the requirements of the hematite process.

2.2 Materials

A hydrometallurgical zinc plant solution, obtained from indium precipitation process, was used for the studies. The initial solution contained approximately 32 g/L iron (II) and 100 g/L zinc and had a pH of 3.95. Table 1 shows a typical feed analysis of the initial solution.

Table 1 Chemical composition of initial solution

TFe[a]	(Fe^{2+})[a]	Zn[a]	Na[a]	K[a]	As[a]	Si[a]
32.1	31.7	99.6	1.33	0.75	0.08	0.04
Al[a]	F[a]	Cu[b]	In[b]	Ge[b]	Ni[b]	Co[b]
0.3	0.05	1.14	1.8	0.58	3.87	3.03

[a]: g/L; [b]: mg/L

2.3 Processes and methods

The effects of temperature and the method used for oxygen supply on the redissolution and oxidation–hydrolysis of ferrous sulfate crystal were studied.

The initial solution (1.0 L) was transferred into the autoclave. The autoclave was sealed, and the tightness of the fasteners and general component functioning were checked. Depending on the method of oxygen supply (as detailed below), oxygen was fed into the autoclave and its partial pressure was set at the desired level. The autoclave was heated to the required temperature (170−190 °C) and agitated for 500−600 r/min. The reaction time (0−180 min) was predefined and the temperature was controlled to within ±2 °C. Upon completion of each experiment, agitation was stopped and the autoclave was rapidly cooled using a cooling coil. The slurry was filtered by vacuum filtration through 0.45 μm filters when the temperature of the slurry decreased to 70 °C. The filter cake was washed with water and then dried in an oven at 55 °C for at least 24 h.

Oxygen was supplied using two different methods: pre-oxidation of ferrous sulfate and pre-crystallization of ferrous sulfate. In the pre-oxidation experiments, oxygen was fed into the autoclave once it was sealed and heated, and the oxygen partial pressure was fixed at 0.4 MPa. In the pre-crystallization experiments, nitrogen was fed into the autoclave until it reached the set temperature, following which the nitrogen was shut off and vented out through the vent valve. Oxygen was subsequently introduced through the inlet valve to achieve a partial pressure of 0.4 MPa.

Samples were removed from the autoclave at reaction time of 0, 30, 60, 90, 120, 150 and 180 min. The uniformly mixed fraction of iron that had precipitated with no further mass loss was analyzed to determine the concentrations of Fe^{3+} and Fe^{2+} in the samples and final filtrate. These analyses were used to determine the centration of undissolved ferrous sulfate. Two different sampling methods were employed: stirred sampling (SS) and natural sedimentation sampling (NS). For SS, the samples were removed from the autoclave at a stirring speed of 600 r/min and analyzed to determine the total Fe concentration (dissolved and undissolved ferrous ion) in the bulk solution. Ferrous sulfate crystals were dissolved after natural cooling according to the characteristic solubility and then filtered from the slurry. For NS, samples were removed from the autoclave after switching off the stirrer to allow natural sedimentation to proceed for 3−5 min. The crystals precipitated at the base of the autoclave owing to gravity and the slurry was immediately filtered.

The extent of dissolution (X) of ferrous sulfate was calculated from Eq. (1):

$$X=(m_{\text{fuc,initial}}-m_{\text{fuc},t})/m_{\text{fuc,initial}}\times 100\% \tag{1}$$

where $m_{\text{fuc,initial}}$ is the initial mass of ferrous sulfate crystals and $m_{\text{fuc},t}$ is the mass at time t.

The undissolved ferrous sulfate in the slurry was calculated from Eq. (2):

$$C_{\text{Fe(II),s}}=C_{\text{Fe(tot),SS}}-C_{\text{Fe(tot),NS}} \tag{2}$$

where $C_{\text{Fe(II),s}}$ is the concentration of ferrous sulfate crystals, $C_{\text{Fe(tot),SS}}$ is the total Fe concentration in the sample withdrawn from the autoclave while stirring, and $C_{\text{Fe(tot),NS}}$ is the total Fe concentration in the sample withdrawn from the autoclave after natural sedimentation for 3−5 min.

The concentrations of iron and zinc in solution were determined by complexometric titration with ethylenediaminetetraacetic acid. The precipitation products were characterized by X-ray diffraction (XRD; D/Max 2200, Rigaku, Japan), scanning electron microscopy (SEM; VEGA-3SBH, Tescan, Czech Republic), and chemical analysis.

3 Results and discussion

3.1 Crystallization behavior of ferrous sulfate at elevated temperature

Determining the solubility of ferrous sulfate in $ZnSO_4$−$FeSO_4$−H_2SO_4 solutions at elevated temperature is important in understanding the early stages of hematite precipitation. The concentrations of Zn^{2+} and Fe^{2+} in the samples that were withdrawn from the autoclave after natural sedimentation for 3 min under N_2 flow are plotted as a function of temperature in Fig. 1. The results show that approximately 80% and 60% of the initial iron(II) and zinc, respectively, crystallized when the temperature exceeded 180 °C. The solubility of ferrous sulfate was considerably lower than that of zinc sulfate. The solubility of both salts in the leach liquor displayed the same characteristics. The crystallization reactions of zinc and ferrous sulfates at elevated temperatures are expressed by Eqs. (3) and (4), respectively [18]:

$$Fe^{2+}+SO_4^{2-}+xH_2O \rightarrow FeSO_4\cdot xH_2O \tag{3}$$

$$Zn^{2+}+SO_4^{2-}+xH_2O \rightarrow ZnSO_4\cdot xH_2O \tag{4}$$

The solubility of ferrous sulfate was higher in the leach liquor, which may have resulted from the hydrolysis of various cations, especially Fe^{3+}, during the heating period. The pH of the samples decreased from 2.5 to 1.62 as the temperature rose from 130 to 200 °C. According to HASEGAWA et al [16], the solubility of ferrous sulfate is positively,

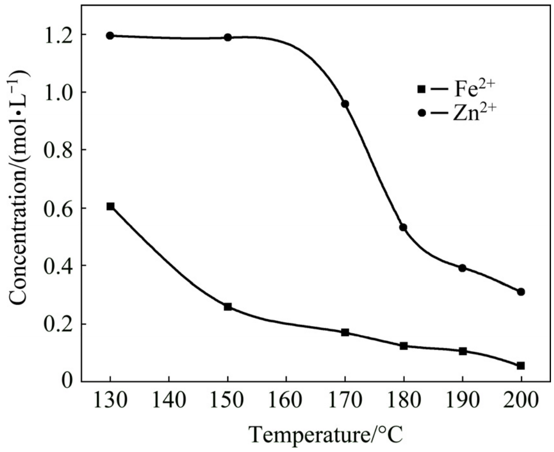

Fig. 1 Solubility of zinc and ferrous sulfates in high-iron leach liquor at various temperatures (pH: 3.95; N_2 atmosphere)

linearly dependent on free acid concentration in the presence of zinc sulfate; a correlation was thus established as Eq. (5):

$$C_{\mathrm{Fe(II),eq}}=A_1C_{\mathrm{H_2SO_4}}+B_1 \tag{5}$$

where $C_{\mathrm{Fe(II),eq}}$ and $C_{\mathrm{H_2SO_4}}$ refer to ferrous concentration at equilibrium and sulfuric acid concentration, respectively, and A_1 and B_1 are constants.

The effect of zinc sulfate concentration on the solubility of ferrous sulfate was evaluated at 190 °C. The result is shown in Fig. 2. The solubility of ferrous sulfate was linearly dependent on the concentration of zinc sulfate, as expressed by Eq. (6):

$$C_{\mathrm{Fe,eq}}=5.932-0.0287C_{\mathrm{Zn,eq}} \tag{6}$$

where $C_{\mathrm{Fe,eq}}$ and $C_{\mathrm{Zn,eq}}$ are the concentrations of iron and zinc ions, respectively, at equilibrium.

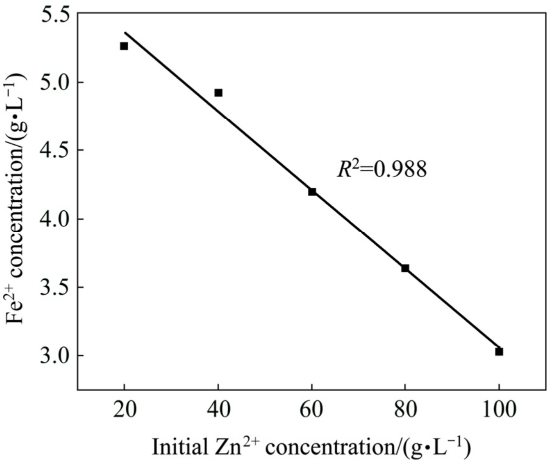

Fig. 2 Effect of zinc sulfate concentration on solubility of ferrous sulfate (Temperature: 190 °C; pH: 3.95; N_2 atmosphere)

Because the concentration of free acid changed in the solution as the reaction proceeded, the solubility of ferrous sulfate displayed a nonlinear relationship with temperature. The solubility of zinc sulfate in the leach liquor was significantly lower than that obtained in water, which indicated that recrystallization of ferrous and zinc sulfates occurred simultaneously at elevated temperatures.

3.2 Effect of method of oxygen supply on dissolution of ferrous sulfate

The influence of the method of oxygen supply on the dissolution of ferrous sulfate was studied. Two methods for oxygen introduction were assessed: pre-oxidation and pre-crystallization during the heating period from 25 to 190 °C. The results are shown in Fig. 3.

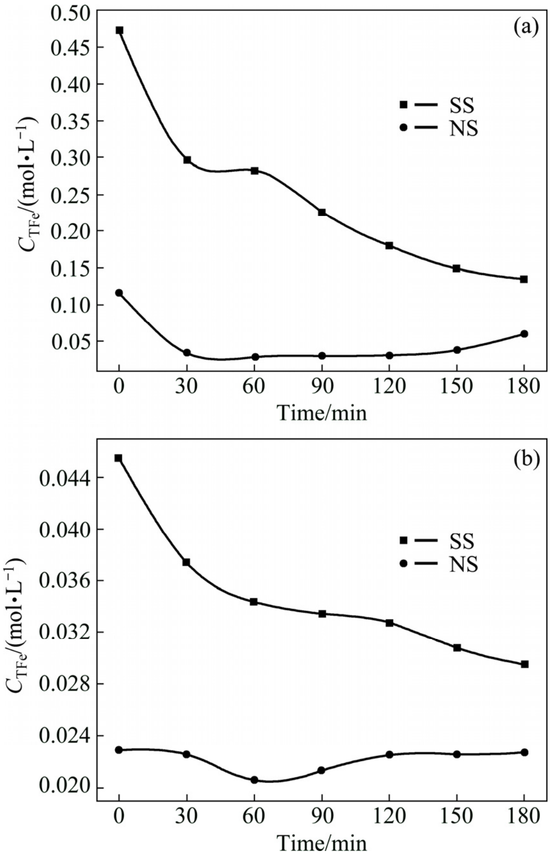

Fig. 3 Iron ions concentration as function of time for two methods of oxygen supply and two sampling techniques (Temperature: 190 °C; pH: 3.95): (a) Pre-crystallization; (b) Pre-oxidation

The amount of undissolved ferrous iron significantly increased when the autoclave was filled with nitrogen during the heating period. The initial concentration of ferrous sulfate under the pre-crystallization and pre-oxidation conditions were 0.37 and 0.02 mol/L, respectively. Using the pre-crystallization method, the concentration of ferrous ion in the final solution increased from 0.01 to 0.07 mol/L and the iron removal efficiency decreased from 93% to 73%. A relatively high concentration of ferrous ion in the final solution not only hindered the iron precipitation process, but also was detrimental to subsequent circuit operation, such as solution after iron removal was returned to neutral leaching in hydrometallurgical zinc.

The effect of the oxygen introduction method on the dissolution of ferrous sulfate crystals is shown in Fig. 4. Under the pre-crystallization condition, the dissolution rate was slow, with the dissolution of ferrous sulfate as the rate-limiting step.

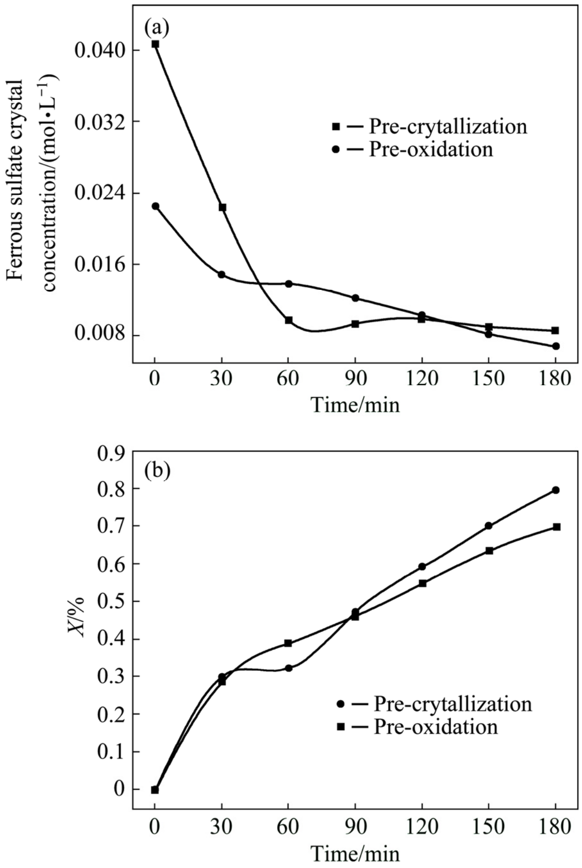

Fig. 4 Effect of method of oxygen supply on dissolution of ferrous sulfate crystals (Temperature: 190 °C; pH: 3.95): (a) Concentration of undissolved ferrous sulfate; (b) Extent of dissolution of ferrous sulfate

3.3 Effect of temperature on dissolution of ferrous sulfate

A series of experiments were conducted at various temperatures to investigate changes in the iron ion concentration during the experiment under the pre-oxidation conditions. Figure 5 shows the iron concentration profiles, obtained by the two sampling techniques, as a function of time at 170, 180, and 190 °C.

Under the pre-oxidation conditions and at an agitation speed of 600 r/min, partial oxidation of ferrous ion occurred. At the start of the process, some of the initial ferrous irons remained undissolved in the form of ferrous sulfate crystals after heating, which became less likely as the temperature increased. The initial ferrous sulfate concentrations at 170, 180, and 190 °C were 0.22, 0.084, and 0.043 mol/L, respectively. Thus, the higher the reaction temperature was, the lower the concentration of undissolved ferrous sulfate remaining at the end of the experiment was.

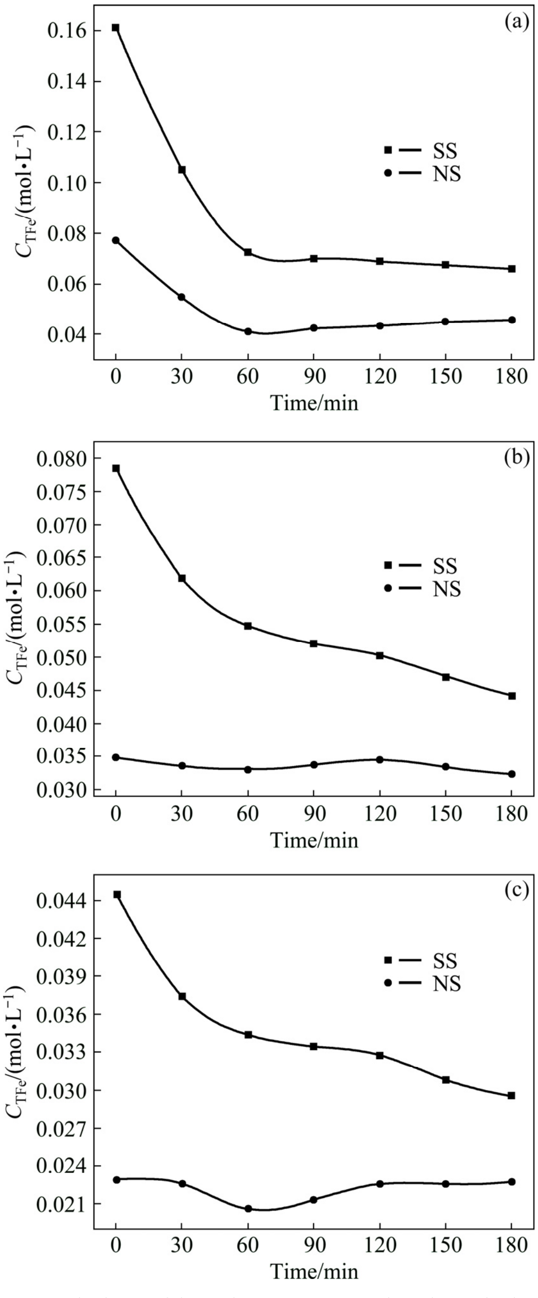

Fig. 5 Variation of iron ion concentration in solution as function of time at different reaction temperatures for two sampling techniques (Pre-oxidation; pH: 3.95): (a) 170 °C; (b) 180 °C; (c) 190 °C

Figure 6 shows that temperature had a pronounced effect on the dissolution of ferrous sulfate—an increase in temperature enhanced the dissolution rate. It is, however, necessary to consider the issues of high cost and corrosion of the autoclave at elevated temperature.

3.4 Characterization of hematite produced by oxydrolysis of ferrous sulfate

The iron and sulfur contents of the different precipitates obtained under different conditions are shown in Fig. 7. Under the pre-oxidation condition, the iron content of the residues increased, whereas the sulfur content decreased with the increase of temperature. Thus, the quality of the hematite precipitate significantly improved using the pre-crystallization condition.

XRD analyses of the precipitates obtained at various temperatures and with different methods of oxygen supply are shown in Figs. 8 and 9, respectively. SEM images of the precipitates obtained at various temperatures and with different methods of oxygen supply are presented in Figs. 10 and 11, respectively.

From Fig. 8, a pure product was obtained with increasing the temperature. Hematite was detected as the main phase, with some traces of jarosite and goethite as the temperature increased from 170 to 190 °C. The XRD pattern at 170 °C featured broad peaks compared with that at 190 °C, which is indicative of a lower degree of crystallization at the lower temperature. The diffraction peaks observed in the XRD patterns of Fig. 8 and the sulfur and iron content results (Fig. 7) indicate that Fe_2O_3 was the main compound precipitated at 190 °C; only a small portion of the precipitate was comprised of goethite (FeOOH) and sodium jarosite ($NaFe_3(SO_4)_2(OH)_6$) because the different thermal stabilities of the impurities in acid increased gradually in the system.

The results showed that some of the iron precipitated in the form of jarosite under the pre-oxidation condition, in particular, K-jarosite and Na-jarosite. Significant amounts of ferrous sulfate were oxidized during the heating period, causing a higher supersaturation of ferric ion, which precipitated as jarosite, especially when treating leach solutions that contained potassium and sodium ions. A metastable phase-like jarosite would

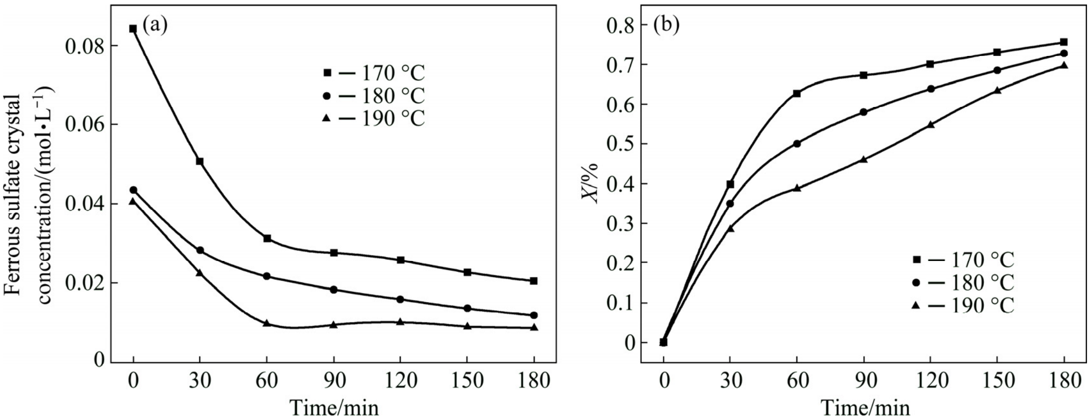

Fig. 6 Effect of temperature on dissolution of ferrous sulfate crystals (Pre-oxidation; pH: 3.95): (a) Concentration of undissolved ferrous sulfate; (b) Extent of dissolution of ferrous sulfate

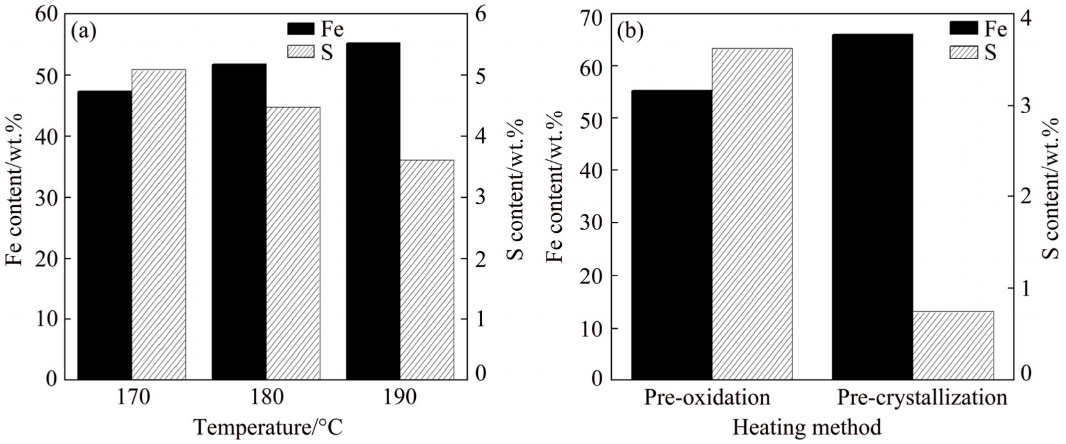

Fig. 7 Iron and sulfur contents of precipitates obtained under different reaction conditions: (a) Pre-oxidation and varying reaction temperature; (b) Temperature of 190 °C and different methods of oxygen supply

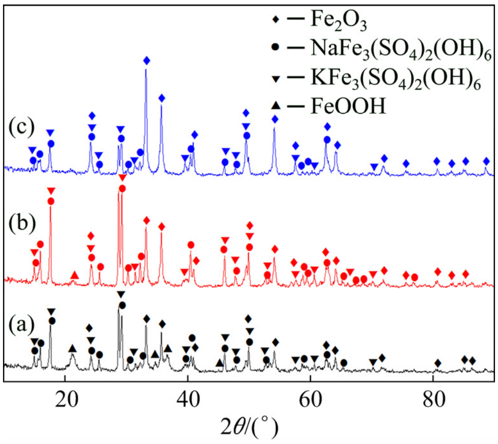

Fig. 8 XRD patterns of precipitates obtained at temperatures of 170 °C (a), 180 °C (b) and 190 °C (c) under pre-oxidation conditions

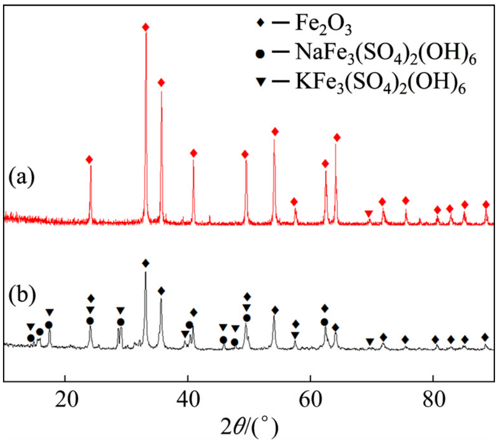

Fig. 9 XRD patterns of precipitates obtained with different methods of oxygen supply: (a) Pre-crystallization; (b) Pre-oxidation (temperature of 190 °C)

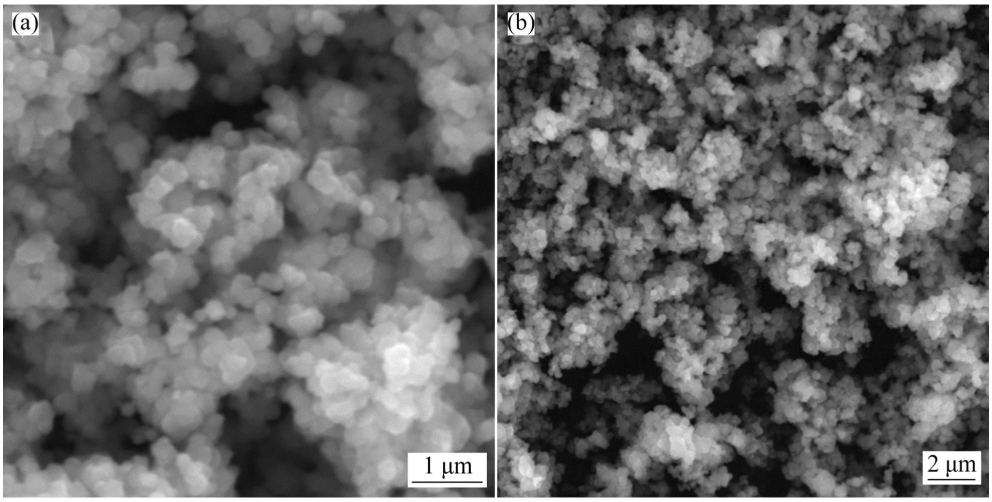

Fig. 10 SEM images of precipitates obtained at temperatures of 170 °C (a) and 190 °C (b) under pre-oxidation conditions

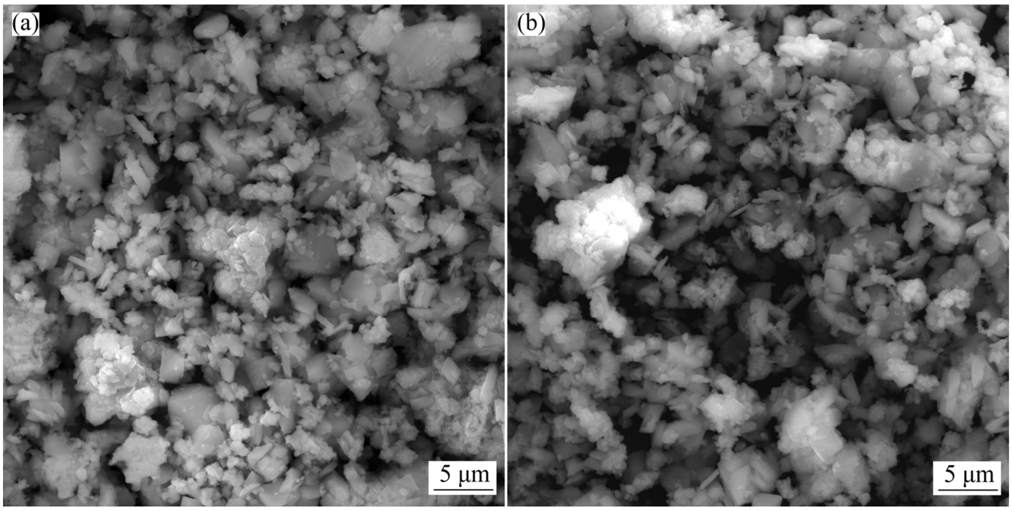

Fig. 11 SEM images of precipitates obtained with different methods of oxygen supply: (a) Pre-crystallization; (b) Pre-oxidation (temperature of 190 °C)

remain in the residue, resulting in a decrease in the iron content and an increase in the sulfur content of the precipitates. Under the pre-crystallization condition, the slow dissolution kinetics of ferrous sulfate helped to maintain a low supersaturation environment that is conducive to the production of high-grade hematite precipitates.

4 Conclusions

(1) The solubility of ferrous sulfate was considerably lower at elevated temperature; the iron(II) concentration was less than 5 g/L when the temperature exceeded 180 °C, and most of the initial iron(II) present was crystallized.

(2) The dissolution behavior of ferrous sulfate was controlled by both the concentrations of free acid and zinc sulfate and the oxydrolysis of ferrous ions. Rapid oxydrolysis of ferrous ions may serve as the dissolution driving force.

(3) The sequential steps involved in hematite precipitation are crystallization, redissolution, oxidation, and precipitation of ferrous sulfate. The dissolution of ferrous sulfate was the rate-limiting step that determined the overall (slow) rate of the entire reaction under pre-crystallization conditions. However, the slow reaction rate helps to maintain a low supersaturation environment that is conducive to the production of high-grade hematite.

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赤铁矿法除铁过程中硫酸亚铁的转化行为

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摘 要：针对湿法炼锌赤铁矿法除铁过程中硫酸亚铁的转化行为，研究供氧方式(硫酸亚铁的预结晶或预氧化)和反应温度(170~190 °C)对硫酸亚铁晶体再溶解−氧化水解的影响，考察赤铁矿产物的析出特性和物相组成。结果表明：硫酸亚铁在高温溶液中的溶解度明显降低；硫酸亚铁晶体的溶解行为受两个因素的控制：游离酸与硫酸锌浓度的变化和硫酸亚铁离子的氧化水解；亚铁离子的快速氧化是硫酸亚铁结晶重溶解的驱动力；赤铁矿的析出经历硫酸亚铁的结晶、再溶解、氧化和沉淀过程；硫酸亚铁的缓慢溶解有助于保持较低的过饱和度，有利于生产高品质的赤铁矿。

关键词：湿法炼锌；硫酸亚铁结晶；赤铁矿法除铁

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