



Dephosphorization of high phosphorus oolitic hematite by acid leaching and the leaching kinetics



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ABSTRACT

It is highly difficult to remove phosphorus from high phosphorus oolitic hematite by the usual dressing process. Acid leaching is an effective method for the dephosphorization of high phosphorus oolitic hematite. The acid leaching experiments were conducted to study the effect of acid concentration, temperature, leaching time, solid-liquid (S/L) ratio and the stirring speed on the dephosphorization of the high phosphorus oolitic hematite. The results demonstrate that hydrochloric acid is the best selection for leaching acid for the dephosphorization, and treatment of the sample in 0.2 mol/L hydrochloric acid at 298 K for 10 min with the S/L ratio of 0.03 g/mL and a stirring speed of 300 rpm is optimum. Thus, the dephosphorization can reach 90% with < 0.18% iron loss. We also investigated the hydrochloric acid leaching kinetics. There were two distinct stages in the leaching process for dephosphorization, and the kinetics of both stages followed a shrinking core model. The apparent activation energy for leaching in leaching stage one (initial 10 min) and stage two (10–60 min) was estimated to be 2.51 kJ/mol and 5.59 kJ/mol, respectively. The results demonstrated that leaching of the two stages was controlled by acid diffusion through the solid product layer. The leaching with iron dissolution was mostly controlled by chemical reaction between Fe_2O_3 and acid.

1. Introduction

The Ningxiang type oolitic hematite, which is widely distributed in Hubei, Hunan, Sichuan, Guizhou and other regions, is one of the largest reserves of sedimentary iron ore in China. The iron ore reserves of 3.72 billion tons, account for approximately 73.5% of the sedimentary iron ore in China (Lu et al., 1994). The high phosphorus oolitic hematite has the characteristics of low-grade iron content and high-level harmful impurities, such as phosphorus and aluminium (Wei, 2010), which limit the development of this oolitic iron ore. In high phosphorus oolitic hematite, phosphorus is mainly in the form of apatite, where the particle size of the crystal is very fine and not easy to separate. Therefore, the high phosphorus oolitic hematite is difficult to utilize as a raw material in ironmaking and is characterized as a refractory ore (Xia et al., 2011). Recently, several phosphorus removal processes for high phosphorus oolitic hematite have been investigated, including physical separation (Wu et al., 2011), flotation (Yan et al., 2011), alkali leaching or acid leaching (Jin et al., 2006; Xia et al., 2011), bio-leaching (Delvasto et al., 2008; Wang et al., 2010), and the pretreatment with hot metal (Wu et al., 2011) etc. Among these

processes, the enrichment process is complicated by the low dephosphorization. The efficiency of bio-leaching dephosphorization is too low. The smelting method incurs a high cost and creates a large volume of slag. For physical separation, crushed ores should be finely ground until the phosphorus minerals are dissociated from the iron minerals and then dephosphorized by gravity separation or magnetic separation, which leads to high energy consumption.

Acid leaching, a more effective dephosphorization method (Forssberg and Adolfsson, 1981), has the advantage that no completely monomeric dissociation of the phosphorus-bearing minerals is needed. Kanungo and Mishra (2000) investigated the dephosphorization of high phosphorus manganese ores in India. In these high phosphorus manganese ores, the phosphorus can be reduced to 0.15% or less by roasting the ores with 2–6 wt% NaCl at 750–800 °C followed by leaching in 0.5–0.75 M HNO_3 at 40–60 °C for a period of 15–20 min. Nwoye (2009) established a valid and precise model to evaluate the concentration of dissolved phosphorus during the leaching of iron oxide ore in oxalic acid solution. Jin et al. (2006) deduced that the sulfuric acid was the most effective acid for the dephosphorization of the Changde iron ore among HCl, HNO_3 and H_2SO_4 . Xia et al. (2011)

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studied the phosphorus removal from high phosphorus iron ores by selective HCl leaching, and the content of phosphorus in iron ore can comply completely with the requirements of steel production after the dephosphorization reaction. Iron ore with Fe < 60% can be used as raw material to produce the direct reduced iron, by coal-based direct reduction, or the slag/metal separation process of the reduced iron from the oolitic high-phosphorus iron ore fines by gas (Tang et al., 2014). Yu et al. (2013) thought sulfuric acid was the most appropriate acid for dephosphorization, and investigated the leaching kinetics of H₂SO₄. However, their studies did not consider the kinetics of iron dissolution, and they did not discuss the HCl leaching kinetics. The cost of the chemical leaching process depends mostly on the consumption of the leaching agents. Some of the literature emphasized that the acid leaching process causes a large amount of waste acid, with the associated environmental problems and the loss of soluble iron (Li et al., 2013; Li et al., 2012). However, the acid leaching process is superior to other methods mentioned for dephosphorization. Therefore, it is necessary to study acid leaching further and simultaneously avoid deficiencies.

In this work, a series of experiments investigated the effects of leaching time, leaching temperature, acid concentration, solid-liquid (S/L) ratio and stirring speed on the dephosphorization by the optimal acid, and the leaching kinetics for the dephosphorization and iron dissolution was also investigated.

2. Experimental

2.1. Materials

The blocks of high phosphorus oolitic hematite ores from western Hubei province, China, were used as raw materials. The raw ore was crushed by a twin roll crusher (PG, Zibo Bo-speed Transmission Machinery Co., Ltd.). After crushing the raw ore powder was planetary ball milled (QM-4F, Nanjing Nanda Instrument) to fine powder, then the fine powder was screened into the desired size by a mechanical vibration sieve. In this experiment, raw ore powder in the range of 0.096–0.120 mm was used, and the chemical composition is shown in Table 1. Hydrochloric acid, sulfuric acid and nitric acid were used for leaching solutions. All the reagents were analytically pure.

2.2. Procedure

The ore sample was put in a 250 mL round-bottom flask with acid at different solid-liquid (S/L) ratio ranges of 0.01 to 0.09 g/mL (with or without stirring), then was heated in the water bath at a preset temperature for the desired time, and subsequently was filtered through a filter paper (FP). The content of phosphorus and iron was analysed from the filtrate by ICP-AES (IRS Advantage ER/S). The characterization of the specimens before and after acid leaching was detected by the scanning electron microscope equipped with an energy diffraction spectrum (SEM-EDS, Nova 400 NanoSEM, le350PentaFET X-3) and the X-ray diffraction (XRD, X' Pert PRO MPD).

2.3. Evaluation indexes

The dephosphorization (η) and iron loss (ϵ) can be defined as follows:

Table 1
Chemical composition (mass%) of the high phosphorus oolitic hematite ore powders (particle size of 0.096–0.120 mm).

Composition	T. Fe	P	CaO	SiO ₂	Al ₂ O ₃	S
Content	56.27	0.57	2.44	8.96	4.92	0.02

$$\eta = \frac{\omega'_P V}{\omega_P m_0} \times 100\% \quad (1)$$

$$\epsilon = \frac{\omega'_{Fe} V}{\omega_{Fe} m_0} \times 100\% \quad (2)$$

where: η is the dephosphorization, %; ϵ is the iron loss, %; ω_{Fe} is the total iron content (T. Fe) of the iron ore powder; ω'_{Fe} is the total iron content in the filtrate, g/mL; m_0 is the weight of the iron ore powder, g; ω_P is the phosphorus content of the iron ore powder, %; ω'_P is the phosphorus content in the filtrate, g/mL; and V is the volume of the filtrate, mL.

3. Results and discussion

3.1. Effect of factors on the acid leaching

3.1.1. Different acids and acid concentration

The samples were leached by HCl, H₂SO₄ and HNO₃, respectively, at 298 K for 10 min, and the solid-liquid (S/L) ratio was 0.03 g/mL. The effect of acid concentration (H⁺ concentration in 0.1 mol/L, 0.2 mol/L, 0.3 mol/L, 0.4 mol/L and 0.5 mol/L, respectively) on dephosphorization and iron loss was investigated.

Fig. 1 shows that dephosphorization increases gradually with the increase in acid concentration for the three kinds of acid. At the range of 0.1 mol/L to 0.4 mol/L, the dephosphorization for H₂SO₄ and HNO₃ is similar, and is significantly lower than the dephosphorization for HCl. When the acidity is > 0.4 mol/L, the dephosphorization capability is HCl > H₂SO₄ > HNO₃. The H₂SO₄ leaching will form CaSO₄, a kind of white viscous colloid, which hinders the reaction of the phosphorus-bearing minerals with H₂SO₄. The reaction between HNO₃ and Fe₂O₃ occurs more easily than the reaction with HCl, possibly resulting in the lower dephosphorization ratio than the ratio with HCl.

Fig. 2 shows that the iron loss increases gradually with the H⁺ concentration increase in the HCl, H₂SO₄ and HNO₃ solution. The trends of iron loss are similar during the acid leaching by HCl and HNO₃, and the iron loss is slightly lower than the iron loss with H₂SO₄. This result is in agreement with the thermodynamic calculation by Zhao et al. (2017), they confirmed that the reaction between H₂SO₄ and Fe₂O₃ occurs the most easily. To reduce the iron loss, HCl or HNO₃ is more appropriate to be used as leaching agent. The market prices of HNO₃ and HCl are approximately 0.12 RMB and 0.033 RMB per mol of H⁺ in China (Meng et al., 2008). Taking into account the dephosphorization, the acid usage and iron loss, HCl is the best choice for the phosphorus removal. To achieve relatively higher dephosphorization and lower iron loss, the acid concentration of HCl was chosen as

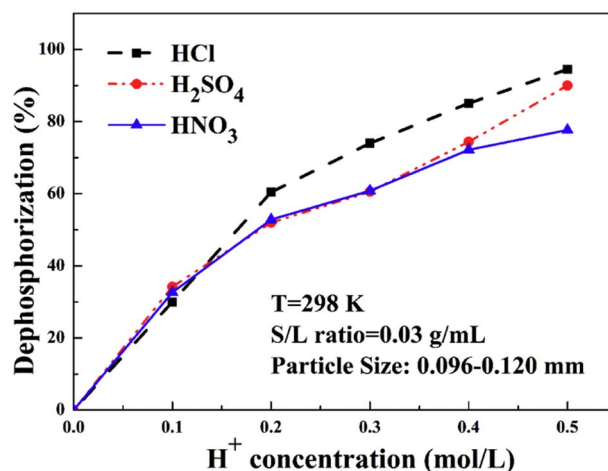


Fig. 1. Effect of acidity of the three acids on dephosphorization in the leaching process with a leaching time of 10 min, S/L ratio of 0.03 g/mL, and particle size of 0.096–0.120 mm, at 298 K.

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