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Influence of phase and microstructure on the rate of hydrochloric acid leaching in pretreated Panzhihua ilmenite

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ABSTRACT

The present study investigated the influence of high temperature oxidation and reduction pretreatments on the leaching rate of Panzhihua ilmenite. The as-pretreated ilmenite was leached with 20% HCl at 105 °C. The leaching process was controlled by the phases and microstructures that evolved during the pretreatment processes. The leaching kinetics of pure hematite, ilmenite and pseudobrookite were characterized to clarify the phase effect on the iron-leaching rate; the rate of iron leaching occurs in the following order in the HCl solution: hematite (ferric iron) > ilmenite (ferrous iron) >> pseudobrookite (ferric iron). Therefore, the often-cited notion that ferrous iron dissolves faster in HCl solutions than ferric iron when explaining the pretreatment effects is inaccurate. Moreover, the oxidation pretreatment (at 600–1000 °C for 4 h) cannot destroy the dense structure of the Panzhihua ilmenite. Therefore, the influence exerted by the oxidation on the leaching process is primarily determined by the phase change; oxidation at 600 and 700 °C slightly increased the rate of iron leaching because the ilmenite was transformed into hematite, while the oxidation at 900–1000 °C significantly reduced the rate of iron leaching because a pseudobrookite phase formed. The reduction effect was subsequently investigated; the as-oxidized ilmenite was reduced under H₂ at 750 °C for 30 min. The reduction significantly accelerated the rate of subsequent iron leaching such that nearly all of the iron had dissolved after leaching for 2 h in 20% HCl at 105 °C. This enhanced iron-leaching rate is mainly attributed to the cracks and holes that formed during the reduction process.

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1. Introduction

Producing synthetic rutile from ilmenite ores is becoming increasingly important because natural rutile resources are becoming depleted, while the demands from the TiO₂ pigment industry have increased (El-Hazek, Lasheen, El-Sheikh, & Zaki, 2007; Itoh, Sato, Ono, Okada, & Nagasaka, 2006; Mahmoud, Affi, & Ibrahim, 2004; Sarker, Rashid, & Kurny, 2006). The conversion of ilmenite to synthetic rutile requires that various impurities, such as iron oxide, MgO, CaO, MnO, SiO₂, and Al₂O₃, be removed (Balderson & Macdonald, 1999). Hydrochloric acid leaching is one of the pre-eminent processes used to obtain synthetic rutile from ilmenite because it removes not only iron oxide but also other impurities, such as MgO, CaO, and MnO; these impurities cannot be efficiently removed using the electro-smelting or the Becher processes. However, directly leaching ilmenite using hydrochloric acid is a slow process (Olanipekun, 1999; Zhang, Hu, Liao, Chen, & Tan, 2011);

only 55% of the iron oxide in an ilmenite ore was removed after leaching in a 20 wt% HCl solution at 105 °C for 10 h (Zhang et al., 2011). Therefore, the hydrochloric acid leaching process must be accelerated. Various methods have been developed during the past several decades, including the addition of metallic iron (El-Hazek et al., 2007; Lasheen, 2005; Mahmoud et al., 2004), mechanical activation (Li, Liang, & Wang, 2008; Wei, Hu, Chen, & Tan, 2009; Zhang et al., 2011) and high temperature pretreatments (Balderson & Macdonald, 1999; Chen, 1976; Ismail, Amarasekera, & Kumarasinghe, 1983; Janssen & Putnis, 2011; Kelly & Rowson, 1995; Ogasawara & Veloso de Araujo, 2000; Sarker et al., 2006; Zhang et al., 2011); the high temperature pretreatment was found to be most effective tactic because under optimal conditions, high temperature pretreatment may both increase the leaching rate and prevent the pulverization of the product during the leaching process.

The influence of high temperature heat treatments, such as oxidation, reduction or sequential oxidation–reduction processes, on the rate of iron leaching has been investigated in detail by many researchers (Balderson & Macdonald, 1999; Chen, 1976; Ismail et al., 1983; Janssen & Putnis, 2011; Kelly & Rowson, 1995;

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Table 1
Chemical composition of Panzhihua ilmenite (wt%).

TiO ₂	MgO	CaO	FeO	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	MnO	H ₂ O
46.76	5.62	1.03	35.59	5.12	1.37	3.56	0.706	<0.1

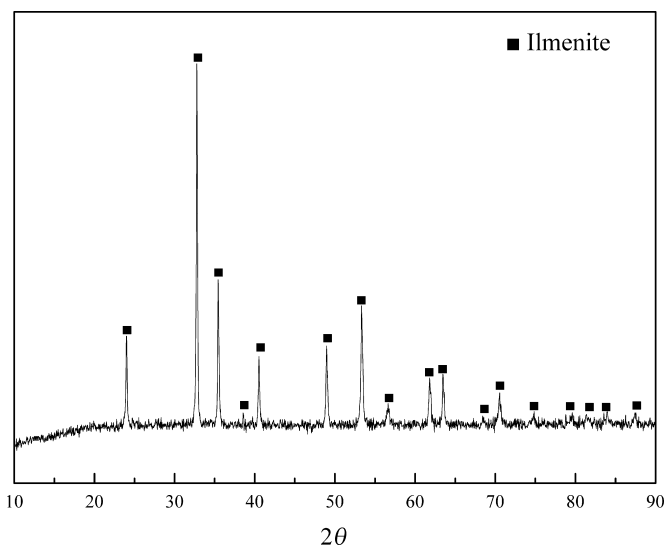
Ogasawara & Veloso de Araujo, 2000; Sarker et al., 2006; Zhang et al., 2011). There is consensus that the reduction of ferric iron in ilmenite to form ferrous iron may enhance the rate of iron leaching; the leaching rate of ferrous iron by hydrochloric acid is thought to be faster than that of ferric iron. However, Chen (1976) reported that two ilmenites with very similar chemical compositions could display varied leaching behavior when using the same reduction process, indicating that mechanisms other than the ferric to ferrous transition may exist. Regarding the purpose of the oxidation, contradictory results have been reported. Sarker et al. (2006) reported that a 1-h oxidation at 950 °C before reducing the ilmenite separated from Bangladesh beach sands increased both the extent and rate of the iron leaching. Sarker et al. attributed the enhanced effect to the microcracks generated by the oxidation process. However, Janssen and Putnis (2011) reported that pre-oxidizing Tellnes ilmenite at 700 °C for 12 h transforms the ilmenite into rutile and hematite, thereby decreasing the rate of iron leaching due to the slower dissolution rate proposed for ferric iron relative to ferrous iron. Ogasawara and Veloso de Araujo (2000) studied the effects of oxidation and reduction treatments on a Brazilian ilmenite on the rate of iron leaching, discovering that oxidation at 820 °C for 2 h before the reduction only marginally affected the rate of iron leaching. This discrepancy might be caused by the difference in both the microstructure and phase formed during the heat-treatment process because these authors employed different ilmenites under different pretreatment conditions. Obviously, more numerous systematic investigations regarding the phase and microstructural evolution of ilmenite during high-temperature pretreatments is require to understand the effects of the pretreatments on the rate of leaching.

In the present paper, the phase and structural evolution during high-temperature oxidation and reduction steps were systematically investigated using the ilmenite from the Panzhihua area (Sichuan Province, China). The leaching rates of the as-heat-treated ilmenites were characterized and compared with those of untreated ilmenite. The influence of the phase and structure on the rate of iron leaching was elucidated for the Panzhihua ilmenite.

2. Experimental

2.1. Materials

The ilmenite concentrate was provided by the Panzhihua Iron and Steel Group Corporation (Sichuan, China). The as-received ilmenite was sieved; only particles in the 100–120 mesh range were used during the study. The ilmenite primarily contained hexagonal FeTiO₃, as illustrated by the XRD pattern in Fig. 1. The chemical composition of the ilmenite is summarized in Table 1; Table 1 also reveals that MgO, CaO, Al₂O₃, and SiO₂ are the primary impurities apart from iron oxide. Most of those impurities may exist as part of a solid solution because they are not observed in the XRD pattern. All the gases (H₂, N₂, and dry air) used in the thermal pretreatment experiments were purchased from Beijing Hua Yuan Gas Chemical Industry Co., Ltd (Beijing, China). Among them, the purity of both the H₂ and N₂ were 99.999%. Ilmenite (purity 99.8%) and pseudobrookite (purity 99.9%) with dense structures were acquired from Alfa Aesar China (Tianjin) Co., Ltd. (Tianjin, China). The ilmenite and pseudobrookite were sieved to obtain

**Fig. 1.** XRD pattern of raw Panzhihua ilmenite.

300–400 mesh particles for the subsequent leaching experiments. Analytical grade, submicron ferric oxide (Sinopharm Chemical Reagent Co., Ltd., Beijing, China) was sintered at 1200 °C for a week to obtain dense, bulk hematite; this material was cooled, crushed and sieved to obtain the 300–400 mesh particles. The as-sieved ilmenite, pseudobrookite and hematite were subjected to the leaching tests and compared. All of the other chemicals used during the experiments were analytical grade and purchased from Beijing Chemical Reagent Company (Beijing, China).

2.2. Thermal pretreatments

The oxidation and reduction thermal pretreatments were carried out in a fluidized bed reactor composed of a quartz tube (15 mm in diameter and 500 mm in height) with a perforated plate for gas distribution. To study the oxidation effect, the ilmenite was oxidized in air at 600–1000 °C for 4 h. The as-oxidized ilmenites were subjected to a reduction pretreatment at 750 °C for 0.5 h to investigate the reduction effect.

During each experiment, 16 g ilmenite was used. Reactant gases (air, N₂, or H₂) were introduced from the bottom of the fluidized bed via the perforated distribution plate. The gas flow rate was approximately 0.3 L/min and was sufficient to fluidize the ilmenite powder. The fluidized bed reactor was heated using a homemade resistant furnace. After the pretreatments, the fluidizing gas was changed to nitrogen and the heating was switched off, allowing the as-pretreated ilmenite to cool naturally to room temperature.

2.3. Leaching

The leaching experiments were carried out using a 20% HCl solution at 105 °C for various durations using a 500 mL three-necked flask equipped with a thermometer, a reflux condenser and a magnetic stirrer that could reach 600 rpm. In the ilmenite leaching experiments, a 30 g sample was added to the flask with 120 mL hydrochloric acid solution to obtain a liquid to solid ratio of 4 mL/g. To measure the leaching kinetics of the pure substances, i.e., the as-sieved ilmenite, pseudobrookite and hematite were leached under nearly identical conditions except that the liquid to solid ratio was 150 mL/g, i.e., 300 mL HCl to 2 g solid. To determine the leaching kinetics, 0.5 mL of leachate was withdrawn from the

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