



Recovery of iron and manganese from iron-bearing manganese residues by multi-step roasting and magnetic separation

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ABSTRACT

Millions of tons of iron-bearing manganese residue are produced as a by-product of the electrolytic manganese industry. And the environmental contamination caused by manganese residue has received increasing attention. This paper focuses on the recovery of iron and manganese from high iron-bearing manganese residues. Manganese ferrite in manganese residues is initially decomposed by oxidative roasting, and the intermediates are magnetized in a reductive roasting step. The roasted product is milled and subjected to multi-stage magnetic separation. The optimum conditions are as follows: roasting at 750 °C under air flow for 30 min, roasting in a CO atmosphere at 750 °C for 30 min, and separating under a magnetic intensity of 1000 G for weak magnetic separation and of 12,000 G for strong magnetic separation. The recovery and grade of iron in the iron concentrate were 72.29% and 62.21%, respectively, and those of manganese in the manganese concentrate were 90.75% and 35.21%, respectively. This study demonstrates that the combination of roasting and magnetic separation provides a promising process for the recovery of iron and manganese from high-iron-bearing manganese residues.

1. Introduction

Electrolytic manganese residues (EMR) are produced in significant amounts from the electrolytic manganese metal (EMM) industry, with 6–9 tons of residue generated per ton of metal (Duan et al., 2010). Most of these residues remain untreated and are generally stockpiled. The hazardous metal elements (Mn, Zn, Cu, Pb, Cd, Cr) (Zhou et al., 2014) associated with these residues result in high environmental risks and waste potentially recoverable valuable resources (Du et al., 2015; Li et al., 2014; Yan and Qiu, 2014).

The recycling and recovery of manganese from EMR has aroused increasing attention in recent years. Manganese is mainly recovered by the hydro-metallurgical route using a hot acid leaching (HAL) process with sulfuric acid as a solvent. The maximum manganese extraction using this process varies because manganese is only soluble in its low-valence form (Elsherief, 2000; Liu et al., 2014). In addition to the HAL process, citric acid leaching assisted by ultrasound (Li et al., 2008; Yuzhu et al., 2007), sulfuric acid leaching with glucose and saccharose (Yao et al., 2003), organic acid leaching (Das et al., 2012) and bio-

leaching (Duan et al., 2011; Xin et al., 2011) have all been applied, with the maximum manganese extraction achieved in the bio-leaching process at above 90%. However, traditional chemical leaching processes have disadvantages, including massive consumption of solvent and generation of secondary high iron-bearing residues in the iron precipitation process. Bioleaching would be the most promising method if the leaching kinetics could be accelerated, as the leaching time of this method was counted in days (Duan et al., 2011).

In this study, roasting and magnetic separation techniques were applied for the recycling of iron and manganese from iron-bearing manganese residues. Manganese ferrite was first decomposed into magnetite and low-valence manganese oxide using a two-stage roasting process. The roasted product was then subjected to ball-milling and multi-stage magnetic separation to recover manganese and iron separately. The decomposition mechanism of manganese ferrite was analysed by thermodynamic calculation and XRD analysis of the roasted product, and the operating parameters of the roasting and magnetic separation procedures were studied.

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Table 1
Element content in EMR (wt%).

Elements	Content	Elements	Content
O	35.01	Mn	15.12
Si	18.63	Fe	16.79
Al	3.08	P	0.734
S	2.32	Cu	0.05
Ca	1.56	Zn	0.24
K	1.41	Ni	0.01
Cl	0.02	Cr	–
Mg	0.39	Cd	–

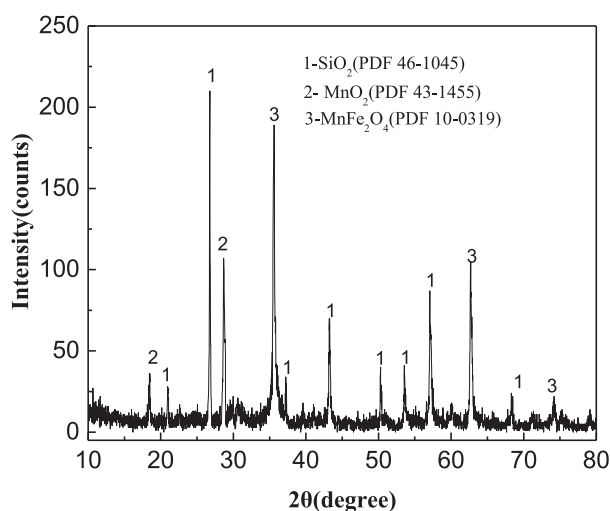


Fig. 1. XRD pattern of EMR.

2. Material and methods

2.1. Materials

A 100 kg sample of EMR was obtained from an electrolytic manganese plant located in Hunan, China. The sample was sieved to produce a fraction of below 75 μm size and dried at 105 $^{\circ}\text{C}$ to a constant weight. ICP analyses of the EMR are shown in Table 1. Its corresponding XRD pattern is shown in Fig. 1. It can be seen Fig. 1 that the iron was associated with manganese in the residues, with MnFe_2O_4 was determined as the main Mn-bearing component in the EMR.

2.2. Analysis

Potassium dichromate titration of Fe and Mn was used for chemical analysis of the samples. XRD analysis with Cu $K\alpha$ -radiation (Rigaku, TTR-III) was applied for analysis of the primary residue and for examining the phase transformations during roasting. The multiple element content was determined by ICP-AES analysis (Baird, PS-6).

2.3. Thermodynamic analysis

The thermodynamic analysis was carried out using FactSage (Källén et al., 2014; Sorensen et al., 2010). The predominance area diagram of $\text{MnFe}_2\text{O}_4\text{-O}_2$ and $\text{MnFe}_2\text{O}_4\text{-CO}$ in the range 100–1000 $^{\circ}\text{C}$ were calculated based on minimization of the Gibbs free energy.

2.4. Roasting

The roasting process was conducted using a pipe furnace (Peng et al., 2017). Crucibles with a volume of 300 ml were plastered with a layer of sample (20 g, 2–3 mm in thickness) on the walls and roasted under a flowing stream of reaction gas ($\text{CO} + \text{Ar}$ or air). The gas was

introduced when the desired temperature was reached. After a certain roasting duration, the reaction gas was replaced by argon, and then the roasting product was quenched in water. The roasting temperature ranged from 600 $^{\circ}\text{C}$ to 850 $^{\circ}\text{C}$ at 50 $^{\circ}\text{C}$ increments, and roasting times ranging from 10 min to 40 min at 5 min increments were examined in the oxidative roasting experiments. In the reductive roasting experiments, the roasting temperature, roasting time and concentration of CO were maintained in the ranges of 600–800 $^{\circ}\text{C}$, 10–40 min and 2–12%, respectively. The material recovered after quenching was directly subjected to mechanical milling and magnetic separation.

2.5. Mechanical milling and magnetic separation

Mechanical milling was performed in a planetary ball mill operating at 500 rpm. Approximately 200 g of steel balls were kept in each cell, along with 20 g of the sample powder. Magnetic separation experiments were carried out with a Kolm-type high-gradient magnetic separator (Oberteuffer, 1974). With this separator, the slurry obtained after mechanical milling was placed into a plastic container equipped with a stirrer and diluted using tap water. Then, the prepared fluid was introduced into the separation chamber under gravity, in which a magnetic field perpendicular to the direction of flow was generated using an electromagnet. The magnetic intensity ranged from 800 to 1400 G for iron separation and from 8000 to 12,000 G for manganese separation. Magnetic particles were enriched on the plates as the concentrate, and non-magnetic particles were collected as the tailings. After separation, the concentrate was washed, filtered and dried for further analyses.

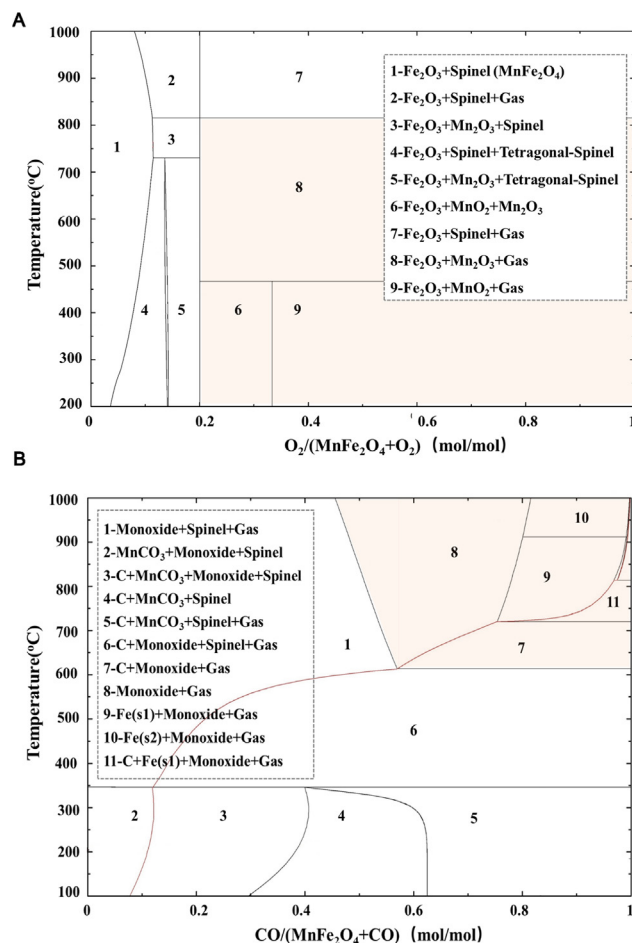


Fig. 2. Predominance area diagram of $\text{MnFe}_2\text{O}_4\text{-O}_2$ (A), $\text{MnFe}_2\text{O}_4\text{-CO}$ (B), the reddish region is the unstable area for MnFe_2O_4 . (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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