



Phase transformation during roasting process and magnetic beneficiation of oolitic-iron ores



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ABSTRACT

Oolitic-iron ore material is one of the most important iron resources in China. However, due to its low grade of Fe, poor liberation of iron minerals and high impurities, no satisfactory processing method has been developed to effectively utilize it for the practical applications. In this work, a coal-based reductive roasting method was applied, where the detail mineral phase transformation and reduction area during magnetic roasting process was quantitatively investigated. Then, low intensity magnetic separation was performed. The results showed that roasting time significantly affected Fe recovery, with a minor influence on Fe content of concentrate. Fe recovery increased from 10 to 60 min before starting to decrease slightly. This may result from the formation of weakly magnetic FeO or Fe₃O₄-FO (solid-melt-body), where the reduction degree increased gradually from 10 to 50 min. During the roasting process, the mineral phase Fe₂O₃ changed to Fe₃O₄, but the original oolitic structure and mineral embedding relation were hardly evolved. This research could enhance our understanding regarding the microscopic view of magnetic phase transformation, and provide help for commercial development of such refractory iron ores.

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1. Introduction

Extracting Fe from raw iron ore materials is considered to be one of the most important topics in thermo-metallurgy field. In present, the blast furnace process is usually applied for extracting iron, where the iron ore needs to be upgraded through a series of pre-treatment [1] processes, including crushing, grinding, separation [2,3], dewatering, and so on [4–6]. Due to the dramatically increasing in crude steel production, there is a rapid rising demand for iron ore. However, high grade and high quality iron ore resources have been nearly exhausted in China.

Oolitic iron ore [7], a type of refractory mineral resource, is being considered with great importance, because of its huge abundance and the increasing demand of iron ores globally. Oolitic ores contain particles with circling layers of iron minerals and gangue.

Considering industrial cost, oolitic iron ores have not been commercially utilized yet. This is mainly due to its chemical complexity of compositions and the poor liberation of iron minerals, which make it difficult to handle in the mineral beneficiation processes.

Generally, some processing methods, e.g., leaching [8–10], microwave roasting [11], sulfidation roasting [12–14], Na₂CO₃-roasting [15], magnetic separation [16], are widely applied to ores or other secondary-resources. For the utilization process [17], some types of oolitic iron ores have been upgraded using conventional methods, such as magnetic separation [18,19] or direct roasting reduction.

Several studies [20–23] have performed reduction and magnetic separation. However, most of them focused on the operational conditions during the reduction process. In this work, the characteristic of mineral phase transformation and roasting behaviour were investigated during roasting process, by monitoring phase change and reduction area versus roasting time. The potential industry application was also discussed to provide a better

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understanding toward the reduction mechanism of such refractory iron ores and scenario for utilization process, respectively.

2. Experimental

2.1. Materials

The representative samples of raw oolitic iron ores were collected from west Hubei province, by notch sampling method, with particle size below 150 mm. The ore particles were crushed to ≤ 6 mm, by Jaw Crusher through open circuit, and then classified, homogenized, and sampled.

After that, samples with size ≤ 2.0 mm were obtained by closed-loop circuit using Roller Grinding machine. The chemical composition is shown in Table 1, using analysis techniques including chemical volumetric method, atomic absorption spectrometry (AAS), inductively coupled plasma (ICP) atomic emission spectrometry and so on, where the TFe (Total Fe content) is $\sim 49\%$ and Ig (Mass loss of ignition) is 6.5%.

The Fe mineral phase and its distribution (by chemical composition method and identification & analysis of ore-minerals) are shown in Table 2, where the main mineral phase is Hematite-Limonite which accounts for 91% of TFe. The recoverable Fe for this type of oolitic iron ore is mainly in the form of Magnetite and Hematite-Limonite, where a theoretical recovery (ϵ) of Fe can be 91.23% (some Fe phase would be lost in tailings). Some amount of Fe in Siderite can be recovered. However, this part is within Ferric silicate, and cannot be utilized.

WG coal powder with size ≤ 1.0 mm was applied as reducing agent in roasting process. It was used to change the iron phase, hence to modify magnetic property for the facilitation of magnetic separation. The main industrial value of WG coal is shown in Table 3. As suggested, the coal contains a high content of fixed carbon (high as $\sim 78\%$) and comparatively low content of ash, volatiles, so the coal is a suitable reducing agent for coal-based reduction.

2.2. Analytical methods and procedures

For the study of magnetic roasting process and its influencing factor, Muffle Furnace (KSY-12D-16) was used, with the roasting outcome checked and validated by Magnetic Tube (XCGS-73). It is noted that Magnetic Tube is widely used in laboratory scale to determine the content of magnetic components in strong magnetic ores. For large scale application, in order to make it as real industrial practice, drum type weak magnetic separator ($\Phi 400 \times 400$) was applied to handle large quantity of feeding.

The detail procedure was as follow: the oolitic iron sample was thoroughly mixed in specific proportions by stirring with reducing agent (WG coal powder). The mixture was then transferred into a heat-resistant stainless-steel container, which was later roasted using Muffle Furnace. After a certain time of roasting, the reduced

Table 1
Chemical composition of raw oolitic iron ore, wt%.

TFe	FeO	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	CaO	MgO	S	P	Mn	Ig
49.02	5.17	64.34	6.52	11.03	7.53	3.06	0.06	1.18	0.22	6.54

Table 2
Fe mineral phases (distribution) within oolitic iron ore.

Mineral	Magnetite	Hematite/Limonite	Siderite	Ferric silicate	Sulphide ores	Total Fe
Fe content (%)	0.06	44.66	1.69	2.55	0.06	49.02
Distribution (%)	0.12	91.11	3.45	5.20	0.12	100.00

Table 3
Proximate quality analysis results of WG (Wugang, Wuhan iron and steel) coal.

Fixed carbon (%)	Calorific value (kJ/kg)	Volatile (%)	Moisture (%)	Ash (%)
78.42	27181	7.9	1.7	11.98

samples were rapidly removed and immediately quenched in water, filtered, dried, and ground by ball mill. Finally, its components were separated using Magnetic Tube.

Observation and counting method were conducted to examine the statistic ratio of reduction area for the roasted oolitic iron. Polarizing microscope (Leiz, Germany) was applied under various roasting conditions with different sizes. The roasted iron sample was firstly added to the adhesive medium (special cement), and then was cut into a test block of 40 mm \times 25 mm. After that, it was treated by rough grinding, fine grinding and polishing. Finally it was investigated under microscope.

Fe recovery (ϵ) is the percentage of the useful component within the concentrate to that of the original oolitic iron sample. It can be calculated as, $\epsilon = \gamma \times \beta / \alpha$, where γ is production yield, β is Fe content of the product and α is Fe content of the original oolitic iron. In order to determine the degree of transformation from Hematite (weakly magnetic) to Magnetite (strong magnetic mineral), reduction degree R was applied, i.e., the ratio between FeO and TFe after reductive roasting, $R = \text{FeO}/\text{TFe}$.

Conical ball mill, XMQ- $\Phi 150 \times 50$ was applied for lab scale grinding during Magnetic Tube test, and XMQ- $\Phi 240 \times 90$ for the case of drum type weak magnetic separator. X-ray diffraction (XRD) test was performed using Rigaku D/max-3c (Japan), with Cu K α radiation ($\lambda = 1.541 \text{ \AA}$), at an operating voltage of 40 kV and a current of 50 mA. The diffraction angle (2θ) was scanned from 10° to 90° , with a scanning speed of 2 (degrees)/min.

3. Results and discussion

3.1. Mineral compositions and mineralogical morphology

The XRD pattern of the raw oolitic iron ore is shown in Fig. 1. It was seen that the main minerals are Hematite, Ankerite, Chlorite, Collophanite, with a small amount of Kaolinite and Quartz. These results are in good agreement with the observation from microscope. The mineral component (by XRD) was listed in Table 4, where the percentages of three main minerals were 64%, 13% and 10%, respectively.

The oolitic particles were composed of Hematite and clay minerals containing layered structures, with Gangue showing circling layer distribution (Fig. 2a).

The inner structure of the oolitic particles varied with their particle sizes, and the sample mostly has 3–5 μm dense concentric wrapped shell, with siliceous quartz or clay like Kaolinite in the middle (Fig. 2b). In the raw iron sample, Al minerals mainly include Chlorite, Kaolinite, and the inclusion of Collophanite. Mg minerals mainly are Dolomite, Chlorite and Calcite.

The SEM image of the sample is illustrated in Fig. 3. The raw iron ores are mainly in the form of oolitic shape from microscopic view, and the iron ore can be readily observed and confirmed to be a typical oolitic iron ore [22] [24]. Hematite was the most

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