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Reduction behavior of boron-bearing iron concentrates by bituminous coal and its magnetic separation



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ABSTRACT

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Keywords: Boron-bearing iron ore Reduction and magnetic separation Metallic iron powder Boron-rich non-magnetic material In this study, the effects of temperature and time on the reduction behavior of boron-bearing iron concentrate briquettes were studied by using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive spectrometer (EDS). Results indicated that reduction temperature played a crucial role in the selective reduction of iron minerals in boron-bearing iron concentrate briquettes with bituminous coal as a reducer. The increase in reduction time facilitated the generation of metallic iron and the grain growth of the metallic phase in the reduced briquettes, which had a beneficial effect on the separation of iron and boron in the subsequent low-intensity magnetic separation process. The magnetic material contained 92.81% Fe with a recovery of 95.58%, while in the boron-rich non-magnetic material, the grade and recovery values of B₂O₃ were 14.11% and 89.59%, respectively. In addition, the efficiency of extraction of boron of the non-magnetic material was 80.76%. The magnetic material and boron-rich non-magnetic material were good raw materials for steel-making and borax production.

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1. Introduction

The boron chemical industry in China has rapidly developed in recent years requiring a large quantity of boron ores. Low-grade, polymetallic, and complex boron ores are currently the overwhelmingly dominant mineral resource for boron in China (Zhao and Wang, 2001; Liu et al., 2006). Therefore, China heavily depends on imported boron ore resources, resulting in an increase in the production costs of boric acid and borax. Using domestic resources to produce a steady supply of high-quality boron ores is an important step toward the sustainable development of the boron chemical industry in China.

Complex boron ores containing iron, magnesium, and other metals are abundant in China (Shen, 2013; Sun et al., 2014; Shao and Xiong, 2010; Zhen, 2009). Iron concentrate could be obtained from the complex boron-iron ores by using combined gravity beneficiation and magnetic separation processes. Such concentrate contains 4% to 6% B_2O_3 , accounting for 30% boron recovery. Thus, the boron recovery would be less than 70% if boron in the iron concentrate could not be utilized as raw material for the boron chemical industry. Several studies have been previously conducted (Lv, 2005; Zhu et al., 2005; Zhang et al., 1995; Liu et al., 1998; Zhao et al., 1996; Ding et al., 2012; Wang et al., 2012) and it was found that, although the associated boron could be effectively separated from the boron-bearing iron concentrates

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with the use of selective leaching processes, environmental pollution and equipment corrosion are the biggest obstacles. High-quality metallic pig iron could be potentially produced for electric arc furnace steelmaking by a blast furnace smelting process that effectively separates boron from iron oxides. However, industrial implementation of a blast furnace smelting process is difficult and characterized by low productivity, high energy consumption, and serious environmental pollution. Therefore, the commercial use of the complex iron ore as a boron source remains a challenge.

The objective of this study is to develop a novel process of selective reduction followed by magnetic separation to prepare high-quality metallic iron powder suitable for steelmaking and boron-rich slag suitable for boron chemical industry from boron-bearing iron concentrates. The reduction behavior of boron and iron oxides in briquettes was characterized by using physico-chemical analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive spectrometer (EDS).

2. Materials and method

2.1. Raw materials

2.1.1. Iron concentrate

The boron-bearing iron concentrate sample with 89.15% passing 0.074 mm was collected from an iron ore dressing plant in Liaoning, China. The sample is a fine-grained concentrate obtained by a combined gravity beneficiation and magnetic separation processes. The chemical

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Table 1	
Chemical composition of boron-bearing iron concentrate.	

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Composition	TFe	FeO	B_2O_3	SiO ₂	Al_2O_3	CaO	MgO	Р	S
Content/wt.%	55.55	25.67	4.22	3.60	0.33	0.10	10.10	0.007	1.44

compositions of the sample, determined by chemical methods combined with X-ray fluoroscopy (XRF), are presented in Table 1.

From the compositions in Table 1, the total iron content was 55.55%, whereas the total B_2O_3 , SiO_2 , and MgO contents were 4.22%, 3.60%, and 10.10%, respectively. The XRD pattern of the boron-bearing iron concentrate suggested that the boron-bearing iron concentrate was mainly composed of magnetite, szaibelyite, serpentine, and ludwigite (in Fig. 1).

The chemical phases of Fe and B and their distributions in the sample concentrate are presented in Table 2. It was observed that 95.19% of the Fe existed as magnetite. The mass fractions of B_2O_3 as szaibelyite and ludwigite were measured to be 95.86% and 4.14%, respectively.

Mineral liberation analyzer (MLA) images of magnetite and szaibelyite in the concentrate samples are shown in Figs. 2 and 3. Magnetite and szaibelyite were the main source minerals for Fe and B_2O_3 in the concentrate. Three forms of szaibelyite were observed in the concentrate: (a) irregular monomer (in Fig. 3), with a fine size varying from 0.01 mm and 0.045 mm; (b) superfine szaibelyite, encircled by the magnetite (in Fig. 3), with a fine size of 0.005 mm–0.02 mm; and (c) szaibelyite connected to the rim of the magnetite or embedded in the coarse magnetite (in Fig. 3) with a size ranging from 0.001 mm to 0.05 mm. These findings indicated a complex association between iron and boron mineral formations.

2.1.2. Coal

A bituminous coal was used as the reducing agent in this study. The results of proximate analysis of the bituminous coal are presented in Table 3. The coal was crushed and screened to a size range of 0 mm to 2 mm.

The fuel ratio of fixed carbon to volatile matter is an important criterion for coal combustibility, which is closely correlated to coal reactivity (Xie, 2002). A low fuel ratio corresponded to improved coal combustibility and reactivity, as well as obvious lingering combustion. As shown in Table 3, bituminous coal had a fuel ratio of 3.68, indicating that this material was a good reducing agent.

2.2. Experimental procedure

The experimental procedure included briquetting, reduction roasting of briquettes, and magnetic separation of reduced briquettes, presented in Fig. 4.



Fig. 1. XRD patterns of the boron-bearing iron concentrate ore samples.

Table 2

Chemical phases of Fe and B and their distribution in the concentrates.

Main elements	Distribution (%)			
Fe	In magnetite 52.87	In ludwigite 0.32	In iron sulfides 2.36	Total 55.55
	95.19	0.57	4.24	100
	In szaibelyite	In ludwigite		Total
B_2O_3	4.04	0.18		4.22
	95.86	4.14		100

A detailed set of bench-scale tests was conducted as follows. First, cylindrical green briquettes with a diameter of 15 mm and a height of 20 mm were produced by pressing 10.0 g of iron concentrate with 5% of water (5.0 mL water: 100.0 g iron concentrate) into a cylindrical mold for 1 min and then dried at 378 K for 6 h in the oven. Second, dry briquettes were mixed with a specific quantity of solid coals, loaded into a steel pot, and then reduced in a closed programmable box-type resistance furnace with the temperature controlled within +1 K. The reduction conditions in these tests included a roasting temperature ranging from 1050 $^\circ$ C to 1200 $^\circ$ C, a C_{Fix}/Fe_{Tot} weight ratio of 2.5 (the dosage of pulverized coal is excess, to ensure that the reduction of magnetite to metallic iron proceed sufficiently), and a coal size of 0 mm to 2 mm. After being reduced for a predetermined length of time, the briquettes were discharged from the furnace and cooled to room temperature in a steel barrel filled with water. Third, the total iron (TFe) and metallic iron (MFe) contents of the reduced briquettes were measured. Finally, magnetic separation was performed in the magnetic separation tube after the reduced briquettes were grounded in the cone ball mill. The models of cone ball mill and the laboratory magnetic equipment were XMQ- $ø150 \times 50$ and XCGS-ø50 Davis tube, respectively.

Chemical methods were used to analyze the total iron (TFe, wt.%) and metallic iron (MFe, wt.%) contents in reduced briquettes. The metallization degree (η) was calculated by using the following equation (Lankford et al., 1985; Qiu et al., 2001):

$$\eta = \frac{MFe}{TFe} \times 100$$

where η is the iron metallization degree, %; *MFe* is the total weight of metallic iron in reduced briquettes, %; and *TFe* is the total weight of iron in reduced briquettes, %.

The phase transformation of iron oxides and structures of the briquettes reduced by bituminous coal was studied using XRD, SEM, and EDS. The mineral compositions of reduction products, metallic iron and non-magnetic material were investigated by XRD (PANalytical X pert PW3040) with Cu K α radiation source. The operating voltage and current were 40 kV and 40 Ma, respectively. The diffraction angle



Fig. 2. MLA photo of irregular monomeric granular szaibelyite (A–szaibelyite, L–ludwigite).

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