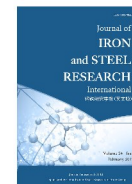




Contents lists available at ScienceDirect

Journal of Iron and Steel Research, International

journal homepage: www.chinamet.cn



# Recovery of boron from high-boron iron concentrate using reduction roasting and magnetic separation

Jian-wen Yu\*, Yue-xin Han\*\*, Peng Gao, Yan-jun Li

College of Resources and Civil Engineering, Northeastern University, Shenyang 110819, Liaoning, China

## ARTICLE INFO

**Key words:**

High-boron iron concentrate  
Carbothermic reduction  
Magnetic separation  
Metallic iron  
Boron-rich non-magnetic product

## ABSTRACT

The comprehensive utilization of abundant high-boron iron concentrate is of particular significance to China, and the high-boron iron concentrate has not yet been utilized as a source for boron at an industrial scale due to its complex mineralogy and fine mineral dissemination. An innovative method was proposed for recovery of boron and iron from high-boron iron concentrate by reduction roasting and magnetic separation. The effects of reduction temperature and roasting time were investigated and their optimum conditions were determined. The mineralogical changes during roasting were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results showed that the pyrrhotite (FeS) contained in the high-boron iron concentrate and the new-formed FeS-Fe solid solution softened or melted at high temperatures owing to their low melting points, and then decreased the metallic iron ratio and accelerated the growth of metallic iron particles. Meanwhile, the magnetite and szaibelyite were converted into metallic iron and suanite, respectively. Consequently, boron was readily enriched into the non-magnetic product and the metallic iron was aggregated to the magnetic concentrate by magnetic separation. Boron recovery of 88.6% with corresponding  $B_2O_3$  content of 14.5% and iron recovery of 95.1% with an iron grade of 92.7% were achieved when high-boron iron concentrate was reduced at 1125 °C for 150 min. Besides, the boron reactivity of the boron-rich non-magnetic product was up to 80.8%.

## 1. Introduction

Boron is widely used in many industries, including metallurgical, chemical, medical, and nuclear industries<sup>[1]</sup>. The szaibelyite ore, as the traditional boron resource in China, is near its depletion. Thus, it is increasingly challenging to meet the growing demand of the boron chemical industry in China. There is a growing interest in the utilization of boron-bearing iron ore, which is abundant in Liaoning province, China, with a reserve of 28.43 Mt  $B_2O_3$  (57% of the total in China)<sup>[2–4]</sup>.

At present, boron-bearing iron concentrate and boron concentrate can be obtained from boron-bearing iron ore via the process of magnetic-gravity separation. The boron concentrate contains 12–16 wt. %  $B_2O_3$ , which can be used as a high-quality raw material for boron chemical industry after activated roasting, but the  $B_2O_3$  content in the iron concentrate reaches 4–6 wt. %<sup>[5,6]</sup>. In order to further separate bo-

ron from boron-bearing iron concentrate, a blast furnace and a solid pre-reduction followed by electric arc furnace melting have been proposed<sup>[7–11]</sup>. However, both of these two current routes face the same crucial problem that molten boron-rich slag has to be cooled slowly to obtain the alkali-dissolution activity of boron, which affects the yield of boron resource.

Reduction roasting followed by magnetic separation is an effective way for the treatment of refractory iron ores<sup>[12,13]</sup>. The aim of this work was to separate boron and simultaneously recover iron from high-boron iron concentrate based on direct reduction roasting followed by magnetic separation process, and a technological route of combined metallic reduction roasting, grinding, and magnetic separation was proposed. The key attribute of this process was the control of reduction roasting conditions, which led to the selective reduction of magnetite into metallic iron rather than boron oxide at low reducing temperatures. After reduction roasting pre-

\* Corresponding author. Ph.D.; Tel.: +86-24-8368 6828.

E-mail address: neu\_mpc237@163.com (J. W. Yu).

\*\* Corresponding author. Prof.; Tel.: +86-24-8368 0162.

E-mail address: dongdafulong@mail.neu.edu.cn (Y. X. Han).

treatment, the metallic iron was effectively aggregated to magnetic concentrate and the boron was enriched into non-magnetic product by magnetic separation. Thus, the effects of reduction temperature and time were investigated. Moreover, the new method eliminated slow cooling and therefore might be potentially more economical in comparison with the existing methods.

## 2. Experimental

### 2.1. Raw materials

The high-boron iron concentrate with 89.2% passing 0.074 mm was taken from Liaoning province of China, and the chemical compositions of the sample, determined by chemical methods combined with X-ray fluorescence (XRF), are presented in Table 1. The high-boron iron concentrate contained 55.55 wt. % Fe and 4.22 wt. %  $B_2O_3$ . The content of main detrimental impurity S was 1.44 wt. %, which was present in the form of pyrrhotite (FeS).

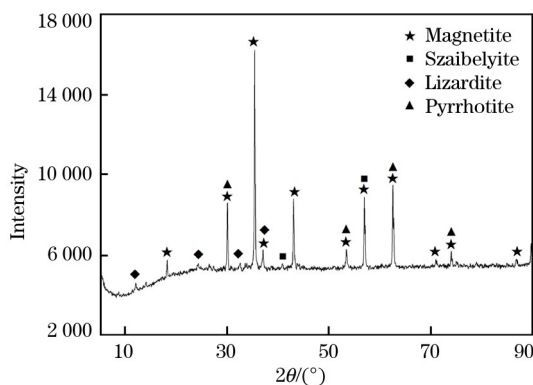
X-ray diffraction (XRD) results (Fig. 1) indicated that the boron-bearing iron concentrate ore was mainly composed of magnetite, szaibelyite, pyrrhotite and lizardite.

The bituminous coal obtained from Jilin Coal Mining Co., Ltd., China was used as reducing agent, which was pulverized and screened to  $-2$  mm. The proximate analysis of the bituminous coal is presented in Table 2, and the fixed carbon is 67.83 wt. %.

**Table 1**

Main chemical composition of boron-bearing concentrate ore sample (wt. %)

TFe	$B_2O_3$	$SiO_2$	MgO	CaO	$Al_2O_3$	P	S
55.55	4.22	3.60	10.10	0.10	0.33	0.007	1.44



**Fig. 1.** XRD pattern of the boron-bearing iron concentrate ore.

**Table 2**

Proximate analysis of the bituminous coal

Fixed carbon/ wt. %	Volatile matter/ wt. %	Ash content/ wt. %	Moisture/ wt. %	Fuel ratio
67.83	18.45	12.00	1.48	3.68

### 2.2. Reduction roasting and magnetic separation

The high-boron iron concentrate was thoroughly mixed with 5% water to prepare non-coal-burdened briquettes with diameter of 15 mm and height of 20 mm. The bituminous coal, as the external reductant, was crushed and screened to a size range of 0–2 mm. After drying, about 40 g dry briquettes were mixed with an excessive amount (82 g) of coal to ensure the sufficient reducing atmosphere (mole ratio of fixed carbon to total iron is 2.5). The mixture of briquettes and coal was loaded into a cylindrical heat-resistant stainless steel pot. Then, the pot was subjected to a box resistance furnace (KJ, model KSL-1400X) and roasted for a given period of time. After roasting, the reduced briquettes were cooled to room temperature in the pot isolated from the atmospheric oxygen. A 10 g sub-sample of the cooled, reduced briquettes was ground to about 65% passing 0.074 mm in an XMQ- $\Phi 150 \times 50$  ball mill. The slurry was then separated in an XCGS-50 Davies magnetic tube using a magnetic field intensity of 0.08 T. Finally, the obtained magnetic and non-magnetic products were metallic iron powder and boron-rich concentrate, respectively. Meanwhile, iron and boron recoveries were obtained through mass balance calculation. The overall experimental flow sheet is presented in Fig. 2.

### 2.3. Measurements

After reduction roasting pre-treatment, a sub-sample of the cooled, reduced briquettes was ground to less than 0.074 mm for determination of metallic iron and total iron content by chemical titration method. Thus, the metallic iron ratio was calculated according to the following equation:

$$\lambda = (W_{MFe} / W_{TFe}) \times 100\% \quad (1)$$

where,  $\lambda$  is the metallic iron ratio of the reduced sample;  $W_{TFe}$  is the total iron content of the reduced sample; and  $W_{MFe}$  is the metallic iron content of the reduced sample.

To evaluate the boron reactivity in the non-magnetic product, the normal pressure alkaline leaching method was applied. The particle size of the non-magnetic material was 90% passing 0.074 mm. The concentration of NaOH was 20% and the volume of lye was 40 mL. The leaching time was 4 h. The boron contents of primary non-magnetic material and filter residue were analyzed by using the inductively coupled plasma-atomic emission spectrometry (ICP-AES) method. The boron reactivity, designated as  $\eta$ , can be defined as follows:

$$\eta = (\beta_0 m_0 - \beta_1 m_1) / \beta_0 m_0 \times 100\% \quad (2)$$

where,  $m_0$  is the amount of primary non-magnetic material;  $\beta_0$  is the  $B_2O_3$  content of primary non-magnetic material;  $m_1$  is the amount of filter residue;

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