



Effects of embedding direct reduction followed by magnetic separation on recovering titanium and iron of beach titanomagnetite concentrate

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ABSTRACT

Embedding direct reduction followed by magnetic separation was conducted to fully recover iron and titanium separately from beach titanomagnetite (TTM). The influences of reduction conditions, such as molar ratio of C to Fe, reduction time, and reduction temperature, were studied. The results showed that the TTM concentrate was reduced to iron and iron-titanium oxides, depending on the reduction time, and the reduction sequence at 1200 °C was suggested as follows: $\text{Fe}_{2.75}\text{Ti}_{0.25}\text{O}_4 \rightarrow \text{Fe}_2\text{TiO}_4 \rightarrow \text{FeTiO}_3 \rightarrow \text{FeTi}_2\text{O}_5$. The reduction temperature played a considerable role in the reduction of TTM concentrates. Increasing temperature from 1100 to 1200 °C was beneficial to recovering titanium and iron, whereas the results deteriorated as temperature increased further. The results of X-ray diffraction and scanning electron microscopy analyses showed that low temperature (≤ 1100 °C) was unfavorable for the gasification of reductant, resulting in insufficient reducing atmosphere in the reduction process. The molten phase was formed at high temperatures of 1250–1300 °C, which accelerated the migration rate of metallic particles and suppressed the diffusion of reduction gas, resulting in poor reduction. The optimum conditions for reducing TTM concentrate are as follows: molar ratio of C to Fe of 1.68, reduction time of 150 min, and reduction temperature of 1200 °C. Under these conditions, direct reduction iron powder, assaying 90.28 mass% TFe and 1.73 mass% TiO_2 with iron recovery of 90.85%, and titanium concentrate, assaying 46.24 mass% TiO_2 with TiO_2 recovery of 91.15%, were obtained.

1. Introduction

Beach titanomagnetite (TTM) ore of Indonesia, the second largest reserve of beach metal mineral resources (approximately 824 million tons in the world), is a complex iron ore consisting of iron and titanium. Given its high Fe content (38–60 mass%), the beach TTM ore is currently used as the main material for the blast furnace process in some countries^[1,2]. Most of the titanium components in the ore are separated from iron and concentrated into the slag in the blast furnace process, forming the titanium slag, which contains 22–25 mass% TiO_2 and 2–6 mass% metallic iron. So far, there is no appropriate and economic method to utilize such slag^[3–5], and these slags also resulted in titanium waste and environmental pollution^[4].

Studies from the past decades focused on developing an alternative route for recovering iron and titanium from the TTM ore; one of the potential choices is the coal-based direct reduction process^[5,6]. In coal-based direct reduction method, the TTM ore mixed

with coal and additives is pressed into composite briquettes, and then the composite briquettes are reduced at high temperature; finally, the reduced briquettes are ground and separated by magnetic separation. The magnetic product is called direct reduction iron powder (DRIP) and used as the raw material for electric arc furnace (EAF)^[4–8]. The non-magnetic products are known as titanium products. Although the process is efficient, coal ash and most additives could fall into the non-magnetic product after direct reduction and magnetic separation, thereby leading to low TiO_2 grade of non-magnetic products (approximately 20 mass%)^[3–5], and the TiO_2 content is even lower than that in the blast furnace slag; the non-magnetic products need to be further refined by chemistry method, including hydrometallurgical processing route, direct acid leaching, or selective chlorination^[9–12]. These chemical methods may cause serious environmental pollution because of poisonous gases and wastewater produced.

Compared with coal-based direct reduction of

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composite briquette, embedding direct-reduction^[13] of pellets is more favorable. In this process, the beach TTM concentrates are pressed into pellets and then are completely embedded into the coal to avoid the impact of coal ash and additives. The reduced pellets are separated by magnetic separation to obtain the DRIP and non-magnetic product. Without the effect of coal ash and additives, the high TiO₂ content of non-magnetic product may be obtained in theory, which could be used as a raw material for titanium-rich material.

A previous study^[13] focused only on the effects of Na₂SO₄ addition and ignored other factors. The main objective of the current paper is to investigate the effects of embedding direct reduction followed by magnetic separation on recovering titanium and iron. The influences of reduction conditions, such as molar ratio of C to Fe, reduction temperature, and time, on the reduction of TTM concentrate were studied.

2. Materials and Methods

2.1. Raw material

Raw beach TTM ore was obtained from Indonesia and ground to 90 wt. % passing 0.074 mm. The TTM concentrate used in this study was obtained from raw beach TTM by using low intensity magnetic separation at a magnetic field intensity of 80 kA/m. The TTM concentrate assayed 57.29 mass% TFe, 11.42 mass% TiO₂, 3.01 mass% SiO₂, 2.90 mass% Al₂O₃, 2.70 mass% MgO, and 0.37 mass% MnO. The phosphorus, sulfur, and vanadium contents were very low. The detailed information of the TTM ore has been described in a previous paper^[13]. Bituminous coal, with proximate analysis results of 54.83% fixed carbon, 13.97% ash, 31.21% volatile matter, and 13% moisture content, was used as a reductant. The sizes of bituminous coal used were less than 2 mm.

2.2. Pellet formation, reduction, and separation

The TTM concentrate was pressed to form pellets before subjecting to reduction roasting by the following procedures: 15 g TTM concentrate and water (5 wt. %) were mixed together; the mixture was then pressed to form a pellet using a die with diameter of 28 mm with the aid of a hydraulic equipment. The pellet is a flat sphere with the height of 8 mm. Two pellets were embedded in the bituminous coal placed in a crucible with a cover. Finally, the crucible was placed into a muffle furnace at a setting temperature for a certain time. After reduction, the reduced pellets were cooled to room temperature in the crucible, crushed to -2 mm, and then separated by two-stage grinding and wet magnetic separation. Grinding was conducted in a rod mill (RK/BM-1.0L).

The reduced ore was ground to about 75.00 wt. % passing 0.074 mm at the first grinding stage and then was ground to about 60.00 wt. % passing 0.043 mm at the second grinding stage in a rod mill. An XCGS-73 magnetic tube with a magnetic field intensity of 151 kA/m was used to recover metallic iron. It is noted that the magnetic field intensity was also an important factor for the recovery of iron and titanium in the magnetic separation process. The effects of magnetic field intensity on recovering iron and titanium were studied in previous study^[14]. Therefore, the optimal magnetic field intensity of 151 kA/m was used in present study. The magnetic and non-magnetic products were designated DRIP and titanium concentrate, respectively. The test flowsheet used referred to a previous study^[13].

The Fe grade, Fe recovery, and TiO₂ content of the DRIP, as well as the TiO₂ grade and recovery of the titanium concentrate were used to assess the effect of embedding direct reduction followed by magnetic separation. The Fe and TiO₂ recoveries were calculated by Eqs. (1) and (2), given as follows:

$$F_r = \frac{m_D \cdot F_D}{m_T \cdot F_T} \times 100\% \quad (1)$$

$$T_r = \frac{m_t \cdot T_t}{m_T \cdot T_T} \times 100\% \quad (2)$$

where, F_r and T_r are the Fe and TiO₂ recovery rates; m_D , m_T and m_t are the mass of DRIP, TTM concentrate and titanium concentrate, respectively; F_D and F_T are iron content of DRIP and TTM concentrate, respectively; T_t and T_T are TiO₂ content of titanium concentrate and TTM concentrate, respectively.

The molar ratio of C to Fe was calculated by:

$$MR = \frac{(m_1 \cdot C_1)/12}{(m_2 \cdot C_2)/56} \quad (3)$$

where, MR is the molar ratio of C to Fe; m_1 and m_2 are the mass of bituminous coal and TTM concentrate, respectively; C_1 and C_2 are the fixed carbon content in bituminous and the iron content in TTM concentrate, respectively; 12 and 56 are the molecular mass of carbon and iron, respectively.

2.3. Analysis and characterization

X-ray diffraction (XRD) patterns were recorded using a diffractometer (Rigaku D/Max2500, Japan) under the following conditions: CuK α radiation, tube current of 150 mA and voltage of 40 kV, scanning range of 10° to 100°, step size of 0.02°, and scanning speed of 5 (°)/min. Polished sections were prepared to observe the morphological changes and to analyze the element content of the roasted pellets using a scanning electron microscope (SEM, Carl Zeiss EVO18, Germany) equipped with an energy dispersive X-ray spectroscopy (EDS) detector (Bruker XFlash Detector 5010, Germany). SEM images were recorded in backscatter electron modes operat-

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