



Chemical and mineral transformation of a low grade goethite ore by dehydroxylation, reduction roasting and magnetic separation



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ABSTRACT

The utilization of abundant low grade goethite (α -FeOOH) ores is potentially important to many countries in the world, especially Australia. These ores contain many detrimental impurities and are difficult to upgrade to make suitable concentrates for the blast furnace. In this paper, chemical and mineral transformations of a goethite ore were studied by dehydroxylation, reduction roasting in CO and CO₂ gas mixtures, and magnetic separation. The goethite sample was taken from a reject stream at an iron ore mine from the Pilbara region, Western Australia. The roasting temperature range investigated was 400–700 °C. Chemical and mineralogical analysis was conducted using XRF, XRD, optical microscope, EPMA, and SEM. Magnetic separation was conducted using a Davis tube tester and a high intensity magnetic separator.

The results show that reduction roasting can remove moisture and impurities but does not significantly change the Fe content in the feed. However, reduction roasting transforms goethite to hematite and eventually maghemite which can be recovered by magnetic separation, allowing upgrading. Further studies are needed to optimize the reduction roasting and correlate it with the magnetic separation to maximize the efficiency of iron upgrading.

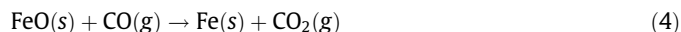
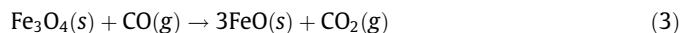
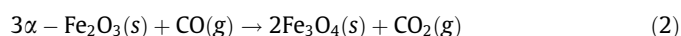
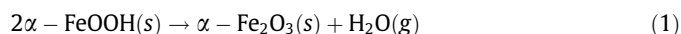
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1. Introduction

Mineral resources including iron ores are non-renewable and are becoming exhausted. As such the difficult-to-process ores such as goethite (α -FeOOH) ores are being investigated for further needs. In fact, there are many goethite ore regions in Australia, such as the Pilbara region in Western Australia which are not currently mined and processed. Moreover, goethite is also the main component of many iron ore plant reject streams. The processing of goethite ores is inevitable. Although variable methods are available to upgrade lower grade iron ores in a number of fields in the world, this study focuses on the chemical and mineral transformation of goethite ores from the Pilbara region by dehydroxylation, reduction roasting and magnetic separation.

There are many stages reported for the transformation of goethite into minerals such as hematite and magnetite which are suitable for magnetic separation. Depending on temperature and reduction conditions, goethite can be transformed to hematite, hematite to magnetite, magnetite to wustite and wustite to pure

iron. The transformation can be described by the following chemical reactions in the presence of a mixture of CO and CO₂ gases:



Reducibility of goethite and hematite can be achieved by using many other reducing agents, including carbon [$\text{C}(s) + \text{O}_2(g) \rightarrow \text{CO}(g)$] and hydrogen [$\text{H}_2(g) + \text{O}_2(g) \rightarrow \text{H}_2\text{O}(g)$]. It is noted that the oxygen partial pressure generated by the agents has to be below that in equilibrium with the solid to be reduced. Hydrogen and carbon monoxide can be relatively cheaply obtained from natural gas or coke. Biomass as a renewable solid fuel has also been studied for the substitution of natural gas and coal (Luo et al., 2011; Ueki et al., 2013; Wu et al., 2012).

Although many other oxidants can be selected for reducing of goethite and hematite, mixtures of carbon monoxide and carbon dioxide are often used in small-scale studies because of safety and low costs. Furthermore, the heating of the reactants is required

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to a temperature at which carbon monoxide becomes more stable than the metal oxide during the reduction of the metal oxide with carbon. According to this principle, when the CO/CO₂ gas mixtures are utilized in the reduction of iron oxide, heat should be provided to increase the temperature to the point which carbon dioxide becomes more stable than the iron oxide. In addition, the content of CO in the CO₂/CO gas mixture could be a significant parameter controlling the appropriate phase transformation of iron oxides. The gas ratio could be determined according to the thermodynamic principles and the phase diagram shown in Fig. 1. The carbon dioxide in the mixture should be high to avoid the unnecessary transformation to Fe₃C and Fe. In the environment of high CO content, many additional phase transformations to iron oxides could take place (Yu and Qi, 2011) as presented in Fig. 1. Furthermore, small amount of the CO gas (lower than 50%) in the mixture can convert goethite to magnetite if the roasting temperature is appropriately chosen. Therefore, the gas ratio and temperature can be the significant parameters of the Fe phase transformation, which has to be controlled, to avoid the unnecessary phase transformation of iron oxides.

Researches have investigated the changes of iron ores as a function of temperature by thermal processing (Gialanella et al., 2010; Ruan and Gilkes, 1995; McCann et al., 2004; O'Connor et al., 2006; Strezov et al., 2011; Valix and Cheung, 2002; Yu and Qi, 2011). The chemical and structural changes of iron ore samples versus the temperature have been identified: The transformation of iron ores starts at the temperature between 120 °C and 140 °C with the free moisture removal. The dehydroxylation of goethite occurs between 250 °C and 350 °C. The decomposing of kaolinite and the magnetic transformation of magnetite happen at around 600 °C and 680 °C, respectively. Finally, the partial reduction of hematite to magnetite occurs at around 850 °C.

Hematite can be created from goethite through dehydroxylation during heating as shown by Eq. (1) (Strezov et al., 2011). This process can occur by roasting at low temperature. It involves the structural transformation leading to the destruction of the structural OH-groups or loss of mineral crystalline bound water (Valix and Cheung, 2002). The decomposition temperature is usually about 337 °C for highly crystalline goethite (Swamy et al., 2003). A strong endothermic reaction occurs in raw limonite ores at a temperature between 210 °C and 370 °C (O'Connor et al., 2006).

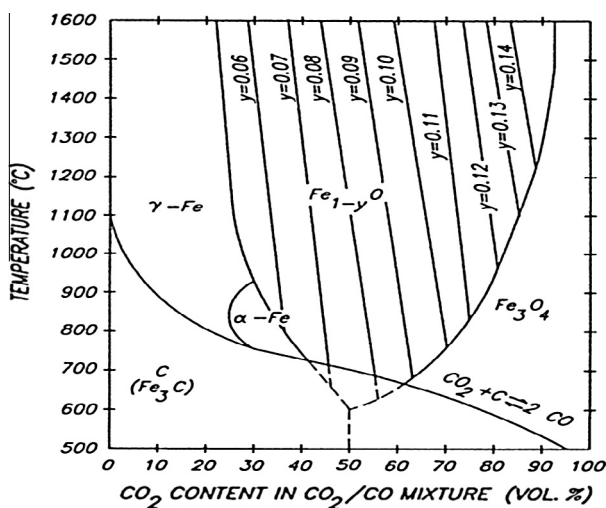


Fig. 1. Phase equilibrium (Baur-Glaessner diagram) between iron, wustite, magnetite, carbon monoxide, carbon dioxide and carbon at total partial pressure $P_{CO} + P_{CO_2} = 1$ atm (Hayes, 2003).

In addition, another literature indicates that at temperatures between 200 °C and 250 °C, the reduction of Fe₂O₃ to Fe₃O₄ starts, and the required temperatures for the reduction of Fe₃O₄ to FeO and FeO to Fe are between 500 °C and 900 °C and between 900 °C and 1300 °C, respectively (Hayes, 2003). Dehydroxylation of goethite occurs by roasting at low temperature to transform goethite into hematite and the reduction of iron oxides is progressed through several stages.

Direct magnetization and magnetic separation of roasted iron ores has also been examined (Wu et al., 2012). The goethite iron ore contains hematite, goethite plus other minerals such as kaolinite and quartz, which contain high levels of silicon and aluminum. In nature, ochreous goethite is mixed with a considerable amount of aluminum through grained and clayed particles, whereas vitreous goethite is hard and crystalline, and rich in iron (Das et al., 2010). Thus, one of the key purposes of this investigation was to remove the impurities such as Si and Al. The existence of iron in some ferromagnetic form, such as magnetite, could create paramagnetic properties of the material, and the paramagnetics could be attracted along the lines of magnetic force to the points of greater field intensity (Napier-Munn and Wills, 2006). The most ferromagnetic iron compound is magnetite although hematite and siderite can be separated by magnetic separation after phase transformation into magnetite by reduction roasting. The magnetic separator divides the samples into two products, magnetic and non-magnetic. Therefore, magnetic separation after reduction roasting was carried out to remove non-magnetic material, such as impurities from the iron ore sample used in this study.

The work conducted aims to help understand the chemical and mineral transformation of goethite ores by reduction roasting. The particular focus is on removing impurities and upgrading the goethite iron ores by reduction roasting and magnetic separation.

2. Experimental

The goethite sample was obtained from an iron ore reject stream from the Pilbara region in Western Australia. The top size of particles in the sample was 2 mm. After dividing the sample into different size fractions by wet screening, the chemical composition and mineralogy of the sample was characterized by XRF (X-ray Fluorescence), optical microscopy, point counting and EPMA (Electron Probe Microanalysis).

Three different roasting studies were conducted. In the first series of experiments, batch roasting was conducted in a tube furnace (Fig. 2) with 50 g of raw sample at seven different temperatures (400, 450, 500, 600, 650, 700 and 750 °C) in 5% CO/95% CO₂ gas environment. The aim of these experiments was to establish the relevant conditions such as temperature, roasting time and gas flow for the phase transformation and dehydroxylation of goethite. The time of each roasting experiment was 1 h with N₂ gas purging and 20 min roasting followed by cooling down to the room temperature for 10 min. The gas flow rate was 2 L/min. The solid products of the roasting were separated and further analysed by low intensity magnetic separation with a Davis Tube Tester (DTT) (Senftle et al., 1994). All the samples were characterized by XRD (X-ray Diffraction), optical microscopy and point counting.

In the second series of experiments, batch bulk roasting of 1000 g sample at 450 °C was conducted in ambient air (i.e., open atmosphere) to investigate dehydroxylation. The bulk roasting took about 2 h and 25 min to reach at the target temperature, and then the temperature was maintained at 450 °C for 30 min for roasting. The roasted ore samples were characterized by Wet High Intensity Magnetic Separation (WHIMS) to define the mineral phase transformation of goethite utilizing optical microscopy and point counting. The most important purpose of these analyses

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