



Synthesis of magnetite powder from the mixture consisting of siderite and hematite iron ores

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ABSTRACT

Phase transformation of weakly magnetic iron minerals into a strongly magnetic magnetite is a promising technique for iron ore beneficiation with the further magnetic separation. Thermal decomposition of siderite leads to both magnetite and carbon monoxide formation. Carbon monoxide, as a reductant, sequentially triggers the transformation of hematite into magnetite. Therefore, this method of magnetite formation does not require any additional reducing agent. In the present paper, the source materials were hematite and siderite ores, which were mixed in different mass ratios. Phase transformation of ores' mixture was performed at various temperatures and for various time intervals. It was shown within the experiments that siderite and hematite transformed to magnetite. The starting temperature of magnetite formation was about 400 °C. X-ray diffraction analysis confirmed the formation of the spinel structure that corresponds to magnetite. The saturation magnetization of transformed samples increased immensely up to 70 Am²/kg for the sample in 1:1 ratio of hematite to siderite. Optimal conditions for the conversion included heating the mixture at 600 °C for 12 min. Under these conditions, siderite and hematite completely transformed to magnetite, since solely magnetite was detected in transformed samples. Simultaneous roasting of hematite and siderite ores is a simple and an economically efficient technique for magnetic powder preparation.

1. Introduction

Iron and steel have been the basis of industry in many countries for centuries. Consequently, a considerable attention in many research works focuses on the exploration and exploitation of iron ore deposits. High-grade iron ore is suitable for the direct reduction process to produce iron. On the other hand, low-grade ore containing magnetite is suitable for beneficiation by magnetic separation. After many years of iron ore mining, magnetite deposits as well as high-grade iron ore deposits are nearly exhausted. There is an increasing necessity to beneficiate low-grade iron ore, which is not suitable for magnetic separation without some pretreatment.

It is known that strongly magnetic magnetite (Fe₃O₄) can be easily separated from nonmagnetic components of magnetite's quartzite. In contrast, ore is difficult to beneficiate if it contains weakly magnetic siderite (FeCO₃), goethite (FeOOH), or hematite (Fe₂O₃) as a main iron-bearing mineral. The aim of beneficiation is to remove undesired impurities and to increase the concentration of iron. Many techniques have been developed for hematite ore beneficiation, such as magnetic separation, gravimetric separation, combined gravimetric/magnetic separation, flotation beneficiation, and others. However, practical

application of the methods listed above is still very limited and requires improvement. In many cases, quality of iron ore concentrate can be rather low to fulfill the requirements of metallurgy.

In order to beneficiate low-grade iron ore, preliminary magnetizing roasting can be used in combination with the further magnetic separation. Magnetizing roasting is a method of thermally induced chemical transformation of weakly magnetic iron-bearing minerals (hematite, goethite) into strongly magnetic materials. The method leads to chemical and structural transformations of iron oxides and hydroxides.

A great variety of chemical techniques (e.g. reduction, oxidation, and thermal decomposition) has been used to transform weakly magnetic minerals into strongly magnetic iron oxides. For example, thermal decomposition of lepidocrocite (γ-FeO(OH)) leads to the formation of maghemite (Cudennec and Lecerf, 2005; Gehring and Hofmeister, 1994; Gendler et al., 2005; Guo and Barnard, 2013; Hanesch et al., 2006). Oxidation of Fe(II) to Fe(III) during siderite decomposition leads to the formation of magnetite (Isambert et al., 2003; Pan et al., 2000; Ponomar et al., 2017a; Qu et al., 2011). Magnetite formation is also possible due to the partial reduction of Fe(III) to Fe(II) in goethite (Dudchenko and Ponomar, 2015; Jang et al., 2014) or hematite structures (Gaviría et al., 2007; Longbottom and Kolbeinsen, 2008;

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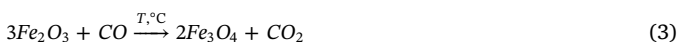
Matthews, 1976; Pineau et al., 2006; Ponomar et al., 2017b; Sakthivel et al., 2010).

Hematite and goethite are the most abundant minerals of low-grade iron ores. They are also the main components of iron waste materials (tailings, red muds, etc.) (Sakthivel et al., 2010; Wu et al., 2011). Hematite and goethite transformation to magnetite requires a reductant such as carbon monoxide (Jang et al., 2014; Monazam et al., 2014), hydrogen (Gaviría et al., 2007) or carbohydrates (Dudchenko and Ponomar, 2015; Ellid et al., 2003) in addition to the high temperatures. However, thermal treatment of siderite leads to the formation of carbon monoxide from the carbonate group. This reaction is well known, but its practical use for the reduction of iron ores has been not previously characterized, with the exception of some works (Chun et al., 2015).

Generally, thermal decomposition of siderite includes two main reactions:



As a result, siderite decomposition produces a molecule of carbon dioxide (CO_2) and a molecule of wüstite (FeO). Besides, according to the Eq. (2), three molecules of wüstite undergo oxidation by carbon dioxide to form one molecule of magnetite (Fe_3O_4) and one molecule of carbon monoxide (CO). Carbon monoxide, consequently, may cause the reduction of hematite to magnetite. This reaction can be written as follows:



Peculiarities of hematite to magnetite reduction are well known as they are widely investigated in the literature (Gaviría et al., 2007; Sakthivel et al., 2010). Oxidation of wüstite may also occur in reaction with hematite. Oxidation properties of hematite have been investigated for chemical looping combustion (Mendiara et al., 2013; Monazam et al., 2014, 2013). Therefore, the reaction described by the following equation may take place:



The formation of hematite from siderite is also possible, but it depends on reaction temperature, gaseous atmosphere, gas flow rate, particle size of the ore, quantity of the sample, and pressure (Dhupe and Gokarn, 1990). Overall, conditions in which siderite transforms into hematite or magnetite, are recognized in the literature (Isambert et al., 2003; Jagtap et al., 1992; McCollom, 2003; Ponomar et al., 2017a,b; Qu et al., 2011).

According to the Eqs. (1), (2), (3), and (4), magnetizing roasting of siderite/hematite mixture can be described by the following generalized scheme:



The efficiency of the process described theoretically with the help of the scheme (5) requires an experimental confirmation. It is also necessary to determine the conditions under which the complete transformation of siderite and hematite into magnetite occurs.

Hence, the aim of this work is to characterize the transformation of ores' mixture of different hematite to siderite ratios, for various times, and at various temperatures by thermomagnetic analysis, X-ray diffraction analysis, and magnetometry measurements.

2. Materials and methods

2.1. Materials

The sample of siderite ore from Bakal deposit (the western slope of the Southern Urals) was ground up to the size of particles < 0.07 mm. Mineral composition of the ore was siderite and ankerite in trace

amounts. The initial sample did not demonstrate strong magnetic properties.

The sample of hematite ore was collected from Kryvyi Rih iron ore basin and was ground to the size of particles < 0.07 mm. A magnetic fraction of the sample was removed by wet low-intensity magnetic separation. The sample consists of hematite and minor quartz.

2.2. Synthesis procedure

Both ores (hematite- and siderite-bearing) were ground to a particle size < 0.7 mm and mixed in various ratios of siderite to hematite. The mixture with a mass of 0.5 g was heated at 400–650 °C for 5–20 min in a quartz reactor using thermomagnetic analysis equipped with gas analyzer. Thus, the transformation experiments were accompanied by a continuous measurement of the sample's magnetization and analysis of the gas emanating from the reactor.

2.3. Thermomagnetic analysis

Thermomagnetic measurements were performed by a custom-built facility that enables measurements of the force affecting the sample in a magnetic field (Fig. 1). This force is proportional to the magnetization of the samples. The applied steady field of 300 mT is sufficiently high to reach the saturation magnetization of magnetite. The heating and cooling runs of the synthesis and analysis were carried out in a quartz reactor with the rate of 65°/min. Thermomagnetic data includes derivative thermomagnetic curves (DTMC) and integral thermomagnetic curves (TMC).

The gas flow rate was measured for 1 g of siderite using differential manometer by comparing the pressure at different points in the pipe. The system was calibrated in such a way that the difference in pressure can be recalculated to the volume per time unit. The concentration of carbon monoxide was measured using an electrochemical CO-110 sensor. The sensor measures the CO concentration in the range of 0 to 1000 ppm. In order to measure higher concentrations, the analyzed gas was diluted with air in known proportions. The content of CO in the outlet gas was estimated for 1 g of pure siderite and for the mixture consisting of 1 g of siderite and 1 g of hematite. The heating rate was 60°/min.

2.4. X-ray Fluorescence analysis

Chemical composition of the samples was determined using X-ray

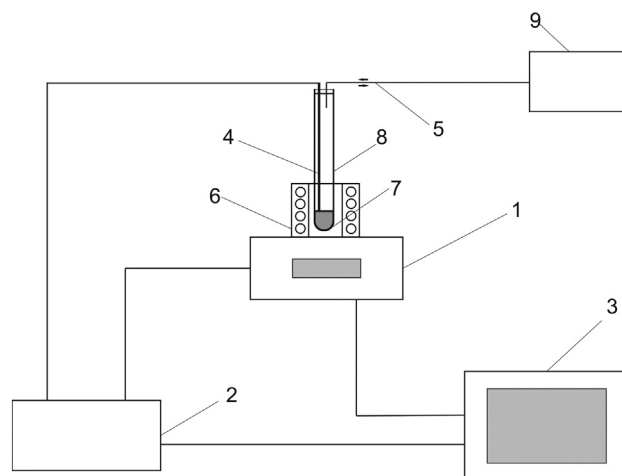


Fig. 1. Scheme of the facility for thermomagnetic measurements: 1 – magnetic digital balance, 2 – digital temperature controller, 3 – personal computer, 4 – thermocouple, 5 – gas outlet, 6 – heating element, 7 – test sample, 8 – quartz reactor, 9 – gas analyzer.

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