



Synthesis and magnetic properties of magnetite prepared by chemical reduction from hematite of various particle sizes



V.P. Ponomar*

M.P. Semenenko Institute of Geochemistry, Mineralogy and Ore Formation of NAS of Ukraine, Kyiv, Ukraine

ARTICLE INFO

Article history:

Received 13 July 2017

Received in revised form

1 December 2017

Accepted 2 January 2018

Available online 4 January 2018

Keywords:

Magnetite

Hematite

Gas-solid reactions

Reduction

Tailings

Magnetic measurements

ABSTRACT

In the process of beneficiating iron ores by the magnetic separation, particles of hematite and quartz accumulate, forming tailings that occupy large areas of agricultural lands. Such man-made deposits are quite common and contain large quantities of iron oxides such as hematite. Therefore the tailings can be used as raw material for the production of magnetite. In this work, magnetite was synthesized by hematite reduction at a temperature of 500 °C in an atmosphere of carbon monoxide. The source hematite was separated initially into ten particle size categories ranging from 0.05 to 2.5 mm. The reduction was carried out with carbon monoxide, which was synthesized by reaction of air and activated carbon at 750 °C. It was determined by X-ray diffraction analysis and magnetometry that, after reduction, samples of each particle size consisted mainly of magnetite. In addition, the magnetic properties of the resulting magnetite were noted to be somewhat different from those of natural magnetite. In particular, remanence and coercivity are higher for synthetic magnetite than for natural magnetite. The saturation magnetization of transformed samples increases to 60–80 Am²/kg, while the source hematite does not have strong magnetic properties. Hematite with a particle size from 0.05 to 0.25 mm is completely converted into magnetite, but if the size of hematite particles increases to from 0.25 to 2.5 mm, incomplete conversion of hematite to magnetite is observed. The obtained results are important for the development of technology for converting hematite to magnetite, which can, in turn, be used to beneficiate iron ores.

© 2018 Elsevier B.V. All rights reserved.

1. Introduction

Magnetite is one of the most important minerals in the Earth's crust due to peculiarities of its composition, structure, and magnetic properties. Magnetite (Fe₃O₄) has an inverse spinel structure in which cations occupy tetrahedral and octahedral sites in the face-centered cubic lattice [1,2]. The tetrahedral sites (A) occupied by Fe³⁺ and the octahedral sites (B) occupied by both Fe³⁺ and Fe²⁺ form the basis for two antiparallel magnetic sublattices. The number of spins at sites A and B is unequal, resulting in ferrimagnetism in magnetite below the Curie temperature of 580 °C [2].

From the Iron Age to the present day, magnetite has been the main component of both high-grade and low-grade iron ores. Magnetite quartzite, for example, is suitable for beneficiation by magnetic separation. Magnetite is widely used for pigments, as a catalyst, for magnetic recording media, for magnetic fluids, for gas

sensors, and for other applications [2,3]. Recently, magnetite has become very important in the biomedical field, with applications in drug delivery systems, magnetic resonance imaging enhancement, medical diagnostics, and cancer therapy [4–7].

The use of magnetite in various fields results from its chemical, structural, and physical properties. Because natural magnetite varies in particle size and chemical purity, most naturally occurring magnetite is unsuitable for some uses. Recently, many methods have been developed for the synthesis of magnetite [5,8–11] including co-precipitation [9–12], sol-gel [13,14], thermal decomposition [15], and hydrothermal synthesis [16,17] to control most of its properties. Thus, the connection between the synthesis procedure and the properties of synthesized magnetite is important – not only theoretically, but also for practical purposes.

The methods listed above have certain disadvantages, however. The particles of magnetite prepared by liquid methods, for instance, have relatively low crystallinity. Organic solvents used in the synthesis of magnetite are undesirable thereafter and should be removed from the suspension. In addition, the reagents involved in

* 34 Palladina Prospect, Kyiv, 03680, Ukraine.
E-mail address: vitaliyponomar.vp@gmail.com.

the reactions are quite expensive. Another method for the synthesis of magnetite is based on the phase transformation of iron oxides, carbonates, or sulfides. Note that iron-containing minerals such as hematite and goethite form waste (tailings) when magnetite ores are beneficiated. Iron ore tailings are promising solid wastes for use in industry. The production of magnetite from industrial waste is attractive for commercial and environmental purposes.

Hematite has the chemical formula of Fe_2O_3 and is one of the most common minerals in iron ore deposits. The spin configuration of hematite is antiferromagnetic below the Neel temperature of $680 \pm 5^\circ\text{C}$. Hence, the hematite has a net magnetic moment of close to zero, because the antiparallel spins completely compensate for each other. Nonetheless, hematite can be converted into highly magnetic magnetite. Features of the synthesis of magnetite by hematite transformation were investigated by several methods, including ultrasonic chemical co-precipitation [10] and chemical reduction with carbohydrates [18,19], with hydrogen [20], with carbon monoxide [21], and with other agents.

Chemical reduction of synthetic hematite with starch in air at $265\text{--}580^\circ\text{C}$ was investigated by means of X-ray powder diffraction, FT-IR, and ^{57}Fe Mössbauer spectroscopies [18]. A mixture of hematite and starch was heated at various temperatures ($265\text{--}580^\circ\text{C}$) for various times (30–120 min). Magnetite and a small fraction of hematite were detected in the transformed samples. Magnetite synthesized at 580°C was highly substoichiometric.

The authors [20] investigated the transformation of hematite into magnetite by heating the sample in a stream of an $\text{H}_2\text{-Ar}$ mixture at a temperature range from 260 to 360°C . The starting material was a pure hematite powder (analytical reagent, Mallinckrodt) consisting of nonporous crystalline grains approximately $0.7\ \mu\text{m}$ in size. Reduction of the starting samples at 320°C for 40 min and 50 min led to complete conversion of hematite to magnetite. Activation energy of $98.74\ \text{kJ/mol}$ was calculated for that reaction.

The formation of magnetite was investigated in Ref. [22] by reduction of hematite by iron in an aqueous solution at $350\text{--}570^\circ\text{C}$ and a pressure of 1–2 kbar. The starting material was synthetic hematite (Koch Light Ltd.). The reaction involved the oxidation of metallic iron with water and the reduction of hematite with hydrogen.

The kinetics of hematite ore reduction with hydrogen at low temperature were studied for various sizes of particles in Ref. [23] by thermogravimetric analysis. Australian iron ore, consisting of hematite and magnetite, was used as the experimental material. The experiments used particle sizes of $2.0\ \mu\text{m}$, $6.5\ \mu\text{m}$, $25.0\ \mu\text{m}$, and $107.5\ \mu\text{m}$. When the particle size of the powder decreased from $107.5\ \mu\text{m}$ to $2.0\ \mu\text{m}$, the reduction process of hematite accelerated by about eight times, resulting in an increase in the activation energy of the reaction from $78.3\ \text{kJ/mol}$ to $36.9\ \text{kJ/mol}$.

Thermomagnetic analyses of natural and synthetic hematite were carried out in air and in vacuum [24]. The starting sample consisted of commercial hematite powder (99.999% Fe_2O_3) with a grain size of about $10\ \mu\text{m}$. It has been shown that intense reduction of hematite occurs when the experiment is performed in a vacuum, but not while the hematite is heated in air.

Despite the fact that there have been several studies concerning the reduction of hematite to magnetite, research on the issue cannot be considered sufficient, especially with regard to industrial applications such as the processing of tailings. First, the process is often complicated because the techniques described above are economically ineffective through many stages and require expensive reagents. Also, because the tailings have quite different iron oxide particle sizes, there is a need for further in-depth study of the effects of various particle sizes on the process of converting

hematite to magnetite.

In this paper, the crystalline structure and magnetic properties were identified for magnetite obtained by chemical reduction of natural hematite. Study samples were divided into ten groups by particle size. The results obtained for synthetic magnetite were compared with the results for natural magnetite.

2. Materials and methods

2.1. Initial materials

The initial sample of hematite was collected from the Kryvyi Rih Basin. The magnetic fraction of the sample was removed by wet low-intensity magnetic separation. The main mineral phases of the remaining sample were hematite and minor quartz. The initial hematite was divided by sieving and grinding into the following particle sizes: <0.05 , $0.05\text{--}0.1$, $0.1\text{--}0.16$, $0.16\text{--}0.25$, $0.25\text{--}0.4$, $0.4\text{--}0.63$, $0.63\text{--}1.0$, $1.0\text{--}1.6$, $1.6\text{--}2.5$, and $>2.5\ \text{mm}$.

The magnetite was represented by natural material obtained by wet low-intensity magnetic separation of magnetite quartzite from the Kryvyi Rih Basin. Mineral phases of the sample were magnetite and minor quartz. The particles of magnetite were ground to a size less than $0.07\ \text{mm}$.

2.2. Procedure for magnetite synthesis

The synthesis process included two main stages. The first stage was the synthesis of carbon monoxide by activated charcoal gasification. For this, activated charcoal was heated to 750°C . After that, air was fed into the gas generator and passed through the layer of activated charcoal. The reaction between charcoal and oxygen contained in the air led to the formation of carbon monoxide (CO). The slow flow of air through the layer of activated charcoal provides conditions for the formation of carbon monoxide:



In the second stage, gas containing carbon monoxide was delivered to a quartz reactor with the particles of hematite. The sample temperature inside the reactor was 600°C . The reduction of hematite may occur according to the equation:



2.3. Thermomagnetic analysis

The thermomagnetic analysis was carried out with a custom-built facility that measures the magnetic interaction between the magnet and the specimens when the temperature changes. The interaction strength between the sample and the magnet is proportional to the magnetization of the sample. The facility is equipped with a digital balance that has a built-in permanent magnet. The steady applied magnetic field that results is $300\ \text{mT}$. Such force is great enough to reach the saturation magnetization of magnetite. Synthesis and analysis of the investigated samples were performed in a quartz mini-reactor at a temperature of 650°C . The rate of the sample's heating/cooling was 65°C/minute . Thermomagnetic data are represented by derivative thermomagnetic curves (DTMC) and integral thermomagnetic curves (TMC).

2.4. Magnetometry measurements

Measurement of magnetization as a function of the applied field was performed for the starting and transformed samples. The

Download English Version:

<https://daneshyari.com/en/article/7993226>

Download Persian Version:

<https://daneshyari.com/article/7993226>

[Daneshyari.com](https://daneshyari.com)