

Reduction roasting of hematite to magnetite using carbohydrates



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ABSTRACT

Thermomagnetic analysis was conducted on phase transformations performed for synthetic and natural hematite by reduction involving various carbohydrates (starch, glucose, fructose, sucrose, and ascorbic acid). Thermomagnetic measurements were carried out using a laboratory facility that allows the automatic registration of a sample's magnetization as the temperature changes (the rate of sample heating/cooling was 65 °/min). The results were then analyzed. The reduction reaction of synthetic hematite for all carbohydrates starts at a temperature of ~350 °C, while the reduction of natural hematite for all carbohydrates starts at a temperature of ~365 °C. The magnetite formed by the reaction has a Curie temperature of 565 °C. Both magnetite and hematite are present in all transformed samples. Saturation magnetization increases to ~50 Am²/kg for the samples obtained from synthetic hematite and ~25 Am²/kg for the sample obtained from natural hematite. The difference in the value of saturation magnetization is attributed to a lower magnetite content for the sample obtained from natural hematite. Synthetic hematite transforms more effectively, suggesting the synthetic sample is characterized by a larger surface area compared to natural hematite.

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1. Introduction

Hematite is one of the most abundant minerals in the Earth's crust, with the chemical formula Fe₂O₃. It is a common rock-forming mineral that is found in sedimentary, metamorphic, and igneous rocks. Hematite and magnetite are the main mineral components of iron ores (e.g. quartzite). However, lower-grade sources of iron ore generally require beneficiation to increase concentration of the iron and remove the impurities (e.g. quartz). Magnetite is highly magnetic and can therefore be easily separated from the gangue minerals of magnetite quartzite, allowing the production of high-grade concentrate with very low levels of impurities. Deposits of magnetite iron ore are becoming exhausted, however.

After the magnetic separation of these types of iron ore, weakly magnetic hematite – as well as quartz – is thrown out, forming gangue. Such deposits, called tailings, are accumulated in large quantities occupying vast areas of agricultural land, and polluting the environment in iron mining regions. Tailings can be considered economically profitable man-made deposits of iron ore, as they require neither grinding nor mine extraction.

Therefore, a number of investigations concerning magnetizing roasting of oxidized iron ore (containing hematite) leading to the formation of magnetic iron oxides (mainly magnetite that is suitable for

magnetic separation) have been conducted in order to develop new technologies for hematite ore beneficiation.

Magnetizing roasting technology, described by Hans et al. (1967), relates to the reduction of Fe₂O₃ to Fe₃O₄ and involves mixing ore with heavy fuel oil in an amount at least theoretically necessary for realizing the above-mentioned reaction. The heating of such a mixture occurs in a closed reactor at 800 °C as is necessary for forming gaseous pyrolysis products.

Ellid et al. (2003) used X-ray powder diffraction and Mössbauer spectroscopy to investigate the high temperature chemical reduction of hematite with starch in the air. In this work, the sample was prepared by thoroughly mixing hematite and starch in agate mortar and then pressing the mixture into pellets at 10⁹ Pa. The hematite to starch ratio was 1:5. The roasting was conducted in temperatures ranging from 300 to 580 °C during time intervals ranging from 30 min to 3 h. Obtained samples consisted of magnetite and hematite, as identified by X-ray diffraction (XRD). However, the magnetite formed at this temperature was substoichiometric, as shown by XRD and Mössbauer spectroscopy.

Gaviria et al. (2007) studied hematite to magnetite reduction by monitoring it with the help of Mössbauer spectroscopy and XRD. This process includes isothermal heating of hematite in a flow of H₂-Ar mixture at temperatures between 260 and 360 °C. The hematite fully transformed to magnetite after 40 min at 320 °C.

A method for obtaining magnetite by means of red mud was investigated by Krause and Rohm (2009). The method consists of hematite or goethite reduction using vegetable oils and/or fats and carbon. This

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method includes mixing red mud with reducing agents and further roasting this mixture with a controlled air supply.

Moreover, the reduction of hematite to wüstite, magnetite, and iron has been widely investigated for chemical looping combustion (Monazam et al., 2013; Monazam et al., 2014; Huang et al., 2013) and for the direct reduction process in the iron industry (Mondal et al., 2004 and Yang et al., 2007). The kinetics aspect of hematite–magnetite transformation is partly considered by Abdouni et al. (1988) and Chakraborty (1999).

Although hematite to magnetite reduction is widely investigated, there is no environmentally friendly and energy efficient method to apply it for commercial purposes in the iron and steel industry.

In this work, the reduction of synthetic and natural hematite (from the Kryvyi Rih region) to magnetite is performed with various carbohydrates, i.e. starch, glucose, fructose, sucrose, and ascorbic acid, and thermomagnetic analysis conducted during transformation. Curie temperatures, magnetization, and phase composition are determined.

2. Materials and methods

The sample of synthetic hematite was synthesized by dehydration of synthetic lepidocrocite in the air at 400 °C. Samples of natural hematite from Kryvyi Rih region were ground up to $0.07 \div 0$ mm.

The initial and obtained samples were investigated by the methods of XRD and magnetometry. XRD measurements were performed with a diffractometer DRON-3 M in filtered emission $\text{CuK}\alpha$ ($\lambda = 0.154184$ nm) and $\text{CoK}\alpha$ ($\lambda = 0.179026$ nm) with recording geometry by Bragg–Brentano. The XRD phase diagnostics were performed using diffraction data cards by detected d-spacing. Measurements of mass magnetization were performed using a magnetometer with Hall sensors.

Changes in magnetization with temperature were determined with a custom built facility (Ponomarenko et al., 2016) that enables measurements of the force affecting the sample in a non-uniform magnetic field. This force is proportional to magnetization and the gradient of the magnetic field.

Initial samples were mixed with 4% (w/w) carbohydrates (starch, glucose, fructose, sucrose, and ascorbic acid (“Chimlaborreactive”, Ukraine)). This mixture (0.2–0.5 g) was then used for the subsequent thermomagnetic analysis.

Reduction of natural and synthetic hematite with different carbohydrates was performed in a quartz mini-reactor with a volume of 4 cm^3 . The samples were isolated from atmospheric oxygen and heated to 650 °C. The rate of sample heating/cooling was $65 \text{ }^\circ/\text{min}$.

3. Results and discussion

The main phase of the synthetic sample measured by XRD-data was hematite (Fig. 1a), and the initial natural sample contain hematite and quartz traces with the characteristic peak (d-spacing) of 3.344 Å (Fig. 1b). The characteristic peaks (d-spacing) of synthetic and natural hematite, and their comparison with the standard d-spacing of hematite from the Power Diffraction File (PDF 89-8104), are given in Table 1.

Initial samples do not demonstrate strong magnetic properties, with a magnetization of $\sim 0.17 \text{ Am}^2/\text{kg}$ for synthetic hematite and $\sim 0.34 \text{ Am}^2/\text{kg}$ for natural hematite in the field of 300 mT.

Thermomagnetic analysis makes it possible to monitor the change of magnetization (σ) as a function of the temperature for samples being heated and cooled in the real time mode. Experimentally-obtained thermomagnetic curves with starch are shown for synthetic hematite (Fig. 2a) and for natural hematite (Fig. 2b).

Thermomagnetic analysis shows that the increase in magnetization starts at the temperature of 350 °C for synthetic hematite and 365 °C for natural hematite. Evidently, this is caused by partial reduction of Fe (III) to Fe (II) and subsequent formation of magnetic phase. The decrease of magnetization on the heating curve after its peak at a

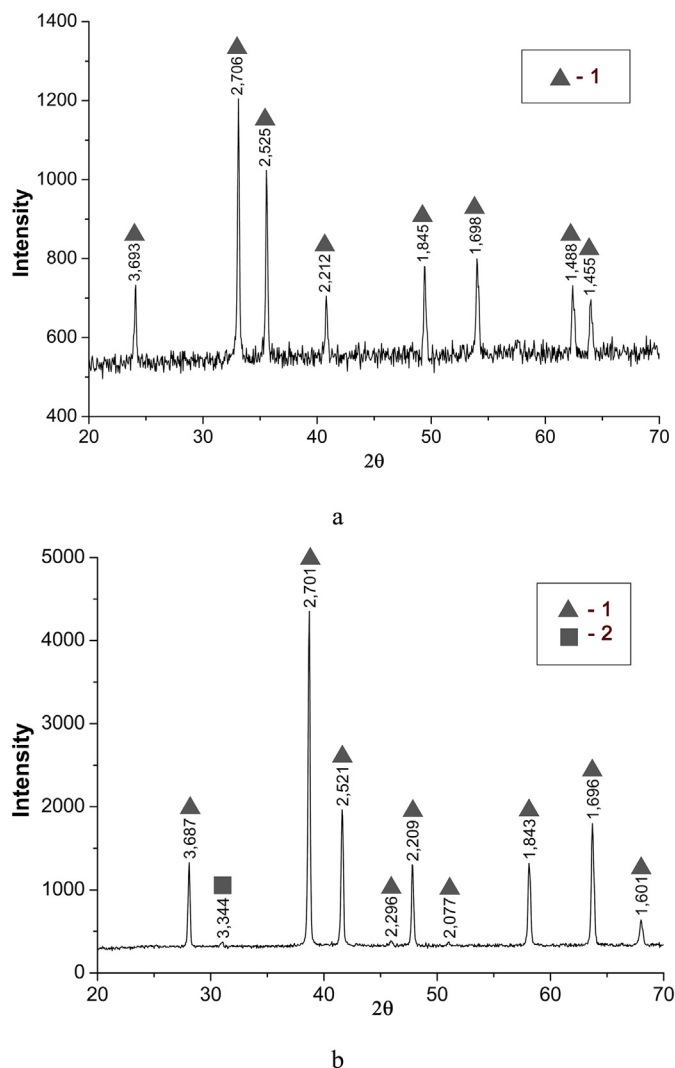


Fig. 1. XRD patterns of the initial samples of synthetic (a) and natural (b) hematite. The numbers correspond to the phases: 1 – hematite; 2 – quartz.

temperature of $\sim 530 \text{ }^\circ\text{C}$ is due to the phase transition of the newly formed ferromagnetic phase into the paramagnetic state. The final, considerable increase in magnetization on the cooling curve is due to the presence of a newly formed magnetic phase in the sample. The magnetic peak on the heating curve is better marked for synthetic hematite than for natural hematite. Therefore, the performed reaction appears to be more effective for synthetic hematite than for natural hematite.

Table 1

Characteristic peaks (d-spacing) (Å) for investigated samples of hematite and standard d-spacing for hematite (PDF 89-8104).

Hkl	Synthetic sample	Natural sample	Standard d-spacing for hematite (PDF 89-8104)
012	3.693	3.687	3.6727
104	2.706	2.701	2.6919
110	2.525	2.521	2.5115
006	n/d	2.296	2.2846
113	2.212	2.209	2.2009
202	n/d	2.077	2.0731
024	1.845	1.843	1.8363
116	1.698	1.696	1.6900
122	n/d	1.601	1.5988
214	1.488	1.487	1.4823
300	1.455	n/d	1.4500

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