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Preparation of Magnetic Fine Particles for Different Applications by Reducing of Non-Magnetic Hematite and Goethite with Biomass

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Phase transformations of natural and synthetic hematite and goethite by reducing with starch at temperature range up to 650°C were investigated. The saturation magnetization of all initial samples was ~1 A*m²/kg, while the saturation magnetization of the samples after phase transformations increases greatly (up to 70 A*m²/kg for synthetic goethite sample). It was shown by X-Ray diffraction method that all phases transformed into magnetite. Rather high saturation magnetization of obtained magnetic particles makes them promising for different medical-biological applications (cells separation, DNA purification, targeted drug delivery, adsorbents of radioactive waste, etc.).

Keywords: hematite, goethite, magnetite, phase transformation, biomass.

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1. INTRODUCTION

During last two decades the intensive research of iron oxides and hydroxides phase transformations are carrying out. Such investigations are very interest because of the possibility to transform the weakly magnetic iron oxides and hydroxides (hematite, goethite, lepidocrocite, etc.) into highly magnetic magnetite with it further usage for creation of new energy effective methods of iron ore beneficiation, selective adsorbents for adsorption of various compounds of inorganic and organic nature that pollute the environment (including radioactive contamination of soil and water), adsorbents for the isolation of RNA/DNA, magnetic carriers for targeted drug delivery and others.

The promising material that can be used to obtain magnetic phases is waste of mining and processing plants that contain large amounts of oxidized iron oxides and hydroxides, that is, in fact, technogenic deposits of iron minerals. These deposits occupy a large area of productive lands and contaminate the environment. So, the utilization of such deposits is very important and could be solved by phase transformations of weakly magnetic iron oxides and hydroxides.

It is known some methods of iron oxides and hydroxides phase transformations. Among them, method of transformation of weakly magnetic iron oxides and hydroxides into magnetite by treatment at high temperatures in the presence of reducing agents (e.g., biomass carbohydrates) [1]. Another article [2] shows the method of reduction of hematite to magnetite using activated coal ZL-302. It was shown, that temperature of hematite reduction with coal is much higher compared to starch (800 °C and 400 °C, respectively). So, reduction of hematite with starch needs less energy. Saturation magnetization of obtained magnetic product was rather high (~50 A*m²/kg). Hydrogen, carbon monoxide [3] and others also could be used as reducing agents.

The aim of this work was to investigate the phase transformations of synthetic and natural hematite and goethite into magnetic phase by reducing with starch.

2. MATERIALS AND METHODS

2.1 Materials

Samples of synthetic goethite were synthesized by procedures [4]. Samples of natural hematite were obtained by grinding of hematite ores from Kryvyy Rih region up to <70 mkm.

2.2 Methods

The main method of the investigation was thermomagnetic analysis. Thermomagnetic analysis was performed using laboratory build facility, that allows to measure the force that effects on the sample in non-uniform magnetic field and which is proportional to magnetization and the gradient of magnetic field. Phase transformations of synthetic and natural hematite and goethite were performed in quartz mini-reactor, isolated from atmospheric oxygen under heating up to 650°C. The rate of sample heating/cooling was 65-80° per minute.

Magnetic characteristics of initial and obtained samples were determined using magnetometry method (magnetometer with Hall sensor). Phase composition was determined by X-ray diffraction (XRD) (X-Ray diffractometer DRON-UM1).

3. RESULTS AND DISCUSSION

According to XRD data, initial samples do not contain admixtures and composed by pure hematite (Fig. 1a) and goethite (Fig 1b). Characteristic peaks (d-spacing) in XRD pattern (Fig. 1a): 5,018; 4,202; 3,381; 2,705; 2,589; 2,459; 2,261; 2,199; 1,729 were attributed to goethite and peaks in XRD pattern (Fig. 1b): 3,687; 2,701; 2,521; 2,209; 1,843; 1,696; 1,601 were attributed to hematite.

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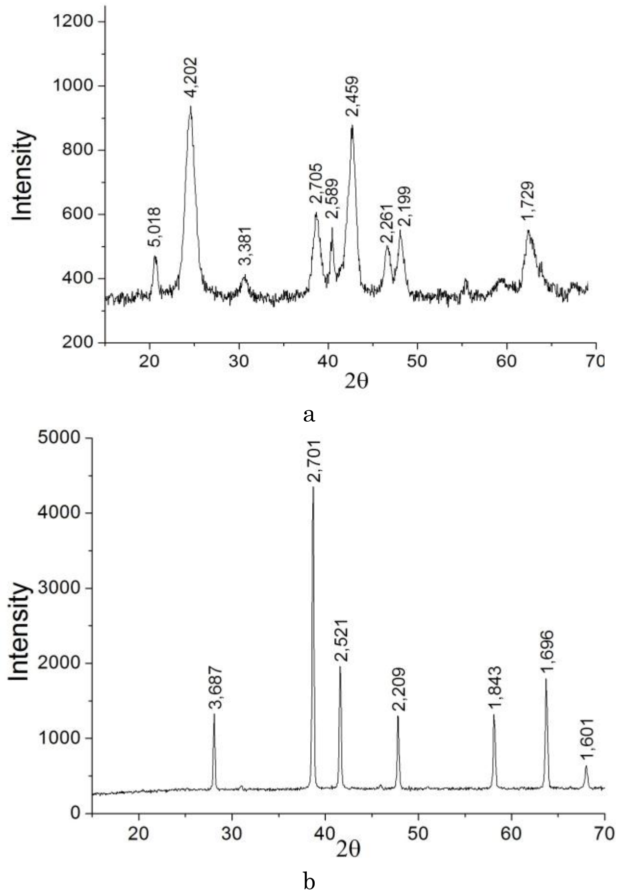

Fig. 1 – XRD pattern of initial samples of synthetic goethite (a) and natural hematite (b).

The broadening of peaks of synthetic goethite (Fig. 1a) indicates that the size of the synthesized particles is in the range of nanometers. Narrow peaks at the XRD pattern of natural hematite indicate high crystallinity of hematite.

The thermomagnetic curves for synthetic goethite and hematite with starch are shown in the Fig. 2. Fig 2a presents cycle of heating/cooling of goethite and Fig. 2b presents cycle of heating/cooling of hematite.

Thermomagnetic analysis of synthetic goethite shows that the reaction of iron reducing with starch starts at the temperature of 260 °C. Magnetization of the sample after cooling increases considerably.

The disappearance of magnetization of the sample above Curie temperature and the cycle of heating and cooling provide us additional information about the present phases. Curie temperature, determined by cooling curve is ~554 °C (marked by the arrow at the Fig. 2a), that is close to Curie temperature of magnetite (580 °C).

Phase transformation of hematite starts at the temperature of approximately 340 °C. The Curie temperature, determined by cooling curve of obtained sample is ~548 °C (marked by the arrow at the Fig. 2b). Therefore one could conclude that the phase of magnetite is formed in the reaction of hematite and goethite reduction by starch.

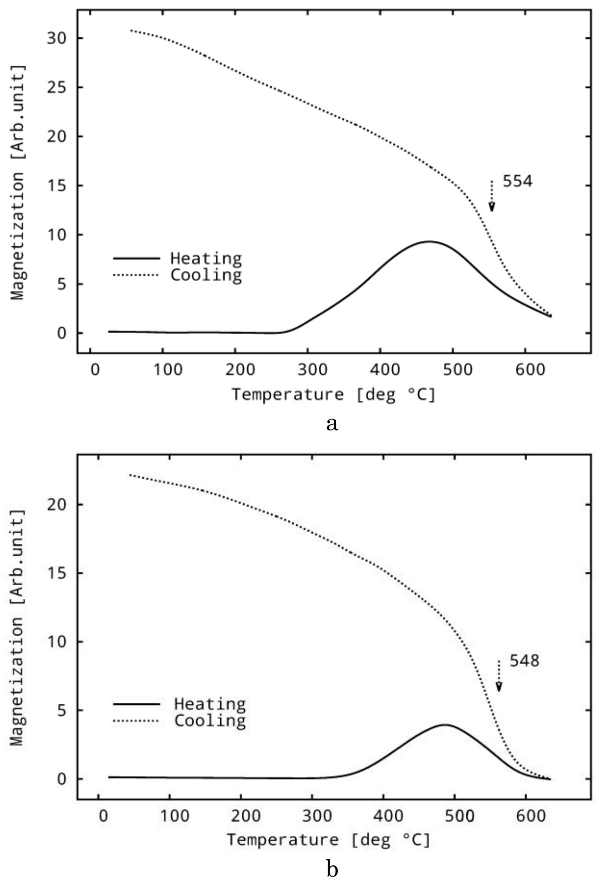

Fig. 2 – Thermomagnetic curves for synthetic goethite (*a*) and hematite (*b*) with starch.

The result of XRD-measurements (Fig. 3, 4) shows that after phase transformation goethite and hematite phases disappeared and new phase of magnetite appears.

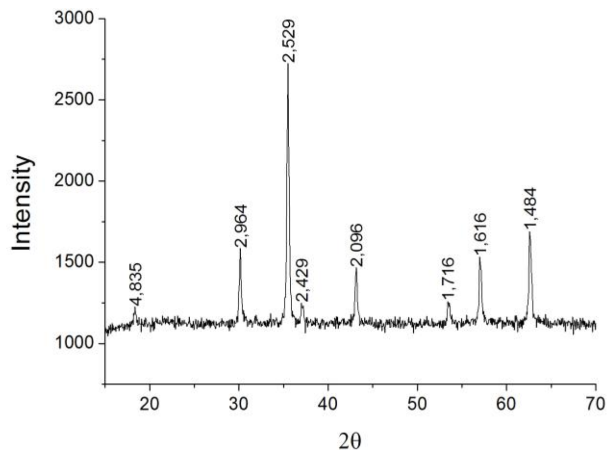

Fig. 3 – XRD pattern of transformed goethite sample.

Characteristic peaks in XRD pattern of magnetic samples, obtained from both goethite (Fig. 3): 4,835; 2,964; 2,529; 2,429; 2,096; 1,716, 1,616; 1,484 and hematite (Fig. 4): 4,822; 2,964; 2,529; 2,423; 2,099; 1,713; 1,616; 1,485 were attributed to magnetite.

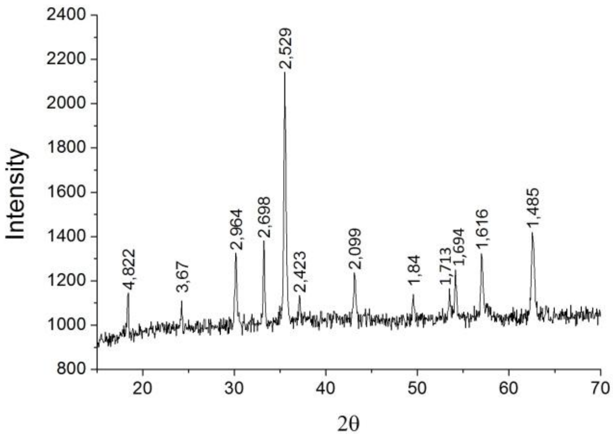

Fig. 4 – XRD pattern of transformed hematite sample.

In the case of goethite it was shown, that complete transformation of goethite structure into magnetite structure occurs. In the case of hematite one could see that after transformation, intensity of hematite peaks decrease and new peaks of magnetite are appearing. Characteristic peaks of hematite in the Fig. 4: 3,67; 2,698; 1,84; 1,694.

Saturation magnetization, determined for magnetite sample, obtained from goethite was ~70 A*m²/kg (magnetization curve is shown at Fig. 5).

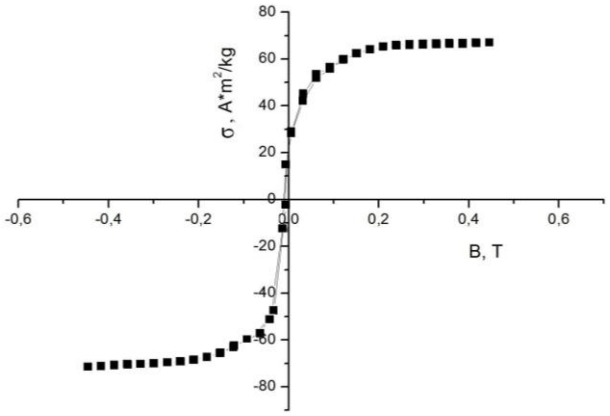

Fig. 5 – Magnetization curve of magnetite obtained from goethite.

Saturation magnetization, determined for magnetite sample, obtained from hematite was ~50 A*m²/kg (magnetization curve is shown at Fig. 6).

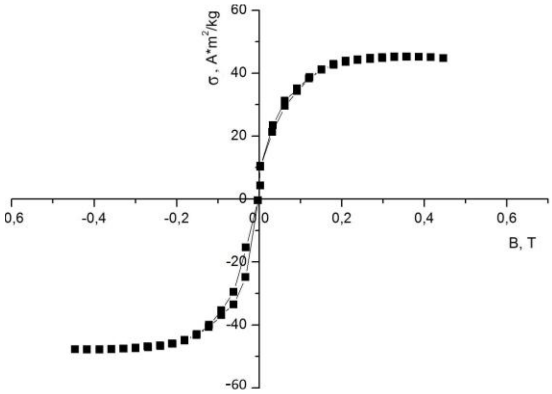

Fig. 6 – Magnetization curve of magnetite obtained from hematite.

The difference between saturation magnetization of pure magnetite (92 A*m²/kg) and magnetic sample, obtained from hematite we could explain by presence of weakly magnetic phase (hematite) in the sample.

4. CONCLUSIONS

It was shown, that synthetic and natural goethite and hematite transformed into magnetite by reducing with starch at the temperature range up to 650 °C. Obtained samples have rather high saturation magnetization: ~70 A*m²/kg for the magnetic sample, obtained from goethite and ~50 A*m²/kg for the magnetic sample, obtained from hematite. The magnetization of obtained samples is rather high and makes them promising for different applications (adsorbents for various compounds of inorganic and organic nature that pollute the environment, adsorbents for the isolation of RNA/DNA, magnetic carriers for targeted drug delivery etc.).

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