RECOVERY AND SEPARATION OF IRON FROM IRON ORE USING INNOVATIVE FLUIDIZED MAGNETIZATION ROASTING AND MAGNETIC SEPARATION

J. Yu, Y. Han*, Y. Li, P. Gao

* School of Resources and Civil Engineering, Northeastern University, Shenyang, China

Abstract

In this investigation, a pilot-scale fluidized magnetization roasting reactor was introduced and used to enhance magnetic properties of iron ore. Consequently, the effects of roasting temperature, reducing gas CO flow rate, and fluidizing gas N_2 flow rate on the magnetization roasting performance were studied. The results indicated that the hematite was almost completely converted into magnetite by a gas mixture of 4 Nm^3/h CO and 1 Nm^3/h N_2 at roasting temperature of 540 °C for about 30 s. Under optimized conditions, a high grade concentrate containing 66.84% iron with iron recovery of 91.16% was achieved. The XRD, VSM, and optical microscopy (OM) analyses revealed that most of the hematite, except some coarse grains, was selectively converted to magnetite, and that the magnetic properties were greatly enhanced. Thus, their separation from non-magnetic gangue minerals was facilitated.

Keywords: Hematite; Fluidized magnetization roasting; Magnetite; Magnetic separation; Magnetic properties

1. Introduction

Research regarding iron ores magnetization roasting began in the early 1900s, and the magnetization roasting was initially applied to the utilization of low-grade iron ores in the USA, Germany and Japan [1]. After several years of theoretical development and engineering practice, magnetization roasting followed by low-intensity magnetic separation was proved to be an effective solution, both technologically and economically when upgrading refractory iron ores [2-7]. According to the type of the roasting reactor, magnetization roasting can be divided into shaft furnace roasting, rotary kiln roasting, and fluidized bed roasting [8].

Shaft furnace magnetization roasting system is mainly composed of heating and reduction processes. The hematite ore is first heated to 700~800 °C in the heating zone, then falls into the reduction zone where the weakly magnetic hematite is reduced to magnetite by coke oven gas at 550~600°C [9-10]. However, several years of engineering practices indicate that the shaft furnace can only process hematite ores with a granularity of 15 ~75 mm, and the shaft furnace roasting is also characterized by heterogeneity of the roasted product. Hence, the shaft furnace magnetization roasting technology is gradually replaced by high-gradient magnetic separation with the development of high-gradient magnetic separator.

The rotary kiln generally handles hematite ores with a grain size of less than 30 mm [8]; however, the formation of clinker ring in the rotary kiln will affect the yield of iron resource [11].

In the fluidized bed furnace, the hematite ore is shaken up by an upward current of gas which maintains the individual particles in suspension [12-13]. The precise adjustment of the gas velocity to the particle size and the specific weight of the hematite ore make it possible to generate a floating mixture of gas and solids which behaves almost like a liquid [14]. The process is reported to have the following advantages over the other roasting techniques: ease of control due to absence of moving parts within the reactor, homogeneity of roasted products, ability to handle fine particles, and high efficiency of heat transfer and mass transfer [15]. However, the heating and reductive magnetizing of hematite ore simultaneously occur at the same location in the conventional fluidized bed reactor. Therefore, the reductive atmosphere, as well as the security and stability of anti-explosion are hard to be controlled. Besides, the roasting temperature for conventional fluidized bed furnace is usually up to 950~1150 °C [16]. Thus, the formation of clinker ring caused by high temperature will affect the operating efficiency.

For this purpose, an innovative fluidized

magnetization roasting reactor, characterized by low roasting temperature (500~600 °C) and high efficiency, where the heating and reduction of hematite ore occurred at different locations was developed and applied successfully for magnetization roasting of iron ores. Due to the high heat/mass transfer efficiency and low energy consumption, fluidized magnetization roasting, by which the weakly magnetic hematite could be transformed into ferromagnetic magnetite quickly (about 30 s), is proven to be the most effective and economical way to deal with the refractory iron ores [17-22].Thus, the new generated magnetite could be captured by a magnet into magnetic concentrate, and non-magnetic gangue materials like quartz could be thrown into tailings after low-intensity magnetic separation. In this investigation, the effects of key variables such as the roasting temperature, and the flow rates of CO and N_2 on fluidized magnetization roasting performance were mainly studied.

2. Experimental
2.1. Magnetization roasting reactor and its working procedure
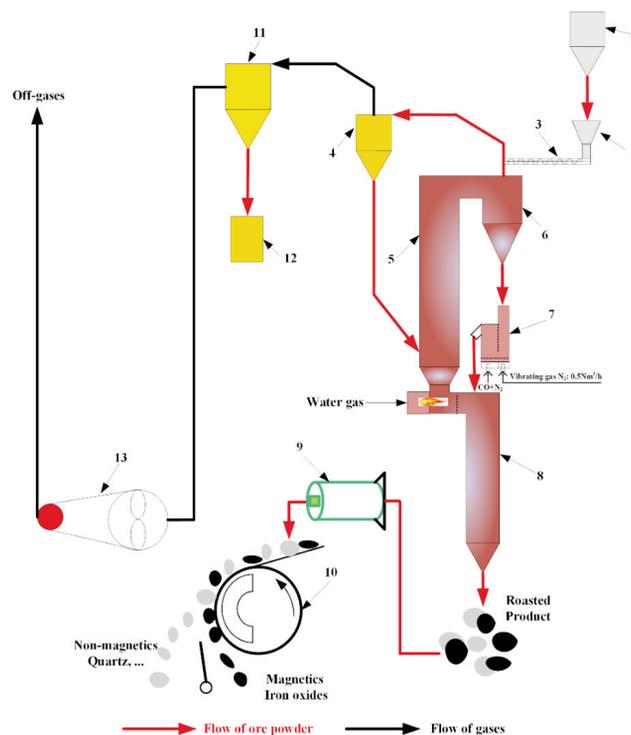
An innovative fluidized magnetization roasting reactor with a throughput rate of 150 kg/h was used in the investigation. As shown in Fig. 1, it mainly consists of a cyclone preheater, a suspension heating furnace, a gas-solid cyclone separator, and a cooling unit. The roasting reactor is equipped with thermocouples and pressure sensors for the precise adjustment of the temperature and pressure [23-24].

The innovative fluidized magnetization roasting reactor is operated under negative pressure conditions. The dry iron ore powder (-0.3 mm) is preheated in countercurrent with the hot off-gas originally from the suspension heating furnace via a number of cyclone stages, and then enters into the suspension heating furnace. The hot gases (about 700~900 °C) produced by the combustion of water gas simultaneously heat and transport the iron ore powder to the gas-solid cyclone separator. Then, under the action of gravity, the heated iron ore powder (450~600 °C) falls into the fluidized magnetization bed. Consequently, the hematite is quickly reduced to magnetite by CO. After reduction roasting, the gas mixture of CO and N_2 transports the iron ore powder to the cooling unit. Meanwhile, the residual CO gas enters into the suspension heating furnace and burns with the water gas [23, 25].

2.2. Description of sample

The iron ore powder used for this investigation was obtained from Donganshan sintering plant in

Figure 1. Sketch of the innovative fluidized magnetization roasting reactor by means of a gas suspension process followed by low-intensity magnetic separation: 1= Feed bin; 2 =Loss-in-weight; 3 = Screw feeder; 4 = Cyclone preheater; 5 = Suspension heating furnace; 6 = Gas-solid cyclone separator; 7 = Fluidized bed; 8 = Cooling unit; 9 = Ball mill; 10 = Drum magnetic separator; 11 = Dust collector; 12 = Dust bin; 13 = Roots blower

Liaoning province of China. Its detailed particle size distribution is presented in Fig. 2 with an average particle size of d(0.5)=82.61 μm, and its chemical compositions are listed in Table 2. Fig. 3 shows the XRD pattern of iron ore powder, indicating that it is mainly composed of hematite, quartz and a small amount of magnetite. Due to its complex mineralogy and fine mineral dissemination, this iron ore has not yet been effectively utilized on an industrial scale. Even if the combined processes of magnetic separation (including low-intensity and high-gradient magnetic separation) and reverse flotation were employed, the iron grade and recovery of the final concentrate were both lower than 65% [26]. Therefore, it is very necessary to develop a new technology to achieve its efficient utilization.

In addition, the combustion fuel for suspension heating furnace is water gas (H_2 50%, CO 40%), the reducing gas is CO with a purity of 99.9%, and the fluidizing gas is N_2 with a purity of 99.9%.
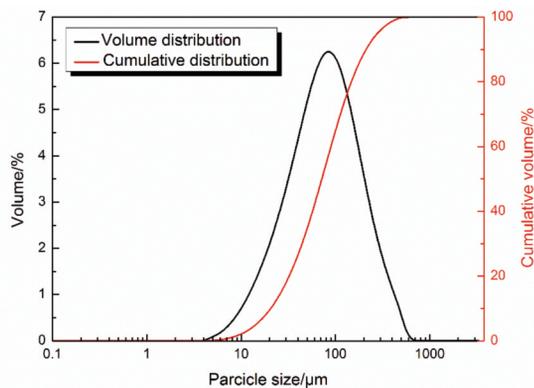

Figure 2. Particle size distribution of iron ore powder

Table 1. Chemical compositions of iron ore powder/wt.%

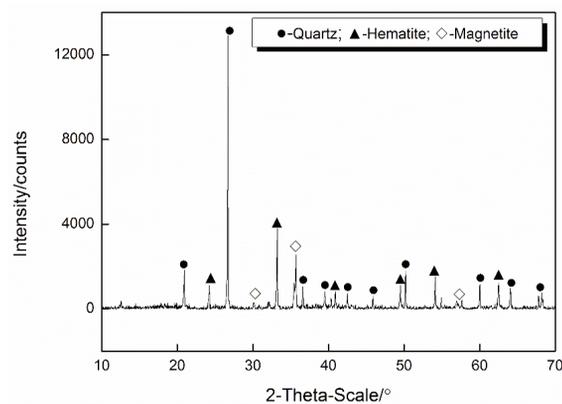| Elements	TFe	SiO_2	Al_2O_3	CaO	MgO	P	S	LOI
Content	41.64	36.62	0.69	0.71	0.57	0.03	0.06	2.63

Figure 3. XRD pattern of iron ore powder

2.3 Methods and evaluation

For all the investigations, the feed velocity for fluidized magnetization roasting reactor was determined to be 120 kg/h, and the roasted iron ore samples at different roasting conditions were ground to 85% passing 0.038 mm. The ground iron ore powder was prepared at a predetermined 30% solid content in a stir tank of 5 L volume. As shown in Fig. 1, a drum magnetic separator (RK/CGS-φ400—300) at a rotational speed of 25 r/min was used for magnetic separation, and the magnetic field intensity was 120 mT. Each time, 5 L slurry was evenly fed to the magnetic separator within 5 min.

Mass weight (γ), iron grade, and recovery (ε) of the magnetic products were used for evaluating the performance of the fluidized magnetization roasting reactor and they were calculated using the following equations:

$$\gamma = \frac{\alpha - \theta}{\beta - \theta} \times 100\% \tag{1}$$

$$\varepsilon = \frac{\gamma \cdot \beta}{\alpha} \times 100\% \tag{2}$$

Where α, β and θ are the iron grades for feed (roasted sample), magnetic concentrate and non-magnetic products (tailing), respectively.

The solid phase structure was analyzed by X-ray diffraction (X' Pert PRO MPD) with Cu Kα radiation, and the operating voltage and current were 40 kV and 40 mA, respectively. The diffraction angle was scanned from 10° to 70°.

A Leica DM4 P optical microscope (Wetzlar, Germany) was used to study the association of hematite with magnetite in the roasted iron ore powder by examining polished thin sections.

A vibrating sample magnetometer (Model 7410, Lake Shore, USA) was used to analyze the magnetic properties of raw iron ore powder and roasted iron ore powder at room temperature (about 25 °C). Once the sample had been positioned correctly, the magnetic field was increased incrementally until 20.0 kOe; this field strength was decreased and then increased again to 20.0 kOe. Thus, a complete hysteresis loop for the sample was given.

3. Results and discussion

The effects of main process parameters, including roasting temperature, flow rates of reducing gas CO and fluidizing gas N_2, on the recovery and separation of iron, were examined in this section.

3.1. Effect of roasting temperature on magnetization roasting performance

The roasting temperature, which was controlled by

adjusting the flow rate of burning water gas in suspension heating furnace, was the most important process parameter. Thus, the influence of roasting temperature on the recovery and separation of iron was investigated in the range of 480~560 °C. The other process conditions, such as the flow rates of reducing gas CO and fluidizing gas N_2, were fixed to be 4.0 Nm^3/h and 2.0 Nm^3/h, respectively. The results are plotted in Fig. 4.
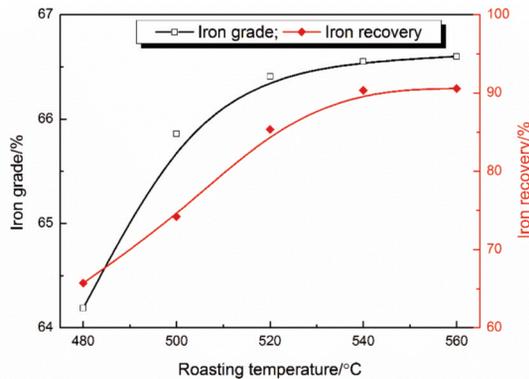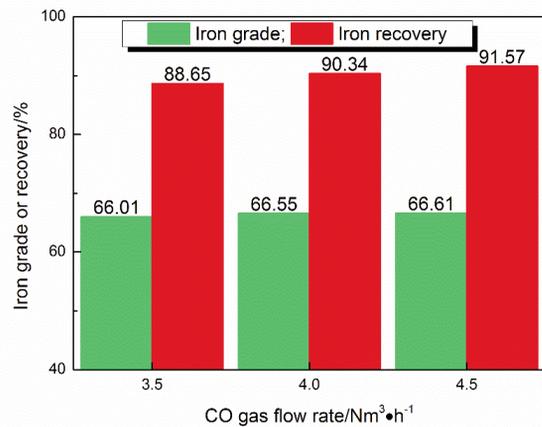

Figure 4. Effect of roasting temperature on magnetization roasting performance: (1) CO flow rate = 4.0 Nm^3/h; (2) N_2 flow rate = 2.0 Nm^3/h

As shown in Fig. 4, both the iron grade and recovery of magnetic concentrate significantly increased as the roasting temperature increased from 480 °C to 540 °C, and then almost remained unchanged when the roasting temperature was further increased to 560 °C. The results indicated that the roasting temperature had a significant effect on the iron grade and recovery of magnetic concentrate. It was inferred that extremely low roasting temperature was not conducive to the conversion of hematite to magnetite due to their limited reaction rate [3, 7], and only a small amount of magnetite was generated, resulting in a low iron recovery of magnetic concentrate. With the increase in roasting temperature, more sufficient reaction rate was available for more hematite being converted to magnetite, and the separation performance was greatly improved. As a result, the roasting temperature of 540 °C is recommended for the optimal condition.

3.2. Effect of CO flow rate on magnetization roasting performance

To optimize the reducing gas CO flow rate required for the process, the experiments were carried out in 3.5~4.5Nm^3/h, and the other process parameters such as roasting temperature and the flow rate of N_2 were fixed to be 540 °C and 2 Nm^3/h, respectively. The results are shown in Fig. 5.

As illustrated in Fig. 5, with the flow rate of reducing gas CO increased from 3.5 Nm^3/h to 4.5

Figure 5. Effect of CO gas-flow rate on magnetization roasting performance: (1) roasting temperature = 540 °C; (2) N_2 flow rate = 2.0 Nm^3/h

Nm^3/h, the iron grade of magnetic concentrate was slightly increased from 66.01 wt.% to 66.60 wt.%, and the iron recovery increased from 88.65 wt.% to 91.57 wt.%. It is easy to understand that an increase in CO flow rate meant a higher CO concentration in the gas mixture of CO and N_2, thus the diffusion of reducing gas CO to hematite particle surface was enhanced, and the conversion of hematite to magnetite was accelerated, resulting in a higher iron recovery and grade of magnetic concentrate. When the flow rate of reducing gas CO increased to 4.0 Nm^3/h, both of the iron grade and recovery of magnetic concentrate were relatively high. Thus, the flow rate of reducing gad CO is selected to be 4.0 Nm^3/h.

3.3. Effect of N_2 flow rate on magnetization roasting performance

In the fluidized magnetization roasting reactor, the fluidizing gas N_2 was mainly used to improve the fluidity of solid materials and to transport the iron ore powder out of the fluidized bed. But for a given roasting temperature, its flow rate and the CO gas flow rate together determined the retention time of iron ore powder in the fluidized bed, thus producing an influence on the separation and recovery of iron from raw ore sample. The results are illustrated in Fig.6.

The results as shown in Fig. 6 revealed that, with the increase of N_2 flow rate from 1.0 Nm^3/h to 5.0 Nm^3/h, both the iron grade and recovery of magnetic concentrate were greatly decreased from 66.84 wt.% to 63.21 wt.% and 91.16 wt.% to 80.64 wt.%, respectively. It is clear that an increase in N_2 flow rate corresponded to a decrease in CO concentration for the gas mixture of CO and N_2, thereby deteriorating the diffusion of CO to the hematite particle surface. On the other hand, the moving speed of solid particles
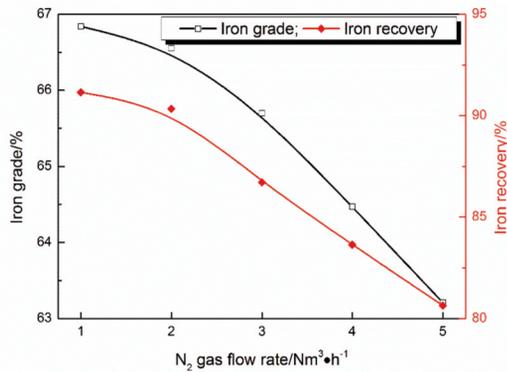
Figure 6. Effect of N₂ gas-flow rate on magnetization roasting performance: (1) roasting temperature = 540 °C; (2) CO flow rate = 4.0 Nm³/h

from the fluidized bed increased with the increase in N_2 flow rate, which led to a significant decrease in reduction roasting time for iron ore powder, resulting in a lower iron recovery and grade of magnetic concentrate. It should be noted that the N_2 flow rate was a key process parameter; if the N_2 was not introduced into the fluidized bed (the flow rate of N_2 was zero), the iron ore powder could not escape from the fluidized bed due to its insufficient moving speed, leading to fluidized bed blockage. Therefore, the N_2 flow rate for this iron ore powder is determined to be 1.0 Nm³/h. In addition, reduction roasting time for this iron ore powder was estimated to be 30 s by using the Wen & Yu and Planoovsky common formulas.

Table 2 shows the magnetic separation results of ground iron ore powder after fluidized magnetization roasting at above optimum conditions (roasting temperature: 540 °C, CO flow rate: 4.0 Nm³/h, N_2 flow rate: 1.0 Nm³/h). As listed in Table 2, a high grade magnetic concentrate containing 66.84% iron with an iron recovery rate of 91.16% was obtained, indicating that the innovative fluidized magnetization roasting was an effective method for the beneficiation of this iron ore powder.

Table 3 shows the chemical analysis for the compositions of magnetic concentrate. It was implied in Table 3 that the magnetic concentrate could be used as qualified raw material for direct reduced iron (DRI) production, and the amounts of impurities like S and P were very low by 0.05 wt.% and 0.02 wt.%, respectively.

Table 3. Chemical compositions of magnetic concentrate/wt.%

Elements	TFe	SiO_2	Al_2O_3	CaO	MgO	P	S	K_2O	Na_2O
Content	66.84	6.41	0.31	0.12	0.32	0.02	0.05	0.06	.0.05

4. Phase transformation and magnetic properties after roasting

Fig. 7 presents the XRD traces for roasted iron ore powder at above optimum conditions. Compared with the results in Fig. 3, the characteristic diffraction peak intensity of hematite was significantly weakened, and that of magnetite was enhanced. It was inferred that most of the hematite (Fe_2O_3) in the raw ore was selectively reduced to magnetite (Fe_3O_4) after roasting with the gas mixture of CO and N_2. As shown in Fig. 7, the hematite peaks did not disappear in the roasted iron ore powder, indicating that hematite was not completely converted to magnetite. This result might be due to the coarse grain size of some hematite; the hematite grain surface was reduced to magnetite, but the inner portion was still hematite crystal lattice.
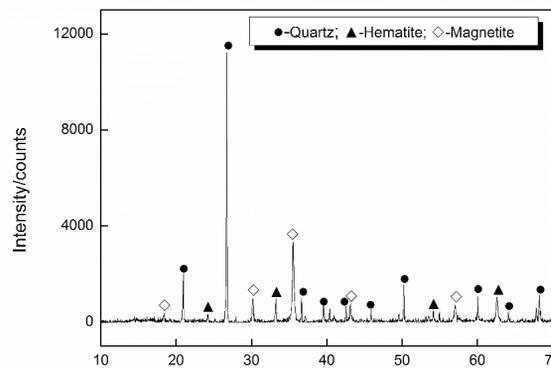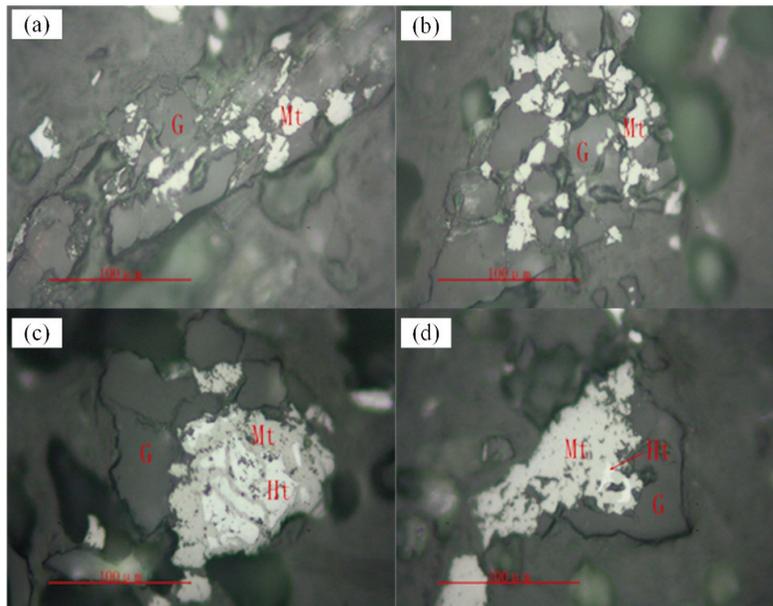

Figure 7. XRD pattern of roasted iron ore powder: (1) roasting temperature = 540 °C; (2) CO flow rate = 4.0 Nm³/h; (3) N₂ flow rate = 1.0 Nm³/h

In order to further verify the above analysis, a Leica DM4 P optical microscope was used to study the association of hematite with magnetite in the roasted iron ore powder by examining polished thin sections. The results are shown in Fig. 8. As illustrated in Fig. 8 (a) and (b). It was clear to see that most of the hematite grains, except for some coarse ones (>100

Table 2. Magnetic separation results of ground iron ore powder after fluidized magnetization roasting

Products	Mass weight (wt.%)	Iron/SiO_2 grade(wt.%)	Iron/SiO_2 recovery(%)
Magnetic concentrate	63.07	66.84/6.41	91.16/10.90
Non-magnetic product (tailing)	36.93	11.07/89.42	8.84/89.10
Feed (roasted iron ore powder)	100.00	46.25/37.10	100.00

Figure 8. Optical microstructure images of roasted iron ore powder: G – gangue minerals; Mt – magnetite; Ht - hematite

μm), were reduced to magnetite, while the inner portion of coarse hematite grains after roasting was still hematite crystal lattice due to the insufficient reaction rate or roasting time, as shown in Fig.8 (c) and (d). It should be noted that these coarse hematite-magnetite interlocking particles could also be effectively recovered by subsequent magnetic separation after liberating from gangue minerals. Meanwhile, the magnetite and hematite could not be distinguished under a scanning electron microscope (SEM), thus the SEM analysis for roasted iron ore powder was not carried out.

Fig. 9 shows the hysteresis loops for iron ore powder before and after fluidized magnetization roasting. It was obvious that the saturation magnetism (Ms) of iron ore powder was increased from 8.96 to 38.08 emu/g after fluidized magnetization roasting. Correspondingly, the residual magnetism (Mr) and

coercive force (Hc) were increased from 0.53 to 4.92 emu/g, and from 79.20 Oe to 146.10 Oe, respectively, indicating that most of the weakly magnetic hematite was converted to ferromagnetic magnetite, and that good magnetic separation would be achieved [27-28]. The results are greatly in agreement with the above analyses.

4. Conclusions

The results demonstrated that the innovative fluidized magnetization roasting reactor, where the heating and reduction of hematite occurred at different locations, was characterized as a low temperature and efficient equipment for beneficiation of refractory hematite iron ores. The optimized fluidized roasting conditions for the selected iron ore powder were obtained as following: roasting at 540 °C with a gas mixture of 4 Nm^3/h CO and 1 Nm^3/h N_2. Under optimized conditions, a high grade concentrate containing 66.84% iron with an iron recovery of 91.16% was achieved, which could be used as qualified raw material for direct reduced iron (DRI) production.

The XRD, VSM, and optical microscopy (OM) analyses revealed that fine hematite grains were completely converted to magnetite, and the magnetic properties were greatly increased. Thus，their separation from non-magnetic gangue minerals was facilitated, and a good low-intensity magnetic separation result was achieved, while some coarse hematite grains (>100 μm) were only partly reduced to magnetite, forming hematite-magnetite interlocking particles, which could also be effectively recovered by subsequent magnetic separation after liberating from gangue minerals.
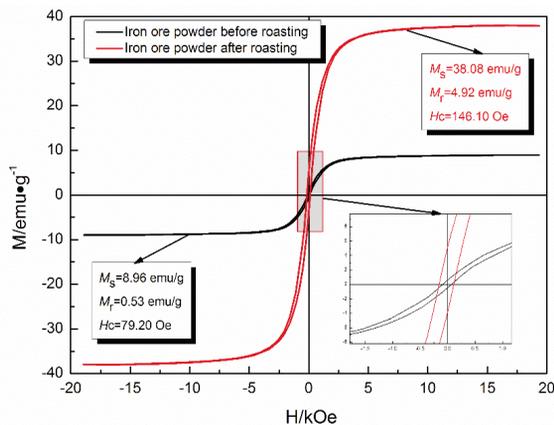
Figure 9. Hysteresis loops of iron ore powder before and after fluidized magnetization roasting

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DOBIJANJE I SEPARACIJA GVOŽĐA IZ RUDE GVOŽĐA KORIŠĆENJEM INOVATIVNOG FLUIDIZACIJSKOG MAGNETIZACIONOG PRŽENJA I MAGNETNE SEPARACIJE

J. Yu, Y. Han*, Y. Li, P. Gao

Fakultet za resurse i građevinarstvo, Severoistočni univerzitet, Šenjang, Kina

Apstrakt

U ovom istraživanju predstavljen je ogledni reaktor za fluidizacijsko magnetizaciono prženje i iskorišćen za pojačavanje magnetnih svojstava rude gvožđa. Dakle, analizirani su efekti koje temperature prženja, smanjenje protoka CO gasa, kao i smanjenje protoka fluidizacijskog gasa N_2 imaju na magnetizaciono prženje. Rezultati su pokazali da je hematit skoro potpuno konvertovan u magnetit mešavinom gasa od 4 Nm^3/h CO i 1 Nm^3/h N_2 pri temperaturi prženja od 540 °C, oko 30s. Pod optimalnim uslovima, dobijen je koncentrat koji sadrži 66,84% gvožđa sa iskorišćenjem gvožđa od 91,16%. XRD, VSM i OM (optička mikroskopija) analize su otkrile da je većina hematita, osim nekih krupnih zrna, selektivno konvertovana u magnetit, i da su magnetna svojstva znatno poboljšana. Na taj način je omogućeno njihovo odvajanje od ne-magnetnih minerala u jalovini.

Ključne reči: Hematit; Fluidizacijsko magnetizaciono prženje; Magnetit; Magnetna separacija