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Effect of fluidized magnetizing roasting on iron recovery and transformation of weakly magnetic iron mineral phase in iron tailings

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Abstract: The eastern tailings of the Anshan mining area are generally categorized as high silicon-bearing iron tailings, and the iron mainly exists in the form of hematite–limonite with an iron grade of 10.60%. In order to recover iron minerals and reduce the influence of the tailings on the environment, a method for pre-enrichment through the combination of low intensity magnetic separation and high intensity magnetic separation with fluidized magnetizing roasting and subsequent low intensity magnetic separation was developed to treat the eastern tailings of the Anshan mining area. The effects of gas-flow rate, H_2 concentration, roasting temperature, and roasting time on the quality of the final iron concentrate were discussed. Moreover, the iron phase transformation and change in magnetism of the sample were studied. The results indicated that an iron concentrate with an iron grade of 65.30% and a recovery of 85.85% could be obtained under the conditions of gas-flow rate of 8 m^3/h, H_2 concentration of 50%, and fluidized magnetizing roasting at 600 ℃ for 20 s. X-ray diffraction analysis, phase analysis, and magnetism analysis on the roasted materials indicated that the hematite–limonite could be transformed into magnetite during the fluidized magnetizing roasting process, and effective separation of iron and gangues could be easily achieved by the weak magnetic separation.

Keywords: iron tailings, fluidized magnetizing roasting, hematite, limonite, iron phase transformation, low intensity magnetic separation

1. Introduction

With the accelerating pace of resource extraction, the quality of resources is decreasing, and the old tailing dams that have served for several decades now have become the focus of attention. Generally, iron ore tailings are stockpiled in tailing dams. The tailing dams not only require a lot of money to construct and maintain but also occupy large tracts of land. In addition, the noxious substances in the tailings, such as residual flotation agents and heavy metals, have a severe impact on the ecological environment in surrounding areas. As a result, comprehensive utilization of iron tailings has been a topic of renewed concern. Many scholars have researched on iron ore tailings (Dauce et al., 2018; Li et al., 2010; Tong et al., 2008). Iron tailings can be treated as a compound mineral, which is an important component for comprehensive utilization of mineral resources. The iron ore tailings could be used to recover iron, to produce building materials, pigments, soil conditioners, mine-fill for goaf and so on (Dauce et al., 2018; Galvão et al., 2018; Li et al., 2013). It is universally acknowledged that the grade of iron ore in China is low, and the output rate of iron ore tailings is high—usually more than 60%. At present, the comprehensive utilization rate of iron ore tailings is still low in China, causing a large amount of iron in the tailings to be wasted; thus, more attention should be paid to the resource utilization of iron ore tailings. The eastern tailings of the Anshan mining area in China contain high silicon, low iron, low sulfur, and low phosphorus and have great recoverable value. However, the iron is difficult to concentrate with traditional mineral processing technologies because of its low grade, weak magnetism, and fine particle size (Zheng et al., 2015; Luo and Nguyen, 2017; Chen, 2011; Ku et

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al., 2015; He et al., 2017). Therefore, new technologies are required to treat this kind of tailings, such as the reduction roasting technology. During the reduction roasting process, the weakly magnetic minerals such as hematite and limonite could be transformed into strong magnetic minerals such as magnetite or metallic iron with the addition of carbon or CO or H_2 as a reductant (Li et al., 2011; Han et al., 2016; You et al., 2018; Yu et al., 2017; Sun et al., 2013).

In the current technical and economic scenario, magnetizing roasting combined with low intensity magnetic separation technology is a typical and effective method to treat low-grade refractory iron ore (Zhu et al., 2018). Owing to fluidization with heat and mass transfer efficiency, low roasting energy consumption, and other advantages that are constantly being discovered, magnetizing roasting has become a popular topic of research in recent years (Li et al., 2013; Zhu et al., 2018; Du et al., 2018; Faris et al., 2017, Chun et al., 2015; Svoboda and Fujita, 2003).

Based on the new technology of 'pre-enrichment through a combination of low intensity magnetic separation and high intensity magnetic separation with fluidized magnetizing roasting and subsequent low intensity magnetic separation' of complex refractory iron ore (Li and Zhu, 2012; Li et al., 2015; Han et al., 2016), a self-made laboratory-scale intermittent fluidized magnetizing reactor was used to systematically dispose the eastern tailings of the Anshan mining area. In the magnetization roasting process, the main factors affecting the roasted material quality were studied so as to determine the appropriate roasting process parameters. Meanwhile, the effects of fluidized magnetizing roasting on the transformation of weakly magnetic iron mineral phase in the iron tailings were characterized by X-ray diffraction analysis combined with vibrating sample magnetometer (VSM) analysis. In general, magnetizing roasting combined with magnetic separation provides a new way for efficient development and utilization of the Anshan iron tailings resources.

2. Materials and methods

2.1. Materials

The sample used in this study was obtained from the eastern tailings of the Anshan mining area located in Liaoning province, China. The mixed tailings sample was composed of three tailings from the eastern tailing dam of the ANSTEEL mining company in a certain proportion. The tailings sample was mixed evenly and air-dried, with 75.43 wt% of particles passing through a sieve of mesh size 0.074 mm. The chemical compositions and XRD pattern of the sample are shown in Table 1 and Figure 1, respectively. Table 2 shows the iron phases of the sample.

Table 1 Chemical compositions of the sample (wt%)

TFe	FeO	SiO_2	Al_2O_3	CaO	MgO	S	P
10.60	2.71	76.22	1.37	1.48	2.42	0.045	0.060

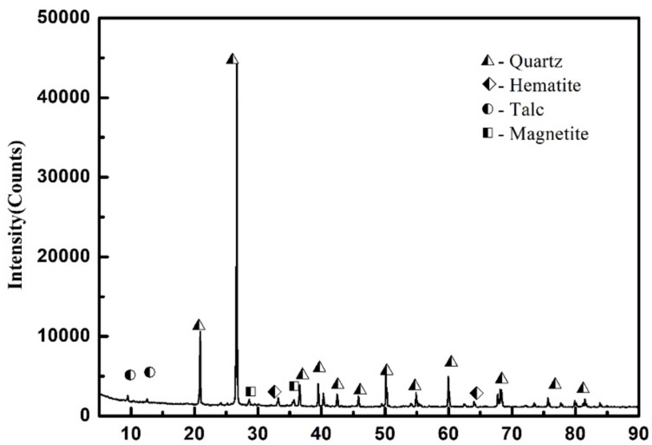

Fig. 1. XRD pattern of the sample

Table 2. Iron phases of the sample

Mineral phase	Magnetite	Iron carbonate	Hematite–Limonite	Iron sulfide	Iron silicate	TFe
Mass/%	2.53	0.11	6.89	0.11	0.87	10.60
Occupancy/%	23.86	1.04	65.85	1.04	8.21	100.0

It can be seen from Table 1 and Table 2 that the total Fe content (TFe) of the sample is 10.60%, and the iron-containing minerals mainly occur in the form of hematite–limonite with a share of 65.85%, followed by magnetite with a share of 23.86%. The main impurity is SiO_2, and its content is approximately 76.22%; the content of harmful elements S and P were low. XRD result in Figure 1 shows that the sample is mainly composed of quartz and hematite. The diffraction peaks of magnetite and talc were also observed. It is seen that the diffraction peaks of magnetite and talc are weak, which indicates that the content of magnetite and talc are low (less than 5%).

2.2. Pre-enrichment with fluidized magnetizing roasting followed by low intensity magnetic separation experiment

According to our previous experience (Li et al., 2015), we could get better results if the TFe grade of the fluidized roasting feed reaches 25%. Therefore, the recovery rate can be raised as high as possible when pre-concentration is used and the pre-concentration product meets the grade. The pre-concentration process is a low intensity magnetic separation followed by vertical ring strong magnetic separation, bulk concentrate grinding, secondary low intensity magnetic separation, and vertical ring strong magnetic scanning process. The pre-concentration experiment flowsheet is presented in Figure 2. Finally, the strong magnetic concentrate (i.e. 2# concentrate) with an iron grade of 24.63% and iron recovery of 43.29% was used as the fluidized magnetizing roasting feeding material.

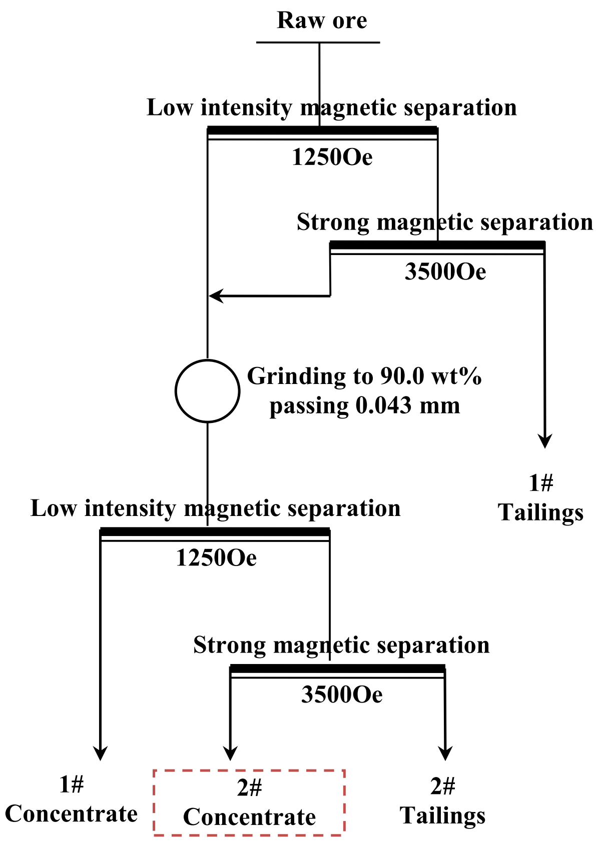

Fig. 2. Flowsheet for preparing samples for fluidized magnetizing roasting experiment

The chemical compositions and XRD pattern of the 2# concentrate are shown in Table 3 and Figure 3 respectively. It can be seen from Table 3 that the total Fe content of the sample is 24.63%, and the main impurity is still SiO_2, but the SiO_2 content is reduced to 62.72% by magnetic pre-concentration. According to Figure 3, the main mineral phases of the pre-enrichment concentrate are quartz and hematite, with a small quantity of magnetite. Compared with Figure 1, it is seen that the diffraction peaks of quartz are obviously weakened and those of hematite are significantly enhanced, which indicates that pre-concentration is beneficial to remove some of the quartz and increase the content of hematite. However, the quartz content in the pre-concentration material is still high, which may be due to the embedment characteristics and symbiotic relationship between quartz and iron ore, leading to a limited reduction in the quartz content.

Table 3 Chemical element analysis of 2# concentrate (wt %)

TFe	FeO	SiO_2	Al_2O_3	CaO	MgO	S	P
24.63	3.02	62.72	1.18	1.04	2.03	0.045	0.031

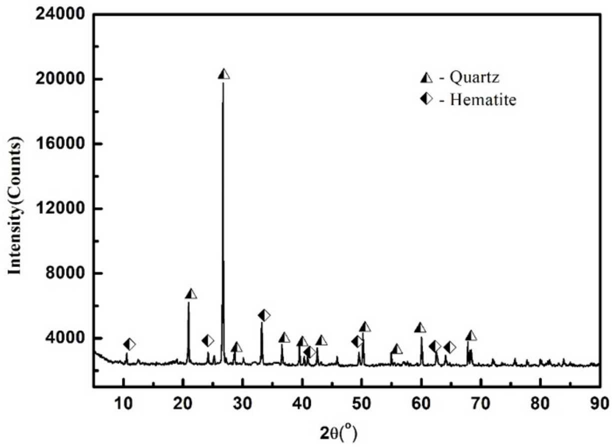

Fig. 3. XRD spectrum of 2# concentrate

The periodic fluidized magnetizing roaster used in the experiment was built with an aim to efficiently utilize weakly magnetic iron minerals. The fluidized magnetizing roasting device consists of a feeding system, electric heating and temperature control system, gas–solid separation system, material collection system, and other components; the structural schematic of the developed device is presented in Figure 4.

The test procedure for fluidized magnetization roasting and low intensity magnetic separation is as follows:

Firstly, the N_2 was introduced from the bottom of the furnace into the roasting chamber to remove air and form a neutral atmosphere when the temperature increased to a predetermined value. Secondly, H_2 was introduced and the mixed gases of N_2 and H_2 (reducing gas) were controlled and regulated by the gas valves. Thirdly, the prepared fine raw material (100 g feeding) was fed to the roasting furnace for magnetizing roasting. Finally, the weak magnetic iron mineral was reduced to a ferromagnetic iron mineral. After a determined time, the heating system was shut off, and the H_2 valve was closed. However, N_2 must be introduced to empty the residual H_2 in the furnace. The roasting product was cooled to 350 ℃ in a nitrogen atmosphere and then exposed to air to continue cooling to room temperature. The main chemical reaction can be expressed by the following equation:

$$3Fe_2O_3 + H_2 (g) = 2Fe_3O_4 + H_2O. \tag{1}$$

Then, the roasting product was finely ground and sieved to -115 mesh (125 μm) accounting for 100% by a laboratory cone ball mill (XMGS-Φ150×50), and divided into 10 smaller samples to be separated by the Davies magnetic tube (XCGS-50), the magnetic field strength was 80 kA/m. The magnetic concentrate and tailing were dried, weighed, and tested for Fe content to calculate the iron recovery.

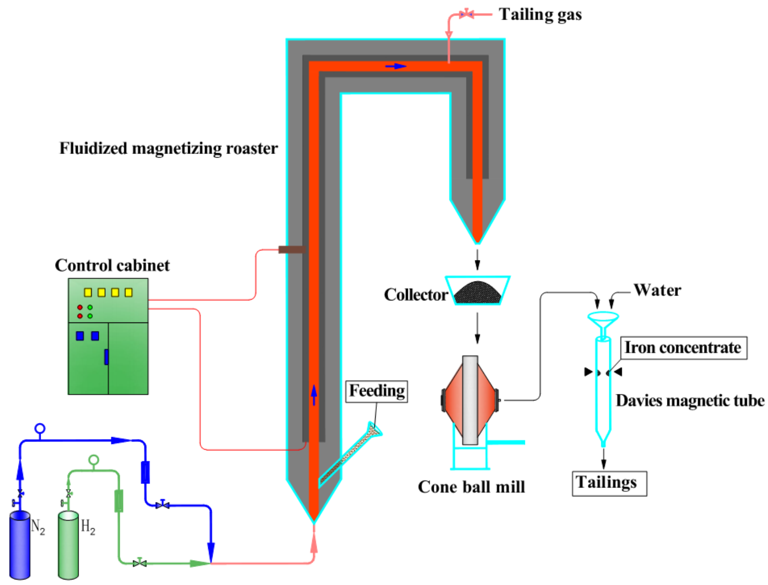

Fig. 4. Schematic diagram of the intermittent fluidized roaster

2.3. Measurement techniques

The raw and obtained roasted samples were both analysed by XRD and magnetometry. The XRD patterns were collected with Cu Kα radiation (λ = 0.154184 nm) using a PW3040 X-ray diffractometer produced by PANalytical B.V. in Holland, and the diffraction angle was scanned from 5° to 90° after setting the operating voltage at 40 kV and current at 40 mA. Then, the XRD patterns were analysed using the software package HighScore Plus.

Measurements of magnetization and specific magnetic susceptibility were performed using a VSM. The sample should be dry and put in a proper place; on the basis of the induced voltage of the sample being directly proportional to magnetic moment, amplitude of vibration, and vibrational frequency, the magnetic moment of the sample could be calculated if the induced voltage was measured with a lock-in amplifier; the specific magnetic susceptibility of the sample could then be obtained by processing the test curve.

3. Results and discussion

In the fluidized magnetizing roasting process, the main factors affecting the roasted material's quality were gas-flow rate, reducing gas H_2 concentration, roasting temperature, as well as roasting time. In order to determine the appropriate fluidized magnetizing roasting process parameters, the effects of gas-flow rate, reducing gas H_2 concentration, roasting temperature, and roasting time on Fe grade and recovery of magnetic concentrate were studied. The results are summarized in Figures 3–5.

3.1. Effect of gas-flow rate

Gas-flow rate is an important factor for the fluidized magnetizing roasting process. The magnetization reaction would be carried out at a constant and full speed if the gas-flow rate is appropriate. When the gas-flow rate is low, the magnetization reaction would be inadequate; when the gas-flow rate is too high, the samples would be over-reduced. The effect of gas-flow rate on the roasted material quality was studied under the conditions of H_2 concentration of 30% and roasting time of 2 s at 600 °C, with the gas-flow rate ranging from 6 to 14 m^3/h. The results are shown in Figure 5. As can been seen from the figure, with the increase of gas-flow rate, the iron grade of the concentrate increases rapidly first and then increase gradually, and the iron recovery shows a tendency to decrease in general. When the gas-flow rate increased from 6 m^3/h to 8 m^3/h, the TFe grade of the concentrate increased from

56.26% to 59.28%, while the recovery decreased from 30.91% to 29.09%. As the gas-flow rate continued to rise to 12 m^3/h, the TFe grade of the concentrate was maintained around 59% and showed little change; however, the iron recovery decreased to 25.54% by approximately 4 percentage points. To sum up, the appropriate gas-flow rate was thus determined to be 8 m^3/h. This result might be due to the fact that hematite could not be transformed into magnetite completely when the gas-flow rate was low, and the reducing gas H_2 was insufficient, resulting in low iron recovery. Whilst further increase in gas-flow rate resulted in a rapid decline in iron recovery, this could be due to over reduction, i.e. some original or new generated magnetite could be reduced to ferrous oxide, which is weakly magnetic.

3.2. Effect of H_2 concentration

In this study, the reducing atmosphere of roasting was affected by the H_2 concentration because H_2 was used as a reductant for the magnetizing roasting. Low concentrations of a reducing agent would make the reducing reaction inadequate, leading to a part of hematite to not be transformed into magnetite, and the recovery rate would be low consequently. However, over reduction might occur if the H_2 concentration increases excessively; this excess H_2 would also be wasted. The effect of H_2 concentration on the roasting effect was investigated under the conditions of total gas-flow rate of 8 m^3/h and roasting time of 2 s at 600 °C, with the H_2 concentration varied from 20% to 50%. The results are shown in Figure 6.

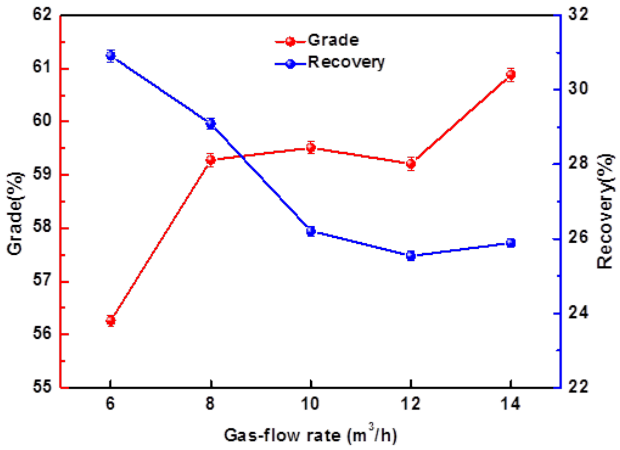

Fig. 5. Effect of gas-flow rate on Fe grade and recovery of magnetic concentrate

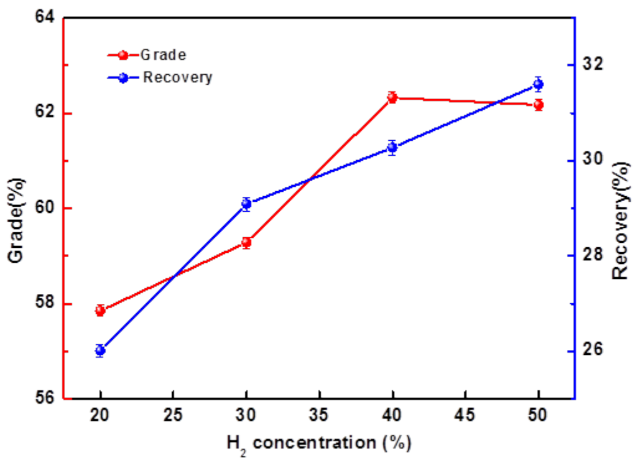

Fig. 6. Effect of H_2 concentration on Fe grade and recovery of magnetic concentrate

It is implied from Figure 6 that the iron recovery increased gradually from 26.01% to 31.60% as the H_2 concentration increased from 20% to 50%; meanwhile, the iron grade of the magnetic concentrate also increased from 57.85% to 62.32% first and then decreased negligibly to 62.17%. In order to ensure that the reduction reaction was adequate, the H_2 concentrations for the following experiments were suggested as 50%.

3.3. Effect of roasting temperature

Temperature is one of the main factors affecting the effect of magnetizing roasting. Appropriate temperature can not only make the reduction reaction proceed quickly and effectively but also produce roasted materials of high quality. Higher temperatures can easily lead to over-reduction, forming weakly magnetic wustite (FeO) (Li et al., 2015). In addition, the higher the roasting temperature, the greater is the cost of production. On the contrary, lower roasting temperature will lead to slow reaction rate, incomplete reduction, and low production efficiency (Yang et al., 2013). Therefore, the roasting temperature should be strictly controlled to ensure optimal roasting effects. The effect of temperature on the reduction of hematite–limonite to magnetite was studied for 8 m^3/h gas-flow rate with 50% H_2 at the temperature range of 500 °C to 700 °C in intervals of 50 °C, reacting for 2 s. The test results are shown in Figure 7.

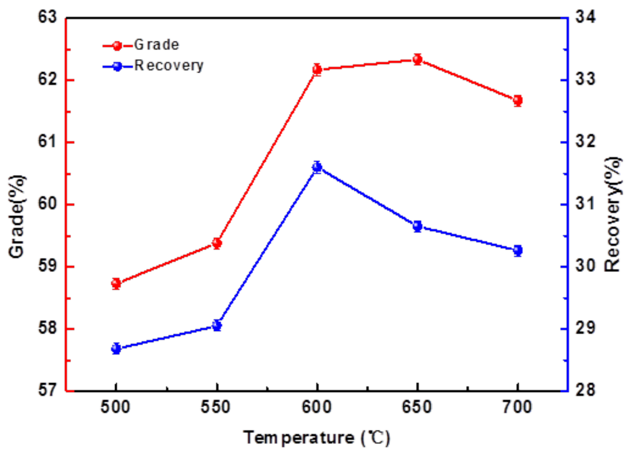

Fig. 7. Experimental results of magnetic separation by fluidized roasting at different roasting temperatures

It can be seen that roasting temperature has a large effect on the TFe grade of the concentrate from Fig. 7. The TFe grade increased from 58.73% to 62.33% when the temperature increased from 500 °C to 650 °C; then, the TFe grade declined slightly to 61.67% when the temperature increased further to 700 °C. However, the iron recovery decreased from the peak point of 31.60% when temperature increased higher than 600 °C. Generally, the rate of reduction increases with the increase of temperature, but if the temperature is too high, it will cause over reduction; that is to say, a portion of the iron oxide could be transformed into weakly magnetic wustite (FeO) or fayalite (Fe_2SiO_4), thereby affecting the subsequent magnetic enrichment effect (Li and Zhu, 2012; Xue, 2008; Zhang, 2007). Therefore, the roasting temperature should be strictly controlled during the fluidized magnetizing roasting process to obtain optimum products. According to the comprehensive consideration of the TFe grade and recovery, the optimum roasting temperature was determined to be 600 °C.

3.4. Effect of roasting time

For the experiment, there must be sufficient and reasonable time corresponding to a certain roasting temperature. The optimum roasting time is fundamentally determined by factors such as roasting temperature, reduction atmosphere, ore properties (size, mineral composition, etc.), as well as heat and mass transfer rates.

Under the selected conditions, further studies were carried out with the roasting time varied from 2 to 20 s. The results are shown in Figure 8.

It can be seen from Figure 8 that with the prolonging of roasting time, the TFe grade and the iron recovery of the concentrate show a trend of growth in general. When the roasting time was extended from 2 s to 20 s, the recovery of iron concentrate decreased a bit at 4 s and then increased quickly from

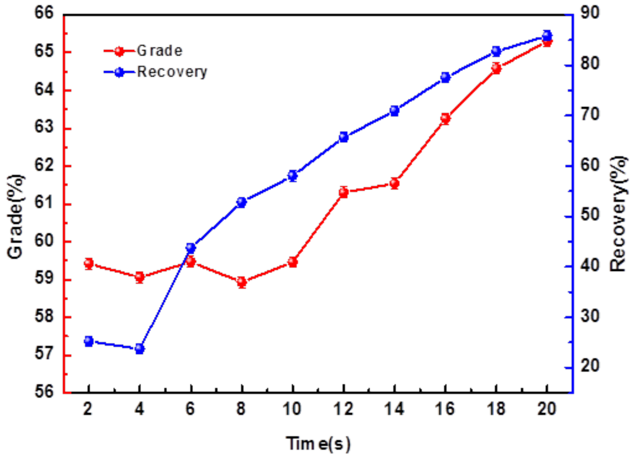

Fig. 8. Experimental results of magnetic separation by fluidized roasting at different roasting times

6 s to 20 s; afterwards, the rate of increase slowed down and the recovery was maintained at about 85%, while the TFe grade of iron in concentrate increased continuously with the increase of reduction time until the maximum value of 65.30% around 20 s, and then declined gently. This observation might be attributed to the following causes. When the roasting time was too short, the reaction was inadequate; this means the magnetic conversion was not adequate, so that the TFe grade and the iron recovery of the concentrate were very low. If the roasting time is too long, it is prone to cause over reduction. Moreover, the formed or original magnetite could be over reduced to wustite (FeO) if the roasting time was overlong, even at suitable temperatures. In addition, FeO easily reacts with gangue minerals such as CaO and MgO, which would result in a decrease in the recovery of iron concentrate from low-intensity magnetic separation (Cui et al., 2002). Therefore, the suitable roasting time should be kept at 16 to 20 s. In this test, 20 s was selected.

According to the test results of the above four single conditions, the determined optimum conditions were as follows: 8 m^3/h gas-flow rate with 50% H_2 at 600 ℃ and maintain roasting for 20 s. At this point, the iron concentrate with grade of 65.30% and recovery of 85.85% could be obtained after the low-magnetic separation. In the following detection and analysis, materials after roasting were all obtained under the optimum conditions.

3.5. Phase transformation after roasting

X ray diffraction (XRD) was used to identify the change of iron minerals and the formation of ferromagnetic materials after fluidized roasting, and the results of X-ray diffraction analysis of materials before and after fluidized roasting are shown in Figure 9.

Compared with the XRD pattern of materials before fluidized roasting in Figure 9, it is obvious that almost all Hematite (Fe_2O_3) in the raw sample transformed into magnetite (Fe_3O_4) after roasting, as shown in Figure 9. The peak shape of magnetite is sharp, which indicates that a lot of strong magnetic substances generated during the process of fluidized roasting, and the degree of crystallization is high (Zhang and Wang., 2014). At the same time, there is no characteristic peak of hematite, which indicates that iron minerals in materials are basically transformed into strong magnetic magnetite after fluidized roasting. It is noteworthy that the phases of wustite (FeO) and fayalite (Fe_2SiO_4) are not found in the XRD patterns of roasted samples, which means that the magnetite is not over reduced to wustite under suitable conditions.

3.6. Magnetism change of iron minerals after roasting

The specific magnetic susceptibility of minerals is an important physical parameter to measure the strength of mineral magnetism, and it is an important basis for judging the possibility of separation of various minerals by magnetic separation. In order to find out the magnetic changes of materials before and after roasting, the magnetization and specific magnetic susceptibility were measured by VSM. The magnetization and specific magnetic susceptibility of the materials before and after the fluidized roasting changed with the external magnetic field intensity, which are shown in Figures 10 and 11.

Figure 10 exhibits the magnetism curves of raw sample. It can be seen that the magnetization of the raw material increases linearly with the increase of the applied magnetic field intensity, and does not reach saturation, which declares the raw sample is paramagnetic. Meanwhile, the specific magnetic susceptibility decreases slowly with the increase of applied magnetic field, indicating that the raw sample is weakly magnetic.

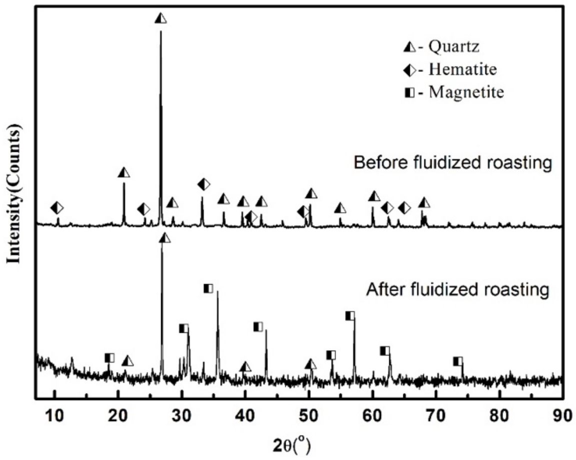

Fig. 9. XRD patterns of materials before and after fluidized roasting

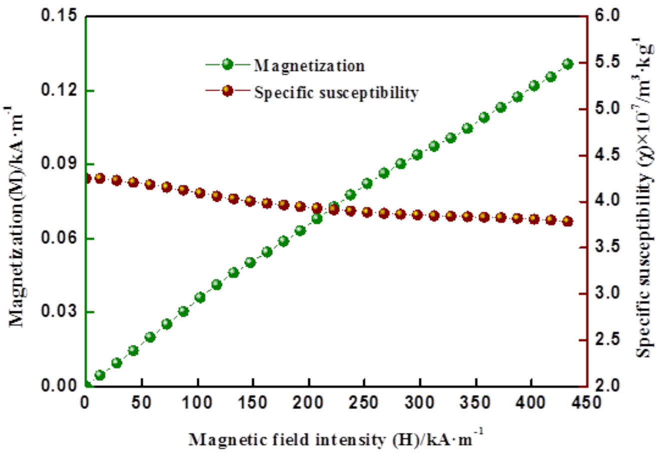

Fig. 10. Analysis of magnetic properties of materials before roasting (raw materials)

From Figure 11, it is known that the magnetization of roasted sample shows a sharp increase at first with the increase of the external magnetic field intensity, and then increased slightly, tends to be constant and reaches saturation. As to the specific magnetic susceptibility, with the increase of the applied magnetic field intensity, its curve increases rapidly to the maximum value of 3.69×10^{-4} m^3/kg at 44.35 kA/m intensity, and then inclined continuously, just making the curve in the shape of a hill. When the external magnetic field is low, the magnetic domain wall in magnetite moves and the magnetic moment turns to the direction of magnetic field until the magnetic saturation state obtained with the increase of magnetic field intensity. After that, with the intensity of magnetic field continues to increase, the magnetic moment does not increase further and becomes constant, so the ratio of magnetic susceptibility decreases. Comparing Figure 11(after roasting) with Figure 10(before roasting), it can be found that the iron minerals in the raw sample were transformed into strong magnetic magnetite after fluidized roasting, and the specific magnetic susceptibility of the roasted sample increased significantly, which expanded the magnetic difference between iron minerals and gangue minerals, and could be effectively separated through weak magnetic separation (Li et al., 2015; Jang et al., 2014).

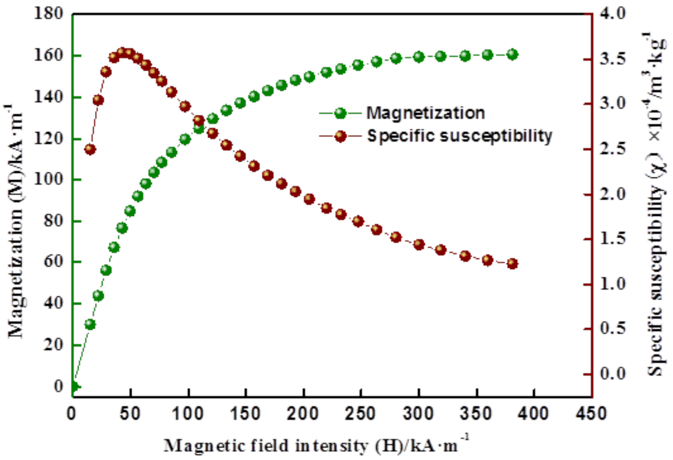

Fig. 11. Analysis of magnetic properties of materials after roasting

4. Conclusions

The sample used in magnetizing roasting experiment was obtained through pre-processing of the eastern tailings of the Anshan mining area. The optimum magnetizing roasting conditions were as follows: gas-flow rate of 8 m^3/h with 50% H_2 at 600 ℃, reacting for 20 s. Under the optimum conditions, the iron concentrate with the iron grade 65.30% and the recovery 39.79% could be obtained when the roasted material was separated by the Davies magnetic tube with a magnetic field intensity of 80 kA/m.

The XRD analysis and magnetic detection results indicate that fluidized magnetizing roasting treatment had a significant effect on magnetic properties of the iron minerals in eastern tailings of Anshan mining area. The main iron minerals in the tailings exist in the form of Hematite-limonite. After roasting, the weakly magnetic minerals (i.e. hematite and limonite) were mostly transformed into strongly magnetic mineral (i.e. magnetite), and the specific magnetic susceptibility of the roasted sample increased significantly, and then the magnetic difference between the iron minerals and the gangue minerals was expanded. Therefore, the iron could easily be concentrated by low intensity magnetic separation.

The effect of fluidized magnetizing roasting on magnetism transformation of tailings containing weakly magnetic iron minerals was prominent. The treatment of East tailings of Ansteel by pre-enrichment - fluidized magnetizating roasting - magnetic separation technology is a new attempt, and has a good concentration effect. Therefore, the application of magnetizing roasting was prospective to the efficient recovery and utilization of iron ore tailings.

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