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An innovative methodology for recycling iron from magnetic concentrate of an iron ore tailing

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Abstract: Iron ore tailing is a kind of hazardous solid waste produced by iron and steel industry. In order to separate and recycle iron from the magnetic concentrate of an iron ore tailing, an innovative technological route of fluidized magnetization roasting followed by low-intensity magnetic separation was proposed in this paper. The effects of roasting temperature, dosages of reducing gas CO and fluidizing gas N₂ on recovery rate of iron were carried out and optimized. The results showed that the hematite was almost reduced to magnetite by a gas mixture of 4 m³/h CO and 1 m³/h N₂ at roasting temperature of 540 °C. Under the optimized conditions, a magnetic concentrate assaying 61.4 wt% Fe with a recovery rate of 81.8% was obtained from the magnetic concentrate of an iron ore tailing. The iron chemical phase, X-ray diffraction (XRD), and optical metallographic microscope analyses revealed that siderite was converted to magnetite successfully after roasting, and some coarse magnetite-hematite interlocking particles were formed due to insufficient reaction time, which could also be recovered by magnetic separation after liberating from gangue minerals.

Keywords: magnetic concentrate, iron ore tailing, iron recycling, fluidized magnetization roasting, magnetic separation

1. Introduction

With the increasing demand in the World, the iron and steel industry has developed rapidly. The total production of iron ore in the World added up to 3320 Tg in 2014 which had increased 3.11% compared with that of 2013. China ranks the first place among iron ore production in the World. Meanwhile, a continuous increase of hazardous industrial wastes such as iron ore tailing generated by iron steel industry is becoming a serious problem (Li et al., 2010; Matschullat et al., 2000). In China, according to statistics, about more than 600 Tg of iron ore tailings are produced each year (Chang, 2010). These residues not only pollute environment and give rise to security risks, but also occupy huge land for the construction of iron ore tailings disposal sites, which will increase the production cost of iron ore dressing plants (Das et al., 2000; Zhang et al., 2006; Giri et al., 2011; Zhao et al., 2014).

In order to alleviate environmental pressures and attain sustainability, one feasible solution is to reuse iron ore tailings as secondary resources. In this regard, numbers of studies on utilization of iron ore tailings were carried out, such as recycling of useful metal like Fe (Li et al., 2010), producing of construction materials directly (Zhang et al., 2006; Yellishetty et al., 2008; Zhao et al., 2014), and using as mine filling materials (Jiang et al., 2014; Ji et al., 2014). However, in this investigation, we mainly introduce the relative contents of iron recovery.

Many researchers have conducted numerous studies on iron recovery from iron ore tailings, and the results indicate that the coal-based magnetizing roasting and direct reduction followed by magnetic separation are the main and effective methods to recycle iron from tailings (Li et al., 2010; Yang et al., 2011; Zhu et al., 2012; Yang et al., 2012; Zhang et al., 2012; Fan et al., 2015). The coal-based magnetizing roasting or direct reduction of iron ore tailings usually occurs in a rotary kiln. However, several years of engineering practice indicate that the rotary kiln magnetizing roasting or direct

reduction is characterized by high energy consumption, low efficiency of heat transfer and mass transfer as well as by heterogeneity of roasted products. Especially, the formation of clinker ring in the rotary kiln affects the yield of iron resource. Thus, it is unreasonable or uneconomic to directly adopt magnetizing roasting or direct reduction technology to process the enormous amounts of iron ore tailings due to their low iron content.

For this purpose, an innovative technological route of combined preconcentration with high-gradient magnetic separator, and fluidized magnetizing roasting followed by low-intensity magnetic separation is proposed. Meanwhile, fluidized magnetizing roasting has become a research focus in recent years due to its high heat/mass transfer efficiency, and low energy consumption (Jiu et al., 2008; Zhu and Li, 2014; Yuan et al., 2016; Yu et al., 2017a, 2017b, 2017c; Chen et al., 2017; Liu et al., 2017). In this paper, the preconcentration of iron ore tailing with high-gradient magnetic separator was not introduced, instead the fluidized magnetizing roasting, by which the weakly magnetic hematite and siderite could be transformed into ferromagnetic magnetite quickly (about 50 s), was investigated in particular. Thus, the new generated magnetite could be captured by magnet into magnetic concentrate, and nonmagnetic substances like quartz could enter non-magnetic tailings after low-intensity magnetic separation.

2. Material and methods

2.1 Raw materials

The magnetic concentrate of an iron ore tailing with 77 wt% passing 0.038 mm were sampled from an iron ore dressing plant in Anshan of Liaoning Province, China. The chemical compositions are given in Table 1, and the XRD pattern of the magnetic concentrate is shown in Fig. 1. The results of XRD and chemical analysis showed that the magnetic concentrate was mainly composed of quartz, hematite and a bit of siderite.

The combustion fuel for suspension heating furnace was water gas (H₂ 50%, CO 40%), and its consumption was about 3 kg/h. The reducing gas was CO with a purity of 99.9%, and the fluidizing gas was N₂ with a purity of 99.9%.

Table 1. Chemical compositions of magnetic concentrate

Constituents	TFe	SiO ₂	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	S	P	LOI
Content (wt%)	33.19	41.19	1.82	1.77	1.78	0.12	0.30	0.05	0.07	5.87

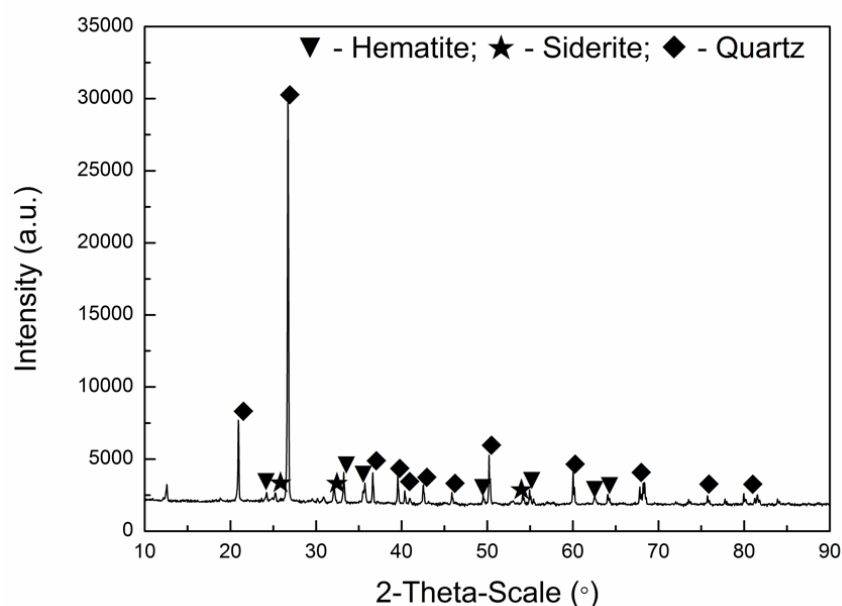


Fig. 1. XRD pattern of magnetic concentrate

2.2 Fluidized magnetization roasting and low-intensity magnetic separation

A pilot plant scale fluidized magnetizing roasting reactor with a throughput of 120 kg/h was used in the investigation. As shown in Fig. 2, it mainly consists of a cyclone preheater, a suspension heating furnace, a gas-solid cyclone separator and a cooling unit. The reactor is equipped with thermocouples and pressure sensors for the precise adjustment of the temperature and pressure.

The fluidized magnetizing roasting reactor was operated under a negative pressure condition. The dry magnetic preconcentrate was preheated in countercurrent with the hot off-gas from the suspension heating furnace, and then entered into the suspension heating furnace. The hot gases (about 700-900 °C) produced by the combustion of water gas simultaneously heated and transported the magnetic preconcentrate to the gas-solid cyclone separator where siderite was thermally decomposed or oxidized to magnetite. Then, the hot magnetic preconcentrate (about 600-750 °C) flowed downward into the fluidized magnetization bed under the action of gravity. Consequently, the hematite was reduced to magnetite quickly by CO, and the retention time was estimated to 50 s. After reduction roasting, the gas mixture transported the magnetic preconcentrate (about 450-600 °C) to the cooling unit. Meanwhile, the residual CO gas entered into the suspension heating furnace and burnt with the water gas.

For all the investigations, the feed velocity for fluidized magnetization roasting reactor was determined to be 120 kg/h. The roasted products under different roasting conditions were ground to 95% passing 0.038 mm in a ball mill, and then were prepared at a predetermined 30 wt% solid content in a stir tank of 5 dm³ volume. As shown in Fig. 2, 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 dm³ slurry was evenly fed to the magnetic separator within 5 min.

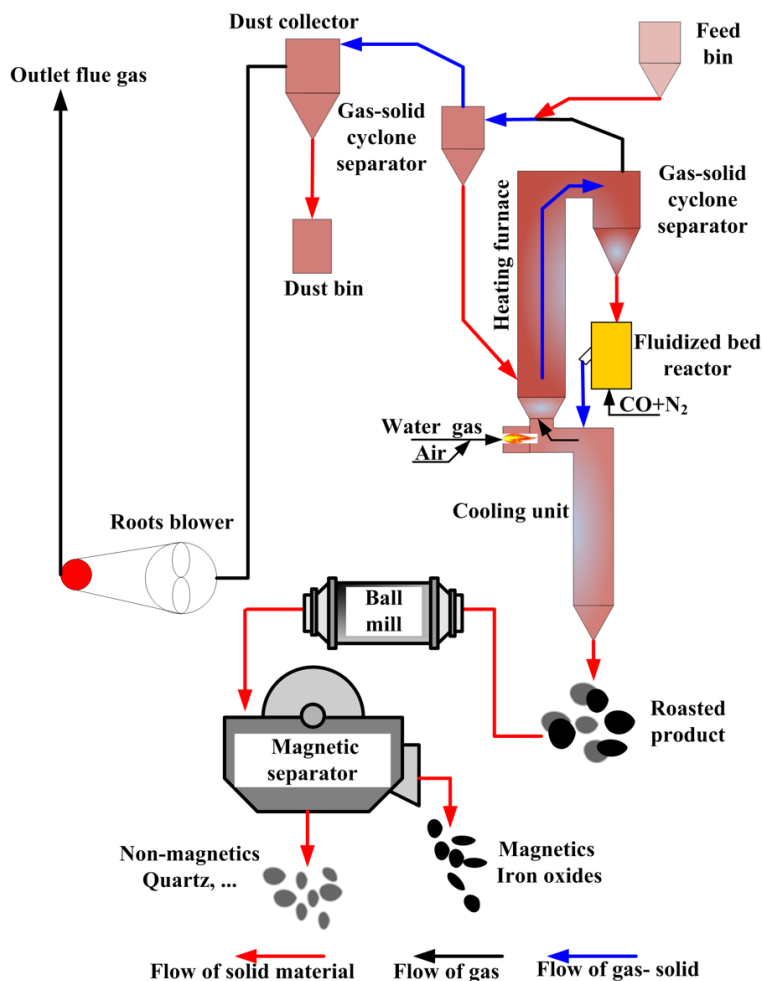


Fig. 2. Sketch of fluidized magnetizing roasting reactor by means of a gas suspension process followed by low-intensity magnetic separation

2.3 Measurements

The grades of magnetic concentrates and tailings were analyzed by chemical method, and the recovery rate of iron was deduced according to mass balance.

The solid phase structures of magnetic preconcentrate and roasted product were analyzed by X-ray diffraction (X' Pert PRO MPD) with Cu K α radiation, and the operating voltage and current were 40 kV and 40 mA, respectively. The diffraction angle was scanned from 10° to 90°.

A Leica DM4 P optical microscope (Wetzlar, Germany) was used to study the association of hematite with magnetite in the roasted product by examining polished thin sections. The magnetite and hematite could not be distinguished under a scanning electron microscope (SEM), thus the SEM analysis for roasted product was not carried out.

3. Results and discussion

3.1 Effect of roasting temperature on iron recovery

The roasting temperature, which was controlled by adjusting the dosage of burning water gas in suspension heating furnace, was the most important process parameter. Thus, the influence of roasting temperature on the recovery of iron was investigated in a range of 480-560 °C. The other process conditions such as the dosages of reducing gas CO and fluidizing gas N₂ were fixed to be 3 m³/h and 2 m³/h, respectively. The results are plotted in Fig. 3.

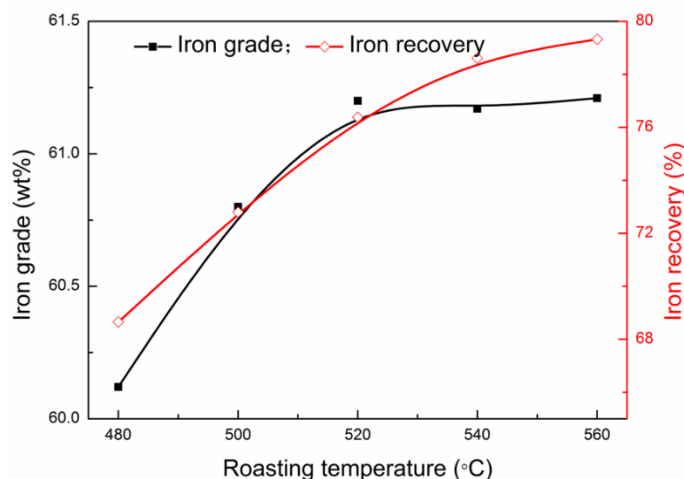


Fig. 3. Effect of roasting temperature on recovery of iron

As shown in Fig. 3, the roasting temperature had a significant effect on the recovery of iron for magnetic product. It was found that low reduction temperature was accompanied by a low iron recovery. When the reduction temperature was low, because of their limited reaction rate (Zhang et al., 2012), only a small fraction of hematite was reduced to magnetite that can be captured by the magnet, which resulted in the low iron recovery of magnetic product. As the reduction temperature was increased, more sufficient reaction rate was available for most of the hematite being reduced to magnetite, and the iron recovery was greatly improved. From Fig. 3, the iron grade of magnetic product almost kept as a constant after a slight increase from 480 °C to 520 °C. When the roasting temperature was 540 °C, the iron grade and recovery were both high. Further raising the temperature, the iron recovery just increased negligibly. Therefore, the roasting temperature was determined to be 540 °C.

3.2 Effect of dosage of reducing gas CO on iron recovery

Fig. 4 shows the effect of CO dosage on the recovery of iron in the magnetic fraction under the conditions of roasting temperature 540 °C and fluidizing gas N₂ 2 m³/h. With increasing CO flow rate from 2.5 m³/h to 4.0 m³/h, both the iron grade and recovery of magnetic product were gradually increased.

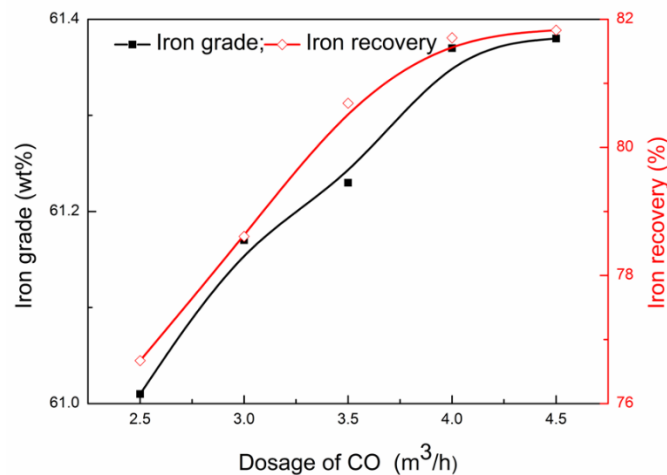


Fig. 4. Effect of CO dosage on recovery of iron

It was easy to understand that increasing in the CO flow rate meant a higher CO concentration in the mixed CO/N₂ gases. Thus, the diffusion of reducing gas CO to hematite particles was enhanced, which resulted in a high iron recovery and grade of magnetic product (Li and Gao, 2017). The dosage of CO was further increased up to 4.5 m³/h, the iron recovery and grade remained almost unchanged. Thus, a CO dosage of 4.0 m³/h was suitable for the magnetic preconcentrate.

3.3 Effect of dosage of fluidizing gas N₂ on iron recovery

Fig. 5 exhibits the effect of N₂ dosage on the recovery of iron in the magnetic fraction under the conditions of roasting temperature 540 °C and reducing gas CO 4 m³/h. With increase in the dosage of N₂, both of the iron grade and recovery of magnetic product were gradually decreased. It was clear that an increase in N₂ dosage corresponded to a decrease in CO concentration for the gas mixture, thereby deteriorating the diffusion of CO to the hematite particles. On the other hand, the flow speed of magnetic preconcentrate in the fluidized bed was improved with the increase in N₂ dosage, which decreased the reduction roasting time for hematite particles. Thus, the iron grade and recovery of magnetic fraction gradually decreased with the increase of N₂ dosage. It should be pointed out that the N₂ flow rate was a key parameter in the operation of fluidized magnetizing roasting, if the fluidizing gas N₂ was not introduced to the fluidized bed (the dosage of N₂ was zero), the magnetic preconcentrate could not escape from the fluidized bed due to their insufficient flow speed, leading to blockage. While, if the dosage of N₂ was too high, the retention time was insufficient for hematite being transformed to magnetite. Therefore, a N₂ dosage of 1 m³/h was selected for the magnetic preconcentrate.

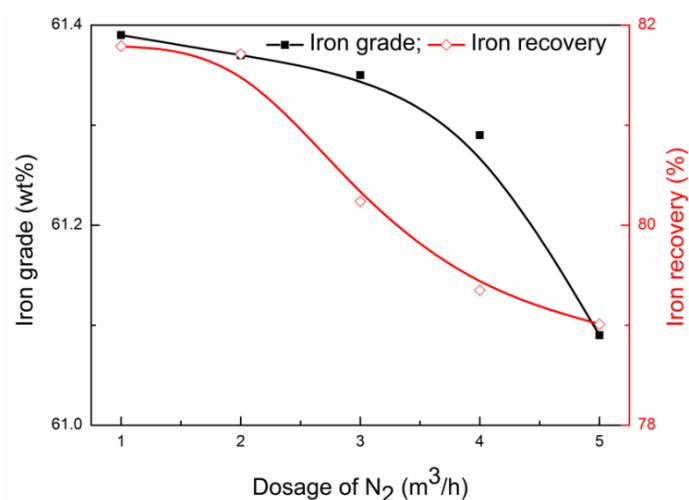


Fig. 5 Effect of N₂ dosage on recovery of iron

It can be seen from Fig. 5 that, a high-grade concentrate containing 61.4 wt% Fe with a recovery rate of 81.8% was obtained through the process fluidized magnetizing roasting and low-intensity magnetic separation, which indicated that the innovative process was effective for recycling iron from iron ore tailing. Meanwhile, the final tailing could be potentially used to make construction materials for creating more economic benefit. The chemical analysis for the compositions of the magnetic concentrate is given in Table 2.

Table 2. Chemical compositions of magnetic concentrate

Constituents	TFe	SiO ₂	Al ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	S	P
Content (wt%)	61.37	10.24	0.88	0.53	0.92	0.05	0.06	0.06	0.03

It was implied in Table 2 that the grade of iron concentrate was qualified for the requirements of companies (Huang 2005), and the amounts of impurities like S and P were very low by 0.063 wt% and 0.034 wt%, respectively. It could be used as a qualified raw material for ironmaking. Meanwhile, it was easy to understand that magnetite was the main mineral phase of magnetic concentrate, but quartz also existed with a content of 10.24 wt%, which proved that iron and quartz were fine-disseminated complexly together and it was hard to be separated.

3.4 Phase transition after fluidized roasting

3.4.1 XRD analysis

Fig. 6 shows the XRD traces for roasted product at above optimum conditions. Compared with the XRD pattern of magnetic preconcentrate (Fig. 1), it was clear to see that the hematite (Fe₂O₃) in the magnetic preconcentrate was almost reduced to magnetite (Fe₃O₄) completely after roasting with mixed CO/N₂ gases due to no finding the peaks of hematite, which indicated that a good low-intensity magnetic separation result would be achieved. Meanwhile, the characteristic diffraction peaks of siderite disappeared, indicating that the siderite was either decomposed or oxidized into magnetite during the fluidized roasting (Zhang et al., 2002).

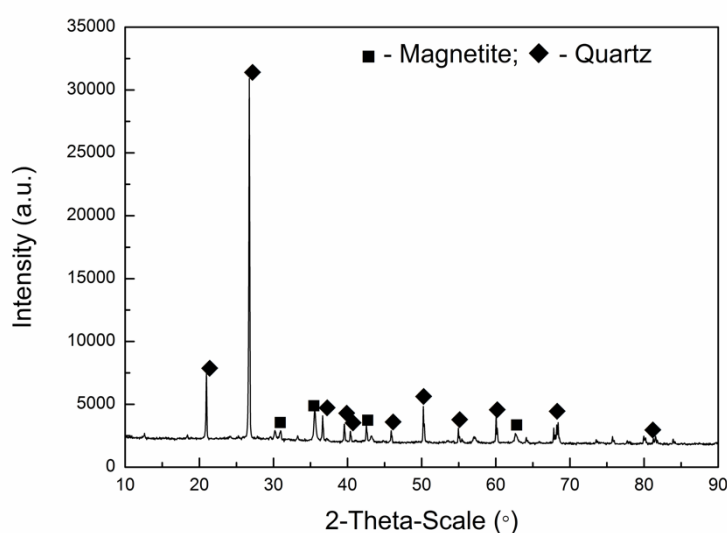


Fig. 6 XRD pattern of roasted product

3.4.2 Iron chemical phase analysis

Table 3 shows the chemical phases of Fe and its distribution for magnetic preconcentrate before and after roasting. It could be seen that iron elements in magnetic preconcentrate were mainly distributed in hematite (66.98%), magnetite (13.99%) and carbonate (17.28%) as well as in iron silicate minerals (in

total 1.75%). After fluidized magnetizing roasting, iron elements in roasted product were mainly distributed in magnetite (85.11%), hematite (8.26%) and carbonate (4.67%) as well as in iron silicate minerals (in total 1.96%), which indicated that most of the hematite was converted to magnetite after roasting. Meanwhile, the distribution of iron carbonate minerals had a great decrease after roasting. This observation might be contributed to the decomposition or oxidization of siderite into magnetite during the fluidized roasting process (Chun et al., 2015). Thus, their separation from non-magnetic gangue minerals was facilitated.

Table 3. Chemical phases of Fe and its distribution for magnetic preconcentrate before and after roasting

Sample	Mineral	Hematite	Magnetite	Carbonate	Silicate	Total grade
Magnetic preconcentrate before roasting	Fe grade, %	22.22	4.64	5.73	0.6	33.19
	Fe distribution, %	66.98	13.99	17.28	1.75	100
Magnetic preconcentrate after roasting	Fe grade, %	2.74	28.25	1.55	0.71	33.25
	Fe distribution, %	8.26	85.11	4.67	1.96	100

3.4.3 Optical microscopy analysis

The optical microstructure images of roasted product are illustrated in Fig. 7. It was clear to see that most of the hematite particles (see Fig. 7(c) and (d)), except for some coarse ones, were reduced to magnetite by CO, i.e., the surface of some coarse hematite particles was reduced to magnetite, but the inner core was still hematite crystal lattice (Gao et al., 2014), as shown in Fig. 7(a) and (b). While such these magnetite-hematite interlocking particles could also be recovered into magnetic concentrate after liberating from gangue minerals due to their strong magnetism. Meanwhile, the results are greatly in agreement with above analyses.

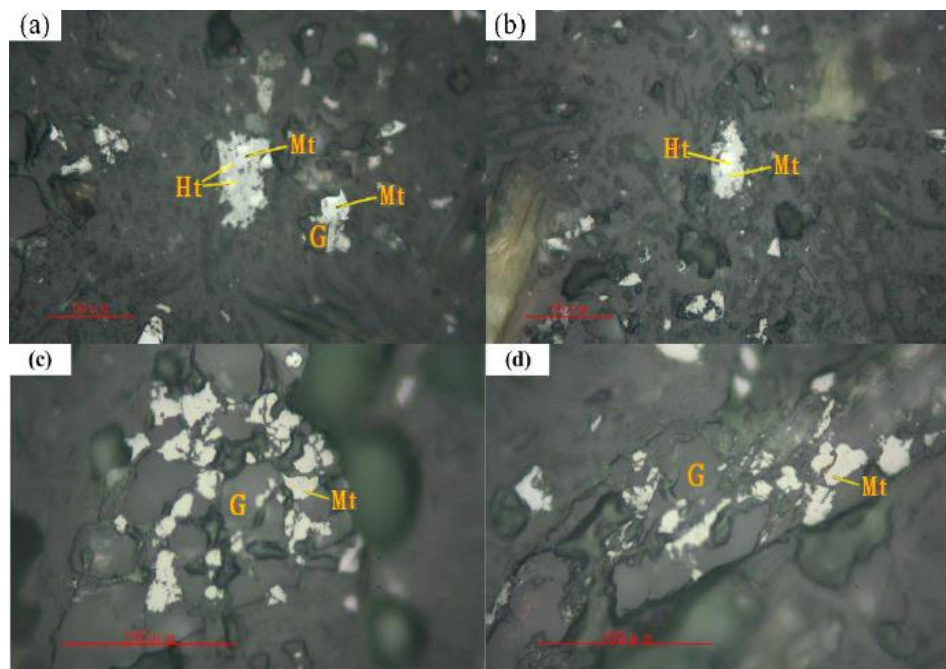


Fig. 7. Optical microstructure images of roasted product: Mt = Magnetite; Ht = Hematite; G = Gangue mineral

4. Conclusions

The optimized roasting temperature, CO flow rate and N₂ flow rate for magnetic preconcentrate were 540 °C, 4 m³/h and 1 m³/h, respectively. Under the optimum conditions, the iron grade and recovery of magnetic concentrate were 61.4 wt% and 81.8%, respectively.

The XRD and iron phase analyses as well as photomicrographs of the roasted product showed that most of the hematite and siderite particles, except for the coarse ones, were converted to magnetite successfully. Thus, their separation from non-magnetic gangue minerals was facilitated, and a good low-intensity magnetic separation result was achieved.

The results demonstrated the technical feasibility of fluidized magnetizing roasting and low-intensity magnetic separation technology for recycling iron from the magnetic preconcentrate of iron ore tailings.

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