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Recovering limonite from Australia iron ores by flocculation-high intensity magnetic separation①

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Abstract: Successful recovery of limonite from iron fines was achieved by using flocculation-high intensity magnetic separation (FIMS) and adding hydrolyzed and causticized flocculants according to the characteristic of iron fines. The separation results of the three iron samples are as follows: iron grade 66.77%-67.98% and the recovery of iron 69.26%-70.70% by the FIMS process with flocculants. The comparative results show that under the same separation conditions the FIMS process can effectively increase the recovery of iron by 10.97%-15.73%. The flowsheet results confirm the reliability of the process in a SHP high intensity magnetic separator. The concentrate product can be used as raw materials for direct reduction iron-smelting. The hydrolyzed and causticized flocculants can selectively flocculate fine feebly-magnetic iron mineral particles to increase their apparent separation sizes. The larger the separation size, the stronger the magnetic force. By comparing the separation results of the three samples it is found that among the three samples the higher the limonite content, the better the separation result. This means that the separation result relates closely to the flocculation process and the adding pattern of the flocculant.

Key words: Australia iron ores; flocculation; high intensity magnetic separation; limonite
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1 INTRODUCTION

In the practice of iron ore beneficiation, high intensity magnetic separation (HIMS) has been successfully applied in recovery of hematite containing fine feebly magnetic iron minerals. There are a great number of researches on the separation principles, both theoretically and practically[1-8], whereas there exist few on the commercial application in recovery of fine limonite. These researches are focused on flocculation and depression of iron oxide minerals and fine hematite separation. In industrial production, there are still some difficulties in separation of the limonite-enriched fine refractory iron minerals using the similar separation process to the processing of hematite.

Australia is abundant in high grade iron ore resources. Among Australia iron ores, most of lump ores are directly exported, and the rest with grade about 60%Fe, and size less than 15 mm are stockpiled for further use and higher content of limonite.

In this paper, the feasibility of separating fine feebly magnetic iron minerals from Australian iron ores by the flocculation-high intensity magnetic separation process was investigated. Main parameters affecting the process, including the magnetic intensity, addition and pulp-condition of polymeric flocculants and limonite content of ore samples on the separation process were studied.

2 PRINCIPLE OF MICRO-FINE PRATICLES SEPARATION

Magnetic force on a mineral particle in a magnetic separation system is expressed as follows:

$$F_m = \kappa_p V_p H_i H_{grad} = \chi_p m_p H_i H_{grad} \quad (1)$$

where F_m is the magnetic force on a particle; κ_p is the volumetric magnetic susceptibility; V_p is the volume of a particle; χ_p is the specific magnetic susceptibility; m_p is the mass of a particle; H_i is the magnetic field intensity of a particle; H_{grad} is the magnetic field gradient of a particle.

Eqn. (1) shows that magnetic particles and non-magnetic particles differ in applied magnetic force, and their applied magnetic forces decrease dramatically with reducing their sizes, resulting in a great reduction in the separation efficiency of small magnetic particles, ever "zero" efficiency in a certain size range. Concrete size ranges depend on the types of minerals to be separated, and the magnetic intensity and magnetic field gradient[9]. For instance, if a Jones high magnetic separator is used to separate hematite ore, its effective lower limit of

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separation size ranges from 10 to 20 μm. The high intensity magnetic separator has a serious defect, namely its low recovery, when it is used to recover feebly magnetic particles below 10 μm.

There are three factors affecting the separation efficiency of fine feebly-magnetic particles, magnetic field intensity, magnetic field gradient and particle size, and the first two types can be controlled by using a super-conducting separator or a high gradient magnetic separator[10, 11], and the last type by using FIMS process. By using the flocculation-high intensity magnetic separation (FIMS) process, fine feebly magnetic mineral particles can be agglomerated selectively by flocculants to increase their apparent size for improving their magnetic force, and then recovered by high intensity magnetic separation, thus achieving the effective recovery of fine feebly magnetic mineral particles using conventional high intensity magnetic separators. There are many ways available to agglomerate fine mineral particles in hydrolyzed suspension[12, 13], such as electrolytic coagulation, polymer flocculation, electrocoagulation, hydrophobic flocculation and magnetic separation, etc.

Natural high polymer is causticized and hydrolyzed to make its carbon chains extend and group. By the action of hydrogen bonds, polar function groups formed by hydrolization can approach and contact fine magnetic particles selectively and effectively, polymerizing and agglomerating them by the bridging effect of carbon chains, and then form aggregates through flocculation polymers[14]. During the period of magnetic separation, magnetic force in magnetic field acts on all aggregates as an organic whole, making fine magnetic particles agglomerated by flocculation. Therefore, they can be separated and enter into concentrate products, resulting in a great increase in the recovery of magnetic particles.

3 EXPERIMANTAL

3.1 Raw materials and equipment

3.1.1 Samples

All the three samples (A, B and C) with a size less than 15 mm, from Australian iron ore Pty Ltd, belonged to the type of low-sulphur and phosphorus high grade iron ores. These fine ores were stockpiled for further use. The samples A, B and C had higher iron contents, 62.18%, 59.22% and 63.02%, respectively. By microscopic observation, X-ray diffraction analysis and electron probe semi-quantitative analysis, the results indicate that their main iron minerals are limonite, martite hematite, hematite and goethite as well as a small amount of magnetite, and their gangue minerals are kaolinite and quartz, which are cloudily disseminated in minerals. Since the limonite was heavily weathered, the samples were porous and had high porosity, resulting in the difficulty in separation. Their main chemical elements and mineral components are shown in Tables 1 and 2. In the small-scale laboratory test, the three samples were crushed to below 2.0 mm.

Table 1 Main chemical compositions of three samples (Mass fraction/%)

Sample	TFe	Fe_2O_3	SiO_2	Al_2O_3	CaO
A	62.18	88.47	3.54	1.83	0.29
B	59.22	84.11	5.24	2.54	0.27
C	63.02	89.70	3.06	1.42	0.32
Sample	MgO	P	S	H_2O^+	Ig
A	0.056	0.057	0.042	4.65	4.98
B	0.210	0.052	0.024	6.24	6.72
C	0.074	0.053	0.030	4.84	4.88

Table 2 Main mineral compositions and content of three samples (Mass fraction/%)

Sample	Hematite	Goethite limonite	Magnetite	Kaolinite	Quartz
A	44.0	48.0	1.0	5.0	2.0
B	32.1	57.6	0.6	7.4	2.3
C	50.6	43.0	0.9	4.0	1.5

3.1.2 Reagents

Sodium hydroxide(NaOH) from the Chemical Reagent Workshop of Hunan Normal University, was chemically pure, and was used as modifying agent for pH value; water glass ($NaSiO_3 \cdot mH_2O$, $m=2.4$), prepared by Changsha Water Glass Factory, was used as pulp dispersant; and modified corn starch, produced by Changsha Starch Mill, was used as flocculent(HSD) for selectively flocculating iron minerals. The flocculent was specially hydrolyzed and causticized and then added according to the test requirements. Tap water was used in this test.

3.1.3 Test equipment

The test equipment mainly included a XMB d200 mm×240 mm rod mill, a high power agitator (adjust table rotary speed), a laboratory high intensity magnetic separator and SHP d700 high intensity magnetic separator.

3.2 Test

The flocculation-high intensity magnetic separation process mainly consists of 4 stages as shown in Fig. 1, pulp conditioning, pulp dispersion, selective polymer flocculation, and magnetic separation. The whole operation process from grinding to pulp conditioning and dispersing, selective polymer flocculation and magnetic separation of the iron samples are shown in Fig. 1.

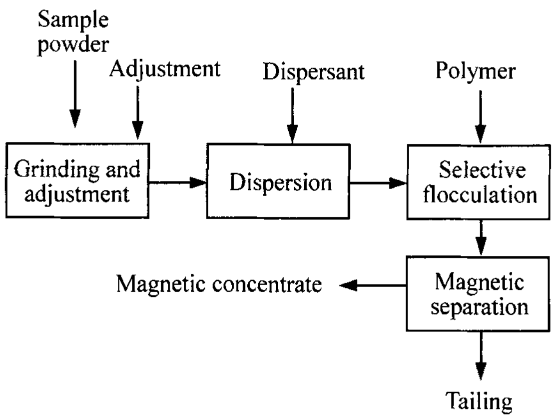

Fig. 1 Schematic diagram of flocculation magnetic separation process

3.2.1 Grinding and pulp conditioning

The prerequisite for mineral separation is that mineral particles should have a proper liberating degree. The liberation of mineral particles is affected by pulp conditioning, and so when the mineral particles are comminuted, their new surfaces have more surface activity, which helps adsorbing ions and function groups. Therefore adding sodium hydroxide as pH value modifying agent into a grinding mill can improve effectively the results of pulp conditioning. Since sample B had the highest content of limonite, it was chosen as the first object of study. During grinding, 1 000 g sample B was added to a grinding mill, then NaOH was added in proportion of 0.12%(mass fraction). The relationship between grinding time and size is listed in Table 3.

Table 3 Relationship between grinding time and content of particles with less than 0.074 mm size for sample B

Grinding time/min	Content
7.00	68.11
7.20	69.68
8.80	81.37
10.00	87.05
10.80	90.85
12.00	93.91
12.25	95.03

3.2.2 Dispersion and selective flocculation

Sample B was ground for 10 min to 87.05% of particle at 0.074 mm and used as feed slurry. The feed slurry was dispersed to separate valuable minerals particles from gangue mineral particles, thus reducing adsorption of slimes onto magnetic mineral particles to prevent flocculent from including and entraining them[15].

The feed slurry was conditioned and fed to a agitator with inner diameter of 140 mm and four-cross-impellers in 10 mm long. In this agitator, the feed slurry was conditioned to a certain concentration, and adjusted for pH value using NaOH and then dispersed using $NaSiO_3 \cdot mH_2O$ at 1200 r/min for a given time. Finally, the feed slurry was stirred at a low velocity and mixed with hydrolyzed and causticized flocculants until polymer flocs of iron minerals were formed in the slurry.

3.2.3 Magnetic separation of iron minerals

The concentration of flocculated slurry was conditioned to 20%, and then separated in a laboratory high intensity magnetic separator, which was equipped with five grooved plates with dimension of 220 mm high and 90 mm wide in its separating box. During the test, its magnetic induction density could be adjusted in the range of 0.4 - 1.5 T, and the slurry was fed at 500 mL/min. After slurry was fed completely, the wash water was fed at 300 mL/min, then a concentrate (i.e. magnetic product) and tailing (non-magnetic product) were obtained.

4 RESULTS AND DISCUSSION

4.1 Properties of sample

By microscopic observation, it is found that the three samples all consist of limonite, hematite and goethite, but their main gangue minerals such as quartz and kaolin, are distributed randomly in iron minerals as small aggregates or intergrowth with goethite in the form of microcrystal. Therefore it is very difficult to liberate them. Moreover the limonite and goethite are very easy to slime during their grinding and mechanical separation operations, worsening the separation operation and resulting in a great loss in iron minerals. For sample B, the goethitized limonite accounts for 2/3 of the total content of the iron minerals. The mining analysis of the samples shows that the higher the gangue content, the lower the iron grade and the smaller the specific magnetic susceptibility. The fine limonite might be recovered in the flocculation-high intensity magnetic separation process. However it is difficult to achieve high iron recovery.

4.2 Analysis of magnetic separation products

In order to investigate the concentrate grades and iron recoveries obtained at various magnetic intensities, the separation conditions are as follows: content of particles with size less than 0.074 mm is 87.05%, pulp density is 20%, and the reagent dosages are NaOH 0.12%, $NaSiO_3 \cdot mH_2O$ 0.1% and hydrolyzed and causticized modified starch NaOH 0.12%. The relationships between the concentrate iron grade and the iron recovery obtained by primary magnetic separation of sample B at magnetic intensities of 0.3 - 1.8 T are shown in Fig. 2. The limonite-enriched iron concentrate is easy to damp by moisture adsorption, affecting the calibration of iron grade. The iron grades in Fig. 2 are obtained by loss on ignition (LOI) at 900 ℃, hereinafter the same. The practical effects of conventional magnetic separation under the same separation conditions are also shown in Fig. 3.

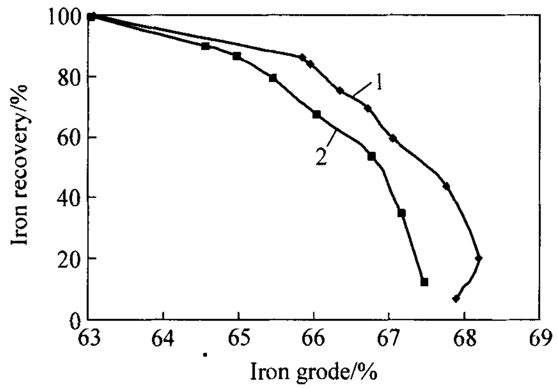

Fig. 2 Relationship between iron grades and iron recoveries of sample B
1—With reagent; 2—Without reagent

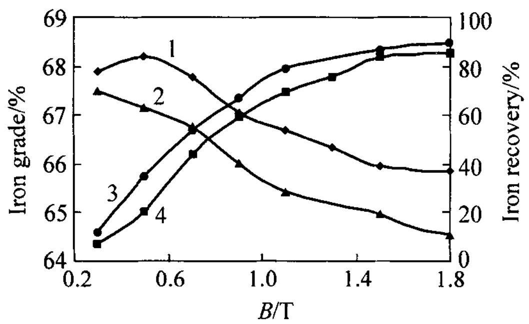

Fig. 3 Relationship between iron grades and iron recoveries with magnetic intensities for sample B
1—Iron grade with reagent; 2—Iron grade without reagent; 3—Iron recovery without reagent; 4—Iron recovery with reagent

From Figs. 2 and 3, it is shown that the changes in the separation results by flocculation-high intensity magnetic separation of sample B are similar to those by conventional magnetic separation, namely, the higher the iron recovery, the lower the concentrate iron grades. However both of them differ greatly in variation range. As shown in Figs. 2 and 3, when the iron grade using the flocculation-high intensity magnetic separation process is 66.70%, the iron recovery is 69.26%; whereas at the iron grade of 66.77%, which is about the same as the iron grade by the flocculation-high intensity magnetic separation process, the iron recovery by conventional magnetic separation is only 53.53%. In other words, the former's iron recovery is 15.73% higher than that of the latter. The sample's iron recovery can be improved greatly by adding hydrolyzed and causticized flocculants after pulp conditioning. From the analysis of the test results, it is shown that dispersing suspension after controlling pH value helps selective adsorption of hydrolyzed and causticized flocculants on the surfaces of limonite. In hydrolyzed suspension, the flocculant has a bridging effect on magnetic particles, which can make their hydrolyzed layers agglomerate, whereby further improving the strength of flocculated particles. With increasing their strength, the flocculated particles can stand the shear and tension of pulp flow, ensuring against their coming off during magnetic separation. The flocculants cannot only increase the agglomerating force for bridging the flocculated particles, but also reduce the porosity between particles and increase their probability of mutual contact due to polymeric agglomeration of the hydrolyzed layers helping formation of flocs of fine feebly-magnetic limonite particles. The flocs are absorbed and recovered on the grooved plates. This is the key reason why the flocculation-high intensity magnetic separation process can improve the iron recovery.

As shown in Fig. 2, when the iron recovery is low (<20%), the iron grade decreases. The main reason is that fine iron monomineral particles which have formed magnetic coagulations can not be separated at a low magnetic intensity of around 0.5 T and that part of coarse iron mineral particles recovered contain microgangue.

4.3 Dosage of flocculants

Some factors, such as particle size, pulp conditioning, flocculation and magnetic separation, can affect the results of FIMS process. However, the selective flocculation process of fine feebly-magnetic mineral particles is the key. For this reason, on the separation effect of the process, we should study only the effect of flocculent dosage on the separation of the sample at constant reagent dosage (HSD) and agitating strength and time. These conditions are as follows: pulp concentration of 20%, NaOH dosage of 0.12%, agitating speed of 1 200 r/min; $NaSiO_3 \cdot mH_2O$ dosage of 0.10%, low velocity agitation and magnetic field intensity of 0.65 T. The effects of various flocculants dosages on concentrate separation results are shown in Fig. 4. From Fig. 4, it can be seen that the optimum dosage of flocculants is about 0.08%.

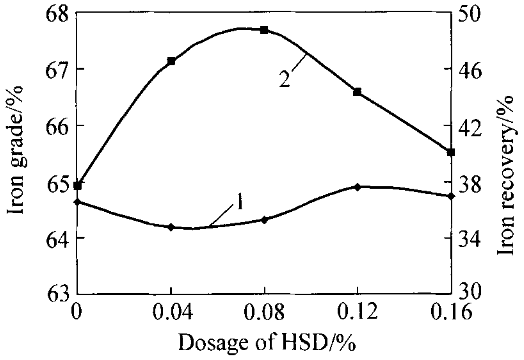

Fig. 4 Separation results obtained at various HSD dosages

1—Iron grade; 2—Iron recovery

4.4 Beneficiation efficiency of iron by FIMS

According to analysis of the three samples' properties, we investigated their separation effects by using FIMS process under optimum reagent-adding and pulp conditioning conditions, the test results are listed in Table 4.

From Table 4, it can be seen that good results can be obtained by flocculation and high intensity magnetic separation. Comparing Table 2 with Table 4, it is found that the contents of limonite in the three samples accord with the increase in their concentrate recovery, namely the higher the limonite content, the higher the iron recovery. The increase of iron recovery is due to the fact that fine limonite particles are flocculated to increase their apparent separation sizes, making them absorbed and recovered on the grooved plates.

Table 4 Results obtained by using FIMS of three samples with or without reagents

Samples	Concentrate	Yield/%	Iron grade/%		Iron recovery/%	Increase in iron recovery/%
			Before LOI	After LOI		
A	With reagent	64.69	66.09	67.98	69.18	12.83
	Without reagent	52.26	66.05	67.56	56.35	
B	With reagent	64.02	63.92	66.70	69.26	15.73
	Without reagent	49.21	63.06	66.77	53.53	
C	With reagent	67.93	65.10	67.46	70.70	10.97
	Without reagent	56.62	65.48	67.50	59.73	

4.5 Separation results

In order to investigate the applicability of the FIMS process to recovery of fine feebly-magnetic minerals and the removal of feebly-magnetic impurities, the process on primary roughing and primary scavenging was carried out using the FIMS process. SHP *d*700 high intensity magnetic separators were used in the test. When the separators' magnetic induction strength was set to be 0.7 T and 0.9 T, and other operation conditions could be properly adjusted. The test results for samples B with 81.40% in feed size less than 0.074 mm are shown in Table 5.

Table 5 Separation results using sample

Magnetic induction strength/T	Product	Yield/%	Iron grade/%		Iron recovery/%
			Before LOI	After LOI	
0.7	Concentrate 1	51.51	63.11	66.49	55.01
0.9	Concentrate 2	12.91	60.34	64.97	13.18

Iron grade 66.19% and iron recovery 68.18% can be obtained using the FIMS process.

Another investigation shows that better results are obtained in the study on direct reduction iron-smelting from the concentrate products obtained by using the FIMS process. This means that the limonite-enriched iron fines obtained using the FIMS process can be used as raw material for direct reduction of iron.

5 CONCLUSIONS

1) Iron fines (with size less than 15 mm) rich in limonite from Australia are easy to slime, and so they are stockpiled for further use. The selective flocculation using hydrolyzed and causticized polymer can be achieved by HIMS. The concentrate with iron grade 66.77%-67.98% and iron recovery 69.26%-70.70% can be obtained by the FIMS process with flocculants. At similar iron grades, their iron recoveries can be increased by 10.97%-15.73%.

2) The selective flocculation and agglomeration of fine limonite particles can be achieved by hydrolyzing polymer's bridging contact and reducing particle's porosity, and increasing the apparent size of the particles can achieve high efficiency recovery of fine feebly-magnetic iron minerals by SHP high intensity magnetic separators.

3) The FIMS process can be used for processing iron fines from Australia and the concentrate product can be used as raw materials for direct reduction iron-smelting.

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