

UPGRADING IRON AND REMOVING PHOSPHORUS OF HIGH PHOSPHORUS OOLITIC IRON ORE BY SEGREGATION ROASTING WITH CALCIUM CHLORIDE AND CALCIUM HYPOCHLORITE

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Abstract

The iron-bearing ore, existing in the form of oolite, was mainly composed of hematite, limonite, daphnite, and collophane. The harmful element phosphorus content was 1.56%, belonging to high phosphorus oolitic iron ore in western Hubei. In this study, segregation roasting and low intensity magnetic separation techniques were applied for upgrading iron and removing phosphorus. The ores, the chlorinating agent, and the reducing agent were mixed into the roasting furnace for segregation roasting. After being transferred from the weak magnetic minerals to the strong ones, the iron was recovered by low intensity magnetic separation. During segregation roasting, new ore phases, metallic iron (Fe), a small amount of ferrous oxide (Fe_3O_4), and ferrous oxide (FeO) could be observed. The results showed that the iron concentrate with the Fe content of 90.3%, the phosphorus content of 0.15%, and the iron recovery of 92.9% were obtained under the segregation roasting temperature of 1273 K, and the roasting time of 90 min, $CaCl_2$ (calcium chloride) 20%, $Ca(ClO)_2$ (calcium hypochlorite) 3%, the dosage of coke 20%, and low intensity magnetic separation field intensity 0.12 T.

Keywords: Low intensity magnetic separation; Ferrous oxide; Calcium chloride; Calcium hypochlorite

1. Introduction

The available reserves of high phosphorus oolitic iron ore [1] located in western Hubei are above 2 billion tons. The ore is considered as a chemical sedimentary iron ore, mainly composed of oolite and detritus. Specifically, oolite consists of oolite core (quartz or iron material), collophane girdle, hematite, and limonite girdles. Detritus is composed of fine-grained hematite and quartz feldspar. Generally, the iron concentrate consists of iron-rich oolite and some amount of hematite, with an iron grade of up to 55%. The phosphorus in the ore is mainly in the form of collophane, where the grain sizes of collophane girdle and monomer quartz are relatively small. The sizes of hematite and limonite in chilimonite are small and difficult to separate. Meanwhile, the low grade of aggregate iron makes it difficult for upgrading iron and removing phosphorus [2].

Conventionally, the direct utilization of oolitic iron ore without any pre-treatment [3-5] is not suitable, since its iron minerals, e.g., Fe_2O_3 , have weak magnetic characteristic which cannot

distinguish them from other gangue minerals. Meanwhile, due to its low Fe content, poor liberation, and complicated composition, satisfactory processing method with acceptable cost that can effectively utilize the ore is still lacking. However, it is feasible and sometimes necessary to modify the properties before direct beneficiation using various roasting methods [6-9], e.g., microwave roasting [10], salt roasting [11, 12], sulfur-fixed roasting [13] and sintering roasting modelling [14], and sulphating roasting [15-17]. Segregation roasting, a technology that applies to the theory of chlorination metallurgy, shows excellent performance in extracting valuable compositions and removing harmful ones from complex refractory iron ore. The methods applied to processing high phosphorus oolitic iron ore are mainly magnetic roasting [18], direct reduction [19], deep reduction, and suspension roasting, along with others [20, 21].

However, emphasize by segregation roasting on high phosphorus oolitic iron ore was rarely investigated. Hereby in this study, calcium chloride as a chlorinating reagent, calcium hypochlorite as an

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auxiliary, and coke as a reducing agent were applied to treat the sedimentary high phosphorus oolitic iron ore in western Hubei by segregation roasting. This work was aimed at gaining a better understanding of the detailed characteristics of the iron phase change and microstructures under various conditions, e.g., segregation roasting temperature and coke dosage. The final chemical composition of iron concentrate with magnetic separation was also examined. The present study aims at providing a basis on upgrading iron and removing phosphorus for the commercial utilization of such oolitic iron ores.

2. Experimental

2.1 Materials and reagents

The sedimentary high phosphorus oolitic iron ore in western Hubei was sampled and processed to the size of -1 mm. The hematite and limonite in the samples exist in the form of oolite, concentric and ellipsoidal structures, taking up 64.26% of total ores. The iron-bearing minerals in the oolite were mainly hematite, limonite, daphnite and collophane. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectrometer (EDS) images of oolite are shown in Fig. 1. SEM and EDS images of collophane are shown in Fig. 2. The main chemical composition of high phosphorous oolitic iron ores is shown in

Table 1, and the iron phase analysis results of sample are shown in Table 2.

The segregation reagents are presented in Table 3. All reagents are of analytical grade. The fineness of coke was -0.5 mm. The quality analysis of coke is listed in Table 4.

2.2 Material characterization

SDTQ600 differential thermal analyzer (TG-DTA) was used to characterize the thermal decomposition behaviors of the ore.

In this experimental study, advanced equipment, including X-ray photoelectron spectroscopy (XPS)-EDS, SEM-EDS, and electron probe EDS analyzer made by EDAX Inc, USA, were used to analyze the mineral morphology and mineral compositions.

2.3 Segregation roasting and low intensity magnetic separation

The ores were crushed to a certain particle size, then the chlorinating agent and reducing agent were mixed and put into the roasting furnace. These mixtures were heated to a certain temperature under the neutral or weak reducing atmosphere, where the chlorinating agent was used to form volatile metal chlorides. The products after roasting were ground to

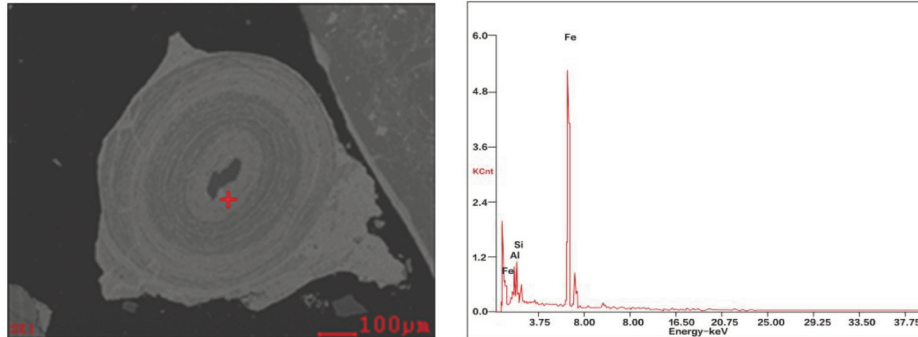


Figure 1. SEM and EDS images of oolite iron ore

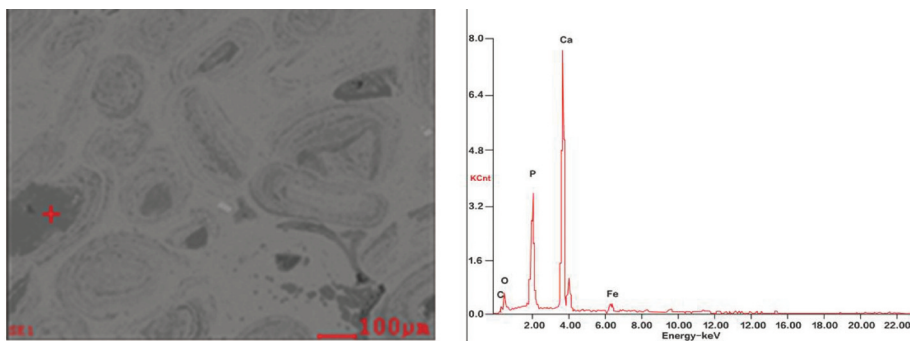


Figure 2. SEM and EDS images of collophane

a suitable fineness, then separated to produce magnetic products and non-magnetic ones through a XCGS-13 magnetic tube under certain magnetic field intensity. Then, the two products were filtered, dried, weighed, sampled, and tested for further evaluation based on the iron grade, phosphorus content and iron recovery in magnetic products.

3. Results and discussion

3.1 Thermal analysis

TG-DTA of high phosphorous oolitic iron ores were conducted from 35 to 900°C in air at a heating rate of 10°C/min, and the results are presented in Fig. 3. It can be seen that the total heat absorption was 313.8 J/g with the temperature increase from ambient temperature 35°C to 900.0°C. The absorption process was mainly divided into three stages, of which the second and the third stage are more pronounced.

In the first stage, the temperature increased from 169.5 to 307.3°C, and the endothermic peak was 265.9°C, the heat absorption occupies 6.612% of the

total heat absorption. It could be observed that a small amount of heat absorption occurred at this stage, and the process was relatively mild, which might be due to the moisture evaporation in the ore. For the second stage from 307.3°C to 588.8°C, the endothermic peak was 469.7°C, and the heat absorption was about 60% of the total heat absorption.

In the third stage, the temperature increased from 588.8°C to 721.9°C. The endothermic peak at this phase was 687.0°C, which was one of the important endothermic peaks. It was speculated that the hematite and limonite were converted into hematite and magnetite by absorbing heat [22]. At the temperature above 721.9°C, the reaction almost stopped and the amount of heat absorbed by the minerals turned to be small.

3.2 Effect of segregation roasting temperature

At the experimental condition of segregation roasting time of 90 min, the 15% dosage of calcium chloride the 5% dosage of calcium hypochlorite, the

Table 1. Main chemical composition of high phosphorous oolitic iron ores (mass, %)

TFe	P	S	SiO ₂	Al ₂ O ₃	CaO	K ₂ O	Na ₂ O	C	H ₂ O ⁺	V ₂ O ₅	MgO
41.7	1.56	0.05	13.58	6.56	5.63	0.4	0.071	0.86	2.89	0.091	1.26

Table 2. Iron phase analysis results of sample (mass, %)

TFe	Magnetite	Hematite and limonite	Pyrite	Siderite	Ferrosilite
41.7	0.056	42.39	0.11	0.69	0.24

Table 3. Roasting reagents used in segregation roasting

Reagent	Chemical formula	Purity (%)	Role
Calcium chloride	CaCl ₂	98	Chlorinating agent
Calcium hypochlorite	Ca(ClO) ₂	95	Auxiliaries

Table 4. The whole quality analysis of coke; M_{ad}: Air drying base content. A_d: ash content. V_{daf}: Volatile content. FC_d: Fixed Carbon content.

M _{ad}	A _d	V _{daf}	FC _d	Characteristic of char residue
0.56	2.19	1.13	94.22	2

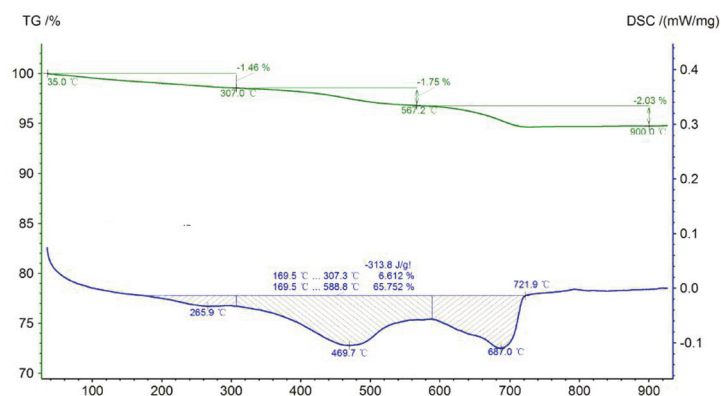


Figure 3. DTA and TG analysis of high phosphorous oolitic iron ores



15% dosage of coke, the SEM analysis, and the iron phase analysis results of different segregation roasting temperatures are shown in Fig. 4 and Fig. 5, respectively.

It was found from Fig. 4 and Fig. 5 that the segregation roasting temperature is one of the key factors that determine whether the chlorinating agent can be decomposed and whether the Fe can be chlorinated to form volatile metal chloride. With the increase of temperature, the content of magnetite decreases dramatically. The content of magnetite increases slightly after the temperature exceeds 1270 K. The change trend of metallic iron is negatively correlated with the change trend of magnetite. An increase in the segregation roasting temperature has a significant effect on the generation of Fe and magnetite within the segregation roasting process [23, 24]. When the segregation roasting temperature exceeded 1273 K, the proportion of Fe has a small range of fluctuations [25]. At 1173 K, the SEM spectra showed a dispersed granular shape which became more concentrated when

the temperature rose to 1223 K. At 1273 K, the dispersed granules disappeared and the maps showed large aggregates. Fig. 5 can clearly show that this is the time when the metal iron content is the largest, and the bulk morphology is mainly the embodiment of metal iron at this time. Meanwhile, the SEM images showed that the content of Fe was also in well degree at segregation roasting temperature of 1273 K. It can be concluded that the segregation roasting temperature of 1273 K can be a suitable condition, under which Fe 32.67% and magnetite 5.68% could be obtained.

3.3 Effect of coke dosage

SEM images of products using various coke dosages are shown in Fig. 6, and the iron phase analysis results are shown in Fig. 7. These results were obtained at conditions of the segregation roasting temperature of 1273 K, the roasting time of 90 min, the 15% dosage of calcium chloride, and the 5% dosage of calcium hypochlorite.

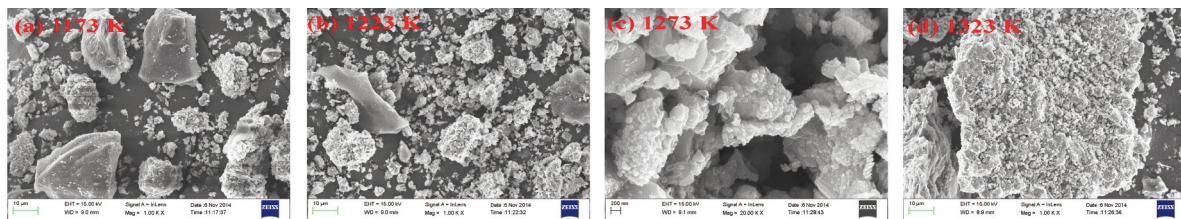


Figure 4. SEM images after roasting at various segregation roasting temperatures

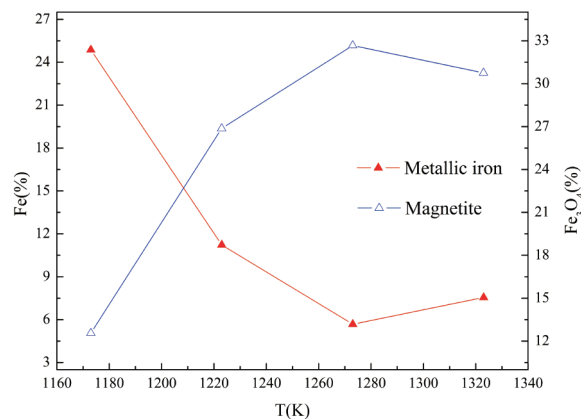


Figure 5. Iron phase after roasting at different segregation roasting temperatures (mass, %)

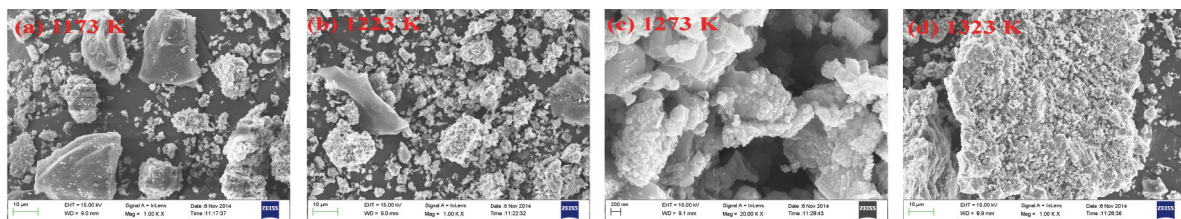
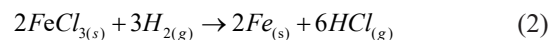
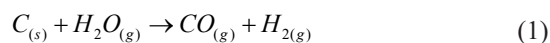


Figure 6. SEM imagines after roasting with various coke dosages

During the segregation roasting process (Fig. 6 and Fig. 7), the coke reacted with water vapor to form H_2 and CO , and volatile $FeCl_3$ was reduced to Fe particles by H_2 over the coke surface, as shown in Eq. (1) and Eq. (2).



As the coke dosage increased, the Fe content in the segregation roasting ore gradually increased. Under the condition that the coke dosage reached 20% or above, the increasing of Fe stopped. For Fe_3O_4 (Fig. 7, red line), when the coke dosage was

below 20%, the smaller coke dosage could lead to the increased amount of magnetite in the roasting ore. Taking costs into account, the excessive or insufficient coke dosage was not beneficial, and the 20% coke dosage could be a choice.

3.4 Effect of calcium chloride

At the condition of the segregation roasting temperature of 1273 K, the roasting time of 90 min, 5% calcium hypochlorite and 20% coke dosage, the SEM images and the iron phase analyses of calcium chloride are shown in Fig. 8 and Fig. 9.

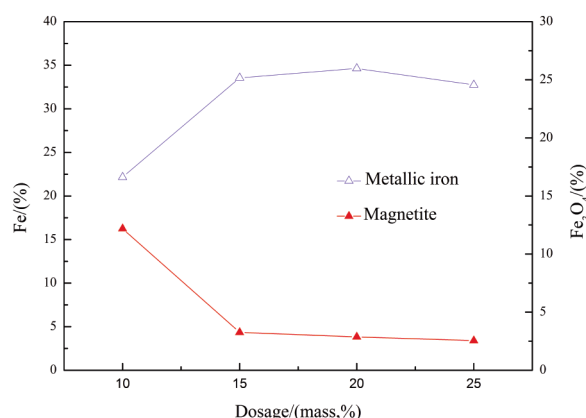


Figure 7. Iron phase results after roasting with various coke dosages

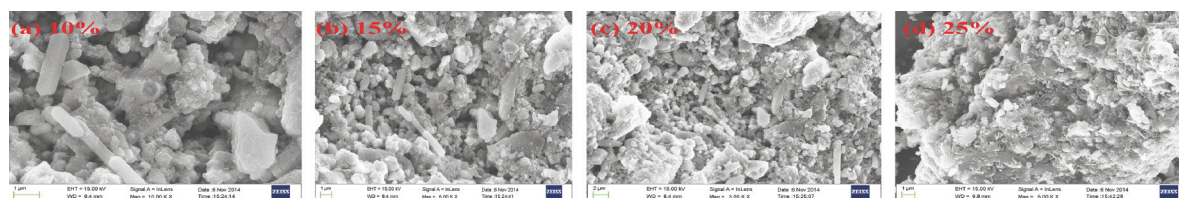


Figure 8. SEM images after roasting with different calcium chloride dosages

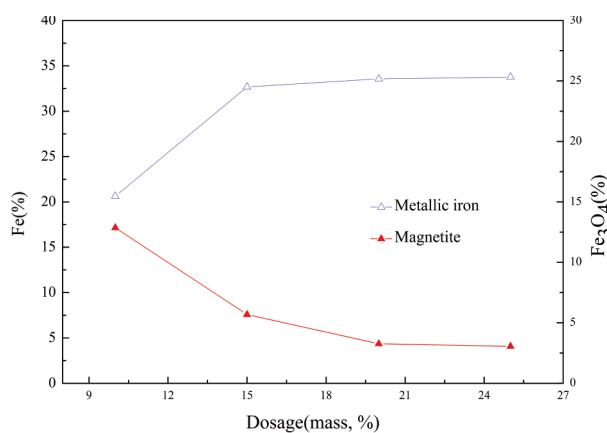
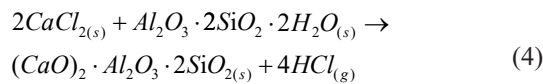
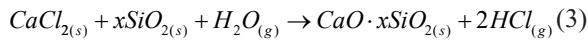


Figure 9. Iron phase results after roasting with different calcium chloride dosages



Firstly, Fe content increased significantly then decreased slightly (Fig. 8 and Fig. 9), with the increasing calcium chloride dosage in the segregation roasting process. Along with the gradually decreased generation amount of magnetite, which indicates that increasing the amount of calcium chloride in a certain range will promote the formation of iron, but after exceeding a certain limit, the amount of calcium chloride is increased, and the formation of iron is not changed much and tend to be stable. During the segregation roasting process, the calcium chloride reacted with the acidic oxides in the ore (e.g., quartz and aluminosilicate) and H_2O , forming hydrogen chloride, as shown in Eq. 3 and Eq.4 [26].

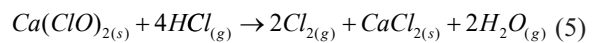


If calcium chloride reached an excessive content, the amount of hydrogen chloride generated in the system would increase. $FeCl_3$ could be generated by chlorination reaction between the Fe and hydrogen chloride. $FeCl_3$ was easily reduced by H_2 to metal particles and then adsorbed on the surface of the coke, due to its instability [27, 28]. However, if calcium chloride is not sufficient, the amount of hydrogen chloride generated would be reduced, which is not conducive to the formation of Fe in the segregation roasting process. It showed that the dosage of calcium chloride 20% could be a good choice.

3.5 Effect of calcium hypochlorite

SEM images and iron phase analysis results after segregation roasting with various calcium hypochlorite dosages are shown in Fig. 10 and Fig. 11, at the conditions of the segregation roasting temperature of 1273 K, the roasting time of 90 min, calcium chloride 20%, and coke 20%.

With the increasing of calcium hypochlorite dosage, a gradual increase of Fe content and a reduction of magnetite in the segregation roasting process (Fig. 10, Fig. 11) can be found. A chemical reaction between calcium hypochlorite (an auxiliary) and hydrogen chloride in the system occurred, forming calcium chloride and chlorine, as shown in Eq. 5 [29].



With the increased dosage of calcium hypochlorite, the chlorine generated by the reaction accelerated the chlorination reaction of Fe to a certain extent, contributing to the formation of $FeCl_2$. In addition, it can be found that excessive calcium hypochlorite might lead to excessive consumption of hydrogen chloride. On the other side, hydrogen chloride involved in the chlorination reaction during the segregation roasting process [30] is the most important chemical reaction.

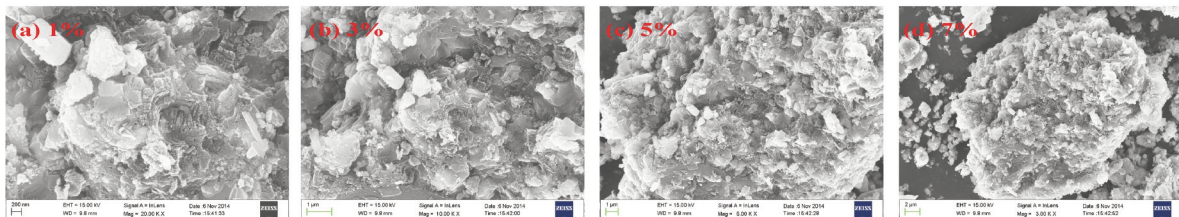


Figure 10. SEM images with various calcium hypochlorite dosages

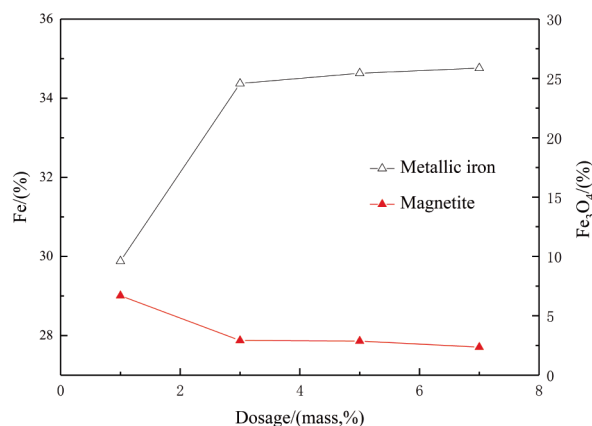


Figure 11. Iron phase analysis results with various calcium hypochlorite dosages

3.6 Effect of segregation roasting time

At the condition of segregation roasting temperature of 1273 K, calcium chloride 20%, calcium hypochlorite 3%, and coke of 20%, the SEM analysis and the iron phase analysis results of different segregation roasting time are shown in Fig. 12 and Fig. 13, respectively.

The extending segregation roasting time was effective for the formation of Fe (Fig. 12, Fig. 13), but when the roasting time was increased to 120 min, the increasing of Fe stopped and decreased slightly. The segregation roasting process had multiple chemical reactions occurring over time, e.g., the decomposition, the chlorination, and the reduction reactions; it also involved a complex phase change. A shorter roasting time will result in an insufficient and non-effective chemical reaction progress [30].

3.7 Low intensity magnetic separation

At condition of the roasting temperature of 1273 K, the roasting time of 90 min, calcium chloride 20%, calcium hypochlorite 3%, coke 20%, 36.18% Fe, and 1.89% magnetite were in the roasted ore (the total was 38.07%), which accounts for 91.29% of all the iron. It is known that the factors affecting low intensity magnetic separation for the roasted ore are mainly grinding fineness and magnetic field intensity. Under the above conditions involved in the segregation roasting process, with the low intensity magnetic

separation field intensity of 0.10T, the effect of grinding fineness of low intensity magnetic separation is presented in Table 5.

From the data in Table 5, with the increase of the grinding fineness, a regular increase of the iron grade and the reduction of the phosphorus content in the iron concentrate can be observed. Due to the increased grinding fineness, the difficulty in the liberation of mineral monomer increased. However, excessively higher grinding fineness may lead to the reduction of iron recovery in the iron concentrate, along with the significantly increased grinding costs. The effect of field intensity is shown in Fig.14, with the grinding fineness less than 0.019 mm accounting for 98%.

From Fig. 14, with the increased magnetic field intensity, the regular decrease of the iron grade and the increase of both the phosphorus content and the iron recovery can be observed. With the decreased magnetic field intensity, the materials will undergo a combined action by mechanical and magnetic forces during the separation process. Under the condition of the magnetic field intensity 0.12T, the iron grade of the iron concentrate decreased to 86.54%, the phosphorus content increased to 0.27%, while the iron recovery was 96.0%, which indicated that the increasing magnetic field intensity had a positive influence on the increase of overall iron recovery. But it was not beneficial to improve the iron grade of the concentrate.

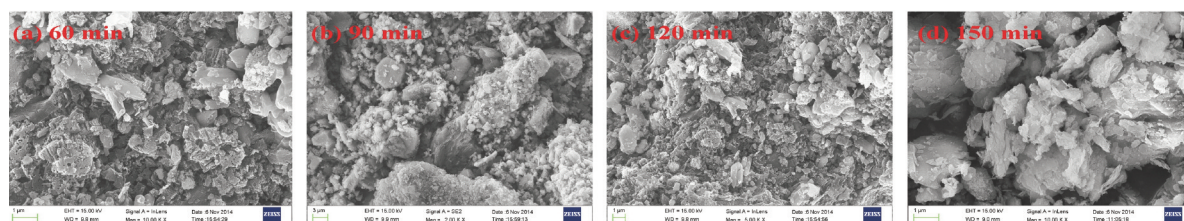


Figure 12. SEM images with various segregation roasting time

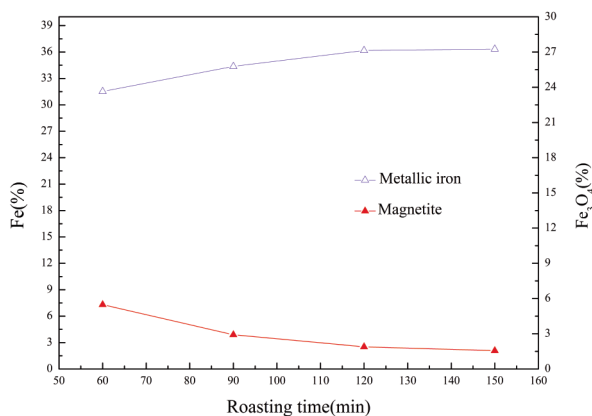


Figure 13. Iron phase results with various roasting time



3.8 Chemical composition of iron concentrate

The effects of CaCl₂ and Ca(ClO)₂ on upgrading iron and removing phosphorus of high phosphorus oolitic iron ore in western Hubei by segregation roasting were tested. The iron concentrate with Fe content of 90.3%, the phosphorus content of 0.15%, and the iron recovery of 93.0% were obtained under the segregation roasting temperature of 1273 K, the segregation roasting time of 90 min, the dosage of calcium chloride of 20%, the dosage of calcium hypochlorite of 3%, the dosage of coke of 20%, and the low intensity magnetic separation field intensity of 0.12 T. To further investigate the final quality of the iron concentrate, chemical

composition analysis, phase analysis, SEM, and electronic probe composition analysis were performed. The results are shown in Table 6 to Table 8, and Fig. 15.

It could be seen (Table 6, Table 7) that the new ore phases were dominated by Fe (86.91%), a small amount of FeO and Fe₃O₄. The iron phase changed compared with the iron phase of the crude ore, which changed from limonite (Fe₂O₃, Fe₂O₃·nH₂O) to a new iron phase dominated by metallic iron. Based on the SEM images and electronic probe composition analysis in Fig. 15 and Table 8, remarkable changes on the iron phases of high phosphorus oolitic iron ore after segregation roasting process can be observed.

Table 5. Effect of grinding fineness on low intensity magnetic separation (mass, %)

Grinding fineness	Products	Productivity	Grade		Recovery	
			Fe	P	Fe	P
< 0.045mm occupying 98%	Iron concentrate	53.4	72.7	0.32	93	10.8
	Tailings	46.6	6.3	3.03	7	89.2
	Total	100	41.7	1.58	100	100
< 0.038mm occupying 98%	Iron concentrate	44.8	85.6	0.28	92	7.9
	Tailings	55.2	6.1	2.65	8	92.1
	Total	100	41.7	1.59	100	100
< 0.019mm occupying 98%	Iron concentrate	38.4	92.4	0.12	85	2.9
	Tailings	61.6	10.2	2.47	15	97.1
	Total	100	41.7	1.57	100	100
< 0.010mm occupying 98%	Iron concentrate	35.4	95.2	0.08	80.7	1.8
	Tailings	64.6	12.5	2.4	19.3	98.2
	Total	100	41.7	1.58	100	100

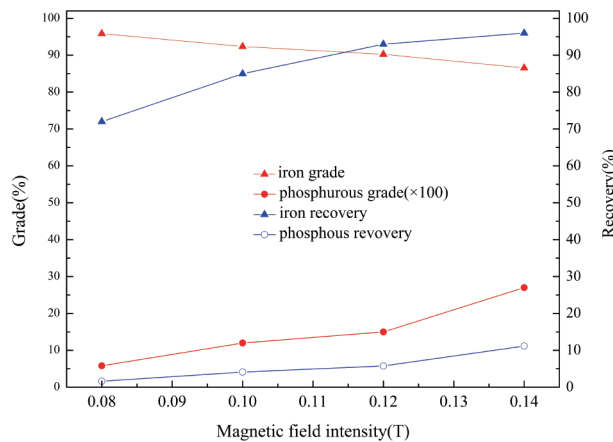


Figure 14. Effect of field intensity on low intensity magnetic separation

Table 6. Main chemical compositions of iron concentrate (mass, %)

TFe	P	S	SiO ₂	Al ₂ O ₃	CaO	K ₂ O	Na ₂ O	MgO
90.25	0.15	0.02	4.12	0.06	1.25	0.08	0.02	0.05

Table 7. Iron phase results of iron concentrate (mass, %)

TFe	Fe	Ferrous oxide	Ferroferric oxide
90.26	86.91	1.22	2.13



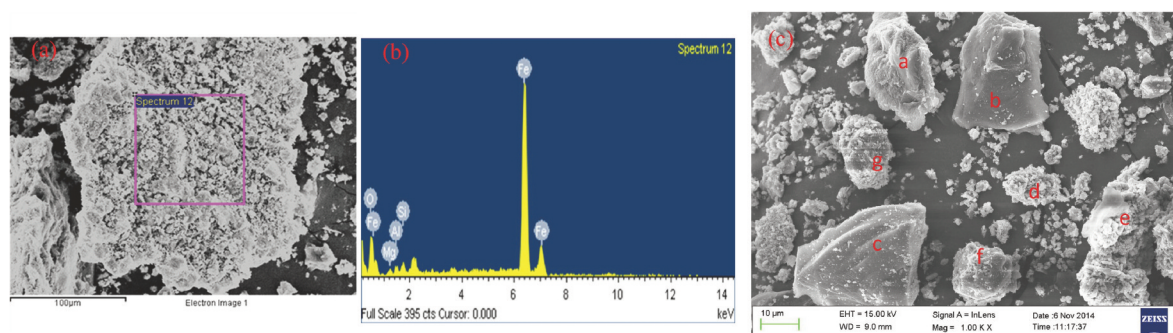


Figure 15. SEM (a)-EDX image (b), and SEM image (c) (10 μm scale) of iron concentrate

Table 8. Electronic probe composition analysis results of iron concentrate (mass, %)

Composition		Fe	Fe ₃ O ₄	FeO	P ₂ O ₅	SiO ₂	CaO	Total
Position	a	91.6	1.65	0.95	0.36	4.33	1.11	100
	b	93.01	1.87	0.87	0.28	2.89	1.08	100
	c	92.42	2.22	1.06	0.31	2.67	1.32	100
	d	89.77	1.08	1.54	0.37	6.13	1.11	100
	e	90.72	0.96	1.73	0.28	5.33	0.98	100
	g	91.97	1.06	0.89	0.33	4.87	0.88	100
Average		91.59	1.47	1.17	0.32	4.37	1.08	100

4. Conclusions

High phosphorus oolitic iron from western Hubei, China, was investigated using segregation roasting and low intensity magnetic separation method to upgrade iron and remove phosphorus. The iron-bearing ores in the oolite are mainly hematite, limonite, chlorite and collophanite. The detailed behaviors of the iron phase change and microstructures during the segregation roasting were examined at various conditions, including the effect of the roasting temperature, the effects of various roasting reagents, e.g., coke, calcium chloride, and calcium hypochlorite. The low intensity magnetic separation after the segregation roasting showed that the iron content of 90.3%, the phosphorus content of 0.15%, and the iron recovery of 93.0% were obtained for the iron concentrate under the segregation roasting temperature of 1273 K, the segregation roasting time of 90 min, the dosage of calcium chloride of 20%, the dosage of calcium hypochlorite of 3%, the dosage of coke of 20%, the low intensity magnetic separation field intensity of 0.12 T and 98% grinding fineness of less than 0.019 mm. The chemical composition analysis, the iron phase analysis, SEM, the electron probe analysis and EDS of the iron concentrate, showed that new ore phases were dominated by Fe (86.9%), and small quantities of FeO and Fe₃O₄ from the original hematite and limonite (i.e., Fe₂O₃, Fe₂O₃·nH₂O) were generated.

Acknowledgments

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OBOGAČIVANJE ŽELEZA I UKLANJANJE FOSFORA IZ OOLITSKE RUDE ŽELEZA SA VISOKIM SADRŽAJEM FOSFORA SEGREGACIONIM PRŽENJEM SA $\text{CaCl}_2 + \text{Ca}(\text{ClO})_2$

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Apstrakt

Ruda železa u formi oolita uglavnom se sastojala od hematita, limonita, dafnita, i kolofana. Sadržaj štetnog elementa fosfora bio je 1.56%, iz oolitske rude sa visokim sadržajem fosfora iz zapadnog Hubeja. U ovoj studiji primenjene su tehnike segregacionog prženja i magnetne separacije niskog intenziteta da bi se uklonio fosfor i obogatilo železo. Ruda, sredstvo za hlorisanje, i sredstvo za redukciju su pomešani u peći za prženje da bi se izvršilo segregaciono prženje. Nakon što su slabo magnetični minerali prešli u jako magnetične, dobijeno je železo pomoću magnetne separacije niskog intenziteta. Tokom segregacionog prženja mogu se uočiti nove faze rude, železo (Fe), male količine železo (II, III) oksida (Fe_3O_4) i železo (II) oksida (FeO). Rezultati su pokazali da su koncentrat železa sa sadržajem železa od 90.3%, sadržajem fosfora od 0.15%, i dobijanje gvožđa od 92.9% dobijeni na temperaturi segregacionog prženja od 1273 K, trajanjem prženja od 90 minuta, CaCl_2 (kalcijum hlorid) od 20%, $\text{Ca}(\text{ClO})_2$ (kalcijum hipohlorit) od 3%, doziranje koksa od 20%, i intenzitetom magnetne separacije niskog intenziteta od 0.12 T.

Ključne reči: Magnetna separacija niskog intenziteta; Železo (II,III) oksid; Kalcijum hlorid; Kalcijum hipohlorit

