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Increase of iron concentration and reduction of impurities in red mud from the Wenshan area of Yunnan Province by segregation roasting-low intensity magnetic separation

Red mud is a solid waste produced during alumina extraction from bauxite. In this study, we pretreated red mud by segregation roasting to convert the iron from weak magnetic minerals to ferromagnetic minerals and then subjected the material to grinding and low intensity magnetic separation to obtain iron concentration. The effects of the chlorinating agent type, chlorinating agent dosage, reducing agent dosage, temperature, reaction time, additive, grinding fineness and magnetic field strength on the iron concentrate grade and recovery rate are studied. At a red mud/KCl/coke/Na_2SO_4 mass ratio of 100:15:15:10, segregation roasting at 1100°C for 60 min, the roasted ore is ground to about 95 wt.% passing 0.045 mm and a magnetic field strength of 0.22 T, the final iron concentrate grade is 78.29% and the recovery is 84.08%, which is an efficient improvement of the valuable metal iron in red mud. The X-Ray Diffraction, Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy analysis of the iron concentrate shows that before the segregation roasting, the precious metal iron in the red mud is mainly in the form of Fe_2O_3. After the segregation roasting, the iron is transformed into a new iron mineral phase mainly composed of metal Fe and Fe_3O_4. The main impurities in the iron concentrate are calcium, silicon and aluminum.

Keywords: Red mud, separation roasting, magnetic separation, metallic iron

1.0 Introduction

Red mud is a residue generated during alumina production. It is red because it contains a lot of Fe_2O_3[1]. According to the Bayer process and the composition of bauxite, approximately 1.0–1.5 tonnes of red mud is produced per 1 tonne of alumina produced [2]. At present, China's emissions of red mud exceed 30 million tonnes/year, and the accumulated stock of red mud exceeds 400 million tonnes. The treatment methods of red mud generally include building materials [3-4], environmentally friendly raw materials [5-7], auxiliary additives [8-9] and filling materials [10], but the consumption is minimal. Red mud is rich in a variety of valuable elements, including Fe, Al, Sc and Ti; it is a useful secondary polyvalent metal resource. Therefore, the overall utilization of alumina red mud has become an urgent problem that needs to be solved. The raw material used in this study is red mud produced during the production of Al_2O_3 by a company in Wenshan, Yunnan, China. It has a high content of iron and scandium. Red mud has potential economic value owing to the high content of rare earth elements and scandium. Utilization of the elements in red mud by hydrometallurgy and mineral processing not only improves the treatment and utilization of red mud but also effectively reduces the environmental pollution and has a significant socio-economic and ecological value. Because iron has a significant influence on the recovery of uncommon and rare elements in red mud, the pre-recovery of iron is very important for further extraction and separation of valuable components such as scandium and titanium in subsequent processes. To this end, we investigated the recovery of iron from red mud produced in the Wenshan area of Yunnan, China by a segregation roasting-low intensity magnetic separation method. The developed process provides a new avenue for the comprehensive utilization of red mud resources.

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2.0 Experimental

2.1. Sample Characteristics

The test ore sample is derived from red mud produced through the Bayer process by Yunnan Wenshan Aluminum Co., Ltd. The red mud retrieved from the mine tailings pond has a high water content. Therefore, sun drying, baking and size reduction are required; the particle size of the raw materials is below 0.15 mm. The samples are subjected to chemical multielement analysis, XRD analysis, particle size composition analysis and red mud micromorphology analysis. The analysis results are shown in Tables 1 and 2 and Figs. 1 and 2.

Table 1. Chemical multielement analysis of red mud (%)

Composition	Fe_2O_3	Al_2O_3	SiO_2	Na_2O	CaO	TiO_2
Content	28.36	17.17	13.70	6.86	16.33	4.40
Composition	MgO	S	P	K_2O	ZrO_2	MnO
Content	0.47	0.37	0.125	0.181	0.227	0.227

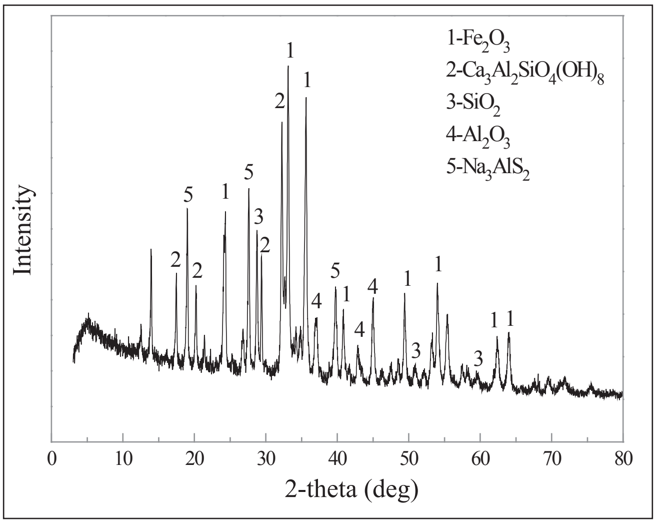


Fig.1: XRD phase analysis of red mud

Table 2. Iron phase analysis of red mud (%)

Composition	TFe	Magnetic iron	Hematite-limonite	Siderite	Pyrite	Iron silicate
Content	19.86	0.02	16.99	0.15	0.03	2.68
Occupancy	100.00	0.10	85.55	0.76	0.11	13.48

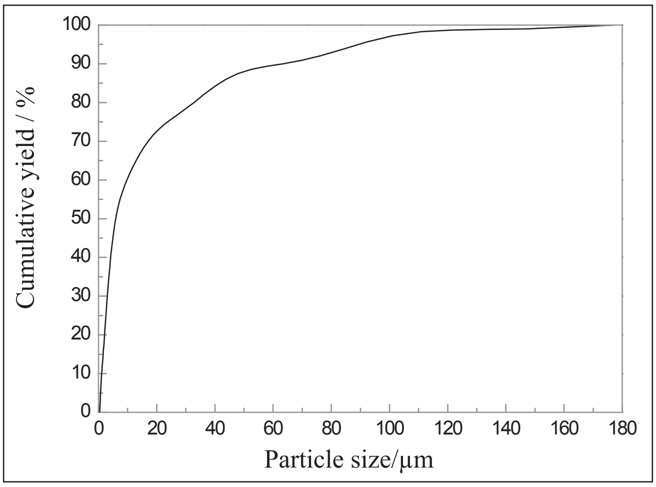


Fig.2: Size analysis of red mud

As indicated in Table 1 and Fig.1, the metals that can be recycled from the red mud are iron, titanium and scandium, and the main gangue mineral components are Al_2O3, SiO_2, CaO and Na_2O. The diffraction spectrum of the red mud ore has many peaks, and the modes of occurrence of the phase are complex, mainly composed of hematite, water, calcium, aluminum, garnet and quartz.

See supplementary Table 2, most of the iron in the red mud ore is in the form of hematite-limonite and a small amount of iron is present in silicates, carbonates and sulfides. Considering the process of segregation roasting-low intensity magnetic separation for iron minerals dominated by hematite-limonite in red mud, the iron is converted into a new ore phase mainly composed of metallic iron and magnetite. See supplementary Fig.2, 90 wt.% of the red mud passes through 0.070mm. One of the typical characteristics of the Bayer process red mud is the extremely fine size. Therefore, roasting is used because it is difficult to recycle the iron element through a conventional sorting method. The occurrence of iron minerals in red mud is changed by roasting, and the enrichment and recovery of iron minerals are obtained through weak magnetic separation.

2.2. Methods

The red mud, chlorinating agent, reducing agent and additives are uniformly mixed in a certain proportion and the appropriate amount of water is added to form a pellet, which is placed in a corundum crucible. The corundum crucible is located in a box resistor furnace, in which the segregation roasting is performed at a designed temperature and duration. The segregation roasting products are quenched with water, dried and ground to about 90.00 wt.% passing 0.045mm and then magnetically separated. Finally, chemical analysis of the magnetically selected concentrates and tailings is performed by XRD analysis (Cu, X Pert pro, Panaco, Netherlands), Scanning Electron Microscopy (S440, Leica Cambridge Ltd, Germany) equipped with an Energy Dispersive X-ray Spectroscopy (EDS) detector (UItra55, Carl zeiss NTS GmbH, Germany) and the use of a SX 2-6-14 Box Resistance Furnace, a XCGS-50 Davies Magnetic Tube, Conical Ball Mill and Grading Sieve. The potassium chloride (KCl), sodium chloride (NaCl), calcium chloride ($CaCl_2$),

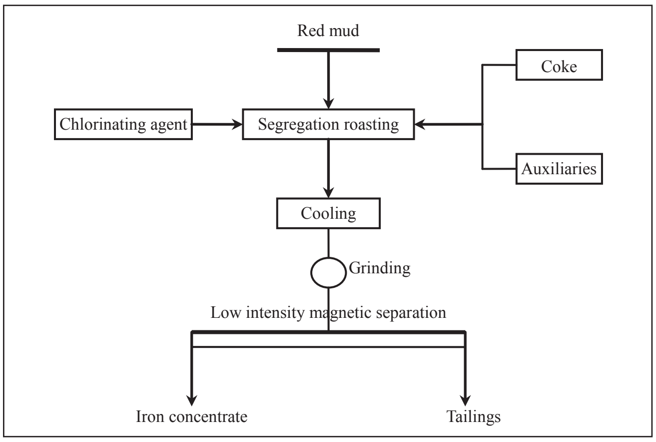


Fig.3: Segregation roasting-low intensity magnetic separation test flow sheet

magnesium chloride ($MgCl_2$), calcium oxide (CaO), calcium fluoride (CaF_2), Na_2CO_3 and Na_2SO_4 used in the study are of chemical grade. Coke is used as reductant. A flowchart of the process is shown in Fig.3.

3.0 Results and discussion

3.1 Effect of chlorinating agent types

We investigated the influence of the type of chlorinating agent on the segregation roasting process under the following conditions: red mud/chlorinating agent/reducing agent (coke) mass ratio of 100:15:15, reaction temperature of 1000°C, reaction time of 90 min, magnetic field strength of 0.22 T and grinding fineness of less than 0.048 mm (95 wt.%) (Table 3).

Table 3: Effect of the type of chlorination agent on the test results (%)

Chlorinating agent	Product	Yield	TFe	Iron recovery
Potassium chloride	Concentrate	20.64	62.57	72.32
	Tailings	79.36	6.23	27.68
	Totals	100.00	17.86	100.00
Barium chloride	Concentrate	25.62	45.92	60.98
	Tailings	74.38	10.12	39.02
	Totals	100.00	19.29	100.00
Calcium chloride	Concentrate	19.32	53.78	57.21
	Tailings	80.68	9.63	42.79
	Totals	100.00	18.16	100.00
Magnesium chloride	Concentrate	13.31	48.76	38.49
	Tailings	86.69	11.96	61.51
	Totals	100.00	16.77	100.00
Sodium chloride	Concentrate	21.93	55.83	63.15
	Tailings	78.07	9.15	26.85
	Totals	100.00	19.39	100.00

The chlorinating agent is a critical component in the segregation roasting process. Under high-temperature conditions, the solid chlorinating agent reacted with the water vapour and silica or aluminosilicate in the metal to decompose it and produce chlorine gas and hydrogen chloride. Hydrogen chloride gas reacted with the iron oxide in the ore to chlorinate and volatilized the iron oxide out of the metal. As shown in Table 3, different chlorination agents have different effects on the results of the segregation roasting. When potassium chloride is used as a chlorinating agent, the iron concentrate grade and recovery are the highest, 62.57% and 72.73% respectively. Therefore, potassium chloride is considered to be the most suitable chlorinating agent.

3.2 Effect of the mass fraction of the chlorinating agent

The influence of the mass fraction of the chlorinating agent on the segregation roasting process is investigated under the following conditions: red mud/calcium oxide/reducing agent (coke) mass ratio of 100:10:15, reaction temperature of 1000°C, reaction time of 90 min, magnetic field strength of 0.22 T and a grinding fineness of less than 0.048 mm (95%) (Fig.4).

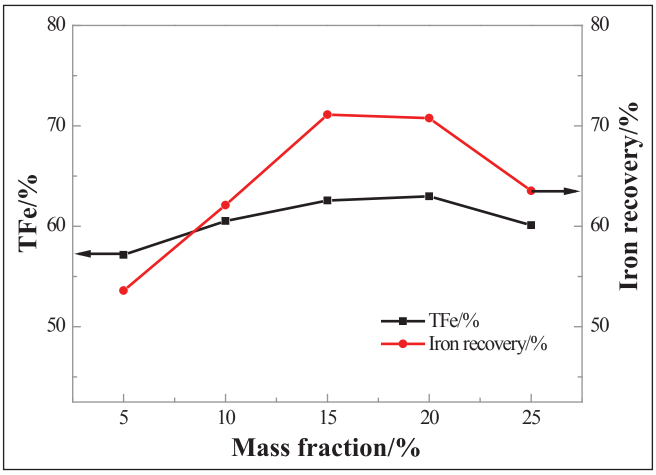


Fig 4. Effect of the mass fraction of chlorination agent on the test results

As shown in Fig.4, as the mass fraction of the potassium chloride increased, the iron grade and recovery rate first increases and then decreases. When the mass fraction of potassium chloride is 20%, the maximum value of the iron grade is 62.98%; when the mass fraction of potassium chloride is 15%, the maximum value of the iron recovery is 72.32%. This occurs because an appropriate increase in the amount of potassium chloride is beneficial for the increase in the amount of hydrogen chloride and chlorine generated during the chlorination and segregation process, which correspondingly increases the amount of volatile metal chloride formed [11,12]. However, if the amount of potassium chloride is excessive, part of the chlorinating agents will react with other metals to form the corresponding metal chloride, which affects the iron separation index. Overall, a potassium chloride mass fraction of 15% provides the best results.

3.3 Effect of the mass fraction of coke

We investigated the influence of the mass fraction of coke on the segregation process under the following conditions: red mud/calcium oxide/potassium chloride mass ratio of 100:10:15, reaction temperature of 1000°C, a reaction time

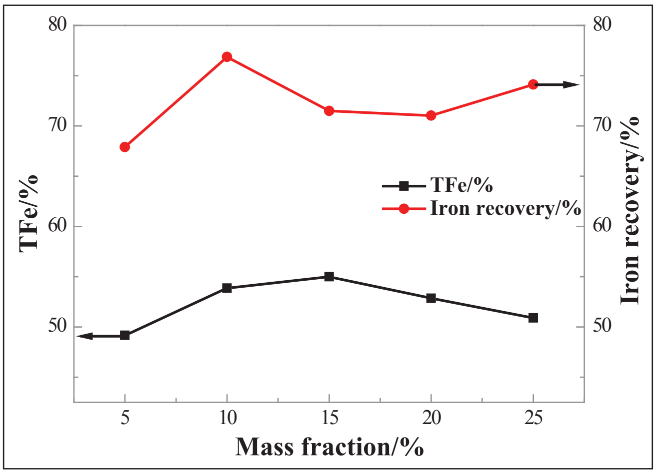


Fig 5. Effect of the mass fraction of coke on the test results

of 90 min, a magnetic field strength of 0.22 T, and grinding fineness of less than 0.048 mm (95 wt.%) (Fig.5).

As shown in Fig 5, as the mass fraction of coke increases, the iron grade and recovery rate first increases and then decreases. When the mass fraction of coke is 15%, the maximum amount of the iron grade is 55.00%; when the mass fraction of potassium chloride is 10%, the maximum value of iron recovery is 76.86%. This is because as a reducing agent, coke plays a dual role of providing a reducing atmosphere and providing a chloride adsorption carrier during the segregation roasting process [13]. An excess amount of reducing agent will cause the reaction between C and H_2O to generate H_2 and CO, which will cause the in-situ reduction of iron oxide, which will affect the segregation roasting. In contrast, the amount of reducing agent is too small to effectively reduce and adsorb iron chloride, which also affects the iron concentrate sorting index [14-17]. Considering these results, a coke mass fraction of 15% is determined to be the most appropriate.

3.4 Effect of segregation roasting time

We investigated the influence of the segregation roasting time on the segregation process under the following conditions: red mud/coke/potassium chloride mass ratio of 100:15:15, reaction temperature of 1000°C, magnetic field strength of 0.22 T, grinding fineness of less than 0.048 mm (95 wt.%) (Fig.6).

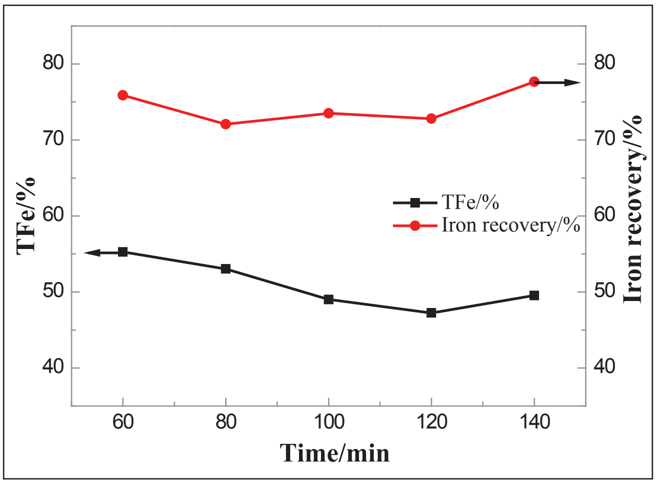


Fig.6: Effect of segregation roasting time on the test results

As shown in Fig.6, the grade of iron concentrate reaches a maximum value of 55.27% at 60 min. When the roasting time is extended, the recovery rate of iron slightly decreases and the grade of the iron concentrate gradually decreases. This is because as the segregation roasting time increased, the reaction is relatively more complete but some side reactions also occurs, forming some compound inclusions. This leads to the magnetic material being easily entrained by nonmagnetic and weak magnetic substances during magnetic separation, resulting in a decrease in the iron concentrate grade. The recovery is slightly increased. Considering these results, a segregation roasting time of 60 min is chosen.

3.5 Effect of segregation roasting temperature

We investigated the influence of the segregation roasting temperature under the following conditions: red mud/coke/potassium chloride mass ratio of 100:15:15, reaction time of 60 min, magnetic field strength of 0.22 T and grinding fineness of less than 0.048 mm (95 wt.%) (Fig.7).

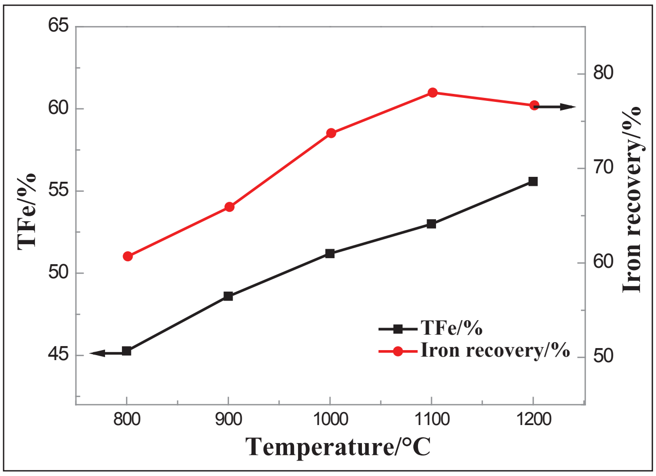


Fig.7: Effect of segregation roasting temperature on the test results

As shown in Fig.7, the grade of the iron concentrate gradually increases with the reaction temperature and the recovery rate of iron first increases and then decreases. This is because the increase in temperature is beneficial for the formation of hydrogen chloride and at the same time increases the reaction rate, making the reaction more complete. From the temperature reaches 1000°C, the recovery of iron is significantly improved. The reason for this is that the reduction reaction is the main reaction that occurs below 1000°C, and the in-situ reduction of iron oxide led to a lower recovery rate [18-20]. When the temperature is higher than 1000°C, the chlorination reaction is preferred and the chlorinated product diffused to the carbon surface and is reduced. When the temperature is 1100°C, the grade of the iron concentrate and the recovery rate of iron are optimal. At this time, the iron concentrate grade is 53.00% and the iron recovery rate is 78.02%. Therefore, a segregation roasting temperature of 1100°C is the most appropriate.

3.6 Effect of additive types

We investigated the influence of additive types on the segregation roasting process under the following conditions: red mud/coke/potassium chloride mass ratio of 100:15:15, reaction temperature of 1100°C, reaction time of 60 min, magnetic field strength of 0.22 T and grinding fineness of less than 0.048 mm (95 wt.%) (Table 4).

Table 4: Effect of the type of additive on the test results(%)

Additive	Product	Yield	TFe	Iron recovery
CaO	Concentrate	24.79	56.10	72.94
	Tailings	75.21	6.86	27.06
	Totals	100.00	19.07	100.00
CaF_2	Concentrate	25.20	51.77	73.15
	Tailings	74.80	6.40	26.85
	Totals	100.00	17.83	100.00
Na_2CO_3	Concentrate	26.36	58.66	76.72
	Tailings	73.64	6.37	23.28
	Totals	100.00	20.15	100.00
Na_2SO_4	Concentrate	22.58	63.19	72.30
	Tailings	77.42	7.02	27.70
	Totals	100.00	19.70	100.00

As shown in Table 4, of the four additives used, Na_2CO_3 and Na_2SO_4 has the best effect. This is explained by the fact that sodium sulfate reacts with most aluminum-silicate minerals to form nonmagnetic sodium aluminosilicate, which reduces the reaction of aluminum-silicate minerals with FeO and improves the activity of FeO [21,22]. At the same time, sodium sulfate reacts with aluminum and silicon minerals to produce SO_3, and the reaction of SO_3 with the chlorinating agent promotes the formation of hydrogen chloride, thereby strengthening the chlorination reaction of iron oxide. Considering these results, Na_2SO_4 is chosen as the additive.

3.7 Effect of the mass fraction of additive

We investigated the influence of the mass fraction of the additive on the segregation roasting process under the following conditions: red mud/coke/potassium chloride mass ratio of 100:15:15, reaction temperature of 1100°C, reaction time of 60 min, magnetic field strength of 0.22 T, grinding fineness of less than 0.048 mm (95 wt.%) (Fig.8).

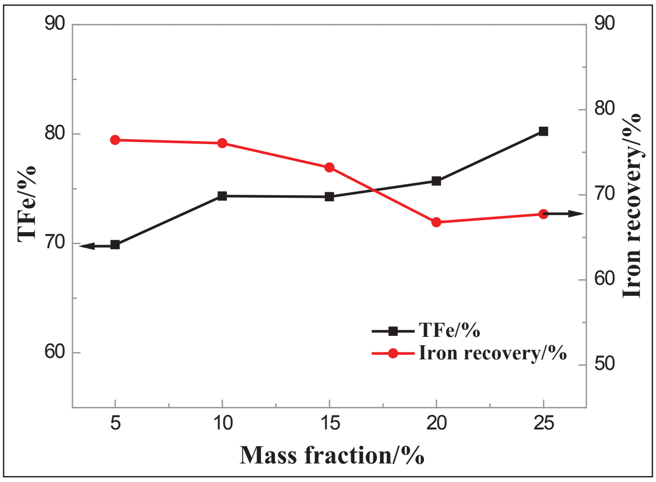


Fig.8: Effect of the mass fraction of additive on the test results

As shown in Fig.8, as the amount of Na_2SO_4 increases the grade of iron concentrate gradually increases, and the recovery rate of iron gradually decreases. This is explained by the fact that both the additive and the chlorinating agent reacted with the aluminum-silicate minerals in the red mud. If the amount of additive is excessive, the amount of hydrogen chloride gas is inordinate. An excessive amount of hydrogen chloride converts the FeO and the reduced metallic iron into $FeCl_3$, and when the carbon content of the coke is exhausted, $FeCl_3$ remains so the grade of the iron concentrate increases, whereas the recovery of iron is gradually decreased. Considering both the grade and recovery, a Na_2SO_4 mass fraction of 10% is considered optimal.

3.8 Influence test of low intensity magnetic separation conditions

The optimum conditions for the segregation roasting of red mud are: reaction temperature of 1100°C, reaction time of 60 min and mass ratio of red mud/sodium chloride/reducing agent (coke)/sodium sulfate of 100:15:15:10. To further improve the separation index of the iron concentrate, the effect of the low intensity magnetic separation conditions on the segregation roasting products is investigated. The principal factors that affect the iron grade and recovery during low intensity magnetic separation are grinding fineness and magnetic field intensity [23-25]. First, the effect of the grinding fineness on the iron concentrate index is tested and the results are shown in Table 5.

Table 5: Effect of grinding fineness on the test results/%

Grinding fineness/mm	Product	Yield	TFe	Iron recovery
-0.075mm occupying 95%	Concentrate	39.55	42.35	68.13
	Tailings	60.45	8.71	21.87
	Totals	100.00	18.99	100.00
-0.048 mm occupying 95%	Concentrate	23.75	70.06	78.77
	Tailings	76.25	5.65	21.23
	Totals	100.00	20.50	100.00
-0.045 mm occupying 95%	Concentrate	22.64	78.29	84.08
	Tailings	77.36	4.34	15.92
	Totals	100.00	21.08	100.00
-0.038 mm occupying 95%	Concentrate	22.35	74.62	80.20
	Tailings	77.65	5.29	19.80
	Totals	100.00	20.79	100.00

As shown in Table 5, the degree of monomer dissociation of minerals increases with the fineness of grinding, which is conducive to improving the grade of the magnetically selected iron concentrate. However, when the fineness of the grinding is too high, some of minerals obtained by the low intensity magnetic separation during the magnetic separation process can easily mix with strong magnetic substances in the magnetic separation tube and some of the ferromagnetic materials are removed by flushing water owing to the weakened magnetic force, thereby reducing the grade of the iron concentrate and the recovery rate of iron. Therefore, the optimum grinding fineness is −0.045mm (95 wt.%).

The effect of the magnetic field intensity on the properties of the iron concentrate is investigated at a grinding fineness of −0.045mm (95 wt.%); the other conditions remained unchanged and the results are presented in Fig 9.

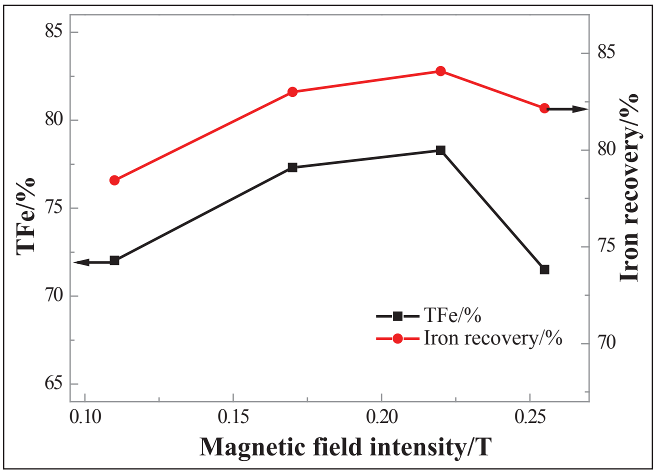


Fig.9

As shown in Fig.9, as the magnetic field intensity increases the grade of the iron concentrate, first increases and then decreases, whereas the recovery of iron gradually increases. This is because as the magnetic field intensity increases, more iron ore ligands intergrowth enter the concentrate, resulting in a decrease of the concentrate grade and an increase in the concentrate recovery [26]. Therefore, a magnetic separation field intensity of 0.22 T is the optimum, at which the grade and recovery of the iron concentrate are 78.29% and 84.08%, respectively.

4.0 Discussion

By considering the influence of the segregation roasting process conditions on the separation of iron, the optimum conditions for the segregation roasting-low intensity magnetic separation are: reaction temperature of 1100°C, reaction time of 60 min, mass ratio of red mud/sodium chloride/reducing agent (coke)/sodium sulfate of 100:15:15:10, magnetic field strength of 0.22 T, and grinding fineness of less than 0.048 mm (95 wt.%). Finally, an iron concentrate sorting index with an iron grade of 78.29% and iron recovery rate of 84.08% is obtained. To further study the le metallic iron in red mud before and after smineral phase transformation of valuable segregation roasting, the magnetic separation concentrate is analyzed by chemical analysis, XRD, SEM and EDS analysis. The primary chemical composition analysis of the iron concentrate is shown in Table 6, the XRD phase analysis is shown in Fig.10 and the SEM-EDS results of red mud before and after the segregation roasting-low intensity magnetic separation is shown in Fig.11 and Table 7.

Table 6. Results of chemical multielement analysis of iron concentrate (%)

Composition	Fe	Al_2O_3	SiO_2	Na_2O	SO_3	TiO_2
Content	75.99	7.37	6.70	4.06	1.90	1.62
Composition	K_2O	MgO	Cr_2O_3	P_2O_5	MnO	Cl-
Content	0.65	0.49	0.28	0.25	0.13	0.10

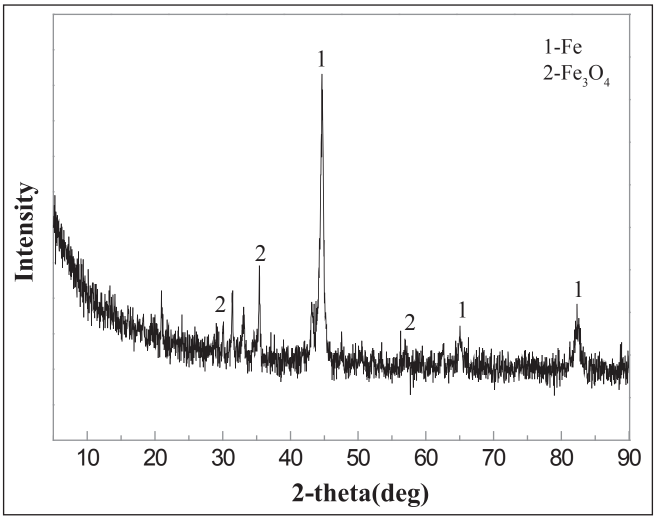
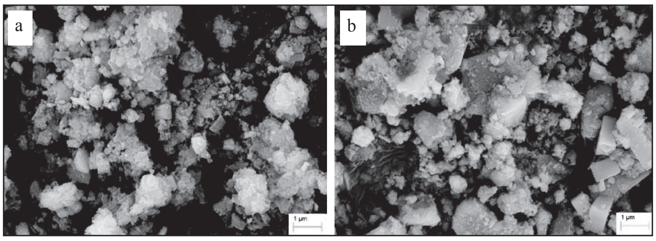


Fig.10: XRD phase analysis results of the iron concentrate



(a) Red mud; (b) Iron concentrate

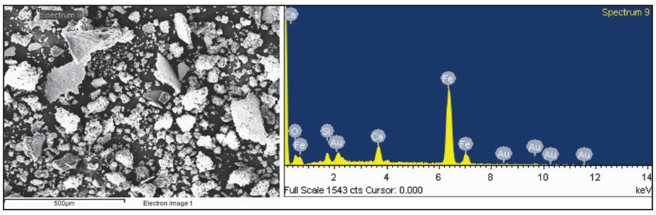


Fig.11: The SEM-EDS results of red mud before and after the segregation roasting–low intensity magnetic separation

Table 7. Results of energy spectrum microanalysis of the iron concentrate

Number	Mass fraction/%				
	O	Ca	Si	Fe	Al
1	8.57	2.05	1.56	84.89	2.03
2	9.08	1.25	2.56	82.43	2.05
3	4.65	5.72	2.18	83.49	3.21
4	8.20	3.66	2.52	81.59	2.55
5	6.57	2.89	1.95	80.37	3.66
Average	7.41	3.12	2.15	82.55	2.70

As shown in Figs. 10 and 11 and in Tables 6 and 7, after the segregation roasting, the iron in the red mud is transformed from Fe_2O_3 to a new iron mineral phase mainly composed of metal Fe and Fe_3O_4. The specific magnetic susceptibility of metallic iron (Fe) and magnetite (Fe_3O_4) is large and the iron ore concentrate is obtained by low intensity magnetic separation after segregation roasting. The composition analysis by EDS shows that the main impurity elements in the iron concentrate are calcium, silicon and aluminum. Owing to the higher fineness of the grinding, the impurity elements in the low intensity magnetic separation process are prone to entrainment and entered the magnetic product and then affected the iron grade of the concentrate.

5.0 Conclusions

(1) The principal chemical components of red mud from the Wenshan area of Yunnan are Fe_2O_3, Al_2O_3, SiO_2, Na_2O and CaO. The majority of the iron in the ore existed as hematite-limonite and a small amount of iron existed as silicates, carbonates and sulfides. The red mud had a fine grain size and the content below 0.070 mm reached 90 %. The conventional method used for sorting and recovery of the metal iron is difficult to realize.

(2) The segregation roasting-low intensity magnetic separation process is used to investigate different segregation roasting conditions and low intensity magnetic separation conditions for the separation of iron. The results showed that the optimum conditions are: reaction temperature of 1100°C, reaction time of 60 min, mass ratio of red mud/sodium chloride/reducing agent (coke)/sodium sulfate of 100:15:15:10, magnetic field strength of 0.22 T and grinding fineness of less than 0.048 (95 wt.%). Finally, an iron concentrate sorting index with an iron grade of 78.29% and an iron recovery of 84.08% is obtained, which demonstrated efficient recovery of the valuable metal iron from red mud.

(3) Before the roasting, the metal iron in the red mud is mainly Fe_2O_3. After the roasting, the iron is transformed into a new iron mineral phase mainly composed of metal Fe and Fe_3O_4. The main impurities in the iron concentrate are calcium, silicon and aluminum.

Acknowledgments

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