

Separation of Kaolinite from Ion-Adsorption Rare Earth Tailings in Southern China and Iron Removal Treatment

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Abstract

Several hundred million tons of ion-adsorption rare earth tailings exist in Ganzhou, Southern China, which is a severe environmental hazard. To reduce and reutilize the tailing, kaolinite has been separated from the tailings by mechanical separation in laboratory scale and pilot scale. The results show that the tailing is mainly composed of fine kaolinite and coarse quart. Quartz and kaolinite can be separated by sieves, shaker, spiral chute or hydrocyclone, which has the similar results in laboratory scale and pilot scale. 30.2% of the tailings can be re-sourced and applied in ceramic industries. 41.7% of kaolinite can be obtained after sorting and iron removal by magnetic separator in pilot scale, which can be applied in ceramic industries according to the Chinese national standard (TC-3). The results give a progressive solution to re-source the tailings economically.

Keywords

Rare Earth Tailing, Kaolinite, Separation, Reutilize, Iron Removal

1. Introduction

Rare earth elements (REEs) have become vital and indispensable components of many high-tech products, devices and technologies. China's production of rare earth minerals has provided more than 90% of the world's supply since 2001 (Su, 2009; Chen, 2011) [1] [2]. However, what is left in China is million tons of tailings because rare earth ore deposits have relatively low REEs concentrations ranging from 10 to a few hundred parts

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per million by weight (Yang, *et al.*, 2013) [3]. Ion-adsorption rare earth reserves only distribute in seven provinces of southern China (Su, 2009; Wübbecke, 2013) [1] [4]. The ion-adsorption rare earth deposits were formed by chemical weathering decomposition and dissolution of granite and granite porphyry (containing relative high abundance of rare earth elements) and subsequent adsorption and enrichment on clay minerals during the migration and penetration process of rare earth mineral solutions. Unlike other rare earth minerals which are in solid state mineral phase and tend to be symbiotic or associated with the radioactive elements uranium and thorium, ion-adsorption rare earth minerals occur at a simple trivalent cationic state which is simply adsorbed onto clay sand and can be readily extracted by a simple leaching technique via anion-exchange process (Yang, *et al.*, 2013) [3]. Hence, the tailings can be and must be re-sourced because there are 191 million tons of tailings just distributed in Ganzhou region (Jiangxi Province, Southern China).

Kaolinite and quartz are the main components of the tailings characterized by Mineral Liberation Analyzer. It is possible to separate of kaolinite from the tailings due to the differences of density and particle size of kaolinite and quartz. In the present work, several methods of mineral separation were applied to the tailings and a systemic separation of the tailing was proposed.

2. Materials and Methods

2.1. Collection and Pre-Treatment of Tailings Samples

The tailings were collected from the ores of ion-adsorption rare earths in Ganzhou, Southern China. Representative sample was made by following Chinese standard method of mixing (China Standard Specification, GB 2007.1-87). No further grinding treatment was conducted in laboratory. The tailings were dried in oven at 100°C overnight, cooled naturally, and then be characterized.

The tailings was suspended in water (1:4 tailings to water ratio) and slurried by stirring with an impeller stirrer without adding any dispersant or adjusting the pH value.

2.2. Characterization

The bulk chemical element analysis in samples melted with lithium tetraborate was performed with an XRF spectrometer (Axios mAX PANalytical, Netherland).

The bulk mineral composition was performed with Mineral Liberation Analyzer (MLA 650, Czech Republic).

The particle size distribution of the tailings was measured by wet sieving method using 20 wt% suspensions. The sieve size of the copper standard sieves were sequentially 0.8 mm, 0.4 mm, 0.25 mm, 0.12 mm, 0.075 mm, and 0.043 mm.

2.3. Sorting Experiments in Laboratory Scale

Sorting of tailing by shaker: 500 ml 20 wt% tailings slurry was put into a shaker (6-S, China). The separation condition kept that: the stroke is 10 mm; the frequency is 220/min.

Sorting of tailing by settlement: 500 ml 20 wt% tailings slurry was sieved by wet sieving. The -0.043 mm fraction of tailings was further slurried in water (about 10 wt% solid) and then put into a cylinder (500 ml in volume). The slurry is stood undisturbed for 1 hr. The height of the slurry was separated according to Stoke equation of 5 μm , 10 μm and 20 μm .

Iron removal: 500 ml 20 wt% tailings slurry was sieved by wet sieving. The -0.043 mm fraction of tailings was put into a cycle type high gradient magnetic separator (SSS-I-145) (Chen, L., 2011, Svoboda, J., 2003) [5] [6]. The magnetic field intensity is in the range of 0.2 T-1.0T.

For the three processes above-mentioned, all the classified tailings were collected, and then dried in oven at 100°C overnight. The dried tailings were weight and characterized by XRF.

2.4. Separation Experiments in Pilot Scale

200 kg tailings were suspended in water to form 20 wt% slurry. The flow chart of the separation of the slurry is shown in **Figure 1**. The slurry was firstly sieved by a high frequency vibrating sieve (GYX11-1210, sieve size is 0.4 mm). The -0.44 mm fraction of the slurry was delivered into a spiral chute (TGL 0610) by a high-pressure pump. The K4, K5 fraction of the slurry were separated by a hydrocyclone. The overflow was separated by the

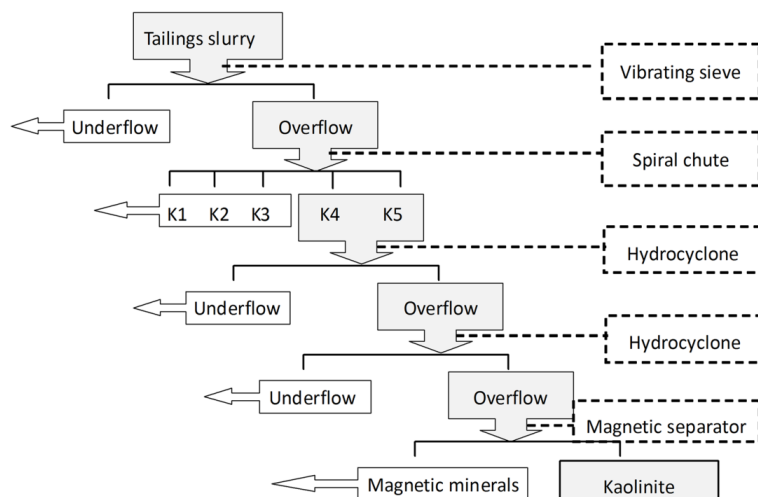


Figure 1. Flow chart of the tailings separation in pilot scale.

hydrocyclone secondly. Finally, the second overflow was put into wet dual band, high intensity, and high gradient magnetic separator (SSS-I-800). The magnetic field intensity is in the range of 0.6 T.

For the processes above-mentioned, all the classified tailings were collected, and then dried in oven at 100°C overnight. The dried tailings were weight and characterized by XRF.

3. Results and Discussions

3.1. Characterization of the Tailings

3.1.1. Chemical Composition of Tailings by MLA

Table 1 shows the mineral composition of the tailings. As can be seen, kaolinite and Quartz is the major mineral of the tailings, which shares 91.98% of the tailings. It implies that the tailings can be re-utilized in ceramic application through the separation of kaolinite or quartz. The other silicates are feldspar, biotite, muscovite and amphibole. These silicates do not need the separation before the ceramic application because the contents are small and they also can be used as sintering aid in the calcinations of ceramic. The iron and titanium minerals include of titanomagnetite, limonite, pyrite, ilmenite, rutile, iron sphene and leucosene, which shares about 1.77% of the tailings. The iron and titanium minerals should be removed down to a limit concentration because they are harmful to the product quality of ceramic. The content of the rare earth mineral is very slow (0.04%), including of monazite, xenotime, parisite and colloidal rare earth. There is no radioactive minerals remained, indicating the re-resource of the tailings is safe in ceramic application.

3.1.2 Chemical Composition of Tailings by XRF

The chemical composition of the tailings sample is presented in **Table 2**. As can be seen, the rich major chemical composition in oxides in mine tailing are SiO_2 (78.32%), Al_2O_3 (17.50%) and Fe_2O_3 (2.11%). Pure kaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) is white in color and its chemical composition is 46.54% SiO_2 , 39.50% Al_2O_3 and 13.96% H_2O (Grimshaw, 1971) [7]. Assumed that all of Al_2O_3 (17.50%) exist in kaolinite, it can be calculated that 44.3% of the tailings is kaolinite, which is similar to the results in **Table 1**. It is deduced that most of Al_2O_3 exist in kaolinite. Hence, the content of kaolinite can be estimated by that of Al_2O_3 in this work.

Minor quantities of transition elements such as iron, titanium and manganese are generally present as ancillary minerals which adversely affect the optical properties of kaolins, especially, Fe-containing minerals (Chandrasekhar and Ramaswamy, 2006; Malengreau *et al.*, 1996) [8] [9]. They cause brown-yellow colorations and reduce the whiteness. The Chinese national standard of kaolin (TC-3, GB/T 14563-2008) applied in ceramic industries requires that $\text{Al}_2\text{O}_3 \geq 28\%$, $\text{Fe}_2\text{O}_3 \leq 1.8\%$, $\text{TiO}_2 \leq 0.6\%$ (mass ratio). Obviously, the Fe_2O_3 content is 2.11% is larger than the requirement, implying that iron removal is required before the utilization of the tailings. There is no further treatment to remove TiO_2 in the separation of the tailings because the content of TiO_2 is low.

The tailing is sieved by the standard sieves. The particle size distribution and the grade of SiO_2 , Al_2O_3 , Fe_2O_3

Table 1. Mineral composition of the tailings.

Mineral	Content %	Mineral	Content %	Mineral	Content %
Kaolinite	42.29	Monazite	0.01	Colloidal rare earth	0.01
Quartz	49.69	Xenotime	0.01	Titanomagnetite	1.10
Feldspar	3.90	Parisite	0.01	Limonite	0.29
Diopside	0.01	Rutile	0.09	Ilmenite	0.13
Muscovite	0.68	Leucoxene	0.10	Iron sphene	0.04
Biotite	1.36	Pyrite	0.01	Remainder	0.14
Amphibole	0.11	Galena	0.01	Total	100

Table 2. Chemical composition of the tailings measured by XRF/%.

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	K ₂ O	Na ₂ O	CaO	MgO
78.32	17.50	2.11	0.38	1.21	0.43	0.22	1.33

and TiO₂ in every distribution are listed in **Table 3**. As can be seen, the particle size of the tailing shows the bi-modal distribution of 0.8mm and -0.043 mm. The grade of SiO₂ is larger than 91.46 for the + 0.4 mm fraction, implying that coarse grains are quartz and can be separated by 0.4 mm sieve. The grade of SiO₂ decreases and the grade of Al₂O₃ increase with the decrease of the particle size. Hence, most of kaolinite exists in the -0.043 mm fraction. Kaolinite and quartz maybe separated by sieving. However, the iron content in the -0.043 mm fraction is 2.16%, implying that the iron-bearing minerals are mixed with kaolinite. Iron removal is required for the kaolinite separated from the tailings (Svoboda, J. and Fujita, T., 2003, Santos, *et al.* 2012, Chandrasekhar and Ramaswamy, 2007, Xia, *et al.* 2012, Zegeye, A. *et al.*, 2013) [6] [10]-[13].

3.2. Sorting Experiments in Laboratory Scale

3.2.1. Sorting of the Tailings by Shaker

The tailing can be sorted by the shaker due to the difference of the density (the density of kaolinite is 1.58 - 1.63 g/cm³; the density of quartz is 2.64 - 2.71 g/cm³). **Table 4** shows the sorting results of the tailings. Obviously, the heavy quartz remains in the underflow K1 and the middling K2, whose gradation is over than 90%. The abnormal data in underflow may be the results that some of kaolinites are entrained by quartz grains in large size. The gradation of Al₂O₃ increases with the decrease of the particle size of the tailing. It is worth mentioning that the gradation of Al₂O₃ is 24.46%, which is far larger than the other zone. The yield of the tailings in overflow K5 is 57.71%, implying that most of kaolinite is fine and locates in the overflow K5. Hence, the tailings can be preliminarily separated into kaolinite and quartz by shaker. The gradation of Fe₂O₃ is 1.94% in the overflow, which is still higher than the national standard of TC-3. The recovery ratio of Fe₂O₃ in overflow is 69.90%. Most of the fine Fe-bearing minerals are mixed with kaolinite. It is no use to remove iron by the shaker.

3.2.2. Sorting of the Tailings by Settlement

The gradation of Al₂O₃ in the overflow of the shaker is 24.46%, which doesn't meet the requirement of TC-3 (in which, Al₂O₃ ≥ 28%). The tailings need to be sorted further.

Table 5 shows the sorting results of the tailings by settlement. The gradation of Al₂O₃ is larger than 28% if the particle size of the tailings is no more than 10 μm. Kaolinite required in ceramic industries can be separated from the rare earth tailing under the favorable sorting condition.

3.2.3. Iron Removal

It doesn't require separating kaolinite from quartz completely because they are both needed in traditional ceramic industries. Kaolinite and quartz share 91.98% of the ion-adsorption rare earth tailings (see **Table 1**). It implies that the tailing can be re-utilized in common ceramic industries if the Fe₂O₃ content can be decreased to 1.8%.

Table 3. Particle size distribution of the tailing and the quality of SiO₂, Al₂O₃, Fe₂O₃ and TiO₂.

Particles size/mm	Yield/ %	Gradation/%				Recovery ratio/%			
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂
0.8	19.1	98.44	0.91	0.31	0.05	23.99	0.99	2.96	2.85
-0.8 - +0.4	7.45	91.46	6.24	1.22	0.15	8.69	2.65	4.54	3.34
-0.4 - +0.25	3.49	79.55	13.28	4.02	0.59	3.54	2.64	7.01	6.15
-0.25 - +0.12	7.01	74.19	16.99	3.81	0.62	6.64	6.79	13.35	12.98
-0.12 - +0.075	4.15	72.70	18.90	3.30	0.72	3.85	4.47	6.85	8.93
-0.075 - +0.043	4.81	73.17	19.41	2.91	0.76	4.49	5.33	7.00	10.92
-0.043	53.99	70.84	25.04	2.16	0.34	48.80	77.12	58.29	54.83
Σ(original ores)	100	78.32	17.50	2.11	0.38	100	100	100	100

Table 4. Sorting results of the tailings by shaker.

Zone label	Yield /%	Gradation/%				Recovery ratio/%			
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂
Underflow K1	0.67	86.42	6.74	4.21	0.76	0.74	0.27	1.79	2.00
Middling K2	21.47	92.32	5.03	1.36	0.25	24.91	6.32	18.23	20.82
Middling K3	17.88	89.30	8.52	0.73	0.14	20.06	8.91	8.15	9.71
Middling K4	2.26	82.28	14.39	1.37	0.29	2.34	1.90	1.93	2.54
Overflow K5	57.71	71.64	24.46	1.94	0.29	51.95	82.60	69.90	64.92
Σ(original ores)	100	78.32	17.50	2.11	0.38	100	100	100	100

Table 5. Sorting results of the tailings by settlement.

Product label	Gradation/%			
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂
5 μm	59.71	31.64	1.38	0.14
10 μm	64.32	28.09	1.54	0.18
20 μm	65.61	24.47	2.5	0.41
Overflow in shaker	71.64	24.46	1.94	0.29

Table 6 shows the distribution of Fe element in the tailings. Fe element distributes mostly in titanomagnetite, kaolinite and limonite. The magnetic titanomagnetite and limonite can be removed by a magnetic separator. If it is done, the total Fe content in the tailing can be reduced to 1.23%, which meets the requirement of the standard of TC-3 in ceramic application. The effect of the magnetic induction on the Fe content is listed in **Table 7**.

As can be seen, the Fe gradation of the sorted tailing decreases with the increase of the magnetic field intensity. However, the ratio of iron removal is not proportional to the magnetic induction. The ratio of iron removal increases sharply with the magnetic field intensity. When the magnetic field intensity is 0.6T, the Fe gradation of the sorted tailing is 1.06% and the ratio of iron removal is 50.41%. Under this condition, most of titanomagnetite and limonite is removed. However, the ratio of iron removal increases slightly (4.68%) when the magnetic induction increases further from 0.6T to 1T. Therefore, the economical magnetic field intensity is 0.6T.

3.3. Separation Experiments in Pilot Scale

It has been proved that the tailing can be sorted by gravity method in laboratory scale. To make the tailing more profitable, kaolinite was separated from the tailing. The tailing slurry was firstly sieved by a high frequency vi-

Table 6. Distribution of Fe element in the tailings.

Mineral	Content of mineral/%	Fe ₂ O ₃ content in mineral/% [*]	Fe ₂ O ₃ distribution in tailing/%	Ratio of Fe ₂ O ₃ distribution/%
Kaolinite	42.29	0.57	0.24	16.99
Quartz	49.69	0.17	0.08	5.98
Feldspar	3.90	0.16	0.01	0.43
Titanomagnetite	1.10	66.28	0.73	51.73
Limonite	0.29	53.19	0.15	10.85
Ilmenite	0.13	32.07	0.04	2.98
Muscovite	0.68	2.24	0.02	1.08
Biotite	1.36	10.34	0.14	9.98
Total	99.44	—	1.41	100

Notes: ^{*} the average value measured by MLA, which is smaller than by XRF due to the crystal water.

Table 7. Effect of the magnetic field intensity on the iron removal.

Magnetic field intensity/T	Fe ₂ O ₃	
	Gradation/%	Ratio of iron removal/%
0.2	1.40	34.78
0.4	1.26	41.02
0.6	1.06	50.41
0.8	0.99	53.80
1	0.96	55.09
Tailing	2.14	—

brating sieve (GYX11-1210, sieve size is 0.4 mm). The coarse quartz was removed. The -0.44mm fraction of the slurry was delivered into a spiral chute (TGL 0610). **Table 8** shows the separation results of the spiral chute.

As can be seen, the gradations of SiO₂ in Ks1 and Ks2 area are higher than the other areas, indicating that quartz can be sorted further by the spiral chute after removal of the coarse quartz. However, the fine quartz is also mixed with kaolinite because the recovery ratio of SiO₂ in Ks5 area is 43.40%. Correspondingly, kaolinite distributes mainly in Ks4 and Ks5 area, whose gradation is about 24% and total recovery ratio is 76.56%. The Al₂O₃ gradation still doesn't meet the standard of TC-3 due to the mixing of fine quartz.

To separate kaolinite from quartz further, the K4 and K5 fractions of the slurry were delivered into the hydrocyclone. The separation result is shown in **Table 9**.

It can be seen that the sorting by the hydrocyclone contributes to separation of kaolinite. However, the aim of the separation is still not obtained. The tailing in overflow needs to be separated second time. **Table 10** gives the separation results of the hydrocyclone secondly.

Table 10 shows that the gradation of Al₂O₃ in the overflow is 28.6%. The sorted kaolinite can be applied in ceramic industries. Correspondingly, the yield of the overflow is 87.13%, implying that the separation by hydrocyclone secondly has the interesting in the point of economical view. The results in pilot scale agree with those in laboratory scale.

The whiteness is one of the most important factors in determining the application and economic value of kaolinite (Xia, *et al.* 2012) [12]. In general, the most deleterious impurities for whiteness of kaolin are iron oxide and titanium oxide minerals. Iron removal is required based on the results in **Table 10** and the national standard of China (TC-3).

Iron removal of the tailing in overflow Kh2 is processed in high gradient magnetic separator (SSS-I-800). **Table 11** gives the separation results of the magnetic separator.

Table 8. Separation results of spiral chute.

Production label	Yield/ %	Gradation/%				Recovery ratio/%			
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂
Underflow Ks1	16.78	89.13	7.54	3.13	0.61	19.19	5.91	23.81	26.06
Middling Ks2	10.12	86.57	10.52	1.00	0.27	11.25	5.74	4.59	6.96
Middling Ks3	12.03	78.76	18.19	1.19	0.27	12.17	11.79	6.49	8.27
Middling Ks4	15.07	72.29	24.00	2.50	0.40	13.99	19.49	17.09	15.35
Overflow Ks5	46.01	73.46	23.02	2.30	0.37	43.40	57.07	48.01	43.36

Table 9. Separation results of hydrocyclone (first time).

Product label	Yield /%	Gradation/%				Recovery ratio/%			
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂
Underflow Kh1	30.75	72.17	24.66	2.45	0.42	31.48	29.35	27.63	37.56
Overflow Kh2	69.25	70.93	26.36	2.85	0.31	68.52	70.65	72.37	62.44
Σ(original tailing)	100	73.17	23.26	1.87	0.29	100	100	100	100

Table 10. Separation results of hydrocyclone (second time).

Product label	Yield/ %	Gradation/%				Recovery ratio/%			
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂
Underflow Kh1	12.87	72.46	24.95	2.66	0.33	13.66	11.41	11.82	13.59
Overflow Kh2	87.13	68.63	28.60	2.93	0.31	86.34	88.59	88.18	86.41
Σ(original tailing)	100	70.93	26.36	2.85	0.31	100	100	100	100

Table 11. Separation results of the high gradient magnetic separator.

Product	Yield/ %	Gradation/%				Recovery ratio/%			
		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂
Magnetic mineral	18.52	68.38	19.29	10.53	0.87	18.70	12.97	66.42	51.00
Diamagnetic mineral	81.48	67.57	29.43	1.21	0.19	81.30	87.03	33.58	49.00
Σ(original tailing)	100	68.63	28.60	2.93	0.31	100	100	100	100

The results in **Table 11** show that 66.42% of Fe₂O₃ can be removed by the magnetic separator. Comparing to the results in **Table 6**, all the magnetic titanomagnetite and limonite have been removed by the magnetic separator. The magnetic separator in pilot scale has the same separation action with that in laboratory scale. The Fe₂O₃ content decreases from 2.93% to 1.21%, which is less than the requirement of TC-3 (1.8%). Obviously, the Fe₂O₃ that exists in diamagnetic minerals must be removed by chemical method if the quality of kaolinite needs to be increased further (Svoboda, and Fujita, 2003, Santos, *et al.* 2012, Chandrasekhar and Ramaswamy, 2007, Xia, *et al.* 2012, Zegeye, *et al.*, 2013) [6] [10]-[13].

In a word, after the above-mentioned separation processes, 30.2% of the ion-adsorption rare earth tailing can be re-sourced and applied in ceramic industries [The total yield is estimated by the yields in every separation process: (46.01 + 15.07)% × 69.25% × 87.13% × 81.84% = 30.2%]. The recovery ratio of kaolinite is 41.7%, which is estimated as the yield. Some of kaolinite cannot be sorted because it is mixed or accompanied by the other minerals. The recovery ratio of kaolinite may be increased by crushing the tailing. However, it is not eco-

conomic considering most of the coarse grains in the tailing are quartz.

4. Conclusions

Kaolinite had been sorted from ion-adsorption rare earth tailings in Southern China in laboratory scale and pilot scale. The conclusions can be drawn as following:

- 1) The ion-adsorption rare earth tailing in Southern China is mainly composed of kaolinite and quartz, which can be re-sourced because no radioactive minerals exist.
- 2) Coarse quartz and fine kaolinite can be separated by sieves, shaker, spiral chute or hydrocyclone. There are the similar results in laboratory scale and in pilot scale. 30.2% of the tailings can be re-sourced and applied in ceramic industries.
- 3) 41.7% of kaolinite can be obtained after sorting and iron removal by magnetic separator in pilot scale, which meet the national standard of China (TC-3). Fe_2O_3 in the sorted kaolinite has the gradation of 1.21%, which should be removed by chemical method if the quality of kaolinite needs to increase further.

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