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Process mineralogy characteristics and titanium preconcentration of Panxi vanadium-titanium magnetite tailings

Chemical and mineralogical characteristics of Panxi vanadium–titanium magnetite tailings are studied to beneficiate the contained ilmenite. A pilot study using a spiral chute to precocentrate the tailings, combined with table enrichment, is carried out. The aluminosilicate content of the tailings is high and the TiO_2 content is only 10.28%. Titanium mainly existed in the form of ilmenite and titanomagnetite, which accounted for 78.02% of total titanium. Although most ilmenite is dissociated, a small amount is embedded in the gangue. More than 90% of the gangue comprised titanaugite, plagioclase, and serpentine. After sorting the sample by spiral chute, a large amount of gangue is discarded and a coarse concentrate with a TiO_2 grade of 23.54% is obtained. This is enriched using a table to obtain a final concentrate of 36.85% TiO_2 with an overall recovery of 71.75%. Titanium pre-enrichment is achieved.

Keywords: *Vanadium-titanium magnetite; process mineralogy; embedding characteristics; preconcentration; gravity beneficiation*

1. Introduction

Titanium is a widely-used metal that has a relatively high concentration in the earth's crust. It is mainly derived from vanadium–titanium magnetite and rutile [1-2]; however, the complex mineral composition of the former and the symbiotic relationship between the minerals makes the extraction of ilmenite from vanadium-titanium magnetite much more complicated than from rutile [3]. About half of the titanium resources in China's vanadium-titanium magnetite are lost to tailings during the beneficiation process [4-6]. This titanium resource is mainly recovered by gravity concentration, magnetic separation, or flotation. Because of the low titanium content in the tailings, the fine size of the ilmenite grains, and the lack of sophistication of technology and equipment, this component is difficult to recover [7-10].

The sample used in this study originated from a concentrator located in Panxi, China. The ore is subjected to magnetic separation and comprised several magnetic minerals, including magnetite and ilmenite. To efficiently recover ilmenite from the tailings, the process mineralogy of titanium, monomer dissociation, and liberation characteristics are studied. This enabled a theoretical basis for the recovery of ilmenite from tailings to be determined to lay the foundation for the subsequent acquisition of titanium concentrate.

2. Experimental

2.1. Sample characteristics

The sample used in this study is derived from tailings produced by a concentrator in Panxi, China. The water content is less than 1% and the particle size is 0.038–20 mm. Large samples included fine-grain agglomeration, so, to avoid large sampling errors, agglomerated samples are broken up before use. Multi-element chemical composition and titanium phase analysis of the selected tailings sample are shown in Tables 1 and 2, respectively.

Tables 1 and 2 show that the main element that could be recovered from the tailings is titanium. The grade is 10.28% TiO_2. The contents of valuable metals, such as iron, manganese, and zirconium, are too low for economic recovery. The gangue components are mainly CaO, Al_2O_3, and SiO_2, the total content of which is 67.68%. For better separation of the titanium minerals, it is necessary to reduce the content of these gangue materials. The presence of titanium in the

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TABLE 1: CHEMICAL COMPOSITION OF TAILINGS SAMPLE (%)

TFe	Fe_2O_3	TiO_2	K_2O	CaO	Co_3O_4	MgO	MnO
10.38	14.12	10.28	1.26	12.56	1.55	0.26	0.18
Al_2O_3	SO_3	SiO_2	ZrO_2	BaO	NiO	ZnO	Nb_2O_5
12.40	1.32	42.72	0.07	0.05	0.04	0.03	0.01

TABLE 2: TITANIUM PHASE ANALYSIS OF TAILINGS SAMPLE (%)

Ingredient	TiO_2	Rutile	Ilmenite and titanomagnetite	Perovskite	Sphene and silicate
Content	10.28	0.05	8.02	1.03	1.18
Occupancy	100.00	0.49	78.02	10.01	11.48

tailings is relatively simple, occurring mainly in metal oxides. The content of titanium in iron oxide minerals is 88.52%.

Based on the above analyses, it is concluded that the titanium grade in the tailings is low and the gangue mineral content is large. If a simple magnetic separation-flotation process is adopted, it is bound to increase the consumptions of energy and flotation reagent, and it will be difficult to obtain a titanium concentrate with a higher grade[11-14]. Therefore, it is necessary to formulate a beneficiation method for the tailings. It is proposed to use gravity beneficiation to first upgrade the tailings to obtain a titanium coarse concentrate, analyze the TiO_2 grade in the beneficiation products by potassium dichromate titration, and then further upgrade the material using a shaking table.

2.2. METHODS

The tailings sample is naturally air dried, then cut and polished to form polished sections for microscopic observation. Image acquisition and monomer dissociation statistics are performed using a transflective polarizing microscope (Ortholux II POL BK, Leitz, Germany). X-ray diffraction (XRD; X'Pert PRO, PANalytical, Nederlands), scanning electron microscopy with energy-dispersive spectroscopy (SEM–EDS; Libra 200, Zeiss, Germany), electron probe microanalysis (EPMA; Libra 200, Zeiss, Germany), and other testing methods are used to study the composition, occurrence, and microscopic characteristics of titanium minerals in the tailings.

3. Results and discussion

3.1. MAIN MINERAL COMPOSITION AND CONTENT

The XRD pattern of the sample is shown in Fig.1, and the main mineral composition and content of the tailings are shown in Table 3. It is concluded that the mineral composition of the ore is complex, the mineral surface is covered with slimes, and the particles are bonded to each other. The main metal-containing minerals in the sample are ilmenite and titanomagnetite, followed by magnetite, limonite, hematite, pyrite, pentlandite, and sulfide ore; gangue mainly comprised titanaugite, plagioclase, and serpentine, followed by chlorite, mica, hornblende, olivine, and calcite.

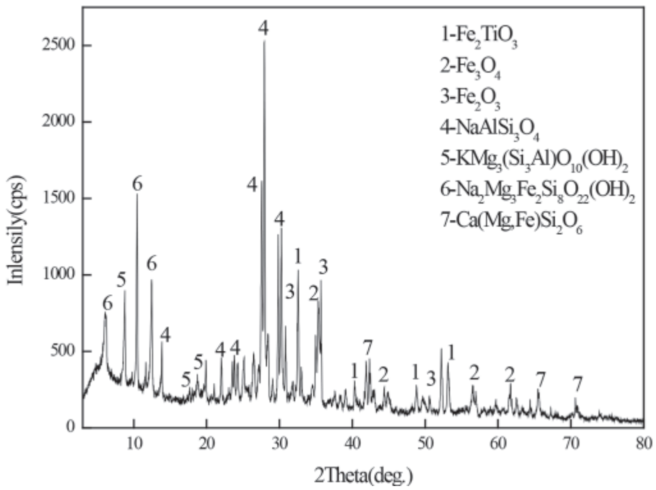

Fig.1 X-ray diffraction pattern of tailings sample

3.2. OCCURRENCE OF MAJOR MINERALS IN TAILINGS

3.2.1. Ilmenite

Ilmenite ($FeTiO_3$) is the main titanium mineral in the tailings and the target mineral to be recovered. The surface of the ilmenite crystals are smooth and they generally appears to be granular or plate-like. Granularilmenite is often closely associated with titanomagnetite or between silicate minerals [15]. Microscopic observation of ilmenite shows polycrystalline and lattice twin crystals, but the crystal grain size is non-uniform and easy to break and dissociate. Microscopic observation shows that the degree of dissociation of ilmenite exceeded 80% (Fig.2a). The remaining ilmenite mainly had the following forms: a small amount of ilmenite and pyroxene formed a eutectic structure (Fig.2b); several ilmenite and plagioclase particles are symbiotic with each other (Fig.2c); small ilmenite grains are closely symbiotic with hornblende (Fig.2d).

3.2.2. Titanomagnetite

Titanomagnetite ($FeO \cdot (Fe \cdot Ti)_2O_3$) is a composite mineral phase composed of magnetite, ilmenite, magnesium aluminate spinel, and ilmenite crystallites. It generally exhibits auto-, hypidio-, and xenomorphic granular textures and can be easily dissociated [16-17]. Microscopic observation of the tailings

Table 3: Main mineral composition and disseminated grain size and content in tailings sample

Type	Mineral	Molecular formula	Granularity (mm)			Content (%)
			Minimum	Maximum	Average	
Sulfide	Pyrite	FeS_2	0.002	0.5	0.03~0.2	3
	Sulfur nickel cobalt	$(Co,Ni)_3S_4$	0.001	0.2	0.02~0.1	0.1
	Linneite	Co_3S_4	0.001	0.2	0.01?0.1	0.3
	Cobalt pyrite	$(Fe,Co)S_2$	0.002	0.09	0.01~0.1	0.5
Carbonate	Calcite	$CaCO_3$	0.01	1	0.05~0.35	3
	Dolomite	$MgCa(CO_3)_2$	0.005	1	0.05~0.3	0.2
Oxide	Quartz	SiO_2	0.01	1	0.05~0.2	7
	Magnetite	Fe_3O_4	0.002	0.8	0.01~0.5	2
	Titanomagnetite	$FeO(FeTi)_2O_3$	0.001	1	0.01~0.3	3
	Ilmenite	$FeTiO_3$	0.02	0.5	0.05~0.25	12
Silicate	Amphibole	$Ca_2(Mg, Fe)_4Al(Si_3AlO_{22})(OH)$	0.05	1	0.1~1.5	20
	Potash feldspar	$(NaCa)Al(AlSi)Si_2O_8$	0.05	0.8	0.1~0.5	4
	Diopside	$CaMg_3(SiO_3)_4$	0.05	1.5	0.1~0.3	11
	Tremolite	$CaMg_3(SiO_3)_4$	0.05	0.3	0.1~1	8
	Chlorite	$Y_3[Z_4O_{10}](OH)_2 \cdot Y_3(OH)_6$	0.001	0.06	0.005~0.05	6
	Montmorillonite	$(Na,Ca)_{0.33}$ $(Al,Mg)_2[Si_4O_{10}]$ $(OH)_2 \cdot nH_2O$	0.001	0.06	0.005?0.05	7
	Talc	$H_2Mg_3(SiO_3)_4$	0.001	0.08	0.005~0.05	10
	Serpentine	$H_2Mg_3Si_2O_9$	0.01	0.2	0.03~0.2	2
Total						99.1

shows that tiny minority crystals are encapsulated in the pyroxene (Fig.2d).

3.2.3. *Magnetite*

The main component of magnetite is Fe_3O_4. The crystals are usually octahedron or rhombohedral dodecahedron. The aggregates are usually granular and dense, without dissociation [18]. The content of magnetite in the tailings sample is small, but particles with a size of 0.02-0.12 mm are observed under microscope (Fig.2a).

3.2.4. *Gangue minerals*

The gangue minerals are mainly titanaugite, plagioclase, and serpentine, which together counted for more than 90% of the gangue. Titanaugite usually exhibits automorphic or xenomorphic granular textures and embedded in metal mineral particles [19-20]. The content of plagioclase in the gangue is second only to that of titanaugite. The plagioclase is usually automorphic or fractal between the metal or gangue minerals (Fig.2b). Some plagioclase is transformed into chlorite and sericite due to secondary changes.

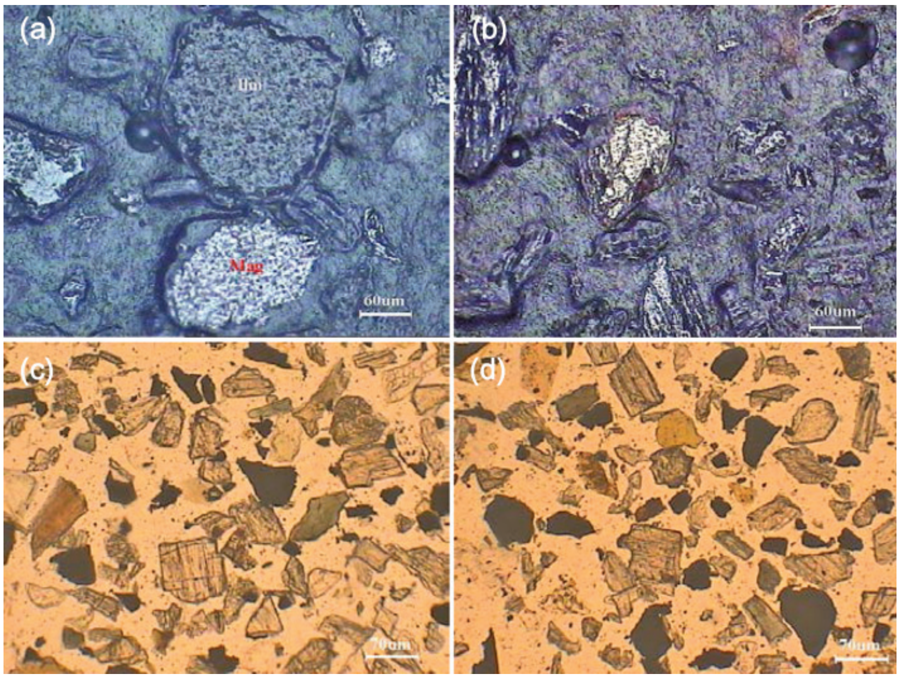

Fig.2 Microscope analysis of ilmenite in tailings sample

3.3. Distribution of target mineral as a function of particle size

The sample is sieved into the following eight particle sizes: +0.38 mm, –0.38+0.25 mm, –0.25+0.15 mm, –0.15+0.096 mm, –

Table 4 Particle size analysis of tailings samples

Particle-size/mm	Yield/%	The grade of TiO_2/%	The distribution ratio of TiO_2/%
–2~+0.38	31.74	8.14	25.58
–0.38~+0.25	16.65	9.80	16.16
–0.25~+0.15	22.41	12.66	28.09
–0.15~+0.096	14.88	11.17	16.46
–0.096~+0.074	4.47	9.65	4.27
–0.075~+0.048	4.74	9.38	4.40
–0.048~+0.038	1.59	9.69	1.53
–0.038~0	3.51	10.07	3.50
Total	100.00	10.28	100.00

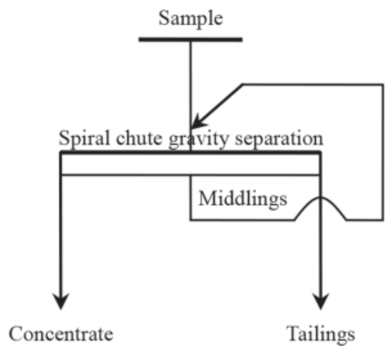

Fig.3 Flow chart illustrating principle of spiral chute gravity separation

0.096+0.074 mm, –0.074+0.048 mm, –0.048+0.038 mm, and –0.038 mm. Each fraction is weighed and the TiO_2 grade chemically determined. The results are shown in Table 4.

These data indicate that TiO_2 is distributed in each particle size at different contents. In the first three fractions, the grade of TiO_2 increases with a decrease in particle size. When the tailings particle size is less than 0.15 mm, the grade of TiO_2 shows a decreasing trend, but, the finer the particle size is, the smaller will be the grade difference of TiO_2. The highest Ti content of 12.66% is measured in the –0.25+0.15 mm size range.

3.4. Effect of spiral chute gravity separation on titanium preconcentration

The main titanium recovery processes used globally include magnetic separation followed by flotation, single-step flotation, coarse particle gravity beneficiation-electrical separation, and fine-grained magneto-flotation [21-22]. The TiO_2 grade in this sample is only 10.28% and it has high gangue and slime contents, so it is difficult to directly separate TiO_2. The density of ilmenite is greater than that of feldspar, plagioclase, serpentine, mica, and other gangue minerals. Microscopic observation confirmed that ilmenite is separated from the gangue minerals; therefore, a two-stage gravity separation process is considered. The large amount of tailings that is discarded by a spiral chute is then enriched using a shaking table to obtain an ilmenite concentrate that is suitable for subsequent sorting.

A spiral chute is suitable for the separation of ores of iron, ilmenite, chromite, tungsten, and tin, where there is a large difference in specific gravity and particle size of 0.074-2 mm [23-25]. Such devices have the advantages of small footprint, simple structure, convenient operation, and large processing capacity. The spiral chute used in this work is a model 5LL-600 (Yifan, China), which has an outer diameter of 600 mm, pitch of 360 mm, and five spiral turns. The flow chart of the principle is shown in Fig.3.

To avoid affecting the test results by agglomeration, all samples are processed to –1.18 mm or less. Although ilmenite is largely dissociated, considering the mutual entrainment between particles, the effect of sample size on the selection of spiral chute is first studied. The tailings sample is grounded to –0.25 mm, –0.15 mm, –0.096 mm, and –0.074 mm using a ball mill and the resulting TiO_2 grade in the spiral chute concentrate product is measured by chemical analysis. The results are shown in Fig.4a.

The TiO_2 grade and recovery to this coarse concentrate first increases and then decreases with a decrease of the particle size. Both parameters were optimal when the particle size is –0.25 mm, so this particle size is selected. The grade of TiO_2 in this concentrate is 23.54%. To improve the recovery, the middle ore component is reground and reprocessed. After this treatment, a crude concentrate with a TiO_2 grade of 23.53% and recovery of 75.03% is obtained. There is still a gap between the TiO_2 grade of the concentrate and the expected value of 47%. To further enrich the ilmenite, a table is used for subsequent sorting.

3.5. Effect of table gravity separation on grade and recovery of titanium from coarse spiral concentrate

A single-layer table is employed. The influences of feed size, table stroke, and vibration frequency on the sorting index are studied. Only two products of concentrate and tailings are produced during the experiment.

3.5.1. Effect of feeding granularity

The coarse concentrate produced by the spiral chute is grounded to –0.25 mm, –0.15 mm, –0.096 mm, –0.075 mm, and –0.048 mm, then subjected to sorting at a fixed table stroke of 15 mm, frequency of 300/min, while other conditions remained constant. Chemical analysis of the product is undertaken. The results are shown in Fig.4b.

As the feed size decreases, the grade of TiO_2 concentrate increases and recovery decreased. The recovery decreases sharply with a decrease of particle size. Considering the relationship between recovery and grade, the final optimum particle size for the separation is determined to be –0.15 mm. For this condition, the grade of the concentrate is 26.95% TiO_2 and recovery is 87.11%.

3.5.2. Effect of table stroke length

Table gravity separation is performed using 8 mm, 12 mm,

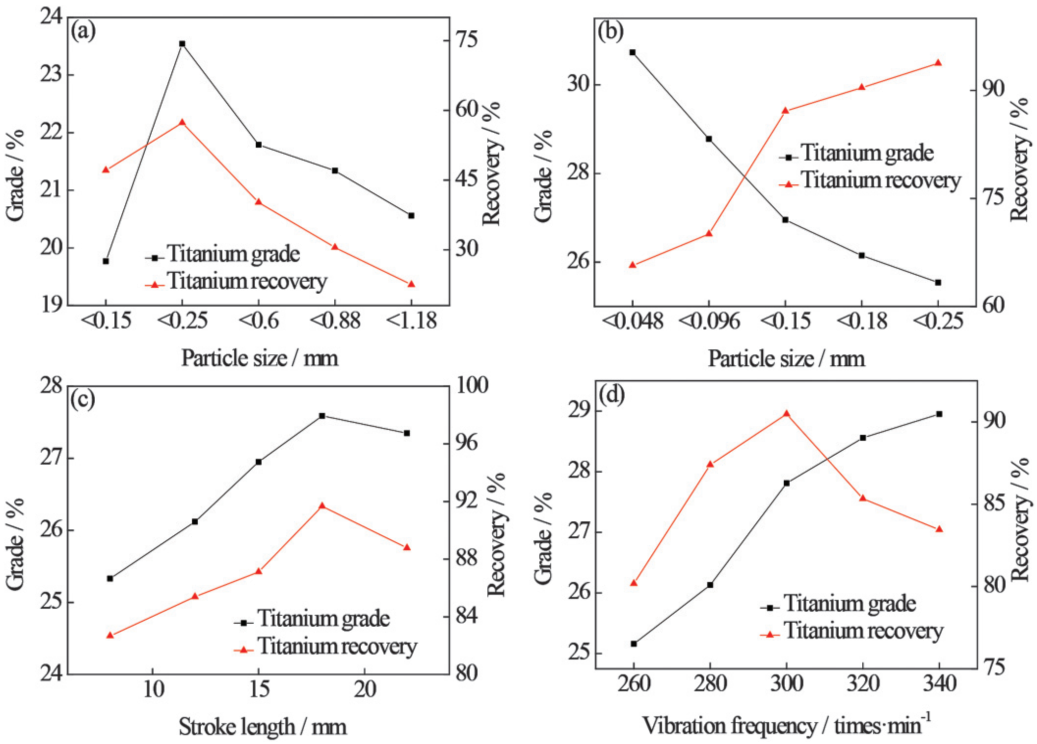

Fig.4 Effect of tailings particle size and equipment parameters on titanium grade and recovery rate

18 mm, and 22 mm stroke lengths for a vibration frequency of 300/min, spiral concentrate particle size of –0.15 mm, and holding other conditions constant. Each product is subjected to chemical analysis. The results are shown in Fig.4c.

As the stroke length increased, both the grade and recovery of TiO_2 to the concentrate first increases and then decreases. When the stroke is 18 mm, the grade and recovery are maximized, giving a concentrate grade of 27.59% TiO_2 and recovery of 91.67%. Therefore, the optimum stroke length is determined to be of 18 mm.

3.5.3. Effect of vibration frequency

Using a fixed table stroke length of 15 mm, spiral concentrate particle size of –0.15 mm, and keeping other conditions constant, the vibration frequency is varied to 260/min, 280/min, 320/min, and 340/min. The product is subjected to chemical analysis. The results are shown in Fig.4d.

As the table vibration frequency increased, the concentrate grade shows an upward trend, while the recovery first rises and then decreases. The recovery of TiO_2 is maximized at 90.48% when the frequency is 300/min, with a concentrate grade of 27.81%. Considering the relationship between recovery and grade, a table shaking frequency of 300/min is selected.

3.5.4. Testing of entire flowsheet using spiral chute and table gravity separation

Based on the above test results, the selected process for ilmenite pre-enrichment from the tailings are shown in Fig.5. The joint sorting test is carried out using a spiral chute feed particle size of –0.25 mm, table feed size of –0.15 mm, table stroke length of 18 mm, and vibration frequency of 300/min. The ilmenite preconcentration test results are shown in Table 5. The final TiO_2 grade is 36.85% and the overall recovery of ilmenite concentrate is 71.75%.

4. Discussion

The morphology and chemical composition of minerals in the final concentrate are identified by X-ray fluorescence (XRF; Axios, PANalytical, Nederlands), XRD (Cu-Ka radiation at a scan rate of 10/min in the range of 3-80), scanning electron microscopy (SEM; Libra 200, Zeiss, Germany), SEM-EDS, and EPMA. The main chemical components are shown in Table 6 and the results of the XRD analysis are shown in Fig.6. The titanium phase analysis of the coarse concentrate are shown in Table 7. The results of SEM, EPMA analysis are shown in Fig.7, and SEM-EDS analysis are shown in Fig.8.

The content of TiO_2 in the final concentrate exceeded 27% and the gangue content (CaO, SiO_2, and Al_2O_3) is significantly

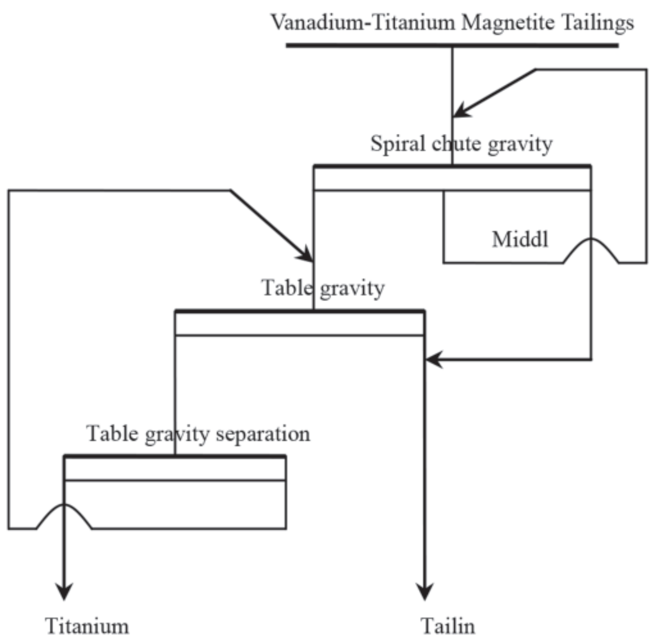

Fig.5 Complete flowsheet used for ilmenitepreconcentration

lower than that of the ore. The ilmenite is irregularly granular, scaly, or thick. Titanium exhibited relatively obvious aggregation and almost no slime is evident. Most ilmenite particles has a size between 0.1 and 0.25 mm. EDS analysis of the final concentrate also shows that the TiO_2 content exceeds 27%, which is similar to the chemical results. This product could therefore be considered a qualified ilmenite concentrate.

TABLE 5 RESULTS FOR FULL FLOWSHEET FOR ILMENITEPRECONCENTRATION BY SPIRAL CHUTE FOLLOWED BY TABLE GRAVITY SEPARATION (%)

Products	Yield	Grade of TiO_2	Recovery of TiO_2
Titanium concentrate	20.01	36.85	71.73
Tailings	79.99	3.63	28.27
Totals	100.00	10.28	100.00

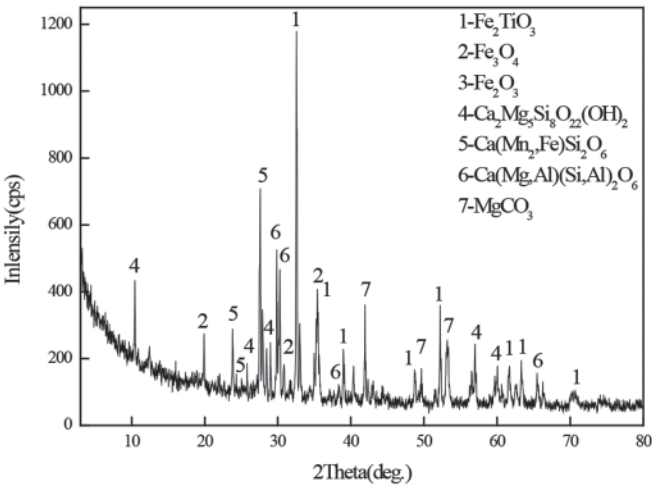

Fig.6 X-ray diffraction pattern of final ilmenite concentrate

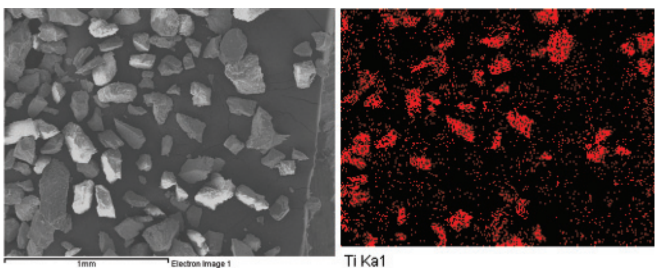

Fig.7 Scanning electron micrograph and electron probe microanalysis image of titanium distribution in final concentrate

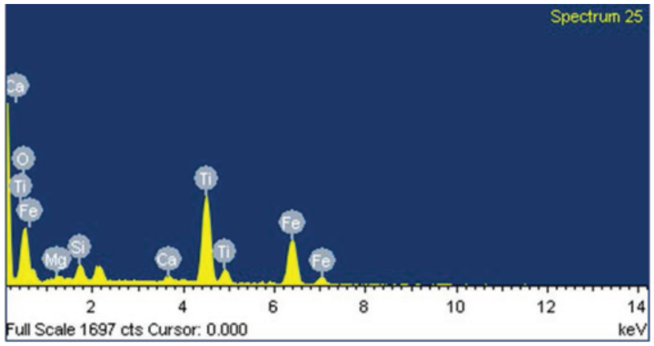

Fig.8 Scanning electron microscopy energy-dispersive spectrum of final concentrate

5. Conclusions

1. A vanadium-titanium magnetite tailings with a large amount of mud, containing 10.38% iron and 10.28% titanium, is studied for recovery of ilmenite. Iron is mainly in the form of titanaugite, ilmenite, and plagioclase; titanium is mainly in the form of ilmenite, titanomagnetite, and hornblende.
2. Ilmenite in the sample is largely dissociated, mainly in the form of granules and flakes. Small amounts of ilmenite formed a eutectic structure with pyroxene or are closely symbiotic with hornblende; some ilmenite and slant feldspar exhibited is symbiosis. Ilmenite is distributed in all particle sizes of tailings and is most abundant in the –0.25+0.096 mm cut.
3. After sorting using a spiral chute, a coarse concentrate with a TiO_2 grade of 23.54% is obtained. This is further concentrates twice using a table. The final TiO_2 grade is 36.85% at an overall recovery of 71.75%. A qualified floating material could therefore be obtained using this beneficiation process.

Author Contributions

Y.P.W.D. and Q.W.contributed to design experiments and experimental operations. J.X. Y.Z. and C.C.contributed to revise the test plan and provide test equipment support. G.J. and W.H. contributed to the interpretation of the results. Y.P. took the lead in writing the manuscript. J.X. revised the manuscript.

TABLE 6: MAIN CHEMICAL COMPONENTS OF FINAL ILMENITE CONCENTRATE (%)

TFe	Fe_2O_3	TiO_2	K_2O	CaO	Co_3O_4	MgO	MnO
20.37	28.12	36.85	0.23	7.25	1.28	5.69	0.32
Al_2O_3	SO_3	SiO_2	ZrO_2	BaO	NiO	ZnO	Nb_2O_5
5.47	0.69	20.98	0.20	0.20	0.04	0.02	0.11

TABLE 7: TITANIUM PHASE ANALYSIS OF FINAL TITANIUM CONCENTRATE (%)

Ingredient	TiO_2	Rutile	Ilmenite and titanomagnetite	Perovskite	Sphene and silicate
Content	36.84	0.12	35.44	0.26	1.02
Occupancy	100.00	0.33	96.19	0.71	2.77

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Conflicts of Interest: The authors declare no conflict of interest.

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