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Function and mechanism of sodium silicate in the cleaning process of ilmenite rough concentrate

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Abstract: The grade of titanium dioxide (TiO_2) from Panzhihua titanium concentrate (defined as the ilmenite rough concentrate in this paper) is generally around 47%. The high impurity content in the titanium concentrate causes difficulties for its successive use in the production of titania pigment. Further purification of the ilmenite concentrate will make it more useful for industrial use. In this study, a further cleaning process of the ilmenite rough concentrate is conducted using sodium silicate as the depressant, and the function of sodium silicate is determined by flotation, absorption measurements, Zeta potential measurements, and infrared spectral analysis. The results indicate that an ilmenite concentrate with a TiO_2 grade of 50.37% can be achieved from 46.78% according to the stages on the flowsheet—one roughing, three cleaning and one scavenging—under the optimal conditions, and can also reflect the advantages of the excellent selectivity of sodium silicate. Sodium silicate can hinder the adsorption of salicylhydroxamic acid (SHA) on the titanaugite surface but has almost no effect on ilmenite. Multiple methods comprehensively confirm that sodium silicate provides a good selective depression effect for the further purification of the ilmenite rough concentrate.

Keywords: ilmenite, sodium silicate, titanaugite, cleaning, rough concentrate

1. Introduction

Titanium (Ti) exhibits a unique and superior performance in coating and anti-corrosion, among other applications (Hou et al. 2011, Yang et al. 2016). The global titanium resources with industrial value are mainly ilmenite, anatase, brookite, perovskite, and rutile; and among these, ilmenite and rutile have been largely used as the main raw materials for producing titania pigment and titanium sponge. (Drzymala et al. 1983, Fan et al. 2000, Yang et al. 2016). Owing to the reserves of rutile being lower than those of ilmenite in China, it is essential to develop viable methods of exploiting and using ilmenite ore. Various methods have been used to obtain a high grade ilmenite product, such as gravity separation, magnetic separation, and electrostatic separation (Mehdilo et al. 2015). Flotation separation is one of the most commonly used methods to efficiently separate ilmenite from associated gangue minerals (Fan et al. 2014, Mehdilo et al. 2014). China has an abundance of titanium deposits, and over 90% of the deposits are in Panzhihua (Zhou et al. 2013, Zhao et al. 2014, Yang et al. 2016), where more than 20 rare metals are also found (Li et al. 2006, Liu et al. 2008, Chen et al. 2011). The ilmenite deposits in Panzhihua occur in the form of basic rock and ultrabasic rock mass, in which Ti, Fe, Ca, Mg, and other elements exist (Jiren et al. 1988, Zhu et al. 2011, Parapari et al. 2017). Recently, extensive research has been conducted to improve the usage of ilmenite and titanomagnetite concentrates (Deng et al. 2012, Fan et al. 2016, Mahmoud et al. 2019). Some processes have been adopted to concentrate these minerals (Jena et al. 1995,

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Sole 1999, Wang et al. 2017). However, the grade of the titanium dioxide in the concentrate provided by many ore-dressing plants is not ideal.

The content of titanium dioxide in titanium concentrate from Panzhihua is generally around 47% (Dong et al. 2012). The high impurity content causes difficulties for the successive production of titania pigment (Li et al. 2006, Chen et al. 2013, Zhao et al. 2019). The main challenge to improve the purity of the titanium concentrate is the difficulty in separating ilmenite from titanaugite (Zhang et al. 2009, Liu et al. 2017, Zhao et al. 2019).

Surface modification in flotation typically entails activating the useful minerals or depressing the gangue minerals (Duan et al. 2018, Deng et al. 2019, Lai et al. 2019). The corresponding agents are termed as activators or depressants. Using a proper depressant is important to enhance the flotability differences between valuable minerals and gangue minerals (Liu et al. 2015, Yang et al. 2016). Sodium silicate is one of the most commonly used modifiers and is the most widely used dispersant of slime, especially for depression of silicate gangue (Silva et al. 2012, Arantes et al. 2013). It is also frequently used as a depressant in the flotation of nonmetallic minerals, such as phosphate-containing minerals (Zhang et al. 2007). Yang et al. (Yang et al. 2016) studied the selective flotation of ilmenite from olivine using an acidified water glass as a depressant. Sodium silicate is also widely used to disperse kaolinite over a wide range of pH values (Al-Wakeel et al. 2006, Ma 2011). Acidized water glass could achieve a higher selectivity between fluorite and gangue minerals (Zhou et al. 2013, Zhou et al. 2013, Yang et al. 2016). Bo et al. (Bo et al. 2015) observed that acidified sodium silicate exhibited selective depression for calcite, and allowed preferential separation of scheelite from calcite. Zhang et al. (Zhang et al. 2009, Zhang et al. 2011) found that sodium silicate exhibits no significant effect on the adsorption of sodium oleate on an ilmenite surface, whereas it significantly decreased the adsorption of sodium oleate on a titanaugite surface, thus facilitating the flotation separation of ilmenite and titanaugite. Deng et al. (Deng et al. 2010) also found that sodium silicate achieved a significant selectivity in the separation of ilmenite and titanaugite.

If a titanium concentrate with a low grade of titanium dioxide is to be more useful, carrying out further cleaning of the titanium rough concentrate is necessary. Many studies have explored different roles of sodium silicate in the recovery of titanium minerals (Deng et al. 2010, Yang et al. 2016). However, the role of sodium silicate in the depth cleaning of titanium rough concentrate has rarely been studied. In this study, closed-circuit flotation tests were conducted, and the function and mechanism of sodium silicate in the cleaning process of ilmenite rough concentrate were estimated using adsorption measurements, Zeta potential measurements, and infrared spectroscopy analysis.

2. Materials and methods

2.1. Materials

The ilmenite used in the flotation test was provided by Panzhihua Iron and Steel (group) Co., Ltd. in China. The ilmenite rough concentrate was obtained by magnetic separation and gravity concentration from the crude ilmenite ore. The results of multi-element analysis of the ilmenite rough concentrate are shown in Table 1. X-ray diffraction analysis was performed to determine the mineral composition. The results showed that the valuable mineral was ilmenite with a content of 83.4%; the gangue minerals were titanaugite and a modicum of amesite with contents of 12.3% and 4.3%, respectively.

Table 1. Results of multi-element analysis of ilmenite rough concentrate

Element	TiO_2	TFe	MgO	CaO	SiO_2	Al_2O_3	S	P
Content(%)	46.78	30.78	5.99	0.59	3.14	1.62	1.69	<0.01

Ilmenite and titanaugite, which were used to study the functional mechanism of sodium silicate, were obtained from the ilmenite rough concentrate by repeated separation and purification using magnetic separation and a table concentrator. The purities of ilmenite and titanaugite were 98% and 89%, respectively.

2.2. Flotation test

A single slot flotation machine, with a volume of 1.5 L, was used for the bench-scale flotation test. All flotation experiments were carried out at 25 °C. For each flotation test the following procedure was followed: 500 g of ilmenite concentrate was placed in the flotation cell, and an appropriate amount of distilled water was added with stirring for 1 min; a pH regulator was added with stirring for 3 min; the depressant agent was added with stirring for 3 min; the collector was added with stirring for 3 min; the frother was added with stirring for 1 min; and finally, the resulting mixture was aerated for 5 min. The dried froth product was employed to assay for the determination of the grade of the titanium dioxide. The regime of agent, the flowsheet, and the flotation time were determined according to the grade and the recovery of titanium dioxide in the concentrate. The closed-circuit flotation tests were conducted based on the above optimal conditions.

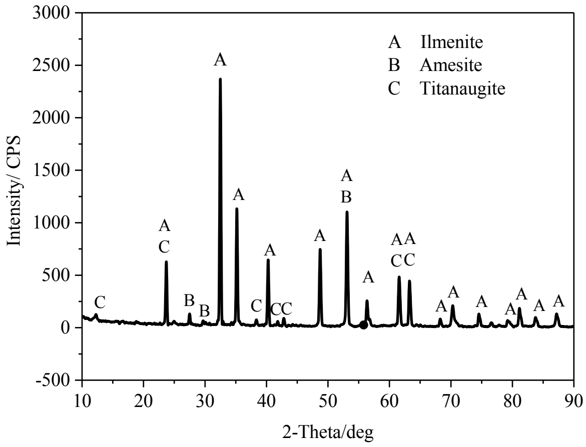

Fig. 1. XRD pattern of ilmenite concentrates

2.3. Adsorption measurement

An ultraviolet-visible spectrophotometer (UV-752, Shanghai, China) was employed to measure the absorbance of the residual agent. The relevant concentration of the residual agent was calculated using standard curves, and then the adsorption of the agent on the mineral surface was calculated from the original concentration. 2 g of the sample was weighed and placed in a 200-mL conical flask, and 40 mL of distilled water was added. The resulting sample was oscillated for 1 h in the oscillator, and thereafter centrifuged to realize the solid-liquid separation. Finally, the supernatant was collected for absorbance measurement.

2.4. Zeta potential measurements

Zeta potential was determined on a Zetasizer Nano Zs 90 (Malvern Instruments Co., Worcestershire, UK). The procedure for the Zeta potential measurement was as follow. The sample was first ground to fine particles of less than 2 μm. 100 mg of the powder sample was placed into a beaker, and 60 mL of deionized water was added. Then, the pH of the solution was adjusted with H_2SO_4 or NaOH to either acidic or basic conditions. Thereafter, various chemical reagents were added in the same pulp conditions as above in the flotation test. The sample was stirred for 12 min with a magnetic stirrer, and the Zeta potential was measured.

2.5. Infrared spectroscopic analysis

Infrared spectroscopic analysis was performed by a Fourier transform infrared spectrometer (Nicolet NEXUS 670, Madison, USA), using the KBr squash technique, in the measurement range 4000–500 cm^{-1}. 2 g of the sample was weighed and placed in the flotation cell. The resulting solution was collected for centrifugation for 30 min to obtain the solid-liquid separation. The liquid phase was separated, and the solid mineral was washed twice with distilled water and dried under a temperature of 60 °C, and the dried sample was used to conduct the infrared spectroscopic analysis.

3. Results and discussion

3.1. Flotation tests

The effect of the dosage of sodium silicate on the TiO_2 grade and recovery is shown in Fig. 2. The result shows that at first, the TiO_2 grade increased as the dosage of sodium silicate increased and then tended to flatten out. The TiO_2 grade reached up to 50.7% when the dosage of sodium silicate was 200 g/t. As the dosage of sodium silicate exceeded 200 g/t, it had little effect on the TiO_2 grade. The TiO_2 recovery decreased slightly with an increasing dosage of sodium silicate and was maintained in the range 57.2%–59.5%. Therefore, the dosage of sodium silicate exhibited a remarkable influence on the TiO_2 grade and the higher the dosage of sodium silicate, the higher the TiO_2 grade and the lower the TiO_2 recovery. Sodium silicate had an obvious depression effect on the gangue minerals, while the depression effect on ilmenite was inconspicuous.

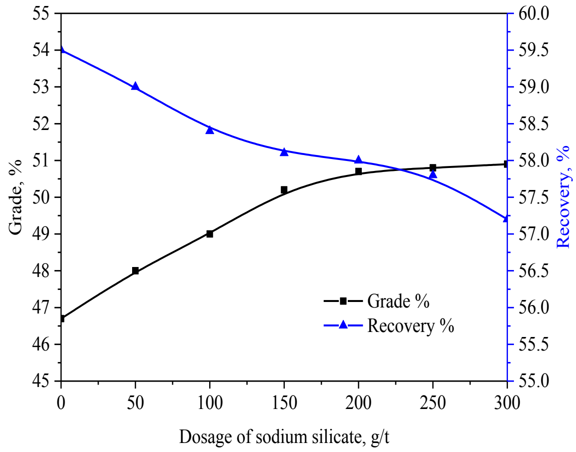

Fig. 2. Effect of the dosage of sodium silicate on the TiO_2 grade and recovery

The closed-circuit flotation test was carried out under the optimal agent conditions to improve the recovery of ilmenite. The closed-circuit flotation flowsheet is shown in Fig. 3 and the flotation result is shown in Table 2. When a depth cleaning process, such as one roughing-three cleaning-one scavenging, was adopted, the TiO_2 grade in the concentrate was improved from 46.78% to 50.37%, while the TiO_2 recovery increased to 77.0%. In addition, a small part of the titanium concentrate (Con. II) was obtained with a TiO_2 content of 41.94% and a corresponding TiO_2 recovery of 15.2%, which can be used as a raw material in titania pigment production through a sulphate process after compounded, with a modicum of high TiO_2 grade titanium concentrate.

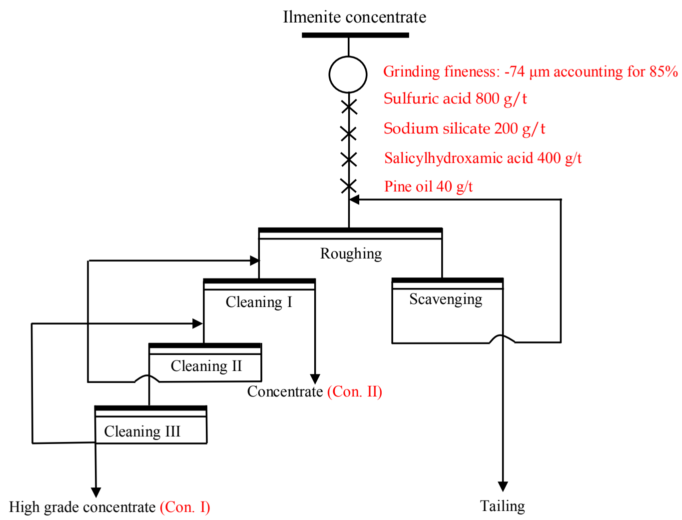

Fig. 3. Closed-circuit flotation flowsheet of ilmenite rough concentrate

Table 2. Result of closed-circuit flotation test

Product Name	TiO_2 Recovery/%	TiO_2 Grade/%
Con. I	77.00	50.37
Con. II	15.20	41.94
Tailing	7.80	24.09
Total	100.00	46.78

3.2. Effect of sodium silicate on absorption of collector

The adsorption of salicylhydroxamic acid on the ilmenite and gangue mineral surfaces, before and after the addition of sodium silicate, were compared using 50 mg/L of sodium silicate and 0.2 mmol/L of salicylhydroxamic acid. During the absorption measurements, the pH of the solution was maintained in the range 5.5–6.5. The main mineral component of the gangue minerals in the experiments was titanaugite, so the gangue mentioned below is referred to as titanaugite. The adsorption measurement results of the collector on the mineral surface are shown in Table 3. The effect of sodium silicate on the absorption of the collector is also shown in Table 3.

Table 3. Adsorption measurement results of the collector on the mineral surface

Sample name	Adsorption capacity (mg/g)
Ilmenite	0.1783
Ilmenite+Na_2SiO_3	0.1621
Titanaugite	0.0716
Titanaugite+Na_2SiO_3	0.0248

As shown in Table 3, the adsorption capacity of salicylhydroxamic acid on both the ilmenite and titanaugite surfaces decreased after sodium silicate was added. The difference is that for ilmenite, the adsorption capacity of the collector slightly decreased after sodium silicate was added, while for titanaugite, the adsorption capacity of the collector decreased sharply, from 17.92% to 6.21%. The addition of sodium silicate significantly decreased the adsorption of salicylhydroxamic acid on the gangue surface. Therefore, the selectivity of the collector was strengthened by sodium silicate in the further cleaning process of the ilmenite rough concentrate. The competitive adsorption capacity of salicylhydroxamic acid on the gangue surface was weakened; therefore, a separation between ilmenite and the gangue was facilitated.

3.3. Zeta potential measurements

Fig. 4 shows the Zeta potentials of ilmenite in the presence and absence of sodium silicate as a function of pH. Additionally, the effects of SHA on the Zeta potentials of ilmenite in the presence of sodium silicate as a function of pH were studied. The concentrations of sodium silicate and SHA were 50 mg/L and 0.2 mmol/L, respectively. In the absence of sodium silicate, the isoelectric point (IEP) of ilmenite was found to be pH 5.8, which was in agreement with a previous report (Yang et al. 2016).

After treatment with sodium silicate, the IEP of ilmenite became more negative and the IEP pH changed to 3.6. The addition of sodium silicate could decrease the Zeta potential values over the entire pH range. Zeta potentials changed more significantly with the addition of 0.2 mmol/L SHA. The Zeta potential of ilmenite pretreated with sodium silicate and SHA in sequence was lower than that of ilmenite directly treated with sodium silicate. The IEP did not occur within the studied pH range, which indicated the specific adsorption of SHA on the ilmenite surface. The sodium silicate did not prevent the adsorption of SHA on ilmenite.

The Zeta potentials of titanaugite in the presence and absence of sodium silicate as a function of pH were examined. Additionally, the effects of SHA on the Zeta potentials of titanaugite in the presence of sodium silicate as a function of pH are shown in Fig. 5. The IEP of titanaugite occurred at pH 3.6.

Titanaugite samples were conditioned with 50 mg/L sodium silicate before SHA addition into the suspensions. Fig. 5 shows that the negative Zeta potentials increased in magnitude when sodium silicate was added. This arose from the strong specific adsorption of sodium silicate on titanaugite surfaces, as indicated by the disappearance of the IEP value in the studied pH range. Meanwhile, the negative change in the Zeta potential of titanaugite was more obvious than that of ilmenite.

However, when the titanaugite samples were conditioned in a 0.2 mmol/L SHA solution with pretreatment of sodium silicate, the addition of SHA did not make the Zeta potential more negative, which indicated that sodium silicate had an obvious depression effect on titanaugite. By comparing Fig. 4 and Fig. 5, it can be seen that the addition of sodium silicate hindered the adsorption of SHA on the surface of titanaugite but had little effect on ilmenite, which further indicated that sodium silicate is a selective depressant for titanaugite. That is to say, sodium silicate still exhibits a good selective depression effect in the process of the further purification of the ilmenite rough concentrate.

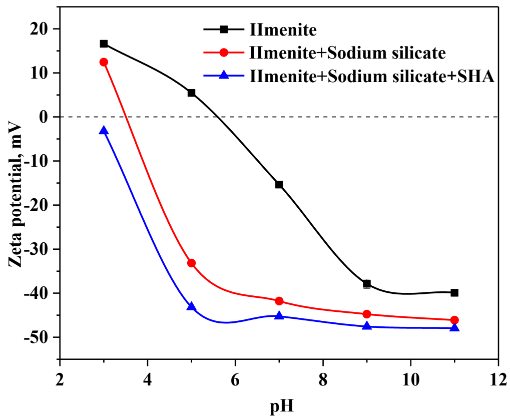

Fig. 4. Zeta potentials of ilmenite as a function of pH

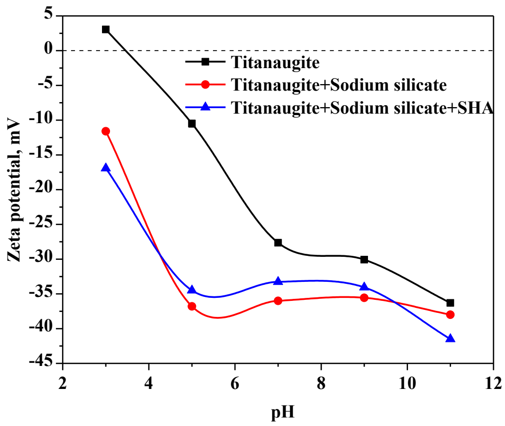

Fig. 5. Zeta potentials of titanaugite as a function of pH

3.4. Infrared spectroscopy analysis

The FTIR spectra of ilmenite and titanaugite before and after conditioning with sodium silicate to determine the adsorption mechanism of sodium silicate on ilmenite and titanaugite are shown in Fig. 6 and Fig. 7. The main functional groups contained in sodium silicate were –OH, -ONa and Si-O-Si, and their absorption peaks were reflected in the infrared spectra in the regions of 3600–2900 cm^{-1}, 1600–1400 cm^{-1} and 1100–900 cm^{-1}, respectively (Xu 2008).

It can be seen from Fig. 6 that the spectrum of ilmenite showed no obvious characteristic peak in the region from 750 to 4000 cm^{-1}. The results illustrated that sodium silicate cannot undergo chemical adsorption on the ilmenite surface in a weak acidic condition of pH 5.5. The IR spectrum of titanaugite interacting with sodium silicate is shown in Fig. 7. Compared with the spectrum of titanaugite, the spectrum of titanaugite treated with sodium silicate exhibited characteristic absorption peaks of sodium silicate. The new peaks at 3600–2900 cm^{-1}, 1600–1400 cm^{-1}, and 1100–900 cm^{-1} were attributed to changes caused by the addition of sodium silicate. Hence, it was inferred that sodium silicate might react with the titanaugite surface.

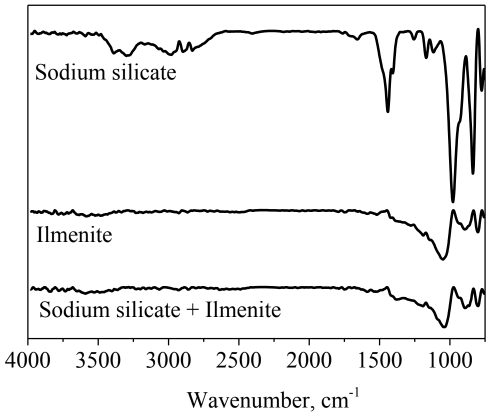

Fig. 6. FTIR spectra of ilmenite before and after conditioning

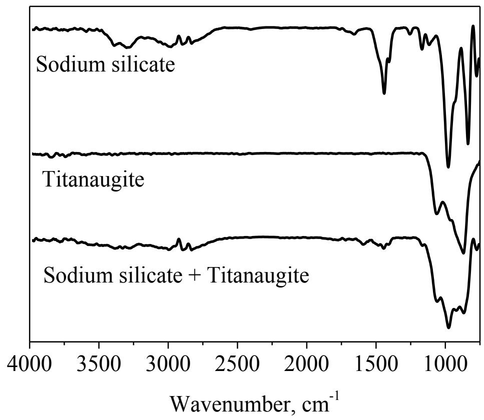

Fig. 7. FTIR spectra of titanaugite before and after conditioning

4. Conclusions

(1) An ilmenite concentrate with TiO_2 grade of 50.37% can be achieved from 46.78% according to the stages on the flowsheet—one roughing, three cleaning and one scavenging—under the optimal conditions, and also reflects the advantages of the excellent selectivity of sodium silicate.

(2) Sodium silicate can hinder the adsorption of SHA on the titanaugite surface but has almost no effect on ilmenite.

(3) Adsorption of sodium silicate can decrease the Zeta potentials of ilmenite and titanaugite. However, the negative change in the Zeta potential of titanaugite was more obvious than that of ilmenite. After adding SHA, the zeta potentials of titanaugite were shifted positively, which indicated that sodium silicate has a depression effect on titanaugite but not on ilmenite. As shown by the FTIR analysis, sodium silicate undergoes chemical adsorption on the titanaugite surface but not on the ilmenite surface, which indicates that a selective depression effect of sodium silicate on the gangue mineral facilitates the separation of ilmenite and gangue mineral in the cleaning process of ilmenite rough concentrate.

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