



Low-temperature performance of cationic collector undecyl propyl ether amine for ilmenite flotation



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ABSTRACT

When conventional anionic collectors (e.g., fatty acids) are used in flotation separation of ilmenite and titanaugite, a high flotation temperature (> 293 K) is generally required. However, it is crucial to develop low-temperature flotation processes for use in cold regions. This study demonstrates that the cationic collector undecyl propyl ether amine (UPEA) has outstanding collecting ability, selectivity, and low-temperature performance for ilmenite ore flotation. Various important factors that influence flotation performance, such as pH, UPEA dosage, and pulp temperature, were investigated by microflotation tests. Compared with a higher temperature (295 K), superior flotation recovery was obtained by closed-circuit flotation of actual ore at 279 K. Zeta potential measurements illustrated that large amounts of UPEA were selectively adsorbed on the ilmenite surface. Thermodynamic calculations and Fourier transform infrared analyses showed that adsorption of UPEA onto both mineral surfaces involved a physisorption process. Moreover, increasing the thermodynamic energy of UPEA molecules diminished interactions between UPEA and the minerals, resulting in decreased adsorption of UPEA with increasing temperature, as confirmed by adsorption analysis. The excellent low-temperature flotation performance of UPEA is consistent with the energy conservation policy of China, and the use of UPEA should be promoted in cold regions. This work provides new insights into the efficient separation of ilmenite from titanaugite at low temperatures.

1. Introduction

Ilmenite (FeTiO₃) is the major TiO₂-bearing mineral. The Panzhihua region (Sichuan, China) has large reserves of ilmenite ore (Jia et al., 2014; Li et al., 2006; Zhao et al., 2014; Zhou et al., 2013). Mineral processing by flotation is conventionally used to separate ilmenite from titanaugite. However, ilmenite and the main silicate gangue minerals, including titanaugite, generally have similar surface properties (Yang et al., 2016; Zhu et al., 2011). For instance, the presence of calcium, magnesium, titanium, iron, and other metal ions on both mineral surfaces increases the difficulty of achieving selective flotation.

Conventional anionic collectors (e.g., sodium oleate, NaOL) are the most widely used collectors in ilmenite and titanaugite flotation (Akbar et al., 2015; Yang et al., 2016; Zhong and Cui, 1987; Zhu et al., 2011). The NaOL collecting mechanism is generally thought to involve chemisorption of oleate ions onto the mineral surface (Fan and Rowson, 2000). However, anionic surfactants have some apparent drawbacks as collectors; for example, either larger dosages at low temperatures or conventional dosages at relatively high temperatures (e.g., > 293 K)

are generally required, which increases production costs, especially in cold areas. The effect of temperature on anionic collector flotation (e.g., when using NaOL) has been frequently reported, and is attributed to the solubility and solution chemistry of surfactants, both of which are temperature-dependent. The solubility and activity of fatty acids in aqueous solution are reduced at low temperatures, reducing their collecting capability (Castro and Borrego, 1996; Ejttemaei et al., 2011).

Other studies have reported the use of cationic collectors for mineral flotation. Examination of the cationic collector dimethyl-dodecyl-ammonium bromide by Huang et al. (2014) revealed that it was adsorbed onto quartz and magnetite surfaces by electrostatic attraction. Ge et al. (2004) and Li et al. (2017) examined amine and octadecylamine hydrochlorides as cationic collectors for magnetite and KCl flotation at lower temperatures (< 288 K) and obtained outstanding flotation results; however, in-depth investigations of the adsorption mechanisms were not conducted. Liu et al. (2015) showed that the cationic collector undecyl propyl ether amine (UPEA) performed well in microflotation tests of ilmenite and titanaugite. Nevertheless, the low-temperature flotation performance and adsorption mechanism of UPEA onto

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Table 1
Multielement chemical analysis of ilmenite ore.

Component	TiO ₂	FeO	Fe ₂ O ₃	CaO	MgO	Al ₂ O ₃	SiO ₂
wt.%	20.4	24.7	3.5	7.2	5.6	5.9	29.4

ilmenite remain poorly understood.

The aim of the present study was to examine the performance of UPEA for ilmenite ore flotation at temperatures below 283 K and determine the underlying adsorption mechanism of UPEA on ilmenite. Microflotation tests, mixed mineral and actual ore flotation tests, zeta potential measurements, Fourier transform infrared (FTIR) analyses, adsorption analyses, and thermodynamic calculations were undertaken to provide new insight into the efficient separation of ilmenite from titanite at low temperatures.

2. Materials and methods

2.1. Mineral samples

The ilmenite ore used in this study was obtained from Panzhihua Iron & Steel (Group) Co., Sichuan Province, China as the rough concentrate product of high-intensity electromagnetic separation. Multielement chemical analysis (Table 1) revealed that the ore contained 20.4 wt% TiO₂ and 29.4 wt% SiO₂. Mineralogical examination using high-power microscopy confirmed that titanite was the predominant gangue mineral in this ore sample.

High-purity ilmenite was obtained by repeated high-intensity magnetic separation using an Eriez magnetic separator (CF-5MM, USA). Purified titanite was obtained from the magnetic separator tailings using a table concentrator (Liu et al., 2015). Chemical analyses of the purified samples were conducted by atomic absorption spectroscopy (AAS) and inductively coupled plasma mass spectrometry (ICP-MS). As shown in Table 2, purified ilmenite contained 50.6 wt% TiO₂ and both ilmenite and titanite were > 90% pure.

The results reported in Tables 1 and 2 are the averages of triplicate chemical analyses (measurement error = ± 5%). The purified samples were dry ground in a ceramic ball mill and subsequently soaked in a 0.05 wt% hydrochloric acid solution for 30 min. Then the samples were naturally dried at ~308 K for about 24 h. The -150 + 45 μm fraction was used in microflotation and adsorption tests, whereas the -20 μm fraction was used for the zeta potential measurements and FTIR analyses.

2.2. Reagents

Sulfuric acid (H₂SO₄) and sodium hydroxide (NaOH) were used as pH regulators. As Liu et al. (2015) reported that oxalic acid (H₂C₂O₄) selectively depresses titanite, H₂C₂O₄ was used as the depressant in the present study. UPEA and NaOL were used as collectors. UPEA was of chemical grade and other reagents were of analytical grade. Tap water was used for flotation of the actual ore, whereas deionized water (resistivity = 18.1 MΩ·cm) was used in all other experiments.

2.3. Microflotation tests

Microflotation tests were conducted using an XFG-II mechanical

Table 2
Multielement chemical analyses of purified ilmenite and titanite (wt.%).

Component	TiO ₂	Fe ₂ O ₃	FeO	CaO	MgO	Al ₂ O ₃	SiO ₂	MnO ₂	S
Ilmenite	50.6	9.0	33.5	1.1	3.6	0.6	1.0	0.3	0.03
Titanite	1.7	1.5	8.3	17.1	13.5	9.4	44.1	0.3	0.09

agitation flotation machine (Jilin Prospecting Machinery, China) (Liu et al., 2015; Shi et al., 2013; Yang et al., 2016; Zhu et al., 2011).

For the single-mineral flotation tests, a mineral suspension was prepared by adding 5.0 g of ilmenite (or titanite) to 80 mL of solution. In the mixed-mineral flotation tests, 2.5 g of ilmenite and 2.5 g of titanite were added to 80 mL of solution; the grade of the mineral mixture was 26.0–26.5 wt% TiO₂. H₂SO₄ or NaOH solution was added as required to adjust the pH to the desired value. The suspension was stirred for 3 min, H₂C₂O₄ was added (if needed) and stirred for 3 min, and then the collector was added and stirred for 3 min. The flotation was conducted for 3 min. The concentrate (or tailing) was collected, filtered, dried, and weighed. Flotation recovery was calculated from the distribution of the solid weight of the two products. In the mixed-mineral tests, the grades of the two products were evaluated by chemical analysis.

All microflotation tests, except temperature-based experiments, were carried out at 293 K. All presented results are the averages of duplicate flotation tests; the standard deviations were calculated using Origin 8.0 software (OriginLab Corporation).

2.4. Grinding and flotation of actual ore

In the batch flotation tests, 250 g of ilmenite ore was ground to -74 μm 40 wt% in an XMB-70 grinding mill (Wuhan Prospecting Machinery, China) with low-carbon alloy steel rods as the grinding media. The pulp was transferred into an XFD-0.75 laboratory flotation machine (Jilin Prospecting Machinery, China) and stirred at 1500 rpm for 3 min. Subsequently, H₂SO₄ was added and the mixture was stirred for 2 min. Then, H₂C₂O₄ was added and the mixture was stirred for 2 min. Finally, UPEA was added and the mixture was stirred for a further 2 min. All reagents were added and the pulp was conditioned without aeration. Flotation was conducted for 3 min. Batch flotation tests were carried out in triplicate under identical conditions. The reported results are the average values, and the standard deviations were calculated using Origin 8.0 software.

Fig. 1 shows the closed-circuit flowsheet and corresponding conditions for ilmenite flotation. The closed-circuit flotation system reached equilibrium after six cycles. The reported yields for the final concentrates and tailings are average values for the last three cycles (cycles six to eight), and the experimental errors for the yields are < 4%. The products were filtered, dried, weighed, and analyzed to determine TiO₂ grade.

2.5. Zeta potential measurements

Suspensions for zeta potential measurements were prepared as described for the microflotation tests in Section 2.3. As the concentration of KCl was fixed at 150 mg/L to maintain ionic strength, the double layer thickness was constant (Liu et al., 2015). After adjusting the pH to the desired value using NaOH or H₂SO₄, UPEA was added. The suspension was agitated with a magnetic stirring apparatus for 10 min. The supernatant was removed and used for zeta potential measurements in a JS94H microelectrophoresis instrument (Shanghai Zhongchen Electronic Technology, China) at 293 K. The presented results are the averages of triplicate measurements, and the standard deviations were calculated using Origin 8.0 software.

2.6. FTIR measurements

FTIR spectra were recorded using a Nicolet G510P instrument (USA) using KBr disks. The pH was adjusted to the desired value using H₂SO₄, and then UPEA was added, if required. The resulting suspension was agitated with a magnetic stirring apparatus for 30 min. All the solution conditioning processes were carried out at 293 K. The samples were naturally dried at about 308 K for 48 h, and then the FTIR spectra were recorded.

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