

The activation mechanism of lead ions in the flotation of ilmenite using sodium oleate as a collector



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ABSTRACT

The flotation behaviors of ilmenite untreated or treated with lead ions using sodium oleate as a collector were studied. The results of micro-flotation experiments reveal that ilmenite shows good floatability at pH 6–8. Compared with the flotation recovery of ilmenite untreated with lead ions, ilmenite floatability can be enhanced, and the resulting increase in recovery can surpass 30% after the addition of lead ions. The results of zeta potential tests show that the isoelectric point of ilmenite can change from 5.2 (untreated) to 7.8 (treated), which is beneficial for the interaction between ilmenite and the collector and increases its floatability. Adsorption measurements show that the adsorption amounts of ilmenite treated with lead ions are higher than those of untreated ilmenite. XPS tests show that after activation with lead ions, the relative contents of iron and titanium on the ilmenite surface, which play leading roles in ilmenite flotation, increase by 5.9% and 4.6%, respectively. At the same time, changes in binding energy are more visible after ilmenite is pretreated with lead ions and subsequently treated with sodium oleate. Within the optimal flotation pH range, oleate species (RCOO^- , $(\text{RCOO})_2^{2-}$, $(\text{RCOO})_2\text{H}^-$) can mainly react with iron on the ilmenite surface through chemical adsorption in addition to electrostatic effects.

1. Introduction

The processing of ores containing rutile and ilmenite, which are the major sources of titanium, has been widely studied for decades (Eidsmo and Mellgren, 1960; Bulatovic and Wyslouzil, 1999; Amer, 2002). Because of its high chemical activity, titanium is always combined with other elements, most commonly oxygen. Additionally, some iron atoms often substitute for titanium in the crystal lattices of ilmenite or rutile, which creates difficulties in the flotation separation of ilmenite from other gangue (Didenko and Efremov, 2004; Deer et al., 1992). As shown in Fig. 1, $\text{TiO}_2\text{-FeO-Fe}_2\text{O}_3$ can form an infinite solid solution. When the proportion of any one of TiO_2 , FeO , or Fe_2O_3 changes, many types of mineral-phases can be formed.

In China, rutile reserves are lower than ilmenite reserves, primarily due to the high consumption of the former in the past. Therefore, it is essential to develop viable means of exploiting and utilizing ilmenite. The Panzhihua area possesses the largest deposits of vanadium-titanium magnetite, with characteristic low-grade and complex mineral components (Zhou et al., 2013; Zhao and Zhou, 2007). Generally, the separation of ilmenite from gangues such as titanite, hematite and forsterite is conducted via high-intensity magnetic separation, gravity separation, and electrical separation, among other methods (Mehdilo

et al., 2015; Zhu et al., 2011; Cui et al., 2002). Nevertheless, the separation results show that these methods do not work as efficiently as expected. Flotation technology has rapidly developed in recent years and provides a new method for enhancing ilmenite beneficiation. Many improvements have made it more efficient.

One of the most important improvements is surface modification, which has many advantages such as adjustment of wettability, surface chemical properties, bio-compatibility, and nanofabrication (Zorn et al., 2005; Foster et al., 2006; Quiñones et al., 2008). Currently, it has been widely applied in mineral engineering, chemistry, material science, and catalysis, and its major functions are selective flotation, anticorrosion and deaggregation (Parikh and Chorover, 2006; Klepser and Bartlett, 2014; Fainerman et al., 2000). Surface modification during mineral processing typically entails activating or inhibiting the surface of useful minerals or gangues, and the corresponding reagents are termed activators or inhibitors. In mineral engineering, surface modification mainly increases or decreases the number of active sites on the mineral surface, which is vital for the adsorption of reagents onto minerals (Li et al., 2016a, 2016b).

In general, two methods are utilized to increase the number of surface-active sites. The first is to change the valence state or structure of the present surface elements. This can be achieved using surface

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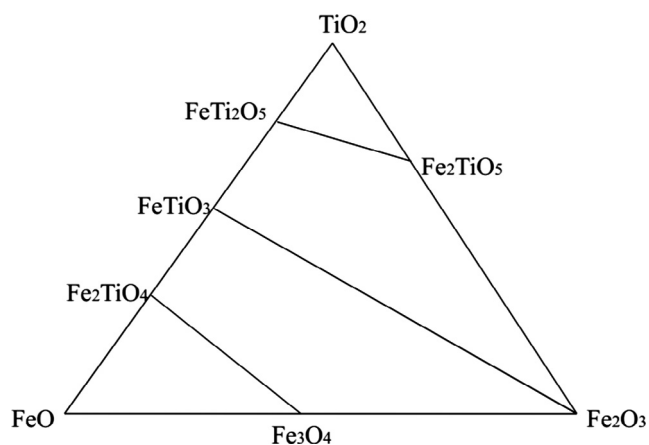


Fig. 1. Three-dimensional phase diagram of the system $\text{TiO}_2\text{-FeO-Fe}_2\text{O}_3$.

dissolution methods, microwave irradiation, oxidation roasting, etc. (Welham and Llewellyn, 1998; Cutmore et al., 2000; Zhang and Ostrovski, 2002). This method usually produces a fresh surface, which is more active in adsorbing reagents such as collectors. The other way to increase the number of surface-active sites is to introduce new active elements to the mineral surface. Generally, ions are introduced via their adsorption on the surface as a potential determining ion (pdi). Compared to a sample with no ions added, this can attract more reagents to improve the flotation behavior. Ions such as Cu(II) , Ca(II) , and Pb(II) have been extensively used as activators, especially in the flotation of sulfide minerals (Dávila-Pulido et al., 2015; Chandra and Gerson, 2009; Finkelstein, 1997). As an activator, Pb(II) has been used in the selective separation of scheelite from other calcium-containing minerals. A recent study (Zhao et al., 2015) revealed that lead ions can be adsorbed onto the surface of scheelite, and benzohydroxamic acid can coordinate with lead ions to enhance the flotation recovery of scheelite compared with calcium, manganese and iron ions.

The ability of Pb(II) to activate the flotation of ilmenite and rutile has been applied in industrial production for a long time (Fan and Rowson, 2000). However, it remains unclear how lead ions affect the flotation of ilmenite. The activation mechanism of lead ions on ilmenite has not been studied systematically. The present work studies the role of lead ions on the flotation behavior of ilmenite using microflotation, zeta potential, adsorption measurement and XPS analysis. Compared to the study by Fan and Rowson (2000), adsorption measurement and XPS analysis are employed to further reveal the activation mechanism of lead ions on ilmenite in present study. Furthermore, solution species are discussed in detail to delineate the flotation behavior in studied system.

2. Materials and methods

2.1. Materials and reagents

Pure ilmenite samples were obtained from Panzhihua, Sichuan province, China. After being transported to the laboratory, the samples were hand-selected, crushed and ground in a porcelain mill with an agate ball. Then, the samples were purified through repeated high- and low-intensity magnetic separation and gravity separation. Following that, the powder samples were sieved, and the $-0.074 + 0.038$ mm size fraction was chosen for single-mineral flotation tests. The results of XRD and XRF are shown in Fig. 2 and Table 1. Based on the results of XRD and XRF it was determined that the purity of ilmenite is very high and meets the criteria for microflotation.

The reagents used in pure mineral flotation, such as sodium oleate, lead nitrate, H_2SO_4 and NaOH , were of analytical grade. Among these reagents, sodium oleate was obtained from Baisaiqin Chemical Technology Co., Ltd., Shanghai, China. Lead nitrate was supplied by Xiya Reagent Co., Ltd., Chengdu, China. The pH was adjusted using

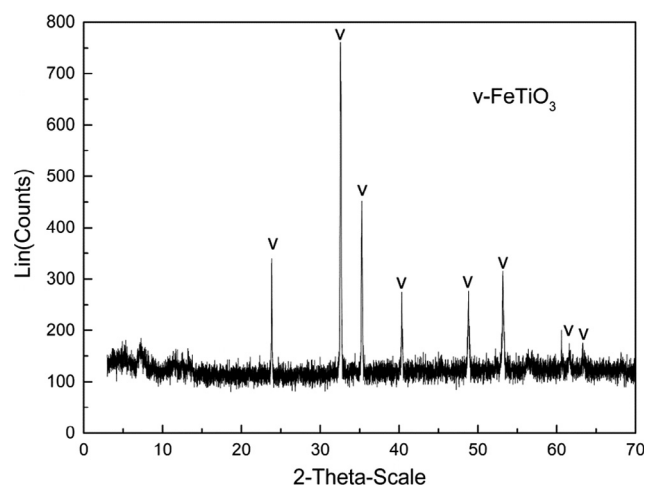


Fig. 2. XRD patterns of the purified samples.

Table 1

Main chemical compositions (wt.%) of the studied ilmenite sample (LE: other elements, mainly as O, Si, S, etc.).

Sample	Ti	Fe	Mn	V	Ca	Mg	Al	LE
Ilmenite	30.58	29.25	0.5	0.33	0.21	3.04	0.25	35.84

H_2SO_4 and NaOH stock solutions. Deionized water was used in the flotation and measurements (Resistivity = $18.3 \text{ M}\Omega \times \text{cm}$).

2.2. Flotation tests

Single-mineral flotation tests of ilmenite were conducted in a 40-mL plexiglass cell using an XFG-type flotation machine purchased from Prospecting Machinery Factory, Jilin, China, each using 2 g of mineral samples at a spindle speed of 1700 rpm. After the desired amount of reagents (pH modifier or collector) was added, the pulp was agitated for 3 min. After the pH of the suspension was adjusted and the collector was completely dispersed, a 3-min flotation period was used. The concentrate and tailing samples were filtered, dried, and weighed to calculate the flotation recovery under various flotation conditions using the following equation:

$$\varepsilon = m_1 / (m_1 + m_2) \times 100\%$$

where m_1 and m_2 are the weights of the concentrate and tailing, respectively. At least three tests were performed in each case, and the mean values and standard deviation were calculated. The calculation results were plotted and reported in the form of mean values \pm standard deviation in this paper.

2.3. Zeta potential measurements

To meet the requirements of particle size in zeta potential measurements, samples for these tests were ground to $-2 \mu\text{m}$. Each time, 16-mg samples were collected in the beaker, and then 80 mL of electrolyte solution ($\text{KNO}_3 = 0.01 \text{ mol/L}$) was poured to adjust the pulp; pH regulators such as H_2SO_4 and NaOH were used to adjust the pH. Next, the respective reagents were added in the order of lead nitrate (when activation occurred) and sodium oleate, and finally, the solution was stirred for 5 min using a stirrer and then allowed to settle for 5 min. The supernatant was used for zeta potential measurements using a Delsa-440sx Zeta meter (Malvern Instruments, UK). Each sample was tested three times, and the average value was taken as the final result. The standard deviation in each case was also calculated and reported.

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