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Study on the activation mechanism of lead ions in the flotation of ilmenite using benzyl hydroxamic acid as collector

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ABSTRACT

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Introduction

Titanium has many extensive applications including medicine, aerospace, function materials and catalytic industries due to its high strength-to-weight ratio and corrosion resistance [1,2]. Two titanium phases, titanium dioxide and metal titanium, are produced presently from rutile and ilmenite. Ilmenite is becoming the major raw material in titanium industry resulting from the excessive consumption of limited rutile resources [3,4]. In the mineral processing of ilmenite ore, gravity separation, highintensity magnetic separation (HIMS), electrostatic separation or a conjunction of them are widely adopted to separate titanium minerals from gangues based on the difference of their physical properties. But for ilmenite freely disseminated in the gangues, the mentioned methods cannot effectively separate ilmenite from associated gangue minerals. In this case, froth flotation, a physicochemical separation process, has been considered suitable for processing ilmenite ore [5–7].

In the flotation process, one of the critical factors is to selectively modify the mineral surface property and improve the hydrophobicity of a desired mineral. Organic surfactants have been widely used as collectors because they can anchor on the mineral surface and render the attachment of particles to air bubbles. Among all surfactants, sodium oleate (aliphatic acids) and succinamate are commonly used in the flotation separation of

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The activation mechanism of Pb^{2+} ions to ilmenite and the subsequent interaction with benzyl hydroxamic acid (BHA) were systematically investigated. The Pb^{2+} ions modification occurred via a chemical adsorption process, in which lead species interacted with iron hydroxyl compounds to form lead-containing complexes on the ilmenite surface. After lead ions activation, the surface of ilmenite became more active, and iron species and lead-containing complexes served as the main active sites to covalently bond with BHA in the form of metal-BHA chelate complexes. As a result, the BHA adsorption increased, giving rise to a concomitant increase in the floatability of ilmenite.

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ilmenite from siliceous gangues [3,8,9]. Successful separation of ilmenite could be achieved using benzyl arsenic acid (BAA) in the presence of acidified silicates, reported by Song and Tsai [10]. Phosphonic acid exhibits a strong affinity toward metal oxides, which is deemed to an impactful collector for the selective flotation of titanium minerals [7,11,12]. The mixed anionic/cationic collector shows more superior hydrophobicity and collecting property than an individual collector because of the remarkable synergistic effect, aiming to improve the flotation efficiency of ilmenite [13–15]. Hydroxamic acids and their salts have also been extensively studied and exploited as collectors due to the active chelation with some transition metal or rare-earth metal ions on mineral surfaces [16–18]. Relevant research using hydroxamate as the collector was reported in the flotation of titanium minerals [19].

Even if collectors are utilized to improve the hydrophobicity of mineral surface, ilmenite yet displays a poor flotation performance compared to rutile. It is because that only half of metallic ions (Ti⁴⁺ and Fe²⁺) on the ilmenite surface are serving as active sites and interact with anionic collectors at different pH ranges [5,20,21]. Therefore, considerable studies have been attempted to supply sufficient active sites for improving the flotation recovery of ilmenite. Surface oxidation, such as microwave irradiation, oxidation roasting, and surface dissolution, is discussed to enhance the ilmenite floatability, which converts ferrous ions to ferric ions in terms of increasing the adsorption of collectors on the ilmenite surface [22–24]. Surface activation by adding new active species on the mineral surfaces is another effective method in the flotation process. Lead ions have been investigated as activators for the

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flotation of sulfide and oxide minerals. As early as 1960, Rey and Formanek firstly reported lead ions as the activator in the sphalerite flotation [25]. Correspondingly, lead ions as the activator of oxide minerals were studied far behind that of sulfide, but they are widely utilized at a plant scale until now. Fan and Rowson reported that Pb²⁺ and Pb(OH)⁺ species of lead ions could selectively adsorb onto ilmenite surfaces and enhance its floatability when sodium oleate was used as a collector [20]. Chen provided further insights to reveal the activation mechanism of lead ions. They found that lead species formed lead-containing complexes on ilmenite surfaces, which served as the main active sites to bond with oleate species [26,27]. Lead ions as the activator combined with hydroxamate collectors have attracted a lot of enthusiasm to apply in the flotation of oxide minerals. Feng discovered that the lead species adsorbed on the cassiterite surface led to the increase in the number of active sites and offered adequate chances for the adsorption of salicyl hydroxamic acid [28]. The effect of lead ions on the flotation of rare earth minerals using benzyl hydroxamic acid and naphthalenic hydroxamic acid as collectors was investigated in virtue of TOF-SIMS analyses by Xia. Lead species reversed the surface charge of ilmenite and promoted the adsorption of collectors [29]. Yue et al. found that the Pb-BHA complexes exercised the synergistic effect between chemical and physical adsorption and facilitated to improve the flotation recovery of tungsten minerals [30]. Zhao reported the activation mechanism of lead ions in the flotation of scheelite and wolframite with benzyl hydroxamic acid by means of DFT and XPS analysis. Their results indicated that benzyl hydroxamic acid preferentially coordinated with Pb²⁺ ions compared with Ca²⁺, Mn² ⁺ and Fe²⁺ ions [31]. Nevertheless, the surface activation of ilmenite by lead ions with hydroxamate as the collector is hardly reported in the published literature.

In the present study, the positive influence of lead ions in the ilmenite flotation with benzyl hydroxamic acid (BHA) as the collector was investigated. The activation mechanism of lead ions on ilmenite surfaces and subsequent response to BHA adsorption were further discussed via adsorption tests, zeta potential measurements, FT-IR and XPS analysis.

Materials and methods

Materials

Ilmenite sample was obtained from the vanadium–titanium magnetite in Panzhihua Mine, Sichuan province, China. Mineral samples were repeatedly purified using low-intensity magnetic separation, high-intensity magnetic separation, and shaking table. The XRD result given in Fig. 1 demonstrated that the sample was composed of ilmenite with a trace of pyrite, and the XRF result presented that the titanium (TiO₂) content was 50.45%, indicating that the purity of ilmenite was above 95%. The purified ilmenite was dry-ground in a porcelain ball mill and sieved to obtain the $-74 \,\mu$ m fractions for the flotation experiments. Samples for measurements were further ground to less than 5 μ m in an agate mortar.

Benzyl hydroxamic acid (BHA), used as the anionic collector, was synthesized in laboratory through the reaction of benzoic acid with hydroxylamine. The element analysis showed that the BHA purity was above 95% after the purification process. Analytically pure lead nitrate ($Pb(NO_3)_2$), used as the activator, was supplied by Tianjin Kermil Reagent Co., Ltd., China. The pH value of aqueous suspensions was adjusted by sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) dilute solutions (1 mol L⁻¹). Analytical grade potassium chloride (KCl) was used as the background electrolyte for zeta potential measurements. All solution and suspension in

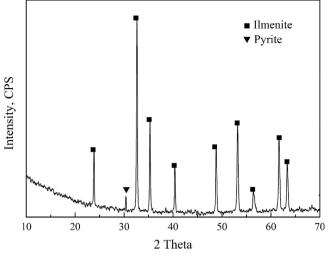


Fig. 1. X-ray diffraction spectra of ilmenite.

the experiments were prepared using deionized water (18.25 $\mbox{M}\Omega$ cm).

Microflotation experiments

Microflotation experiments were conducted in an XFG II flotation machine with a fixed stirring rate of 1800 rpm. 2 g ilmenite samples and 40 mL deionized water were used in each test. The suspension was firstly adjusted to the desired pH value by H_2SO_4 or NaOH solution for 2 min, and then the activator and collector were added one after another and conditioned for 3 min and 5 min, respectively. After that, terpenic oil was added as the frother. The flotation time was limited to 5 min, and the froth was scraped out every 10 s. The floated and sank fractions were filtered, dried, and weighed, respectively. The flotation recovery of ilmenite was calculated according to Eq. (1),

$$R = \frac{M_f}{M_f + M_s} \times 100\% \tag{1}$$

where *R* is the flotation recovery, M_f is the mass of the floated fractions (concentrates), and M_s is the mass of the sank fractions (tailings).

Adsorption tests

The adsorption amount of BHA on the ilmenite surface was determined by the UV1901PC UV–Vis spectrophotometer (China). BHA concentration was measured using UV absorbance at the wavelength of 520 nm according to the ferric hydroxamate method [32,33]. 1.0 g ilmenite sample was dispersed into 40 mL aqueous phase in the absence and presence of lead ions at a desired pH value and conditioned for 3 min. Sequentially, the BHA stock solution was added to interact with the mineral surface, and the mixing was stirred for 30 min. The conditioned mineral particles were separated out by centrifugation, and the BHA concentration of the separated supernatant was measured by the UV–Vis spectrophotometer. The adsorption amount was calculated by Eq. (2) based on the difference between the initial and final BHA concentrations.

$$Q = \frac{V(C_0 - C_f)}{M} \tag{2}$$

where, Q is the adsorption amount of BHA on ilmenite surfaces $(mg g^{-1})$, C_0 is the initial BHA concentration $(mg L^{-1})$, C_f is the final

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