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journal homepage: www.elsevier.com/locate/jiec1 Adsorption mechanism of lead ions at ilmenite/water interface and its
2 influence on ilmenite flotability

3 Qi Pan Chen, Jihua Zhai, Wei Sun*, Yuehua Hu*, Zhigang Yin, Xiangsheng Lai

4 School of Minerals Processing Bioengineering, Central South University, Changsha 410083, China

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ABSTRACT

In order to get further understanding of lead ions adsorption onto ilmenite surface, zeta potential analysis, adsorption density calculation, FT-IR and XPS analysis were employed. The results showed that the adsorption of lead ions onto ilmenite surface was a chemically dominating process. Lead species could interact with iron-hydroxyl complex compounds to form a Fe–O–Pb complex. The hydrophobic complex of Pb(OL)₂ was also observed. Iron and adsorbed lead ions on ilmenite surface served as the main active-sites via chemisorption with oleate species. Introducing lead ions, as a surface modification means, can efficiently improve ilmenite flotability.

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5 Introduction

6 Titanium industry has recently become more and more
7 important and attracting for its extensive applications including
8 medicine, navigation, aerospace, function materials and catalytic
9 industries [1–3]. In contemporarily industrial system, titanium
10 metallurgy is composed of mass production of titanium white,
11 titanium sponge and titanium metal using the rutile (TiO₂) and
12 ilmenite (FeTiO₃) as raw materials. Compared with ilmenite, rutile
13 is relatively easy to be utilized and extracted from raw ores. Duo to
14 the level limit of original technology and science, rutile is the main
15 object as the extraction of TiO₂ and most have run out. Therefore,
16 ilmenite extraction has become promising and many studies focus
17 on it [4–6]. However, ores containing ilmenite are recognized as
18 refractory resources for TiO₂ extraction because there are few
19 active sites on ilmenite surface [5,7]. Study showed that a series of
20 hydroxyl complex compounds of Fe or Ti on ilmenite surface
21 served as active sites within the pH range from 2 to 12. Fe or Ti
22 could not serve as active sites at the same time, resulting in less
23 active sites on ilmenite surface [8]. Besides, ilmenite and its gangue
24 like titanite have similar active sites on the surface, which is
25 another contribution to the difficult extraction of ilmenite. Prior to
26 ilmenite hydrometallurgy, pre-enrichment of TiO₂ grade from low-
27 grade raw ores is a necessary procedure. In mineral processing,

28 magnetic separation, gravity concentration and flotation are
29 usually combined to the pre-enrichment of TiO₂ grade due to its
30 refractory characteristics [9,10]. Fatty acid is used as the popular
31 collector to modify the physicochemical properties of mineral
32 particles [11,12].

33 Surface modification is recognized as a meaningful tool to the
34 adjustment of biocompatibility, wetting and adhesion, fabrication,
35 etc. [13,14]. For ilmenite, surface modification methods such as
36 microwave irradiation, oxidation roasting and surface dissolution
37 have been previously documented and discussed to improve its
38 flotability [11,15,16]. These methods were all confirmed to have an
39 increase in ferric abundance on ilmenite surface after surface
40 modifications. Because the ferric species could form more
41 insoluble layers on ilmenite surface and increased the chance of
42 ilmenite particles to capture by air bubbles, ilmenite flotability was
43 improved [7].

44 Introducing ions, as a pretreatment method of surface
45 modification, is also attracting in minerals processing. The
46 mechanism during the activation of metal ions on various minerals
47 is always one of the hottest topics and frequently reported.
48 Sphalerite could be activated by copper ions and it was first
49 discovered by Bradford in 1911 [17]. Besides, the activation
50 mechanism of calcium on sphalerite using xanthate as the
51 collector was systematically discussed by Dávila-Pulido et al. in
52 2015 [18]. Liu reported the effects of Ca and Mg ions on spodumene
53 using NaOL as the collector through FT-IR and XPS analyses [19].
54 Lead ions, as an activator, were also reported by previous studies.
55 The activation of sphalerite by lead ions was first demonstrated by
56 Rey and Formanek in 1960 [20]. There were many subsequent

* Corresponding authors.

E-mail addresses: sunmenghu@csu.edu.cn (W. Sun), huyuehuacsu@126.com (Y. Hu).

investigations to reveal the mechanism in this activation system [21–23]. Patrick et al. [21] summarized two mechanisms for the activation between lead ions and minerals. One is the formation of Me–O–Pb complex and the other is the formation of PbS(O) via ion exchange. For oxide minerals, lead ions could be used as the activator and were reported by some literatures. Xia reported the activation mechanism of lead ions on rare earth minerals [24]. DFT calculation and XPS measurements were used by Zhao to further reveal the activation mechanism of scheelite and wolframite by lead ions [25]. Feng et al. reported the activation mechanism of lead ions in cassiterite flotation using salicylhydroxamic acid as collector [26]. Even so, it could be clearly seen that studies on the activation mechanism of lead ions on oxide minerals are far behind the corresponding studies on sulfide minerals. More fundamental researches are still necessary to consummate the activation theory. The effect of lead ions on ilmenite flotation was first reported by Fan and Rowson in 2000 [8]. However, the mechanism of lead ions activation on ilmenite has not received further discussions and the adsorption behavior of lead ions at ilmenite/water interface is still unclear.

In present study, the adsorption of lead ions onto ilmenite surface and its influence on the adsorption of sodium oleate (the collector) were further discussed via zeta potential analysis, adsorption density calculation, FT-IR and XPS analysis. A potential reaction mechanism was established, hoping to provide further insight into the adsorption of lead ions at ilmenite/water interface.

Materials and methods

Materials and reagents

Pure ilmenite samples were obtained from Panzhihua, Sichuan province, China. The minerals were ground in a porcelain mill with an agate ball. Following that, the products were sieved, and the samples of $-0.074+0.038$ mm size fraction were used for the micro-flotation experiments. The results of XRF showed that the grade of titanium element was 30.58%, showing that the purity of ilmenite was above 90%. Fig. 1 gives the XRD pattern of pure ilmenite samples used in the study, indicating that the main mineral-phase is ilmenite. On the basis of the results of XRF and

XRD, it was determined that the purity of ilmenite was very high and met the requirements of micro-flotation.

Analytical grade sodium oleate was used as the collector in the flotation and supplied by Baisaiqin Chemical Technology Co., Ltd., Shanghai, China. Analytically pure lead nitrate was supplied by Xiya Reagent Co., Ltd., Chengdu, China. The pH was adjusted using sodium hydroxide and sulphuric acid stock solutions. Deionized water (Resistivity = $18.3 \text{ M}\Omega \times \text{cm}$) was used in the whole experiments.

Flotation tests

A flotation machine of XFG-1600 type (mechanical agitation) with the volume of 40 ml was used in micro-flotation tests. The impeller speed was fixed at 1700 rpm. The mineral suspension was prepared by adding 2.0 g of single mineral to 30 ml of solutions in single mineral flotation tests. The pH of the mineral suspension was adjusted to the desired operating value by adding H_2SO_4 or NaOH stock solutions. The general reagent addition scheme involved activator and collector addition with each stage having a 2 min conditioning period prior to the next reagent addition. Flotation concentrates were then collected for a total of 3 min. The floated and unfloated particles were collected, filtered and dried. In single mineral flotation, the recovery was calculated based on solid weight distributions between the two products. In order to assess the accuracy of flotation tests, the errors of the recovery were found to be within 2.0% after at least three tests at each condition, and the average values were reported.

Zeta potential experiments

Zeta potential experiments were conducted using a Delsa-440sx Zeta meter (Malvern Instruments, UK). Zeta potential measurements for each mineral were carried out in 0.01 M KNO_3 electrolyte solution. A freshly-ground 0.05 g mineral sample ($-2 \mu\text{m}$) was dispersed in 500 ml of electrolyte solution and mixed for 5 min. The coarse mineral particles were allowed to settle and the supernatant containing the fine particles were transferred into a folded capillary cell, ensuring that there were no bubbles in the cell. The results presented here were the average of three independent measurements.

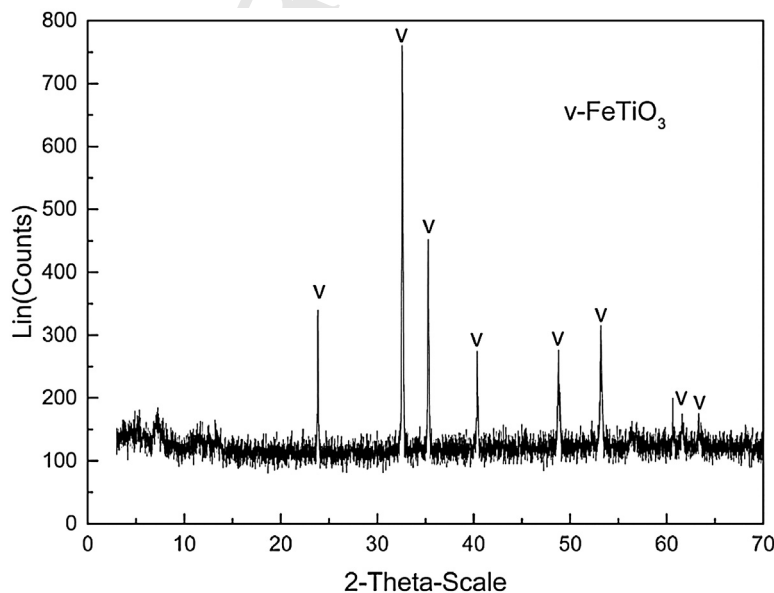


Fig. 1. XRD pattern of pure ilmenite samples used in the study.

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