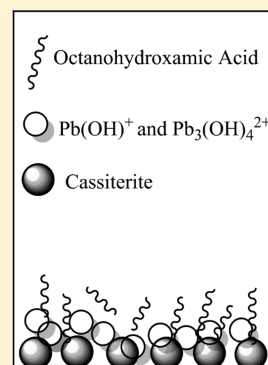


# Behavior of Lead Ions in Cassiterite Flotation Using Octanohydroxamic Acid

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**ABSTRACT:** The effects of lead ions on the flotation performance of the cassiterite system have been investigated using microflotation tests. Adsorption experiments, X-ray photoelectron spectroscopy (XPS), contact angle measurement, and zeta ( $\zeta$ )-potential measurements were conducted. Octanohydroxamic acid and lead ion solution speciation simulations were used. These were used to correlate surface properties of lead-activated with cassiterite flotation performance. Flotation results indicate that the addition of lead ions activates the cassiterite flotation. The addition of lead ions enhanced the adsorption of octanohydroxamic acid on cassiterite, and the wetting characteristic of cassiterite decreased in the presence of octanohydroxamic acid and the addition of lead ions.  $\zeta$ -Potential measurements and solution chemistry analyses confirmed that flotation recovery of the cassiterite increases due to  $\text{Pb}(\text{OH})^+$  and  $\text{Pb}_3(\text{OH})_4^{2+}$  sites on the cassiterite surface. XPS results indicated the adsorption of octanohydroxamic acid onto the cassiterite surface was through chemisorption, which combined with lead ions changed the chemical properties of the cassiterite surface.



## 1. INTRODUCTION

Tin is an important metal which has been widely used in industry such as in ceramics, coating cans, and pigments.<sup>1</sup> Cassiterite is considered to be the only economically significant tin mineral in the earth's crust. It is hard and heavy; hence gravity technology such as jigs, spirals, and tables has been the dominant method for use.<sup>2</sup> However, once cassiterite particle sizes are below 40  $\mu\text{m}$ , recovery by gravity-concentration processes decreases markedly. Due to the extreme brittleness of cassiterite, significant quantities of fine particles can be produced during crushing and grinding, resulting in loss of tin in the succeeding processing stages.<sup>3,4</sup> Therefore, flotation technique is recommended for the recovery of fine and ultrafine cassiterite particles from low-grade tin ores and gravity tailings.<sup>5</sup>

Flotation is a physicochemical separation process that utilizes the difference of surface properties between the valuable minerals and the unwanted gangue minerals.<sup>6</sup> The flotation environment is very complicated in real industry, and many metal ions such as  $\text{Ca}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Al}^{3+}$ , and  $\text{Pb}^{2+}$  may exist in the pulp, which is mainly from the use of recycle water, or the presence of semisoluble minerals.<sup>7,8</sup> There has been a growing suspicion that metal ions play an important role by depressing or activating target minerals in mineral flotation. A number of studies have been conducted to evaluate the effects of metal ions on the flotation. Irannajad et al.<sup>8</sup> investigated that quartz can be activated with  $\text{Zn}(\text{II})$  and  $\text{Pb}(\text{II})$  ions. Honaker et al.<sup>9</sup> found that the presence of calcium ions suppresses the floatability of the monazite using hydroxamic acid as the collector. Wang et al.<sup>10</sup> investigated that the floatability of spodumene, albite, and quartz was enhanced by adding  $\text{Fe}(\text{III})$  ions. Many studies have found that lead ions can act as an activator to improve the recovery of cassiterite,<sup>5</sup> hemimorphite,<sup>11</sup> and wolframite.<sup>12</sup> However, no one has reported

the effect of metal ions on cassiterite flotation when octanohydroxamic acid are used as a collector.

The aim of this work is to evaluate the effect of  $\text{Pb}^{2+}$  on cassiterite flotation with octanohydroxamic acid as collector. The experiments were carried out in a mechanical agitation flotation machine, and the activation mechanism was investigated by adsorption experiments, zeta ( $\zeta$ )-potential determinations, and X-ray photoelectron spectroscopy (XPS) analysis.

## 2. MATERIALS AND METHODS

**2.1. Materials.** The cassiterite samples were purchased from Yunnan Bao Yun Jewelry Company, China. Chemical analysis showed that the resultant powder contained 95.58%  $\text{SnO}_2$ . For the microflotation tests, the cassiterite concentrate was screened to obtain material having a particle size smaller than 38  $\mu\text{m}$  and larger than 20  $\mu\text{m}$ . The remaining samples were ground continuously for  $\zeta$ -potential, adsorption, and X-ray photoelectron spectroscopic tests.

Octanohydroxamic acid of 99% purity was purchased from Aladdin Bio-Chem Technology, China.  $\text{Pb}(\text{NO}_3)_2$  was used as a source of  $\text{Pb}^{2+}$ .  $\text{H}_2\text{SO}_4$  and  $\text{NaOH}$  were used for pH adjustment of the suspension. All the chemicals were of analytical grade except for the frother pine oil (commercial grade), and distilled water was used for all experimental work.

**2.2. Microflotation.** Microflotation tests were conducted in a mechanical agitation flotation machine, and the cell volume is 40 mL. The mixtures were stirred at 1650 rpm by a four-bladed

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impeller. The microflotation tests involved the following procedures: (1) Pure mineral particles (2.00 g) with 30 mL of distilled water were placed in the cell and then agitated for 1 min. (2) The pH was adjusted by adding pH regulator and agitated for 3 min. (3) The metal ions were added and agitating took place for 3 min. (4) The collector was added and agitating occurred for 3 min. (5) After the addition of frother, the floated materials were collected for 4 min. (6) The collected materials were filtered, dried, and weighed for recovery calculation.

**2.3. Measurements of  $\zeta$ -Potential.**  $\zeta$ -Potential measurements of cassiterite were carried out using a Zetasizer Nano ZS90 (ZEN3690, Malvern Instruments Ltd., U.K.). All measurements were conducted in a 1 mM KCl background electrolyte solution. A solids concentration of 0.05% by weight was used, and conditioning procedures were similar to that used for the microflotation tests. The results presented in this paper are the average of three independent measurements with a typical variation of +2 mV.

**2.4. Adsorption Tests.** Hydroxamic acid concentration was determined using a Shimadzu 2600 UV–visible spectrophotometer at 507 nm based on the ferric hydroxamate method.<sup>9</sup> Adsorption tests were conducted in a 100 mL glass beaker. Conditioning procedures were similar to that used for microflotation tests. After that, 10 mL of the suspension was centrifuged at 9000 rpm for 10 min, and the separated liquid was collected to quantitatively analyze the octanohydroxamic acid concentration.

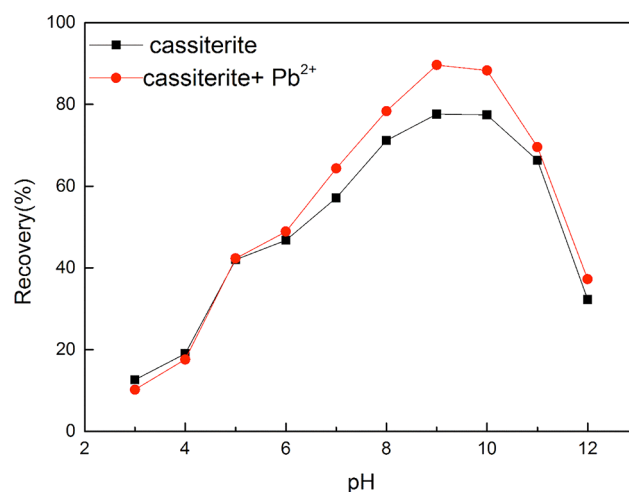
**2.5. X-ray Photoelectron Spectroscopic Study.** XPS analysis was conducted using an ESCALAB250Xi (Thermo Scientific, U.K.) with Al K $\alpha$  X-ray source. The mineral samples were taken from flotation experiments and then washed with distilled water to a similar pH to remove any suspended colloidal particles. Finally they were introduced immediately into the fore vacuum of the spectrometers as a slurry.

**2.6. Contact Angle Measurement.** The contact angles of cassiterite were measured before and after treatment with octanohydroxamic acid with metal ions using an instrument (JC2000D, Shanghai Chenzhong Digital Technology Limited Company). Conditioning procedures were similar to that used for microflotation tests, and modified cassiterite powder was taken from flotation experiments. The samples were vacuum-dried in a desiccator model Sotelem at approximately 25 °C and then transferred into a tablet machine to press into a wafer with diameter of 12 mm. Then it was determined by a contact angle analyzer.

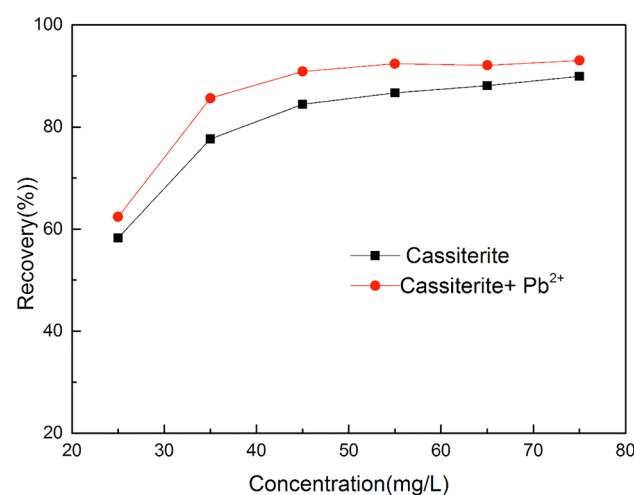
### 3. RESULTS AND DISCUSSION

**3.1. Microflotation Tests.** Mineral floatability is closely related to the solution pH value, which influences the component of collectors and ions present in solution. Figure 1 shows the flotation recovery of cassiterite with a collector dosage of 35 mg/L and lead ions of 20 mg/L. A maximum recovery of 77.63% was obtained for cassiterite at pH 9.0 in the absence of lead ions. Cassiterite floatability was significantly enhanced by lead ions. The recovery of cassiterite increased about 12% compared to solution in the absence of lead ions.

While maintaining the solution pH at a value of 9.0, octanohydroxamic acid dosage was varied in flotation systems. Figure 2 shows the flotation recovery of cassiterite with collector concentration from 25 to 75 mg/L and metal ion concentration of 20 mg/L at pH 9.0. The recovery of cassiterite approached 90% at high collector dosage levels without the use



**Figure 1.** Effects of pH on cassiterite flotation when using 35 mg/L octanohydroxamic acid and 20 mg/L lead ions.

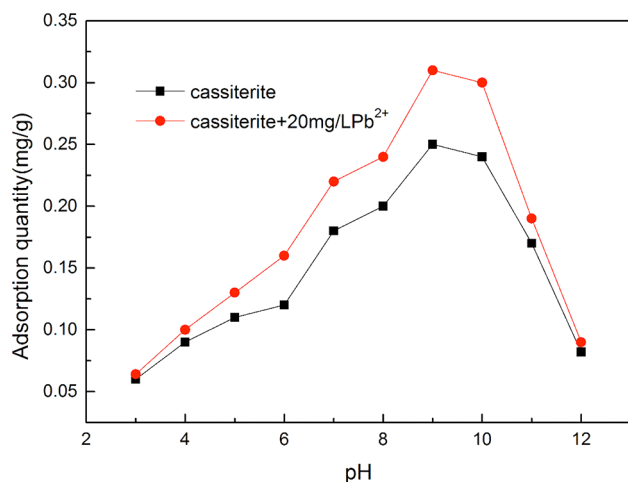


**Figure 2.** Effects of octanohydroxamic acid concentration on cassiterite flotation at pH 9.0.

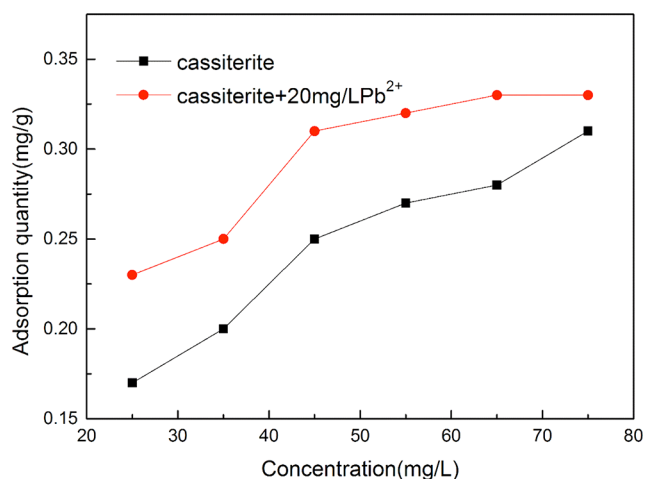
of metal ions. The maximum recovery reached 93.03% in the presence of octanohydroxamic acid and lead ions.

**3.2. Adsorption Tests.** Adsorption density was measured over a range of pHs and octanohydroxamic acid concentrations. Figure 3 shows the effect of pH on the adsorption of octanohydroxamic acid on cassiterite before and after treatment with lead ions. As can be seen, the adsorption capacity first increased and then decreased when the pH increased, and the maximum value is obtained within pH 9–10. The addition of lead ions enhanced the adsorption of octanohydroxamic acid on cassiterite over the whole pH range, which agreed well with the microflotation tests of cassiterite (Figure 1). Figure 4 shows the adsorbed amounts of octanohydroxamic acid onto the cassiterite surface as a function of octanohydroxamic acid concentrations before and after treatment with lead ions. As shown in Figure 4, the adsorption capacity of octanohydroxamic acid increased slightly from 0.17 to 0.31 mg/g with an elevation in the octanohydroxamic acid concentrations, while in the presence of octanohydroxamic acid and lead ions, the adsorption capacity increased from 0.23 to 0.33 mg/g.

**3.3. Contact Angle Measurement.** The contact angle is closely related to the floatability of minerals since it is the reflection of the wettability of solid mineral surface.<sup>13</sup> The



**Figure 3.** Effect of pH on adsorption of octanohydroxamic acid on cassiterite when using 45 mg/L octanohydroxamic acid.

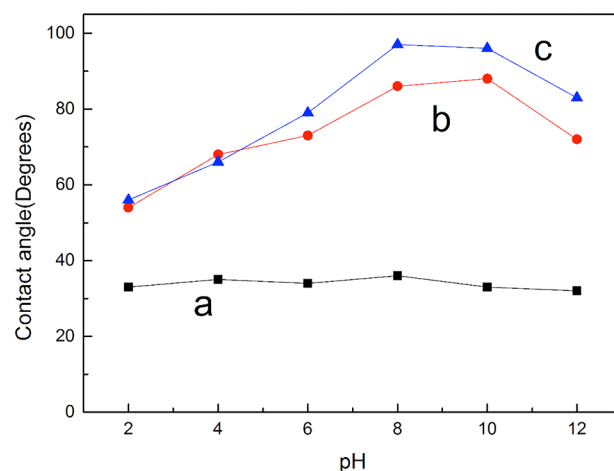


**Figure 4.** Effect of octanohydroxamic acid concentration on adsorption of octanohydroxamic acid on cassiterite at pH 9.0.

contact angles of cassiterite are depicted in Figure 5. Before the interaction with octanohydroxamic acid, the surface of the cassiterite was completely hydrophilic, and values of the contact angle were approximately 35°. The contact angle values of cassiterite sample increased to between 54 and 88° after the interaction with octanohydroxamic acid. Moreover, the contact angle reached higher values in the presence of octanohydroxamic acid and lead ions; for pH values between 8.0 and 10.0, the values of the contact angle are higher, demonstrating a larger interaction between the cassiterite cells and octanohydroxamic acid with lead ions in this pH range.

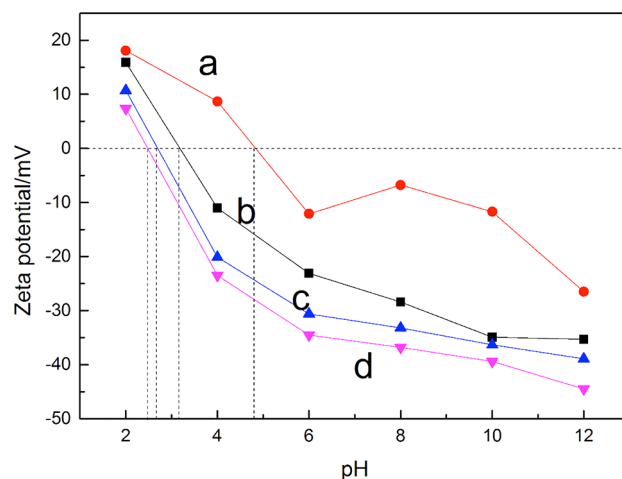
The results in Figure 5 illustrate that the cassiterite was completely hydrophilic in the absence of octanohydroxamic acid and lead ions while the addition of collector enhanced the contact angle. The addition of lead ions made this result more obvious especially within pH values between 8.0 and 10.0. These results confirmed that the wettability of cassiterite decreased in the presence of octanohydroxamic acid and more obviously with the addition of lead ions, which corresponds to the results of the flotation recovery of cassiterite and the adsorption of octanohydroxamic acid on cassiterite.

**3.4.  $\zeta$ -Potential Measurements and Solution Chemistry Analyses.** Electrokinetic tests are normally widely used



**Figure 5.** Contact angles of cassiterite as a function of pH: (a) cassiterite alone; (b) cassiterite in the presence of 45 mg/L octanohydroxamic acid; (c) cassiterite in the presence of 45 mg/L octanohydroxamic acid and 20 mg/L Pb<sup>2+</sup>.

in the interpretation of flotation phenomena based on the isoelectric point (IEP) of mineral particles, and the  $\zeta$ -potential typically shifts when specific adsorption occurs in the Stern layer.<sup>9</sup> Figure 6 shows the  $\zeta$ -potential of cassiterite as a function



**Figure 6.**  $\zeta$ -Potential of cassiterite as a function of pH: (a) cassiterite in the presence of 20 mg/L Pb<sup>2+</sup>; (b) cassiterite alone; (c) cassiterite in the presence of 45 mg/L octanohydroxamic acid; (d) cassiterite in the presence of 45 mg/L octanohydroxamic acid and 20 mg/L Pb<sup>2+</sup>.

of pH, and the IEP of cassiterite alone was located at a solution pH of 3.2, which is lower than that reported in the literature.<sup>14–16</sup> This might be due to SO<sub>4</sub><sup>2-</sup> being adsorbed on the surface of cassiterite which influences the IEP of cassiterite.<sup>17</sup> The IEP of cassiterite shifted toward more acidic pH from pH 3.2 to 2.7 after octanohydroxamic acid was added to the pulp suspension. Depression in the electrokinetic curve, toward the more negative direction, was also observed. We can attribute the shifts of the IEP values to a specific adsorption of octanohydroxamic acid on cassiterite surface. In the presence of lead ions, the  $\zeta$ -potential of the cassiterite was shifted to the right significantly and the IEP of cassiterite shifted from pH 3.2 to 4.8. This might be attributed to the lead ions being adsorbed/precipitated onto the negatively charged cassiterite surfaces through electrostatic interactions. Moreover, the IEP

shifted to pH 2.5 in the presence of octanohydroxamic acid and lead ions, which is more negative compared to octanohydroxamic acid alone. In the presence of lead ions and octanohydroxamic acid, the  $\zeta$ -potential of cassiterite is more negative compared to octanohydroxamic acid alone. It can be concluded that the presence of lead ion stimulates the adsorption of octanohydroxamic acid on cassiterite surface.

The species distribution diagram of 45 mg/L ( $2.83 \times 10^{-3}$  mol/L) octanohydroxamic acid as a function of pH is shown in Figure 7. As can be seen, the concentration of ionic

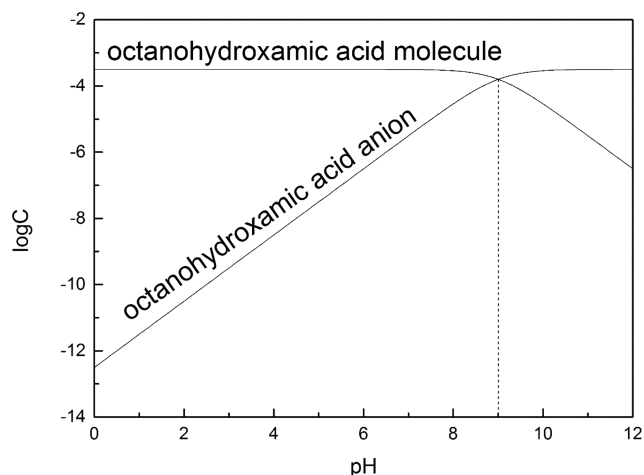


Figure 7. Relation between log C and pH of hydrolytic species of octanohydroxamic acid.

octanohydroxamic acid gradually increased while the concentration of molecular octanohydroxamic acid decreased with the increase of pH. As shown in Figures 1 and 3, the flotation recovery of cassiterite and the adsorption of octanohydroxamic acid on cassiterite were highest at pH 9–10. As shown in Figure 7, the ionic components and molecular components of octanohydroxamic acid were balanced in this pH range. This indicates that the ionic octanohydroxamic acid coadsorbed with molecular octanohydroxamic acid on the cassiterite surface. When pH < 9.0, molecular octanohydroxamic acid dominated and the adsorption of octanohydroxamic acid on cassiterite surface was very limited. Competitive adsorption occurred between octanohydroxamic acid and hydroxyl ions on cassiterite when molecular octanohydroxamic acid was the predominant species at a high pH. Along with Figure 6, we can infer that the adsorption of octanohydroxamic acid on cassiterite is due to chemisorption at pH > IEP.

Calculation of metal speciation helps in better understanding of metal behavior in mineral flotation. The analytical technique VISUAL MINTEQ is usually used to calculate metal speciation in water.<sup>18</sup> The species distribution diagrams for 20 mg/L ( $6 \times 10^{-5}$  mol/L) lead ions as a function of pH computed by VISUAL MINTEQ are shown in Figure 8. Free ionic  $\text{Pb}^{2+}$  is the most concentrated form under acidic conditions, whereas  $\text{PbOH}^+$  dominates within the pH range between 7 and 10. The concentrations of  $\text{Pb}(\text{OH})_2(\text{aq})$ ,  $\text{Pb}(\text{OH})_3^-$ , and  $\text{Pb}_3(\text{OH})_4^{2+}$  gradually increase as the pH increases, while this is accompanied by a drop in  $\text{Pb}^{2+}$  concentration. At pH 8–10, the  $\zeta$ -potential of cassiterite in the presence of  $\text{Pb}^{2+}$  shifted to the right significantly compared to the cassiterite alone, which can be attributed to the adsorption of positively charged  $\text{PbOH}^+$  and  $\text{Pb}_3(\text{OH})_4^{2+}$  on cassiterite surface. In addition,

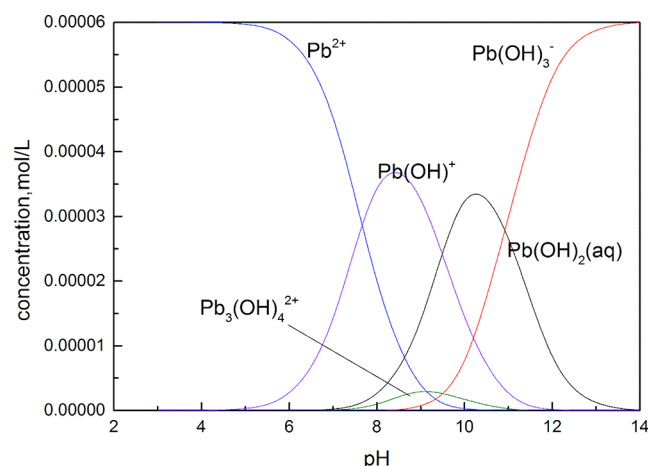


Figure 8. Distribution coefficients of various lead hydroxyl complexes in aqueous solutions as a function of pH.

microflotation tests in Figure 1 indicate that cassiterite has good floatability at pH 8–10. After positively charged  $\text{PbOH}^+$  and  $\text{Pb}_3(\text{OH})_4^{2+}$  adsorbed on cassiterite surface, the ionic octanohydroxamic acid coadsorbed with molecular octanohydroxamic acid onto the positively charged cassiterite surface, which overcomes electrostatic repulsion. Based on these findings, the solution species resulting in the specific adsorption were mainly  $\text{Pb}(\text{OH})^+$  and  $\text{Pb}_3(\text{OH})_4^{2+}$ .

**3.5. X-ray Photoelectron Spectroscopy.** X-ray photoelectron spectroscopy is the major method for establishing the chemical state of elements. It is based on the chemical shift of photoelectron lines on the change of the core–electron binding energy induced by a change in the chemical state of the atom.<sup>19</sup> XPS analysis was carried out to explore the effect of octanohydroxamic acid and lead ions on the cassiterite. Spectra of N 1s of untreated cassiterite and octanohydroxamic acid treated cassiterite are shown in Figure 9. As can be seen, no obvious N 1s peaks were detected on untreated cassiterite. However, after treatment by octanohydroxamic acid, the N 1s peaks were observed and the spectrum was made of two components: one at 397.28 eV assigned to the N–H group in the organic octanohydroxamic acid molecule and the other at 400.08 eV due to the deprotonated head groups. The XPS

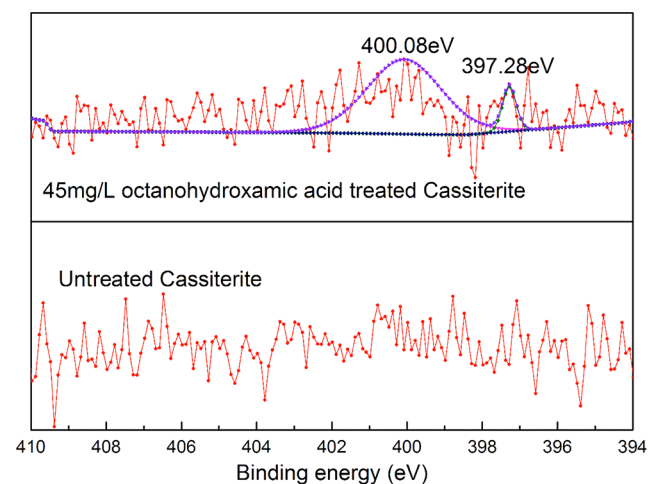
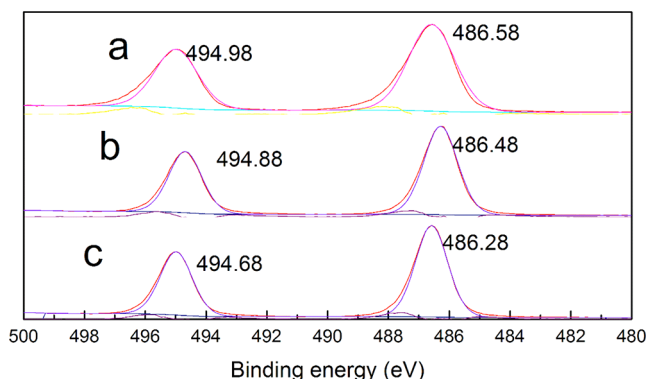


Figure 9. N 1s spectra of untreated cassiterite and octanohydroxamic acid treated cassiterite.



spectrum of octanohydroxamic acid treated cassiterite indicated that the adsorption of octanohydroxamic acid onto cassiterite surface was through chemisorption.<sup>20–22</sup>

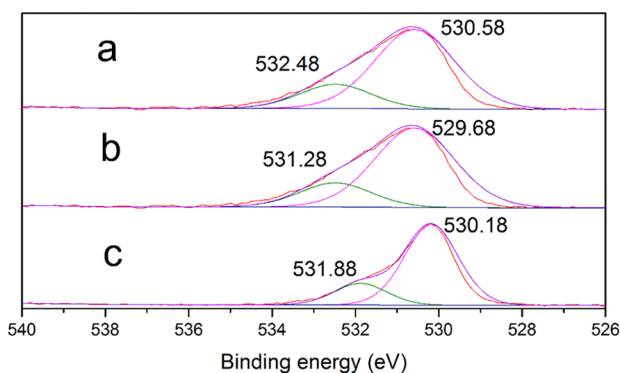
Figure 10 shows the binding energies of the Sn 3d electrons of cassiterite alone, cassiterite treated with lead ions, and



**Figure 10.** Sn 3d XPS spectra of (a) cassiterite alone, (b) cassiterite in the presence of 20 mg/L  $\text{Pb}^{2+}$ , and (c) cassiterite in the presence of 45 mg/L octanohydroxamic acid and 20 mg/L  $\text{Pb}^{2+}$ .

cassiterite treated with lead ions and octanohydroxamic acid. As can be seen from Figure 10a, there are two pairs of Sn 3d doublets, with binding energies of 486.58 eV for Sn  $3d_{5/2}$  and 494.98 eV for Sn  $3d_{3/2}$ . As can be seen from Figure 10b, the binding energies of Sn  $3d_{5/2}$  and Sn  $3d_{3/2}$  on lead ions treated cassiterite surface were both decreased by 0.1 eV, which indicates that lead ions affect the chemical environment of Sn atoms on cassiterite surface. Moreover, the binding energies of Sn  $3d_{5/2}$  and Sn  $3d_{3/2}$  on lead ions and octanohydroxamic acid treated cassiterite surface were both decreased by 0.2 eV compared to the one treated with lead ions. Therefore, Sn atoms on cassiterite mineral surface could be active sites for lead ions and octanohydroxamic acid to form chemical bonds.

Figure 11 shows the binding energies of the O 1s electrons of cassiterite alone, cassiterite treated with lead ions, and

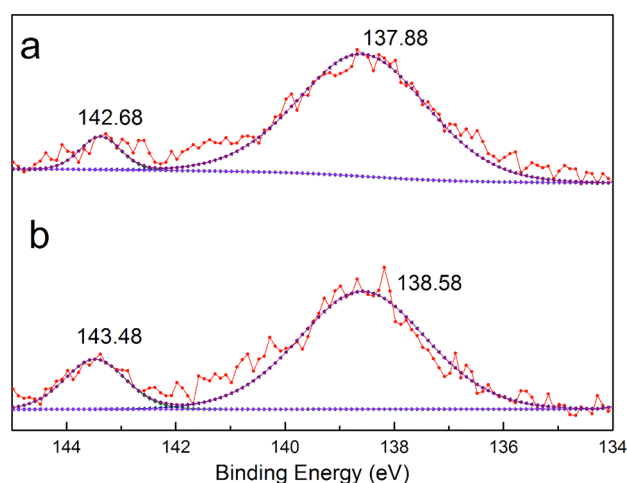


**Figure 11.** O 1s XPS spectra of (a) cassiterite alone, (b) cassiterite in the presence of 20 mg/L  $\text{Pb}^{2+}$ , and (c) cassiterite in the presence of 45 mg/L octanohydroxamic acid and 20 mg/L  $\text{Pb}^{2+}$ .

cassiterite treated with lead ions and octanohydroxamic acid. There are two spectral peaks at 530.58 and 532.48 eV on the spectrum of cassiterite alone; the peak at 530.58 eV is assigned to the oxygen in the cassiterite and the peak at 532.48 eV is attributed to the oxygen in the hydroxyl species.<sup>23</sup> The binding energy of the O 1s spectrum on lead ions treated cassiterite

surface was decreased by 0.9 eV, which indicates that the electronic environment of O atoms has been changed. As can be seen from Figure 11c, the O 1s spectrum also displays two peaks in the presence of octanohydroxamic acid and lead ions. The peak at 531.88 eV is assigned to  $\text{O}=\text{C}$ , the oxygen atom of octanohydroxamic acid molecule;<sup>23</sup> this shows that octanohydroxamic acid adsorbed on the surface of cassiterite. The peak at 530.18 eV is assigned to the oxygen of the cassiterite and the binding energies of this peak increased 0.5 eV relative to the lead ions treated cassiterite surface, which, on the other hand, is 0.4 eV lower than cassiterite alone. This phenomenon indicates that the chemical environment of O 1s has been changed under the combined action of lead ions and octanohydroxamic acid.

The Pb 4f peak of cassiterite treated with lead ions and octanohydroxamic acid consisted of two peaks as illustrated in Figure 12. As can be seen from Figure 12a, there are two pairs



**Figure 12.** Pb 4f XPS spectra of (a) cassiterite in the presence of 20 mg/L  $\text{Pb}^{2+}$  and (b) cassiterite in the presence of 45 mg/L octanohydroxamic acid and 20 mg/L  $\text{Pb}^{2+}$ .

of Pb 4f doublets on the spectrum of lead ions treated cassiterite. With a binding energy of 137.88 eV for the Pb  $4f_{7/2}$  and 142.68 eV for the Pb  $4f_{5/2}$ , the binding energy of the Pb  $4f_{7/2}$  and the Pb  $4f_{5/2}$  increased 0.7 and 0.8 eV, respectively. This result indicates that lead ions adsorbed on cassiterite surface and a chemical reaction take place under the action of octanohydroxamic acid, in agreement with the analysis result of the Sn 3d and O 1s spectra.

#### 4. CONCLUSION

Flotation results showed that octanohydroxamic acid contributed to good floatability of cassiterite and the addition of lead ions yielded higher recovery of cassiterite. Adsorption tests and contact angle measurements confirmed the flotation results: the adsorption capacity of octanohydroxamic acid increased and the wettability of cassiterite decreased in the presence of octanohydroxamic acid and lead ions compared to the cassiterite with octanohydroxamic acid.  $\zeta$ -Potential measurements and solution chemistry analyses confirmed that the adsorption of octanohydroxamic acid onto cassiterite surface was through chemisorption and the hydrophobicity and flotation recovery of the cassiterite increased due to  $\text{Pb}(\text{OH})^+$  and  $\text{Pb}_3(\text{OH})_4^{2+}$  sites on the cassiterite surface. XPS analysis indicated that tin and oxygen atoms on cassiterite

mineral surface could be active sites for lead ions and octanohydroxamic acid to form chemical bonds.

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### Notes

The authors declare no competing financial interest.

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