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Research Article

Keywords: Stibnite, Flotation, DFT, Cu2+, Pb2+

Posted Date: February 8th, 2023

DOI: https://doi.org/10.21203/rs.3.rs-2544656/v1

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Additional Declarations: No competing interests reported.
Activation mechanisms of Cu$^{2+}$ and Pb$^{2+}$ in stibnite flotation

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Abstract

Different activation mechanisms of Cu$^{2+}$ and Pb$^{2+}$ in stibnite flotation were studied via microflotation and adsorption capacity tests and density functional theory (DFT). Microflotation experiments indicated that the recovery rate of stibnite flotation with butyl xanthate (BX) was activated 7.82% higher by Pb$^{2+}$ than Cu$^{2+}$. The presence of Pb$^{2+}$ increased the amount of adsorbed BX on the stibnite surface. The results of DFT also suggested stronger activation by Pb$^{2+}$. Cu$^{2+}$ activation induced the formation of Cu-S bonds between the double-bonded S1 atom of BX with Cu, resulting in electron gain. Pb$^{2+}$ activation was followed by the formation of a stable chelating structure between single-bond S1 and double-bond S2 atoms of BX, resulting in electron loss. This system also facilitated dixanthate formation on the mineral surface via interaction between BX and oxygen, thus improving the floatability of stibnite. This study lays a theoretical foundation for the efficient recovery of stibnite.

Keywords: Stibnite; Flotation; DFT.; Cu$^{2+}$; Pb$^{2+}$

1. Introduction

Stibnite is the primary natural source of antimony, which can be effectively floated by xanthates in an acidic medium at pH < 5[1,2]. Stibnite does not respond well to xanthates owing to the presence of alkaline gangues in most deposits. Further, it is uneconomical to float stibnite in an acidic slurry at pH > 5[3]. During production, xanthate flotation is typically used to separate stibnite from other gangues after activation at pH 6.5[1,4].

CuSO$_4$ and Pb(NO$_3$)$_2$ are effective stibnite flotation activators[5,6]. Cao et al. reported that the recovery rate of stibnite was only 45% when butyl xanthate (BX) was used as a collector without an activator. The recovery rate of stibnite increased to 75% when copper sulfate was used as the activator, and to 85% when lead nitrate was used[7,8]. Richards et al. obtained similar results with stibnite in a flotation experiment[9]. Copper sulfate and lead nitrate may promote stibnite flotation using a xanthan acid collector, possibly because Cu$^{2+}$ and Pb$^{2+}$ are adsorbed on the lattice of stibnite, forming a complex hydrophobic layer comprising lead/copper xanthan acid and antimony xanthan acid on the stibnite surface[7]. Further, Cu$^{2+}$ and Pb$^{2+}$ are reduced on the stibnite surface to form a more hydrophobic Cu$_2$S or PbS layer, facilitating the adherence of stibnite to the bubbles and thus improving its recovery rate[4,8]. Lead nitrate enhances the activation better than copper sulfate, because Pb$^{2+}$ undergoes surface adsorption on the stibnite surface, whereas Cu$^{2+}$ undergoes volume adsorption[10,11]. Pen'kov et al. attributed this to the larger charge on lead atoms than on copper atoms, whereas Solozhenkin proposed Pb$^{2+}$ migration to the relatively deep stibnite surface layer, which enabled binding with sulfur atoms, as the primary factor contributing to the differential activation[12].

The aforementioned papers mainly studied the effect of stibnite activators on mineral surface from a macroscopic perspective based on chemical characterization[13,14]. Quantum chemistry has been increasingly used to obtain microscopic information about minerals[15]. First-principles calculations based on density functional theory (DFT) have become an important research tool in flotation
chemistry[16,17]. DFT is reliable in evaluating the chemical characteristics of flotation agents and the surface energy of minerals[18,19]. More importantly, this method has been widely used to analyze chemical interactions between agents and mineral surfaces[20-22].

In this work, the adsorption characteristics of butyl xanthate (BX) on the 010 surface of stibnite before and after the activation were investigated using DFT to simulate the interaction between Cu$^{2+}$ and Pb$^{2+}$ on the 010 surface. The results of the microflotation test provide a theoretical basis for the efficient recovery of stibnite by elucidating the activation mechanism of lead nitrate and copper sulfate in stibnite flotation.

2. Materials and methods

2.1. Samples and reagents

Stibnite samples were provided by Yunnan, China. Pure stibnite samples with particle sizes ranging from 45 μm to 74 μm were used for X-ray diffraction (XRD) and chemical element analysis. Based on the chemical analysis, the stibnite sample contained 68.73% Sb and 30.95% S. The XRD pattern indicated that the purity of stibnite was > 95% (Fig. 1). The reagents used in the experiment were BX, Pb(NO$_3$)$_2$, CuSO$_4$, hydrochloric acid, sodium hydroxide (all AR grade), and industrial-grade pine oil. Deionized water was used in all experiments.
2.2. Microflotation test

An XFGCII flotation machine with a 40-mL flotation cell was employed for the microflotation study. This flotation machine was purchased from Jilin Province Ore Exploration Machinery Factory, China. Pure stibnite (2.0 g, −200 mesh +325 mesh) was used in each experiment. The sample was first conditioned, and the pH of the slurry was adjusted to 6.5 using NaOH and HCl, followed by the addition of the activator and collector to the solution, further stirring for 3 min. The concentration of pine oil in the solution was 20 mg/L, and the frother conditioning time was 2 min. The flotation period was limited to 2 min. Each experiment was repeated three times, and the froth and sink products were collected to calculate the recoveries. The experimental process is outlined in Fig. 2.
2.3. Adsorption experiments

A UV-vis spectrophotometer (UV-2007, Shimadzu, Germany) was used to study the adsorption capacity of the collector on the stibnite surface. The residual amount of BX was determined by measuring its concentration in the supernatant by ultraviolet spectroscopy following centrifugation. The xanthate concentration adsorbed on the mineral surface was calculated from the residual xanthate concentration using the following equation:

$$\Gamma = \frac{V(C_0 - C)}{m}$$

where $C_0$ is the initial concentration of xanthate in the pulp solution; $C$ is the final concentration after the interaction between the reagents and the mineral surface; $V$ represents the solution volume; and $m$ denotes the mass of the stibnite sample.

2.4. Computational details

In this study, the CASTEP module in Material Studio 2019 software was used for structural optimization and energy calculations. According to published computational results, this is a first-principles calculation based on DFT\cite{17,21}, using the stibnite structure ($\text{Sb}_2\text{S}_3$) with a Pbnm space group. A generalized functional gradient was used
to approximate the GGA functional, and the mitigation correlation energy was described using the gradient correction function (PW91) proposed by Perdew and Wang. The valence electron plane wave function truncation energy (energy cutoff) was set to 380 eV. The valence electrons selected to calculate the pseudopotential of the atom were H 1s, C 2s 2p, O 2s 2p, S 3s 3p, Sb 5s 5p, Pd 5d 6s 6p, and Cu 3d 4s. All calculations were performed in the reciprocal space, and the Brillouin zone was integrated using the Monkhorst–Pack scheme. The k-point set grid size was 3 × 3 × 1; the convergence accuracy was $1.0 \times 10^{-6}$ eV/atom; the maximum force was 0.1 eV/Å; and the maximum stress was 0.2 GPa. Because stibnite itself is not magnetic, all atoms were allowed to relax. The non-spin results were used to analyze the data, regardless of the effect of spin on the calculated results.

Previous studies of the surface energy of stibnite showed that the (010) surface was the most stable[20]. Therefore, the (010) surface was selected for analysis in this study. The stibnite (010) surface was obtained by directional cutting. Subsequent simulation of the adsorption of BX molecules on the stibnite surface used a 2 × 1 × 2 supercell model, with a vacuum layer of 15 Å and a total of two atomic layers. The main and side views of the stibnite (010) surface model after geometrical optimization are depicted in Fig. 3.

Figure 3. Geometrically optimized stibnite (010) model. The “main” and side views are indicated by the red and dotted lines.

Cao et al. presented a comparative study of the possible adsorption sites of Pb$^{2+}$ on stibnite and found that the six-fold hollow site (6FH) exhibited the strongest
adsorption capacity[23]. Therefore, the geometry for DFT calculations was optimized by setting all the ion adsorption sites as 6FH sites on the stibnite (010) surface, as shown in Fig. 4.

![Figure 4. Six hollow sites on the stibnite (010) surface.](image)

The adsorption energy ($\Delta E_{ads}$) of Pb$^{2+}$ and Cu$^{2+}$ at the 6FH point of the stibnite (010) surface is expressed by

$$\Delta E_{ads} = E_{slab+Pb/Cu}^{tot} - E_{Pb/Cu} - E_{slab}^{tot}$$

(2)

Here, $E_{slab}^{tot}$ and $E_{slab+Pb/Cu}^{tot}$ represent the total energy of the stibnite supercell model before and after the adsorption of Pb$^{2+}$ or Cu$^{2+}$, respectively; and $E_{Pb/Cu}$ is the total energy of Pb$^{2+}$ or Cu$^{2+}$.

To compare the activation mechanisms of Pb$^{2+}$ and Cu$^{2+}$, BX was added to the model. The optimized BX model is shown in Fig. 5, where S1 and S2 represent S atoms with single and double bonds in BX.

![Figure 5. Molecular model of butyl xanthate (BX).](image)

After adding BX to the stibnite (010) surface, the adsorption energy of Pb$^{2+}$ or Cu$^{2+}$ activated by BX is expressed by
\[ \Delta E_{\text{ads}} = E_{\text{slab+Pb/Cu+BX}}^{\text{tot}} - E_{\text{BX}} - E_{\text{slab+Pb/Cu}}^{\text{tot}} \] (3)

where \( E_{\text{slab+Pb/Cu}}^{\text{tot}} \) and \( E_{\text{slab+Pb/Cu+BX}}^{\text{tot}} \) represent the total energy levels before and after the adsorption of BX by the stibnite (010) surface model, respectively. \( E_{\text{BX}} \) is the total energy of BX.

The change in enthalpy of the reaction between substances is represented by \( \Delta E_{\text{ads}} \). A higher negative value indicates rapid progress of the reaction. All quantum mechanical simulations were conducted under vacuum, which is different from the actual production and testing environments. Therefore, the results are not completely consistent with the experimental observations of the interaction between minerals and reagents. Accordingly, the calculated results only provide a general reference for the adsorption of \( \text{Pb}^{2+} \) or \( \text{Cu}^{2+} \) on stibnite (010) surfaces and subsequent BX adsorption on the activated surface.

3. Results and discussion

3.1. Effect of \( \text{CuSO}_4 \) and \( \text{Pb(NO}_3\text{)}_2 \) on the flotation recovery of stibnite

![Graph showing the dependence of stibnite recovery on the activator concentration in stibnite flotation](image)

*Figure 6.* Dependence of stibnite recovery on the activator concentration in stibnite flotation (10 mg/L of BX and 20 mg/L of pine oil at pH 6.5).

The flotation behavior of stibnite depends on both the amount of BX and the type
and amount of the activator[7]. First, the effect of various activators on stibnite was studied via microflotation experiments at a pH of 6.5; the results are shown in Fig. 6.

Without the activator, the recovery rate of stibnite was only 63%. With the addition of the activator, the recovery of stibnite first increased with increasing concentration of the activator and then plateaued. At an activator concentration of 3 mg/L, the recovery rate of stibnite reached 84.22% with copper sulfate as the activator and 92.04% with lead nitrate as the activator (7.82% higher than in the presence of copper sulfate). Cao et al. studied the flotation of stimonite, which yielded a recovery rate of 85% with lead nitrate as the activator and only 75% with copper sulfate as the activator, which was similar to the results of this test[8]. Under other process conditions, both activators significantly increased the recovery of stibnite, with a slightly better performance using the lead nitrate activator.

3.2. Effect of activator dosage on BX adsorption

![Graph](image)

**Figure 7.** Consumption of BX on stibnite surface from a pulp solution as a function of the activator concentration (BX concentration of 10 mg/L and pH of 6.5).

The amount of adsorbed BX on the stibnite surface was evaluated at various dosages of the activator and a fixed optimal concentration of BX (10 mg/L) to investigate the effect of the activator on BX adsorption.
UV analysis was performed to determine the BX collector consumption during the flotation of stibnite with various activators [19]. As shown in Fig. 7, under the optimal dosage of BX, with an increasing initial concentration of the activator, the amount of BX adsorbed by stibnite gradually increases and then slightly decreases. Moreover, over the experimental concentration range, the BX adsorption capacity on stibnite was higher with lead nitrate than copper sulfate as the activator.

3.3 DFT results

3.3.1 Adsorption of Cu$^{2+}$ and Pb$^{2+}$ on the Sb$_2$S$_3$ (010) surface

The adsorption of Cu$^{2+}$ and Pb$^{2+}$ on the 6FH sites of an Sb$_2$S$_3$ (010) surface was simulated. After the optimization of DFT geometry, the adsorption energies of the systems with different activators were calculated, and the results are listed in Table 1. The magnitude of the adsorption energy of Pb$^{2+}$ was greater than that of Cu$^{2+}$, indicating that Pb$^{2+}$ was more stably adsorbed than Cu$^{2+}$ on the mineral surface. Before the addition of BX, both Cu$^{2+}$ and Pb$^{2+}$ exhibited high adsorption energy, which exerted an adsorption effect on Sb$_2$S$_3$.

<table>
<thead>
<tr>
<th>Adsorption model</th>
<th>$\Delta E_{\text{ads}}$, kJ/mol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu$^{2+}$</td>
<td>$-228.45$</td>
</tr>
<tr>
<td>Pb$^{2+}$</td>
<td>$-250.08$</td>
</tr>
</tbody>
</table>

3.3.2 Adsorption of BX on the Sb$_2$S$_3$ (010) surface

The adsorption configuration of BX on the Sb$_2$S$_3$ (010) surface activated by Cu$^{2+}$ or Pb$^{2+}$ after DFT geometry optimization is shown in Fig. 8.
Figure 8. Adsorption of butyl xanthate (BX) on Sb$_2$S$_3$(010) surface after activation by (a) Cu$^{2+}$ or (b) Pb$^{2+}$.

It can be seen that the S1 and S2 atoms in BX form adsorption structures on the surface of Sb$_2$S$_3$ (010). The surface activation by Cu$^{2+}$ and Pb$^{2+}$ occurs via three pathways, with different values of $\Delta E_{\text{ads}}$, as clearly summarized in Table 2. After Cu$^{2+}$ and Pb$^{2+}$ activation, the magnitudes of the $\Delta E_{\text{ads}}$ values of the Sb$_2$S$_3$(010) surface with BX are smaller than before BX adsorption. Therefore, the adsorption of BX on the surface of Sb$_2$S$_3$(010) activated by Cu$^{2+}$ and Pb$^{2+}$ is somewhat weakened, although the strong adsorption still persisted. The adsorption energy of S1 and S2 atoms in BX in the presence of Pb atoms on the surface of Sb$_2$S$_3$(010) was the largest, indicating that BX has a strong adsorption capacity after Pb$^{2+}$ activation, which is consistent with the results of the flotation tests. The following section compares the various activation mechanisms of the two ions.

Table 2 Adsorption energy of the (010) stibnite surface after Cu$^{2+}$ and Pb$^{2+}$ activation

<table>
<thead>
<tr>
<th>Adsorption models</th>
<th>$\Delta E_{\text{ads}}$, kJ/ mol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu+Sb$_2$S$_3$+BX</td>
<td>−146.54</td>
</tr>
<tr>
<td>Pb+Sb$_2$S$_3$+BX</td>
<td>−192.95</td>
</tr>
</tbody>
</table>
3.3.3. Density of states analysis

Density of states (DOS) is one of the essential parameters used to describe the motion of electrons in solid state physics. It has also been used in surface science and interfacial adsorption studies, where partial DOS (PDOS) revealed the role of each orbit[24].

A comparative DOS analysis was performed to further analyze the adsorption mechanism of BX after Cu\textsubscript{2+} and Pb\textsuperscript{2+} activation on the Sb\textsubscript{2}S\textsubscript{3}(010) surface. The DOS of BX atoms S1+S2 and Cu before and after the activation of Sb\textsubscript{2}S\textsubscript{3} (010) Cu\textsuperscript{2+} are shown in Figs. 9(a) and (b). The local DOS of S1 + S2 and Pb after BX adsorption on Pb\textsuperscript{2+}-activated Sb\textsubscript{2}S\textsubscript{3}(010) surface are shown in Figs. 10(a) and (b).

![Figure 9. Density of states (DOS) of S1 + S2 atoms in BX before and after Cu\textsuperscript{2+} activation of](image-url)
the Sb₂S₃(010) surface. (a) S1 + S2. (b) Cu.

As can be seen in Fig. 9, the S1 and S2 atomic orbitals and the Cu atomic orbitals primarily comprise S 3p 3s orbitals and Cu 3s 3p 3d orbitals, respectively. Addition of Cu²⁺ led to a significant change in the adsorption of BX on the Sb₂S₃(010) surface. The 3p orbital of S1 + S2 overlaps with the Cu 3d orbital at −3-0 eV (close to the Fermi level). The intensity of the main peak is close to 4 eV. Thus, the addition of Cu²⁺ resulted in a strong hybridization of the 3p and 3s orbitals of S1 + S2 atoms of BX with Cu 3d orbitals, which facilitated the adsorption reaction, suggesting that Cu²⁺ activates stibnite collection.

Figure 10. Density of states (DOS) of S1 + S2 atoms in BX before and after Pb²⁺ activation on the Sb₂S₃(010) surface. (a) S1 + S2. (b) Pb.

To further study the adsorption mechanism of BX on the Sb₂S₃ (010) surface activated by the different ions, the surface model of BX adsorption on the Pb²⁺-activated
Sb$_2$S$_3$(010) surface was constructed. The PDOS results of S1 + S2 and Pb atoms are shown in Fig. 10. It can be seen that the Pb atomic orbitals are composed mainly of Pb 5d 6s 6p orbitals, while the S1 and S2 atomic orbitals are composed mainly of S 3p 3s orbitals. Compared with the surface activated by Cu$^{2+}$, the intensity of the main peak of S1 + S2 atoms at the Fermi level was higher (exceeding 5 eV), indicating that S atoms exhibit higher activity after Pb$^{2+}$ addition. Further, the 3p orbitals of S1 + S2 and Pb 6s 6p orbitals cross the Fermi level and undergo stronger hybridization in the valence band position, which is conducive to the adsorption reaction and formation of new bonds. These results demonstrate that the reaction between BX and stibnite is greatly enhanced when stibnite is activated by Pb$^{2+}$ rather than Cu$^{2+}$, and the difference between the activators can be described on an atomic scale.

### 3.3.4. Mulliken population and electron density

The Mulliken overlapping population (or bond population) reflects the overlap of electrons between two atoms and determines the ionic and covalent properties of bonds between atoms[13]. The Mulliken bond populations of the Sb$_2$S$_3$ (010) surface for the Cu$^{2+}$–BX and Pb$^{2+}$–BX systems are presented in Table 3. BX forms stable and strong covalent bonds with Pb$^{2+}$ and Cu$^{2+}$ when added to the system, regardless of Pb$^{2+}$ or Cu$^{2+}$ activation. However, the Mulliken bond population and bond length of the two types of chemical bonds in the Pb$^{2+}$–BX system are similar, while the corresponding difference between the two types of chemical bonds in the Cu$^{2+}$–BX system is larger. Therefore, the adsorption structures of the two systems differ. The adsorption process in the Pb$^{2+}$–BX system mainly leads to the formation of a chelation structure with S1 and S2 atoms, while the adsorption of BX in the Cu$^{2+}$–BX system mainly results in the formation of a double-bond structure with S2 atoms.

<table>
<thead>
<tr>
<th>Adsorption model</th>
<th>Chemical bond</th>
<th>Mulliken population</th>
<th>Bond length/Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sb$_2$S$_3$+Pb+BX</td>
<td>Pb-S2</td>
<td>0.20</td>
<td>2.78</td>
</tr>
<tr>
<td></td>
<td>Pb-S1</td>
<td>0.12</td>
<td>2.84</td>
</tr>
</tbody>
</table>

Table 3 Mulliken key populations and bond lengths
Differential electron density analysis was performed to further explain the different mechanisms of action between BX and the Sb₂S₃(010) surfaces activated by Cu²⁺ or Pb²⁺. Based on the electron density presented in Fig. 11, the S₁ and S₂ atoms react with Cu and Pb atoms. Electron densities show that the electron cloud overlaps at the junction between Cu and double-bond S₂ atoms, while the electron cloud overlaps partially at the intersection between single-bond S₁ and Cu atoms (Fig. 11(a)). This indicates that during the activation by Cu²⁺, Cu and BX form stable chemical bonds (Cu–S₁), but not a stable structure. As shown in Fig. 11(b), Pb has a high electron-cloud overlap density with both single-bond S₁ and double-bond S₂ atoms, indicating that Pb forms stable chemical bonds with BX to form Pb–S₁ and Pb–S₂ bonds, resulting in a stable chelating structure. This conclusion is consistent with the Mulliken population results.

**Figure 11.** Differential electron density of Sb₂S₃(010) surfaces after BX adsorption and Cu²⁺ and Pb²⁺ activation.

In Fig. 11, the Sb₂S₃(010) surfaces activated by Cu²⁺ or Pb²⁺ reveal blue and pink backgrounds of the differential electron densities, respectively, indicating electron gain and electron loss, respectively. Under the activation of Pb²⁺, the entire system is in a
state of electron loss, which facilitates the interaction between the mineral surface and oxygen to form double xanthate on the mineral surface, thus enhancing the collection.

4. Conclusions

CuSO$_4$ and Pb(NO$_3$)$_2$ are both effective activators of stibnite. However, Pb(NO$_3$)$_2$ exhibits a more substantial activation than CuSO$_4$. Microflotation experiments show that both activators effectively increase the recovery of stibnite when BX is used as the collector. The recovery with Pb(NO$_3$)$_2$ as the activator is 7.82% greater than in the presence of CuSO$_4$. Compared with Cu$^{2+}$, Pb$^{2+}$ induces higher adsorption of BX on the stibnite surface.

DFT results show that the adsorption effect of Pb$^{2+}$ is stronger than that of Cu$^{2+}$ because BX is adsorbed on the surface of Sb$_2$S$_3$ (010) after Cu$^{2+}$ and Pb$^{2+}$ adsorption. Further, the double-bond S2 atom of BX forms a stable adsorption structure with Cu atoms. The single-bond S1 and double-bond S2 atoms of BX form a strong chelation adsorption structure with Pb atoms. Further, the 3p orbitals of S1 and S2 atoms is strongly hybridized with the d orbital of Pb atoms, forming new Pb–S1 and Pb–S2 bonds. The differential charge density maps show that the adsorption of Cu$^{2+}$ and Pb$^{2+}$ leads to the formation of electron-gain and electron-loss systems on the BX–Sb$_2$S$_3$ (010) surface, respectively. The Pb$^{2+}$ electron-loss system facilitates the interaction between the mineral surface and oxygen to form double xanthate on the stibnite surface, thus enhancing the collection and improving mineral flotation. This study demonstrates the potential for developing an efficient strategy for activation of Cu$^{2+}$ and Pb$^{2+}$ in stibnite flotation.

Declarations

Competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this
Authors' contributions

Youming Xiao: Investigation, Software, Formal analysis, Validation, Writing-original draft. Yiqi Cui: Writing-review & editing, Funding acquisition. Xiong Tong: Methodology, Funding acquisition. Jing Wang: Writing -review & editing, Conceptualization. Dianqiang Huang: Methodology. Yang Zhang: Conceptualization, Methodology.

Funding

We gratefully acknowledge the Yunnan Major Scientific and Technological Projects (NO. 202202AG050010).

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