

Article

Flotation Performance and Adsorption Mechanism of Cerussite with Phenylpropenyl Hydroxamic Acid Collector

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Abstract: In this paper, the flotation separation of cerussite and quartz under different experimental conditions was studied by using phenylpropenyl hydroxamic acid (PHA) as a collector. The flotation performance of PHA was studied through experiments involving pure minerals and artificial mixed minerals, and the results showed that PHA has good selectivity and collecting ability for cerussite. The adsorption mechanism of the collector on the surface of cerussite was investigated via adsorption capacity experiments, zeta potential tests, FTIR spectroscopy, and XPS analysis. The adsorption capacity results showed that PHA can be well adsorbed on the surface of cerussite. The results of the zeta potential tests showed that PHA has strong chemical adsorption on the cerussite surface. FTIR and XPS analysis showed that PHA may form a Pb–PHA complex on the cerussite surface to improve the floatability of cerussite.

Keywords: phenylpropenyl hydroxamic acid (PHA); cerussite; flotation; adsorption



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1. Introduction

As an essential non-ferrous metal with favorable ductility and corrosion resistance, lead is now widely used in alloys and the mechanical and pharmaceutical industries [1,2]. Lead is normally extracted from lead sulfide minerals (such as galena) [3]. However, with the increasing depletion of lead sulfide minerals and the growing national demand for lead minerals [4–7], the importance of rational mining and the utilization of lead oxide minerals has gradually increased; typical lead oxide minerals include cerussite (PbCO₃), anglesite (PbSO₄), etc. [8,9]. Anglesite (PbSO₄) has very low solubility and is easily transformed into cerussite under the action of carbonated water.

At present, the main beneficiation method of cerussite is flotation, which is a process of separation through the difference in surface properties between target minerals and gangue [10]. The selection of flotation agents (collector, inhibitors, etc.) plays a critical role in the flotation process. The collector enhances the hydrophobicity of the mineral surface by changing the chemical properties of the mineral surface, and makes the mineral particles adhere to the bubbles in the aeration process of the flotation machine, which is a key link in the foam flotation. A high-efficiency selective collector has always been the focus of research on the separation of cerussite via flotation. In the past, oleic acid was commonly used as a collector for the flotation of oxide minerals [11], but its selectivity is poor and requires a large number of inhibitors in the flotation process, which is not environmentally friendly. The xanthate–sulfide method is also widely used in the flotation of cerussite; after

sodium sulfide treatment, lead sulfide will form on the surface of minerals, which increases the active site of xanthate and promotes the flotation of xanthate to cerussite [12–15]. Tang X et al. investigated the interface reaction on the surface of cerussite by using the DFTB+ method, established the vulcanization model of PbS film forming on the PbCO_3 surface, and revealed the vulcanization mechanism of cerussite. The results of the DFTB+ calculation confirmed that vulcanization promoted the adsorption of cerussite on the surface of xanthate, thus promoting the interaction between cerussite and xanthate [1]. It has been further verified that vulcanization is a crucial process to realize effective recovery of cerussite. However, when xanthate is used as a collector, a small amount of sulfiding agent will greatly reduce its effect, while an excessive amount of sulfiding agent will lead to a sharp decrease in floatability [16]. Therefore, the flotation of sulfide minerals is extremely difficult, and the sulfidation process must be strictly controlled [17]. R. Herrera-Urbina et al. found that a low dosage of sulfide can precipitate cerussite into lead sulfide via the consumption of lead dissolved in water, while excessive sulfide oversulfides the mineral surface and inhibits the cerussite [14]. Moreover, xanthate is not a stable compound, and it decomposes to produce carbon disulfide (CS_2), which is a toxic substance [18,19].

In recent years, hydroxamic acid has been gradually employed as a collector for oxidized minerals [20–22]. Hydroxamic acid is stable [23], does not decompose easily, and is environmentally friendly. Hydroxamic acid collectors have special molecular structures that allow them to form complexes with the surface metal atoms in the oxide minerals [24,25]. The intricate structure of complexes changes the properties of the mineral surface [26–28], increasing surface hydrophobicity and making the oxide minerals more susceptible to bubble attachment, which improves their selectivity and collection ability [29–31]. Z. Huang et al. prepared a novel “carb” structure sebacoyl hydroxamic acid called SHA [32]; a recovery of 75.3% and a grade of 55.6% were obtained via the flotation of wolframite and kaolinite using SHA, showing that the collection capacity and selectivity of SHA-assisted wolframite flotation are better than that achieved by flotation through the use of benzo-hydroxamic acid (BHA), and the dosage is half less than BHA. Zhao C et al. separated scheelite from fluorite and calcite by Fe-BHA. The results show that scheelite and calcite can achieve a good separation effect when $\text{pH} = 8$, and the scheelite grade is 74.2%, the recovery is 46.9%, the calcite grade is 25.7%, and the recovery is 17.9%. When $\text{pH} = 11$, scheelite and fluorite can achieve a good separation effect, and the scheelite grade is 7.17%, the recovery is 5.40%, the fluorite grade is 92.2%, and the recovery is 69.4% [25]. This provides an important research direction for the flotation collectors of oxide minerals. Wei Z et al. investigated the collection performance of BHA, p-methyl-benzohydroxamic acid (MBHA), and p-bromine-benzohydroxamic acid (BBHA) for scheelite through flotation experiments. The adsorption behavior of MBHA and BBHA was studied via zeta potential tests and XPS analysis, and the electron effect in MBHA and BBHA molecules was studied via the use of Density Functional Theory (DFT) calculation. Finally, it was concluded that the adsorption effect and collection performance of MBHA is better than that of BHA and BBHA [29]. In the flotation process of lead oxide ore, gangue minerals such as quartz and calcite affect the recovery of lead ore, among which quartz is a gangue mineral with high content that is difficult to separate, and Pb^{2+} dissolved in water can promote the flotation of quartz, which makes the flotation process more difficult. Through inductively coupled plasma mass spectrometry (ICP-MS), XPS, time-of-flight secondary ion mass spectrometry (ToF-SIMS), zeta potential, UV–visible spectroscopy, and flotation experiments, Zhang Q et al. explained the difficulty of separating quartz by using dissolved species of cerussite in a xanthate sulfidization system. The existence of $\text{Pb}(\text{OH})^+$, $\text{Pb}(\text{OH})_2$, and PbCO_3 provide active sites for xanthate and activate the flotation of quartz. The separation difficulty of cerussite and quartz is increased [12].

For the creation of this paper, the flotation behavior of PHA as a collector in cerussite was studied [33]. The unique properties of Phenylpropenyl hydroxamic acid (PHA) in cerussite were investigated via pure and artificial mixed minerals experiments. The adsorption mechanism of PHA on the surface of cerussite was studied through zeta potential

measurements, Fourier-Transform Infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy analysis (XPS analysis).

2. Materials and Methods

2.1. Materials and Agents

The samples of pure cerussite and quartz used in this research were purchased from the Pb-Zn mine in Yunnan province, China. By hand selection, the pure samples were crushed and ground in an agate mortar. After screening, $-74\ \mu\text{m} + 38\ \mu\text{m}$ pure minerals were used for the flotation experiments; the specific surface area of cerussite was $56.8\ \text{m}^2/\text{kg}$, and the specific surface area of quartz was $33.3\ \text{m}^2/\text{kg}$. Then, $-38\ \mu\text{m}$ pure minerals were used for mechanism research. XRD sample analysis was conducted with pure mineral samples with a particle size of $-74\ \mu\text{m}$. As shown in Figure 1, both pure minerals have high purity and satisfied the requirements of the flotation experiments. The results of our chemical composition analysis are shown in Table 1. The chemical composition analysis indicated that the pure mineral samples of cerussite contained 98.98% PbCO_3 and 0.18% SiO_2 , and the pure mineral samples of quartz contained 97.63% SiO_2 . In the experiment, sodium hydroxide (NaOH), sodium hexametaphosphate, PHA, and MIBC were all analytical-grade agents, the pH regulator used in the experiments was NaOH, the inhibitor was sodium hexametaphosphate, the collector was PHA, and the foaming agent was MIBC.

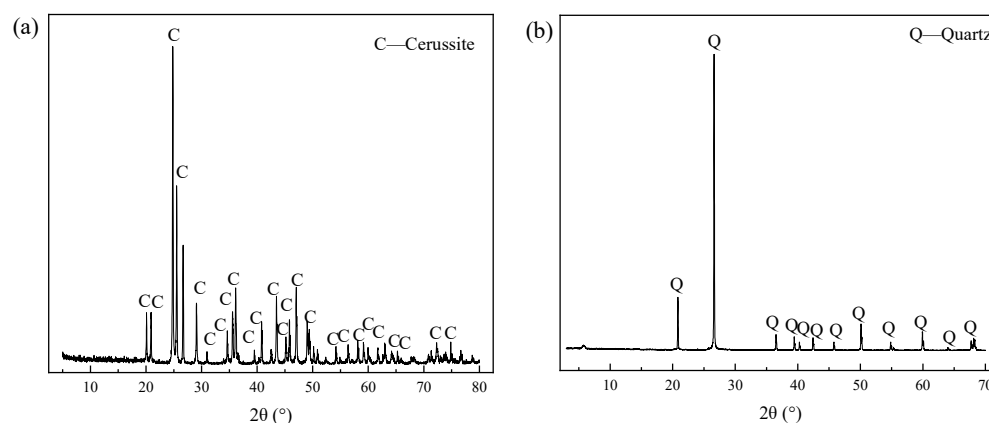


Figure 1. XRD patterns of pure mineral samples: (a) cerussite; (b) quartz.

Table 1. Chemical composition analysis results regarding cerussite and quartz.

Sample	$\text{PbCO}_3/\%$	$\text{Fe}/\%$	$\text{SiO}_2/\%$	$\text{Al}_2\text{O}_3/\%$	$\text{CaCO}_3/\%$	Others/%
Cerussite	98.98	-	0.18	0.22	0.13	0.49
Quartz	-	0.11	97.63	0.23	-	2.03

2.2. Micro-Flotation Tests

Micro-flotation tests on pure minerals were performed in the XFG-II flotation machine, and Figure 2 shows a flow chart depicting the procedure followed for the flotation experiments. A 35 mL flotation tank was filled with 2 g of pure minerals and 30 mL of deionized water, and the speed of the flotation machine was set at 1992 r/min. After stirring for 1 min, the pH was adjusted with NaOH, and the collector was added into the pulp for 2 min; then, the foaming agent was added, the mixture was stirred properly for 2 min, and the bubble was scraped manually. The filtered, dried, weighted, and recovery values for the bubble products were subsequently estimated.

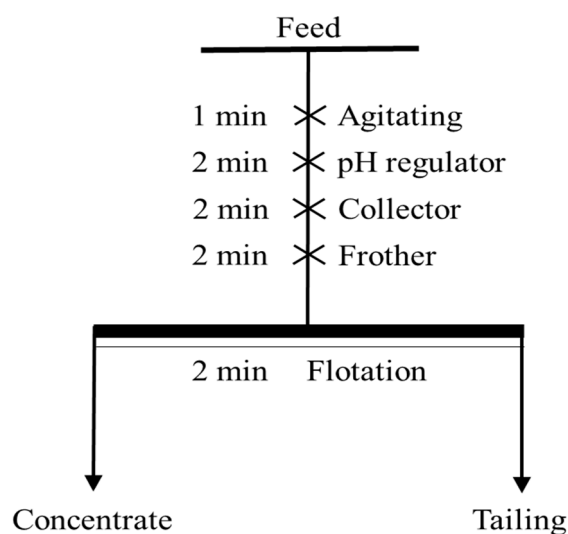


Figure 2. Flow chart depicting the process of the flotation experiments.

2.3. Adsorption Capacity Measurement

The functional connection between the concentration of leftover agent and the absorbance in the pulp's supernatant following the action of the minerals and agent was identified using a UT6-A UV spectrophotometer. The adsorption capacity of the agent on the minerals' surfaces was calculated via the determined function. A total of 2 g of pure minerals and 30 mL of deionized water were used to create the pulp. The pulp's pH was adjusted to obtain the optimal flotation; different concentrations of collector were added, and the pulp was agitated on a magnetic agitator at constant temperature for 1 h. After 30 min of standing, the centrifuge was spun at a speed of 5000 rap/s to separate the solid from the liquid. An appropriate amount of the supernatant was put into the quartz colorimetric dish for absorbance measurement.

2.4. Zeta Potential Tests

The zeta potential of the surface of the minerals under pure mineral and different chemical systems was measured via JS94H electrophoresis (Beijing Starwood Technology Co., Ltd., Beijing, China). The pure minerals of cerussite were added into deionized water to form a pulp solution with 0.01% concentration. This solution was agitated for 30 min and left undisturbed for 48 h. Zeta potential was measured using the standing supernatant.

During the measurement, the pH regulator was added to adjust the pH of the supernatant to be measured, and the influence of pH on the minerals' surface potential under different agent systems was measured. We repeated the measurements five times and took into account the error before calculating the averages of the results.

2.5. FTIR Spectrum and XPS Analysis

Fourier-transform infrared spectroscopy (Nicolet iS5, Thermo Fisher Scientific, Waltham, MA, USA) with a measuring range of 400–4000 cm^{-1} was used to analyze the FTIR spectrum of PHA, cerussite, and quartz before and after PHA treatment. The Thermo Scientific EscaLab 250Xi (Thermo Fisher Scientific, Waltham, MA, USA) measuring instrument was used to analyze the XPS spectrum of cerussite before and after PHA treatment. The pulp was prepared with 2 g of pure minerals and 30 mL of deionized water. After 1 h of stirring using a constant-temperature magnetic agitator, the filtered products were progressively rinsed with deionized water. The acquired solid samples were dried in a low-temperature vacuum drying oven and subsequently used for FTIR and XPS analysis. The date of infrared was analyzed through the use of OMNIC 8.2 software, and the XPS spectrum was fitted through the use of Advantage 5.9929 software.

3. Experimental Results

3.1. Micro-Flotation Experiments

3.1.1. Effect of pH

Figure 3 shows the effect of pH on the floatability of cerussite and quartz at a dosage of PHA, BHA, and sodium oleate (NaOL) of 20 mg/L. With an increase in the pulp pH value, the recovery of cerussite gradually decreases, and when the pH is greater than 9, the recovery of cerussite is less than 10%. It can be seen that the floatability of PHA, BHA, and NaOL for cerussite is basically the same, and the flotation effect is best at pH 7–8. Due to the increase in the pH value, the activity of the hydroxamic group decreases, and the ability of the $\text{Pb}(\text{OH})^+$ and $\text{Pb}(\text{OH})_2$ binding sites on the cerussite surface decrease, and the collecting ability of cerussite decreases accordingly [34]. At pH = 7, the recovery of PHA, BHA, and NaOL was 82.0%, 67.5%, and 56.0% for cerussite and 28.0%, 19.5%, and 1.0% for quartz, respectively. The flotation effect of PHA and BHA on quartz was not ideal at any pH value, which indicated that hydroxamic acid did not undergo chemical adsorption on the quartz surface because hydroxamic acid has no chemical affinity with the quartz surface. However, when the pH value was greater than 9, NaOL's ability to collect quartz significantly improved, and this result is consistent with that of a previous study [35]. The results showed that PHA has good collecting ability for cerussite and that PHA separates cerussite and quartz well at pH = 7.

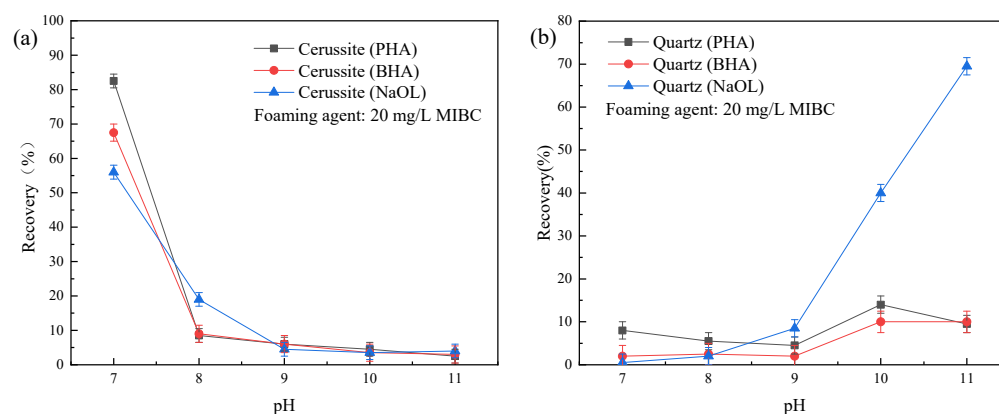


Figure 3. Effect of pulp pH on flotation recovery of cerussite (a) and quartz (b).

3.1.2. Effect of Collector Dosage

According to the pH flotation experiments, the optimal flotation pH of cerussite was selected as 7 for the collector dosage experiments, and the experimental results regarding collector dosage are shown in Figure 4. The recovery of cerussite increased with the increase in collector dosage. In Figure 4a, when the dosage of PHA was 30 mg/L, the recovery of cerussite reached 91.5%. Under the same conditions, the recovery of cerussite reached 81.5% when BHA was used as the collector, and the recovery of cerussite reached 60.5% when NaOL as the collector. These results show that the hydrophobic product is not formed by the collector in full contact with the surface of the cerussite at a low dosage and that the flotation recovery is not high. With the increase in the collector dosage, the chelation of the hydroxamic group and Pb ion is closer in order to promote the flotation process. Overall, although NaOL has a certain collecting ability for cerussite, it has a poor effect on the separation of cerussite and quartz, BHA has good selectivity but poor collecting ability for cerussite, and under the same conditions, PHA has better selectivity and collecting ability.

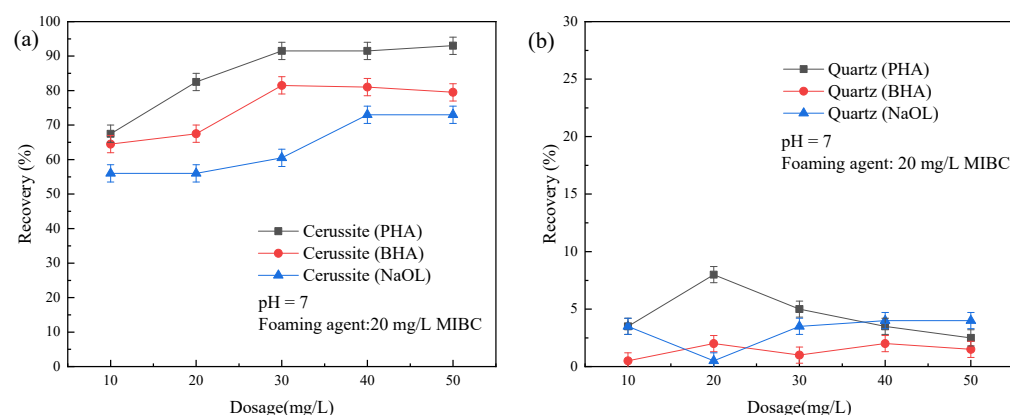


Figure 4. Effect of collector dosage on flotation recovery of cerussite (a) and quartz (b).

3.1.3. Artificial Mixed Minerals Flotation

To further explore the flotation performance of the collector, artificial mixed minerals experiments were conducted. This type of experiment simulates the occurrence conditions of minerals, meaning that it could more directly reflect the collector's ability and selectivity of cerussite and further highlight the advantage of the collector for the selective collection of cerussite. The artificial mixed minerals were composed of 1 g cerussite and 1g quartz, both with a Pb grade of 38.4%, and the results are shown in Figure 5. Under the conditions of pH = 7 and 20 mg/L of collector, the Pb grades of cerussite concentrates with PHA, BHA, and NaOL collectors were 44.5%, 25.4%, and 37.7%, respectively, and the recovery of Pb was 72.4%, 21.0%, and 39.4%, respectively. Comparing the effect of BHA and NaOL, PHA has better collecting ability, and it can also effectively separate cerussite and quartz.

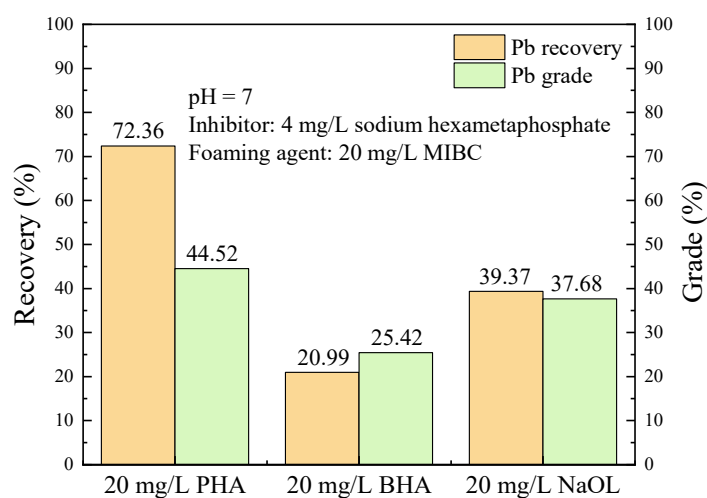


Figure 5. Grade and recovery rate of artificial mixed ore with different agents.

3.2. Adsorption Capacity Measurement

An ultraviolet spectrophotometer was used to measure the adsorption amount of residue on the minerals' surfaces. The standard absorbance curve was established based on the absorbance of different concentrations of PHA, and the measured PHA absorbance curve is shown in Figure 6a. The fitted function curve was $Y = 0.06408X - 0.04033$, and the variance was $R^2 = 0.99842$.

To further comprehend the quantitative adsorption capacity of PHA on mineral surfaces, adsorption measurement experiments were carried out on cerussite and quartz. The amount of agent adsorption of PHA on the minerals' surfaces is depicted in Figure 6b, and it can be clearly seen that the amount of agent adsorption on the minerals' surfaces showed an increasing trend as the concentration of PHA increased. When the concentration of PHA

was 50 mg/L, the adsorption capacity of PHA on the surface of cerussite was 0.59 mg/m², which was significantly higher than the surface adsorption capacity of quartz. This indicated that PHA was more adherent to the surface of cerussite compared with quartz, which was in alignment with the results of the flotation experiments. However, there is no clear linear relationship between the adsorption capacity and the flotation recovery, which may be explained by the fact that the effect of PHA is both related to its adsorption capacity and solubility.

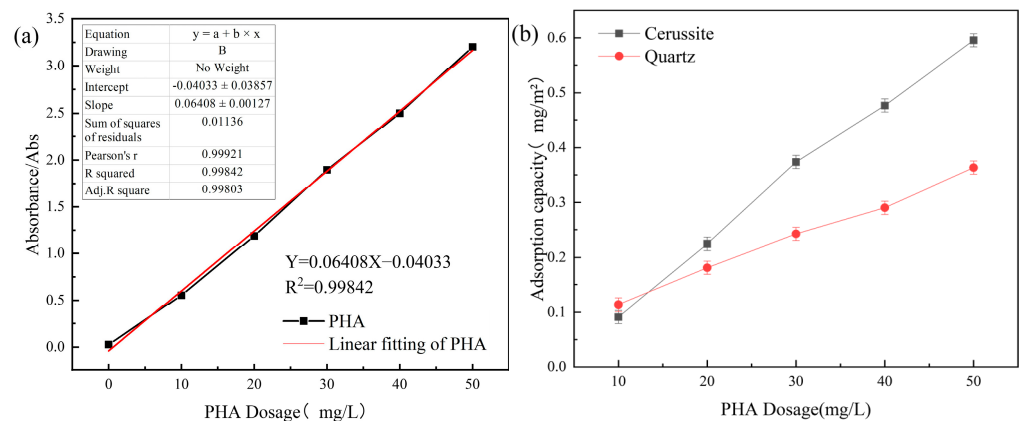


Figure 6. Relevance of the absorbance (a) and mineral adsorption capacity (b) of the PHA dosage.

3.3. Zeta Potential Results

The determination of zeta potential is an important measure to clarify the adsorption mechanism between the collector and the mineral surface in the flotation process. Figure 7 shows the variation in the zeta potential of cerussite and quartz at different pH values. As shown in Figure 7a, the isoelectric point of cerussite is 7.6, and this is consistent with previous reports [36]. The change in zeta potential values could reflect the interaction between the collector and minerals [20,22]. When $\text{pH} > 7$, the potential of cerussite decreases significantly, and the rate of decrease first showed a plateau effect and then an increase with the increase in pH value. In the presence of PHA, BHA, or NaOL, the PHA-treated cerussite shifted in the negative direction. This indicates that phenylpropenyl hydroxamic anions may be more tightly adsorbed on the surface of cerussite [29]. As shown in Figure 7b, the potential of quartz showed a decreasing trend in the experimental pH range, and the potential of the PHA-treated quartz had a small shift in the negative direction. This indicates that the adsorption of phenylpropenyl hydroxamic anions on the quartz surface was weak. The results indicated that PHA can adsorb more on the surface of cerussite compared to quartz.

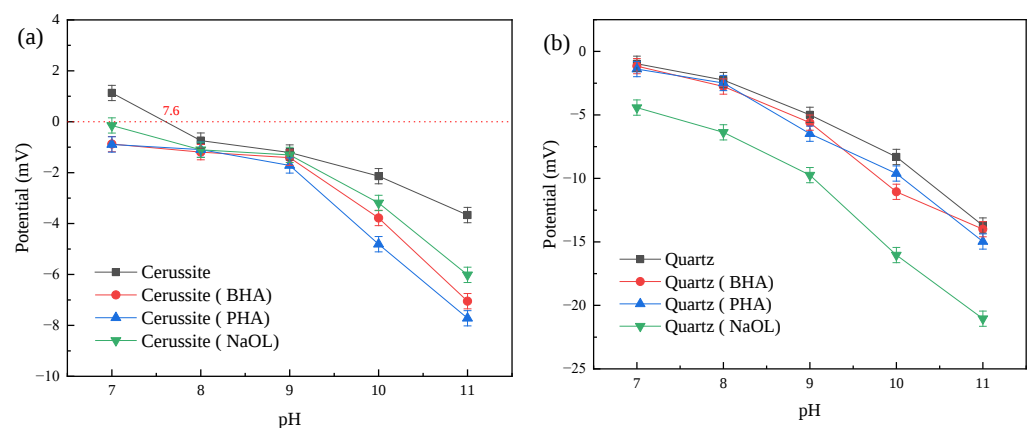


Figure 7. Effect of pH on the zeta potential of both cerussite (a) and quartz (b).

3.4. Infrared Spectrum Analysis

The FTIR spectrum of PHA and cerussite and quartz before and after PHA treatment are shown in Figure 8. In Figure 8a, the IR spectrum of PHA shows characteristic peaks corresponding to the benzene ring structure at 1424 cm^{-1} , 1449 cm^{-1} , 1494 cm^{-1} , the stretching vibration peak corresponding to the C-N at 1025 cm^{-1} , and the stretching vibration peak corresponding to the N-O at 969 cm^{-1} . The characteristic peaks of cerussite were 3630 cm^{-1} , 2409 cm^{-1} , 1725 cm^{-1} , 839 cm^{-1} , 782 cm^{-1} , and 679 cm^{-1} , while the characteristic absorption peaks of the PHA-treated cerussite were shifted to 3622 cm^{-1} , 2404 cm^{-1} , 1730 cm^{-1} , 840 cm^{-1} , 778 cm^{-1} , 680 cm^{-1} . The new absorption peaks correspond to stretching vibration of the C-N at 1055 cm^{-1} and the characteristic peaks of the benzene ring structure after PHA treatment at 1447 cm^{-1} . The above results show that the adsorption of PHA on the cerussite surface is chemical adsorption.

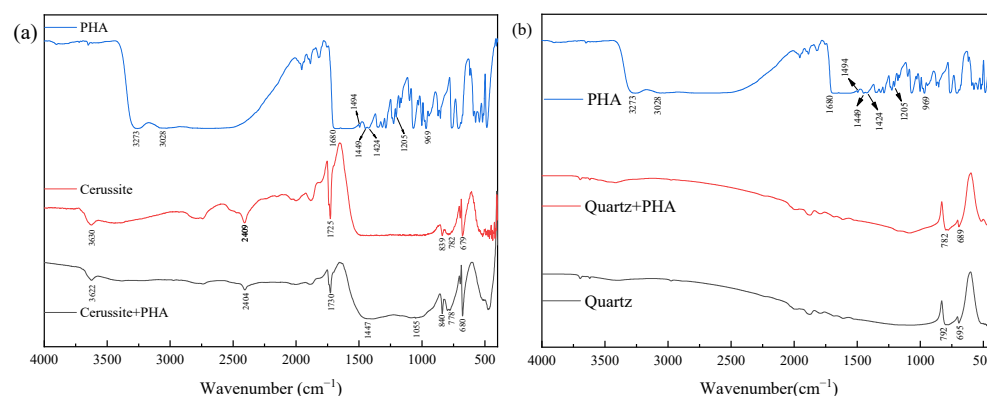


Figure 8. Infrared spectrum results for (a) cerussite and (b) quartz.

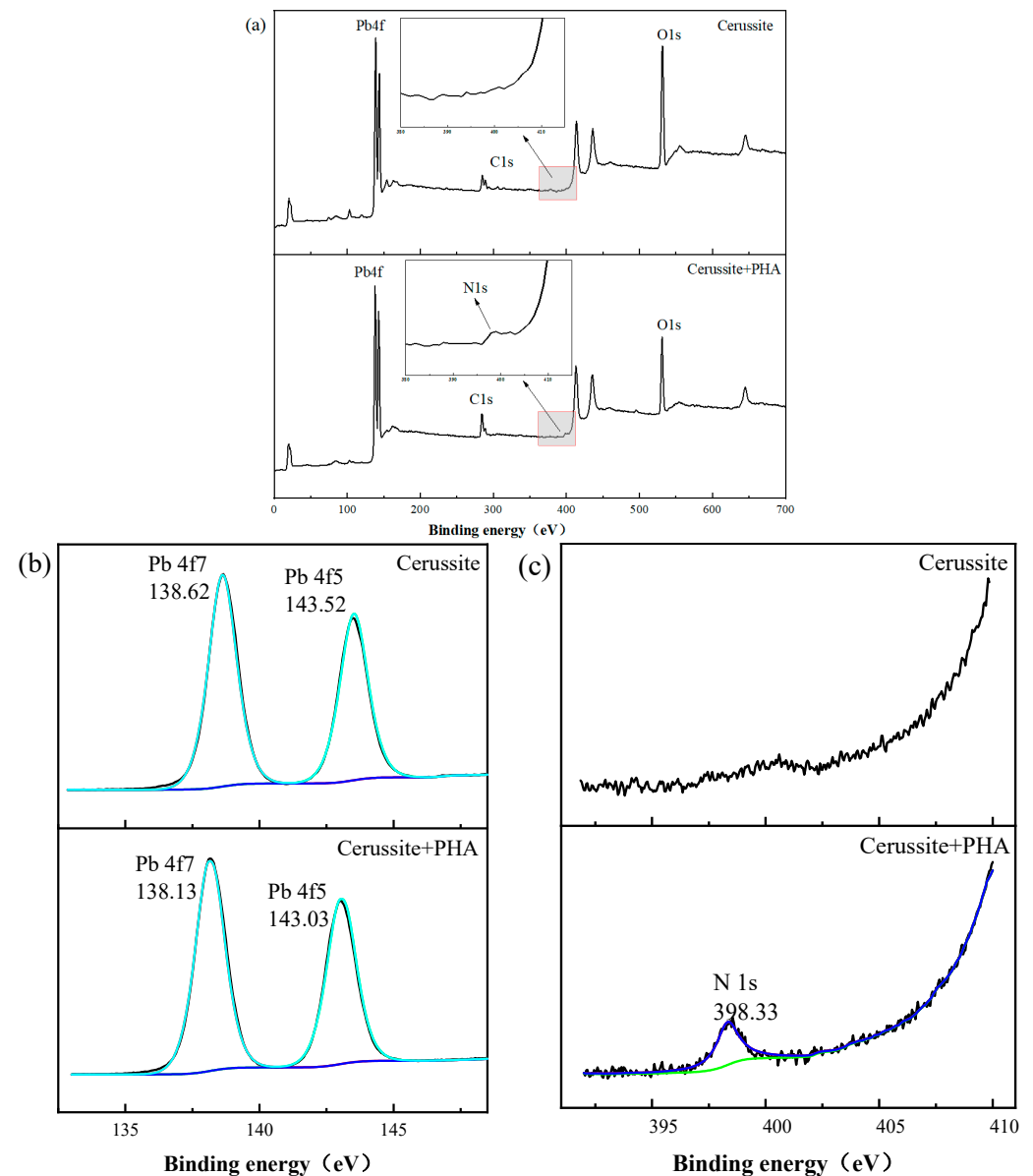
It can be seen from Figure 8b that after PHA treatment, the FTIR spectrum of quartz was essentially unchanged. Therefore, it can be concluded that PHA was not adsorbed on the quartz. This is consistent with the flotation results.

3.5. XPS Analysis

To further investigate the mechanism of PHA on the flotation separation of cerussite, the chemical changes on the mineral surface after PHA treatment (20 mg/L dosage) were determined via XPS analysis. The atomic relative concentration and binding energy of cerussite and the PHA-treated cerussite are shown in Table 2. The full XPS spectrum is shown in Figure 9a, in which it can be seen that the characteristic peaks of elements Pb4f, C1s, and O1s mainly exist in cerussite, and when the cerussite was adsorbed by PHA, the characteristic peaks of the N element appear near 400 eV. The high-resolution narrow region diagram of Pb is shown in Figure 9b; there are two characteristic peaks of Pb 4f7 and Pb 4f5 in its XPS spectrum because of the spin-orbit splitting of the outer electron of Pb. The binding energy of Pb 4f7 of the pure mineral is 138.62 eV, which was the characteristic peak of the Pb element in metal carbonate; after the addition of PHA, the binding energy of Pb 4f7 shifts to 138.13 eV, which was the characteristic peak of Pb-O, indicating that after the reaction, the effect of PHA on the surface of cerussite leads to the formation of a new chemical bond on the Pb atom on the surface of cerussite via combining with the O atom. The high-resolution narrow region of the N element is shown in Figure 9c. Regarding the pure cerussite mineral, the characteristic peak of the N element could not be detected via XPS, indicating that the N element was not present on the surface of cerussite, which was consistent with the results of the full spectrum; after the addition of PHA, a characteristic peak of metal nitride appeared at 398.33 eV for N 1s, indicating that the characteristic peak of Pb-N appeared on the surface of cerussite after the reaction, and that the surface of the cerussite is chelated with the N atom in PHA, which made PHA adsorbable on the surface of the cerussite, thus enhancing the hydrophobicity of the cerussite.

Table 2. Atomic relative concentration and binding energy of cerussite interacting with PHA.

Sample	Atomic Orbitals	Relative Concentration/%	Binding Energy/eV	Binding Energy Offset/eV
Cerussite	Pb 4f7	42.46	138.62	$\Delta\text{Pb } 4f7 = -0.49$
	N 1s	0	-	
Cerussite + PHA	Pb 4f7	53.32	138.13	$\Delta\text{N } 1s = 398.33$
	N 1s	1.17	398.33	

**Figure 9.** The survey scan and XPS analysis results of cerussite before and after PHA treatment. (a) the full XPS spectra. (b) the XPS spectra of Pb 4f5 and Pb 4f7. (c) the XPS spectra of N 1s.

4. Conclusions

In this paper, a kind of high-efficiency hydroxamic acid collector (PHA) was used in the separation flotation of cerussite and quartz to study the effect of PHA on cerussite and quartz. The effective separation of cerussite and quartz was achieved via micro-flotation experiments; the adsorption mechanism of PHA on the surface of cerussite was determined by carrying out adsorption capacity experiments, zeta potential tests, FTIR spectroscopy, and XPS analysis. The results are as follows:

- (1) The pure mineral flotation experiments showed that PHA has a higher collecting ability than BHA and NaOL, and the artificial mixed mineral experiments proved that PHA has a good flotation separation effect on cerussite and quartz. At pH = 7, the grade of cerussite increased from 38.4% to 44.5%, and the flotation recovery was 72.4%.
- (2) The adsorption capacity and zeta potential experiments indicated that PHA was more adsorbed on the surface of cerussite than quartz; the adsorption capacity of PHA is better than that of BHA and NaOL.
- (3) Our infrared results showed that new absorption peaks appeared in the PHA-treated cerussite, while the peak intensity of quartz did not change significantly, indicating that PHA promoted the selective flotation of cerussite.
- (4) Our XPS analysis showed that PHA was adsorbed on the surface of cerussite through the complexation between the hydroximic group and Pb ions on the surface of cerussite, changing its surface properties and improving its floatability.

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