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Sedimentation of Fine Arsenopyrite with PEI and the Flotation Significance

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Abstract: The flotation of fine mineral particles is always a difficult problem. The flotation of fine arsenopyrite particles (−20 μm) in a sodium butyl xanthate (SBX) system was studied by using polyethyleneimine (PEI) as a flocculant. The flocculation properties of PEI on fine arsenopyrite were studied using sedimentation tests. The results showed that the optimum pH for the sedimentation of PEI was approximately 7.5; the higher the molecular weight (M.W.) of the flocculant, the better the sedimentation effect. In the flotation experiments, it was found that the flotation recovery of PEI-3 with high M.W. as flocculant was only 57%, while the flotation recovery of PEI-2 with medium M.W. was 90% under respective optimum conditions. The contact angle tests showed that the natural contact angle of arsenopyrite was 37°; the addition of moderate PEI-2 had a slightly negative influence on the hydrophobicity of arsenopyrite in the SBX system. From the size analysis results, the maximum particle size (D_{100}) and median size (D_{50}) of the arsenopyrite increased from 20 and 11 μm to 48 and 28 μm after treatment with 40 mg/L PEI-2, a size more conducive to bubble capture. From the combination of these results, it can be concluded that PEI-2 improved the flotation of fine arsenopyrite mainly by increasing the particle size to a suitable range through flocculation. The XPS results indicated that the adsorption of PEI-2 on the arsenopyrite surface was due to the chemisorption between the imino group and the active Fe/As sites. Applying PEI-2 to a fine disseminated arsenopyrite-type gold ore, a concentrate containing 36 g/t Au with a Au recovery of 88% can be obtained.

Keywords: flocculation; arsenopyrite; particle size; adsorption; flocculant

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1. Introduction

The Wulonggou gold mine is located in Qinghai Province, Western China. It is one of the largest ore concentration areas in the east Kunlun metallogenic belt [1]. The ore is mainly disseminated and veined disseminated and has a brecciform, fine-grained structure. The main mineral assemblage is sericite, (white) mica, quartz, pyrite and arsenopyrite. Gold elements mainly exist in metallic sulfides, such as arsenopyrite and pyrite, in an ultra-microscopic state, and the distribution ratio of Au in arsenopyrite is approximately 70% [2]. Since the disseminated size of arsenopyrite and pyrite in ore is becoming finer, the grind fineness must be finer so that Au-bearing minerals can be liberated from gangues; now, the flotation indexes are deteriorating, highlighted by the decrease in the flotation recovery of Au (arsenopyrite) and the increase in the Au grade of tailings. The main approach under consideration at present is to seek new effective flotation reagent regimes that can improve the recovery of fine particles, as the existing flotation equipment must be adequately utilized.

Fortunately, researchers have a number of methods to make fine particles grow, by which the flotation of fines would become relatively easy [3,4]. Suitable electrolytes can make fine particles grow to large sizes through the mechanism of compressing double-layer particles (agglomeration) [5]; some long-chain polymers can make fine particles grow to large sizes through the mechanism of the bridging effect (flocculation) [6]; some hydrophobic organics can make fine particles grow to large sizes through the mechanism of the hydrophobization of the mineral surface (hydrophobic attraction) [7]. Among them, the flocculation of fine particles through long-chain polymers is the most frequently used method. The linear polymer polyacrylamide (PAM) is freely soluble in water, so it is widely applied in mining and environment industries, etc. [8]. PAM can be grafted in different groups to form cationic polyacrylamide (CPAM) or anionic polyacrylamide (APAM), by which the chemical interaction between the grafted group of PAM and mineral surface becomes possible [9]. However, the depression of such PAM-based flocculants is relatively strong, which would negatively affect the recovery of other valuable minerals. For example, a PAM-based flocculant was used in fine copper sulfide mineral flotation to improve Cu recovery, but the Mo recovery showed a significant decrease [10].

Therefore, it is important to search for new flocculants. Polyethyleneimine (PEI), a cationic flocculant, is often used to remove metal cations in wastewater through chelation, so it has the potential to be used as a flotation reagent because the interaction of a reagent with a mineral also takes place through the adsorption between the special groups of the molecule and the active sites (metal sites) on the mineral surface [11]. Here, it is employed as a new reagent in a fine arsenopyrite flotation system to mainly reflect its flocculation effect. The adsorption of PEI on the arsenopyrite surface and its effect on the flotation performance of sodium butyl xanthate (SBX) are studied through flotation tests, and the improving mechanism is also discussed.

2. Experimental Procedure

2.1. Materials

The arsenopyrite used in the experiment was obtained from Dulan Jinhui Mining Co., Ltd. Parts of the ore samples were handpicked and then ground in a ceramic ball mill. Finally, −20 μm arsenopyrite was obtained through the elutriation method and was used for sedimentation, flotation, size analysis and X-ray photoelectron spectroscopy (XPS) tests. The X-ray diffraction (XRD) spectra (Figure 1) of the samples showed that the purity of arsenopyrite met the requirement through comparison with the corresponding PDF cards in Jade (version 6.0, Materials Data, Inc., Leominster, MA, USA) [12]. The high purity of the arsenopyrite sample was also demonstrated by the chemical element analysis results of 43.46% As, 19.64% S and 32.44% Fe, which were very close to the stoichiometric ratio of arsenopyrite. A massive arsenopyrite was cut into a 3 cm × 3 cm × 1 cm cube, with one of the 3 cm × 3 cm surfaces being polished using an automatic target surface processor (EM TXP, Leica Microsystems, Wetzlar, Germany), which was used for the contact angle measurement.

NaOH and H_2SO_4 stock solutions were used as the pH regulators, except for bench scale flotation, where Na_2CO_3 was the pH modifier. Sodium butyl xanthate (SBX) was used as the collector in the micro-flotation tests, while in the bench-scale flotation of the real ore, s mixture of ammonium dibutyl dithiophosphate (ADD) and SBX was employed. PEIs with different M.W. (PEI-1, 3500; PEI-2, 10,000; PEI-3, 50,000) were used as the flocculants. Methyl isobutyl carbinol (MIBC) was used as the frother for all the flotation tests. The monomer molecular structure of PEIs is shown in Figure 2. Deionized water (18.25 MΩ·cm) was used for pure mineral tests; tap water was used for bench-scale flotation tests.

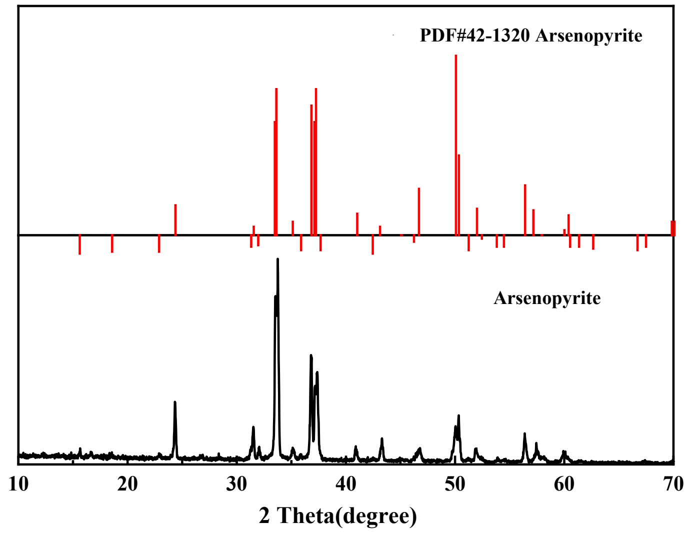


Figure 1. X-ray diffraction patterns of arsenopyrite with corresponding PDF cards.

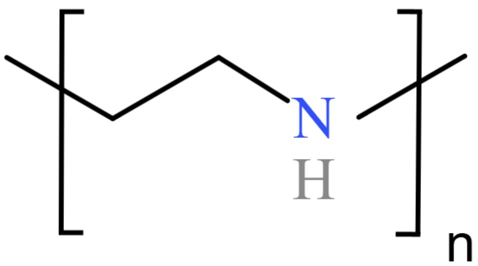


Figure 2. Monomer structure of linear polyethyleneimine.

2.2. Sedimentation Tests

Sedimentation characteristics could be used to understand the flocculation properties of fine arsenopyrite under different flocculation conditions. In the sedimentation tests of arsenopyrite, 5 g of powder, together with 90 mL of deionized water, was added to a 100 mL beaker. After adjusting the slurry pH to the desired value, the corresponding reagent(s) were added. Each interaction time was 2 min for the reagent with arsenopyrite particles under magnetic agitation. Then, the slurry was transferred to a 100 mL graduated cylinder and agitated vigorously to remain fully suspended. After the stirring was stopped, the settling behavior of the slurries was observed after a specified time by recording the solid–liquid interface. The fraction over the interface was then pored off, and the remaining part was filtered, dried and weighed to calculate the sedimentation yield [13].

2.3. Flotation

2.3.1. Micro-Flotation

Floatability tests were conducted in a 60 mL micro-flotation cell. Pure mineral particles (2.0 g) were placed in a plexiglass cell for 2 min to be conditioned, and then, a pH regulator was added to adjust it to the required pH. Under this desired pH, PEI flocculant was added, followed by SBX collector, with 3 min of stirring time after each addition. After all of the above was finished, the flotation process was performed for 4 min. After being filtered and dried, the froth products together with the tails were separately weighed. Then, the recovery was calculated in accordance with the dry weight of the products (Equation (1)). The researchers established three flotation tests in the same conditions to report the average values of the three methods.

$$R = W_C/(W_C + W_T) \quad (1)$$

where Wc is the weight of the concentrate and W_T is that of tailings.

2.3.2. Bench Scale Flotation

The bench-scale flotation experiments of the actual ore were carried out in an XFD-63 flotation cell, with 1500 mL rougher–scavenger flotation and 500 mL cleaner flotation. The impeller speed was 1650 rpm. Froth products were collected using an automatic froth scraping device. In the flotation stage, the required reagents were successively added to the flotation cell with an interval of 3 min and a flotation time of 6 min. The final products of concentrate and tailings were filtered, dried, weighed and analyzed with Au. The bench-scale locking cycle test process is shown in Figure 3. The experimental system of the real ore used the SBX + ADD as the collector.

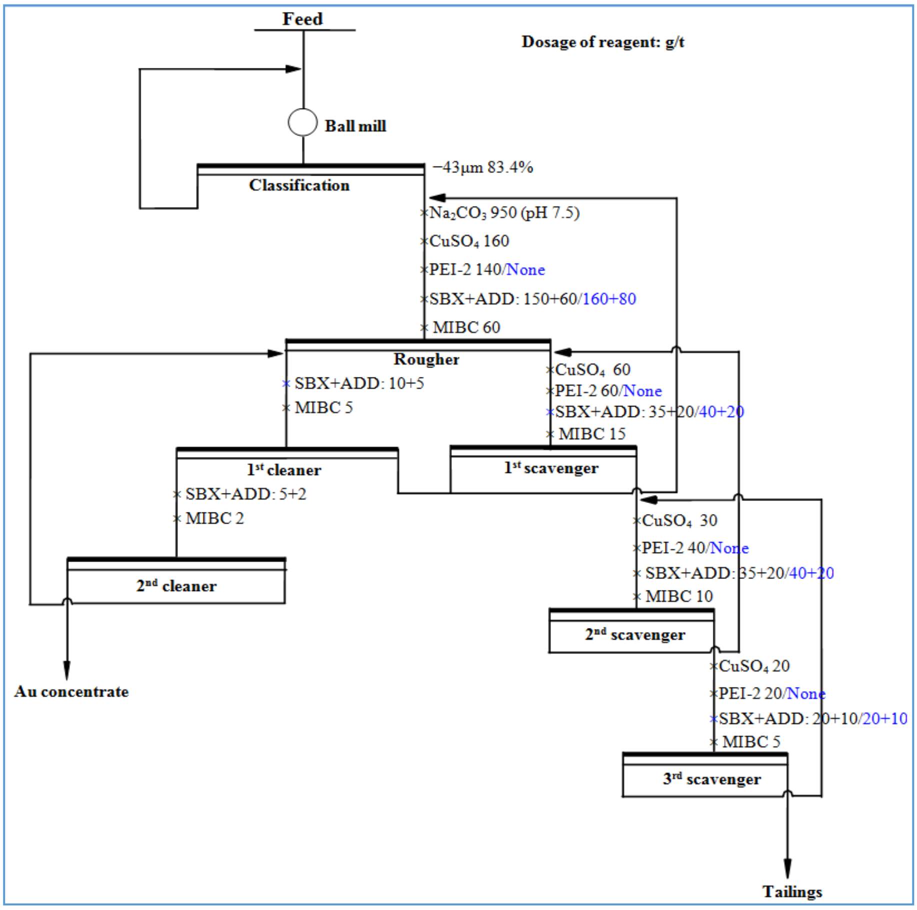


Figure 3. Flowsheet and corresponding conditions of bench-scale locked cycle tests.

2.4. *Particle Size Distribution Analysis*

Particle size distribution could directly reflect the flocculation behaviors between arsenopyrite and PEI-2. The −20 μm samples were treated with different concentrations of PEI-2 for 3 min to obtain the flocculated suspension. The laser-based particle size analysis was carried out on the treated and untreated samples with a Master-size 2000 instrument (Malvern Panalytical Ltd., Malvern, UK). Ultrasonics were not applied to all the suspensions to prevent damage to the flocs.

2.5. *Contact Angle Measurement*

The contact angle was measured using a Rame-Hart goniometer (Model 590, Rame-Hart, Succasunna, NJ, USA) through the sessile drop method [14]. The cube arsenopyrite (3 cm × 3 cm × 1 cm cube with one polished 3 cm × 3 cm surface) was conditioned with different reagents by immersing it in the corresponding reagent solution for 3 min. After, the washing and drying of the tablets were conducted. The contact angle value was determined by recording the image of the prepared sample with the water droplet. For each specific measurement, at least three tests were conducted at different locations on the surface. The reported value was the average of these measurements.

2.6. XPS Detection

A 1.0 g sample of −20 μm arsenopyrite powder was added into the 60 mL microflotation cell together with 40 mL distilled water. The pH was adjusted to 7.5, upon which the desired amount of PEI-2 was added and stirred for 10 min at 1900 r/min. After filtration and vacuum drying, the XPS spectra for arsenopyrite samples treated with or without PEI-2 were recorded using a K-Alpha 1063 spectrometer (Thermo Scientific Co., Waltham, MA, USA), which employs Al Kα as a sputtering source at 12 kV and 6 mA with 1.0×10^{-9} Pa pressure in the analytical chamber. The curve was fitted using XPSPEAK 4.1 software (version 4.1, Taiwan, China).

3. Results and Discussion

3.1. Sedimentation Tests

PEI dosage, pH and sedimentation time were the key factors affecting the settling. The influence of these factors on arsenopyrite (−20 μm) settlement was studied using systematic sedimentation tests. The experimental results are shown in Figure 4. As could be seen from Figure 4a, except for PEI-1, the sedimentation efficiency increased with the increase in the dosage of flocculant. According to the curve trend, PEI-3 had the best effect, with the highest sedimentation yield of 96% when the dosage was 200 mg/L. When the dosage surpassed 120 mg/L, the sedimentation yield increased slowly, and it was selected for further tests.

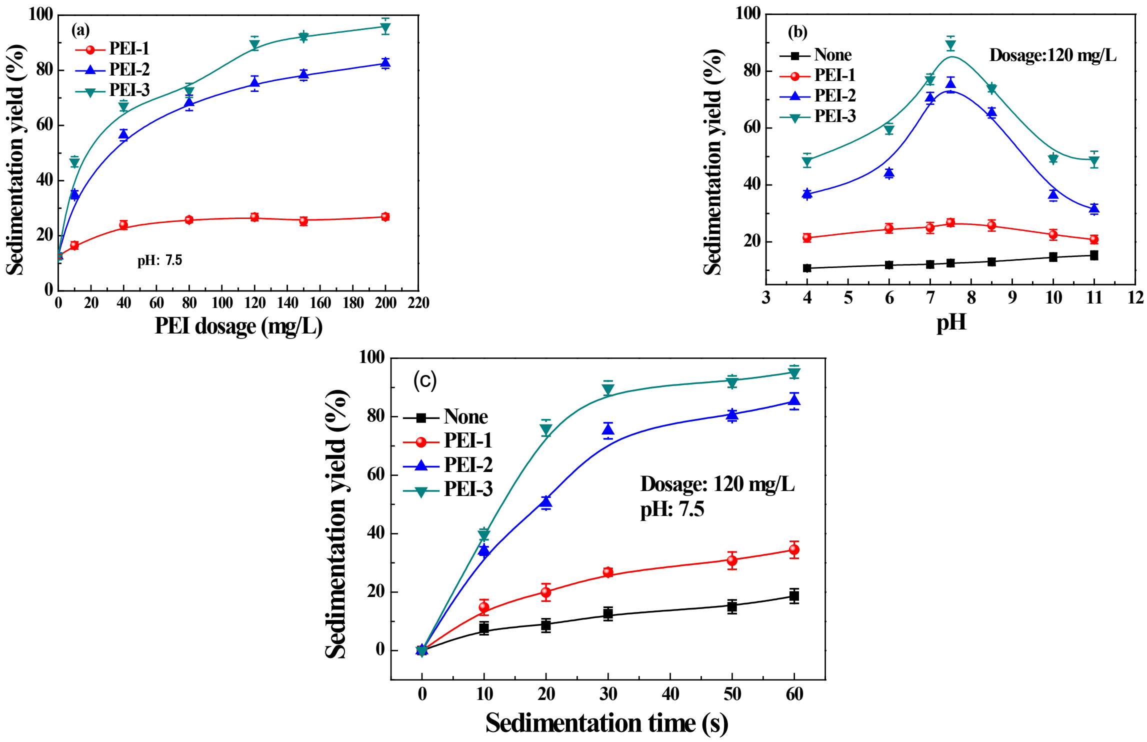


Figure 4. Effects of PEI dosage (pH = 7.5; sedimentation time 30 s) (**a**), pH (dosage, 120 mg/L; sedimentation time 30 s) (**b**) and sedimentation time (pH = 7.5; dosage, 120 mg/L) (**c**) on sedimentation yield.

The influence of pH on PEI sedimentation efficiency is shown in Figure 4b. The sedimentation efficiency of minerals in PEI-2 and PEI-3 systems increased first and then decreased with the increase in pH. The optimal value of PEI-3 was 90%, while the sedimentation yield without flocculant was only 12.6%. When pH exceeded 7.5, the decrease in sedimentation efficiency may have been caused by the decationization of the PEIs under alkaline conditions [15,16]. Therefore, the pH of 7.5 was determined as the optimum condition for the sedimentation tests.

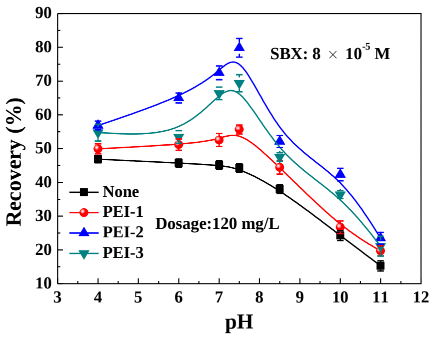
If the sedimentation time was set as the independent variable, it was found that regardless of PEIs, the sedimentation efficiency increased with the increase in time (Figure 4c). When the sedimentation time was 60 s, the sedimentation yield was only 18.66% without flocculant, but it was as high as 95% with the addition of PEI-3.

In conclusion, under the same conditions, the higher the molecular weight of the flocculant, the better the sedimentation efficiency. In flotation experiments, however, a different result was achieved.

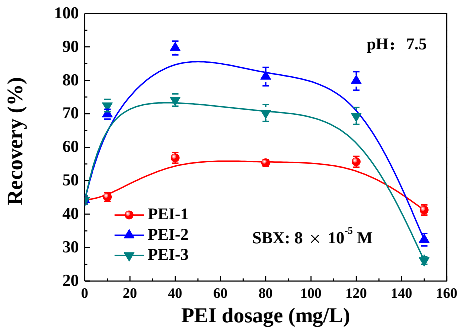
3.2. Flotation Tests

3.2.1. Micro-Flotation

A single mineral flotation test was conducted to explore the effects of PEI dosage, pH and SBX concentration on the flotation recovery of arsenopyrite with SBX as the collector, and the results are shown in Figure 5. As shown in Figure 5a, without PEI, the flotation recovery of arsenopyrite was lower than 50% and decreased with the increasing pH, especially when the pH was higher than 7.5. The addition of PEI-1 slightly improved the recovery of arsenopyrite in the pH range of 4–11, while the addition of PEI-2 and PEI-3 significantly improved the flotation recovery of arsenopyrite. At pulp pH 7.5, the recovery of arsenopyrite was only 44% without flocculant, while the optimum recovery increased to 80% with 120 mg/L PEI-2 as the flocculant in 8 × 10^{-5} M SBX solution.



(**a**)



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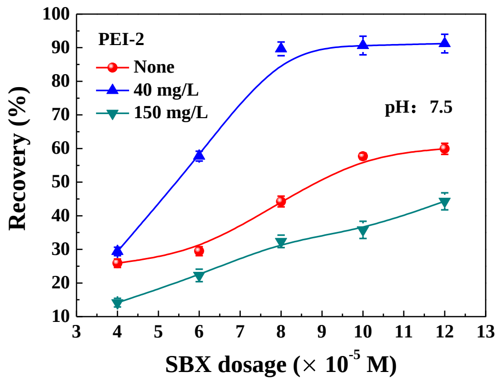

(**c**)

Figure 5. Effects of (**a**) pH (PEI, 120 mg/L; SBX, 8 × 10^{-5} M), (**b**) PEI dosage (pH, 7.5; SBX, 8 × 10^{-5} M) and (**c**) SBX dosage (pH, 7.5) on the recovery of arsenopyrite.

Fixing the pH to 7.5, the influence of the PEIs dosage on mineral recovery was investigated. As illustrated in Figure 5b, with the increasing PEI dosage, the flotation recovery increased first and then declined. The best dosage of PEI was 40 mg/L at the conditions of pulp pH 7.5 and SBX dosage 8×10^{-5} M. When the dosage of flocculant exceeded 120 mg/L, the recovery of arsenopyrite decreased. Evidently, adding moderate flocculant could improve the flotation of fine arsenopyrite particles, which was consistent with the previous study [17]. Additionally, in the two flotation tests, moderate PEI-2 showed the best improvement in the flotation of arsenopyrite.

With different dosages of PEI-2, the influence of SBX dosage on the recovery of arsenopyrite was researched, and the results are shown in Figure 5c. The recovery increased with the increasing SBX dosages. When the dosage of PEI-2 was 40 mg/L, its addition improved arsenopyrite flotation, while with 150 mg/L PEI-2, the recovery was even lower than that without additives, which was ascribed to the excessive flocculant, resulting in a particle agglomeration size that is too large, thus causing bubble rupture [18].

In the settlement experiment, the best settlement effect was PEI-3 (maximal M.W.). However, in the micro-flotation experiment, the best flotation recovery of arsenopyrite was PEI-2 (medium M.W.). The reason for this phenomenon was that the high molecular weight of PEI-3 resulted in the optimal flocculation ability and made the particle size of the flocculated fine particles of arsenopyrite too large, which would increase the probability of particle desorption from the bubble [19].

3.2.2. Bench-Scale Flotation

Based on the results of micro-flotation tests and the subsequent flotation condition tests on the real ore, locked cycle testing was carried out at batch scale using SBX + ADD as a mixed collector in weakly alkaline pulp (pH 7.5). The closed flotation circuit included one roughing, three scavenging, and two cleaning steps (shown in Figure 3), and all the reagent dosages and grinding fineness presented are the optimum values. The flotation results achieved with and without PEI-2 are displayed in Table 1. A concentrate containing 31.44 g/t Au with the Au recovery of 77% was achieved without PEI-2 in the flotation process at pH 7.5. When PEI-2 was added into the pulp before collectors in the roughing and scavenging stage, a concentrate with the Au grade of 36 g/t and Au recovery of 88% was obtained. For the fine-grained disseminated refractory gold ore, the flocculant PEI-2 improved the flotation of the gold-bearing arsenopyrite-type ore, and with PEI-2, a higher grade and recovery of gold concentrate could be obtained in the weak alkaline pulp. It is of great industrial application potential in fine particle flotation.

Table 1. Values of closed flotation circuit.

System	Products	Ratio (w/%)	Au Grade (g/t)	Au Recovery (%)
Without PEI-2	Au concentrate	5.39	31.44	77.28
	Tailing	94.61	0.53	22.72
	Feed	100	2.19	100
With PEI-2	Au concentrate	5.35	35.99	87.92
	Tailing	94.65	0.28	12.08
	Feed	100	2.19	100

3.3. Size Analysis Results

The addition of PEI-2 could increase the particle size of fine arsenopyrite according to the results of sedimentation tests. Here, the size testing was conducted on the particles treated with 0, 40 and 150 mg/L PEI-2 using a laser particle sizer.

The results shown in Figure 6 provide intuitive data. The maximum particle size (D_{100}) and median size (D_{50}) of the arsenopyrite without PEI treatment were 20 and 11 μm, which were far below the sizes that can be effectively floated [20], while the corresponding data increased to 48 and 28 μm after treatment with 40 mg/L PEI-2. This increase in particle size was responsible for the improvement in sedimentation yield (Figure 4) and flotation recovery (Figure 5). When the PEI-2 dosage was 150 mg/L, the flocculated arsenopyrite suspension presented the D_{100} and D_{50} of 116 and 69 µm, respectively. These values increased to approximately six times those of the original ones, which resulted in a greater sedimentation yield but lower flotation recovery [21]. These size analysis results further demonstrated the conclusion regarding the size increase according to the sedimentation tests.

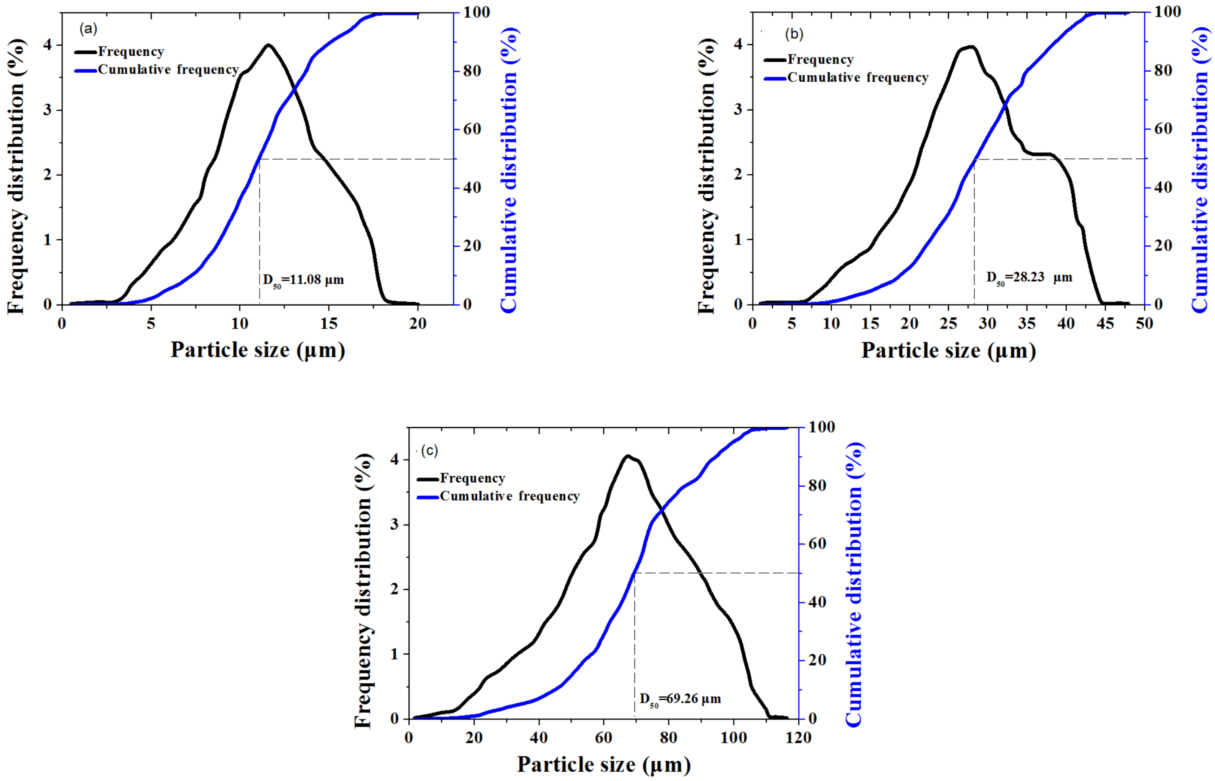

Figure 6. Size distribution of the arsenopyrite particles treated with (**a**) 0, (**b**) 40 and (**c**) 150 mg/L PEI-2. (Dotted line represents 50% cumulative yield corresponded particle size.)

3.4. Contact Angle Measurement

Enhancing the recovery of fines can be accomplished by increasing the particle size to a suitable range or improving the hydrophobicity of the particle surface [3]. The contact angles of the arsenopyrite surface under different conditions were measured, which, combined with the size analysis results, are shown in Figure 6 to determine the relationships between the good flotation recovery and the two factors.

The contact angle results are shown in Figure 7. The natural contact angle of arsenopyrite was 36°, which is close to some of those reported in the literature [22]. When it was treated with distilled water of pH 7.5, the contact angle decreased slightly, mainly due to the formation of hydrophilic metal hydroxyl compounds [23]. For the surface treated with 8×10^{-5} M SBX (pH 7.5), the contact angle shifted to 62°, showing that SBX could greatly improve the hydrophobicity of arsenopyrite. At this condition, the recovery was only 44%, as shown in Figure 5a. However, when the arsenopyrite surface was treated with 40 mg/L PEI-2 solution, the contact angle dropped to 28°, indicating that it resulted in a decrease in the hydrophobicity of the surface due to its hydrophilicity. When the surface was treated with 40 mg/L PEI-2 solution first and 8×10^{-5} M SBX after, the contact angle of 57° was obtained, slightly lower than that with a single SBX. However, the flotation recovery was 79% under this condition (Figure 5). Therefore, the improvement of PEI-2 upon arsenopyrite flotation in the SBX system was not due to increasing the hydrophobicity of the arsenopyrite surface but due to increasing the particle size to a suitable range via the flocculation effect.

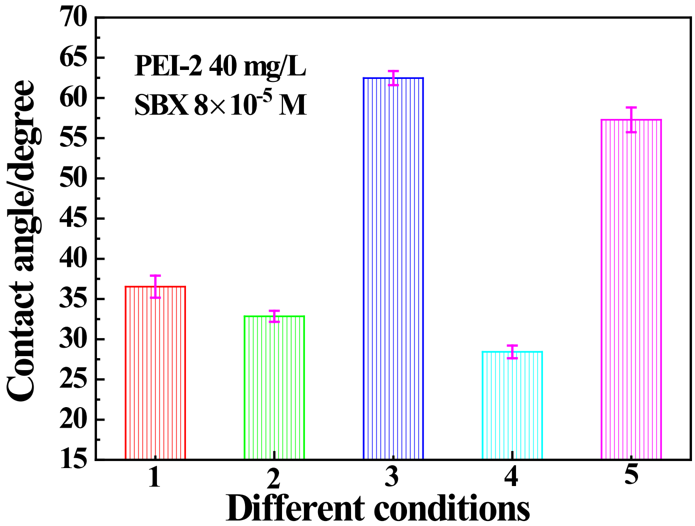

Figure 7. Contact angle measurement results under different conditions (1—natural; 2—treated with aqueous solution at pH 7.5; 3—treated with 8 × 10^{-5} M SBX at pH 7.5; 4—treated with 40 mg/L PEI-2 at pH 7.5; 5—treated with 40 mg/L PEI-2 then 8 × 10^{-5} M SBX, at pH 7.5).

3.5. XPS Analysis

As shown by the size analysis and contact angle results, with moderate PEI-2 flocculant (40 mg/L), the arsenopyrite size had a modest increase (D_{50} 28 μm), while the contact angle had only a very mild decrease in the SBX system. This is the reason for the improvement in PEI-2 upon the xanthate flotation of fine arsenopyrite. Studies on the adsorption interaction between SBX and sulfides are relatively abundant [24], but those on the PEI/arsenopyrite are few. Therefore, the XPS data of the arsenopyrite surface before and after treatment with PEI-2 were detected and analyzed. The As 3d and Fe 2p spectra are shown in Figure 8.

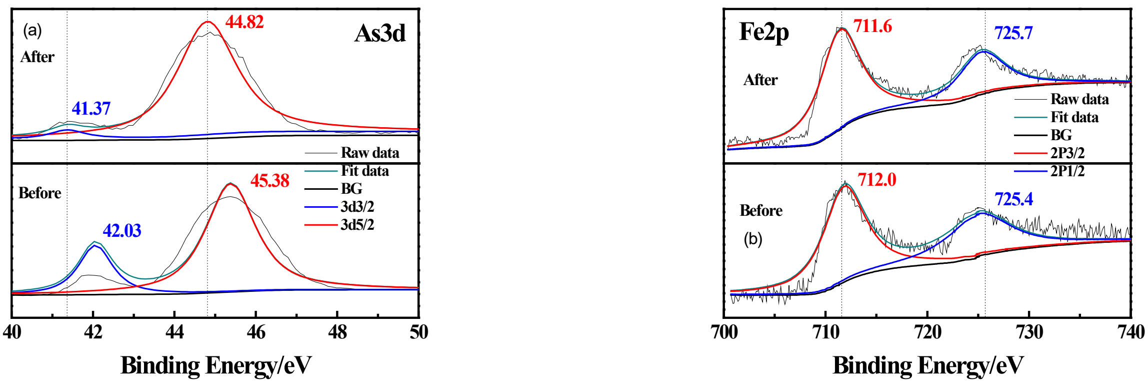

Figure 8. The (**a**) As 3d and (**b**) Fe 2p spectrum before and after interaction with PEI-2.

According to previous reports, the As and Fe atoms on the arsenopyrite surface were all likely to be the active sites for flotation reagent adsorption [25]. Without PEI interaction, the As 3d 3/2, As 3d 5/2, Fe 2p 1/2 and Fe 2p 3/2 peaks occurred at 41.37, 44.82, 711.6 and 725.7 eV, respectively. The corresponding BE shifted by 0.66, 0.56, 0.4 and −0.3 eV (all over the instrumental error of 0.2 eV), indicating the occurrence of chemical changes in the Fe and As active sites, i.e., the chemisorption of PEI-2 on the arsenopyrite surface. In the treatment of wastewater containing heavy metal ions, PEIs have been used as flocculants to collect these cations through coordination with the imino groups [26]. The minerophilic group of PEIs is the imino group. Different imino groups in the polymer can coordinate with the Fe/As atoms on the mineral surface, and some of the remaining imino groups are adsorbed with the active sites into other particles, leading to the bridging flocculation of the fine arsenopyrite particles.

3.6. Interaction Mechanism Model

Figure 9 shows the possible mechanism of flocculant PEI-2 in the flotation of fine arsenopyrite (−20 μm, D_{50} 11 μm) with collector SBX. The mineral particles with small sizes had a low collision probability with bubbles and thus had a low recovery (Figure 5), which was the reason to consider adding flocculants. When PEI-2 was added, these fines would form bigger particles through the bridging action of PEI-2 molecules. The adsorption of PEI-2 on the arsenopyrite surface was due to the coordination adsorption between the imino group and the active Fe/As sites. Different agglomerations can also further grow via the hydrophobic force between the hydrophobic group of different PEI-2 molecules. These resulted in an appropriate particle (agglomeration, D_{50} 28.23 μm) size for flotation. The hydrophobicity of the arsenopyrite surface did not reduce to a large extent (Figure 6), so it still had enough active sites for the collector SBX adsorption. Hence, the flotation of fine arsenopyrite in the SBX system could be improved by the addition of PEI-2 (Figure 5).

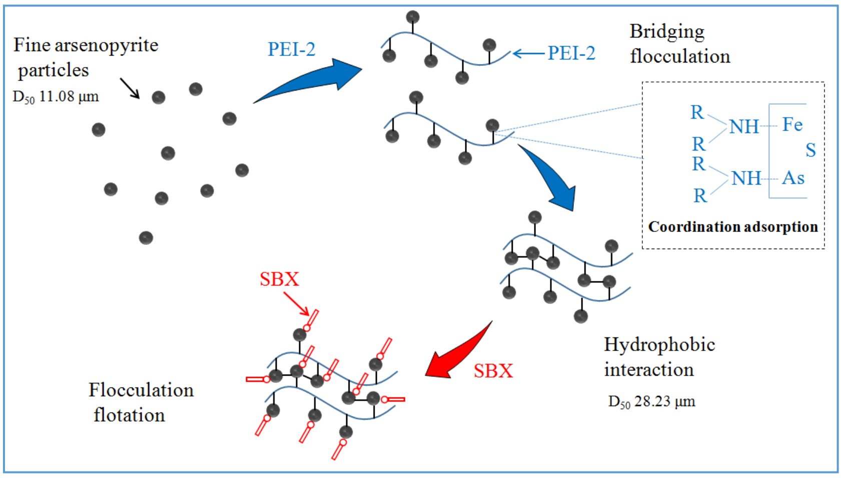

Figure 9. The possible flocculation mechanism of PEI-2 improved the flotation system of arsenopyrite.

4. Conclusions

In the xanthate system, arsenopyrite with a small particle size (−20 μm) had a poor flotation effect. This could be improved by adding moderate flocculant PEI-2, which effectively increased the particle size from D_{50} 11 μm to 28 μm at optimum dosage. Therefore, it improved the flotation of fines mainly by increasing the particle size to a suitable range through flocculation. The adsorption of PEI-2 on the arsenopyrite surface was through the coordination adsorption between the imino group and the active Fe/As sites. For a disseminated arsenopyrite-type gold ore, with the application of PEI-2, a better concentrate containing 36 g/t Au with Au recovery of 88% can be obtained with PEI-2.

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