Flotation Dephosphorization of High-Phosphorus Oolitic Ore

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Abstract: The feasibility of dephosphorization using the flotation process and its mechanism of high-phosphorus oolitic hematite were discussed in this paper. The results showed that phosphorus minerals mainly exist in the form of collophane, which can be divided into three types. Block collophane and band collophane could be effectively removed through flotation, while the third type could not be eliminated through physical concentration alone. A lab-made fatty acid was identified as an effective collector for high-phosphorus oolitic hematite, resulting in a product containing 57.67% iron and 0.14% phosphorus with a flotation recovery rate of 82.43%. The study of the flotation mechanism revealed that, in the presence of starch and the lab-made fatty acid, there was both physical absorption and chemisorption occurring on the surfaces of apatite. However, only very weak physical absorption was observed on the surface of hematite.

Keywords: dephosphorization; oolitic hematite; flotation; mechanism

1. Introduction

In recent decades, iron ore resources have been consumed in large quantities, and an increasing number of complex refractory iron ores have attracted attention. Among these, oolitic ore is particularly valuable due to its large reserves. These resources are widely distributed in countries such as Pakistan, the United States, Canada, United Kingdom, France, Germany, Nigeria, Saudi Arabia, Algeria, and China, with a total reserve of more than 140 billion tons [1–5]. However, a large number of oolitic hematite resources have not been fully utilized due to the high phosphorus content. Phosphorus is typically considered a harmful element in the steelmaking process, necessitating dephosphorization as a necessary step prior to steel production. Several methods, such as smelting dephosphorization [6–9], bio-leaching [10–12] and chemical leaching [13–15], have been developed to remove phosphorus. However, there have also been reports on the simultaneous recovery of iron and phosphorus to prepare Fe–P alloys for high-phosphorus steel production [16].

Zhou [17] proposed a microwave-fluidized bed pretreatment technology (MFP) to enhance the separation of oolitic hematite. Compared with those obtained under traditional roasting pretreatment methods, the magnetic properties of the roasting products treated via MFP are significantly enhanced, and the iron grade of the concentrate can reach 58.72%. The phosphorus content in the final leaching residue can also be reduced from 0.17% to 0.11%. However, this method is currently limited to laboratory applications and cannot be introduced into industrial production.

Yehia [18] conducted a study on the application of fungal cellulase as an inhibitor in the reverse flotation of high-phosphorus iron ore. The results showed that cellulase has a good inhibitory effect on hematite, while it has no inhibitory effect on phosphorite at pH = 10, allowing for the effective separation of the two.
Nunes [19] studied the influence of anionic, cationic and amphiteric collectors on the flotation effect of wavelet flotation under alkaline conditions. The results show that the amine collector has better selectivity for phosphorus and iron, and an iron concentrate with only 0.201% phosphorus content can be obtained.

Junhui Xiao [20] used the combined process of low-intensity magnetic separation–reverse flotation to treat oolitic hematite after suspension roasting, and found that the flotation reagent combination of starch and toluenesulfonamide had a good effect on iron extraction and phosphorus reduction, and that the phosphorus content in the iron concentrate could be reduced to 0.11%. However, this method is suitable for oolitic magnetite after suspension magnetization roasting rather than being directly used in the flotation process of raw ore.

In some iron and steel smelters have indeed studied and developed methods for dephosphorization of hot metal, such as SARP, SMP and ORP, but these methods usually only have a good effect on low phosphorus (less than 0.15%) hot metal, and the removal effect is limited for iron ore with high phosphorus content, and can not get higher quality products [21–28]. Bacterial leaching dephosphorization, on the other hand, is time-consuming, and chemical leaching dephosphorization may pose environmental contamination risks. Generally speaking, flotation dephosphorization [29,30] is considered the most economical and efficient method. However, while some research has focused on the flotation dephosphorization of oolitic hematite [31–34], a systematic and in-depth analysis is lacking, which is a major shortcoming. Chemical dephosphorization may offer higher efficiency, but physical dephosphorization offers lower costs. Few studies have been conducted on traditional flotation dephosphorization, primarily due to the unique nature of oolitic hematite. Despite previous research efforts, a suitable method to address oolitic iron ore has not yet been identified.

This paper will fundamentally study the feasibility of dephosphorization using the characteristics of the oolitic hematite and place emphasis on the specific flotation reagents and flotation mechanism.

2. Materials and Methods

2.1. Materials

The raw materials were taken from western Hubei. The chemical composition and iron phase composition are shown in Tables 1 and 2.

Table 1. Chemical composition of high-phosphorus oolitic hematite (%).

<table>
<thead>
<tr>
<th></th>
<th>Fe total</th>
<th>FeO</th>
<th>Fe₂O₃</th>
<th>P</th>
<th>S</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>K₂O</th>
<th>Na₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>48.80</td>
<td>1.70</td>
<td>67.83</td>
<td>1.32</td>
<td>0.028</td>
<td>15.22</td>
<td>4.77</td>
<td>2.58</td>
<td>0.54</td>
<td>0.25</td>
<td>0.085</td>
</tr>
</tbody>
</table>

Table 2. Iron phase composition of high-phosphorus oolitic hematite (%).

<table>
<thead>
<tr>
<th>Iron Phase</th>
<th>Fe in Magnetite</th>
<th>Fe in Siderite</th>
<th>Fe in Hematite or Limonite</th>
<th>Fe in Iron Pyrite</th>
<th>Fe in Iron Metasilicate</th>
<th>Fe in Iron Sulfate</th>
<th>Total Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>0.24</td>
<td>0.54</td>
<td>44.45</td>
<td>0.06</td>
<td>3.52</td>
<td>0.11</td>
<td>48.92</td>
</tr>
<tr>
<td>Distribution</td>
<td>0.49</td>
<td>1.10</td>
<td>90.86</td>
<td>0.12</td>
<td>7.20</td>
<td>0.22</td>
<td>100.00</td>
</tr>
</tbody>
</table>

Mineralogy research indicates that oolitic hematite ore belongs to a high-phosphoric-acid orebody. The primary valuable minerals are hematite and limonite, accompanied by small amounts of magnetite and siderite. The main gangue minerals include daphnite, quartz, biotite, calcite and collophane, among others. The complex mineral composition, fine dissemination, and intricate intergrowth relationship between iron minerals and gangue minerals, as well as the unique oolitic texture, contribute to making oolitic hematite one of the most refractory ores globally. Figure 1 displays the oolitic texture and Figure 2 displays the collophosphate in oolitic hematite.
As can be seen from Figure 2, the phosphorus mineral in the ore mainly exists in the form of collophane, which can be divided into three types. The first type, known as block collophane, is easily removable as it occurs in debris and cement and has a particle size ranging from 0.02 mm to 0.10 mm. The second type, called band collophane, is more difficult to remove as it is distributed within the banded oolitic structure. Removing this type of phosphorus mineral often results in some loss of iron recovery. The third type of phosphorus mineral is dispersed within hematite crystals and cannot be removed through physical concentration alone. Electron probe analysis reveals that block collophane contains 30.02% $P_2O_5$ and 45.89% CaO, while band collophane contains 41.18% $P_2O_5$ and 38.90% CaO. Therefore, flotation can effectively remove block collophane and band collophane.

2.2. Methods

Optical microscopes, scanning electron microscopes, electron probes and mineral liberation analyzers were employed to determine mineral composition. Electric potential analysis and infrared spectroscopy were utilized to investigate the mechanism of action of
the reagents. The flotation process is illustrated in Figure 3. Following flotation, iron (Fe) content and phosphorus (P) content were analyzed.

![Figure 3. The flowsheet of flotation.](image_url)

### 2.3. Test Characterization

A zeta potential analyzer (ZS90, Malvern Instruments Ltd., Malvern, UK) was used to measure the electro-kinetic potential changes before and after the action of the agent on the mineral surface to determine whether or not the agent shows characteristic adsorption on the mineral surface, so as to determine the action mechanism of the agent. Infrared spectroscopy (Frontier, Perkinelmer, Waltham, MA, USA) was used to study the structure and chemical bonds of the chemical molecules, so as to determine the adsorption state of the chemical agents on the mineral surface. Micromorphology, particle size composition and mineral composition were determined using Mineral Liberation Analyser (MLA) (MLA250, JKMRC, Indooroopilly, Australia). The composition and distribution of elements in the samples were determined via energy dispersive spectroscopy (EDS) (EDAX, FEI Company, Hillsboro, OR, USA). The microchemical composition of the sample was quantitatively analyzed via electron probe analysis (JXA-733, Nippon Electronics Co., Ltd., Tokyo, Japan).

### 3. Results and Discussion

#### 3.1. Experimental Study of Dephosphorization

After closed-circuit comminution to $-2$ mm, the raw materials were further ground to $-0.074$ mm, accounting for 92.5% of the total. To minimize the impact of slime on flotation, the pulp prepared for flotation underwent a desliming process using a spiral chute at a concentration of 30%. The results are presented in Table 3.

The results of process mineralogical analysis for oolitic hematite reveal that the ore consists of a complex composition of various minerals, with a fine particle size and an intricate micro-symbiotic relationship between iron minerals and gangue minerals. Therefore, fine grinding is necessary to liberate the iron-bearing minerals from the ore. However, oolitic hematite ore is highly susceptible to argillization during the crushing and grinding process. Consequently, significant amounts of slime are produced during the liberation of oolitic hematite, which adheres to coarse particles (Figure 4) and severely hampers the flotation process.
The detrimental effect of very fine slime on the flotation process. EDS analysis demonstrates that the main elements present in the adsorbed fine particles on large particles at point A are Fe, Al, Si, Ca, etc., while Fe is present at point B. This indicates that fine iron particles and gangue particles can adsorb onto coarse particles. In order to mitigate the impact of slime on flotation, two methods are commonly employed: desliming or selective flocculation [35]. The latter method requires a more precise flocculation process but achieves higher recovery rates. However, if the flocculation process is not adequately controlled, the flotation process can rapidly deteriorate. Desliming, on the other hand, is a simpler and more stable approach. The SEM image of the conditions after desliming is shown in Figure 5, where the slime content is significantly reduced and has a minimal effect on the flotation process.

Table 3. The results of pulp desliming (%).

<table>
<thead>
<tr>
<th>Products</th>
<th>Yield</th>
<th>Grade</th>
<th>Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>Desilting</td>
<td>89.71</td>
<td>49.10</td>
<td>1.340</td>
</tr>
<tr>
<td>Slime</td>
<td>10.29</td>
<td>48.92</td>
<td>1.300</td>
</tr>
<tr>
<td>Feeding</td>
<td>100.00</td>
<td>49.08</td>
<td>1.336</td>
</tr>
</tbody>
</table>

Figure 4. SEM–EDS images before desliming. (a) is the SEM image before desliming, (b,c) are the EDS images of point A and point B in (a) respectively.

A significant number of flotation studies have demonstrated that as slime content increases, the flotation system undergoes sharp deterioration, primarily due to fundamental changes in the flotation characteristics resulting from the reduction in ore particle size [36–39]. Due to the size effect, fine particles possess a larger surface area and higher surface energy. As a result, they not only selectively adsorb onto the surface of coarse particles (Figure 4), but also undergo non-selective agglomeration, which adversely affects flotation performance.

In order to further mitigate the adverse impact of slime on subsequent operations, a desliming treatment was implemented for the fine grinding products. Figure 5 illustrates the scanning electron microscope analysis results of the desliming materials. The analysis shows that the desliming products contain fewer fine particles, and the surfaces of the ore particles do not adsorb fine particles. EDS energy spectrum analysis verifies that the main element present is Fe at both point A and point B, with small amounts of Al, Si and Ca. This further confirms the cleanliness of the desliming products’ surfaces. Through de-sliming treatment, the detrimental effects of very fine slime on the flotation process can be eliminated. It is evident that slime removal not only reduces the burden on subsequent flotation operations but also facilitates the smooth progress of the flotation process. Therefore, the desliming operation is of utmost importance.
Figure 5. SEM–EDS images after desliming. (a,b) are the SEM images after desliming, (c,d) are the EDS images of point A and point B in (b) respectively.

3.1.1. The Collector Selection Tests

The collector of phosphate ores is usually fatty acid [40], which has a good selectivity. Conventional fatty acid and lab-made fatty acid are used as collectors of phosphorus mineral. The flotation flowsheet is shown in Figure 3. The dosages of the starch and the collector are 600 g/t and 400 g/t in the roughing, respectively, and the dosages in the scavenging are added at half of the original. The results of flotation tests are shown in Figure 6.

Figure 6. Results of dephosphorization agent tests.
Figure 6 illustrates that under the present flotation conditions, the use of lab-made fatty acid as a collector can reduce the phosphorus content in the iron concentrate to 0.16%, which is significantly lower compared to that of products obtained using conventional fatty acids. Therefore, lab-made fatty acid was chosen as the dephosphorization collector for the subsequent tests.

3.1.2. The Starch Dosage Tests

Starch is commonly utilized as an inhibitor in the reverse flotation of iron minerals [41–43]. Its addition increases the hydrophilicity of iron minerals and reduces the adsorption between iron minerals and collectors. Additionally, starch can also function as a flocculant. The dosage of starch significantly impacts the inhibitory effect on iron minerals, hence the importance of investigating the appropriate amount of starch. Dosage tests of starch were conducted with a roughing collector dosage of 600 g/t and a cleaning collector dosage of 300 g/t. The results are presented in Figure 7.

Figure 7 demonstrates that as starch dosage increases, iron content remains relatively stable, while iron recovery and phosphorus content reach stability after certain degrees of increase. When the starch dosage reaches 400 g/t, the iron content and iron recovery in the concentrate are 56.81% and 82.48%, respectively, with a phosphorus content of 0.16%. These results indicate a favorable flotation index. After comprehensive consideration, a starch dosage of 400 g/t was selected.

3.1.3. The Collector Dosage Tests

Dosage tests of the collector were conducted with a roughing starch dosage of 400 g/t and a cleaning starch dosage of 200 g/t. The results are depicted in Figure 8.

As shown in Figure 8, with an increase in collector dosage from 400 g/t to 750 g/t, the iron grade exhibited a slight increase from 57.01% to 57.71%. However, both the iron recovery and phosphorus content showed significant decreases. Specifically, at a collector dosage of 400 g/t, the iron recovery and phosphorus content were 84.84% and 0.18%, respectively. These values decreased to 80.99% and 0.11%, respectively, at a collector dosage of 750 g/t. Taking all into account, a collector dosage of 600 g/t was selected.

At the starch dosage of 400 g/t, the collector dosage of 600 g/t in the roughing, and half of them in the scavenging, with a reverse flotation flowsheet of one roughing and one scavenging, a good index of iron content of 57.67%, phosphorus content of 0.14% and
flotation recovery of 82.45% was obtained. Taking desliming into account, the ultimate recovery was 74.18%.

Figure 8. Influence of collector dosage on test results.

3.2. Flotation Product Characteristics

3.2.1. Flotation Tailings

The scanning electron microscope (SEM) analysis results of the tailings products are depicted in Figure 9, while the mineral liberation analysis (MLA) results are presented in Figure 10. The results shown in Figure 9 show that the particle size of tailings products is relatively fine, and the fine particles are mainly needle-shaped and flocculent particles. An energy-dispersive X-ray spectroscopy (EDS) energy spectrum analysis of the tailings reveals that the main elements at point A are Ca and P, with small amounts of Fe, Si and Al. This suggests that apatite is the primary component at point A. The MLA analysis results depicted in Figure 10 indicate that the tailings mainly consist of colloidal phosphorus mineral aggregates. Additionally, they contain a certain amount of hematite limonite aggregates with Fe₂O₃ ≥ 50%, layered silicate mineral aggregates with Fe₂O₃ ≤ 30%, quartz aggregates and their conjoined bodies. These findings demonstrate that the use of lab-made collectors effectively removes phosphorus minerals from high-phosphorus oolitic hematite, which aligns with the results of the dephosphorization flotation test.

Figure 11 displays the test results regarding the dissociation degree of the main minerals in the tailings. Table 4 presents the quantitative analysis results of the main minerals in the tailings. The analysis results indicate that the content of collophane aggregate in the tailings reaches 80.44%. The dissociation degrees of collophane aggregate, quartz, hematite limonite aggregate and silicate mineral aggregate are 93%, 56%, 68% and 71%, respectively. Collophane aggregates are primarily associated with quartz, hematite limonite aggregates, silicate mineral aggregates and carbonate minerals. Therefore, this further proves that the lab-made collector can effectively recover collophanite aggregates but may also introduce some quartz, hematite aggregates and silicate aggregates into the tailings.
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**Figure 9.** SEM–EDS analysis of phosphorus tailings. (a,b) are the SEM images of phosphorus tailings, (c) is the energy spectrum analysis of point A in (b).

**Figure 10.** MLA image of phosphorus tailings.

**Table 4.** Quantitative analysis of the main mineral in the phosphorus tailings (%).

<table>
<thead>
<tr>
<th>Minerals</th>
<th>Quartz</th>
<th>Collophanite Aggregate</th>
<th>Hematite Limonite Aggregate</th>
<th>Silicate Mineral Aggregate</th>
<th>Sulfide</th>
<th>Carbonate</th>
<th>Manganese Oxide</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Contents</td>
<td>4.09</td>
<td>80.44</td>
<td>10.39</td>
<td>2.13</td>
<td>0.04</td>
<td>2.88</td>
<td>0.02</td>
<td>100.00</td>
</tr>
</tbody>
</table>
3.2.2. Analysis of Iron Concentrate Products

The chemical composition and iron phase composition of iron concentrate products are shown in Tables 5 and 6. The results show that the main iron-bearing minerals in the iron concentrate are hematite and limonite, accounting for 98.43% of the total iron, while the content of P, Si and other impurity elements is low, and the enrichment and separation effect is good.

Table 5. Chemical composition of iron concentrate products (%).

<table>
<thead>
<tr>
<th></th>
<th>Fetotal</th>
<th>P</th>
<th>S</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>K₂O</th>
<th>Na₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>58.86</td>
<td>0.24</td>
<td>0.03</td>
<td>6.29</td>
<td>4.39</td>
<td>0.95</td>
<td>0.54</td>
<td>0.17</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Table 6. Iron phase composition of iron concentrate products (%).

<table>
<thead>
<tr>
<th>Iron Phase</th>
<th>Fe in Magnetite</th>
<th>Fe in Siderite</th>
<th>Fe in Hematite or Limonite</th>
<th>Fe in Iron Metasilicate</th>
<th>Fe in Iron Sulfate</th>
<th>Total Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Content</td>
<td>0.31</td>
<td>0.45</td>
<td>57.89</td>
<td>0.096</td>
<td>0.067</td>
<td>58.81</td>
</tr>
<tr>
<td>Distribution</td>
<td>0.53</td>
<td>0.77</td>
<td>98.43</td>
<td>0.16</td>
<td>0.11</td>
<td>100.00</td>
</tr>
</tbody>
</table>

The iron concentrate was examined using a scanning electron microscope (SEM), and the analysis results are presented in Figure 11. The SEM analysis results reveal that the particle size of the iron phosphate concentrate is uniform, with a noticeable reduction in micro-fine acicular and flocculent particles. Furthermore, the surface of the product displays a smooth and clean appearance. Point A in the scanning electron microscope atlas was selected for EDS detection. The analysis results indicate that the main elements at point A are Fe, Si, Al and Ca, suggesting that point A consists of hematite aggregates. On the other hand, the main elements at point B are Ca, P, Al and Si, indicating the presence of apatite aggregates. This observation implies that a small portion of apatite with larger particle sizes did not enter the foam layer with the collector but eventually mixed into the floating iron phosphate concentrate at the bottom of the tank.
3.3. Mechanism of Dephosphorization

3.3.1. Influence of Floatation Reagents on Zeta Potential of Mineral Surface

Hematite and apatite monominerals with a purity above 98% were obtained and ground to −2 μm for zeta potential measurement. The zeta potential measurements of apatite and hematite were conducted before and after the addition of the collector, and the results are presented in Figures 12 and 13, respectively. It can be observed that the isoelectric point of apatite changed from 4.1 to 3.4 after the addition of the collector, indicating that the collector exhibits characteristic adsorption on the surface of apatite. Similarly, the isoelectric point of hematite also shifted from 6.8 to 3.8, suggesting the occurrence of characteristic adsorption between hematite and the collector.

![Figure 12. The potential of apatite before and after being soaked in the collector solution.](image1)

![Figure 13. The potential of hematite before and after being soaked in the collector solution.](image2)

3.3.2. Adsorption Characteristics of Flotation Reagents on Mineral Surface

Hematite monomineral and apatite monomineral, with a purity of over 98%, were obtained to test the adsorption characteristics of flotation reagents. Infrared spectrograms of the collector, starch, apatite and hematite were obtained for comparison. Prior to
testing, the monominerals were briefly soaked in the reagent solution. Some samples were subsequently washed, while others were not. All samples were then air-dried and used for infrared spectroscopy measurements. The results are shown in Figures 14 and 15.

Figure 14. Infrared spectra of collector and minerals.

Figure 15. Infrared spectra of collector and starch and minerals.

As in Figures 14 and 15, under the action of the collector, the infrared spectra of apatite changed significantly. The -OH stretching vibration absorption peak of the hydrogen bond at 3421.34 cm\(^{-1}\) in the infrared spectra of the collector appeared in the infrared spectra of apatite, which was the same as what happened in the -CH\(_2\)- stretching vibration absorption peak at 2930.51 cm\(^{-1}\) and 2850.07 cm\(^{-1}\). This shows that the collector can be effectively adsorbed on the surface of apatite. After apatite was soaked in the starch solution and the collector solution, the absorption peak still existed, and at 2930.51 cm\(^{-1}\) and 2850.07 cm\(^{-1}\), the absorption peak moved 6.57 cm\(^{-1}\) and 4.10 cm\(^{-1}\), respectively. Then, apatite was washed in water, and the absorption peak became stronger. This indicates that physical
absorption and chemisorption exists on the surfaces of the apatite, and this is also the reason flotation achieves good results.

Similar to what was found from the previous analysis, the collector can also be adsorbed onto the surface of hematite. However, when starch is introduced, the absorption is significantly weakened. After washing, almost no new peaks are detected on the surface of hematite. This indicates that only very weak physical absorption occurs on the surface of hematite in the presence of starch and the collector. This phenomenon explains why starch can effectively depress hematite and facilitate the separation of apatite and hematite.

4. Conclusions

Mineralogy research indicates that oolitic hematite is one of the most challenging types of iron ore due to its complex mineral composition, fine dissemination, intricate intergrowth relationship between iron minerals and gangue minerals, and unique oolitic texture. Phosphorus minerals in the ore predominantly exist in the form of collophane, which can be classified into three categories. Block collophane and band collophane can be eliminated through flotation, whereas the remaining type cannot be physically separated during concentration.

Flotation research reveals that the laboratory-produced fatty acid serves as an effective collector for high-phosphorus oolitic hematite. A favorable result was achieved, with an iron content of 57.67%, phosphorus content of 0.14% and a flotation recovery of 82.43%.

The study on the flotation mechanism demonstrates that in an experimentally prepared starch and fatty acid system, both physical adsorption and chemical adsorption occur on the surface of apatite, while weak physical adsorption is observed on the surface of hematite. Consequently, selective separation and extraction of apatite and hematite are achieved, thereby presenting a novel approach for the development and utilization of oolitic hematite.

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