Analysis of the Application Potential of Coffee Oil as an Ilmenite Flotation Collector

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Abstract: Coffee grounds are the most significant production waste in the coffee industry and contain about 15% coffee oil. Coffee oil is rich in fatty acids and polyphenols, which have great application potential in the flotation of oxidized minerals. In this study, coffee oil as a green flotation collector for ilmenite was investigated by micro-flotation, zeta potential measurement, and foam stability analysis. The results of zeta potential reveal that both coffee oil and MOH can be adsorbed on the ilmenite surface at pH 6.7, and the chemical adsorption mode is dominant. However, when the pH is 2.8, the adsorption capacity of coffee oil on the ilmenite surface is much larger than that of MOH. The pH value of the pulp has little effect on the foam properties in the coffee oil solution and has a great influence on the foaming performance and foam stability of the MOH solution. When coffee oil is used as a collector, the grade of TiO2 in ilmenite concentrate is increased from 21.68% to 46.83%, and the recovery is 90.22%, indicating that the potential of coffee oil in the application of ilmenite flotation is large.

Keywords: coffee oil; green flotation reagent; microbubble stability; ilmenite flotation

1. Introduction

As one of the most popular beverages in the world, coffee has become the second-largest commodity after oil [1]. Coffee grounds are by-products of instant coffee processing. The weight is about two-thirds of the coffee beans, that is, 2 t of coffee grounds per 1 t of instant coffee. Coffee grounds are rich in fats, sugars, and proteins, of which coffee oil is 14% or more [2,3]. The use of coffee grounds as a raw material for extracting surfactants, such as coffee oil [3,4], biodiesel [5], and polyphenols [3,6], has been reported. These surfactants are mainly used in energy [4,7,8], skin care [9–11], and as food additives [12–14]. However, this natural green surfactant has not been reported in the field of mineral flotation.

Mineral flotation is based on the difference between physical and chemical properties of different mineral surfaces. By adding surfactants, the difference in hydrophobicity of different mineral particles is expanded to achieve the enrichment of the target minerals in foam products [15–17]. Surfactants used in mineral flotation are mainly divided into two categories. One is natural surfactant, which are derived from natural resources such as petroleum, animals, and plants [18–20]. The other is a chemically synthesized surfactant [17,21,22]. Since the launch of China’s green mine protection program, chemically synthesized surfactants have been increasingly restricted due to their adverse effects on health and the environment. Therefore, the extraction of environmentally friendly green natural surfactants from waste is a trend in the field of mineral flotation.

There are abundant ilmenite resources in China, and its flotation mainly relies on fatty acids as collectors [26,27]. The concentration of ilmenite requires a strong acidic pulp environment for its successful application. However, existing foaming agents such as pine oil and methyl isobutylcarbinol
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(MIBC), have poor foam performance under acidic conditions. Coffee oil contains a large amount of fatty acid, including palmitic acid, stearic acid, oleic acid, and linoleic acid, as well as rich polyphenols [3,13]. Due to the strong ability of fatty acids to collect ilmenite and the excellent foaming properties of polyphenols [3], coffee oil has an excellent application potential in the flotation of ilmenite.

In this study, we used multiple analytical methods, such as zeta potential and foam stability tests, to investigate the solution properties of coffee oil and the adsorption mechanism of coffee oil on the ilmenite surface. Then, the application behavior of the most commonly used collectors for ilmenite flotation and coffee oil in the flotation of Panzhihua ilmenite were compared. The results of this study can provide direction for the comprehensive application of coffee grounds, and also provide reference for the development of green flotation collectors.

2. Materials and Methods

2.1. Materials and Reagents

The single minerals and actual ilmenite samples used in this study were from Panzhihua, Sichuan province, China. The single samples were manually selected, crushed by cone crushers, and finally ground and sieved to obtain powdered samples with particle sizes in the range 37 to 74 µm. The actual ilmenite sample was the concentrated product after separation by gravity and magnetic separation in the beneficiation plants. The results of the chemical composition of the single and actual samples are presented in Table 1, and the results of XRD are shown in Figure 1. The results showed that the purity of the single ilmenite used was greater than 91%, and the actual ilmenite contained 21.68% TiO₂. Coffee oil was purchased from Shandong Minghao Chemical Co., Ltd. (Linyi, China), and chemical composition information is shown in Table 2. The results in Table 2 demonstrate that fatty acids in the coffee oil were mainly composed of palmitic acid (saturated fatty acid) and linoleic acid (unsaturated fatty acid), and their total content exceeded 58%. NaOH, H₂SO₄, and KNO₃ were of analytical grade and were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) As a control experiment, the collector MOH was purchased from Jingzhou Flotation Reagent Co., Ltd. (Jingzhou, China) MOH is a commercial collector, widely used in fine ilmenite upgrading, and its detail composition was reported in the literature [28]. Ultra-pure water with a conductivity of 18.2 MΩ·cm was obtained from a USF-ELGA Maxima (USF Elga, High Wycomb, UK) water purification system and used in all experiments.

Table 1. The results of chemical composition analysis of mineral samples (%).

<table>
<thead>
<tr>
<th>Sample</th>
<th>TiO₂</th>
<th>FeO</th>
<th>S</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single</td>
<td>49.97</td>
<td>41.28</td>
<td>-</td>
<td>1.68</td>
<td>0.51</td>
<td>0.13</td>
<td>4.26</td>
<td>2.17</td>
</tr>
<tr>
<td>Actual</td>
<td>21.68</td>
<td>19.45</td>
<td>0.85</td>
<td>25.65</td>
<td>4.68</td>
<td>11.12</td>
<td>5.39</td>
<td>11.18</td>
</tr>
</tbody>
</table>

Figure 1. XRD pattern of single ilmenite samples in this study.
Table 2. Composition and content of fatty acid, content of polyphenolic compound in the coffee oil.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Palmitic acid (C16:0)</td>
<td>21.4</td>
</tr>
<tr>
<td>Stearic acid (C18:0)</td>
<td>4.2</td>
</tr>
<tr>
<td>Oleic acid (C18:1)</td>
<td>5.6</td>
</tr>
<tr>
<td>Linoleic acid (C18:2)</td>
<td>26.2</td>
</tr>
<tr>
<td>Arachidic acid (C20:0)</td>
<td>1.3</td>
</tr>
<tr>
<td>Polyphenolic compound</td>
<td>About 1.1</td>
</tr>
</tbody>
</table>

2.2. Methods

2.2.1. Actual Ilmenite Flotation

The actual ore powder sample was treated with a BMZ-1 rod mill to obtain 75% of 74 µm particle size content in the ground material. The flotation experiments were carried out in a series of XFD type flotation machines, including 0.5 L, 1.5 L, and 3.0 L volume. The experiment used pre-flotation of sulfide minerals, and then the bottom product of the tank underwent a closed-circuit phase with one rougher, three cleanings, and one scavenging process, in which the medium ore was returned in sequence. The specific dosage of the reagents and the flotation process are shown in Figure 2. After the closed-circuit experiment was balanced, the concentrate and tailings were separately filtered, dried, and weighted, and the grade of TiO$_2$ was determined by chemical titration. Finally, the flotation recovery rate was calculated by Equation (1).

$$R = \frac{\beta_1(\beta_0 - \beta_2)}{\beta_0(\beta_1 - \beta_2)} \times 100\%$$

where $R$ is the flotation recovery rate, and $\beta_0$, $\beta_1$, and $\beta_2$ indicate the grades of TiO$_2$ in raw ore, concentrate, and tailings, respectively.

2.2.2. Zeta Potential Measurements

The zeta potentials on the ilmenite surface before and after coffee oil adsorption were determined with a ZetaSizer 3000 Malvern Instrument (Brookhaven, MS, USA). The single ilmenite samples were
ground to less than 5 µm by agate grinding, and then 20 ± 1 mg was added to a beaker containing 50 mL of distilled, deionized water with 10^{-3} mol/L KCl, wherein KCl was used as the electrolyte. The slurry was adjusted for 15 min by electromagnetic stirring at 150 rpm. Then different concentrations of coffee oil were added to the beaker, while stirring for 30 min to reach equilibrium. Finally, the pH of the slurry solution was adjusted using NaOH or HCl to obtain a pH range of 2 to 11. After the stabilization of the pH value, the zeta potential was measured in accordance with the procedures described in the instrument manual [29]. The reported results were the average of at least three full repeats of the experiment.

2.2.3. Foam Property Measurements

The effect of coffee oil on the properties of the solution foam was measured using a chromatography column. The measurement principle is found in our previous research [30], and its test schematic is shown in Figure 3. Solutions of different surfactants were prepared, and the pH of each solution was adjusted to the prescribed value. The prepared solution was added to the chromatographic column. Before the beginning of the experiment, the chromatographic column containing the solution was inflated with N₂, and a pressure value was fixed in all the experiments. The whole process of the foam layer was recorded from the initial maximum to the final elimination. The foaming capacity was based on the maximum volume (V_{max}) of the foam layer, and the foam stability was analyzed through the reciprocal of the average disappearance velocity of the foam layer 1/(V_{max}/τ). The inner diameter of the chromatography column used in this study was 2.15 cm. The parameters of air pump power and supply air rate remained the same, at 3.75 W and 3.5 L/min, respectively.

![Figure 3. The simple device diagram of bubble property tests [30]: 1. Chromatography column; 2. Sand core; 3. Stent; 4. Hose; 5. Air pumps; 6. Camera.](image)

3. Results and Discussion

3.1. Zeta Potential Measurements

It is known that the adsorption of anions on the mineral surface will change the charge distribution on the mineral surface. The adsorption characteristics of the anionic collector on the mineral surface are revealed by studying the distribution of the surface charge of the mineral under different conditions [31–33]. Figure 4 shows the relationship between the zeta potential of the ilmenite surface and the pH value with coffee oil or MOH before and after adsorption on the ilmenite surface. When there is no collector, the isoelectric point of the ilmenite surface is about pH 5.0, which is similar to that reported in the literature [26]. When the coffee oil is adsorbed (Figure 4a), the zeta potential on the ilmenite surface is significantly reduced. With the increase in pH, the zeta potential gradually decreases,
which indicates that coffee oil is mainly electrostatically adsorbed on the ilmenite surface. With the
increase in negative charge on the ilmenite, the electrostatic adsorption of the negatively charged anion
in coffee oil and the ilmenite surface is gradually reduced, and electrostatic repulsion between the same
kind of charge occurs. However, when the pH value is greater than 8, the zeta potential on the ilmenite
is $-16$ mV, and the zeta potential on the ilmenite surface after coffee oil adsorption is also reduced by
nearly 22 mV, which indicates that the characteristic adsorption occurred between the anion in coffee
oil and the ilmenite surface. The bond energy of the characteristic adsorption is generally greater than
the electrostatic repulsion [34]. However, after MOH adsorption, the trend of zeta potential on the
ilmenite surface is significantly different from that after coffee oil adsorption. With the increase in
pH value, the zeta potential on the ilmenite surface increased first and then decreased. When the pH
value was about 6.8, the zeta potential on the ilmenite surface decreased more than 40 mV. At this time,
the ilmenite surface is negatively charged, indicating that the anion in MOH forms a strong chemical
structure with the ilmenite surface, and the characteristic adsorption is more obvious. Compared with
the anion in MOH, when the pH is less than the isoelectric point, the anion in the MOH has a stronger
adsorption ability on the surface of positively charged ilmenite, indicating that the anion in the MOH
is mainly chemisorbed on the ilmenite surface, accompanied by electrostatic adsorption.

![Figure 4. Zeta potentials of the ilmenite surface before and after adsorption with different collectors as
a function of pH: (a) coffee oil, (b) MOH.](image)

Since the flotation of ilmenite is carried out under acidic concentrations, the cleaning process
requires a lower pH. Therefore, we selected the pH values of rougher (pH 2.7) and cleaning (pH 6.8)
to analyze the adsorption behavior for coffee oil and MOH on the ilmenite surface. The adsorption
density of collector ions can be calculated using the Stern-Grahame Equation [35]:

$$
\Gamma = kC \exp \left( \frac{\Delta G_{ads}^0}{RT} \right)
$$

(2)

where $k$ (constant less than 1) is the adsorption coefficient; $C$ is the collector concentration in aqueous
solution; $R$ is the gas constant; $T$ is the solution temperature; and $\Delta G_{ads}^0$ is the standard free energy of
adsorption. $\Delta G_{ads}^0$ is estimated by the following Equation [26]:

$$
\Delta G_{ads}^0 = ZF\Delta \zeta
$$

(3)

where $Z$ is the valence of the adsorption ions (when the pH is 2.7, $Z$ is 2; when the pH is 6.8, $Z$ is 1);
$F$ is the Faraday constant; and $\Delta \zeta$ is the difference value in zeta potential after and before the collector
is adsorbed.

Based on the results in Figure 4, the adsorption density and standard free energy of coffee oil or
MOH are shown in Table 3. $\Delta G_{ads}^0$ were negative values in all conditions, indicating that coffee oil and
MOH could both be spontaneously adsorbed on the ilmenite surface in all experiments. The value of $\Delta G_{ads}^0$ represented the adsorption ability of the collector on the mineral surface. The smaller the value, the stronger the adsorption ability is [36,37]. It is worth noting that in rougher stage, the dosage of the collector was chosen higher under neutral conditions of ilmenite. Nevertheless, the reason beyond this selection should be made clearer because the dosage used in this stage was six times compared to cleaning stages, which made it discussable in terms of selectivity. Therefore, we chose 800 mg/L of collectors for the neutral condition and 200 mg/L for the acidic condition. Table 3 shows that when the pH was 2.7, the value ($-6.48$) of $\Delta G_{ads}^0$ for 200 mg/L of the coffee oil adsorbed on the ilmenite surface was much less than that ($-1.48$) for 200 mg/L of MOH. This reveals that under strong acid conditions, the adsorption capacity of coffee oil on the ilmenite surface is much higher than that of MOH, which is mainly caused by electrostatic adsorption. When the pH was 6.8, the value ($-2.96$) of $\Delta G_{ads}^0$ for 800 mg/L of the coffee oil adsorbed on the ilmenite surface was more than that ($-3.60$) for 200 mg/L of MOH, but the difference between them was very small. This suggests that under neutral conditions, the adsorption capacity of coffee oil and MOH on the ilmenite surface is similar, and MOH is slightly higher than coffee oil.

### Table 3. The results of different parameters at pH 2.8 and 6.7 under various conditions.

<table>
<thead>
<tr>
<th>Condition*</th>
<th>Number</th>
<th>$\zeta$ (mV)</th>
<th>$\Delta \zeta$ (mV)</th>
<th>$\Gamma$ (mol/L)</th>
<th>$\Delta G_{ads}^0$ (kJ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coffee oil pH 2.8</td>
<td>1</td>
<td>19.13</td>
<td>-33.63</td>
<td>kC exp(0.00262)</td>
<td>-6.48</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>-16.50</td>
<td>-52.85</td>
<td>kC exp(0.00412)</td>
<td>-10.20</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>-33.72</td>
<td>-17.22</td>
<td>kC exp(0.00134)</td>
<td>-3.32</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>-11.59</td>
<td>-21.31</td>
<td>kC exp(0.00083)</td>
<td>-2.06</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>-32.90</td>
<td>-30.72</td>
<td>kC exp(0.00119)</td>
<td>-2.96</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>-42.31</td>
<td>-9.41</td>
<td>kC exp(0.00037)</td>
<td>-0.91</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>18.77</td>
<td>-7.66</td>
<td>kC exp(0.00058)</td>
<td>-1.48</td>
</tr>
<tr>
<td>Coffee oil pH 6.7</td>
<td>8</td>
<td>11.11</td>
<td>-14.63</td>
<td>kC exp(0.00114)</td>
<td>-2.82</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>4.14</td>
<td>-6.97</td>
<td>kC exp(0.00054)</td>
<td>-1.34</td>
</tr>
<tr>
<td>MOH pH 2.8</td>
<td>10</td>
<td>-11.17</td>
<td>-34.15</td>
<td>kC exp(0.00133)</td>
<td>-3.30</td>
</tr>
<tr>
<td>MOH pH 6.7</td>
<td>11</td>
<td>-45.32</td>
<td>-37.28</td>
<td>kC exp(0.00145)</td>
<td>-3.60</td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>-48.45</td>
<td>-3.13</td>
<td>kC exp(0.00012)</td>
<td>-0.30</td>
</tr>
</tbody>
</table>

* $\Delta \zeta = \zeta_2 - \zeta_1$, $\Delta \Delta \zeta = \zeta_3 - \zeta_1$, $\Delta \zeta = \zeta_3 - \zeta_2$, $\Delta \xi = \xi_5 - \xi_4$, $\Delta \zeta = \zeta_6 - \zeta_4$, $\Delta \zeta = \zeta_6 - \zeta_5$, $\Delta \zeta = \zeta_8 - \zeta_7$, $\Delta \zeta = \zeta_9 - \zeta_7$, $\Delta \zeta = \zeta_9 - \zeta_8$, $\Delta \xi = \xi_11 - \xi_10$, $\Delta \xi = \xi_12 - \xi_11$, $\Delta \xi = \xi_12 - \xi_10$, $\Delta \xi = \xi_12 - \xi_11$.  

### 3.2. Foam Property Measurements

Mineral flotation is the use of bubbles to adhere to the hydrophobic mineral particles, which rise to the slurry surface under the action of buoyancy, thereby achieving separation between the hydrophobic particles and the hydrophilic particles. Therefore, the nature of the foam plays a pivotal role in the flotation process [38,39]. The foam properties of different concentrations of coffee oil and MOH solution at pH 2.8 and 6.7 are shown in Figure 5. In Figure 5a, it can be observed that for either coffee oil or MOH, the foaming volume at the higher concentration solution was higher than the lower concentration under the same pH condition. When the concentration of the collector was 800 mg/L and the pH was 6.7, the foaming volume of the MOH solution was larger than that of the coffee oil solution, but the difference between the two was small (the former foam height was 17.86 cm, and the latter was 15.93 cm). However, at a pH of 2.8, the coffee oil solution had a much higher foaming volume than the MOH solution (the former foam height was 12.68 cm and the latter was 3.56 cm). Under the same conditions, the quantity of bubbles in the solution directly affects the probability of the mineral particles being trapped by the bubbles [40–42]. In the rough selection stage (pH 6.7), when MOH was used as the collector, the capture efficiency of the ilmenite particles in the slurry was trapped by the bubbles. When it was slightly larger than coffee oil as a collector, most of the ilmenite particles could be trapped by bubbles because both of them had a very large quantity of bubbles. However, in the selection stage (pH 2.7), when coffee oil was used as a collector, the probability of trapping the ilmenite particles in the slurry by the bubbles was much larger than that of MOH.
Figure 5. The volume of foam (a) and foam stability; (b) as a function of the dosage of collectors.

The capture of mineral particles by bubbles does not mean that they will eventually enter the concentrate product [40,43]. Bubbles trapped with mineral particles are easily broken during the ascent. The factors of bursting are related to the stability of the bubbles. Figure 5b shows the results of the stability test of the foam in solution at various concentrations of coffee oil and MOH solution at pH 2.8 and 6.7. It was found that under any conditions, the stability of the foam in the coffee oil solution was stronger than that of the MOH solution, especially under strong acidic conditions. Based on the results in Figure 5a, we found that the bubbles generated in the coffee oil solution during the roughing stage met the requirements of ilmenite flotation, but in the cleaning stage, the bubbles generated in the coffee oil solution were stronger than those generated in the MOH solution in terms of foaming volume and foam stability, and the difference between the two was large.

3.3. Actual Ilmenite Flotation

The effect of collector coffee oil and MOH on the ilmenite index was compared. Diesel was used as the auxiliary collector, and sulfuric acid was used as the pH adjuster and inhibitor. The closed-circuit test procedure of the actual ore is shown in Figure 2 and the test results after the process balance are shown in Table 4. From the perspective of product indicators, we found that coffee oil and MOH each had their own advantages. When coffee oil was used as a collector, the grade of TiO_2 in the concentrated product was slightly lower than that of MOH (46.83% vs. 47.37%), but the recovery was 1.72% higher than that of MOH (90.22% vs. 88.50%). The grade of TiO_2 in the tailing was also relatively low (3.64% vs. 4.19%). From the perspective of the reagent dosage, the dosage of MOH was higher than that of coffee oil (2418 g/t vs. 2146 g/t) under the condition of a similar flotation index, but the dosage of sulfuric acid was relatively small. The main reason is that the bubble performance of coffee oil is superior to that of MOH, especially under strong acid conditions, and the ability of coffee oil to capture ilmenite is stronger than that of MOH. MOH is the most widely used collector for China’s ilmenite flotation. Whether it is for the production of pharmaceuticals or the application of flotation, there is already a mature process. However, coffee oil is extracted from discarded coffee grounds. There are many types of extraction processes, and which extraction process is most beneficial to the flotation process needs to be further investigated to optimize the test. This study only proposed a way to extract coffee oil from waste coffee grounds and convert them into ilmenite flotation collectors, and there are still many places for further optimization of its specific industrial applications.
Table 4. Experiment results of closed-circuit tests on ilmenite using MOH and coffee oil as collectors.

<table>
<thead>
<tr>
<th>Collector</th>
<th>Concentrates</th>
<th>Tailings</th>
<th>Dosage of Collector (g/t)</th>
<th>Dosage of Sulfuric Acid (g/t)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MOH</td>
<td>47.37</td>
<td>88.50</td>
<td>4.19</td>
<td>11.50</td>
</tr>
<tr>
<td>Coffee oil</td>
<td>46.83</td>
<td>90.22</td>
<td>3.64</td>
<td>9.78</td>
</tr>
</tbody>
</table>

Note: $\beta$ and $\varepsilon$ represent the grade and recovery of TiO$_2$, respectively, and the unit is %.

4. Conclusions

In summary, the application potential of coffee oil as an ilmenite flotation collector was studied. The advantages and disadvantages of coffee oil and MOH in the ilmenite flotation process were analyzed from two aspects: zeta potential and foaming performance of the solution. The conclusions are as follows:

1. In neutral pulp solution (pH 6.7), both MOH and coffee oil can produce strong chemical adsorption on the ilmenite surface. Their solutions have excellent foaming properties and the foam is very stable with little difference between the two.

2. In strong acid slurry solution (pH 2.8), the adsorption capacity of coffee oil on the ilmenite surface is much stronger than that of MOH and the adsorption is mainly electrostatic. The foaming performance of the coffee oil solution is also much stronger than that of the MOH solution.

3. In laboratory experiments, it is possible to replace MOH with coffee oil as a collector for ilmenite flotation, and the grade (46.83%) and recovery rate (90.22%) of TiO$_2$ in the concentrate product meet the experimental requirements. In addition, coffee oil has obvious advantages in environmental protection and further optimization.

Author Contributions: W.X. conceived of and designed the experiments; S.W. prepared the samples and performed the experiments; H.Y. and X.M. analyzed the data; W.X., J.L., and L.C. contributed to the writing and revising of the paper.

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Conflicts of Interest: The authors declare no conflict of interest.

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