Research Article

An Experiment Study on Flotation Process of Fine Scaly Graphite from Dandong in Liaoning

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Abstract: The aim of this study is to acquire graphite from the ore of 3.21%, a technological process including an open circuit experiment of six grinding and seven concentrating steps is testified and in the final concentrate the graphite with fineness (325-mesh) raise to 82.63%. The fine scaly graphite ore from low-grade mine in Dandong, Liaoning contains quartz, sericite, muscovite, feldspar and other impurities. As the only valuable element in the mineral, graphite with small grain size is closely symbiotic with gangue minerals. The technological process also includes a close circuit flotation experiment using sodium silicate as dispersing agent and depressant agent of gangues and using kerosene as inhibited agent and the final rate rises to 86.32% and recovery rate to 82.50%. This experimental study will provide support and guidance on process of graphite minerals with similar characters.

Keywords: Concentrating, flotation, grinding, recovery rate, scaly graphite

INTRODUCTION

Based on the degree of crystallinity, graphite minerals are divided into two categories, crystalline (flake) graphite and Aphanitic (earthy) graphite (Dong, 1998). In flake graphite gangue minerals, sericite and muscovite are the most popular contents with flaky or scaly appearance like the target graphite. They are always mudding and attaching to the surface of graphite, to affect the graphite flotation. There are also some other metallic minerals, which are mainly metal oxide ores like limonite. The metallic minerals are always out of recovery value because of low grade.

Hard to be infiltrated on surface by water, graphite has good floatability, so it is easy to be separated from mineral impurities (Yu et al., 2000). Flotation process is a commonly used method for the purification of minerals (Zhang et al., 2012; Wakamatsu and Numata, 1991; Yue, 2007; Yuan, 2013). The usual technical process includes one roughing step, multiple regrinding and concentrating steps.

In this study, the row graphite mineral are floatation processed with six grinding, two roughing, one scavenging and seven washing steps in experiment and the grade raises from 3.21% in ore to 86.32% in concentrate. The final yield is 3.07% and recovery rate is 82.50%.

MATERIALS AND METHODS

In the mineral samples from Dandong, Liaoning, graphite occurrence is in forms of plates, sheets, leaf-shaped, scaly, fibrous or aggregate, with good crystalline and mostly bands-disseminated in gangue. The size of graphite is in a range of 0.01~0.075 mm, but size of the graphite aggregate reaches 1~5 mm. There are also little micro-size fibrous or hair-like graphite smaller than 0.01 mm. Part of the graphite is in high-dense distribution with small gangue particles inside. The particles cannot be separated from the graphite easily and will affect the grade and recovery rate of the concentrated graphite mineral.

Chemical analysis: After crushing, sieving, blending and shrinkage processes, the final size of raw ore sample are less than 2 mm. The x-ray spectrum analysis result is shown in Table 1 and chemistry analysis result is in Table 2.

From the x-ray spectrum analysis result, the main chemistry elements in row ore are rock-forming minerals, including SiO$_2$, Al$_2$O$_3$, K$_2$O and poor metallic elements. The high content of CO$_2$ is the direct result of graphite. The chemical composition analysis result confirms graphite as the only valuable element.

Process mineralogy study:

Mineral composition and content: The only valuable mineral in sample is graphite 4.46%. The accompanying metallic minerals are too rare, including limonite, pyrrhotite and anatase, etc. Gangue minerals include quartz, sericite, feldspar, muscovite, biotite, chlorite and so on, which is listed in Table 3.

Texture and structure: Texture and structure of the ore are carefully considered. The texture mainly refers...
to the self-morphology of the mineral and aggregates
and the structure refers to the special distribution.

The observations show that, the metal minerals are
mainly in banded, disseminated and filling vein
structure. Banded structure, graphite aggregates are in
alternating parallel bands along the same direction.
Disseminated structure, metallic minerals in varying
thickness granular disseminated in gangue minerals.
Filling vein structure, graphite fills the fracture in the
ore as veins.

Mineral’s morphology has a significant influence
on the dissociation of minerals. To the sample ore, there
are euhedral crystal texture of graphite, hypidiomorphic
crystal texture of pyrite, xenomorphic crystal texture of
pyrrhotite, chalcopyrite and the poikilitic texture
including two or more minerals. Euhedral crystal
texture means that graphite is in self-shaped flake
graphite, leaf-shaped, plate with intact crystalline
shape. Hypidiomorphic crystal texture means pyrite has
partial crystalline in ore. Xenomorphic crystal texture,
pyrrhotite and chalcopyrite have no complete
crystalline, but in shape of other materials. Poikilitic
texture refers to the mutual wrapped phenomenon
between metallic minerals or between metal and gangue
minerals.

**Occurrence characteristics of main minerals:**

**Graphite:** Being the target mineral to be recovered,
graphite is mainly in self-shaped plates, sheets, leaf-
shaped, scaly, fibrous and aggregate occurrence, with
good crystalline arranged in parallel extension
direction. Most graphite crystallines are in band-
disseminated distribution in gangue and some graphite
crystals are in sheets and hair-like because of bending
stress. Graphite has small particle size with crystal
width in the range 0.01-0.075 mm, but the aggregate’s
size can reach 1-5 mm and very few fiber or hair-like
graphite crystals are micro-fine particles with width less
than 0.01 mm. Some graphite can be in dense gangue
like distribution with gap filling by tiny crystals gangue
minerals. Most graphite aggregates often are mixed with
fine gangue minerals. Graphite and limonite has a
close disseminated relationship, such as graphite
interspersed with limonite, filling the edge of limonite,
or in a shape of fine flaky crystals distributed in
limonite. The disseminated relationship between
graphite and anatase is relatively simple, including

**Limonite:** Limonite is mainly in shapes of grainy,
irregular, hull and porous distributed in gangue. It has a
close disseminated relationship with graphite, including
interspersed with graphite, edge filling and small flake
graphite crystals inside.

**Quartz:** Quartz is the most content in the ore and its
occurrence is aggregate of euhedral crystal,
hypidiomorphic crystal in coarse and medium grained
size. Between grains, there are filling and cemented
with bladed muscovite and scaly sericite and
disseminated with small amount of biotite, feldspar and
chlorite.

**Sericite:** Sericite is in small flake or scaly aggregate
occurrence, banded filling, coarse grained size. Some
sericite are mixed with muscovite in a room to fill
between quartz grains.

**Muscovite:** Muscovite is mostly filling and cementing
quartz grains in the shape of flake or leaf. Some
muscovite forms mixed aggregates with sericite, in a
banded distribution in the ore.

**Biotite:** Biotite occurrence is euhedral crystal texture in
shape of plate, disseminated in quartz aggregates. Some
weathered biotite turns to chlorite.

**Feldspar:** Feldspar occurrence is euhedral and
Hypidiomorphic crystal texture in shape of plate and
granular, disseminated among the quartz grains. Partial
weathered feldspar turns to sericite in feldspar.

**Chlorite:** Chlorite occurrence is similar with biotite,
because it is the weathering result of biotite. Chlorine is
mainly disseminated among quartz and feldspar in a
shape of plate with grains coarse size.

**Disseminated size of main minerals:** As the main
mineral to be recovered in ore, graphite is measured and
statistical result of graphite size is shown in Table 4.

The graphite is mostly disseminated at the medium
to fine size of 0.037-0.075 mm, accounting for 57.56%.
Fig. 1: Experiment process including roughing and grinding

26.79% of graphite is bigger than 0.075 mm and 15.65% are smaller than 0.037 mm. The micro-fine graphite, especially those smaller than 0.01 mm, is hardly to be dissociated from gangue ore.

**EXPERIMENTAL RESULTS AND DISCUSSION**

The floatation processing experiment focuses on the factors which affect graphite sorting process, including grinding fineness, grinding number, reagent system, separation process flow, etc.

**Roughing grinding fineness experiment:** Grinding is the process of crushing and dissociating ore into monomer. To graphite ore, it is not only to get a higher separation rate by crushing graphite ore, but also as much as possible to improve rate of flake graphite and to avoid over crushing (Zhang et al., 2011; Fang and Xiao, 1996). Therefore, multi-stage grinding and multi-stage separation process is widely used on graphite ore (Ding et al., 2011). Roughing grinding fineness will ensure high graphite recovery rate. The technical processes and experiment conditions are shown in Fig. 1 and the results shown in Fig. 2.

In Fig. 2, to the concentrate ore after 1st Roughing-Concentrating Process (RCP), grade and recovery rate both increase with the raise of grinding fineness and graphite and gangue minerals are gradually dissociated. But for graphite grained is too fine without monomer dissociation, the concentrate grade is relatively low. After the grinding fineness (200-mesh) reached 80%, concentrate recovery rate will not raise but turn to drop with the increase of grinding fineness and at the same time the tailing grade is basically stable. Therefore, the target roughing grinding fineness (200-mesh) is 80%.

**Water glass dosage experiment:** Water glass is widely used in graphite ore processing as common inhibitor for gangue minerals such as silicate (Li, 2013). This experiment testified the effects of water glass. The experiment processes are shown in Fig. 1 and result is shown in Fig. 3.

In Fig. 3, the increase dosage of water glass has positive effect on improve of concentrate grade, but negative effect on concentrate recovery rate. During roughing when dosage is less than 1500 g/t, the concentrate’s grade is lower. But if the water glass dosage is greater than or equal to 2500 g/t, the concentrate recovery rate is significantly lower. Therefore, proper dosage should be in the range of 1500-2000 g/t. During concentrating, increasing of water glass dosage will improve concentrate grade. Finally the dosages of water glass are set as, 2000 g/t during roughing and 1000 g/t during concentrating.
Collector dosage experiment: Graphite is a natural non-polar mineral with good natural floatability. Generally non-polar oils are used as graphite flotation collectors (Jin et al., 1995) and in this experiment kerosene is select. Test procedures and conditions are shown in Fig. 1 (water glass dosage, roughing as 2000 g/t, concentrating as 1000 g/t), the results are shown in Fig. 4.

According to Fig. 4, with the increase of collector kerosene, tailing grade drops and concentrates recovery rate increases while concrete grade decreases. The result can be explained as that the increasing of kerosene will decrease the selectivity, so the gangue minerals, especially mica, come into concentrates and bring extra difficulties for concentration. To middling, the increase of production rate and drop of mineral grade also indicate that large amounts of gangue minerals come into the rougher concentrate. Therefore, kerosene dosage is set as, rougher 200 g/t for 1st rougher process and 100 g/t for 2nd rougher process.

Fineness experiment of 1st regrinding process: After 1st RCP, the grade of concentrate is about 36–38%. Two reasons are considered, including the fine grain size and the fine gangue minerals between graphite grains or aggregates. To improve the concentrate grade and to protect the graphite flake, multiple Rerinding Processes (RP) are taken after 1st RCP.

In Fig. 5 the experiment result of 1st RP shows that, concentrate grade increase with fineness degree improvement. When fineness (325-mesh) rises from 49.74 to 67.43%, recovery rate does not change much. But on the way fineness rising to 78.9%, the concentrate degree keeps increase, but the recovery rate shows an obviously decrease. So target fineness (325-mesh) of 1st RP is set as 67.43%.

Fineness experiment of 2nd regrinding process: After 1st RCP and 1st RP, grade of the concentrate ore is still not enough and the dissociation between graphite and gangue minerals is still insufficient. So the 2nd regrinding process is performed on current concentrate ore and fineness result is shown in Fig. 6.

The result shows that, the concentrate grade rise distinctively with the increase of fineness, which indicates the disassociation of gangue minerals. When fineness (325-mesh) is 71.46%, concentrate grade reaches 68.37% and continues increasing obviously. At the same time, concentrate recovery rate shows significant reduction. Taken together, the target fineness (325-mesh) of 2nd RP is set as 71.46%.

Open circuit experiment: To fully disassociate the fine graphite, multiple regrinding and concentrating
Table 5: Open circuit experiment conditions

<table>
<thead>
<tr>
<th>Operation index</th>
<th>1&lt;sup&gt;st&lt;/sup&gt;</th>
<th>2&lt;sup&gt;nd&lt;/sup&gt;</th>
<th>3&lt;sup&gt;rd&lt;/sup&gt;</th>
<th>4&lt;sup&gt;th&lt;/sup&gt;</th>
<th>5&lt;sup&gt;th&lt;/sup&gt;</th>
<th>6&lt;sup&gt;th&lt;/sup&gt;</th>
<th>7&lt;sup&gt;th&lt;/sup&gt;</th>
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</thead>
<tbody>
<tr>
<td>Regrinding target fineness (%, 325-mesh)</td>
<td>67.43</td>
<td>71.46</td>
<td>75.40</td>
<td>78.82</td>
<td>82.63</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentrating dosage of water glass (g/t)</td>
<td>1000</td>
<td>500</td>
<td>500</td>
<td>200</td>
<td>200</td>
<td>200</td>
<td>200</td>
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</table>

Table 6: Open circuit experiment result

<table>
<thead>
<tr>
<th>Type</th>
<th>Yield (%)</th>
<th>Grade (C, %)</th>
<th>Recovery rate (C, %)</th>
</tr>
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<tbody>
<tr>
<td>Clean ore</td>
<td>2.23</td>
<td>86.32</td>
<td>82.50</td>
</tr>
<tr>
<td>Tailing</td>
<td>96.93</td>
<td>0.58</td>
<td>17.50</td>
</tr>
<tr>
<td>Raw ore</td>
<td>100.00</td>
<td>3.21</td>
<td>100.00</td>
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</table>

Table 7: Close circuit experiment result

<table>
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<tr>
<th>Type</th>
<th>Yield (%)</th>
<th>Grade (C, %)</th>
<th>Recovery rate (C, %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentrate</td>
<td>3.07</td>
<td>86.32</td>
<td>82.50</td>
</tr>
<tr>
<td>Tailing</td>
<td>96.93</td>
<td>0.58</td>
<td>17.50</td>
</tr>
<tr>
<td>Raw ore</td>
<td>100.00</td>
<td>3.21</td>
<td>100.00</td>
</tr>
</tbody>
</table>

processes are required. An open circuit experiment including five times of regrinding processes and 7 times of concentrating processes is taken. The experiment conditions are shown in Table 5 and result are in Table 6.

**Close circuit experiment:** Based on the experiment result of 5-regrending and 7-concentrating open circuit experiment, a close circuit floatation experiment is taken to examine the impact of middlings’ return and circulation.

In the close circuit experiment, some kinds of middlings will be combined and returned to operation with similar grade. And considering the multiple intergrowths inside, middling composition will be sent to grinding operation. In operation, low grade middlings from 1<sup>st</sup> to 5<sup>th</sup> were mixed and returned to roughing grinding operation and middlings from 6 to 8<sup>th</sup> were mixed and returned to 1<sup>st</sup> regrinding operation. Result of close circuit experiment is in Table 7.

**CONCLUSION**

By means of rock-mineral identification and chemical analysis, sample raw ore is determined as low grade fine scaly graphite with complex compositions, including rich gangue minerals like quartz, sericite, muscovite and feldspar, main minerals like limonite which is not worth for recovery.

As the only valuable mineral, graphite is in occurrences of plates, sheets, leaf-shaped, scaly, fibrous or aggregate. The fine size of graphite is in high-dense distribution with inside of small gangue particles which are hard to be separated and will affect the grade and recovery rate of final concentrate ore. The high content Sericite and Muscovite in ore have distinctive negative effect on the improvement of graphite grade for their close occurrence.

Since graphite’s fineness and close symbiosis with gangue minerals, multiple grinding processes are required along with high fineness standard are required to disassociate graphite with gangue minerals. After a technological process including six grinding and seven grading, the final graphite concentrate is of grade as 86.32% and floatation recovery rate as 82.50%.

**REFERENCES**


