Advance Journal of Food Science and Technology 6(8): 989-993, 2014
DOI:10.19026/ajfst.6.145
ISSN: 2042-4868; e-ISSN: 2042-4876

Submitted: April 09, 2014 Accepted: May 09, 2014 Published: August 10, 2014

Research Article

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

Dai Shujuan and Zhang Meng
University of Science and Technology LiaoNing, LiaoNing, Anshan, 114051, China

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_2O_3, K_2O 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

Corresponding Author: Dai Shujuan, University of Science and Technology LiaoNing, LiaoNing, Anshan, 114051, China

Table 1: X-ray spectrum analysis result of row ore sample

Element	SiO_2	CO_2	Al_2O_3	K_2O	Fe_2O_3	MgO	TiO_2
Content (%)	49.230	24.020	14.4200	5.8800	3.7600	1.2700	0.460
Element	Na_2O	CaO	SO_3	P_2O_5	BaO	MnO	Rb_2O
Content (%)	0.440	0.120	0.1100	0.0940	0.0660	0.0360	0.021
Element	Cr_2O_3	ZrO_2	SrO	CuO	ZnO	As_2O_3	Cl
Content (%)	0.021	0.014	0.0062	0.0049	0.0045	0.0054	0.011

Table 2: Chemical composition analysis result of row ore sample

Element	C	SiO_2	Al_2O_3	K_2O	Na_2O	MgO	CaO	TFe	TiO_2	S
Content (%)	3.40	61.65	17.74	7.33	0.54	2.54	0.35	3.19	0.56	0.052

Table 3: Mineral composition and content in row ore

Minerals	Graphite	Limonite	Quartz	Sericite	Feldspar	Muscovite	Biotite	Chlorite
Content (%)	4.46	0.51	55.46	23.43	7.51	4.69	1.87	0.93

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 graphite filling between anatase and gangue mineral grains.

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%.

Table 4: Statistical result of graphite size distribution

Size (mm)	>0.15	0.15~0.10	0.10~0.075	0.075~0.053	0.053~0.037	0.037~0.01	<0.01
Distribution (%)	11.43	7.11	8.25	28.44	29.12	13.27	2.38
Total (%)	11.43	18.54	26.79	55.23	84.35	97.62	100.00

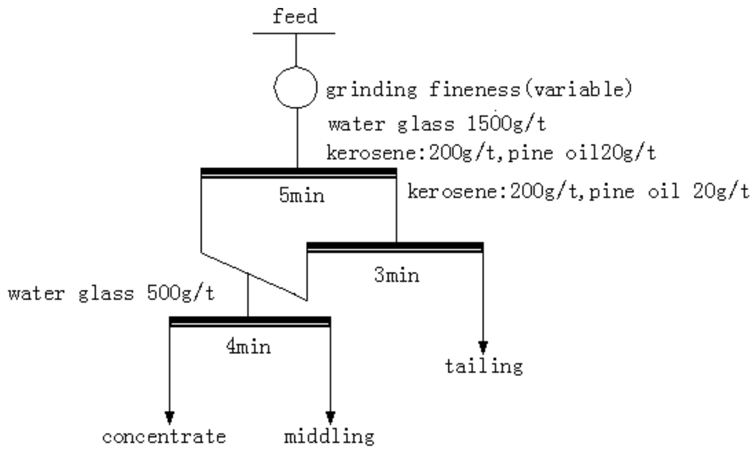

Fig. 1: Experiment process including roughing and grinding

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%.

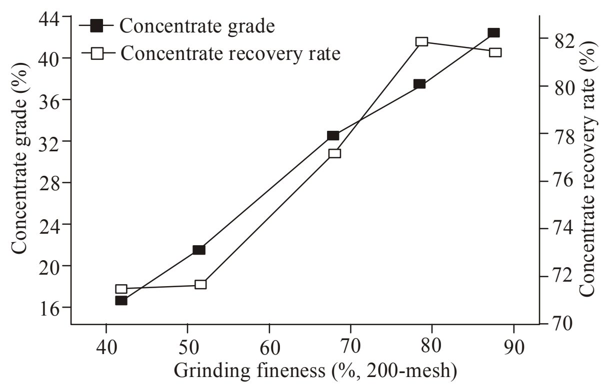

Fig. 2: Grinding fineness after 1st roughing-concentrating process

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.

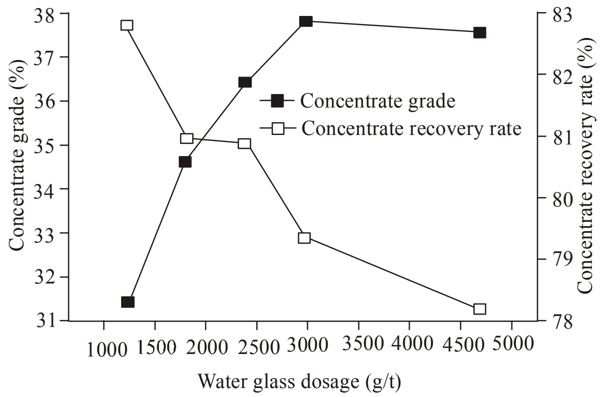

Fig. 3: Experiment results on water glass dosage

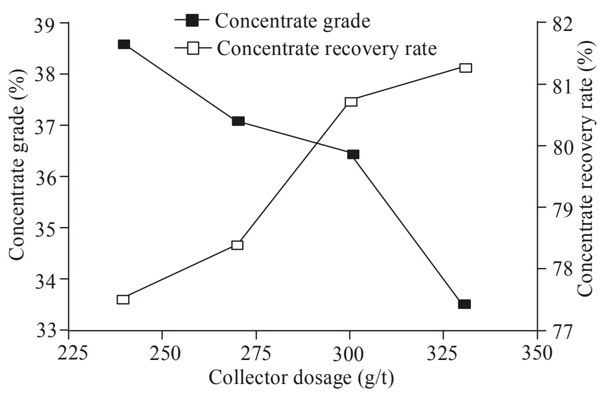

Fig. 4: Experiment results on collector dosage

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 Regrinding Processes (RP) are taken after 1st RCP.

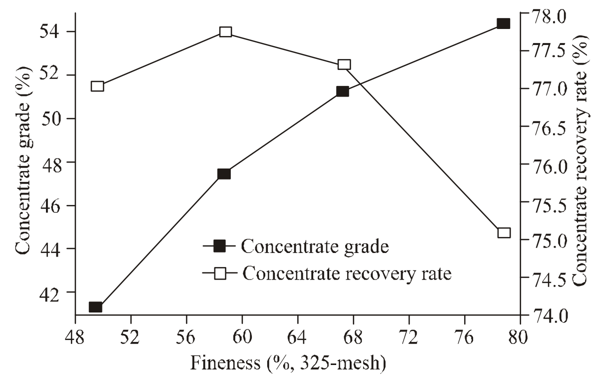

Fig. 5: Fineness of concentrate after 1st regrinding process

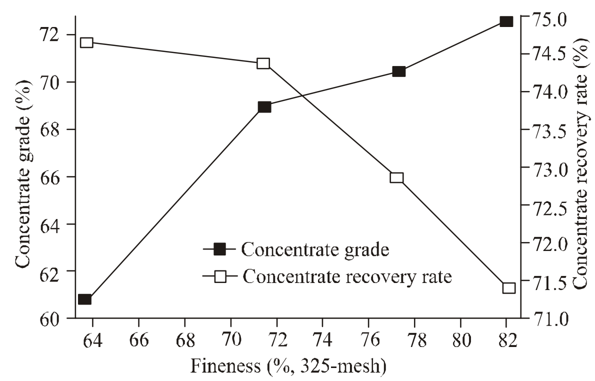

Fig. 6: Fineness of concentrate result after 2nd regrinding process

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

Operation index	1st	2nd	3rd	4th	5th	6th	7th
Regrinding target fineness (%, 325-mesh)	67.43	71.46	75.40	78.82	82.63		
Concentrating dosage of water glass (g/t)	1000	500	500	200	200	200	200

Table 6: Open circuit experiment result

Type	Concentrate	7th middling	6th middling	5th middling	4th middling	3rd middling	2nd middling	1st middling	Cleaner	Tailing
Yield	2.23	0.22	0.30	0.43	0.62	1.37	2.28	5.11	2.31	85.13
Grade	87.62	32.38	18.11	11.41	7.32	5.56	4.86	5.73	5.64	0.39
Recovery rate	62.71	2.290	1.74	1.57	1.46	2.44	3.56	9.40	4.18	10.65

Table 7: Close circuit experiment result

Type	Yield (%)	Grade (C, %)	Recovery rate (C, %)
Concentrate	3.07	86.32	82.50
Tailing	96.93	0.58	17.50
Raw ore	100.00	3.21	100.00

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^{st} to 5^{th} were mixed and returned to roughing grinding operation and middlings from 6 to 8^{th} were mixed and returned to 1^{st} 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

Ding, X.B., W. Yu and K.H. Zhou, 2011. Study on contrastive experiment for flotation of aplanatic graphite. Min. Proces. Equip., 39(11): 82-85.

Dong, Y.Q., 1998. Review on the world graphite market. Build. Mater. Ind. Inform., 5(1): 26-27.

Fang, H.P. and Y.J. Xiao, 1996. Experimental study on purification of large-scales carbon graphite. China Non-Metallic Min. Ind. Herald, 1: 31-33.

Jin, C., Y. Xie, Y.H. Qin *et al*., 1995. A study on the mineral separation using compound graphite floating agent. J. Harbin Inst. Technol., 27(6): 109-111.

Li, H.M., 2013. A study on inhibitor for graphite floatation. Public Commun. Sci. Technol., 16: 95-96.

Wakamatsu, T. and Y. Numata, 1991. Flotation of graphite. Miner. Eng., 4(7): 975-982.

Yu, X.B., H.P. Fang, Y.J. Xiao and L.Y. Zhang, 2000. Experimental study on purification of a fine-scaled graphite ore by flotation. Conserv. Util. Miner. Resour., 1: 14-16.

Yuan, L.M., 2013. Experimental research on purification process of Flake graphite from Liaoning by floatation. Modern Mining, 6: 94-96.

Yue, C.L., 2007. Research on speed flotation of flake graphite. Non-Metallic Mines, 30(5): 40-42.

Zhang, L.Y., D.P. Zhang, Y.L. Qiu *et al*., 2012. Experimental research on beneficiation of a refractory fine graphite ore in Korea. Mult. Util. Miner. Resour., 2: 28-32.

Zhang, L.Y., W. Huang, Y.S. Qiu, L. Pan and W.M. Tu, 2011. Experimental study on purification of a low-carbon graphite ore by flotation. J. Wuhan Univ., Technol., 11: 113-117.