



Shahrood University of
Technology



Iranian Society of
Mining Engineering
(IRSM)

Preparation of High Purity Graphite using Comminution, Pressure Leaching and Flotation

Satya Sai Srikant^{1*}, and Raghupatruni Bhima Rao²

1. Department of ECE, SRM Institute of Science and Technology, Delhi-NCR campus, Modinagar, Ghaziabad, India

2. CSIR-Institute of Minerals and Materials Technology, Bhubaneswar, India

Article Info

Received 13 July 2023

Received in Revised form 18
August 2023

Accepted 30 August 2023

Published online 30 August 2023

DOI: [10.22044/jme.2023.13371.2461](https://doi.org/10.22044/jme.2023.13371.2461)

Keywords

Graphite

Rougher concentrate

Alkali digestion

Flash flotation

Pressure leaching

Abstract

The present paper deals with the development of process flowsheet for recovery of high-grade graphite from rougher graphite concentrate for use in industrial applications. In the present investigation, since the coarser graphite flakes have a higher demand, an attempt is made at every stage of comminution, and flash flotation experiments have been carried out, and the end product obtained by stage comminution followed by flotation has been further subjected to alkali pressure leaching followed by grinding and flotation to recover industrial grade graphite concentrate. During this stage grinding, an additive, a depressant that is being used for depression of silica during flotation, has been used as grinding aid. The data indicates that at any given time of grind, the effect of grinding aid is significantly better as far as the separation efficiency of graphite. Hence, it is concluded that excessive size reduction of graphite is minimised while using a grinding aid. Based on these observations, further studies have been planned for flash flotation using a comminution circuit with grinding aids. The results of the present study reveals that number of cleanings of rougher concentrate alone will not fetch more than 75% of fixed carbon (FC) with less than 8% yield and 41% recovery. The effect of four stage cleanings continued with starvation dosage in grinding circuit followed by flash flotation indicate that the end product achieved contains 97.8% FC with 11.6% yield and 78% recovery and the overall values loss in tailings contain 3.6% FC. The end product achieved by alkali digestion method from a flotation product [97.8% FC] contains 99.2% FC with overall 10.4% yield and 71% recovery. The process adopted in the present investigation is friendly environment and process flowsheet is an innovative. The end product obtained from this process is useful for various industrial applications.

1. Introduction

In India, there are about thirty-seven beneficiation plants located in the states of Andhra Pradesh, Bihar, Gujarat, Odisha, Rajasthan, and Tamil Nadu. Only two beneficiation plants at Rajasthan and Tamil Nadu are state-own; all others are being operated by private agencies [1]. Few plant owners claim to have produced product containing as high as 95% of fixed carbon (FC), while 92% FC is reported to have been obtained by many producers after repeated cycles of beneficiation. Unless like other ores, graphite needs several stage flotation

cleaning to obtain desired grade of graphite [2-6]. This concept is universally accepted. Detailed beneficiation studies were carried out by different agencies to develop a flowsheet of commercial beneficiation plant. The generalized flow sheet on beneficiation of lean and off grade graphite is shown in Figure 1.

The practice, in general, is to reduce the crude graphite ore to the desired size by crushing and stage grinding in rod mills in closed circuits with screening, followed by flotation of a rougher concentrate [7]. The rougher concentrate is milled

*Corresponding author: satya.srikant@gmail.com (S.S. Srikant)

as required and cleaned several times for recovering marketable grade graphite. However, there are many problems that exist during graphite beneficiation, which are enumerated below. Graphite ore associated with schistose rocks are more amenable for flotation than associated with gneisses and metamorphous lime stones [1].

- Schistose rock-bearing graphite deposits possess long, elongated, flatten graphite flakes. These flakes are more hydrophobic. The gneiss rock deposits contain fine crystalline graphite flakes or sometimes amorphous. These re-crystallised flakes need addition of reagents for flotation. Graphite of metamorphosed lime stone deposits origin possesses invariably re-deposited calcite solutions in the graphite flakes and recrystallizes. Such flakes need thermal shock treatment prior to flotation.
- Relatively pure grains of quartz, calcite, and other associated gangue minerals present in the graphite ore inadvertently become smeared with soft fine graphite during grinding and tend to float with graphite necessitating multistage cleaning.

- Despite depression of silicate minerals by sodium silicate and maintaining the pulp pH by sodium carbonate are established practice, the depression of carbonate minerals is yet a problem in graphite ore flotation.
- The depression of carbonate minerals depends on the modes of formation of graphite. The common carbonate minerals are calcite, dolomite, and marble. Calcite (CaCO_3) is a pure mineral, dolomite an analogous composition (CaMgCO_3), and marble (rock) is recrystallized mineral of calcite. The physical and chemical characteristics of these minerals affect its adsorption, electro-kinetic properties and its hydrophilic character.
- The depression of mica minerals also depend on the modes of formation of graphite. The common mica minerals are muscovite and biotite group of minerals. These minerals co-float along with graphite due to sheet structure. The common reagent to depress the phlogophyte mica is lactic acid.
- Gaseous pollution is inevitable since hydrofluoric acid treatment is a common method adopted by many industrialists for obtaining high purity graphite from a concentrate.

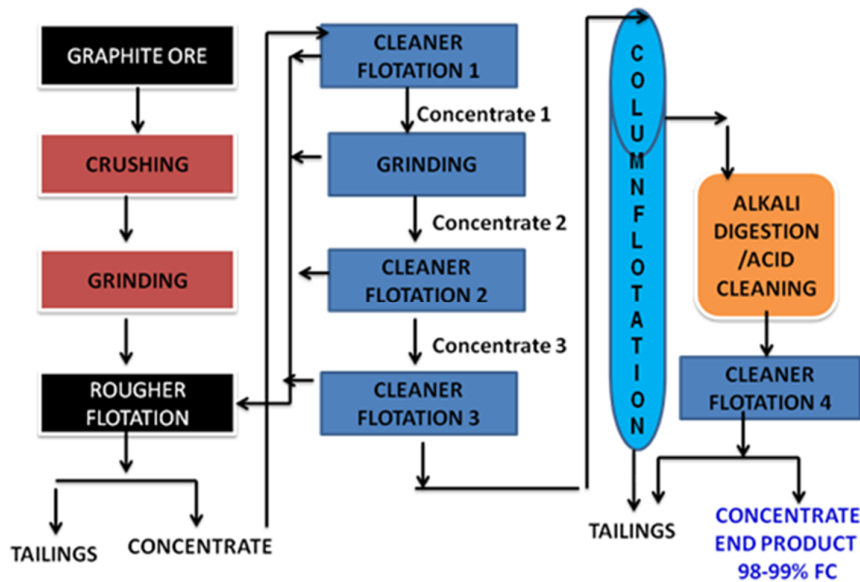
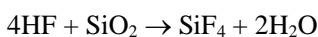
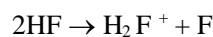


Figure 1. Schematic flowsheet to achieve 98% fixed carbon from lean and off grade graphite ore

Most of the plant manufacturers follow chemical beneficiation using hydrofluoric acid for removal of silica content in order to achieve high pure graphite (with 98% fixed carbon). The chemical reactions involved in this process are as follows:



The self-ionisation equilibria process in acid-liquid phase is given below:



During chemical beneficiation, gaseous pollution is inevitable. This may cause

considerable damage to skin and respiratory track of human body. The maximum permissible occupational limit for fluorine is 3 ppm. In view of this, use of fluorine for chemical beneficiation is restricted. Hence, it is necessary to look for an alternate method for beneficiation. The main objective of this work is to develop economical and environment friendly route for the production of high purity graphite. It is very easy to obtain a crude or rougher concentrate but very difficult to refine it up to a desired extent. Unlike other ores, the graphite flakes which reports to coarse fractions are easily liberated whereas the graphite, which report to the fine fractions are not easily liberated. Because of high premium for coarse flaky graphite, the problem in milling is of grinding to liberate graphite from the gangue minerals without excessive size reduction. The preventing of damage of graphite flake is rather difficult as during grinding; the calcite flakes are destroyed by associated abrasive quartz minerals and other sharp edged gangue minerals. Since physical beneficiation has its own limitations, the present work has been aimed to upgrade the rougher graphite to a high purity level by a novel pressure leaching route. A few researchers attempted chemical route to upgrade the graphite by scarifying the flake size [8-15]. The literature is restricted on stage grinding [16, 17], ultra-sonic treatment [18, 19], scrubbing [20], flash flotation followed by alkali treatment [8, 14, 15] and comminution followed by further flotation to achieve industrial grade graphite at possible higher flake size. Hence, an attempt is made in the present investigation to stage grinding with additives followed by flash flotation to upgrade the graphite ore to a desired grade and then subjected to alkali digestion for obtaining high grade and high pure graphite.

2. Materials and Methods

A rougher feed size d_{80} having 600 microns and the optimum variables such as pH 9.5, sodium silicate one kg/t, sodium silicate for depression of silicate minerals, and light diesel oil 0.4 kg/t as the best collector dosage and MIBC 0.15 kg/t as the best frother dosage for flotation, a large

quantity of rougher graphite concentrate sample was prepared for the present study to prepare high purity graphite. At first instance, the rougher graphite concentrate was cleaned five times for obtaining high-grade graphite. In all the experiments, only at first cleaner cell the pH 9.5 was adjusted, sodium silicate one kg/t for depression of silicate minerals and light diesel oil 0.4 kg/t as the collector dosage and MIBC 0.15 kg/t as frother were used. In the subsequent cells, no reagent was used for cleanings. Since the desired grade of graphite (FC%) content had not been obtained, further studies planned with flash flotation studies using starvation dosages for flotation of clean graphite.

In the subsequent experiments, the rougher graphite concentrate was cleaned without grinding and with grinding aid. Rougher graphite concentrate was ground for a minute at 75% pulp density using sodium silicate as grinding aid as dispersant for particles in the mill and at the same time diesel oil, also used in the mill for surface coating of the graphite particles in the mill. Since the results obtained with grinding aids are relatively better than no additive used for obtaining a cleaner product, further experiments were carried out with grinding aids in stage comminution circuit followed by flotation for obtaining clean graphite.

In the present investigations, grinding aids were used as starvation dosages. It may be noted here that, in the previous experiment, the depressant/dispersant sodium silicate was used one kg/t. Starvation dosage is nothing but a dosage to be equally divided to n number of times, and each dosage will be added at regular intervals, like a human being takes his food three or four times in a day is expressed here as starvation dosage. Regular dosage means required dosage to be added at one time only. During starvation dosages, this dosage was equally distributed for four stage cleanings of rougher graphite concentrate. Thus rougher graphite concentrate was ground at four stages with equal distribution of grinding aids followed by cleaning, as shown in the experimental set up Figure 2.

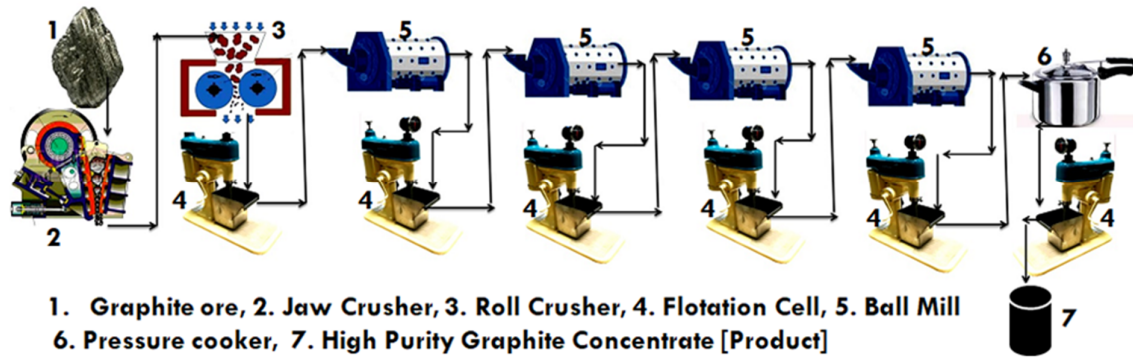


Figure 2. Experimental set up for preparation of high purity graphite

A question may rise why are four grinding steps necessary? In order to avoid excessive size reduction graphite flakes, short time grinding and flash flotation is recommended for recovery of graphite. The graphite concentrate is again further ground for a short time and subjected to flash flotation. This process is continuous till the constant grade achieved. In the present investigation, a four stage grinding was found to recover industrial grade graphite. The fourth cleaned graphite concentrate was initially cleaned with dilute HCl for dissolving the calcite and goethite particles if any present, and then this graphite concentrate subjected to pressure leaching studies using domestic pressure cooker with sodium hydroxide for leach out silicate minerals present within in the graphite flakes. The process in use of sodium hydroxide in pressure cooker with graphite sample is known as alkali pressure leaching. In these experiments, sodium hydroxide (0.1 kg/t) in one hundred ml of water was used for half kg graphite concentrate sample for pressure leaching in the pressure cooker. The sample was kept on hot plat till pressure cooker built pressure. Once pressure was built, the cooker was removed from the hot plate. After cooling the sample was drawn from the cooker and subjected

to one minute grinding followed by flotation. The chemicals used in the present investigation were commercial reagents. Denver flotation cell was used, and standard ball mill 300 X 300 mm size was used in all batch experiments. Grinding was carried out at 75% pulp density and just for one minute of grinding (stage grinding), so that excessive degradation of particles could not occur. It is essential to explain the stage grinding here. Stage grinding does not lead excessive degradation of graphite flaky particles. Continuous grinding may lead to over grinding and excessive soft production. As a result, increase the dosage of chemicals used in flotation and production unwanted finer size. Hence it is recommended for stage grinding.

Fixed carbon analysis was carried out in a standard method using conventional muffle furnace. Typical graphite concentrate samples were collected for petrological and scanning electron microscopic studies using standard laboratory model microscopes. The products and tailings were assessed by yield%, grade (FC)%, and recovery% as well as separation efficiency of unit operation. The assessment formulas are given below:

$$\text{Yield\%} = (\text{feed FC\%} - \text{tailing FC\%}) / (\text{concentrate FC\%} - \text{tailing FC\%}) \times 100$$

$$\text{Recovery\%} = \text{Concentrate Weight \%} \times \text{Concentrate FC\%} / \text{Feed FC\%}$$

$$\text{Enrichment\%} = 100 (\text{concentrate FC\%} - \text{feed FC\%}) / \text{maximum FC\%} - \text{Feed FC\%}$$

$$\text{Separation efficiency\%} = \text{Enrichment\%} \times \text{recovery\%}$$

3. Results and Discussion

3.1. Mineralogical studies

The study of mineralogy is more important to understand the presence of valuable and gangue minerals present, especially micro-crystalline graphite [21]. The association and size of the gangue and valuable minerals are also important to know before take up the project. The mineralogical characteristics of graphite sample can be seen in Figure 3. The study reveals that the graphite rougher concentrate exhibits most of the graphite flakes are coarse to fine needles and

present as independent as well as within or associated with the gangue minerals such as quartzite, goethite, calcite, etc. This observation is very much important for recovery of micro-crystalline graphite from quartz [22]. The morphology of the typical graphite sample indicates that graphite has flakes and the gangue minerals present on the surface of the flakes as well as within the flakes. Hence, this observation indicates that the graphite flake has to be crushed or ground carefully to release the gangue minerals present within the flakes.

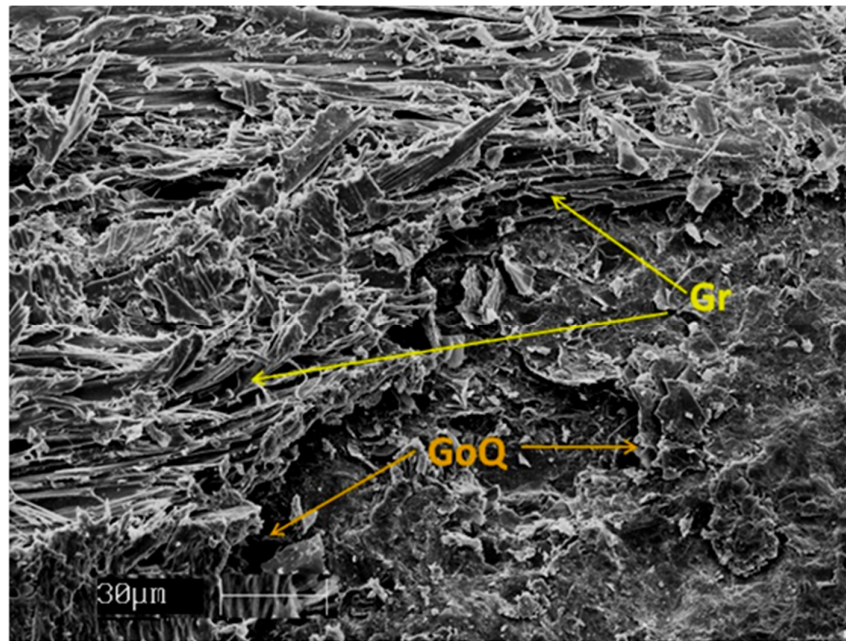


Figure 3. Graphite needles (Gr) within Geothitic Quartz (GoQ) associated with the gangue minerals.

3.2. Flotation

The results of rougher concentrate subjected to five stage cleaning for recovery of high grade graphite is given in Table 1. The data indicate that at first stage of flotation, using flotation parameters constant [pH 9.5, sodium silicate one kg/t for depression of silicate minerals and light diesel oil 0.4 kg/t as the collector dosage and MIBC 0.15 kg/t as frother] a product obtained contains 43.9% FC with 87% recovery from a rougher feed sample contain 26.1% FC. The second stage cleaned product obtained contains 59.5% FC with 91% recovery from a feed contain 43.9% FC. The third and fourth stage cleaned products obtained contains 66.9% FC and 74.7% FC with variable recoveries 90% and 87%, respectively. However, it is observed that at the fifth stage of cleaning a marginal upgrade of product obtained containing 75.7% FC with a

reduction of recovery to 75%. Separation efficiency of graphite with number of cleaning of graphite is shown in Figure 4. The data seen from Figure 4 reveals that as expected with increasing number of cleanings of rougher concentrate, the separation efficiency of graphite is increasing at each stage of cleaning but at the fifth stage cleaning it is observed a sharp fall in separation efficiency. Bu X, Zhang *et al.* also studied the multi-stage flotation for the removal of ash from fine graphite using mechanical and centrifugal forces [23] and suggested for number of cleanings. This may be a fact that the gangue minerals are smeared with graphite coating. The smeared gangue minerals with graphite are easy to float along with graphite, because these minerals are also become hydrophobic due to coating of graphitic carbon, and hence, it has affected the quality of the product.

Table 1. Results of five stage cleaning of rougher concentrate.

Details	Weight, %	FC, %	Recovery, %	Separation efficiency, %
Rougher Concentrate				
[sodium silicate Kg/t, diesel oil 0.4 kg/t, and pH 9.5]				
Concentrate	48.7	26.1	88	16
Tailing	51.3	3.5	12	84
Total	100.0	14.5	100	100
Cleaner concentrate 1 [No reagents used]				
Concentrate	51.9	43.9	87	39
Tailing	48.1	6.9	13	61
Total	100.0	26.1	100	100
Cleaner concentrate 2 [No reagents used]				
Concentrate	67.5	59.5	91	63
Tailing	32.5	11.5	9	37
Total	100.0	59.5	100	100
Cleaner concentrate 3 [No reagents used]				
Concentrate	79.8	66.9	90	72
Tailing	20.2	30.1	10	28
Total	100.0	59.5	100	100
Cleaner concentrate 4 [No reagents used]				
Concentrate	78.2	74.7	87	80
Tailing	21.8	38.9	13	20
Total	100.0	66.9	100	100
Cleaner concentrate 5 [No reagents used]				
Concentrate	73.9	75.7	75	70
Tailing	26.1	71.9	25	30
Total	100.0	74.7	100	100

The results of these studies on number of cleaning of rougher concentrate can clearly be seen in the flowsheet with material balance in Figure 5. Hence, it is concluded that cleaning of

rougher concentrate will not fetch more than 75% FC. In view of this, alternate approaches are made to recovery better grade of graphite.

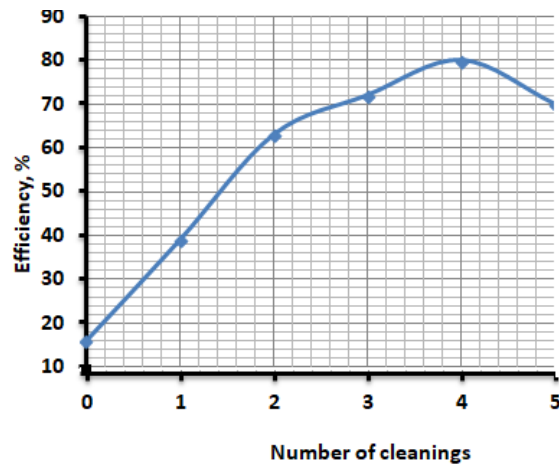


Figure 4. Effect of number of cleaning of rougher concentrate on separation efficiency of graphite.

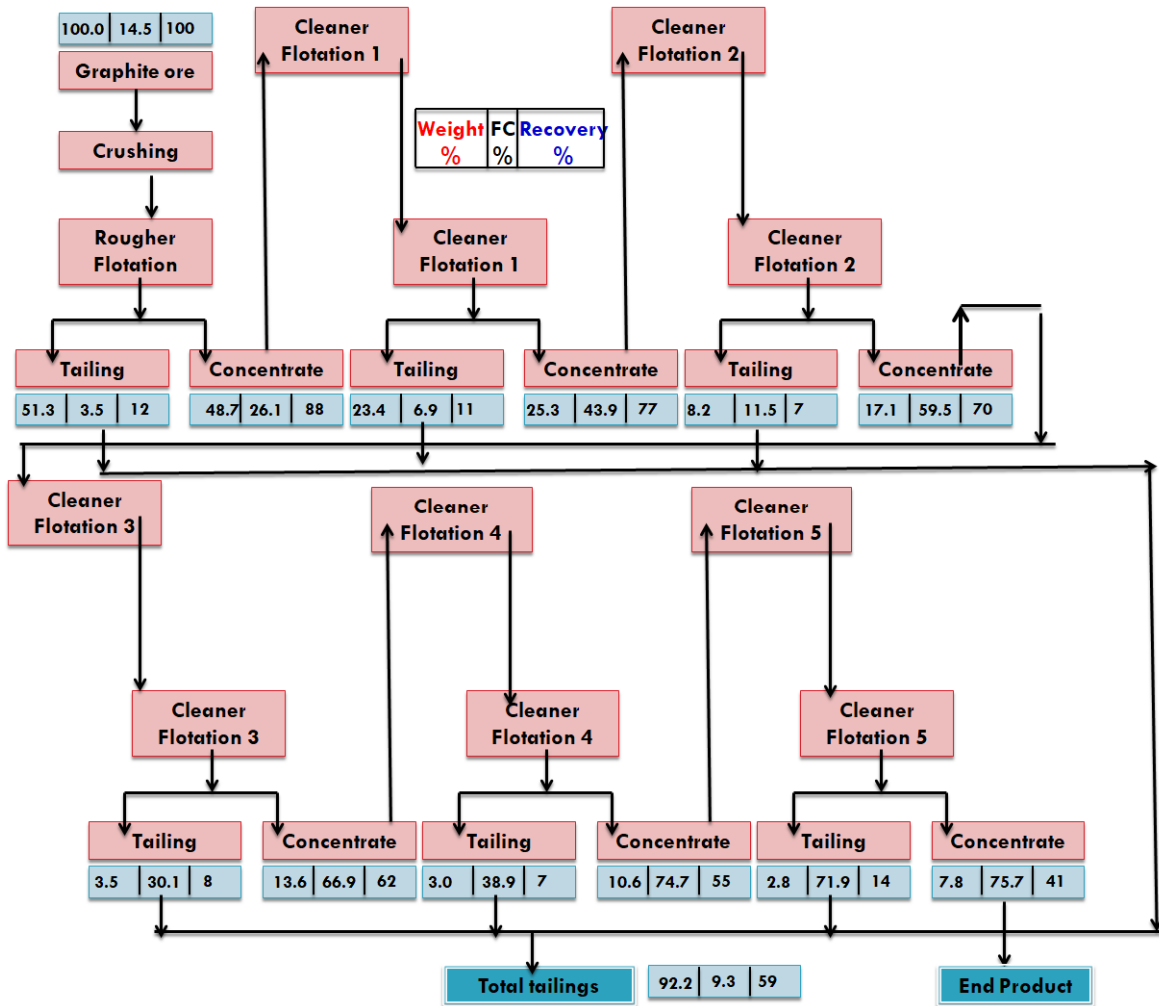


Figure 5. Flowsheet with material balance for recovery of high grade graphite by 5 stage cleanings.

3.3. Grinding aid

A mini-review on flotation techniques and reagents used in graphite beneficiation and reducing the entrainment of gangue fines in low grade microcrystalline graphite ore flotation using multi-stage grinding-flotation process are studied by a few researchers [24 -25] but not studied on grinding aids in flotation. Effect of grinding aid on flash flotation has been studied at different time ground product in the present investigation.

Here, the additive used is sodium silicate, which is being used in the flotation circuit as a depressant for silica. Different time ground products obtained with and without additive have been size analysed and the d_{80} passing size versus separation efficiency of graphite has been assessed. The effect of grinding time with and with additive on production of d_{80} passing size and efficiency of separation of graphite is shown in Figure 6.

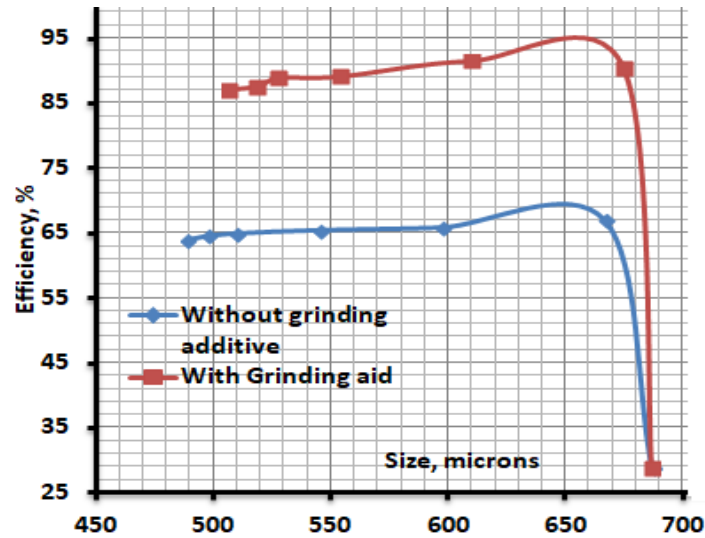


Figure 6. Effect of grinding aid on separation efficiency of graphite.

The data indicates that at any given time of grinding, the effect of grinding aid is significantly better as far as the separation efficiency of graphite. Hence, it is concluded that excessive size reduction of graphite is minimised while using a grinding aid. Based on these observations, further studies have been planned for flash flotation using a comminution circuit with grinding aids.

3.4. Flash flotation and development of flowsheet

Flash flotation term is being used, when the graphite flakes are crushed or ground, a new surface area is being generated. At such condition, the fresh ground feed if it is used for flotation, is known as flash flotation. The regular flotation, is process the feed material can be processed at any time. Flash flotation results with grinding aids on development of flowsheet to recover high grade graphite are given in Table 2. In this table, the results of rougher graphite concentrate obtain in earlier test results [Part 1 of this paper] are repeated for the convenience of the reader. The effect of grinding aid on flash flotation is also given in this Table 2. Results reveal that without grinding aid the product obtained by flash flotation contains 43.1% FC with 92% recovery,

whereas the results obtained with grinding aid, a product achieved contains 49.1% FC and 94% recovery. Hence, further cleanings are continued with starvation dosage in grinding circuit followed by flash flotation.

In all five cleaning of graphite concentrate it is noted that prior to cleaning of each cleaner graphite concentrate, the concentrate sample has been ground for a minute with starvation dosage sodium silicate 250 gr/t for depression of silicate minerals and light diesel oil 0.1 kg/t as the collector dosage at pH 9.5 and subjected to flash flotation. It can clearly be seen from Figure 7 and Table 2 that the end product achieved contains 97.8% FC with 11.6% yield and 78% recovery and the overall tailings contain 3.6% FC by stage comminution with grinding aids followed by flash flotation. When we look into the detailed results of development of flowsheet, the cleaner flotation concentrates ground for a minute with grinding aids as data are given in Table 2 indicate that a product [cleaner concentrate 2] has been upgraded to 70.1% FC from a feed [cleaner concentrate 1] a contain 49.1% FC. Similar way, the products obtained in series that the cleaner concentrate 3 contains 86.5% FC and cleaner concentrate 4 contains 94.7 % FC and the cleaner concentrate contains 97.8% FC.

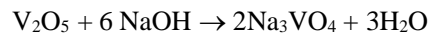
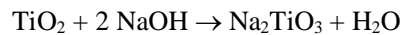
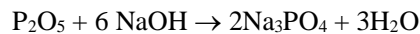
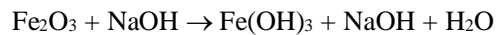
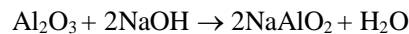
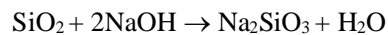
Table 2. Flash flotation results with grinding aids on development of flowsheet to recover high grade graphite

Details	Weight, %	FC, %	Recovery, %
Rougher concentrate			
Concentrate	48.7	26.1	88
Tailing	51.3	3.5	12
Total	100.0	14.5	100
Cleaner concentrate [without grind aid]			
Concentrate	55.6	43.1	92
Tailing	44.4	4.8	8
Total	100.0	26.1	100
Cleaner concentrate 1			
[with grinding aid, starvation dosages sodium silicate 250 gr/t, diesel oil 0.1 kg/t, pH 9.5, and grinding time 1 minute]			
Concentrate	50.1	49.1	92
Tailing	49.9	3.1	8
Total	100.0	26.1	100
Cleaner concentrate 2			
[with grinding aid, starvation dosages sodium silicate 250 gr/t, diesel oil 0.1 kg/t, pH 9.5, and grinding time 1 minute]			
Concentrate	68.1	70.1	98
Tailing	31.9	3.9	2
Total	100.0	49.1	100
Cleaner concentrate 3			
[with grinding aid, starvation dosages sodium silicate 250 gr/t, diesel oil 0.1 kg/t, pH 9.5, and grinding time 1 minute]			
Concentrate	79.9	86.5	99
Tailing	20.1	5.1	1
Total	100.0	70.1	100
Cleaner concentrate 4			
[with grinding aid, starvation dosages sodium silicate 250 gr/t, diesel oil 0.1 kg/t, pH 9.5, and grinding time 1 minute]			
Concentrate	90.5	94.7	99
Tailing	9.5	8.4	1
Total	100.0	86.5	100
Cleaner concentrate 5			
[with grinding aid, starvation dosages sodium silicate 250gr/t, diesel oil 0.1 kg/t, pH 9.5, and grinding time 1 minute]			
Concentrate	96.5	97.8	99
Tailing	3.3	3.7	1
Total	100.0	94.7	100

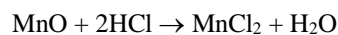
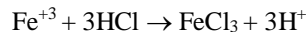
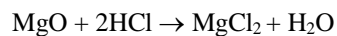
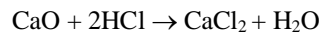
3.5. Pressure leaching and flotation

The high-grade graphite obtained by five stage cleanings has been studied under Scanning Electron Microscope [SEM] to identify the presence of gangue minerals. The image analysis of the graphite sample is shown in Figure 8. The individual graphite grains show that the smeared gangue minerals are on the surface of the graphite [Figure 8 (a)] as well as within the graphite flake Figure 8 (b)]. Figure 8(c) clearly exhibits that the gangue minerals are within the graphite flake. Hence the high grade graphite has been further subjected to pressure leaching using alkalis. The pressure leached sample after cooling, subjected to dilute acid leaching prior to flotation.

The chemical methods adopted for purification of graphite by acid and alkali leaching follows the reactions.



The HCl acid dissociation proceeds as follows:



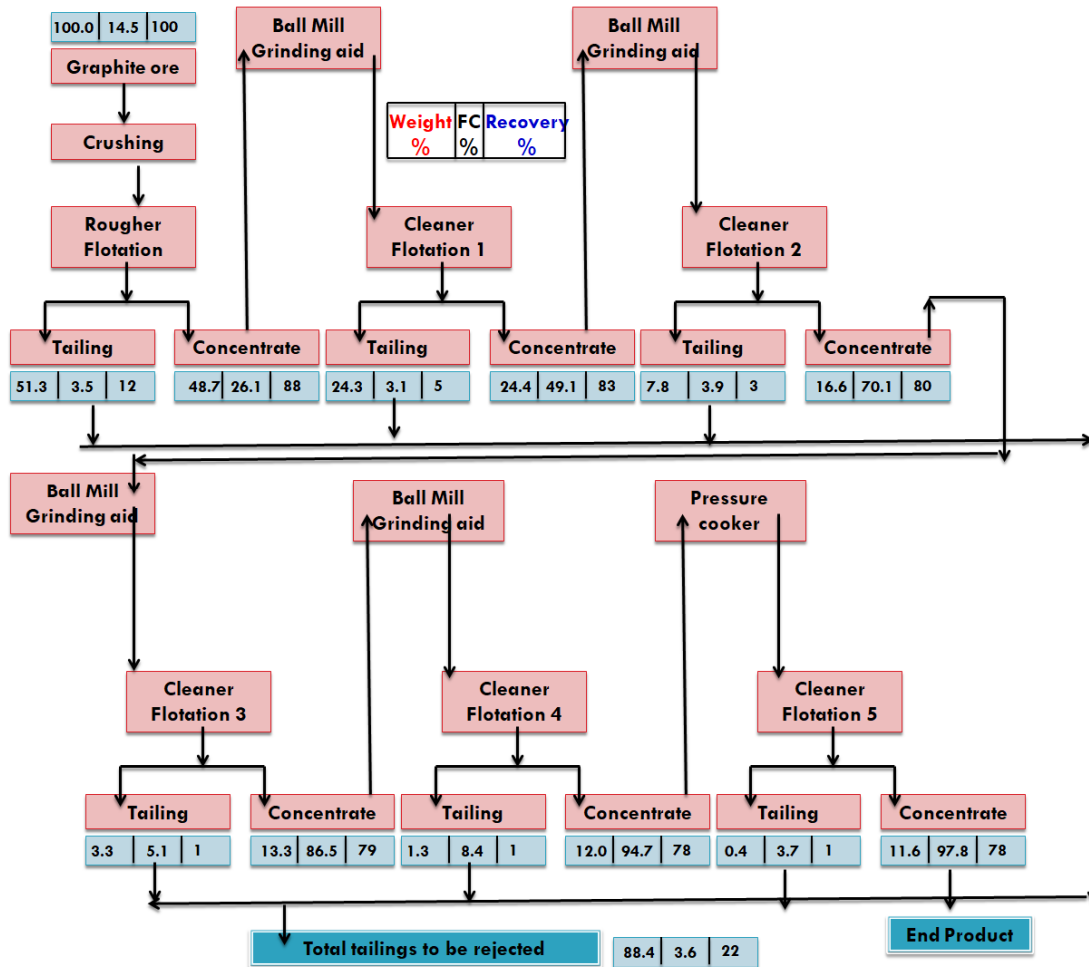


Figure 7. Development of flowsheet to recover high grade graphite.

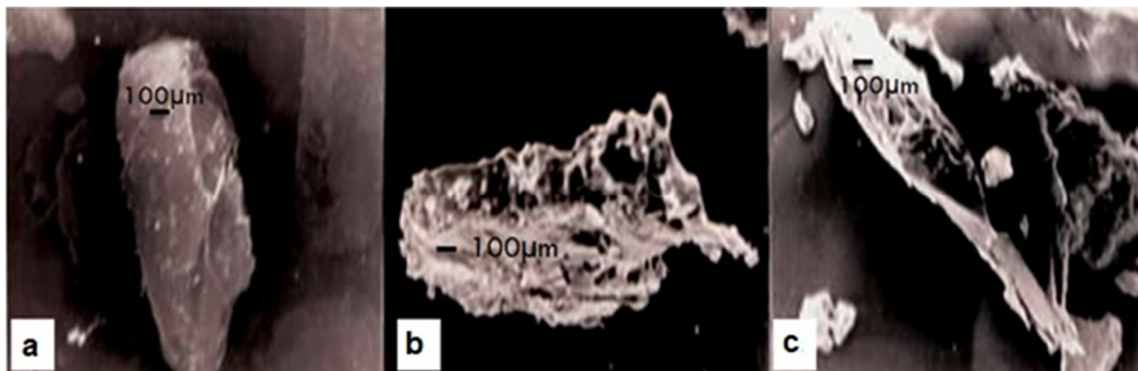


Figure 8. Morphology of graphite flakes with associate gangue minerals seen under SEM showing. a) Graphite flakes on the surface fine smeared gangue. b) Flakes of graphite with inherent gangue. c) Gangue minerals within the graphite flakes.

Alkali flotation process has been carried out with diesel oil 0.05% Kg/ton at natural pH because sodium hydroxide keeps the flotation environment in alkali condition, and it may be noted here further that no depressant has been used for flotation. This is due to a fact that when pressure leaching has been carried out with

sodium hydroxide, a chemical reaction has been taken such that quartz mineral has been formed as sodium silicate, which in the process helped further to depress the liberated silicates. During alkali pressure, the graphite flakes are opened up to little extent and there by the gangue minerals releases from the flakes and liberated freely from

the graphite. Thus during flotation, the gangue minerals will further depressed and enhance the grade of the product. The end product achieved contains 99.2% FC with overall 10.4% yield and 71% recovery. The complete chemical analysis of the end product [99.2% FC] is given in Table. 3. The complete chemical analysis of the sample indicate that the ash content of the high purity

graphite 99.2% Fixed Carbon (FC) contain mostly silicate minerals followed by iron and calcium carbonate minerals. As shown in Table 4, the SiO₂ content of the ash is 70.1%, Al is 12.7%, Fe and Ca contents are 5.3% and 9.6%, respectively, for assessing the mineral composition in the ash content.

Table 3. Proximate analysis of the final graphite concentrate

Details	Moisture	Volatile matter	Ash	Fixed carbon
percentage %	0.03	0.40	0.40	99.20

Table. 4. Chemical analysis of ash.

Details	SiO ₂	Fe	Al	Ca	Mg	P	S	K	Na	Pb	Cu
Percentage, %	70.1	5.3	12.7	9.6	0.002	0.38	0.43	0.72	0.76	0.002	0.005

In the flotation of graphite, in general, hydrocarbons such as parafn, diesel, and kerosene are used, as well as ionic collectors such as potassium amyl (or ethyl) xanthates, di-thiocarbamates, and di-thiophosphate are being used. The future work on recovery of graphite instead of using traditional collectors such as kerosene, which is a ration item in India, diesel oil which is being used in automobiles are to be restricted and to see for alternative chemicals because the traditional chemicals are in high demand for mankind. In view of this it is suggested to use bio collectors by which national economy can be saved by avoiding the demanded chemicals and additionally it satisfy the man need. A few researchers [26-31] had already attempted for purification and published their work in terms for recovery of graphite. Vasumathi and her teammates [30] developed a bio-collector to recover graphite from a low grade deposit and reported that the end graphite product obtained contain 4.24% ash and 14.42% yield by using the developed bio-collector on flotation of low-grade graphite ore. Similarly, Gamaralalage *et al.* [31] studied a chemical-free flotation technology for recovery of high grade graphite, in which a suspension of pristine powdered graphite (PG) is dispersed and stirred in water without adding froth formers or supportive chemicals and recommended that the technique is helpful for the value enhancement of graphite, the reduction of the chemical cost of the conventional techniques, environmental friendliness, and improvement of its applications.

4. Conclusions

The developments of process flowsheet for recovery of high grade graphite for industrial applications with special reference to prepare high grade or high pure graphite in the present investigations, the following conclusions are drawn:

- The general practice is for obtaining a desired graphite grade; the rougher concentrate has to be subjected for number of cleanings. The present study reveals that number of cleanings of rougher concentrate will not fetch more than 75% FC with less than 8% yield.
- Rougher concentrate ground with and without additives and subjected to flash flotation. The results indicate that that without grinding aid the product obtained by flash flotation contain 43.1% FC with 92% recovery, whereas the results obtained with graining aid, a product achieved contains 49.1% FC and 94% recovery.
- The effect of further four stage cleanings continued with starvation dosage in grinding circuit followed by flash flotation. It can clearly be seen that the end product achieved contains 97.8% FC with 11.6% yield and 78% recovery and the overall tailings contain 3.6% FC.
- The end product achieved by alkali digestion method from a flotation product [97.8% FC] contains 99.2% FC with overall 10.4% yield.
- The ash analysis of this end graphite concentrate contain around 70.1% SiO₂, Al is 12.7%, Fe and Ca contents are 5.3% and 9.6%, respectively.
- The process adopted in the present investigation is friendly environment and process flowsheet is an innovative.

- The end product obtained from this process is very useful for different industrial applications.
- Future studies are in progress with alternate collectors such as bio collectors as compared to the traditional collectors.

Acknowledgement

The authors are thankful to the Director CSIR IMMT for giving facilities to carry out the experiments and encouraged to publish the paper.

References

- [1]. Indian Minerals Yearbook, 2018 (Part- III: Mineral Reviews) (2019). 57th Edition graphite, government of India, Ministry of Mines. *Indian Bureau of Mines*, 14.1-14.13.
- [2]. Sengupta, R., Bhattacharya, M., & Bandyopadhyay, S. (2011). A review on the mechanical and electrical properties of graphite and modified graphite reinforced polymer composites *Progress in polymer*, 36(5), 638-670.
- [3]. Chelgani, S.C., Rudolph, M., Kratzsch, R., Sandmann, D., & Gutzmer, J. (2016). A review of graphite beneficiation techniques. *Min Processing and Extractive Metallurgy Rev*, 37(1), 58-68.
- [4]. Al-Ani, T., Leinonen, S., Ahtola, T., & Salvador, D. (2020). High-Grade Flake Graphite Deposits in Metamorphic Schist Belt, Central Finland—Mineralogy and Beneficiation of Graphite for Lithium-Ion Battery Applications. *Minerals*, 10(8), 1-16.
- [5]. Zhang, L., Peng, W., Li, X., & Qiu, Y. (2014). Sichuan [J]. *Multipurpose Utilization of Mineral Resources*, (1), 40-44.
- [6]. Floreana, F.F., Syarifuddin, F., Hanam, E.S., Trisko, N., Kustiyanto, E., Enilisiana, Rianto, A., & Arinton, G. (2016). Floatability study of graphite ore from southeast Sulawesi (Indonesia). *AIP Conference Proceedings*, 1712, 050005.
- [7]. Peng, W., Qiu, Y., Zhang, L., Guan, J., & Song, S. (2017). Increasing the Fine Flaky Graphite Recovery in Flotation via a Combined Multiple Treatments Technique of Middlings. *Minerals*, 208(7), 1-13.
- [8]. Hao, W., Qiming, F., Liu, K., Zuo, K., & Tang, X. (2018). A novel technique for microcrystalline graphite beneficiation based on alkali-acid leaching process. *Separation Science and Tech*, 53(6), 982-989.
- [9]. Ziming, P., Dianshun, Li., Wenjie, F., Zhang, J., Zhang, R., Qiu, Y., & Sun, K. (2023). Improved Flotation of Fine Flake Graphite using a Modified Thickening Process, *Separations*. 10(5), 275.
- [10]. Wang H., Feng, Q., Liu, K., & Zuo, K. (2018). A novel technique for microcrystalline graphite

beneficiation based on alkali-acid leaching process. *Separation Sci and Tech*, 53, 982-989.

- [11]. Jara, A. D., & Kim, J.Y. (2020). Chemical purification processes of the natural crystalline flake graphite for Li-ion Battery anodes. *Mat Today Comm*. 25, 1-30.
- [12]. Syarifuddin, F., Floreana, F.F., & Hanam, E. S. (2016). Effect of acid leaching on upgrading the graphite concentrate from West Kalimantan (Indonesia). *AIP Conference Proceedings*, 1712, 050022: 1-7.
- [13]. Ravichandran, V. (2012). A PhD thesis on Studies on the effect of chemical reagents on beneficiation of natural graphite, Alagappa University, India.
- [14]. Thi, H. T., & Hong, N. D. (2017), Sulfuric Acid Leaching Process for Producing High Purity Graphite from 92.6% C to 98% C. *World Jr of Res and Rev*, 5(1), 23-26.
- [15]. Wang, H., Feng, Q., Tang, X., & Liu, K. (2016). Preparation of high-purity graphite from a fine microcrystalline graphite concentrate: Effect of alkali roasting pre-treatment and acid leaching process. *Separation Sci and Tech*, 51, 2465-2472.
- [16]. Sun, K., Qiu, Y., & Zhang, L. (2017). Preserving flake size in an African flake graphite ore beneficiation using a modified grinding and pre-screening process. *Minerals*, 7(7), 1-13.
- [17]. Quast, K. B., Hall, S. F., & Skinner, W.M. (2018). Comminution and flotation of Renascor graphite ore from Arno Bay, South Australia. *47th Conference- Chemeca 2018; Queenstown, New Zealand*, 120.1-120.11.
- [18]. Barma, S. D., Baskey, P. K., Rao, D. S., & Sahu, S. N. (2019). Ultrasonic-assisted flotation for enhancing the recovery of flaky graphite from low-grade graphite ore. *Ultrasonics sonochemistry*, 56, 386-396.
- [19]. Kang, K., & Li, H. (2019). Enhancement of flaky graphite cleaning by ultrasonic treatment. *The Royal Society*, 6 (12), 1-11.
- [20]. Wang, X., Bu, X., Ni, C., Zhou, S., Yang, X., & Zhang, J. (2021). Effect of scrubbing medium's particle size on scrubbing flotation performance and mineralogical characteristics of microcrystalline graphite. *Int Jr of Coal Prep and Utilization*, 163(15), 106766-1066776.
- [21]. Hu, P., Liang, L., Peng, Y., Yu, H., & Xie, G. (2020). Recovery of Microcrystalline Graphite from Quartz using Magnetic Seeding. *Minerals*, 24(10), 1-13.
- [22]. Sinha, N., & Singh, R. (2014). Beneficiation and purification of a low grade and finely

disseminated graphite ore. *Jr of Metal and Mat Science*, 56(1), 15-22.

[23]. Bu, X., Zhang, T., Peng, Y., Xie, G., & Wu, E. (2018). Multi-stage flotation for the removal of ash from fine graphite using mechanical and centrifugal forces. *Minerals*, 8(1), 1-15.

[24]. Vasumathi, N., Sarjekar, A., Chandrayan, H., Chennakesavulu, K., Reddy G.R., Kumar. T.V. V, El-Gendy, N S., & Gopalkrishna, J. (2023). A Mini Review on Flotation Techniques and Reagents Used in Graphite Beneficiation. *International Journal of Chemical Engineering*, 1007689, 1-15.

[25]. Weng, X., Li, H., Song, S., & Liu, Y. (2017). Reducing the entrainment of gangue fines in low grade microcrystalline graphite ore flotation using multi-stage grinding-flotation process. *Minerals*, 7(3), 1-12.

[26]. Zhang, L., Zhang, D., Qiu, Y., & Pan, L. (2012). Experimental Research on Beneficiation of a Refractory Fine Graphite Ore in Korea Area. *Multipurpose Utilization of Mineral Res*, 2, 28-32.

[27]. Ding, S. F., & Niu, Y. P. (2023). Research on purification techniques of some flake graphite. *Advanced Materials Research*. 753-755, 119-123.

[28]. Zhang, L. Y., Huang, W., Qiu, Y. S., & Pan, L. (2011). Experimental study on purification of a low-carbon graphite ore by flotation. *Journal of Wuhan Univ of Tech*, 33(11), 107-111.

[29]. Gabriela, L. L., Henriques, A. B., & da-Silva, G. R. (2023). Graphite ore flotation efficiency using bio ester collector formed by transesterification. *REM - International Engineering Journal*, 76 (02), 195-201.

[30]. Vasumathi, N., Chennakesavulu, K., Cassandra, A. I., Kumari, A., Kalidoss, J., Suseelamma, S. S., Tadiparthi, V. V., & Sirigeri, G. (2023). Development of a novel biocollector for flotation of low grade graphite ore. *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects*, 45(1), 229-245.

[31]. Gamaralalage, R. A. K., Herath, M. G. T. A. P., Karunarathne, B., Mantilaka, M. M. G. P. G. M., Rajapakse, M. G. R., Hsin-Hui, H., De-Silva, K. K. H., & Yoshimura, M. 2021, Development of a chemical-free floatation technology for the purification of vein graphite and characterization of the products. *Scientific Reports*, 11, 22713-22719.

تهیه گرافیت با خلوص بالا با استفاده از خرد کردن، شستشو با فشار و فلوتاسیون

ساتیا سای سریکانت^{1*} و راگوپاترونی بهیما رائو²

1. گروه ECE، موسسه علم و فناوری SRM، پردیس دهلی-ان سی آر، مودیناگر، غازی آباد، هند
2. موسسه فناوری مواد معدنی و مواد (CSIR)، بوبانسوار، هند

ارسال 2023/07/13، پذیرش 2023/08/30

* نویسنده مسئول مکاتبات: satya.srikant@gmail.com

چکیده:

مقاله حاضر به توسعه برگه فرآیند برای بازیابی گرافیت با عیار بالا از کنسانتره گرافیت خشن‌تر برای استفاده در کاربردهای صنعتی می‌پردازد. در تحقیق حاضر، از آنجایی که ورقه‌های درشت گرافیتی تقاضای بالاتری دارند، در هر مرحله از خرد کردن تلاش می‌شود و آزمایشات فلوتاسیون فلاش انجام شده است و محصول نهایی به دست آمده از خردایش مرحله و سپس فلوتاسیون بیشتر مورد بررسی قرار گرفته است. شستشو با فشار قلیایی و به دنبال آن آسیاب و فلوتاسیون برای بازیابی کنسانتره گرافیت صنعتی. در این مرحله سنگ زنی، یک افزودنی، یک ماده کاهش دهنده که برای فرورفتگی سیلیس در حین شناورسازی استفاده می‌شود، به عنوان کمک آسیاب استفاده شده است. داده‌ها نشان می‌دهند که در هر زمان آسیاب، اثر کمک آسیاب به میزان قابل توجهی بهتر از کارایی جداسازی گرافیت است. از این رو، نتیجه‌گیری می‌شود که کاهش اندازه بیش از حد گرافیت در هنگام استفاده از کمک سنگ زنی به حداقل می‌رسد. بر اساس این مشاهدات، مطالعات بیشتری برای فلوتاسیون فلاش با استفاده از یک مدار خرد کردن با کمک سنگ‌زنی برنامه ریزی شده است. نتایج مطالعه حاضر نشان می‌دهد که تعداد تمیز کردن کنسانتره خشن‌تر به تنهایی بیش از 75 درصد کربن ثابت (FC) با عملکرد کمتر از 8 درصد و بازیابی 41 درصد دریافت نمی‌کند. اثر پاک‌سازی چهار مرحله‌ای با دوز گرسنگی در مدار آسیاب و به دنبال آن فلوتاسیون فلاش نشان می‌دهد که محصول نهایی به دست آمده حاوی 97,8 درصد FC با 11,6 درصد عملکرد و 78 درصد بازیابی و افت ارزش کلی در باطله‌ها حاوی 3,6 درصد FC است. محصول نهایی به دست آمده با روش هضم قلیایی از یک محصول شناور [FC %97,8] حاوی 99,2 درصد FC با عملکرد کلی 10,4٪ و 71٪ بازیابی است. فرآیند اتخاذ شده در تحقیق حاضر، محیطی دوستانه است و برگه جریان فرآیندی نوآورانه است. محصول نهایی به دست آمده از این فرآیند برای کاربردهای مختلف صنعتی مفید است.

کلمات کلیدی: گرافیت، کنسانتره خشن، هضم قلیایی، فلوتاسیون فلاش، شستشوی فشاری.