

Effect of Grinding Media on the Preparation of Rougher Flake Graphite Concentrate

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Abstract

Protecting the coarse-flake graphite is one of the most important tasks during the grinding of graphite ore because coarse-flake graphite is more valuable than fine-flake graphite. The graphite ore sample contains 14.50% FC, 71.82% ash, and 13.68% volatile matter + moisture content. In the present study, the test work has been carried out on the graphite ore to maximize the recovery of rougher coarse flake graphite in a rod mill by varying the grinding media such as (i) rods, (ii) cylindrical metallic pebbles and (iii) combination of rods and cylindrical metallic pebbles in the weight ratio of 1:1, followed by rougher flotation. The effect of various flotation parameters such as a Mesh of Grind (MOG), pH, and dosages of Sodium silicate, Kerosene, and Methyl Isobutyl Carbinol (MIBC) on rougher flotation was carried out, and optimal parameters were also established. A comparative study of varying grinding media followed by rougher flotation at optimal parameters was carried out concerning the grade and recovery of coarse flaky graphite. The results indicated that the rod mill grinding with rods as a grinding media followed by rougher flotation of mill products produced a better grade (43.74% FC) and recovery (43.3%) of rougher coarse flaky graphite concentrate as compared to the other two grinding media.

Keywords: Rod mill, Grinding Media, Rods, Cylindrical Metallic Pebbles, Rougher Flotation.

1.0 Introduction

Generally, graphite occurs in nature in the form of flaky, vein, and amorphous based on the difference in crystalline morphology. It has a unique function in various industries because of its physical and chemical properties.^{1,2} It generally arises due to the metamorphism of organic matter in sediments, and the carbonaceous material converts to microcrystalline graphite when the metamorphic grade increases.^{3,4} Graphite is one of the most important minerals used in the industry manufacturing products such as crucibles, refractories, foundry facings, paints and pigments, electrodes, pencils, inks and coatings, two-dimensional renderings, 3D carvings etc. It is used in friction, molding, and conductive applications due to its high thermal and electrical conductivity and low spring-

back properties. Graphite ore comprises coarse and fine flakes, and the flake graphite can only be noticed in nature. Coarse flake graphite cannot be synthesized using existing modern technology.⁵ Flake graphite has a flaky morphology, and it is formed due to the metamorphic geological environment by the heat and pressure metamorphism of dispersed organic material^{6,7}. The flake graphite concentrate contains above 80.0% Fixed Carbon (FC), and its size varies from less than 1 μ m to 800 μ m. The size of the coarse flake graphite is plus 150 μ m in diameter, and the fine flake graphite is minus 150 μ m in diameter¹. Coarse flake graphite has a more significant industrial value than fine flake graphite due to some exclusive properties such as excellent thermal conductivity and lubricity etc.⁶. The floatability of coarse flake graphite ore is more than fine flake graphite ore during the flotation process. The priority is to avoid over-grinding and thereby enhance the weight fraction of coarse flake graphite⁸.

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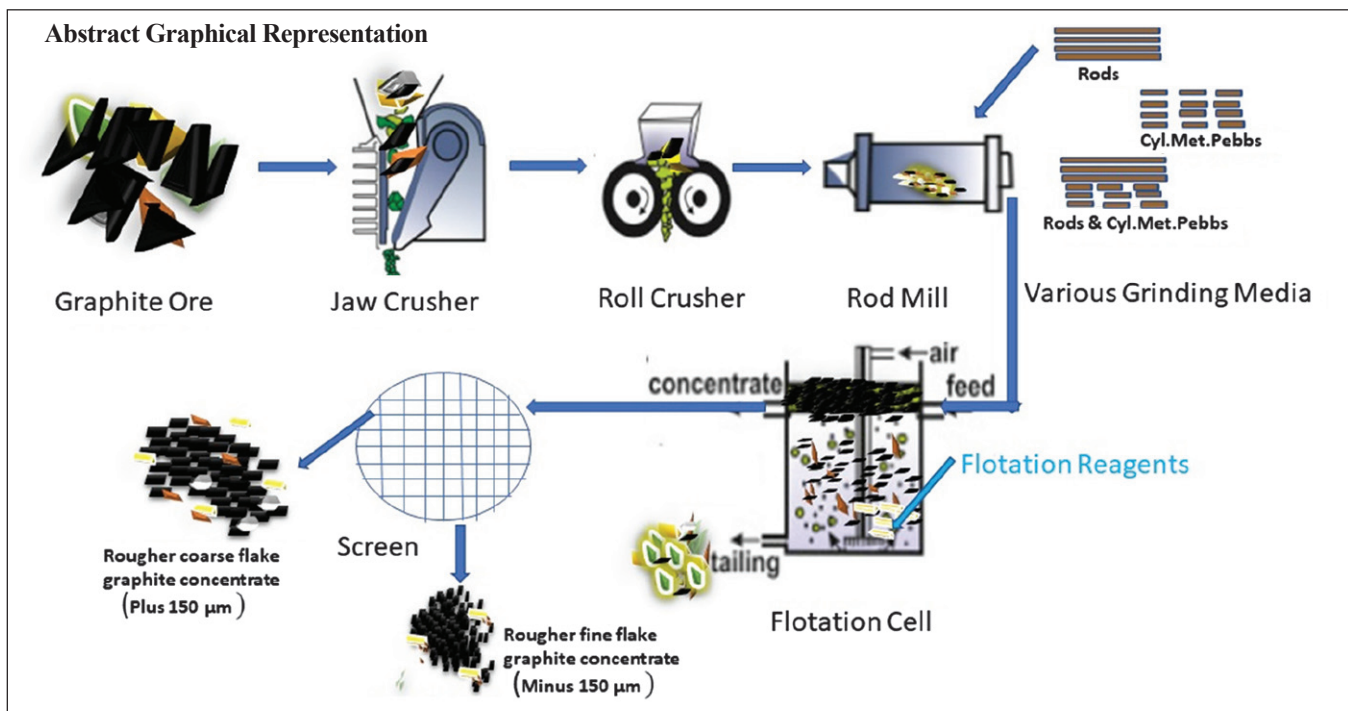


Figure 1: Graphical representation of Abstract

Two main factors, such as over-grinding and the abrasive action of harder gangue minerals (e.g., quartz and pyroxene), affect the yield of coarse flake graphite during the grinding operation^{9,10}. A small Wits laboratory mill with grinding media as balls (B), cubes (C), eclipsoids (E), and their combinations (B-E, B-C) of were used to carry out the batch grinding tests on the quartz sample. The breaking rate is more in the combination of grinding media. It is due to increasing the volume of grinding zones and media surface area¹¹. The product of plus 150µm coarse flake graphite containing 92.9% FC is achieved by five times regrinding and six times cleaning in a closed circuit¹². The graphite concentrate grade obtained by rod mill grinding followed by froth flotation is between 67.0% and 83.0%¹³. The recovery of flake graphite is more in low-frequency ultrasound-assisted flotation when compared to conventional flotation¹⁴.

In the present scenario, protecting the coarse flakes in the graphite ore is a challenging task during the grinding process. Once the flakes in the graphite ore are wrecked during the grinding operation, they cannot be restored. Hence, there is a strong need to ameliorate the grinding process to protect the flakes of the graphite ore. Hence, a study has been undertaken using a rod mill for primary grinding with various grinding media such as rods, cylindrical metallic pebbles, and rods & cylindrical metallic pebbles in the weight ratio of 1:1 followed by rougher flotation to protect the coarse flakes of the graphite ore.

Effects of various process parameters such as a mesh of

grind (MOG), pH, and dosage of Sodium silicate, Kerosene, and Methyl Isobutyl Carbinol (MIBC) on the rougher flotation were studied. The maximum liberated coarse flake and fine flake graphite from the associated gangue minerals, such as quartz, calcite, dolomite, etc., were recovered during rougher flotation in two stages at optimal process parameters with minimum graphite losses in the rougher tailings.

2.0 Materials and Methodology

2.1 Materials

The bulk graphite ore sample was collected from Tamil Nadu Minerals Limited (TAMIN), Sivaganga, Tamil Nadu. The entire graphite ore sample was initially crushed using a Jaw crusher, and the crushed product was sent to a Roll crusher to reduce it to all passing 1680 µm size. The representative samples were drawn from the bulk sample for studying the mineralogical characteristics and chemical composition and used in the rod mill grinding followed by rougher flotation test works.

2.2 Methodology

2.2.1 Microscopic Studies

The low-grade calcareous graphite sample's mineralogy was studied using the optical microscope (CARL ZEISS AXIO

Scope A1 model). The sample size for the preparation of Mould and Grain mounting sections is 210 μ m. These sections were prepared as per the Standard Operating Procedure (SOP) to identify the opaque and transparent minerals by using a polarized microscope.

2.2.2 Proximate Analysis

The composition of the original graphite sample was determined by using proximate analysis as per SOP. The original graphite sample size (i.e., 1680 μ m) was reduced to 100 μ m. This sample was subjected to proximate analysis.

2.2.3 Rod Milling

The grinding action in the rod mill is by attrition. Conventional rod mill acts as a grinder and as well as a classifier. Rod mill acts as a classifier when the larger and finer particles come into contact between two rods. The larger particles will be gradually reduced to finer particles while the existing finer particles will be escaped from voids between the rods. Therefore, the generation of finer particles will be minimized. The breakage of coarse flakes was minimized due to the line contact along the rods with the ore particles. The rod mills operations are suitable for the preparation of feed to specific processes such as flotation, gravity separation, magnetic separation, Etc.,

The grinding operation was carried out on the calcareous graphite ore in the rod mill (180 mm by 350 mm, Denver make, 0.5 HP) using different grinding media such as rods, cylindrical metallic pebbles, and a combination of rods and cylindrical metallic pebbles in the weight ratio of 1:1. The grinding test work was carried out with 1kg of minus 1680 μ m size of graphite sample with a constant weight of various grinding media, feed, constant speed of rod mill and maintaining the pulp density at 66.6% by weight separately. The rod mill product was sent through a 210 μ m sieve size, and the oversized particles were sent back to the rod milling, called a closed circuit. This procedure was repeated till all the particles pass through the 210 μ m sieve size.

2.2.4 Rod Mill Feed and Product Analysis

The rod mill feed sample and ground products obtained with different grinding media were subjected to wet sieve analysis separately. The standard set sieves such as 1680, 840, 590, 420, 300, 210, 150, 105, 75, and 53 μ m were used for the feed sample to determine the size distribution. Similarly, the ground products were sieved over 150, 105, 75, and 53 μ m sizes. Wet sieve analysis was done on the rod mill ground products using different grinding media over the 150 μ m sieve for each experiment separately. The coarser (plus 150 μ m) and finer (minus 150 μ m) weight fractions were determined. Each mill product was subjected to proximate analysis to quantify the FC, ash, and volatile matter + moisture content.

2.3 Parametric Study

The effects of various parameters were examined for the rougher flotation of conventional rod mill-grounded products. The ground products from the rod mill grinding with various grinding media were subjected to rougher flotation at optimal parameters such as Mesh of Grind (MOG), pH, the dosage of Sodium silicate, Kerosene, and MIBC separately. The maximum graphite minerals were recovered during the rougher flotation in two stages with minimum loss of graphite in the rougher tailings. The final rougher graphite float products were subjected to sieve analysis over 150 μ m standard sieve to separate the coarse and fine flake graphite size fractions. Proximate analysis has been carried out on these rougher flotation products. The study of process parameters on rougher flotation is explained below.

2.3.1 Mesh of Grind

Sodium carbonate, sodium silicate, kerosene, and MIBC were used in the graphite flotation as pH modifiers, depressants, collectors, and frothers, respectively¹⁵. To determine the liberation size, One Kilogram of the test sample (i.e., 1680 μ m size) was subjected to a rod mill grinding to reduce the particle size, which can pass through 420, 300, 210, and 150 μ m sieves separately. The rougher flotation was carried out for each grind product separately at constant process parameters such as pH 8.0, Sodium silicate 0.55 kg/tonne, Kerosene 0.161kg/tonne, and MIBC 0.05 kg/tonne.

2.3.2 pH

The pH is one of the essential parameters in flotation operations. Majority of mineral flotation operations, there is an optimum pH range in which optimum results could be obtained. In most of the graphite flotation operations, the alkaline environment is maintained because the floatability of graphite is more in the alkaline region rather than in the acidic region¹⁵. Hence, the rougher flotation test work trials were carried out on the graphite ore at the pH range of 8.0 to 9.5, keeping other process parameters constant. The pH of the pulp was modified with the addition of sodium carbonate. The sodium carbonate is not only used as a pH modifier but also as a dispersant which improves the performance of the flotation process.

2.3.3 Reagent Dosages

The rougher flotation test works were carried out by varying the dosage of sodium silicate (0.55 kg/tonne to 0.85 kg/tonne), kerosene (0.114 kg/tonne to 0.184 kg/tonne), MIBC (0.05 kg/tonne to 0.125 kg/tonne) while keeping other process parameters constant. Generally, most of the silicate minerals are effectively depressed in the flotation process by using sodium silicate^{16,17}. Graphite is a naturally floatable mineral;

even then, it requires a reagent called a collector, which provides adequate strength to the graphite mineral to float in the flotation process. Kerosene is a suitable collector to float graphite minerals^{16,17}. Light diesel oil can also be used as a collector apart from kerosene. Kerosene has produced a better graphite concentrate grade and recovery than the other collectors used. Hence, the rougher flotation test works were carried out by varying the dosages of kerosene. MIBC is used as a frother in the flotation process. Its presence increases the stability of air bubbles and helps keep air bubbles dispersed to facilitate improving the conditions for partial mineral attachment. MIBC is an efficient frother compared to pine oil and polyglycolic ethers.^{18,19} The rougher flotation test works were carried out by varying the dosage of MIBC.

2.3.4 Rougher Flotation of Rod Mill Products

The rougher flotation test was carried out on the rod mill products at optimal parameters of reagents such as sodium carbonate, kerosene, sodium silicate, and MIBC to separate the graphite minerals from gangue minerals effectively and produce a desired quality and quantity of rougher graphite concentrate. The flow sheet for the preparation of rougher flake graphite concentrate is shown in Figure 2.

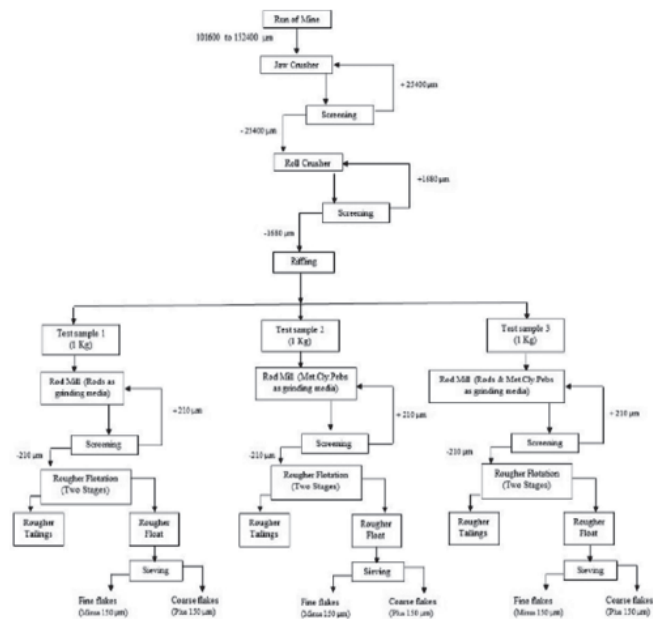


Figure 2: Flow sheet for Rougher Graphite concentrate preparation

3.0 Results and Discussions

3.1 Characterization Studies

3.1.1 Microscopic Studies

The microscopic studies indicated that it contains graphite, quartz/feldspar, and carbonates (calcite and dolomite) as major minerals. Goethite, limonite, mica (muscovite/biotite), hematite, magnetite/martitized magnetite as minor minerals, and traces of ilmenite, pyrite, rutile, garnet, etc. were also noticed. The photographs of the original graphite sample for the mould and grain mounting section are shown in Figures 3(a) to (d).

3.1.2 Proximate Analysis

The proximate analysis results indicated that the original graphite ore sample contains 14.50% FC, 71.82% ash, and 13.68% volatile matter + moisture content.

3.1.3 Rod Mill Grinding with various Grinding Media

The wet sieve analysis of the feed sample and products of rod mill grinding performed by varying the grinding media such as rods, cylindrical metallic pebbles, and a combination of rods and cylindrical metallic pebbles are shown in Figure 4 (a) and (b). This figure shows that there is not much difference in the size distribution of ground products obtained with rods, cylindrical metallic pebbles, and the combination of rods and

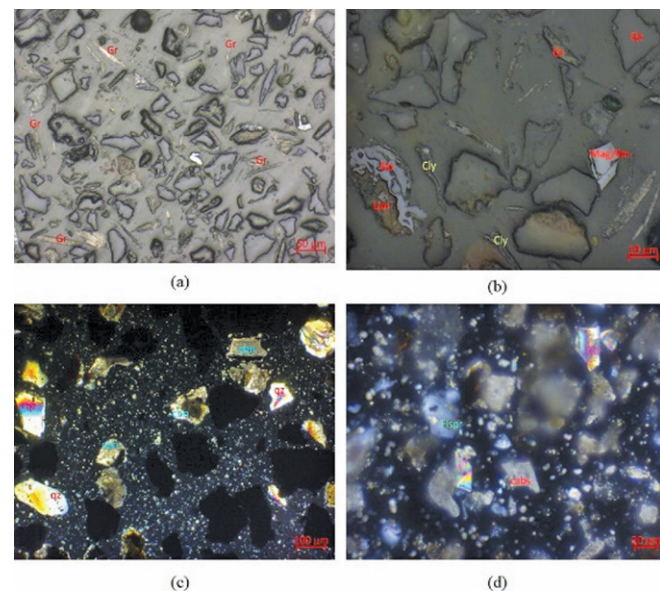


Figure 3: Microscopic images (a) & (b) Mould section under reflected light (c) & (d) Grain mounting section under plain polarised light with crossed nicols. (Notation: Gr: Graphite, Go: Goethite, Lim: Limonite, Cly: Clay, Mag/ilm: Magnetite/ilmenite qz: Quartz, Cbn/cabs: Carbonates, Flspr: Feldspar)

cylindrical metallic pebbles with a weight ratio of 1:1 used as grinding media in the mill. However, the product size distribution of graphite ground with only rods has shown that the size degradation is marginally less than the other two grinding media. The weight yield of plus 150µm size fraction

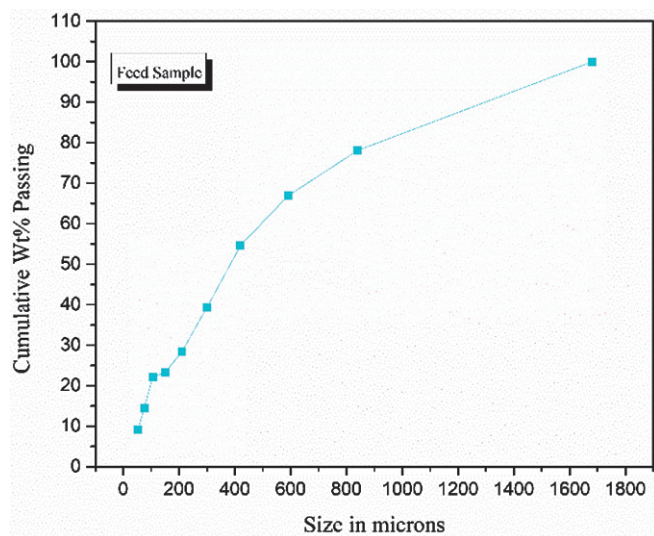


Figure 4(a): Sieve analysis of feed sample

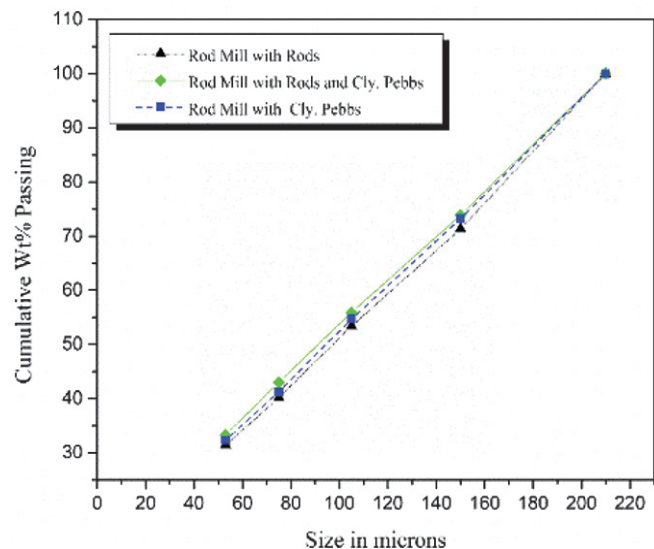


Figure 4(b): Sieve analysis for products of Rod mill with various grinding media

particles decreased marginally from 28.7% to 26.1% using rods and a combination of rods and cylindrical metallic pebbles, as shown in Figure 5. From the test results, it is clearly understood that the coarse flakes are marginally protected. At the same time, the production of fines is minimized during rod mill grinding using rods compared to other grinding media because of attrition action. The marginal improvement in the grade and recovery of plus 150 μm size fractions was observed in the rod mill grinding using rods compared to the other two grinding media. The assay characteristics of products of rod mill grinding with different grinding media are shown in Figure 5.

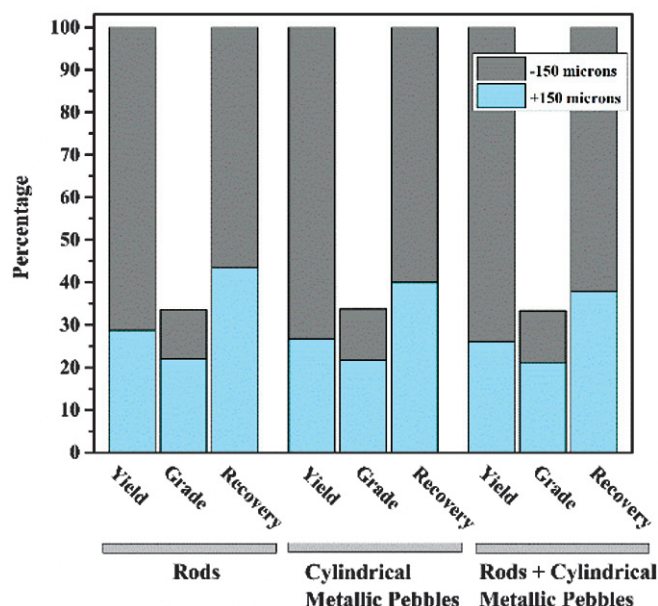


Figure 5: Results of Rod mill product with different grinding media

3.2 Parametric Studies on Rougher Flotation

3.2.1 Mesh of Grind

MOG plays a vital role in beneficiation studies as it provides information about the liberation size of the graphite mineral from unwanted minerals. The graphite minerals are interlocked/intermixed with gangue minerals such as iron, silicates, carbonates, etc. The interlocked/intermixed gangue particles are detached gradually from the graphite minerals during the grinding operation. Selective adsorption of flotation reagents on the surface of a particle mainly depends on its surface characteristics and size²⁰. The results of the mesh of grind are shown in Figure 6. The results clearly show that the recovery of FC content increased significantly from 74.9 % with a grade of 35.89% to 96% with a grade of 40.62% in 420 μm and 150 μm grind, respectively. A fair degree of graphite liberation could be achieved in the range of 210 μm to 150 μm . The grade and recovery of graphite marginally increased in the 150 μm grind, i.e., 40.85% FC with a recovery of 96.0% compared to the 210 μm grind, i.e., 40.62% with a recovery of 95.7%²¹. But the entire coarse flake graphite in the ore was reduced to fine flake graphite during the 150 μm grind, which has less economic value than coarse flake graphite. So, the optimal mesh of the grind was determined as 210 μm .

3.2.2 Effect of pH

The effect of pH is important in improving the floatability of graphite minerals in the flotation process as graphite is a naturally floatable mineral due to its hydrophobic nature.

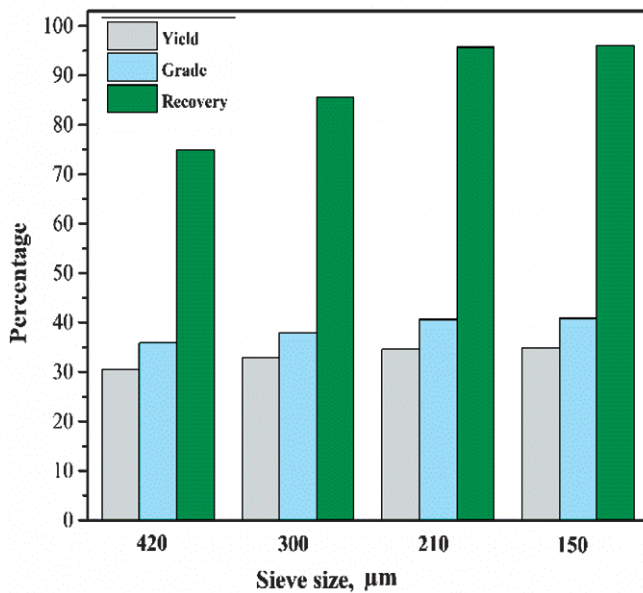


Figure 6: Results of rougher flotation for various MOG at pH: 8, Sodium silicate: 0.55 kg/tonne, Kerosene: 0.161 kg/tonne MIBC: 0.05 kg/tonne

However, its floatability can be improved by the addition of specific pH regulators. The interactions between collector and graphite or vice versa can be improved due to varying the concentration of the hydrogen and hydroxyl ions in the pulp with the help of a pH modifier. pH modifier is also used to reduce the interactions of the collector with the gangue minerals.²⁰ The effect of pH was studied in rougher flotation for an optimal MOG of 210 μm with the dosage of 0.55 kg/tonne sodium silicate, 0.161 kg/tonne kerosene, and 0.05 kg/tonne MIBC as shown in Figure 7. It is observed that the

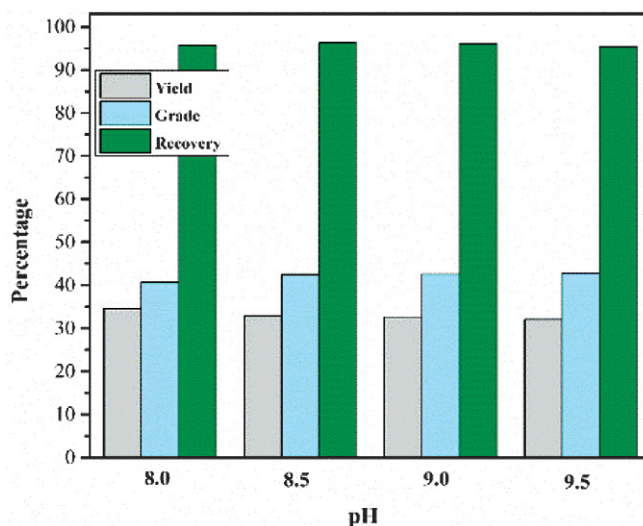


Figure 7: Results of rougher flotation for various pH at MOG: 210 μm , Sodium silicate: 0.55 kg/tonne Kerosene: 0.161 kg/tonne MIBC: 0.05 kg/tonne

grade of the rougher graphite concentrate increased significantly from 40.62% to 42.51% FC for pH 8 and 8.5, respectively. Beyond pH 8.5, the improvement in the rougher graphite concentrate grade was very minimal. Hence, the optimal pH value was determined as 8.5.

3.2.3 Effect of Sodium Silicate Dosage

The effect of sodium silicate dosage was studied for the rougher flotation of graphite ore at optimal MOG (210 μm), pH (8.5), and the dosage of 0.161 kg/tonne Kerosene, and 0.05 kg/tonne MIBC. Most of the silicate minerals were depressed in the rougher flotation of graphite ore. The grade of rougher graphite concentrate increased significantly from 42.51% to 44.84% FC by varying the dosage of sodium silicate from 0.55 to 0.75 kg/tonne, respectively, as shown in Figure 8. It is also observed that there is not much improvement in the graphite grade with the addition of more than 0.75 kg/tonne of sodium silicate. The quantity of sodium silicate (i.e., 0.75 kg/tonne) could be sufficient for the maximum rejection of silicate gangue minerals into rougher tailings. Hence, the optimal dosage of sodium silicate was determined as 0.75 kg/tonne.

3.2.4 Effect of Kerosene Dosage

The effect of kerosene dosage was studied for the rougher flotation of graphite ore at optimal MOG (210 μm), pH (8.5), and sodium silicate (0.75 kg/tonne) with the dosage of 0.05 kg/tonne MIBC. The rougher graphite concentrate grade was increased significantly from 42.86% to 44.84% FC with a dosage of kerosene from 0.115 to 0.161 kg/tonne, respectively as shown in Figure 9. The grade of graphite is significantly reduced at 0.184 kg/tonne of kerosene. It could be due to reduced selectivity at this dosage resulting in the flotation of gangue minerals, interlocked particles, and very

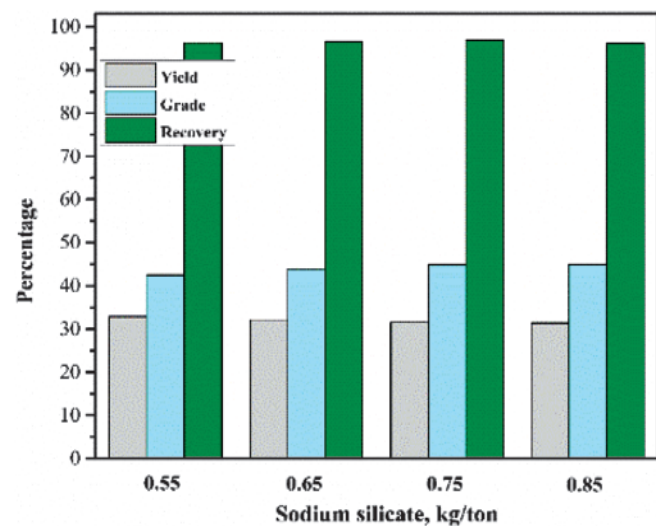


Figure 8: Results of rougher flotation for various dosages of Sodium silicate at constant MOG: 210 μm , pH: 8.5 Kerosene: 0.161 kg/tonne MIBC: 0.05 kg/tonne

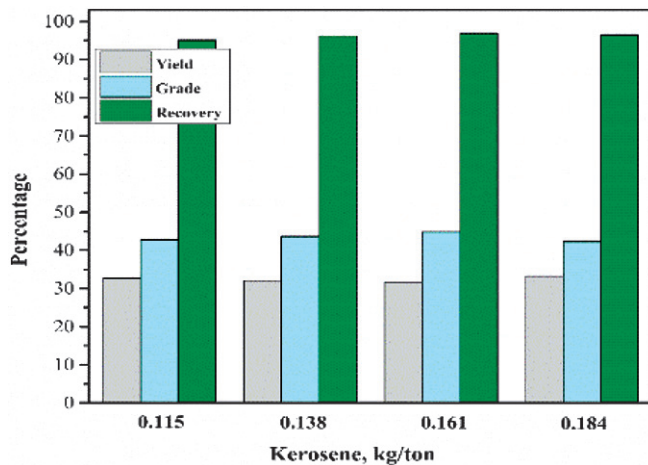


Figure 9: Results of rougher flotation for various dosages of Kerosene at MOG: 210µm, pH: 8.5 Sodium silicate: 0.75 kg/tonne MIBC: 0.05 kg/tonne

fine and slimy unwanted minerals that are floated along with rougher graphite float, thus diluting its grade. Hence, the optimal dosage of kerosene was determined as 0.161 kg/tonne.

3.2.5 Effect of Methyl Isobutyl Carbinol (MIBC) Dosage

MIBC is used as a frother and plays a vital role in graphite flotation. The rougher flotation test work was carried out on the graphite ore sample at optimal MOG (210 µm), pH (8.5), sodium silicate (0.75 kg/tonne), and kerosene (0.161 kg/tonne). The graphite rougher flotation results revealed that the grade of rougher graphite concentrate was increased significantly from 43.09% to 46.01% FC with the dosage of MIBC from 0.05 kg/tonne to 0.1 kg/tonne, respectively, as shown in Figure 10. The grade of rougher graphite concentrate was reduced

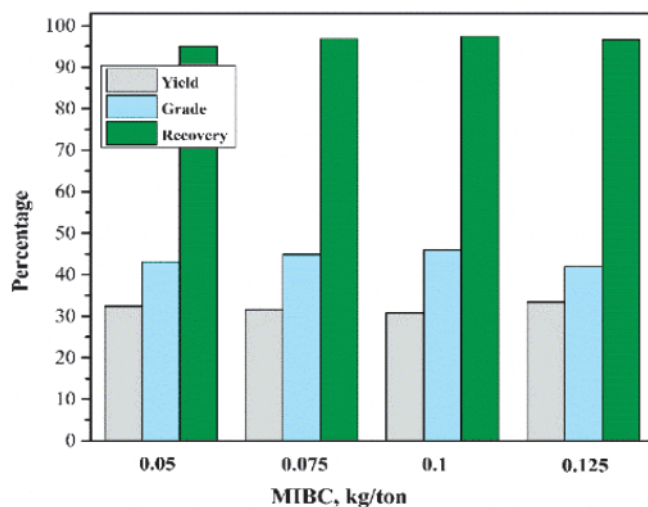


Figure 10: Results of rougher flotation for various dosages of MIBC at MOG: 210 µm, pH: 8.5, Kerosene: 0.161 kg/tonne, Sodium silicate: 0.75 kg/tonne.

significantly with an increased dosage of MIBC beyond 0.1 kg/tonne. It could be due to the physical entrainment of gangue minerals along with the graphite mineral into the float fraction²². Hence, the optimal dosage of MIBC was determined as 0.1 kg/tonne.

3.3 Rod Milling followed by Rougher Flotation

3.3.1. Rougher Flotation of Mill Products

The rougher flotation test work was carried out on the rod mill products at optimal parameters to separate the graphite minerals effectively from gangue minerals and to obtain better-grade of graphite concentrate at a satisfactory level of FC recovery. The recovery of the coarse flake graphite fraction in the rougher float is improved significantly from 36.80% to 43.74% with the rougher flotation carried out rod mill grinding with a combination of rods and cylindrical metallic pebbles (weight ratio of 1:1) and rods as grinding media respectively as shown in Figure 11. It is also observed that the coarse flake graphite size is protected considerably and effectively recovered by applying optimum flotation parameters during the rougher flotation stage than the other two types of grinding media. The recovery of fine flake graphite fraction in the rougher float is more in, the rougher flotation of rod mill grinding with a combination of rods and cylindrical metallic pebbles, and cylindrical metallic pebbles compared to rods as grinding media. It may be due to increasing the volume of grinding zones 11, and some portion of the coarse flake graphite was also ground along with other gangue minerals, which pass through a minus 150 µm sieve size. Rougher flotation product's assay characteristics for rod mill grinding with different grinding media are shown in Figure 11.

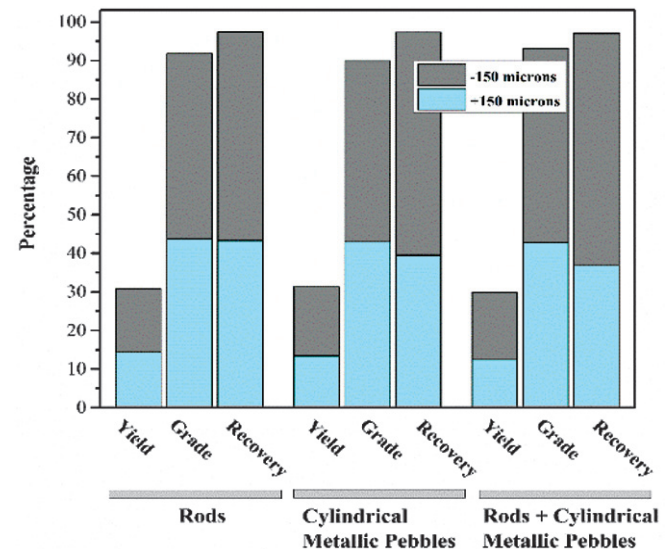


Figure 11: Results of rougher flotation of Rod mill with different grinding media

4.0 Conclusions

This work focused on studying various grinding media such as rods, cylindrical metallic pebbles, and a combination of rods and cylindrical metallic pebbles in the rod mill to protect the coarse flake graphite during the grinding stage and recover effectively during the rougher flotation stage. Various process parameters such as MOG, pH, the dosage of sodium silicate, kerosene, and MIBC were studied and optimized. The rougher flotation of mill products was performed at these optimal parameters. The wet sieve analysis and proximate analysis were also carried out. The proximate analysis of the representative sample was 14.5% FC, 71.82% ash, and 13.68% volatile matter + moisture content. The rod mill using rods as grinding media protected the coarse flake graphite significantly more than the other two grinding media. The amount of coarse flake graphite (i.e., plus 150 μm) more as compared to using cylindrical metallic pebbles, and the combination of rods and cylindrical metallic pebbles in the weight ratio of 1:1. The optimal parameters were determined as 210 μm (MOG), 8.5 (pH), 0.75 kg/tonne (sodium silicate), 0.161 kg/tonne (kerosene), 0.1 kg/tonne (MIBC). The liberated coarse flake graphite minerals in the graphite ore sample were recovered during the rougher flotation conducted on these rod mill products with different grinding media at optimal parameters. A better grade (43.74% FC) and recovery (43.3%) of plus 150 μm rougher coarse flake graphite concentrate were obtained using rods as grinding media in a rod mill grinding followed by rougher flotation of rod mill products compared to the other two grinding media.

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