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Upgrading graphite quality from Chenjere-Ruangwa ore through froth flotation techniques

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Abstract

Tanzania is endowed with graphite deposits found in Mahenge-Morogoro, Bunyu-Mtwara, Nachu and Chenjere-Ruangwa. In Chenjere, the graphite resource is approximately 500 million metric tons with 7.75% of total graphitic carbon grade (TGC) content. The demand in the global market needs a high-grade graphite of at least 90% TGC content. However, the graphite found in Chenjere is of low grade (7.75%). The present study aims to upgrade Chenjere's graphite to meet global market standards. To attain this objective, froth flotation was selected as an upgrading method. The sample was collected at Chenjere-Ruangwa, well prepared, and later upgraded through flotation. Flotation experiments were designed using the Box Behnken design, with controlled variables being particle size, collector, and frother concentrations while the TGC was the response. The analysis of variance (ANOVA) was utilized to assess the relationship between the experimental factors and model response. The results revealed that the graphite from Chenjere can be upgraded to > 90% TGC content under the optimal experimental conditions of -75 µm particle size, 990 g/t kerosene as a collector, and 150 g/t methyl isobutyl carbinol (MIBC) as a frother, hence meeting the required standard. In addition, the total graphitic recovery of 84% was obtained as the maximum recovery. In light of the potential benefits of graphite in advancing industrialization, this study strongly recommends using froth flotation technology to upgrade the graphite ore from Chenjere to meet the global market specifications in terms of quality.

Keywords: *Chenjere-Ruangwa; Graphite quality; Froth-flotation; Total graphitic carbon grade; Upgrading*

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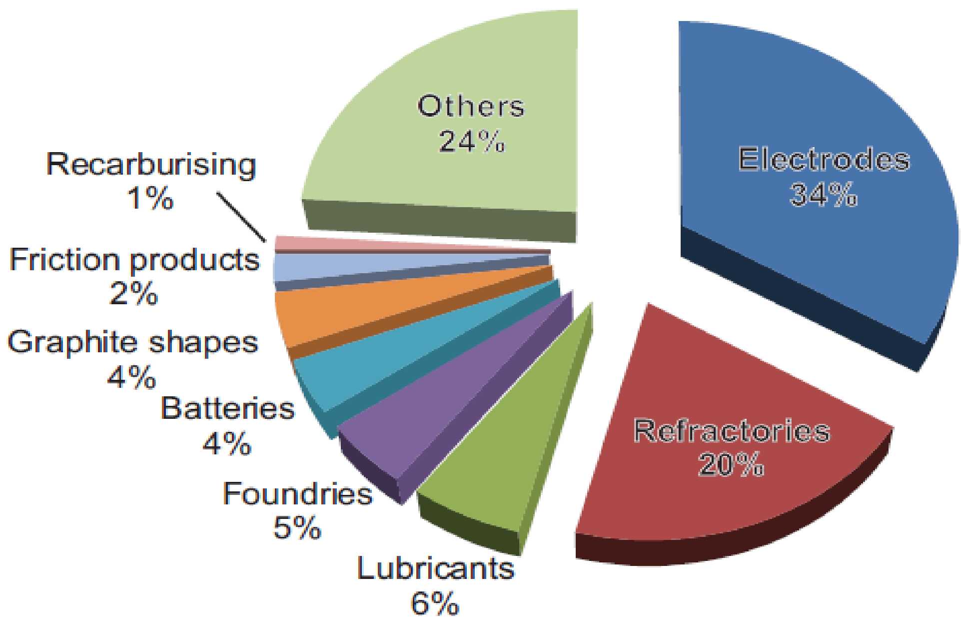
Introduction

Graphite, a naturally occurring crystalline form of carbon, is typically greenish-black in colour and has a relatively soft and greasy texture, with a hardness rating of 0.5–1.0 on the Mohs scale (Hoffmann *et al.*, 2016; Barma *et al.*, 2019). It is commonly found in three main commercial varieties based on its mode of occurrence and origin. These varieties include crystalline flake graphite, crystalline (lumpy) graphite, and microcrystalline (amorphous) graphite (Öney and Samanli, 2016; Jin et al., 2018; Vasumathi et al., 2023). Natural graphite possesses a unique combination of properties, some resembling metals (such as thermal and electrical conductivity) and others characteristic of non-metals (including inertness, thermal resistance, and high lubricity). These dual characteristics make natural graphite highly versatile and suitable for various industrial applications. The characteristics of different forms of natural graphite, including their origin, ore grade, product grade, and main uses, were presented in studies by Keeling & John (2020) and Gautneb and his core authors (2023).

Natural graphite deposits are found worldwide. Mozambique holds the largest flake graphite reserves, approximately 1,420 tons, with a grade of 10%. The Chenjere deposit in Tanzania contains about 500 tons, with a grade of 7.8% (Simandl et al., 2015; Keeling & John, 2020; Case et al., 2023). Globally, most natural graphite is utilized in electrodes, refractories, lubricants, foundries, batteries, graphite shapes, recarburizing, steelmaking, and friction products like brake linings, as shown in Figure 1 (Keeling and John, 2020; Gautneb *et al.,* 2023).

Figure 1

Main Uses of Natural Graphite



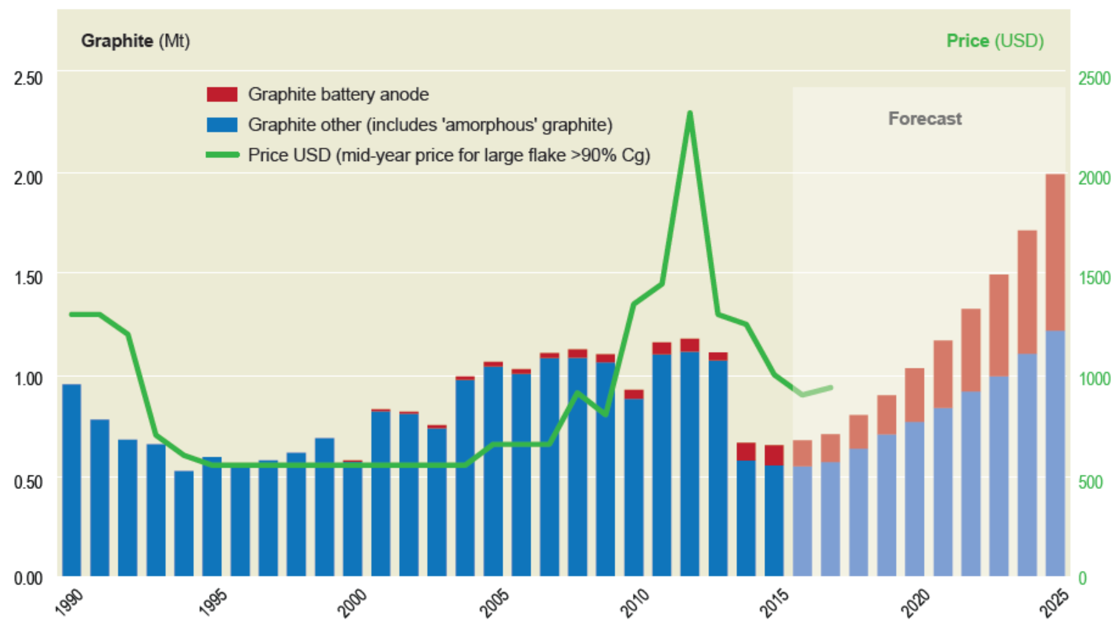
Demand for flake graphite in 2012 was under 400,000 tons per annum, but it is expected to increase significantly due to the growing production of lithium-ion batteries for portable devices, electric vehicles, and energy storage. The Historical and forecast demand for natural graphite (1990–2025) is shown in Figure 2. By 2040, the demand for battery graphite is expected to be 25 times higher than it was in 2020 (Simandl et al., 2015; Survey, 2019; Keeling and John, 2020; Gautneb et al., 2023).

The economic viability of graphite is determined by two crucial factors: the particle size of graphite (this dictates the liberation size) and the total graphitic carbon grade (TGC) content (Chelgani et al., 2016; Moye and Msabi, 2021; Vasumathi et al., 2023). These characteristics play a central role in determining the market price of graphite. International standards typically mandate that natural graphite should have a purity level of at least 90%. However, since finding natural graphite with purity exceeding 90% is relatively rare, there is a necessity to upgrade lower-grade graphite to meet the desired grade (usually >90%).

Froth flotation is a widely employed method for concentrating low-grade graphite ore due to its high natural hydrophobicity (Oney *et al.,* 2017). In the froth flotation process, graphite ores are often treated with a suitable hydrocarbon oil to change their hydrophobicity, improve recovery, and enhance selectivity. The amounts of the collector and the frother significantly impact the flotation performance (Vasumathi *et al.*, 2014; Oney *et al.*, 2017). In this process, hydrocarbons such as kerosene, fuel oil, paraffin, and diesel oil, or ionic collectors like potassium amyl xanthate and dithiophosphate are typically used as collectors. Pine oil and MIBC are used as frothers, while sodium silicate, quebracho, and starch are used to depress gangue minerals (Kaya and Canbazoglu, 2007; Öney and Samanli, 2016; Jin *et al.*, 2018; Qiu *et al.*, 2022).

Figure 2

Historical and Forecast Demand for Natural Graphite (1990–2025)



Note. Graphite used for battery anode is highlighted in red. Historical price trend for natural graphite, large flake >90% Cg, is shown in green.
Source. Simandl et al., 2015; Keeling & John, 2020; Mykhailov et al., 2021.

The Chenjere – Ruangwa graphite resource, situated in South-Eastern Tanzania, has a total of 500 million metric tons (District, 2010). The graphite mineralization consists of medium to fine-grained crystalline flake-type graphite, with individual flakes having a long axis size of up to 1000 micrometers (Moye and Msabi, 2021). This resource contains graphite with a total graphitic carbon (TGC) grade ranging from 3.03% to 7.75%, categorizing them as low-grade deposits (Thomas *et al.*, 2014; Leger *et al.*, 2015). To meet the demands of global markets, this ore needs to be upgraded to contain at least 90% TGC. Apart from a study conducted in 2021 by Moye and Msabi to investigate the Mineralogy and Geochemical Characteristics of Graphite-Bearing Rocks in Chenjere area, the authors did not find any other study conducted to upgrade this ore. Therefore, this study aimed to improve the quality of the graphite ore using the froth flotation method. The primary goal was to produce a concentrate with a TGC content of at least 90% while maintaining a graphitic recovery rate of 80% or higher.

Materials and Methods

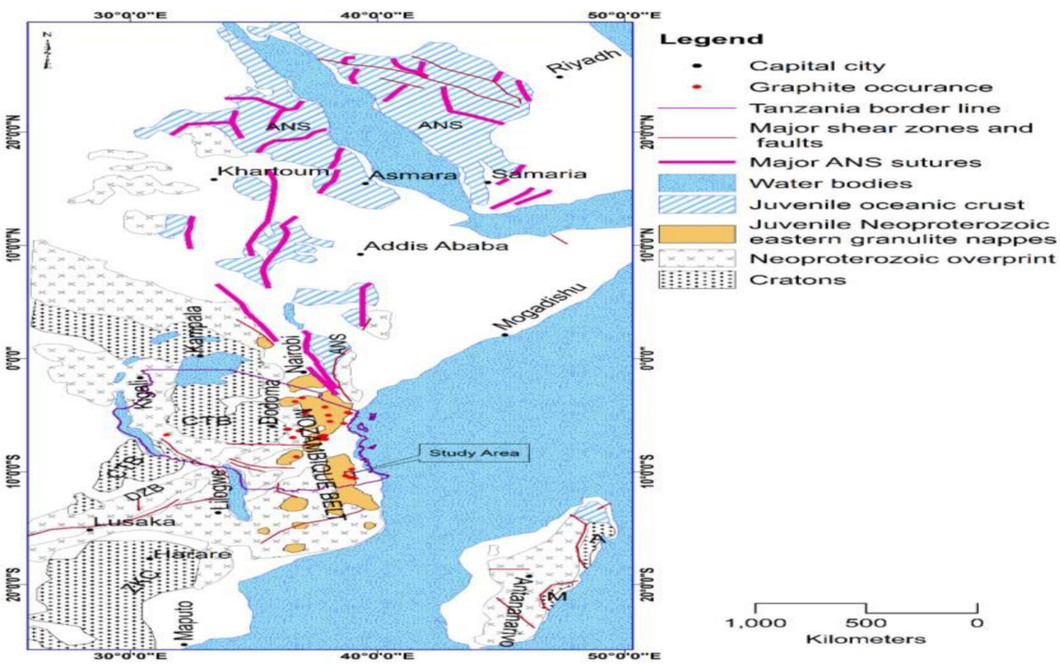
Sample Collection and Preparation

The representative samples used in this study were collected in the Chenjere area in Ruangwa district. This area is situated over metamorphic basements that have origins in both sedimentary and igneous processes within the Neoproterozoic Mozambique Mobile Belt (NMMB) of Tanzania.

This geological belt extends from the northern to the southern regions on the eastern side of the country (see Figure 3). The Chenjere area is located within the Eastern Granulite's domain, specifically excluding the Neoproterozoic "Eastern Granulite's" nappes (Thomas *et al*., 2014). This Eastern domain is characterized by a predominant presence of granulites, gneisses containing graphite, marbles, quartzites, and schist, as well as post-orogenic granites and pegmatites bearing gemstones (Moye and Msabi, 2021).

Figure 3

Distribution of Crustal Domains in the EAO (East African Orogen) and Graphite Occurrences in Tanzania



Note. CTB = Congo-Tanzania-Bangweulu Cratons; ZKC = Zimbabwe-Kalahari Cratons; A = Antogil Craton; M = Masora Craton; ANS = Arabian Nubian Shield and DZB = Damara-Zambezi Belt.
Source. Thomas et al. 2014, Leger et al. 2015; Moye and Msabi, 2021.

A total of 10 kg samples were collected from eight locations using a systematic sampling approach which is suitable for accounting all sample representativeness issues. The collected samples were homogenized, split using coning and quartering techniques, and sent to the Geological Survey of Tanzania (GST) Laboratory for preparation and analysis. A rotary sample splitter was used to divide the sample into two portions. The first portion was reserved for reference purposes, while the second one was used for various analyses. To prepare the sample for analysis, any debris was manually removed and dried in an oven at 105 degrees Celsius to remove moisture. Then, the sample was pulverized to 80% minus 75 microns using a jaw crusher and swimming mill machines. The sample was riffled again to obtain sub-samples for froth flotation test works, TGC, and mineralogical analysis.

Determination of Total Graphitic Grade Content

At GST, the TGC was analyzed using the loss on ignition (LOI) method. This involved heating and weighing the samples in an oven and muffle furnace. The pulverized samples were weighed using an electrical analytical balance with an accuracy of 0.0001 g to obtain 1 g. The sample was heated at 800 °C for seven minutes in the muffle furnace to remove volatile materials like sulphides and chlorites that may be present. After weighing the cooled sample, it was then heated at 900 °C for ignition for four hours. Replication was done to comply with GST quality assurance/quality control policy and the data presented were the means of the recorded values. Grades of graphite from the head, concentrate and tailing streams after flotation were determined in terms of percentages using Equation 1, as suggested by Heiri *et al.,* (2001).

$$TGC = \frac{W_{800} - W_{900}}{W_{800}} \times 100 \qquad \text{Equation (1)}$$

Where: W_{800} was weight at 800 °C, and W_{900} was weight at 900 °C.

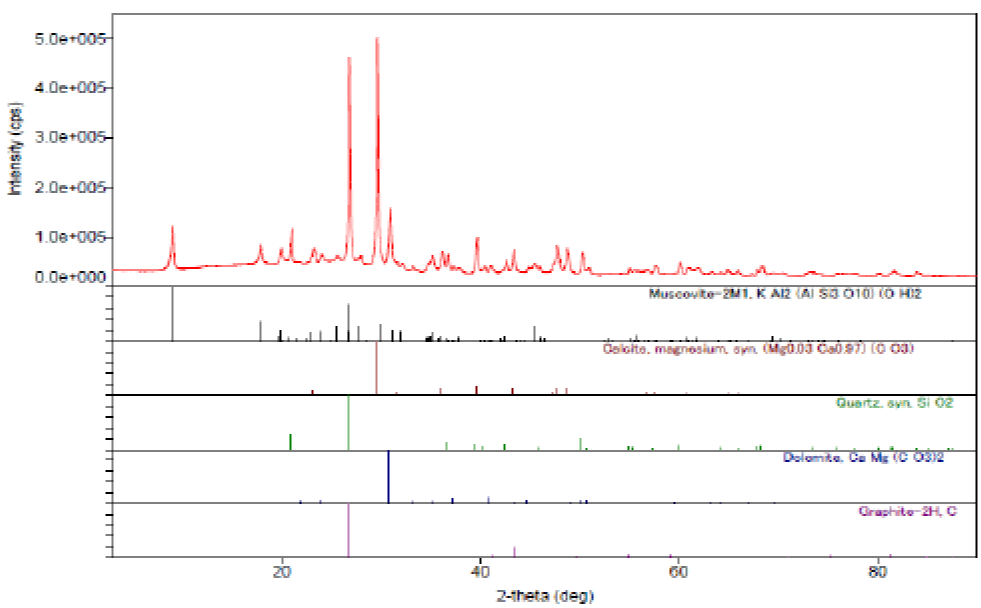
Analysis of Mineralogical Compositions

To determine the mineralogical phase of the graphite ore sample and identify any associated non-graphite minerals, X-ray diffraction (XRD) analysis was conducted. The analysis was carried out using a Bruker AXS D2PHASERA26-X1-A2B0D2C- with a copper anode as the x-ray source (CuKα 1.54060Å), operating at 30.0 kV and 10.0 mA.

The XRD analysis results indicated quartz as the predominant mineral (about 81%) with minor amounts of graphite (8%), muscovite (6%), and K-feldspar (3%). Biotite, calcite, and magnesite calcite were also present in trace amounts (less than 1% each). Most of the graphite was crystalline flake graphite in nature, usually present in close association with the silicate gangue. A diffractogram illustrating these findings is shown in Figure 4. The head grade of the investigated sample was found to contain 7.75% of total graphitic grade content.

Figure 4

XRD Profile of Chenjere Graphite Ore



Froth Flotation Test Works to Upgrade the Ore

The main objective of carrying out flotation experiments was to upgrade the ore to have a concentrate with over 90% TGC and a graphitic recovery of at least 80%. Various parameters were analyzed, including particle size (ranging from 212 to 75 microns), collector concentration (between 500 and 1000 g/t kerosene), and frother concentration (between 100 and 300 g/t MIBC). To maintain consistency, we kept other flotation parameters constant, such as pulp density (12% solids), pH (8), impeller rotation speed (1400 rpm), and depressant concentration (1200 g/t sodium silicate). The depresent helps to improve the product selectivity by depressing the gangue minerals and allowing only graphite to collect in the concrentrate product. The experimental parameter levels were established based on the findings from previous studies (Chelgani *et al.,* 2016; Öney and Samanli, 2016; Oney *et al.,* 2017; Barma *et al.,* 2019; Jara *et al.,* 2019; Qiu *et al.,* 2022).

Design of Experiments

This study employed the Response Surface Methodology (RSM) technique to assess the impact of various independent variables on the TGC response. RSM was chosen as the preferred method due to its effectiveness in identifying new operational conditions that could lead to demonstrated process improvements (Radojković *et al.,* 2012).

To design the experiments, a Box-Behnken Design (BBD) with three levels (as outlined in Table 3) was utilized. The choice of the Box-Behnken design was motivated by its ability to ensure that all experimental points fall within a safe operating zone, as recommended by Oney *et al.,* (2017). The experimental design and subsequent statistical analyses were carried out using Design-Expert (DX) software, specifically version 13.0.0 by Stat-Ease, Inc. Some experiments were replicated in accordance with the GST quality assurance/quality control policy.

For predicting the response, a regression model was developed. The suitability of the developed regression model was assessed using the coefficient of determination (R^2). Additionally, an Analysis of Variance (ANOVA) test was conducted to establish the relationship between the independent variables and the response. The optimal conditions were determined using Design-Expert software (Demo v.13.0.0, Stat-Ease, Inc.), and three-dimensional response graphs were generated to enhance understanding of the results.

Flotation Procedure

Flotation experiments were carried out using a mechanical flotation cell, Denver laboratory type. About 200 g of a representative sample (80% passing through 75 μm) was fed to the flotation cell, and water was added to maintain a pulp density of 12% solids. All experiments were conducted at a pH of 8. The impeller speed of the flotation machine was kept constant at 1400 rpm for both conditioning and flotation. The suitable flotation conditions were maintained by adding flotation reagents as per Table 3. The pulp was mixed for three minutes prior to the addition of kerosene and sodium silicate. After an additional mixing of three minutes, the MIBC was added. Following a three-minute mixing period, air was introduced into the cell at the aeration rate of 0.2 m^3/h, and the froth products were collected for five minutes. The concentrate obtained was then, filtered in a vacuum filter, dried in an air oven, and then subjected to LOI analysis for determining the concentrate and tailing's TGC. Finally, the TGR was computed using Equation 2.

$$\text{TGR} = \frac{(f-t)}{(c-t)}\left(\frac{c}{f}\right)\times 100 \qquad \text{Equation (2)}$$

Where,

TGR	Total graphitic recovery (%)
f	TGC, head
c	TGC, concentrate (froth)
t	TGC, tailing

Results

Regression Model and Statistical Evaluation of the Experimental Test Results

Table 3 presents the experimental conditions and the resulting concentrate TGC (%) content for all experiments. The highest concentration with a purity of 95.4% TGC was achieved during the 5th run, utilizing the following conditions: -75 µm particle size, 1000 g/t kerosene, and 200 g/t MIBC.

Table 1

Experimental Design Layout with Actual Levels of Parameters and the Upgraded Concentrates

Run	Particle Size (µm)	Kerosene (g/t)	MIBC (g/t)	Concentrate TGC (%)
1	75	750	100	94.0
2	150	1000	200	85.8
3	75	500	200	90.5
4	150	500	200	78.3
5	75	1000	200	95.4
6	112.5	500	300	89.7
7	112.5	1000	300	92.8
8	112.5	750	200	87.4
9	112.5	750	200	87.7
10	112.5	750	200	88.1
11	112.5	500	100	85.8
12	150	750	100	79.3
13	75	750	300	92.1
14	150	750	300	83.7
15	112.5	1000	100	90.1

Table 2

Analysis of Variance (ANOVA) of the Quadratic Regression Model

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Model	336.98	9	37.44	33.41	0.0006
A (Particle size)	251.66	1	251.66	224.53	< 0.0001
B (Kerosene)	49.35	1	49.35	44.03	0.0012
C (MIBC)	10.53	1	10.53	9.40	0.0279
Residual	5.60	5	1.12		
Cor Total	654.12	14			

Discussions

Regression Model and Analysis of Variance

Using the results for the TGC (%) as the model response, a second order regression model with a coefficient of multiple determinations of (R^2) 98.4% (Eq. 3) was established. The statistical analysis results indicated a strong fit of the model. According to Joglekar and May (1987), a model is considered to have a good fit when the R^2 value is at least 0.80. In this case, the R^2 value for the concentrate TGC was 0.984, suggesting that the model explains 99% of the variation in the studied response, demonstrating a comprehensive explanation of the response by the model. Furthermore, the adjusted R^2 (0.954) and predicted R^2 (0.8920) values also produced satisfactory results. The f-value of the TGC of the concentrate was found to be 33.41. The lack-of-fit values of 14.48 resulted in no significant F-values for the response variables. By using the established model (Eq. 3), an unknown response can be calculated at any coded levels of particle size (A), kerosene (B) and MIBC (C). Based on the model, the response surface plots for interaction effects of two parameters at a fixed level of third parameter were generated as shown in Figure 7.

$$TGC = +87.73 - 5.61\,A + 2.48\,B + 1.15\,C + 0.645\,AB + 1.56\,AC - 1.29\,A^2 + 1.06\,B^2 + 0.816\,C^2 \quad \text{Equation (3)}$$

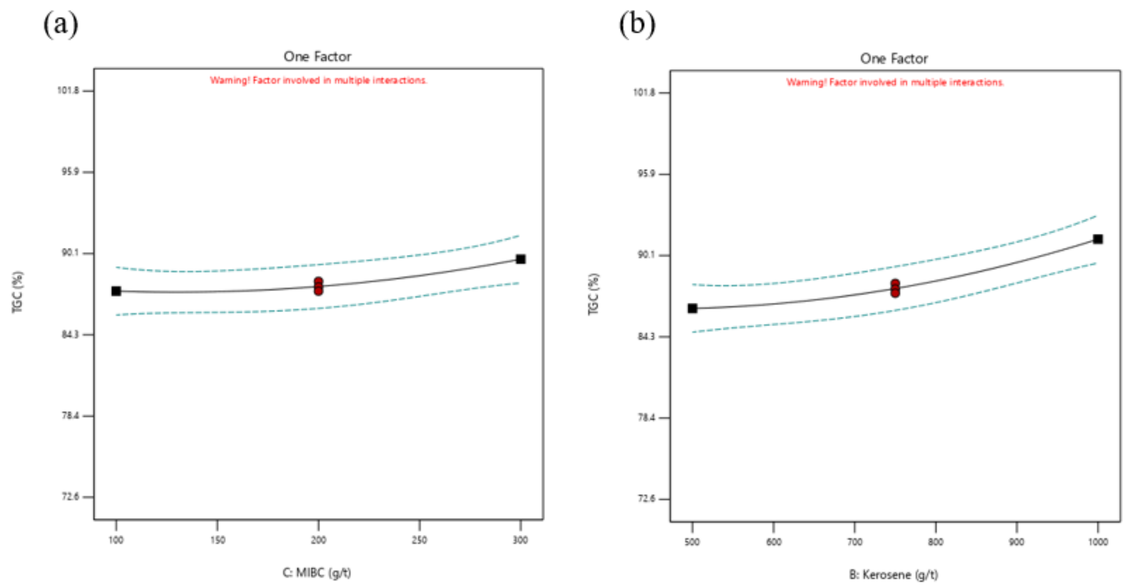
To determine the linear/quadratic/interaction effects of parameters on the response, the analysis of variance (ANOVA) was used (Table 4). The regression model is determined to be statistically significant at 95% confidence level. The effects of the studied parameters on the model response were determined to be statistically significant at a 95% confidence level. This indicates that the (TGC) content in the graphite concentrate is indeed influenced by the particle size, kerosene, and frother concentrations across the investigated levels as further explained in section 4.2.

Effects of Experimental Parameters on the Response

In Figure 5(a), the impact of MIBC concentration on the TGC content of the concentrate is illustrated under the conditions of a 112.5 µm particle size and 750 g/t kerosene. The Figure reveals an increase in TGC content as the MIBC concentration rises. This trend is consistent with the widely recognized role of frothers like MIBC in predominantly influencing flotation responses. Previous research has indicated that the maximum effective concentration of MIBC typically hovers around 300 g/t, beyond which the impact on the process becomes negligible, as documented in studies by Chelgani *et al.,* (2016) and Vasumathi *et al.,* (2023). Frothers, including MIBC, play a crucial role in reducing surface tension at the air-liquid interface, facilitating the formation of stable bubbles within the system. Furthermore, they exert influence over the kinetics of adhesion between bubbles and particles, contributing to the stability of bubble-particle aggregates.

Figure 5

Effects of (A) MIBC and (B) Kerosene Concentrations on the Concentrate TGC Content

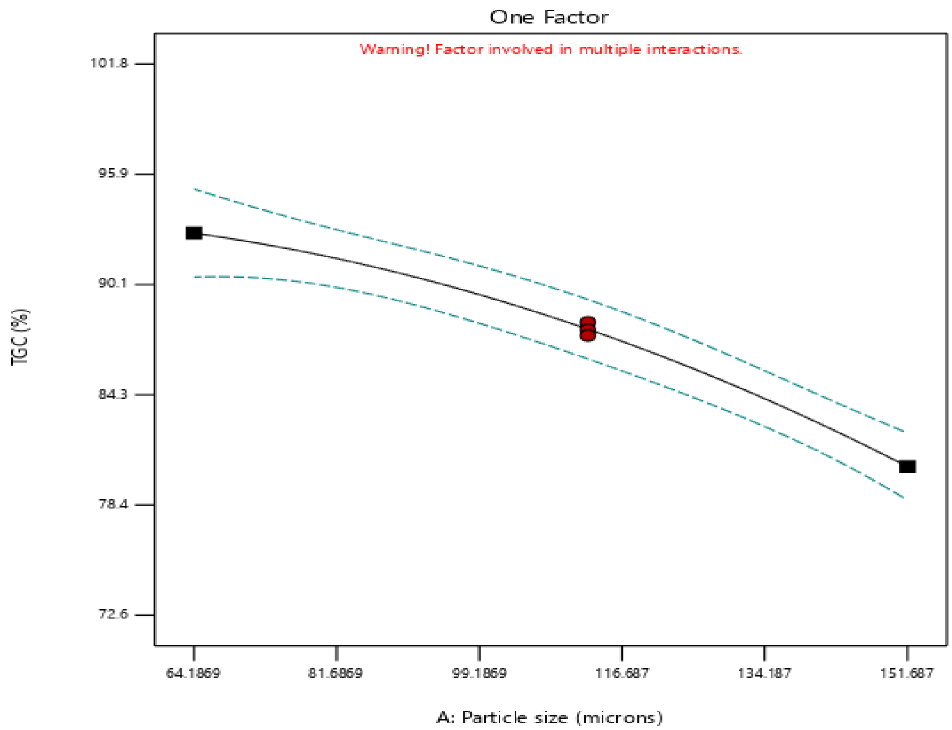


Similarly, the impact of kerosene concentration on the TGC content demonstrated a clear linear relationship, as depicted in Figure 5(b). The TGC content exhibited a consistent increase with higher kerosene concentrations. Various studies have highlighted an optimal kerosene concentration of 1400 g/t in graphite flotation processes, as documented by Hoffmann et al. (2016), Öney and Samanli (2016), and Barma et al. (2019). Collectors, such as kerosene, typically enhance the selectivity of separating graphite from associated gangue minerals like mica, quartz, feldspar, and carbonate.

Moreover, the particle size of the material emerged as a critical factor in determining the purity of the concentrate, which is corroborated by previous research findings (Jin *et al.,* 2018; Jara *et al.,* 2019). Figure 6 provides supporting evidence for this assertion, indicating that as particle size decreases, the TGC content increases. Generally, graphite liberation is achieved at particle sizes of 80% pass 75 microns, underscoring the significance of finer particle sizes in obtaining higher-purity graphite concentrates.

Figure 6

Effects of Particle Size on the Concentrate TGC Content

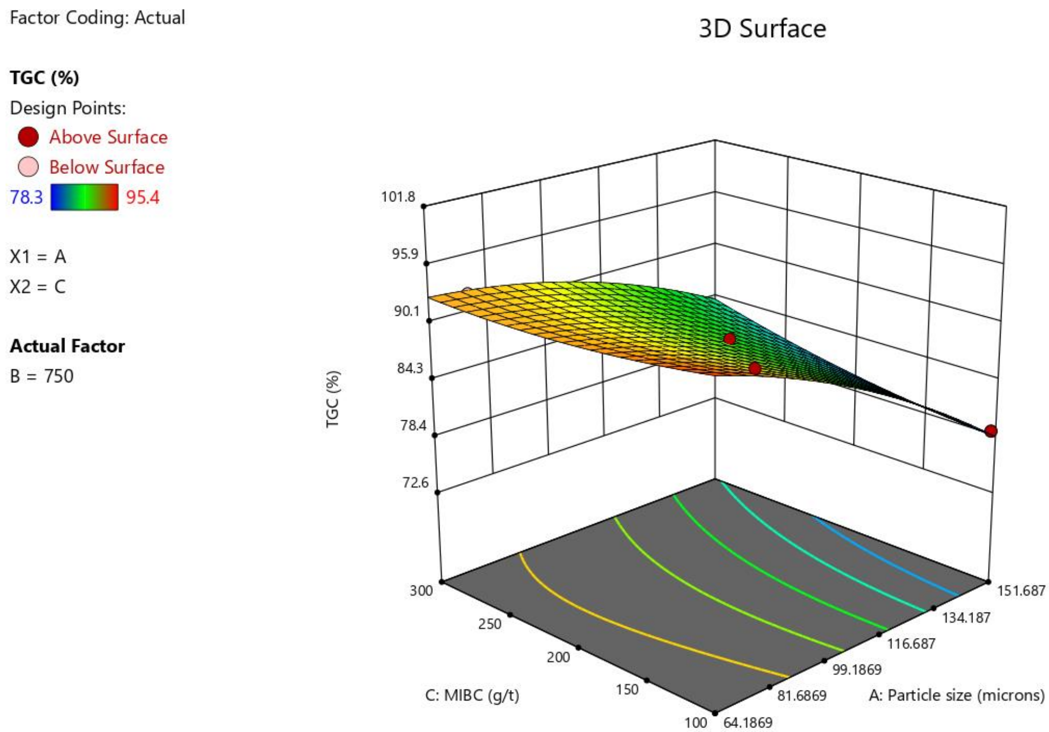


Interaction Effects of the Experimental Factors to the Model Response

Interaction effects of experimental factors (particle size, kerosene, and MIBC) on the TGC content are clearly presented in terms of response surface plots (Figure 7). The interaction effects of particle size and MIBC on the TGC content were found to be statistically significant at a 95% confidence level. The TGC quality appears to increase with the respective increase of MIBC concentration and decrease in particle size at the fixed level (750 g/t) of kerosene. This is apparently attributed to the fact that MIBC as the frother is a crucial factor as it plays a crucial role in reducing surface tension at the air-liquid interface, facilitating the formation of stable bubbles within the system and exert influence over the kinetics of adhesion between bubbles and particles, contributing to the stability of bubble-particle aggregates (Chelgani *et al.*, 2016; Vasumathi et al. 2023). Furthermore, the decrease in particle size means the increase in the degree of liberation and thus obtaining higher-purity graphite concentrates. The graphite mineralization at Chenjere consists of medium to fine-grained crystalline flake-type graphite. Therefore, fine grinding, mostly at 80% pass 75 microns is required to liberate the graphite from the associated gangue (Jin *et al.*, 2018; Jara *et al.*, 2019). On the other hand, the interaction effects of particle size and kerosene, MIBC and kerosene levels to the TGC purity were found to be statistically not significant.

Figure 7

Response surface plot versus varying levels of MIBC and particle size at 750 g/t kerosene



Response Optimization

Normally, response optimization is done to establish the optimum levels of the experimental parameters relative to the experimental response. The response optimizer used the fitted quadratic model (Equation 3) at 95% confidence level for optimization. The concentrate TGC content maximization was the main target whereby the optimal TGC of 93% was predicted at the optimum levels of -75 µm, 1000 g/t kerosene, and 200 g/t MIBC. The total graphitic recovery (TGR) was computed using Equation 2, where the optimized value of the concentrate grade (93 TGC) was used. The other inputs to the equation were TGC, head (7.75), and TGC, tailing (1.28). The TGR was found to be 84.6%.

Potential Limitations of the Present Study Results. The main findings of this study such as the upgrading technique (froth flotation) and its parameter levels might be applied to upgrade the other graphite deposits if and if the deposits share the same geological settings. The mineralogical and chemical compositions of the ore have the great influence on the upgrading efficiency (Gautneb *et al.*, 2023). Therefore, the current study's findings can be applicable to other graphite deposits if those deposits have the same ore compositions as the Chenjere's graphite ore.

The primary focus of the present study was on laboratory-scale froth flotation tests to assess the efficiency of flotation technique on upgrading the

Chenjere's graphite ore. The achievement of study would be the motives for focusing on the process economic analysis and scaling up to commercial production scale. Therefore, process scaling up and economic analysis are not part of the current study, rather they have been left as the next potential studies.

Conclusion

The purpose of this study was to upgrade the low-grade graphite ore from Chenjere area to a concentrate with at least 90% TGC and a total graphitic recovery of over 80%. The graphite ore under investigation had a head grade of 7.75% TGC and was associated with minerals such as quartz, muscovite, k-feldspar, biotite, calcite, and magnesite calcite as gangue minerals.

The optimal TGC content of the concentrate was projected to be 93%, which was achieved through the following experimental conditions: particle size of -75 µm, kerosene of 990 g/t, MIBC of 150 g/t, sodium silicate of 1200 g/t, pH of 8, and pulp density of 12% solids. The maximum graphitic recovery was found to be 84%.

Based on the study's most favourable results, it is highly recommended to employ froth flotation technology for upgrading Chenjere graphite ore to align with the quality standards demanded by the global market.

Recommendations

For upgrading the graphite ore from Chenjere so as to meet the global market requirements of at least 90% TGC and 80% TGR, the froth flotation technique is recommended to be used with the following flotation parameter levels: particle size of -75 µm, kerosene of 990 g/t, MIBC of 150 g/t, sodium silicate of 1200 g/t, pH of 8, and pulp density of 12% solids.

As the primary focus of the present study was on laboratory-scale froth flotation tests, and the promising outcome were obtained, a study focusing on the scaling up the process to a commercial production level is recommended to be done. Here factors such as process optimization, equipment selection, and operational considerations have to be addressed.

A study focusing on the detailed economic analysis for the froth flotation upgrading technique is recommended to be conducted. Here, factors such as operating costs, energy consumption, and market demand should be considered to assess the economic feasibility of implementing this process on a large scale

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