

Research Article

Upgradation of Chimiari Black Shale Khyber Pakhtunkhwa Region of Pakistan by Froth Floatation

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Abstract

Extensive reserves of black shale in Pakistan are enriched with economically important metals such as vanadium (V), titanium (Ti), manganese (Mn), zinc (Zn), rubidium (Rb), and copper (Cu), alongside an appreciable content of organic carbon (OC). This study investigates the effectiveness of flotation techniques in upgrading the content of these metals and OC, while simultaneously examining the behavior of impurities. Two sets of flotation experiments were conducted utilizing kerosene oil as a collector and sodium hydroxide (NaOH) to control pH. Pine oil was introduced as a frothing agent in Experiment-II. The results demonstrate a significant increase in the concentration of total organic carbon (TOC) and TiO_2 . Experiment-I achieved enrichment ratios of 1.44 for TOC and 1.57 for TiO_2 , while Experiment-II achieved factors of 1.50 and 1.09, respectively. The flotation process selectively removed impurities such as SiO_2 , SO_3 , CaO, and Fe_2O_3 into the tailing's fractions of both experiments. Additionally, X-ray fluorescence (XRF) analysis revealed the successful upgrading of ZnO, CuO, and Rb_2O , which were detected in either the tailings or concentrate but not in the feed samples. This research highlights the promise of optimized flotation processes for enhancing the value of black shale deposits, providing a foundation for further refinement and industrial application.

Keywords

Black Shale, Chimiari, Froth Floatation, Recovery, Upgradation

1. Introduction

Extensive black shale deposits in Pakistan represent a significant economic opportunity due to their enrichment in several valuable metals. These metals include copper (Cu), zinc (Zn), rhenium (Re), germanium (Ge), gold (Au), palladium (Pd), and platinum (Pt). Additionally, these shales hold promise 2. as unconventional reservoirs for gaseous hydrocarbons [1, 2]. On a global scale, black shales play a critical role in hosting a wide variety of metallic mineral resources. These resources encompass precious metals (Au, Ag, Platinum Group

Elements), transitional metals (Mo, Cu, Ni, Cr, V, Zn), and the radioactive element uranium (U) [3, 4]. These deposits are critical to the global natural fuel-resource economy [5].

The Chamiari black shale deposit, located within the Khyber Pakhtunkhwa region of Pakistan, represents a multifaceted orebody. This deposit is characterized by notable enrichments in several economically valuable metals, including vanadium (V), titanium (Ti), manganese (Mn), copper (Cu), and uranium (U). [6]. It is well-documented that metals in black shales are

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often bound within stable organic complexes, such as metal-lopophyrin [3] with metal-bearing compounds dispersed as fine particles within clay fractions or slimes [7]. Historically, the extraction of metals from black shales and sedimentary ores has been documented in several regions, including Sweden, Estonia, Canada, and Central Asia. A notable example of such operations are the Ronneburg Gera mines in Germany. [8-11].

Sieve size analysis of Chamiari black shale indicates a broad distribution of minerals across all fractions [6]. The upgrading of black shale ores with high organic content presents a unique challenge due to the presence of clay minerals and disseminated metal grains. Efficient recovery of metals necessitates the disaggregation of this clay matrix. Globally, researchers have explored various methodologies for upgrading and metal recovery from black shale deposits. Sulfuric acid leaching has demonstrated success in the collective recovery of nickel (Ni), zinc (Zn), and vanadium (V), while conventional carbon-in-leach cyanidation offers a viable option for gold (Au) recovery.

Furthermore, gravity concentration techniques have been successfully applied to specifically target and concentrate heavy minerals and metals from black shale deposits in the Birch Mountains, Athabasca Region, Alberta, Canada [12, 13]. In Guizhou Province, China, researchers achieved vanadium recovery from black shale through a three-step process involving leaching with a mixture of sulfuric acid (H_2SO_4), hydrofluoric acid (HF), and sodium chlorate ($NaClO$) at atmospheric pressure [14]. Bioleaching has also been investigated for the Chamiari black shale; however, this method resulted in slow recovery rates for zinc (Zn) and limited recoveries for cobalt (Co), zinc (Zn), and copper (Cu) [15].

Froth flotation represents a widely employed and versatile technique for the separation, collection, and concentration of valuable minerals from their associated gangue materials. This approach leverages the differences in surface properties between the desired minerals and the gangue. In direct flotation, the target minerals selectively adhere to air bubbles and are carried to the froth layer at the top of the flotation cell, effectively separating them from the gangue material that remains suspended in the pulp and ultimately reports to the tailings. [16].

This study aims to systematically evaluate the physicochemical response of the Chamiari polymetallic black shale ore to flotation, targeting the production of a metal-rich concentrate enriched in vanadium (V), titanium (Ti), manganese (Mn), copper (Cu), and carbon (C). Effective froth flotation relies heavily on the surface properties of minerals. Therefore, this research investigates the modification of these properties to achieve optimal separation of the valuable components

from the gangue minerals [1]. In this research, kerosene oil was employed as a collector for carbon content [17, 18] and pine oil is a frother to get persistent, stable and best froth in float [19]. The initial experiment utilized only kerosene oil, while the subsequent experiment incorporated pine oil as a frother. NaOH was consistently used as a pH controller in both experiments. The effects of the collector, frother, and pH controller (NaOH) were meticulously monitored throughout the experimental process, and comprehensive data was collected to inform downstream processing strategies for the recovery of carbon (C), vanadium (V), titanium (Ti), copper (Cu), zinc (Zn), and rubidium (Rb).

2. Materials and Methods

2.1. Materials

A representative sample of 3.5 kg was meticulously collected from the Chamiari black shale deposit site. The sample was then transported to the mineral processing laboratory at the Department of Mining Engineering, University of Engineering and Technology Lahore, for further processing and analysis. This sample was designated for a series of flotation experiments aimed at evaluating the physicochemical response and potential for metal and carbon recovery.

Particle size analysis was performed using a Tyler Series sieve set for precise grading. A Denver Laboratory Flotation Machine, Model 533000, was employed for the froth flotation experiments. A JENWAY pH meter (Model No. 3305) was used for continuous monitoring of the slurry pH.

The flotation process employed kerosene oil (local supplier) as the collector and pine oil (local supplier) as the frothing agent. Analytical grade sodium hydroxide (NaOH) from Fluka, UK, was used for pH control.

The chemical composition of both the flotation concentrates and tailings was determined through X-Ray Fluorescence (XRF) analysis. This technique provided precise and reliable quantitative data on the elemental composition of the samples, facilitating subsequent evaluation and interpretation.

2.2. Methodology

The initially crushed black shale sample (3.5 kg) consisted of sub-samples with a size range of 1.25 to 0.5 inches (31.75 mm to 12.7 mm). These sub-samples were subsequently size-reduced in a Denver Laboratory Batch Ball Mill (cylindrical, stainless-steel lined) to achieve a final particle size of less than 212 μm (equivalent to 95 wt.% of the initial sample weight).

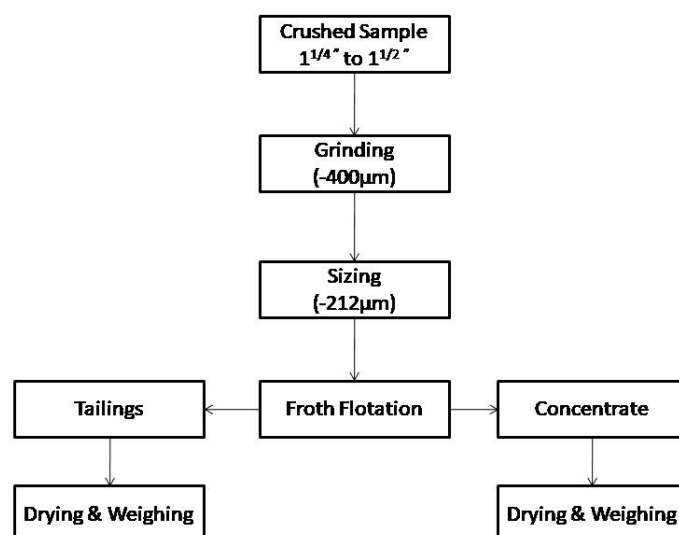


Figure 1. Flowsheet of flotation experiment performed to upgrade the Chimiari Black shale (experiment 1).

A slurry with a pulp density of 25 wt.% was prepared by mixing 200 g of the ground black shale sample with 600 mL of fresh water. The initial pH of the slurry was maintained at its natural level of 7.01 before the addition of any chemicals (collectors, frothers, or desliming agents). Subsequently, 3 mL of kerosene oil was incrementally added as the collector, followed by a three-minute conditioning step.

The flotation cell was operated at a rotational speed of 1300 RPM and an air flow rate of 1.0 liters per minute, chosen based on previous studies to optimize bubble-particle collision efficiency. A stable, dark gray froth with minimal entrainment of gangue minerals was observed (Figure 1). The concentrate was continuously collected using a froth scraper, with concurrent washing achieved by adding fresh water. This collection

process continued for fifteen minutes. The decanted tailings were collected in a separate container. Both the concentrate and tailings fractions were subsequently dried in an oven at 105°C for six hours. The dried samples were then weighed using an electronic balance to determine concentrate yield and recovery efficiencies.

Building upon the procedures established in Experiment I, Experiment II introduced the following modifications: the sample weight was increased to 300 g, and 2 g of sodium hydroxide (NaOH) was introduced as a pH regulator. The addition of NaOH increased the pH of the slurry from 7.01 to 7.40 (a Δ pH of 0.39).

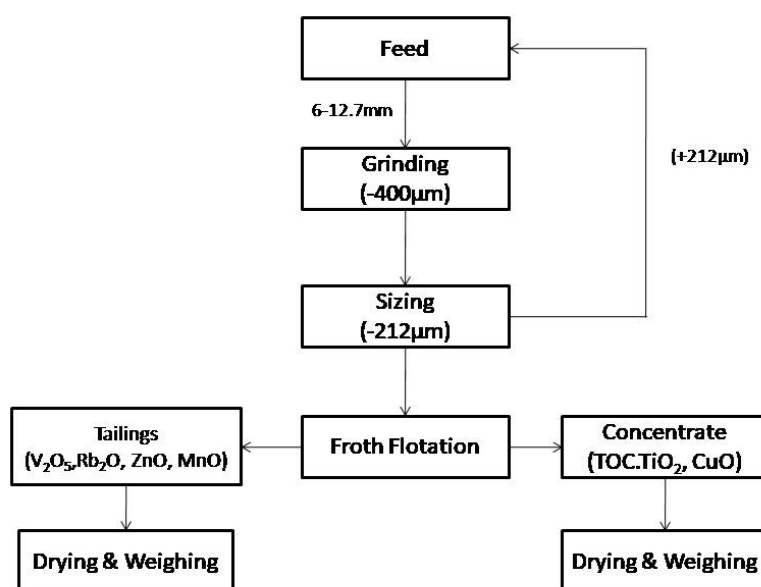


Figure 2. Flowsheet of flotation experiment performed to upgrade the Chimiari Black shale (experiment 2).

Maintaining consistency with Experiment I, the flotation cell was again operated at a rotational speed of 1300 RPM and an air flow rate of 1.0 liters per minute. A stable froth, similar to that observed in Experiment I (Figure 2), was produced, indicating potential for good bubble-particle attachment. The concentrate was continuously removed using a froth scraper, with concurrent washing achieved by adding fresh water. This collection process continued for fifteen minutes. The decanted tailings were collected in a separate container.

Following the flotation process, both the concentrate and decanted tailings were dried in an oven at 105°C for six hours to ensure complete moisture removal. The dried samples were

then weighed using an electronic balance for precise measurement. The weight data was subsequently used to calculate mass losses throughout the experiment. The overall process flow for both experiments is visually illustrated in Figure 2.

3. Results and Discussion

3.1. Experiment-I

The results of concentrate and tailings were compared with the results of feed/composite sample and furnished in Table 1. From the table following assessments were derived:

Table 1. Percentage weight and material balance of concentrate and tailings for experiment-I.

Minerals/ Metals	Feed Grade %	Grade in Con- centrate %	Grade in Tailing %	Wt. in Concen- trate %	Wt. in Tailing %	Total Wt. (Conc+Tailings) %	Unaccounted Ma- terial For (MUF) %
SiO ₂	49.86	45.52	51.72	32.59	65.61	98.20	1.80
Al ₂ O ₃	11.30	11.11	11.54	35.10	64.57	99.67	0.33
Fe ₂ O ₃	7.53	6.65	8.01	31.55	67.33	98.88	1.12
CaO	5.18	4.02	5.85	27.72	71.35	99.08	0.92
MgO	0.56	0.33	0.66	20.65	73.83	94.47	5.53
K ₂ O	2.55	2.71	2.47	37.96	61.39	99.34	0.66
SO ₃	4.12	2.37	5.10	20.55	78.26	98.82	1.18
V ₂ O ₅	0.26	0.17	0.31	22.74	75.95	98.69	1.31
MnO ₂	0.14	0.10	0.17	24.56	74.46	99.02	0.98
TiO ₂	0.81	1.28	0.56	55.92	43.75	99.66	0.34
Rb ₂ O	NR	0.022	NR	CC	CC	CC	CC
ZnO	NR	NR	0.031	CC	CC	CC	CC
CuO	NR	0.015	0.009	CC	CC	CC	CC
TOC	17.92	25.76	13.77	51.32	48.60	99.92	0.08

3.1.1. TOC Dynamics

The flotation process effectively concentrated total organic carbon (TOC). The TOC content increased from 17.92% in the feed to a refined 25.76% in the float concentrate, representing an enrichment factor of X (Enrichment factor = TOC_{concentrate} / TOC_{feed}). Notably, 51.32% of the TOC was recovered in the concentrate, which translates to 35.7% of the initial feed mass. However, the presence of 48.60% TOC in the tailings (62.75% of the original feed mass) suggests a complex interplay between surface chemistry and hydrodynamics govern-

ing the partitioning of organic species during flotation. A residual loss of 0.08% of the initial TOC highlights the intricacies of material tracking and process efficiency. This loss could potentially be influenced by factors such as frother type and dosage, pulp pH, and particle size distribution, which warrant further investigation.

3.1.2. TiO₂ Enrichment

Flotation yielded a moderate enrichment of titanium dioxide (TiO₂). The TiO₂ content increased from 0.81% in the feed to 1.28% in the concentrate, representing a concentration factor of approximately 1.57. This translates to the recovery of

0.91 g of TiO_2 in the concentrate, capturing 55.92% of the initial TiO_2 content. However, 43.74% of the TiO_2 remained in the tailings (assaying 0.563%), highlighting the complex interplay between surface properties, mineral liberation, and froth stability. Further optimization is likely achievable through investigating the influence of collector selection, pH manipulation, and pulp density on flotation kinetics and the resulting concentrate mineralogy.

3.1.3. Mineralogical Dynamics

Analysis of other metal oxides revealed interesting trends. Zinc oxide (ZnO), while absent from the composite and concentrate, exhibited a notable concentration of 0.031% in the tailings, suggesting potential for further upgrading. Similarly,

rubidium oxide (Rb_2O) and copper oxide (CuO), undetectable in the initial sample due to low abundance, became discernible in the float concentrate, indicating selective targeting by the flotation process. Vanadium pentoxide (V_2O_5) showed a modest increase in concentration within the tailings (enrichment factor of 1.2) and a more significant enrichment in the float concentrate. However, this limited upgrading in the tailings necessitates further investigation into the underlying physico-chemical mechanisms governing flotation.

3.2. Experiment-II

The results of concentrate and tailings were compared with the results of feed/composite sample and furnished in Table 2. From the table following assessments were derived:

Table 2. Percentage Weight and material balance of concentrate and tailings for experiment-II.

Minerals/ Metals	Feed Grade %	Grade in Con- centrate %	Grade in Tailing %	Wt. in Concen- trate %	Wt. in Tailing %	Total Wt. (Conc+Tailings) %	Unaccounted Ma- terial For (MUF) %
SiO_2	49.86	45.32	50.31	21.87	75.24	97.12	2.88
Al_2O_3	11.60	10.12	12.19	21.00	78.31	99.31	0.69
Fe_2O_3	7.53	7.61	7.58	24.33	75.08	99.41	0.59
CaO	5.18	3.81	5.67	17.69	81.59	99.28	0.72
MgO	0.56	0.25	0.67	10.49	88.90	99.39	0.61
K_2O	2.55	2.08	2.70	19.70	78.99	98.69	1.31
SO_3	4.12	2.26	4.76	13.19	86.03	99.22	0.78
V_2O_5	0.26	0.19	0.28	17.75	80.32	98.07	1.93
MnO_2	0.14	0.11	0.15	19.12	80.38	99.50	0.50
TiO_2	0.81	0.89	0.78	26.31	71.27	97.58	2.42
Rb_2O	NR	NR	NR	CC	CC	CC	CC
ZnO	NR	NR	0.021	CC	CC	CC	CC
CuO	NR	0.023	NR	CC	CC	CC	CC
TOC	17.92	25.87	15.21	36.09	63.29	99.38	0.62

3.2.1. TOC Concentration Dynamics

Flotation effectively concentrated total organic carbon (TOC). The TOC content increased from 17.92% in the composite sample to 26.87% in the concentrate. This enrichment resulted in the recovery of 19.4 g of TOC in the concentrate, with 40.08 g remaining in the tailings. A small amount of unaccounted material highlights the inherent complexity of material tracking during processing. Notably, 36.09% of the TOC was recovered in the concentrate, representing 24.1% of the

initial mass. The remaining 62.2% (40.08 g) resided in the tailings, constituting 74.55% of the initial feed. This distribution underscores the complex interplay between hydrodynamic forces and the chemical affinity of organic matter during the flotation separation process.

3.2.2. V_2O_5 Concentration Dynamics

Vanadium Pentoxide (V_2O_5) exhibited a contrasting behavior during flotation. While a larger portion (0.624g) ended up in the tailings (representing approximately 80.3% of the initial mass), the concentration itself slightly decreased from 0.26%

in the feed to 0.19% in the concentrate. This suggests that a lower enrichment factor was achieved for V_2O_5 , warranting further investigation into the separation mechanisms for this particular metal oxide.

3.2.3. TiO_2 Concentration Dynamics

Similar to V_2O_5 , the concentration of Titanium Dioxide (TiO_2) increased slightly in the concentrate (0.81% to 0.89%) but decreased in the tailings (0.78%). Consequently, the concentrate captured 0.64 g of TiO_2 out of a total of 2.43 g. This translates to an enrichment of only 26.31% of the TiO_2 in the concentrate, with the remaining significant portion (71.27%, or 1.79 g) found in the tailings. The unaccounted TiO_2 highlights the importance of precise measurements for accurate material balance assessments.

3.2.4. Other Mineral Dynamic

Copper oxide (CuO) was detected in the concentrate at a concentration of 0.023% (230 ppm), indicating its affinity towards the flotation process. Zinc oxide (ZnO), previously undetectable in the composite sample, was identified in the tailings at 0.021% (210 ppm). Interestingly, the concentrations of silicon dioxide (SiO_2), sulfur trioxide (SO_3), magnesium oxide (MgO), and ZnO in the tailings mirrored those observed in Experiment I, suggesting consistent mineral partitioning behavior. Aluminium oxide (Al_2O_3) exhibited slightly higher

concentrations in the tailings compared to both the feed and concentrate, implying preferential distribution towards the tailings fraction. Notably, rubidium oxide (Rb_2O) was not detected in either the concentrate or tailings, suggesting its negligible presence or limited interaction with the flotation process.

3.3. Comparison of Experiment I&II

A rigorous examination of data acquired from flotation Experiments I and II (Table 3) elucidates significant observations pertaining to mineral recovery and enrichment behavior. These findings hold substantial value for the optimization of mineral processing methodologies. Material balance assessments reveal a noteworthy disparity in concentrate recovery rates between the experiments. Experiment I achieved a superior yield of 35.7%, whereas Experiment II exhibited a lower recovery of 24.1%. However, a more intriguing observation lies in the contrasting distribution of Total Organic Carbon (TOC). Despite its lower overall yield, Experiment I demonstrate a higher recovery of TOC (31.32%) compared to Experiment II (36.09%). This divergence underscores the intricate interplay between process variables and emphasizes the necessity for a comprehensive understanding of flotation kinetics to achieve selective recovery of the target species.

Table 3. Percentage Weight and material balance comparison of Experiment I & II.

Minerals/ Metals	Feed Grade %	Grade in Con-1 %	Grade in Con-2 %	Grade in Tailing-1 %	Grade in Tailing-2 %	Upgrad. Factor in Con-1	Upgrad. Factor in Con-1	Upgrad. Factor in Tailings-1	Upgrad. Fac- tor in Tail- ings-2
SiO_2	49.86	45.52	45.32	51.72	50.31	0.91	0.91	1.04	1.01
Al_2O_3	11.30	11.11	10.12	11.54	12.19	0.98	0.9	1.02	1.08
Fe_2O_3	7.53	6.65	7.61	8.01	7.58	0.88	1.01	1.06	1.01
CaO	5.18	4.02	3.81	5.85	5.67	0.78	0.74	1.13	1.09
MgO	0.56	0.33	0.25	0.66	0.67	0.59	0.44	1.17	1.19
K_2O	2.55	2.71	2.08	2.47	2.70	1.06	0.82	0.97	1.06
SO_3	4.12	2.37	2.26	5.10	4.76	0.58	0.55	1.24	1.15
V_2O_5	0.26	0.17	0.19	0.31	0.28	0.64	0.74	1.20	1.08
MnO_2	0.14	0.10	0.11	0.17	0.15	0.69	0.79	1.18	1.08
TiO_2	0.81	1.28	0.89	0.56	0.78	1.57	1.09	0.69	0.96
Rb_2O	NR	0.022	NR	NR	NR	CC	CC	CC	CC
ZnO	NR	NR	NR	0.031	0.021	CC	CC	CC	CC
CuO	NR	0.015	0.023	0.009	NR	CC	CC	CC	CC
TOC	17.92	25.76	25.87	13.77	15.21	1.44	1.50	0.77	0.85

Flotation experiments revealed distinct enrichment behaviour for targeted minerals. The enrichment factor for Total Organic Carbon (TOC) was 1.44 in Experiment I and 1.50 in Experiment II. Conversely, Experiment II demonstrated a more pronounced removal of Iron Oxide (Fe_2O_3) and Copper Oxide (CuO) from the Chimiari black shale, as evidenced by their higher concentrations in the tailings compared to Experiment I. This suggests a greater partitioning of these oxides towards the tailings fraction.

In contrast, Experiment I exhibited superior conditions for upgrading Titanium Dioxide (TiO_2) in the concentrate. The enrichment factor for TiO_2 reached 1.57, significantly higher than the 1.09 achieved in Experiment II. This highlights the crucial role of process optimization in maximizing the recovery and concentration of specific minerals during flotation. Further analysis of mineral concentrations revealed consistent flotation behaviour for Silicon Dioxide (SiO_2), Sulphur Trioxide (SO_3), Vanadium Pentoxide (V_2O_5), Magnesium Oxide (MgO), Titanium Dioxide (TiO_2), Manganese Dioxide (MnO_2), and Zinc Oxide (ZnO) across both experiments. These observations suggest minimal variations in the partitioning of these minerals between the concentrate and tailings fractions.

Notably, Rubidium Oxide (Rb_2O) was only detected in Experiment I, implying its negligible presence or limited interaction with the flotation process in Experiment II. Conversely, V_2O_5 exhibited selective upgrading, with an enrichment factor of 1.20 in Experiment I and a slightly higher value of 1.18 in Experiment II. This finding suggests a moderate improvement in V_2O_5 recovery during Experiment II. The exclusive detection of Zinc Oxide (ZnO) in the tailings of Experiment-II, despite its minimal concentration in the initial feedstock, underscores the sensitivity of flotation separations to trace elements. This observation emphasizes the importance of meticulously controlling process parameters to optimize the recovery of even low-abundance minerals.

4. Conclusions

A comprehensive comparison of the two flotation experiments unveils a complex interplay between chemical modifications and process parameters, offering valuable insights for optimizing mineral processing methodologies. Notably, the introduction of sodium hydroxide (NaOH) as an alkali modifier in Experiment II significantly influenced surface charge properties, leading to a noticeable reduction in froth volume. This suggests a potential mechanism for tailoring froth stability for improved selectivity.

Furthermore, the strategic, batch-wise addition of kerosene oil as a collector in both experiments underscores its crucial role in enhancing float yield. Kerosene oil likely facilitates selective mineral recovery through hydrophobic interactions with targeted mineral surfaces. These observations highlight the importance of optimizing collector selection and dosage

strategies for efficient mineral separation. Moreover, the incorporation of pine oil as a frother in Experiment-II catalyzed enhanced gas-liquid interfacial dynamics, augmenting bubble stability and facilitating the preferential entrainment of valuable organic compounds within the float fraction. This underscores the critical role of frother chemistry in modulating flotation kinetics and organic compound recovery.

Both flotation experiments yielded promising results for the enrichment of critical elements. Total Organic Carbon (TOC), Titanium Dioxide (TiO_2), Rubidium Oxide (Rb_2O), and Copper Oxide (CuO) exhibited significant enrichment in the concentrate fraction. Conversely, Vanadium Pentoxide (V_2O_5), Manganese Dioxide (MnO_2), and Zinc Oxide (ZnO) displayed a preference for partitioning into the tailings. This suggests a potential separation strategy for these targeted minerals. However, a key challenge remains in achieving effective separation of gangue minerals. The consistent presence of Silicon Dioxide (SiO_2) in the tailings fraction of both experiments highlights the difficulty of separating it from the desired minerals within the complex ore matrix.

The calculated enrichment factors for Total Organic Carbon (TOC) of 1.44 in Experiment I and 1.50 in Experiment II under modified conditions highlight the significant impact of frother chemistry on organic compound recovery. These findings suggest that optimizing frother selection and dosage can be a valuable strategy for enhancing TOC enrichment. Along with that, Experiment II's conditions yielded a slightly higher enrichment factor for Vanadium Pentoxide (V_2O_5) compared to Experiment I. This observation warrants further investigation into the specific process parameters that influence V_2O_5 recovery during flotation. These insights pave the way for the development of tailored reagent selection and potentially selective precipitation techniques to improve the recovery of targeted minerals.

Abbreviations

Cu	Copper
Zn	Zinc
Re	Rhenium
Ge	Germanium
Au	Gold
Pd	Palladium
Pt	Platinum
TOC	Total Organic Carbon
NaOH	Sodium Hydroxide
XRF	X-Ray Fluorescence
H_2SO_4	Sulfuric Acid
HF	Hydrofluoric Acid
NaClO	Sodium Chlorate

Author Contributions

Muhammad Tariq: Conceptualization, Methodolgy, Resources

Shahab Saqib: Data curation, Methodology, Supervision
Muhammad Mansoor Iqbal: Formal Analysis, Investigation, Supervision

Adil Hussain: Formal Analysis, Investigation

Ahmad Raza: Formal Analysis, Writing – original draft, Writing – review & editing

Data Availability Statement

Further data can be provided upon request by the reader to the corresponding author respectively.

Conflicts of Interest

The authors report there are no competing interests to declare.

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