The present work was submitted to the Faculty of Engineering

Simulation and Optimization of Column Leaching Processes for Oxidized Zinc Ore

Bachelor Thesis

by

Munkhzul Bilegt

Supervisor 1 / M.Sc Baasandorj.M
Supervisor 2 / M.Sc Dorjsundui.G

Ulaanbatar/Nalaikh, 11.01.2024

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Statutory Declaration

Bilegt , Munkhzul Student ID Number B2000403

I hereby affirm in lieu of an oath that I provided the submitted bachelor thesis

Simulation and Optimization of Column Leaching Processes for
Oxidized Zinc Ore

I did not use any sources other than those stated. In case the work is additionally submitted on a data medium, I declare that the written and the electronic form are completely identical. The work was not submitted in the same or similar form to any examination authority.

Place, Date Signature Ulaanbaatar 2024/11/1 Munkhzul

Acknowledgment

I extend my deepest gratitude to my thesis supervisors, Mr. Baasandorj and Mr. Dorjsundui, for their invaluable guidance and support throughout this research. Mr. Baasandorj's depth of knowledge in raw material processing engineering has been a guiding light, and his constructive feedback has challenged me to refine my approach and elevate the quality of this thesis. Mr. Dorjsundui’s expertise in data analysis has been essential, providing me with the skills and tools needed to develop a robust analytical framework for this work. Their unwavering support, encouragement, and dedication have been instrumental at every stage of this journey, and I am profoundly grateful for their mentorship.

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1. Introduction

1.1 Brief History of the Zinc Ore

Zinc ore, in combination with copper, was used thousands of years before zinc was identified as a distinct element. Zinc alloys, especially brass, have been historically significant and are now primarily used to prevent rust. Today, zinc is crucial for galvanizing other metals, like iron, in large quantities. Additionally, zinc casting plays an essential role in manufacturing automotive, electrical, and other equipment components. Zinc sulfide is also utilized to produce luminescent paints, fluorescent lighting, and X-ray screens. Over the past 40 years, global zinc production has increased significantly due to growing demand in the construction, automotive, and electronics industries. In the early 1980s, global zinc production was approximately 6 million metric tons per year. Since then, production has steadily risen, reaching around 12 million metric tons annually in recent years.

1.2 Technology of Zinc production

Zinc ranks as the fourth most consumed metal globally, mainly due to construction demand. Zinc is primarily sourced from zinc sulfide (ZnS) ore, which supports over 85% of global production, though oxide ores are gaining interest due to sulfide depletion. Oxide ores, however, are challenging to process through conventional flotation due to impurities. The electrolytic zinc process, involving sulfuric acid leaching and purification, is used for low-impurity ores, while high-impurity oxide ores may require direct leaching, although this is costly and complex. This thesis aims to examine the column leaching process and the potential of the flotation process, for enriching low-grade oxidized zinc ore that is challenging to process using traditional methods.
The detailed aims are ;

The Bond Work Index (BWI) determines the hardness and energy consumption of ore during comminution, which is crucial for selecting the appropriate equipment.

The potential of the flotation process to enhance the enrichment of low-grade oxidized zinc ore, as well as the effects of reagents and temperature on this process.

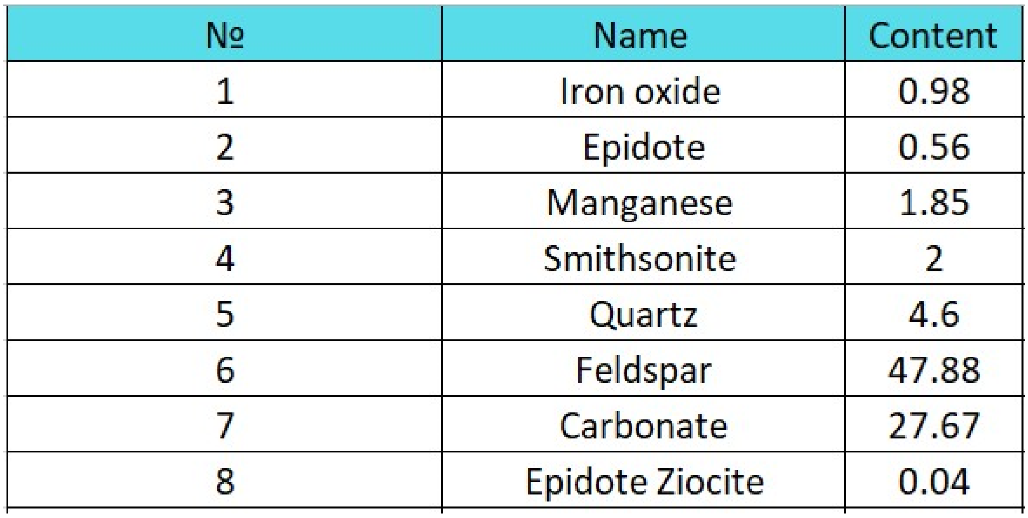
Permeability tests are conducted to ensure the appropriate flow rate of acid through the ore in the column.

Bottle roll leaching is employed to assess how particle size and leaching time impact metal recovery, with the goal of identifying the optimal leaching duration and feed particle size for column leaching.

Vat leaching is applied to identify the optimal concentration for effective leaching.

1.3 Feed Introduction

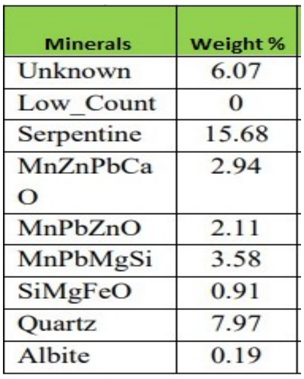
The feed material was prepared through a three-stage crushing process, followed by the cone and quartering method and riffling. It was then sent to the Central Geological Laboratory in Mongolia for analysis. Results indicate elevated levels of carbonate and magnesium content. XRD analysis was also conducted; however, no zinc content was detected in samples analyzed at both the Erdenet Mining Corporation Laboratory and the Central Geological Laboratory.



№	Name	Content
1	Iron oxide	0.98
2	Epidote	0.56
3	Manganese	1.85
4	Smithsonite	2
5	Quartz	4.6
6	Feldspar	47.88
7	Carbonate	27.67
8	Epidote Ziocite	0.04

Table 1.3.1 Element Analysis Result

The samples were subsequently sent to Freiberg for in-depth analysis, where advanced techniques were employed to determine the precise zinc content and identify the specific zinc-bearing minerals, which were successfully detected and documented, providing valuable insights into the mineral composition of the feed material.



Minerals	Weight %
Unknown	6.07
Low_Count	0
Serpentine	15.68
MnZnPbCaO	2.94
MnPbZnO	2.11
MnPbMgSi	3.58
SiMgFeO	0.91
Quartz	7.97
Albite	0.19

Table 1.3.2 Freiberg laboratory results

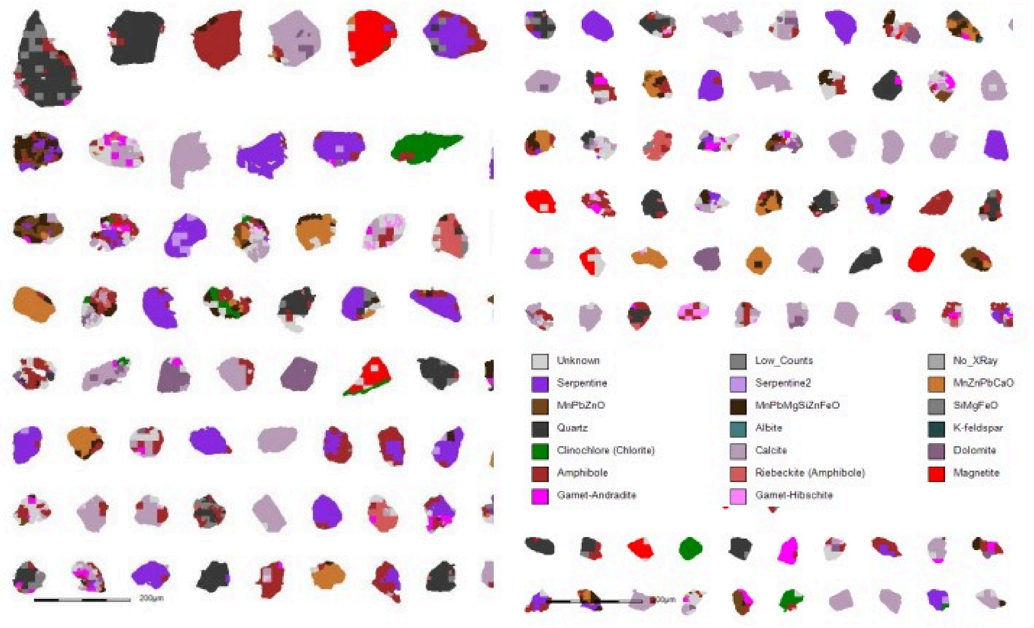


Figure 1.3.1 Zinc Containing Minerals

2. Methodology

2.1 Sample preparation

Zinc ore samples were received from "Mongolrostsvetmet SOE," with each sample weighing 200 kg, resulting in a total of 400 kg. Particles larger than 10 cm were separated from the bulk material, followed by manual crushing with a hammer to reduce the size to less than 10 cm. The ore was then crushed using a jaw crusher, reducing the particle size to a maximum of 14 mm. The crushed ore, containing 1.31% zinc, was prepared for further analysis using the coning and quartering method. Additionally, the riffling splitter was applied after coning and quartering to further ensure a representative sample for analysis.

2.1.1 Coning and Quartering for sample preparation technique

The sampling method employed for large quantities of material, such as 20 kg, involves forming the material into a conical heap on a solid surface. This technique relies on the radial symmetry of the heap to produce four quarters when divided by perpendicular axes. Two opposite quarters are selected as the representative sample, while the remaining two quarters are set aside. The selected portions can be further reduced by repeating this process until the desired sample size is achieved.

Figure 2.1.1 - Zinc Ore Feed Preparation Using the Coning and Quartering Method

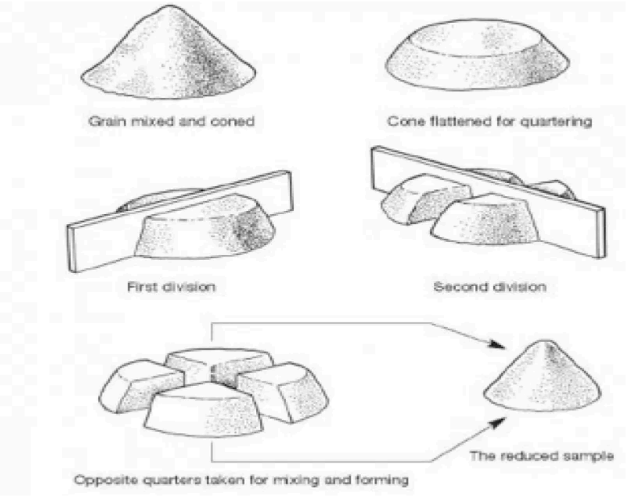


Figure 2.1.2 - Coning and Quartering method

<u>The procedure of Coning and Quartering Method</u>

1. Prepare the work area adjacent to the sampling location.
2. Ensure that the floor is clean for the sampling process.
3. Spread the sample on the floor and thoroughly mix it to form a conical heap.
4. Perform the quartering procedure to divide the heap into four equal parts.

5. Repeat the quartering process as needed to reduce the sample size.

   The entire process, from steps 1 to 5, should be completed within 30 minutes.

2.1.2 The Riffle Splitter in sample preparation method

The sample splitter is designed as an open V-shaped container equipped with a series of chutes positioned at right angles to the long axis, creating a series of rectangular slots of equal area. These slots alternatively direct material into two collection trays. The sample, which contains particles sized to allow free movement through the slots (with the largest particle being no greater than one-third the width of the riffle openings), is poured into the feeder and is subsequently split into equal portions. Through repeated cycles of this process, the desired sample size is achieved.

Procedure for Riffle Sampling:

1. Set up the sample and the riffle splitter, ensuring that the riffle splitter is clean before use.
2. Conduct the riffle-splitting process once.
3. Repeat the riffling, discarding every alternate sample to ensure representativeness.
4. Bag the selected sample and label it appropriately; store any excess material.
5. Clean the riffle splitter after completion of the sampling process.

Figure 2.1.3: a) Process of using a riffle splitter to divide crushed feed into portions with the same particle size distribution, b) Riffle splitter

After applying these methods to ensure a representative sample, half of the sample is tested at the Central Geological Laboratory of Mongolia. *The feed containing 1.31% zinc and particle size of -1.44 mm will be used in the subsequent stages.*

2.2 Bond Work Index

The Work Index is a metric employed in the mineral processing field to assess the grind ability of different materials, estimate the energy necessary for grinding, and assist in the sizing and selection of ball milling equipment. It serves as a reference for comparing how materials vary in their ease of grinding. It is expressed as the energy in kilowatt-hours per short ton needed to reduce the material from an infinite feed size to a point where 80% of it passes through a 100 µm screen (Will's Mineral Process Technology, Elsevier 2005). The Bond Work Index /BWI/ is determined using the following formula:

determined using the following formula:

$$BWI = \frac{45.1}{P1^{2} \times 0.23 \times Grp^{0.82} \times (\frac{10}{\sqrt{p}} - \frac{10}{\sqrt{F}})}$$

Formula 2.2.1; Bond Work Index

$P1$- $closing\ screen\ size$

$Grp\ -\ net\ grams\ of\ screen\ undersize\ \ per\ mill\ revolution$

$P\ -\ 80\ percent\ passing\ size\ of\ the\ product$

$F\ -80\ percent\ passing\ size\ of\ the\ feed$

In the given formula, the closing screen size $P1$ is specified as 125 microns. The parameters Gbp, P, F are obtained through comprehensive sieve analysis and the execution of the Bond Work Index test.

2.2.1 Sieve analysis

The initial ore sample was prepared using the coning and quartering method and then subdivided for the Bond Work Index test. Sieve analysis should be conducted on both the feed to the milling operation and the product of milling to determine the particle size at which 80 percent of the material passes, indicating the changes in the ground material during the operating time. The results / *P80 and F80* / are then used in the Bond Work Index formula to calculate the energy requirements for comminution.

2.2.2 Grindability test

The grindability test is a closed-cycle laboratory procedure used to measure a material's resistance to ball milling. Results from this test are used to determine the Bond Ball Mill Work Index through Bond's formula. The primary procedure for the grind ability test is outlined below.

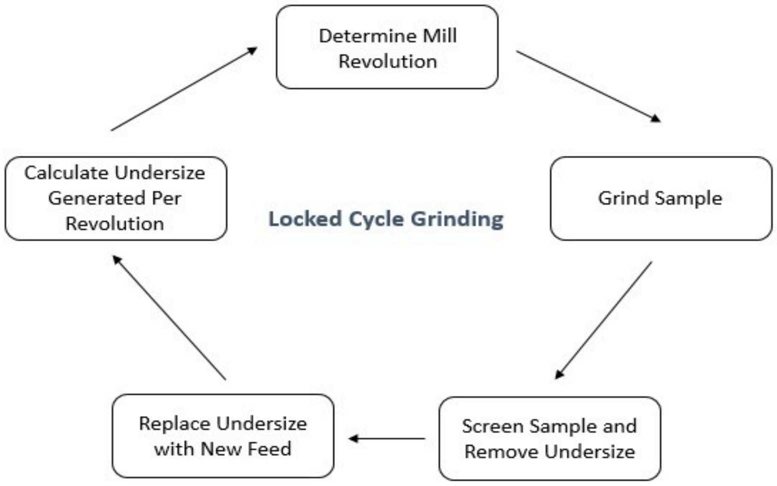


Figure 2.2.1: Grindability Test Procedure

2.3 Flotation process

The flotation method is the most commonly used technique for extracting zinc from its ore, especially from zinc sulfide ores. This process utilizes the differences in hydrophobicity between zinc-bearing minerals and the surrounding gangue. The choice of an appropriate enrichment method depends on the characteristics of the ore, its mineral associations, and the desired enrichment levels. In flotation, activators, collectors, and frothers each serve distinct roles to enhance the separation process:

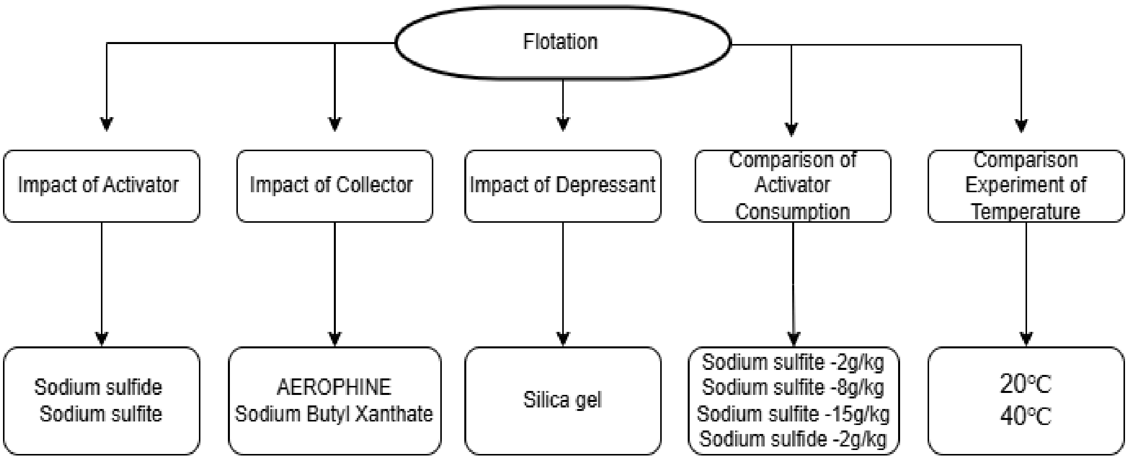
1. *Activators*: These substances modify the surface characteristics of certain minerals, making them more amenable to collector attachment. By improving mineral receptiveness, activators help previously non-reactive or weakly reactive surfaces better adhere to air bubbles, enhancing mineral recovery.
2. *Collectors*: Collectors make mineral particles hydrophobic, encouraging them to attach to air bubbles and rise to the surface. By selectively binding to particular minerals, collectors improve both the selectivity and efficiency of the separation process.
3. *Frothers*: Frothers manage bubble size and stability in the flotation cell, maintaining a balanced froth layer that supports mineralized bubbles. By preventing bubbles from becoming too large or collapsing too quickly, frothers enable the formation of a stable froth layer that assists with effective mineral separation.

Together, these reagents ensure an optimized flotation process, facilitating specific mineral-bubble attachment (through collectors and activators) while stabilizing bubbles for maximum recovery (frothers).

In this project, our goal was to determine the most effective zinc enrichment process through laboratory testing and to compare zinc recovery rates under various methods and conditions.

Aim: The potential of the flotation method for low-grade oxidized zinc ore, along with the influence of reagents in the flotation process, will be examined. Additionally, a comparison of different reagents will be conducted to achieve higher metal recovery.

Figure 2.3.1 Procedure for Conducting Flotation Experiment



2.4 Permeability test

The permeability of a material is a metric that shows the rate at which liquid flows through the material. This is one of the important parameters that should be considered during column dissolution experiments. The coefficient of permeability (k) refers to the ability to conduct fluids through the matrix of the pore size of the material or the voids between them. Two general methods of permeation testing are routinely used in the laboratory: "fixed level" testing and "variable level" testing methods.

Aim: to ensure the appropriate flow rate of acid through the ore in the column.

2.5 Vat Leaching

Vat leaching in the laboratory is a controlled process in which ore is treated with a leaching solution to extract valuable minerals. This process involves placing finely crushed ore into a container, or "vat," and submerging it in a leaching solution, typically an acidic or basic chemical solvent.

In laboratory conditions, vat leaching helps to study leaching kinetics, reaction rates, and the influence of variables such as particle size, concentration, temperature, and pH. The key steps typically include:

1. *Ore Preparation*: The ore is crushed or ground to increase surface area, improving the interaction between ore particles and the leaching solution.
2. *Solution Addition*: The vat is filled with a leaching solution, such as cyanide for gold extraction or sulfuric acid for copper, allowing it to seep through the ore and dissolve the targeted metals.
3. *Leaching Time and Conditions*: The leach time, temperature, and concentration of the leaching agent are carefully controlled. The ore is often agitated or stirred to maximize contact.
4. *Filtration and Analysis*: After leaching, the solution is drained and analyzed to measure metal concentration, giving insights into leaching efficiency and recovery rates.

Figure 2.5.1 Vat Leaching Tank

2.6 Bottle Roll Leaching

Bottle roll leaching is a laboratory technique used to assess the leaching efficiency of minerals. In this process, ore samples are placed in a container filled with a leaching solution, typically an acid or alkaline solution; in our case, sulfuric acid is used. The container is then rotated to ensure thorough mixing and optimal contact between the ore and the solution. This method enables the evaluation of various factors, such as leaching time, particle size, and reagent concentration, on metal recovery, thereby assisting in optimizing conditions for column leaching.

Figure 2.6.1 Bottle Rolling Machine

Figure 2.6.2 Containers for bottle roll leaching

3. Experimental Parts and Results

3.1 Bond Work Index Calculation

To determine the Bond Work Index, the BICO F.C. Bond Ball Mill 395-50X is utilized, along with 285 balls of varying sizes, ranging from 19 mm to 38.1 mm in diameter. A sieve with an opening size of 125 microns was used for the process.

Experimental procedure diagram.

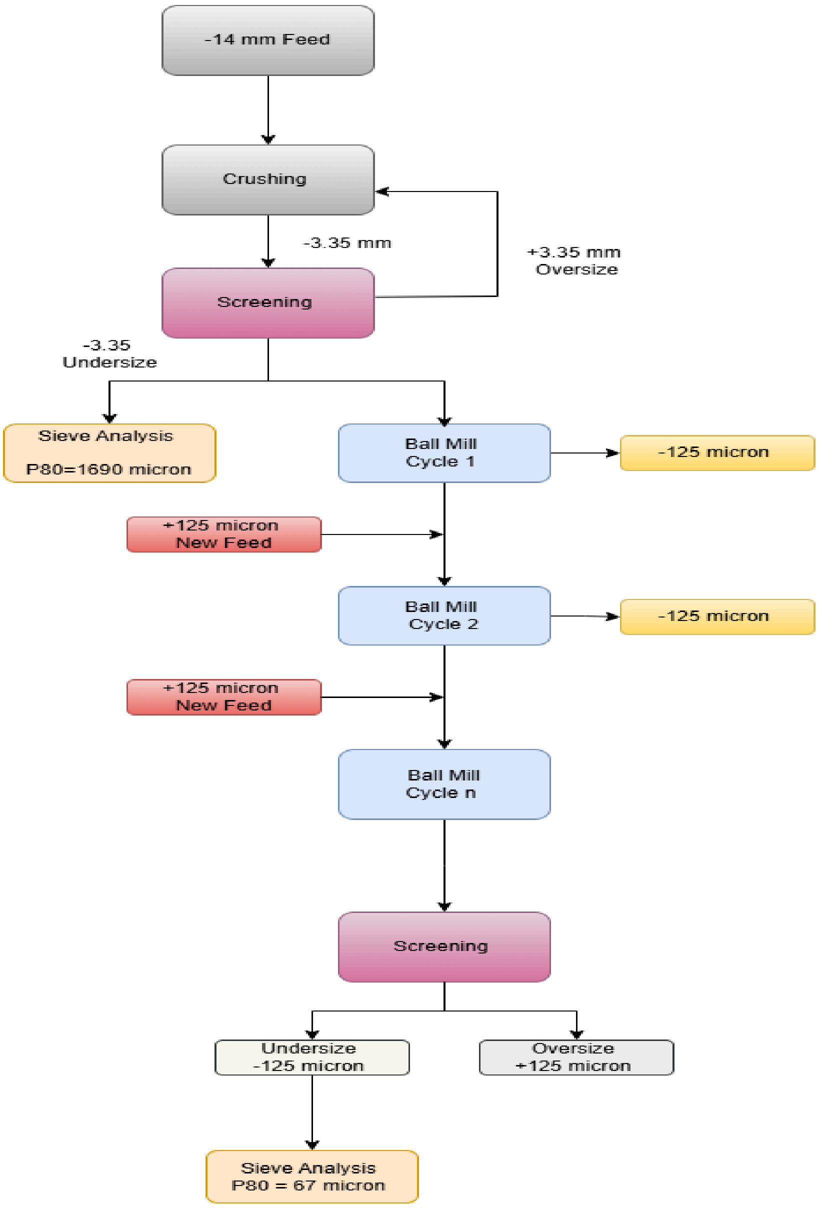
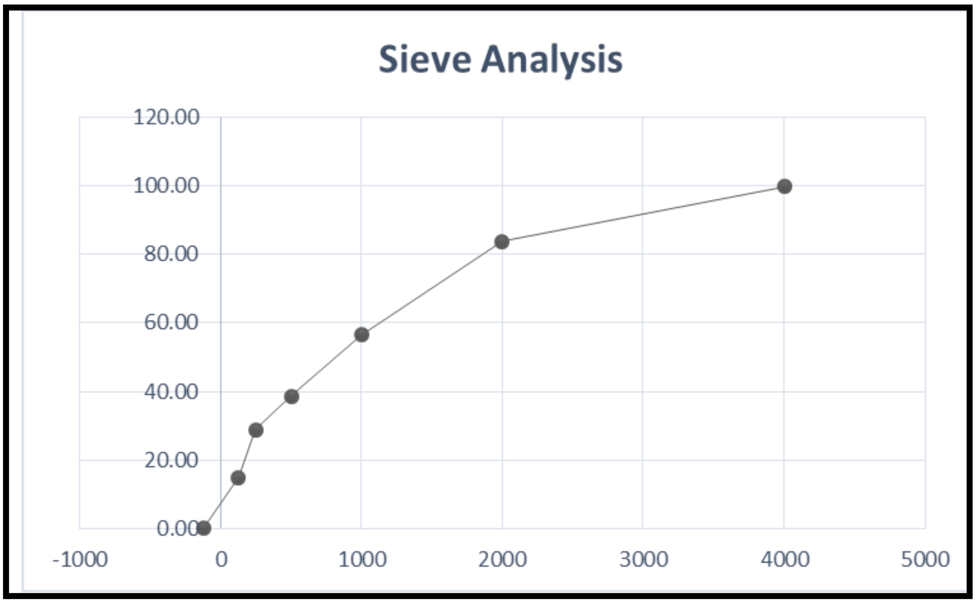
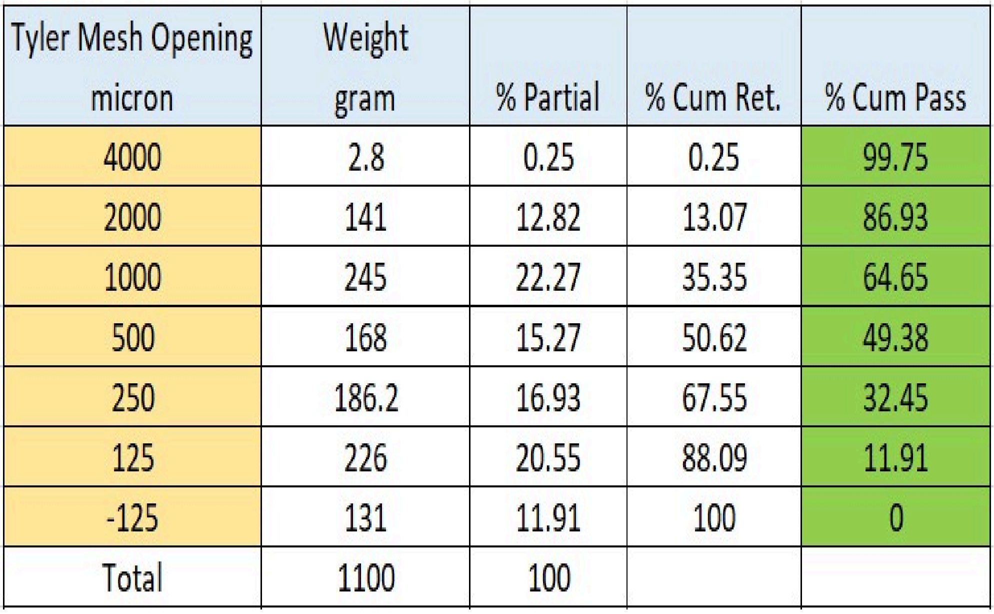


Figure 3.1.1 Experimental procedure diagram



Graphic 3.1.1 Sieve analysis for the feed ore



Tyler Mesh Opening micron	Weight gram	% Partial	% Cum Ret.	% Cum Pass
4000	2.8	0.25	0.25	99.75
2000	141	12.82	13.07	86.93
1000	245	22.27	35.35	64.65
500	168	15.27	50.62	49.38
250	186.2	16.93	67.55	32.45
125	226	20.55	88.09	11.91
-125	131	11.91	100	0
Total	1100	100		

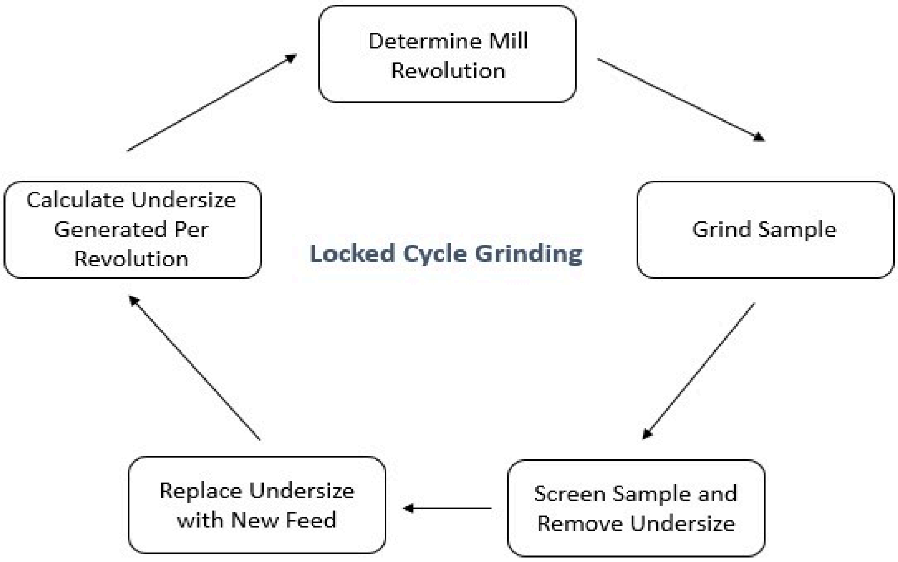
Table 3.1.1 Sieve Analysis data for the feed ore

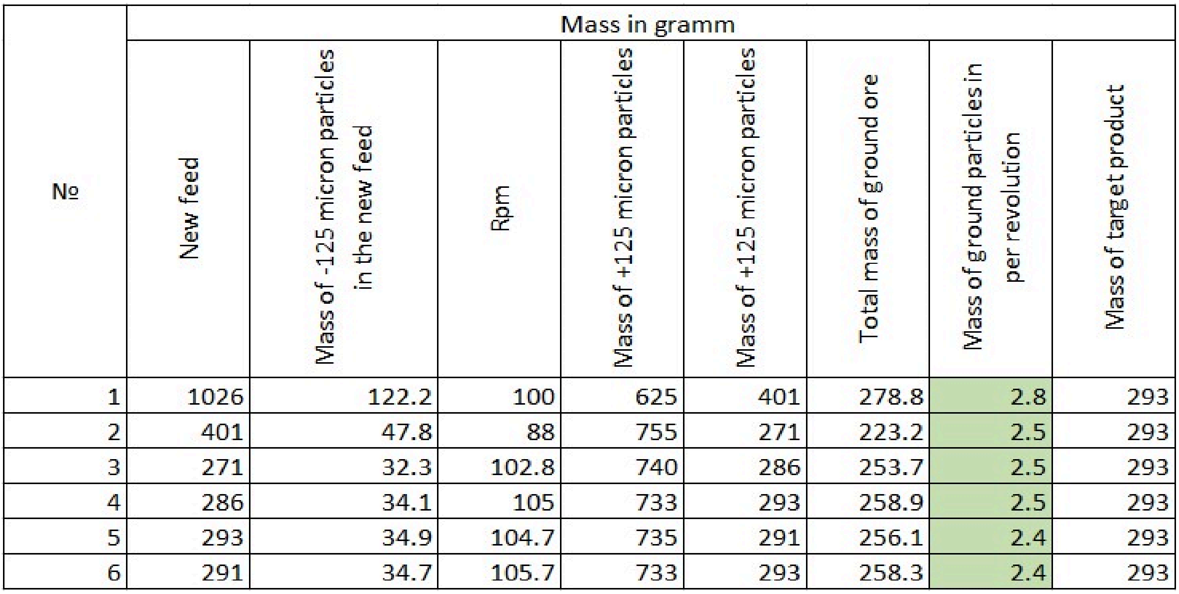
The initial ore sample was prepared using the coning and quartering method and subsequently subdivided for the Bond Work Index test. The subdivided samples underwent primary crushing with a jaw crusher to achieve a particle size of -14 mm. This was followed by secondary crushing using a gravel crusher to ensure that 100% of the material passed through a 3.35 mm sieve. Finally, the processed samples were subjected to sieve analysis for further evaluation. As illustrated in the graphic, the feed size at which 80% of the material passes is 1850 microns.

3.1.1 Ball Mill Grinding Result

During the initial cycle, a 700 ml feed sample of ore, weighing 1,026 grams, is ground at a speed of 100 revolutions per minute. Following the grinding process, the material is subjected to sieving, and the particles measuring less than 125 microns are weighed. These finer particles are subsequently replaced with an equivalent mass of new feed ore. This procedure is repeated until the mass of the particles below 125 microns stabilizes across consecutive cycles. By the fifth and sixth cycles, the mass of the ground ore smaller than 125 microns per revolution reaches a consistent value of 2.4 grams, indicating that a steady state has been achieved. After completing the sixth grinding cycle, wet screening is conducted on the resulting product, revealing that 80 percent of the material successfully passes through a sieve with a mesh size of 67 microns.

Figure 3.1.2 Locked Cycle Grinding Procedure



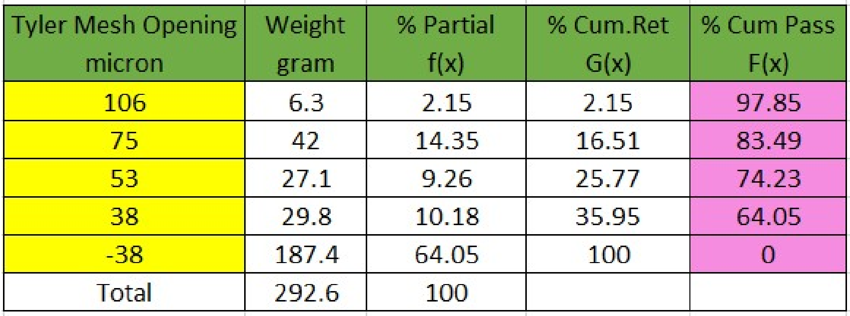


№	Mass in gramm							
	New feed	Mass of -125 micron particles in the new feed	Rpm	Mass of +125 micron particles	Mass of +125 micron particles	Total mass of ground ore	Mass of ground particles in per revolution	Mass of target product
1	1026	122.2	100	625	401	278.8	2.8	293
2	401	47.8	88	755	271	223.2	2.5	293
3	271	32.3	102.8	740	286	253.7	2.5	293
4	286	34.1	105	733	293	258.9	2.5	293
5	293	34.9	104.7	735	291	256.1	2.4	293
6	291	34.7	105.7	733	293	258.3	2.4	293

Table 3.1.2 Ball mill grinding test result

For the feed material, 80% passing size ***F80 was initially 1690 microns.*** After six grinding cycles, particle size analysis showed a ***P80 of 67 microns***.

Shown in the *table 3.1.3* and graphics below.



Tyler Mesh Opening micron	Weight gram	% Partial f(x)	% Cum.Ret G(x)	% Cum Pass F(x)
106	6.3	2.15	2.15	97.85
75	42	14.35	16.51	83.49
53	27.1	9.26	25.77	74.23
38	29.8	10.18	35.95	64.05
-38	187.4	64.05	100	0
Total	292.6	100		

Table 3.1.3 *Sieve analysis for the product obtained from the ball mill.*

Bond Work Index Calculation

$$BWI = \frac{45.1}{P1^{2} \times 0.23 \times Grp^{0.82} \times \left(\frac{10}{\sqrt{p}} - \frac{10}{\sqrt{F}}\right)}$$ *[formula 2.2.1]*

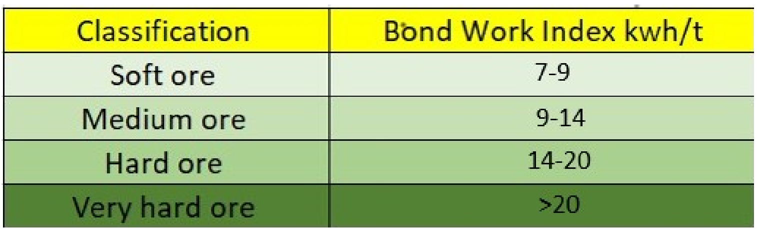
$P1$- 125 $microns$

$Grp\ -\ 2.4\ grams$

$P\ -\ 67\ microns$

$F\ -1690\ microns$

$$BWI\ =\ \frac{45.1}{(125\ microns)^{2}\times(0.23)\times(2.4^{0.82})\times(\frac{10}{\sqrt{67\ microns}}-\frac{10}{\sqrt{1690\ microns}})} = 7.4\ kwh/t$$



Classification	Bond Work Index kwh/t
Soft ore	7-9
Medium ore	9-14
Hard ore	14-20
Very hard ore	>20

Table 3.1.4 Ore Classification Based on Bond Index Values

Following the grind ability test and screen analysis, the feed, product, and grams per revolution were determined at an 80% passing size. These values were then applied to calculate the Bond Work Index. As a result, the Bond Work Index for the zinc ore was found to be 7.4 kWh/t, indicating the energy (in kilowatt-hours per short ton) required to reduce the material size to -125 microns. Based on this index and the reference table, the zinc ore is classified as a soft ore. This finding will assist in assessing the ore's resistance and hardness and aid in selecting appropriate ball milling equipment.

3.2 Flotation Experiment Result and Procedure

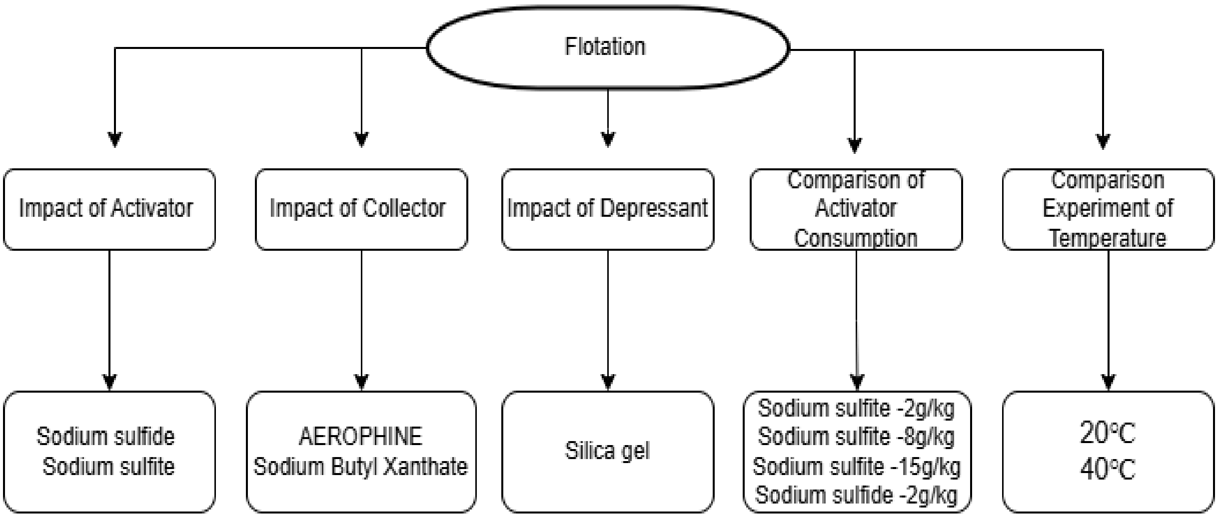


Figure 3.2.1 Working Scheme for Flotation Experiment

Sample Preparation

Sample preparation plays a crucial role in laboratory processes, as it ensures accuracy, consistency, and reliability in analytical results. Properly preparing the sample allows it to accurately represent the entire material, which is vital for obtaining meaningful and reproducible data. Therefore, the sample preparation scheme is outlined below.

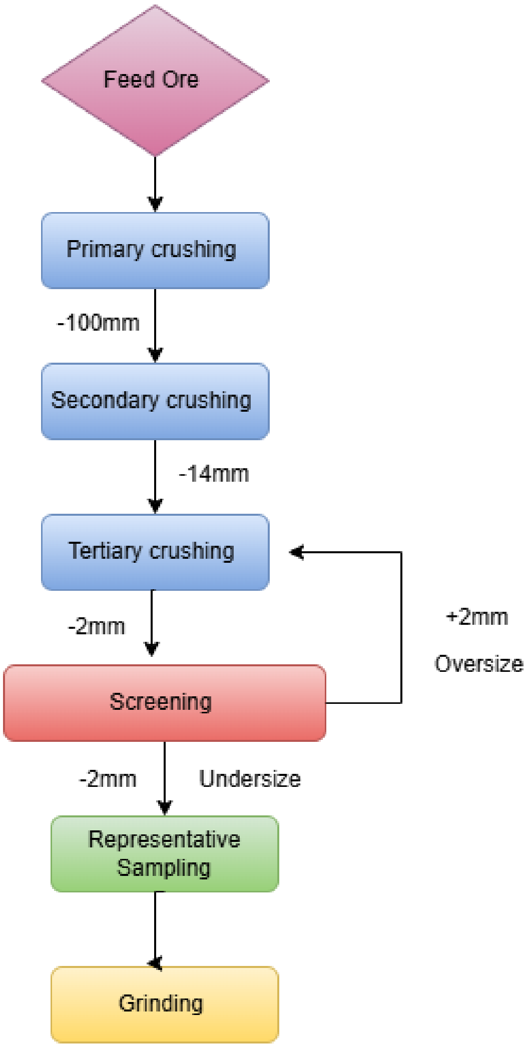


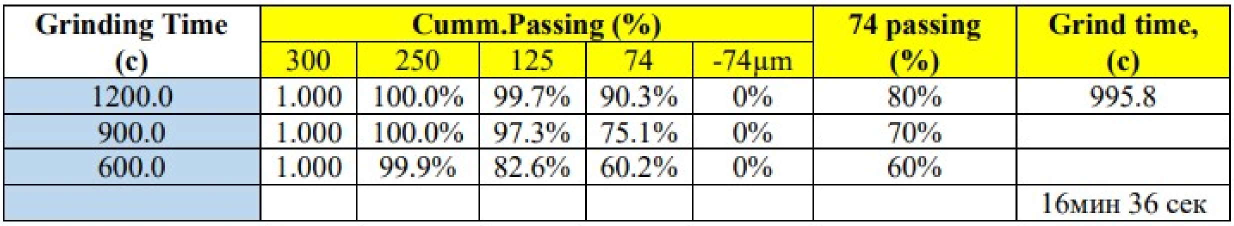
Figure 3.2.2 Feed preparation

Grindability test procedure

- *A total of 325 mL of water is measured using a glass cylinder*
- *A representative sample of zinc ore, weighing 1 kg, is prepared.*
- *The rod mill is positioned vertically within its container.*
- *Then, 325 mL of water and the 1 kg sample are added to the vertically placed rod mill container. S/L = 6/4*
- *Finally, the container is positioned on the rolling machine.*
- *A timer or similar device was set for intervals of 1200, 900, and 600 seconds.*
- *Once the designated time elapsed, the water was separated from the product using a filter, and the product was drained in the laboratory.*
- *Sieve analysis was performed after the product had been fully drained.*

Figure 3.2.3: Rod Mill Container and Rod Mill Used in the Rod Milling Process

Figure 3.2.4 Screen Analysis for the product of rod milling



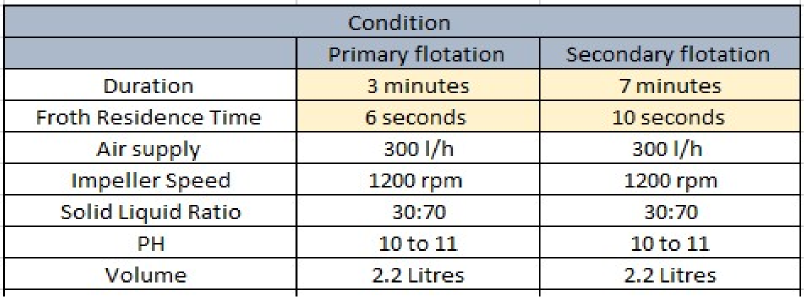
Grinding Time (c)	Cumm.Passing (%)					74 passing (%)	Grind time, (c)
	300	250	125	74	-74μm		
1200.0	1.000	100.0%	99.7%	90.3%	0%	80%	995.8
900.0	1.000	100.0%	97.3%	75.1%	0%	70%	
600.0	1.000	99.9%	82.6%	60.2%	0%	60%	
							16мин 36 сек

Table 3.2.1: Sieve Analysis of Grinding Products at Three Selected Time Intervals

The grinding time required to achieve a P80 of 74 microns is 16 minutes and 36 seconds. Therefore, for subsequent sample preparation for the flotation process, the duration using the roll mill will be set to 16 minutes and 36 seconds.

Flotation Experiment

The Metso D12 flotation cell is utilized in the flotation process, incorporating both main and secondary flotation stages to validate the results of the primary flotation. The flotation occurs under specific conditions.



	Condition	
	Primary flotation	Secondary flotation
Duration	3 minutes	7 minutes
Froth Residence Time	6 seconds	10 seconds
Air supply	300 l/h	300 l/h
Impeller Speed	1200 rpm	1200 rpm
Solid Liquid Ratio	30:70	30:70
PH	10 to 11	10 to 11
Volume	2.2 Litres	2.2 Litres

Table 3.2.2 Flotation conditions

Impact of Activators on the Flotation Efficiency of Oxidized Zinc Ore

In the flotation process, MIBC (Methyl Isobutyl Carbinol) is used as a frother, while sodium butyl xanthogenate serves as the collector, and silica gel acts as the depressant. The process conditions, detailed in Table 1, provide a controlled environment for flotation. To evaluate the impact of different activators, varying concentrations of sodium sulfide and sodium sulfite are applied, allowing for a comparative analysis of each activator's effect on flotation efficiency and selectivity.

Activator	*Concentration*	*Yield %*	*Metal Recovery %*	*Zinc content %*
Na2SO3	*2 gr/kg*	*9.5*	*15*	*1.58*
Na2SO3	*8 gr/kg*	*36.1*	*42.8*	*1.19*
Na2SO3	*15 gr/kg*	*30.6*	*38*	*1.24*
Na2S	*2gr /kg*	*7.7*	*11.6*	*1.51*

Table 3.2.3: Comparison of Activator Impact at Various Concentrations and Types

The results presented in the table indicate that the use of sodium sulfide as an activator at a concentration of 2 g/kg was less effective than employing sodium sulfite at the same dosage. Consequently, further experimentation was conducted with sodium sulfite at elevated concentrations of 8 g/kg and 15 g/kg. Notably, the 8 g/kg concentration yielded the highest recovery rate and overall performance relative to the other concentrations evaluated.

Impact of on the Flotation Efficiency of Oxidized Zinc Ore

Collector	*Concent %*	*Yield %*	*Metal Recovery*	*Zinc Content %*
AEROPHINE	*0.4 g/kg*	*2.6*	*4.2*	*1.63*
Sodium Butyl Xanthogenate	*0.4 g/kg*	*7.7*	*11.6*	*1.51*

Table 3.2.4: Comparison of Collector Impact at Various Concentrations and Types

All experimental conditions were strictly adhered to as specified in [Table 3.2.4], incorporating OTZ100, sodium sulfite, and silica gel into the process. To facilitate a comparison of collectors, AEROPHINE and sodium butyl xanthogenate were tested at identical concentrations. Given previous flotation experiments that demonstrated enhanced recovery with sodium sulfite, it was chosen as the activator, while sodium butyl xanthogenate was utilized as the collector.

In both experiments, all conditions remained consistent, with the exception of the frother: Experiment 1 utilized MIBC, whereas Experiment 2 incorporated OTZ100, a frother widely used in flotation processes at Oyu Tolgoi. The observed differences in recovery between the two experiments may be attributed to the variation in frother used.

Comparison Test of Flotation Temperatures

Temperature	Yield %	Metal Recovery %	*Zinc Content %*
20°C	30.6	38	1.24
40°C	23.2	29.6	1.28

Table 3.2.5: Influence of Temperature on Metal Recovery in the Flotation Process

Raising the temperature did not lead to improved outcomes in this experiment. A temperature of 20°C yielded the highest recovery and overall performance, suggesting that higher temperatures may not enhance yield or recovery under these specific conditions.

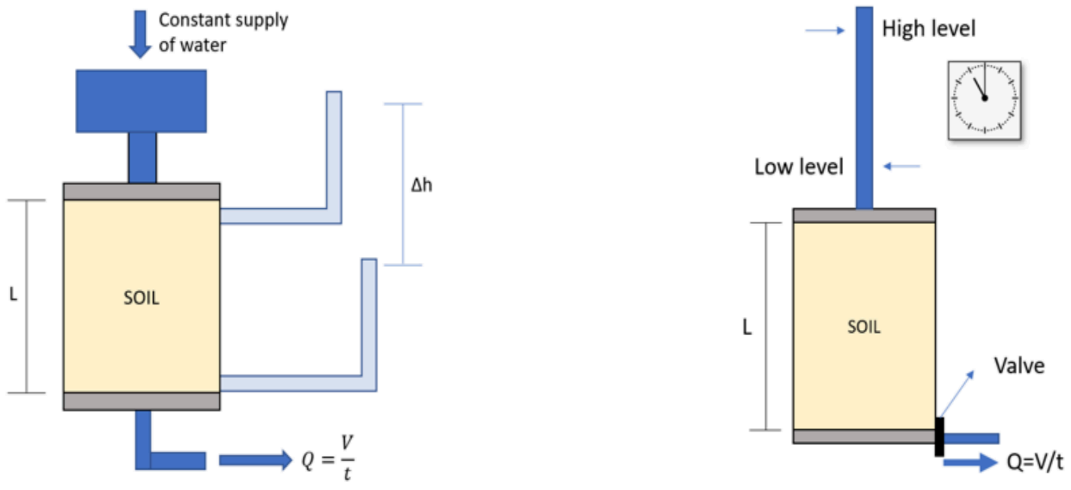
Overall, the results demonstrate that a temperature of 20°C positively affects flotation performance. Sodium sulfite at a concentration of 8 g/kg was found to be more effective than both other concentrations of sodium sulfite and sodium sulfide. Two distinct experiments were performed: one examining the influence of the activator and the other investigating the effect of the collector.

In the experiment focused on the activator, the metal recovery rates were as follows: 15% for sodium sulfite at 2 g/kg, 42.8% for 8 g/kg, and 38% for 15 g/kg concentrations. In contrast, the experiment investigating the impact of the collector resulted in a metal recovery of only 11.6%. This discrepancy in recovery rates can be attributed to the differing frothers utilized in the experiments. MIBC, when used as the frother, yielded superior results compared to OTZ100, particularly for zinc-oxidized ore. Although the highest metal recovery achieved was 42.8%, there was no significant increase in the zinc content of the product. Therefore, it appears that the flotation process may not be effective for low-grade zinc ore.

3.3 Permeability Test

In laboratory settings, two predominant methodologies are employed to evaluate permeability: the constant rate test and the variable rate test. The constant rate test maintains a steady flow of liquid through the material, allowing for the determination of permeability under controlled conditions. In contrast, the variable rate test involves changing the flow rate during the experiment, providing insights into the material's response to differing hydraulic gradients. Both methods contribute valuable data for characterizing the flow dynamics within porous materials and enhancing our understanding of fluid transport mechanisms.

Figure 3.3.1 Constant and Variable Level Experiment Schematics



In a constant-level test, fluid is allowed to percolate through the column by maintaining a consistent water level at the inlet, which is kept equal to the bottom water level, thereby ensuring constant pressure throughout the experiment (see Figure 40-1). Conversely, the variable-rate test, as illustrated in Figure 2, operates on a similar principle to the constant-rate test; however, it differs in that the flow rate of the feedwater is not held constant.

In our laboratory experiments, we employed the constant-level method to determine the permeability coefficient (k) of the ore. This approach allowed us to estimate the permeability effectively by maintaining stable hydraulic conditions, facilitating accurate measurement of fluid flow through the material.

Testing methodology

1. The ore is carefully filled and compacted in the 35 cm long column with a diameter of 12.5 cm as shown in the following picture.

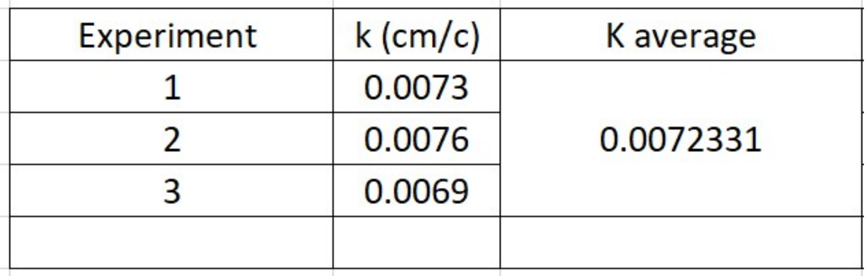
2. The sample loaded in the column was filled with water for about 15 minutes until it was saturated, and the test was started.

3. After starting the experiment, we waited until the water coming out of the bottom of the column became constant.

4. After the outlet water level became constant, the difference in level between the 2 points was recorded.
5. After the level difference became constant, the time it took for the outlet water to fill the 10 mL volumetric cylinder was recorded.

This experiment was repeated 3 times and the results are shown in the following table.

Table 3.3.1 Test results for determination of seepage rate



Experiment	k (cm/c)	K average
1	0.0073	0.0072331
2	0.0076	
3	0.0069	

According to Darcy's law, if the quantity of water flowing through a material per unit of time q is:

$$q = v * A = k * i * A \quad \textit{Formula 3.3.1}$$

Formula 3 Volume of liquid flowing through ore per unit of time

$$v = k * i$$

$$i = \frac{\Delta h}{L}$$

Equation 4 Hydraulic gradient

$$q = \frac{Q}{t}$$

Above :

q - the amount of liquid flowing through the ore per unit of time (volume/time).

k - coefficient of permeability (length/hour).

i - hydraulic gradient.

A - Cross-sectional area of the ore column mass (m^2)

Q - volume of collected water.

Δh - height difference of the water level inside the piezometer tube.

L - the distance between piezometer connections.

t - time of water collection.

Expressing Darcy's law as a coefficient k:

$$\frac{Q}{t} = k * \frac{\Delta h}{L} * A$$

$$k = \frac{QL}{A\Delta ht}$$

Formula 5 to find the coefficient of permeability

Using the table showing the test results and Darcy's law above, the permeability coefficient k can be found.

Table 3.3.2 Permeability coefficient

	k (cm/s)	k average (cm/s)
Test 1	0.0072	0.0072331
Test 2	0.0076	
Test 3	0.0069	

The average permeability coefficient obtained from the experiment can be inserted into Darcy's equation to find the appropriate flow rate for the column leaching test.

$$q = k * \frac{\Delta h}{L} * A$$

Equation 3.3.2 Darcy's equation

But in the case of column dissolution, $\Delta H = L$ or $i = 1$, so the equation becomes $q = k * A$.

A or the cross-sectional area of the column is A = 362.87cm^2

$$q = 0.007233\frac{cm}{s} * 362.87cm^2 = 2.646\frac{cm^3}{s} = 9.44\frac{L}{h}$$

According to this, the flow rate of the solution during the column dissolution is 9.44 l/h, and if the flow rate is lower than that, the test will be conducted normally.

3.4 VAT Leaching Result and Procedure

Feed preparation

The feed is specifically prepared for the flotation process, aiming to produce a product in which 80% of the particles are 74 microns or smaller following grinding. The grinding duration is carefully adjusted to achieve this target particle size, with the grinding time determined when the P80 reaches 74 microns.

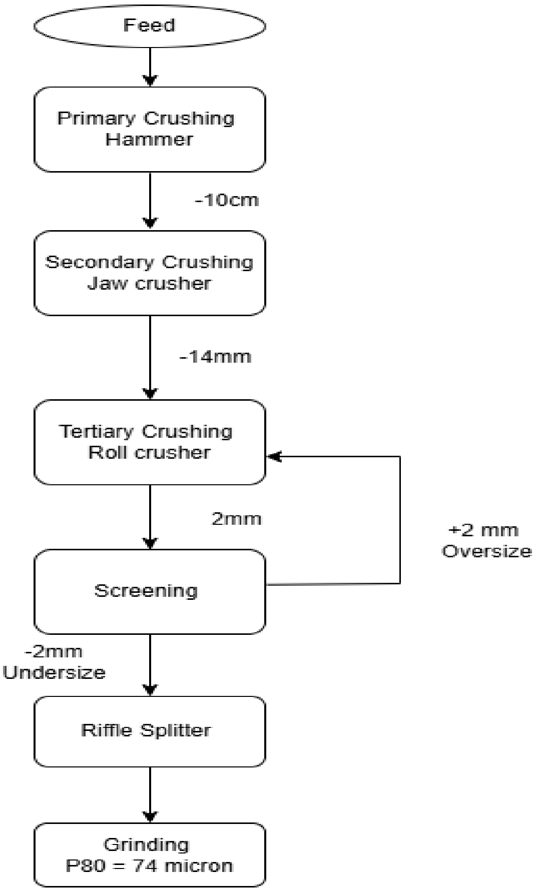
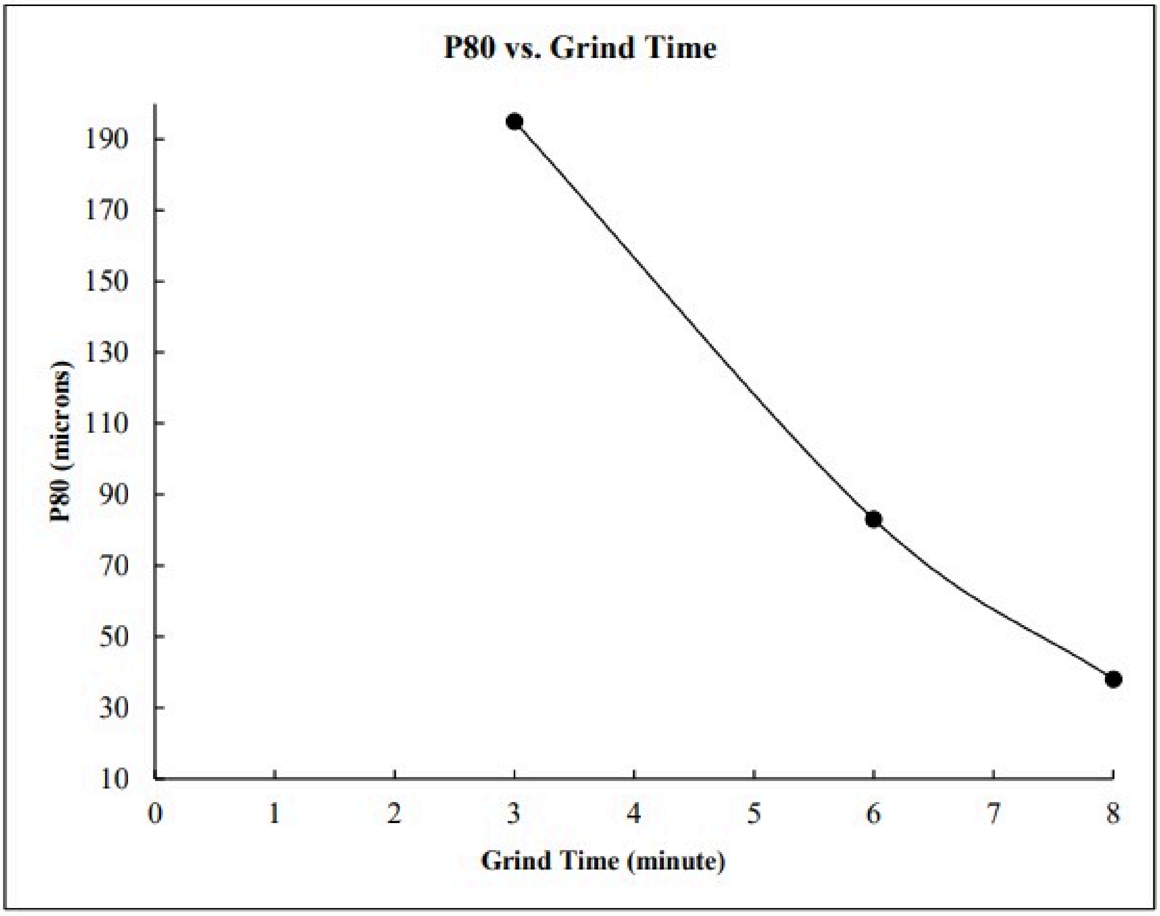


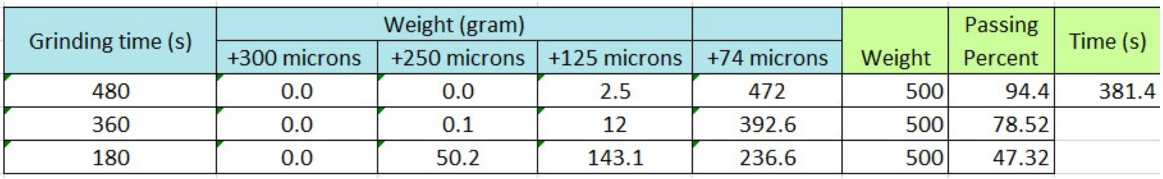
Figure 3.4.1: Working Scheme for Feed Preparation in Vat Leaching

Grinding Result

The grinding duration is optimized to achieve the target particle size, with the grinding time established when the P80 value reaches 74 microns.



Graphic 3.4.1: Time Required for Grinding to Achieve P80 = 84 Microns of Product



Grinding time (s)	Weight (gram)				Weight	Passing Percent	Time (s)
	+300 microns	+250 microns	+125 microns	+74 microns			
480	0.0	0.0	2.5	472	500	94.4	381.4
360	0.0	0.1	12	392.6	500	78.52	
180	0.0	50.2	143.1	236.6	500	47.32	

Table 3.4.1: Sieve Analysis of the Grinding Product

The grinding results demonstrate that a processing time of 381.4 seconds is necessary for the feed material to achieve a P80 of 74 microns. This grinding duration will be utilized in preparing the feed material for the vat leaching process.

Vat leaching Experiment

To identify the optimal concentration of sulfuric acid for maximizing metal recovery in the vat leaching process, sulfuric acid was evaluated at concentrations of 25 g/L, 50 g/L, 100 g/L, 150 g/L, 200 g/L, 250 g/L, and 300 g/L. The table below summarizes the specific conditions employed in each vat leaching test.

- A sample of 500 grams is prepared through a three-stage crushing process and ground until achieving a P80 of 74 microns.
- Various concentrations of sulfuric acid solution are prepared by adding acid to water using a pipette.
- The sample is then placed in a tank, and the prepared solution is added.
- The vat leaching mixer is set to a rotational speed of 1200 RPM.
- After 20 minutes, 50 mL of the pregnant solution is collected.
- The sample is poured onto filtration paper, neutralized, and then sent to the laboratory for elemental analysis following the leaching process.

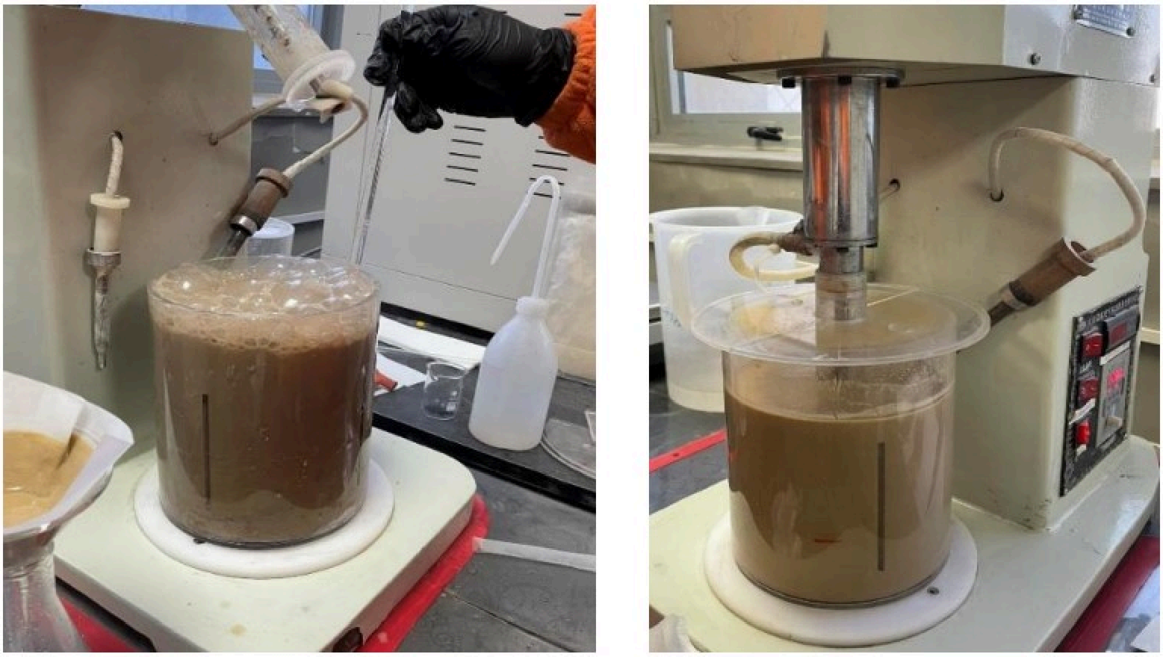
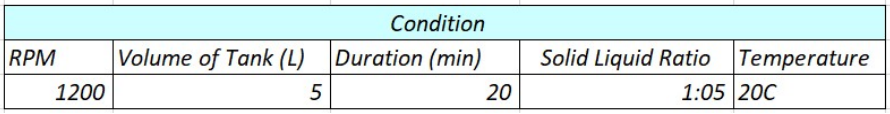
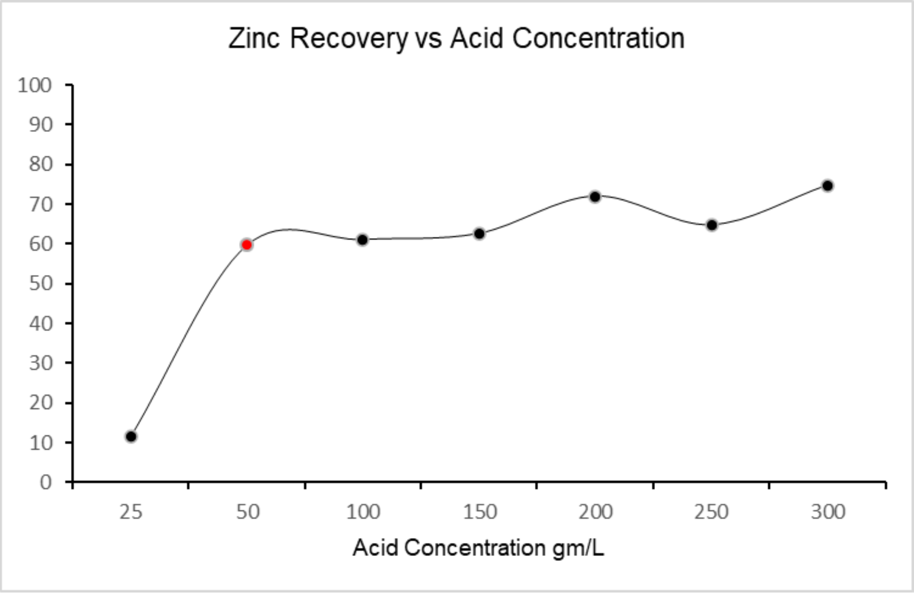


Figure 3.4.2 Vat leaching Experiment



Condition				
RPM	Volume of Tank (L)	Duration (min)	Solid Liquid Ratio	Temperature
1200	5	20	1:05	20C

Table 3.4.2: Conditions for Vat Leaching



Graphic 3.4.2 Zinc recovery at different acid concentrations

Acid Concentration gm/L	*Zinc metal content, %*
25	*11.45*
50	*59.54*
100	*61.07*
150	*62.6*
200	*71.98*
250	*64.77*
300	*74.69*

Table 3.4.3: Metal Content at Various Acid Concentrations

According to the data from *Graph 3.4.2* and *Table 3.4.3*, metal recovery was lowest at a sulfuric acid concentration of 25 g/L, yielding only 11.45%. However, increasing the concentration to 50 g/L resulted in a substantial improvement in recovery, reaching

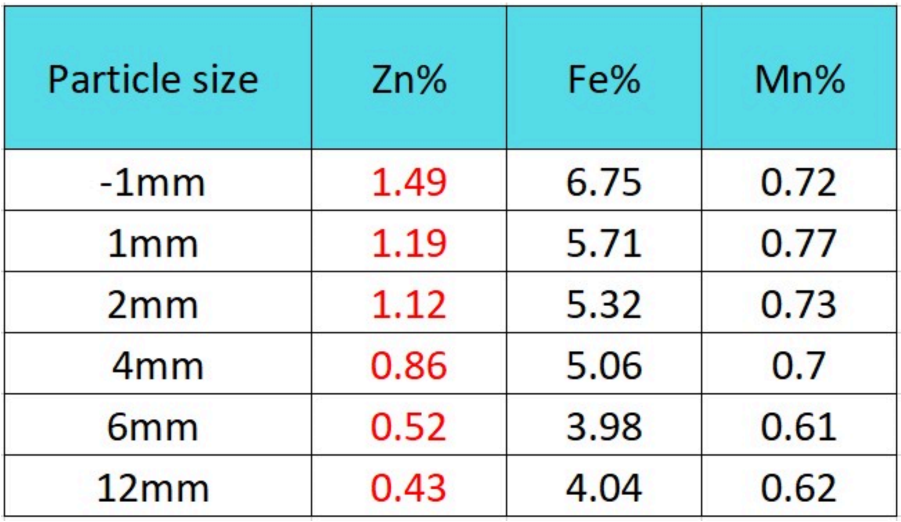
59.54%. Between 100 g/L and 300 g/L, recovery increased gradually, ranging from 61.07% to 74.5%, with the highest recovery observed at 300 g/L.

While the peak recovery occurred at 300 g/L, this concentration is economically unfeasible due to the high cost of sulfuric acid. Therefore, a concentration of 50 g/L is deemed the most appropriate, providing nearly 60% recovery—a result sufficiently close to 74.5% to be considered an efficient and cost-effective option.

3.5 Bottle Roll Leaching Experiment

Feed preparation

A total of 20 kg of zinc ore undergoes a two-stage crushing process. Following this, a screen analysis is performed, and particles ranging from +12 mm to -1 mm are collected for bottle roll leaching to evaluate how particle size affects leaching efficiency. Each 300-gram sample of the collected material is mixed using a riffle splitter to ensure uniform size distribution. A small portion of the collected samples is then analyzed using an XRF device, which provides information on the zinc content and the presence of other elements in the samples. The XRF results in Table 3.5.1 indicate that zinc content increases as particle size decreases.



Particle size	Zn%	Fe%	Mn%
-1mm	1.49	6.75	0.72
1mm	1.19	5.71	0.77
2mm	1.12	5.32	0.73
4mm	0.86	5.06	0.7
6mm	0.52	3.98	0.61
12mm	0.43	4.04	0.62

Table 3.5.1 XRF result

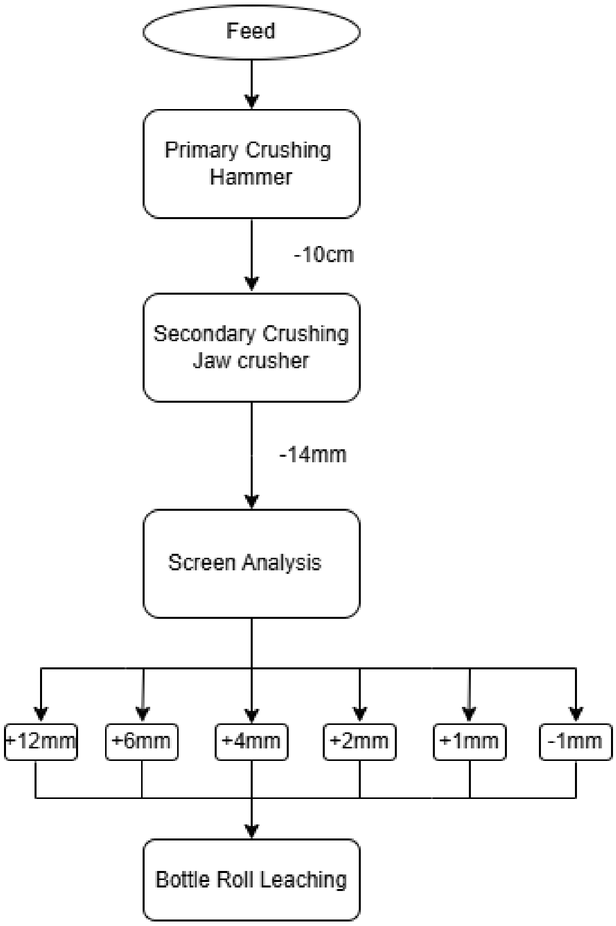
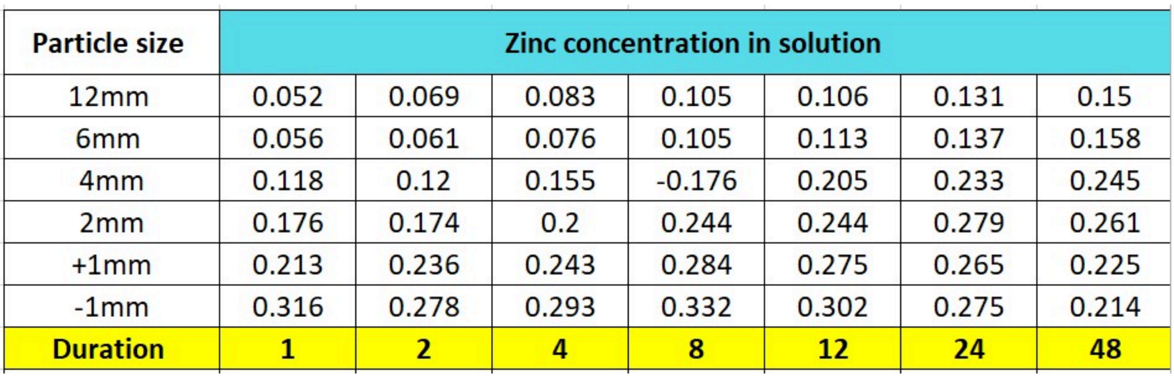


Figure 3.5.1 Feed preparation for bottle roll leaching

Experimental part

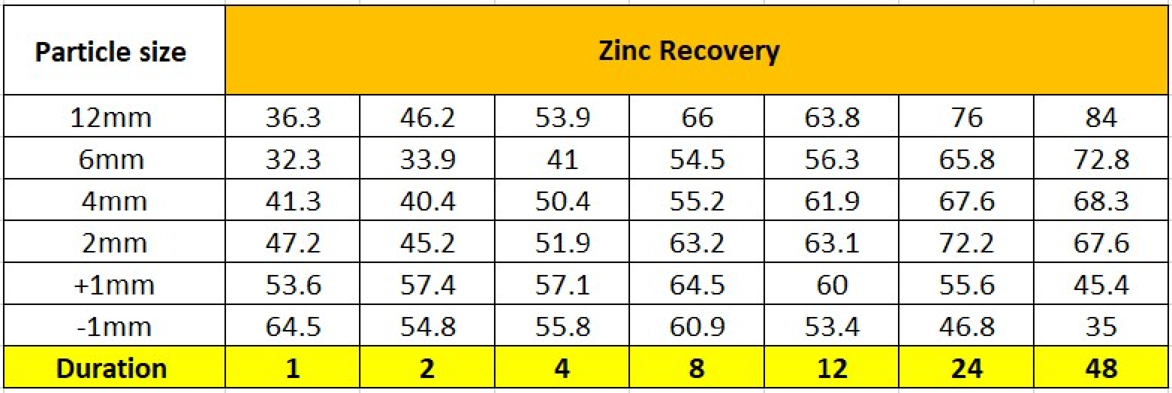
The bottle roll leaching process was performed on 300-gram samples for each particle size, using a solid-to-liquid ratio of 1:3 and a leaching speed of 80 rpm. The experiment ran for a total duration of 48 hours, with 30 ml samples collected at 1, 2, 4, 8, 12, and 48 hours after the process began. These samples were analyzed in the laboratory, and the results, detailing changes in zinc concentration in solution over time, are provided in Table X . The zinc concentration in the pregnant solution initially increases, then gradually decreases over time for the -1 mm particle size. During this process, gas is released from the finer particles, causing slight swelling in the bottle

Figure 3.5.2: Bottle Roll Leaching Procedure



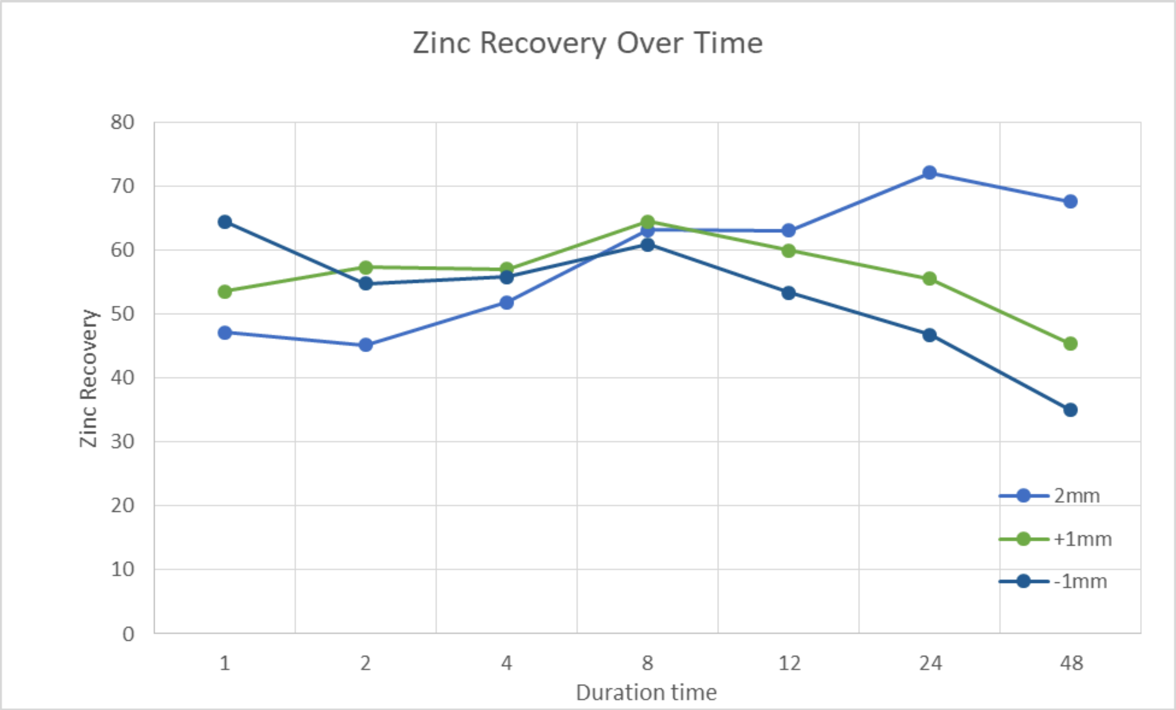
Particle size	Zinc concentration in solution						
12mm	0.052	0.069	0.083	0.105	0.106	0.131	0.15
6mm	0.056	0.061	0.076	0.105	0.113	0.137	0.158
4mm	0.118	0.12	0.155	-0.176	0.205	0.233	0.245
2mm	0.176	0.174	0.2	0.244	0.244	0.279	0.261
+1mm	0.213	0.236	0.243	0.284	0.275	0.265	0.225
-1mm	0.316	0.278	0.293	0.332	0.302	0.275	0.214
Duration	**1**	**2**	**4**	**8**	**12**	**24**	**48**

Table 3.5.2: Zinc Concentration in Solution of Various Particle Sizes at Different Duration Times

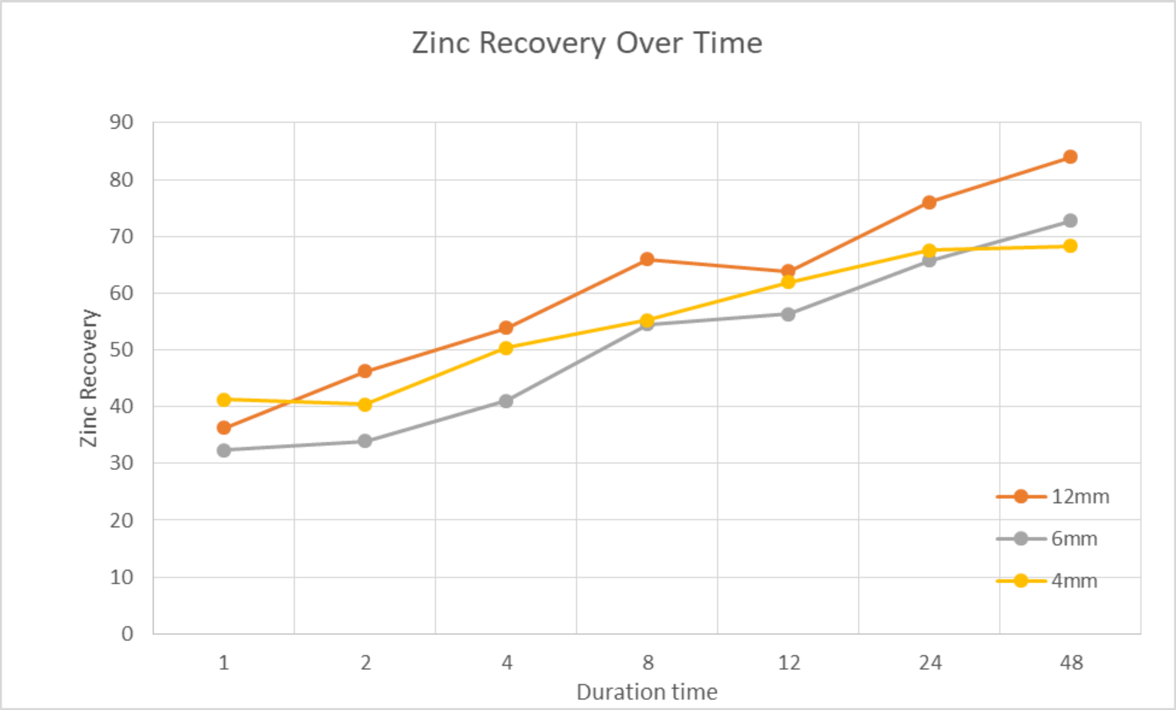


Particle size	**Zinc Recovery**						
12mm	36.3	46.2	53.9	66	63.8	76	84
6mm	32.3	33.9	41	54.5	56.3	65.8	72.8
4mm	41.3	40.4	50.4	55.2	61.9	67.6	68.3
2mm	47.2	45.2	51.9	63.2	63.1	72.2	67.6
+1mm	53.6	57.4	57.1	64.5	60	55.6	45.4
-1mm	64.5	54.8	55.8	60.9	53.4	46.8	35
Duration	**1**	**2**	**4**	**8**	**12**	**24**	**48**

Table 3.5.3: Zinc Recovery of Various Particle Sizes at Different Duration Times



Graphic 3.5.1: Zinc Recovery Versus Duration Time for Leaching Products of -1 mm, 1 mm, and 2 mm Particle Sizes



Graphic 3.5.2: Zinc Recovery Versus Duration Time for Leaching Products of 4 mm, 6 mm, and 2 mm Particle Sizes

3.6 Column leaching

The column in the GMIT laboratory has a height of 2.2 meters, a base height of 10 centimeters, and a diameter of 22 centimeters. In the column leaching process, quartz is positioned at the bottom, occupying the 10-centimeter base. Incorporating quartz at the base of a column leaching system significantly improves both the efficiency and effectiveness of the leaching process. Its characteristics as a stable, inert, and permeable material render it an excellent choice for providing support to the column while optimizing metal recovery.

To ensure the uniform distribution of sulfuric acid in all directions, glass-wool pad is placed at the top of the column, and a JLM pump is utilized for acid pumping.

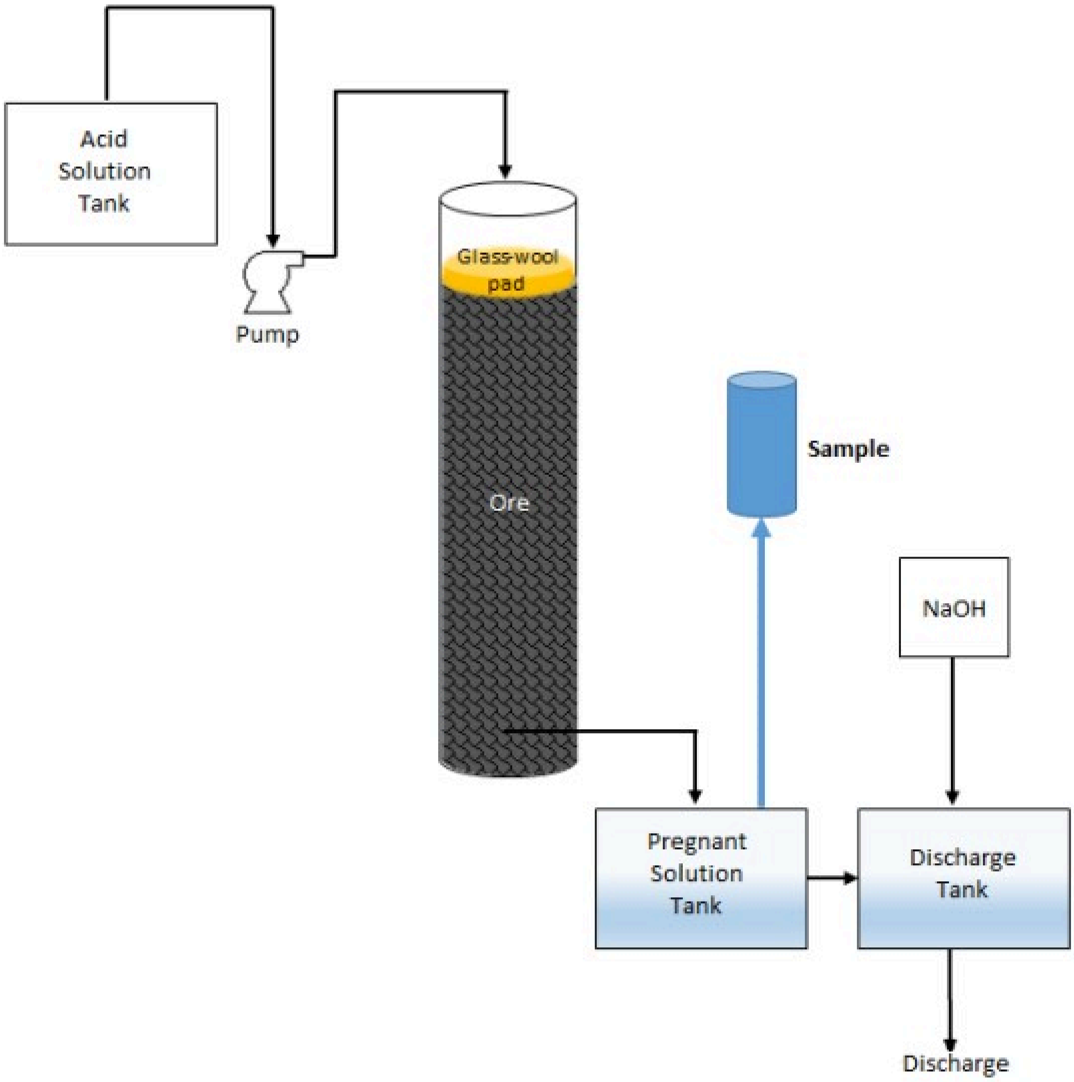
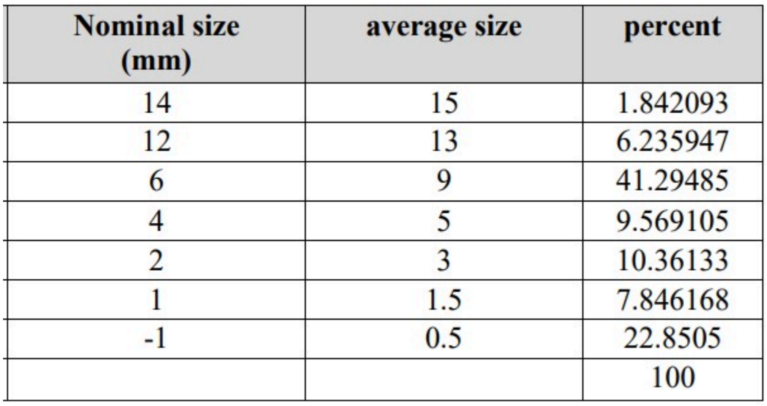


Figure 3.5.1 Column leaching scheme

Feed for Column Leaching

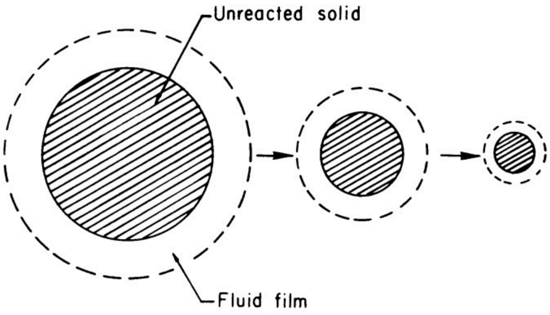
The 160 cm column is filled with zinc ore that weighted 104.1 kg with zinc content of 1.31%. The size distribution of the feed for the column leaching process is presented in Table 3.6.1.



Nominal size (mm)	average size	percent
14	15	1.842093
12	13	6.235947
6	9	41.29485
4	5	9.569105
2	3	10.36133
1	1.5	7.846168
-1	0.5	22.8505
		100

Table 3.6.1 Size distribution of feed to column leaching

3.7 Mathematical Simulation of Column Leaching Design



$$(A)_{fluid} + b(B)_{solid} = c(C)_{fluid} + d(D)_{removable\ solid}$$

Figure 3.7.1 Shrinking Core Model

Shrinking core model is widely used in chemical engineering, this model describes the kinetics of solid-fluid reactions (like leaching and gas-solid reactions) by showing how solid particles react with a fluid, shrinking inward from the surface and leaving an unreacted core.

The kinetics of leaching depend on various experimental parameters, and the table below presents the formulas for each set of conditions.These two formulas are applied and reaction control is defined

	Kinetics $G(a)$	K
Liquid film diffusion	$1 - (1 - a)^{2/3}$	$\frac{2bDC_a}{\rho R_0^2}$
Solid Product Diffusion	$1 - (1 - a)^{2/3} + 2(1 - a)$	$\frac{2bDC_a}{\rho R_0^2}$
Chemical Reaction	$1 - (1 - a)^{1/3}$	$\frac{2bDK_0C_a}{\rho R_0^2}$

Table 3.7.1 Reaction kinetic formula and parameters

$G(a)\ -\ reaction\ controls$

$a\ -\ conversion$ $/\ in\ our\ case\ its\ 50\%\ =\ 0.5/$

$b\ -\ coefficient$

$D\ -\ effective\ diffusion\ coefficient$

$C_a\ -\ concentration\ of\ acid$

$\rho\ -\ density\ of\ ore$

$R_0\ -\ average\ radius\ of\ ore$

Arrhenius equation

$$k = Aexp(-\frac{E}{RT})$$

Equation 3.7.1 Arrhenius equation

$k\ -\ reaction\ rate\ constant$ $A\ -\ pre\ exponential\ factor$

$E - activation\ energy\ \ (J)$

$R - gas\ constant\ \ \ (J/mol * K)$ $\quad T - \ temperature\ in\ Kelvin$

The reaction rate constant is defined by the Arrhenius equation, which demonstrates the dependence of the reaction rate on activation energy and temperature. An increase in activation energy results in a decrease in the reaction rate, indicating that a higher energy input is necessary for the chemical reaction to occur. Conversely, a reduction in temperature leads to a slower reaction rate, as outlined by collision theory, which attributes this to diminished particle collisions. The parameters A (the pre-exponential factor) and E (activation energy) can be determined through the shrinking core model.

Data from vat leaching and bottle roll leaching are utilized as primary datasets to highlight how variations in concentration and particle size influence the leaching process. The latest Arrhenius equation is employed to quantify the reaction rate within these contexts.

For the column leaching , recovery dependency on time equation is expressed as

$$Recovery\ =\ C_{nominal} * (A) * exp(\frac{v_{fluid}}{t_{day} * \rho_{solid}})$$

Equation 3.6.2 Recovery Dependency on Nominal Concentration and Duration Time in Leaching

$v_{fluid} - \ acid\ flow\ rate$

$t_{day} - \ duration\ of\ leaching\ process$

Using this equation, we can determine the zinc recovery at each day of the process based on the acid flow rate and nominal concentration. If we maintain a constant acid flow rate throughout the experiment, the recovery will be contingent upon the leaching time (measured in days). The recovery is linearly correlated with the Arrhenius equation, indicating a dependence on time. As time progresses, the efficiency of the reaction improves. Therefore, the time dependency of the nominal concentration can be expressed as follows:

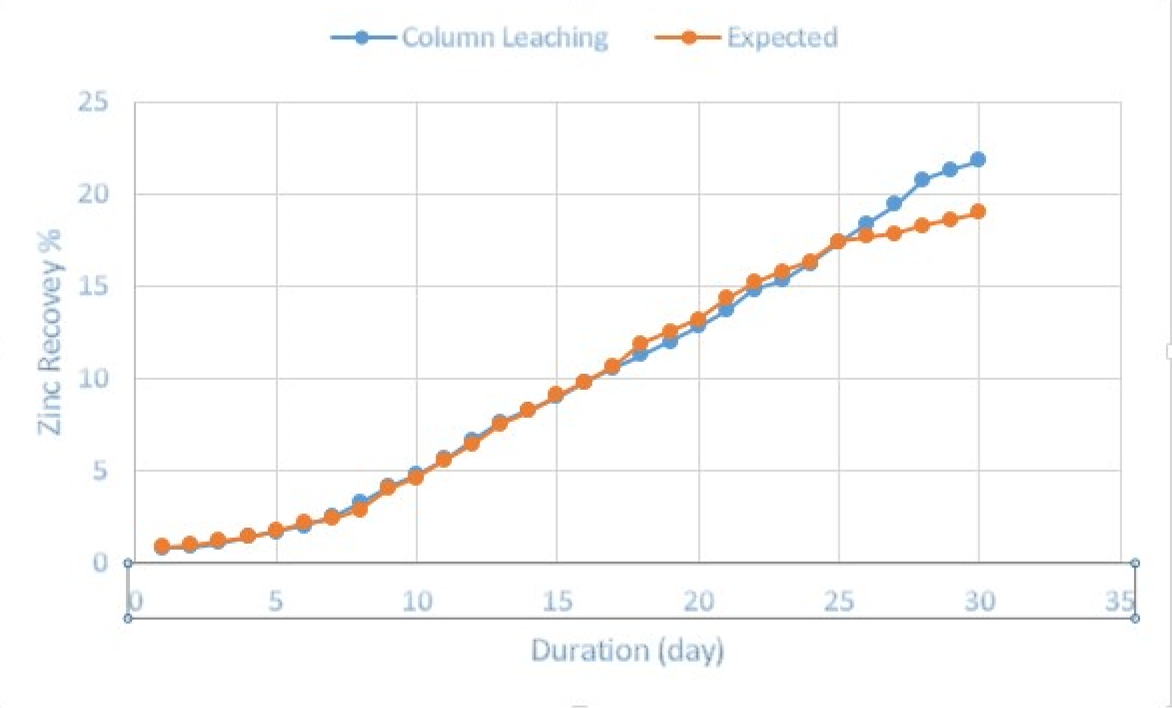
$$C_{nominal} = k_0 \times \frac{\rho_{solid} \times V_{fluid}}{2b} \times C_{actual}$$

Equation 3.6.3: Nominal Concentration Dependence on Reaction Rate Constant

$k_o = k(t)$ *Equation 3.6.4: Rate Constant Variation Over Time*

To minimize acid consumption, we initiated our leaching process with an acid flow rate of 2 liters per hour and a concentration of 6 grams per liter.

Result



Graphic 3.6.2 Comparison of Expected Zinc Recovery with Real-Time Results from Column Leaching

4.Conclusion

My thesis research concentrated on establishing effective conditions for the enrichment of zinc. The zinc content in the ore was determined to be 1.31%. To assess the hardness of the zinc ore, the Bond Work Index was utilized, yielding a result of 7.4 kW/h, which classifies the ore as soft and easy to grind. This information will aid in the selection of the appropriate crusher for processing.

Subsequently, both vat leaching and bottle roll leaching processes were conducted using an optimal concentration of 50 g/L sulfuric acid to achieve higher metal recovery. However, at concentrations of 200 g/L and 300 g/L, gas was released from the leaching solution, accompanied by heat emission. This created challenges in maintaining a consistent reaction temperature, complicating the continuation of the process.

In the bottle roll leaching process, a particle size of 2 mm yielded higher recovery results compared to other particle sizes at the specified duration times. Additionally, gas was released due to the presence of carbonate-containing minerals. This finding contributes to the development of a mathematical simulation model for the leaching of zinc ore.

For the column leaching process, a total of approximately 202.57 kg of zinc ore is required, with Feed-2 exhibiting a higher manganese content. Feed-2 underwent enrichment during the leaching process in Column 2. It is important to note that Feed-2 was not involved in the bottle roll leaching or vat leaching experiments, as those processes were not conducted on this particular feed. Despite both feeds originating from the same location, the mathematical simulation for leaching does not apply to Feed-2 due to insufficient data.

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