



Beneficiation Consequences on the Influence of Potential Hydrogen Variation in the Froth Flotation of Farin-Lamba (Plateau State) Cassiterite

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ABSTRACT

This study explores the influence of pH variation on the froth flotation performance of cassiterite ore obtained from the alluvial deposits of Farin-Lamba, Plateau State. Bulk ore samples were acquired via random sampling across the active mining site and subsequently homogenized to ensure uniformity. A 20 kg head sample was prepared, from which a representative 5 kg sub-sample was subjected to comminution and sieving. The processed material underwent chemical analysis, particle size distribution assessment, and beneficiation through froth flotation under controlled pH conditions. Initial analysis showed a tin oxide (SnO₂) grade of 20.22%. Sieve analysis across various mesh sizes identified -180+125 μ m fraction as the optimal liberation size, recording the highest assay value of 23.28% SnO₂. Accordingly, sieve fractions -250+180, -180+125, and -125+90 μ m were selected for flotation experiments conducted using a Denver D-12 mechanical agitator at 1200 rpm and gas flow of 0.5-1.0 m²/h. Experiments were carried out at pH 5, 7, and 9 to determine the best condition Farin-Lamba cassiterite to be beneficiated. Post-flotation analysis revealed that the highest SnO₂ concentration of 65.62% was achieved at pH 9 within the -180+125 μ m size fraction, indicating this condition as optimal for tin beneficiation from Farin-Lamba cassiterite.

INTRODUCTION

Nigeria is endowed with a diverse range of solid mineral resources, including cassiterite, columbite, lead/zinc, barite, gold, and gypsum, which occur in economically viable grades and quantities (Ayeni et al., 2011). Historically, tin ore (cassiterite) was particularly abundant in Jos, Plateau State, and played a central role in Nigeria's colonial mining economy. Formal tin mining in Plateau commenced in 1904 under the British colonial regime. At its peak, the industry supported railways, education, and hospitals before experiencing a decline following Nigeria's shift to a petroleum-based economy (Foraminifera Market Research Ltd, 2017).

In recent decades, African nations such as Ghana and Tanzania have demonstrated how strategic reforms can revitalize solid mineral sectors (Bridge, 2008). Similarly, Nigeria's Federal Government has embarked on diversification efforts, with renewed focus on non-oil sectors, including solid mineral exploitation. Cassiterite, as a critical raw material for tin production, presents economic potential if properly characterized and processed using efficient beneficiation methods.

The global tin reserve as of 2017 was over 4.80 million tonnes, with Nigeria contributing 2,400 tonnes, predominantly from secondary alluvial deposits (USGS, 2018). Despite a large untapped reserve, tin mining in Nigeria is still largely

artisanal. Farin-Lamba, located in Jos North LGA of Plateau State, is one such region with cassiterite-rich alluvial deposits, yet its ore characteristics remain underreported.

Cassiterite (SnO_2), the primary source of tin, forms through hydrothermal processes and often occurs in association with granitic rocks, pegmatites, and in placer deposits. It contains up to 78.6% tin and is valued for industrial applications ranging from plating and soldering to catalysis and alloys (Ebikemefa, 2020; Tian et al., 2016). Beneficiation techniques such as gravity separation and flotation are essential in upgrading cassiterite to meet industrial purity requirements, with efficiency dependent on the ore's mineralogical and surface chemistry (Wills, 1997).

There exists a gap between tin demand and supply in Nigerian metallurgical industries due to the depletion of high-grade ores and inefficiencies in processing. The limited data on the chemical and mineralogical composition of Farin-Lamba cassiterite ores necessitate this study. Understanding the liberation characteristics and optimal beneficiation conditions, especially with respect to pH, could yield economically viable tin concentrates and reduce processing waste.

This study aims to determine optimal froth flotation parameters for the beneficiation of Farin-Lamba cassiterite at varying pH conditions, alongside its chemical and mineralogical characterization.

The efficiency of cassiterite beneficiation depends on its mineralogical and chemical profile, which varies by deposit. Despite its significance, Farin-Lamba cassiterite remains understudied. This research addresses this gap and supports industrial demand for tin by identifying high-yield, cost-effective processing conditions, particularly across different pH regimes. It also considers secondary applications of gangue material, thereby promoting

environmental and economic sustainability. This study involves sourcing cassiterite ore from Farin-Lamba, Plateau State, followed by homogenization, chemical and mineralogical characterization, liberation size analysis, and beneficiation via froth flotation under controlled pH conditions using particle sizes of 180, 125, and 90 μm .

MATERIALS AND METHODS

Cassiterite samples were collected from ten distinct pits in Farin-Lamba, each excavated to dimensions of 1.0 m \times 1.0 m \times 2.0 m. The samples were thoroughly mixed to achieve homogenization, from which 50 kg was obtained using the cone and quartering sampling technique. A 20 kg portion of the homogenized crude ore was sampled for the research.

The 20 kg sample was ground using a Denver Laboratory Milling Machine (Model D-12) to prepare it for characterization analyses and liberation size determination. For chemical characterization, 20 g of the finely ground sample (passing through a 200-250 mesh sieve) was mixed with 1.0 g of cellulose flakes binder and pelletized under a pressure of 10-15 tons/in².

The pelletized sample was stored in a desiccator prior to analysis. Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRFS, Model: PANalytical Minipal 7) was employed for elemental analysis, with the instrument warmed up for two hours before use. Elemental concentrations were reported in percentage for both major and minor elements.

Mineralogical characterization was conducted using X-ray Diffractometer (XRD), Scanning Electron Microscope equipped with Energy Dispersive Spectroscopy (SEM-EDS), and a petrological analyzer. For XRD analysis, 20 g of the sample was finely ground to pass through a 200-250 mesh sieve. Analysis was performed using a PANalytical X'Pert

Pro powder diffractometer with X'Celerator detector, variable divergence and receiving slits, and Fe-filtered Co-K α radiation. Phases were identified using X'Pert Highscore Plus software, with a receiving slit of 0.040°, a counting range from 5° to 70° on a 2 θ scale, and a count time of 1.5 seconds. Temperature-scanned XRD data were obtained using an Anton Paar HTK 16 heating chamber with a platinum heating strip. Relative phase amounts in weight per cent were estimated using the Rietveld method (Gbadamosi et al., 2021).

Morphological and micro-analytical studies were carried out using an ultra-high-resolution field emission scanning electron microscope (UHR-FEGSEM) equipped with EDS. Pulverized samples were examined using a secondary electron detector. For petrological analysis, 10 g of the sample was impregnated in resin, mounted on slides, and examined under a Leica petrological microscope to determine microstructure and identify mineral constituents.

To determine particle size distribution and liberation size, 100 g of the ground sample was subjected to sieve analysis. Series of sieves ranging from 600 μ m to 63 μ m were arranged in descending order, with the coarsest sieve on top and a receiver below the finest sieve. The sieve stack was placed on an Automated Pascal Denver sieve shaker (Model 16153) and operated for 30 minutes. After sieving, the material retained on each sieve was weighed, and the data were tabulated to determine the economic liberation size (Gbadamosi et al., 2017; Wills, 2006). The work index of the cassiterite ore was determined using the modified Bond's method based on the Gaudin-Schumann expression. Silica ore from the Ibule River side in Ondo State served as the reference material. Both the test and reference ores were pulverized using the Denver Laboratory Milling Machine. Subsequently, 100 g of each sample was subjected to sieve analysis using the

Automated Pascal Denver sieve shaker for 15 minutes. The weight of material retained on each sieve was recorded, and the data were analyzed using the Gaudin-Schumann equations to evaluate the grinding characteristics of the cassiterite ore.

Flotation experiments were conducted in a mechanically agitated flotation cell. Three size fractions of the cassiterite ore (-250+180 μ m, -180+125 μ m, and -125+90 μ m) were selected based on liberation size. For each experiment, 0.5 kg of ore was mixed with 1000 ml of distilled water to form a slurry with a 1:2 ratio. The slurry was agitated for one minute, and the pH was adjusted to 5, 7, or 9 as required. Sodium silicate (0.2 g) was added as a depressant, followed by one minute of agitation. Copper sulfate (0.2 g) was introduced as an activator, with another minute of agitation. Two drops of oleic acid, serving as the collector, were added using a 5 ml syringe, followed by one minute of agitation. Subsequently, two drops of methyl isobutyl carbinol were added as a frother, and the mixture was agitated for one minute. Air was then introduced to the system to generate and stabilize the froth (Alabi, 2020). The froth and tailings were collected separately, allowed to settle for 24 hours, and then decanted. The solid products were filtered and dried in a Carbolite oven (Model OV95C) at 110°C to reduce moisture content to below 5%. The dried samples were weighed, and representative portions were analyzed to determine their chemical compositions.

RESULTS

Table 1 presents the chemical composition of the crude Farin-Lamba cassiterite ore, as determined through Energy-Dispersive X-Ray Fluorescence Spectroscopy (ED-XRFS). The table provides a comprehensive breakdown of the ore's chemical compounds and their corresponding weight percentages.

Table 1: Chemical Composition of the crude Farin-Lamba cassiterite ore via ED-XRFS

Sample/Assay (%)	Al ₂ O ₃	SiO ₂	ZrO ₂	Nb ₂ O ₅	TiO ₂	SnO ₂	MnO	Fe ₂ O ₃
Crude	3.240	52.194	3.774	2.066	3.389	20.221	0.365	5.021

Chemical compositional analysis of Farin-Lamba cassiterite ore using energy dispersive X-ray fluorescence spectroscopy (ED-XRFS)

The result of the chemical composition of the as-received mineral is presented above in Table 1, showing that SiO₂ has the highest chemical compound present, having 52.194% SiO₂ with 5.021% Fe₂O₃, 3.240% Al₂O₃, 3.389% TiO₂, 20.221% SnO₂ and other constituent compounds in trace form. The result obtained further showed that the Farin-Lamba Cassiterite ore does not meet the metallurgical required grade of not less than 60–70% SnO₂, consequently, the need for an upgrade (Alabi et al., 2024). In addition, the chemical composition of the Froth and Depressed samples of Farin-Lamba Cassiterite ore presented in Tables 4 and 5 shows that 65.62% SnO₂, 5.03% Fe₂O₃, 4.46%

SiO₂ and 6.21% SnO₂, 9.06% Fe₂O₃, 60.37% SiO₂, respectively, on sieve size 125 µm and pH 9. This shows that Farin-Lamba Cassiterite is a potential source of Tin oxide and the characterization also reveals that the Cassiterite ore reserve is of commercial quantity. This research further shows that Farin-Lamba Cassiterite ore can be beneficiated to metallurgical grade before utilization for industrial purposes. Flotation performance is most commonly represented using the grade-recovery curves.

Mineralogical characterization analysis of Farin-Lamba cassiterite ore

Figure 1 shows the X-ray diffraction (XRD) pattern of the crude Farin-Lamba cassiterite sample, highlighting the characteristic diffraction peaks of the major minerals present.

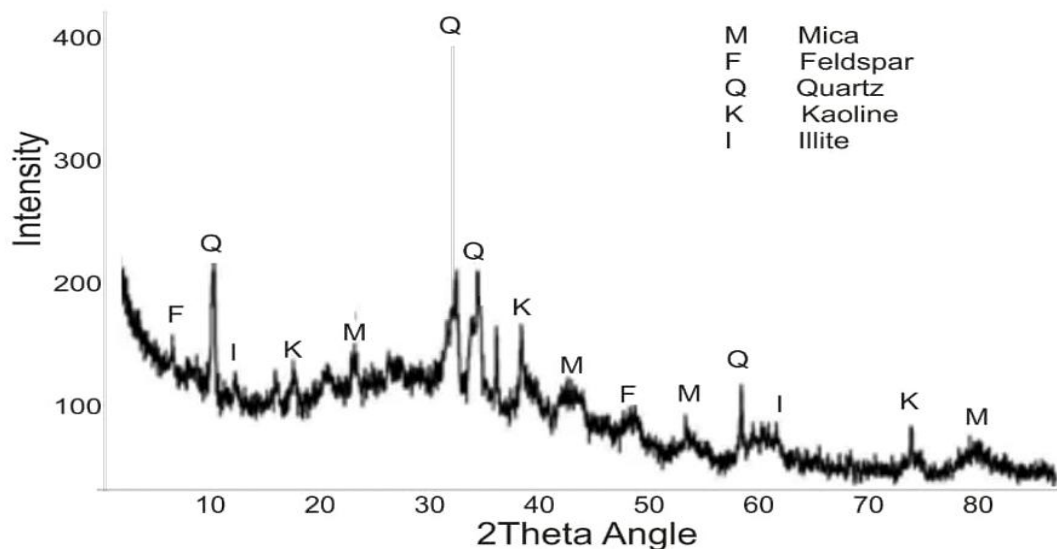


Figure 1: XRD pattern of Farin-Lamba cassiterite crude sample showing the diffraction peaks of the major minerals present

Figure 1 presents the mineralogical association of Farin-Lamba Cassiterite ore via XRD. The

diffractogram reveals the peaks of different minerals present within the Farin-Lamba Cassiterite ore

matrix. The minerals existing are as follows: Mica, Quartz, SnO₂, Kaolin, and Feldspar. From the diffractogram, Farin-Lamba Cassiterite ore was observed to be complex, composed of spessartine quartz. The silica was found to be predominant in

the ore matrix. Therefore, the ore will require extensive beneficiation to obtain a metallurgical grade of Tin oxide. The characterization study therefore affirms that silica is the major gangue which needs to be separated from the ore.

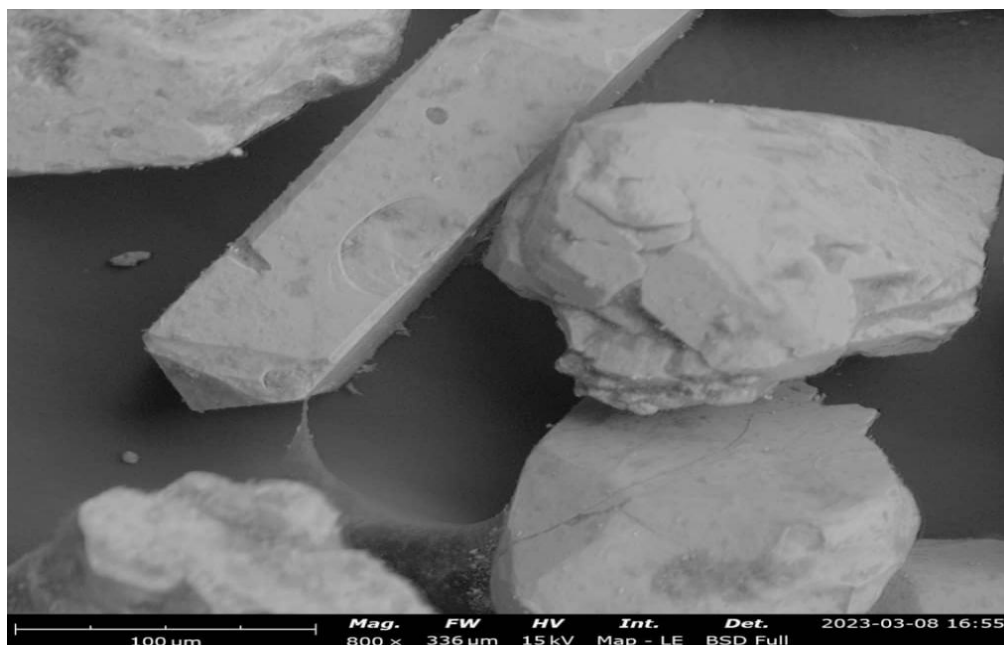


Plate 1: Scanning Electron Microscopy (SEM) images of the crude sample at 100 μm via 800 magnification of the Farin-Lamba cassiterite ore

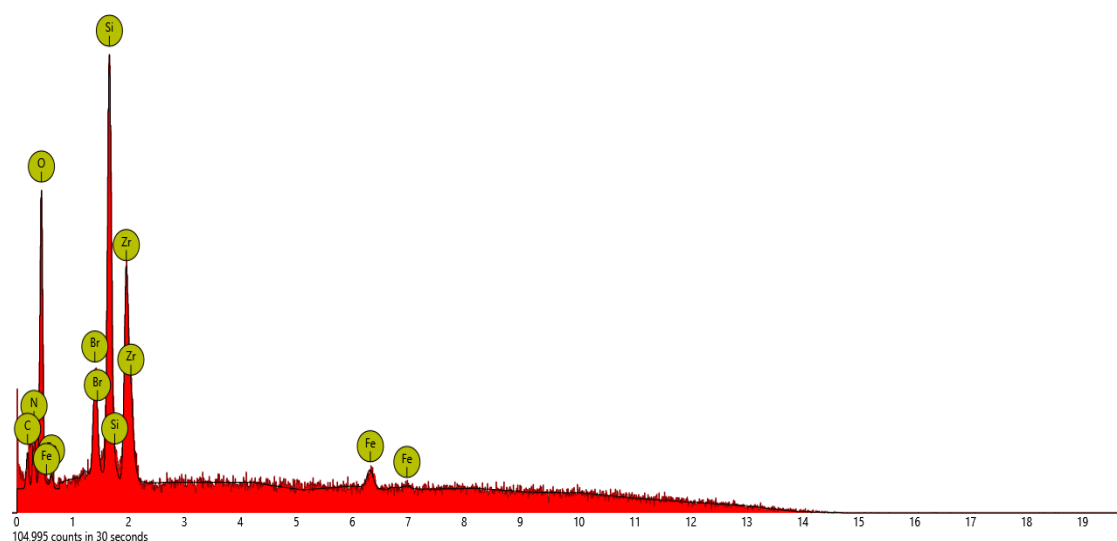


Figure 2: Energy Dispersive X-ray Spectroscopy (EDS) peaks for the various elements present in the crude sample SEM Micrograph

Plate 1 displays the Scanning Electron Microscopy (SEM) image of the crude Farin-Lamba cassiterite sample, showcasing its morphology at a magnification level of 800x at a scale of 100 μm.

Plate 1 reveals the SEM image at 100 μm and Figure 2 shows the EDS qualitative analysis of the Farin-Lamba Cassiterite ore. The SEM morphology shows that the minerals available in the ore matrix are

loosely packed which enhances easy liberation via comminution because of its loose nature. Figure 2 displays the Energy Dispersive X-ray Spectroscopy (EDS) spectrum of the crude Farin-Lamba cassiterite sample, showing the characteristic peaks corresponding to the various elements present, providing a qualitative and quantitative analysis of the elemental composition.

The EDS qualitative analysis of the image certainly revealed the presence of Br, Fe, O, Zr, C, N, and Si.

The point analysis carried out further reveals that oxygen, iron and silica are the predominant elemental constituents of minerals in the ore matrix. While SnO_2 is present, because of the population of silicon in the matrix, it falls below the detectable level. Plate 2 presents photomicrographs of polished sections of the Farin-Lamba cassiterite crude ore, captured under plane-polarized light. The image reveals the ore's mineralogical features, with heavy and light minerals labelled.

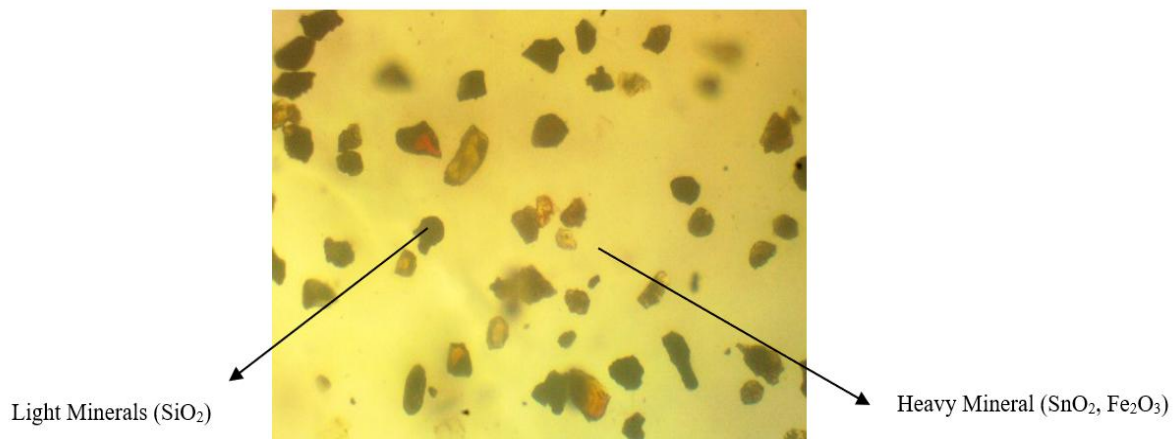


Plate 2: Photomicrographs of polished Farin-Lamba cassiterite crude ore under plane-polarized light

Plate 2 presents the petrological analysis carried out on the crude of Farin-Lamba Cassiterite ore, showing the presence of heavy and light minerals that are reflective or opaque in nature when they interact with light rays. Photomicrograph indicates the segregation distribution of the heavy mineral (Sn, Fe) and the associated mineral, like silica. The photomicrograph also shows a non-evenly distributed heavy mineral and its associated minerals. The results obtained are in line with the analysis done on the crude sample using Energy Dispersive X-ray Fluorescence Spectrometer (ED-XRFS), X-ray Diffractometer (XRD) and Scanning Electron Microscope (SEM) equipped with EDS detector which reveals the presence of minute Tin oxide in Cassiterite which is the mineral of interest in the Farin-Lamba Cassiterite ore, where all other associated minerals will be removed via

beneficiation process. Table 2 presents the result of the fractional sieve size analysis of crude Farin-Lamba cassiterite ore.

Size/assay analysis and liberation size determination of Farin-Lamba cassiterite ore

Table 2 presents the fractional sieve size analysis of crude Farin-Lamba Cassiterite ore. It can be observed that 28.86 g of the total 100 g was retained on sieve size 500 μm , (17.28 g on 355 μm), (21.15 g on 250 μm), (8.01 g on 180 μm), (15.24 g on 125 μm), (6.97 g on 90 μm), (1.78 g on 63 μm), and (0.71 g on -63 μm). The cumulative percentage passing following the order of sieve arrangement from the most coarse - 71.14%, 53.86%, 32.71%, 24.70%, 9.46%, 2.49%, and 0.71%. Table 2 also shows the chemical analysis of the ore sample conducted on all the sieve size fractions.

Table 2: Fractional Sieve Size Analysis of crude Farin-Lamba Cassiterite ore

Sieve size range (μm)	Weight retained (g)	% Weight retained (g)	Cumulative weight retained (g)	Cumulative weight passing (g)	SnO ₂ content (%)
-600+500	27.78	28.86	28.86	71.14	20.17
-500+355	16.63	17.28	46.14	53.86	20.14
-355+250	20.36	21.15	67.29	32.71	20.18
-250+180	7.71	8.01	75.30	24.70	21.33
-180+125	14.67	15.24	90.54	9.46	23.28
-125+90	6.71	6.97	97.51	2.49	21.43
-90+63	1.71	1.78	99.29	0.71	20.51
-63	0.68	0.71	100	0.00	20.16

The result in its elemental form also shows the percentage composition of Cassiterite in oxide and metal constituents in each of the sieve sizes, which is as follows: -600+500 μm (20.17% SnO₂), -500+355 μm (20.14% SnO₂), -355+250 μm (20.18% SnO₂), -250+180 μm (21.33% SnO₂), -180+125 μm (23.28% SnO₂), -125+90 μm (21.43% SnO₂), -90+63 μm (20.51% SnO₂), and -63 μm (20.16% SnO₂). The actual liberation size of mineral of interest (Cassiterite) could be achieved at a sieve size fraction of -180+125 μm with a percentage

assay of 23.28% SnO₂ being the highest percentage of Cassiterite when compared to other sieve sizes. This indicates that a reasonable quantity of Sn can be obtained if the ore is ground to -180+125 μm when compared to other sieve fractions. Hence, -180+125 μm is the actual liberation sieve size of Farin-Lamba Cassiterite ore. Figure 3 shows the graph (log-log plot) of percentage cumulative weight retained and passing against sieve sizes (μm) of the fractional analysis of Farin-Lamba cassiterite ore.

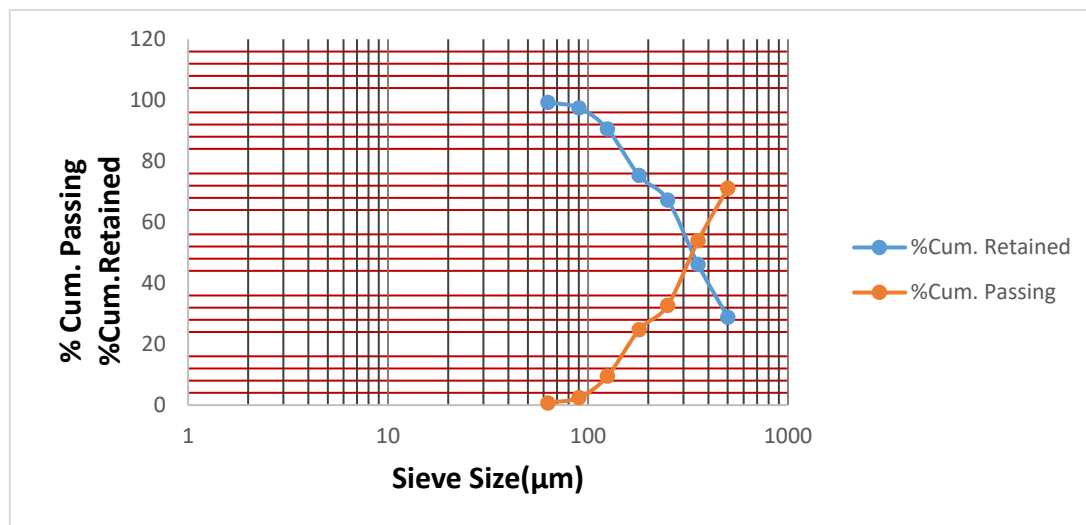


Figure 3: A plot of log-log of percentage cumulative weight retained and passing against sieve sizes (μm) of the fractional analysis of Farin-Lamba cassiterite ore

Figure 3 shows that the two curves, which are mirror images of each other, intersect at -500+355 μm , which indicates the theoretical liberation size of Farin-Lamba Cassiterite ore. Therefore, -180+125

μm is the liberation size for Farin-Lamba Cassiterite. Tables 3–5 show the results of the froth flotation beneficiation process of the Farin-Lamba cassiterite ore using the Denver Flotation Cell.

Table 3: Yield Results of Froth Flotation Process

pH μm	9.0		7.0		5.0	
	Froth (g)	Depressed (g)	Froth (g)	Depressed (g)	Froth (g)	Depressed (g)
180	75.76	424.24	20.41	479.59	15.15	484.84
125	31.25	468.75	40.82	459.18	42.11	457.89
90	38.46	461.54	56.71	443.29	126.26	373.74

Table 4: Chemical Composition of the Froth Processed Samples of Farin-Lamba Cassiterite Ore

pH μm	5.0			7.0			9.0		
	SnO ₂	Fe ₂ O ₃	SiO ₂	SnO ₂	Fe ₂ O ₃	SiO ₂	SnO ₂	Fe ₂ O ₃	SiO ₂
90	57.90	5.86	8.15	57.92	5.17	9.17	58.90	5.81	7.15
125	56.15	5.60	9.21	56.07	5.71	9.35	65.62	5.03	4.46
180	54.85	5.42	8.26	54.14	5.75	8.04	59.11	5.90	5.17

Table 5: Chemical Composition of the Depressed Processed Samples of Farin-Lamba Cassiterite Ore

pH μm	5.0			7.0			9.0		
	SnO ₂	Fe ₂ O ₃	SiO ₂	SnO ₂	Fe ₂ O ₃	SiO ₂	SnO ₂	Fe ₂ O ₃	SiO ₂
90	6.27	6.82	58.24	6.27	8.35	54.92	7.04	6.63	54.87
125	6.37	5.28	59.27	6.13	7.23	59.32	6.21	9.06	60.37
180	6.87	8.70	60.31	6.94	8.91	60.01	7.21	6.54	59.86

Tables 6 and 7 show the metallurgical accounting balance of the froth flotation process carried out on Farin-Lamba cassiterite ore at varying sieve size

ranges and pH values for the resulting products (Froth and Depressed, respectively).

Table 6: Metallurgical Accounting Balance for the Froth Sample

Sieve sizes (μm)	pH	Feed weight (g)	Feed assay (%)	Concentration weight (g)	Concentration Assay (%)	Recovery Cc/Ff	Enrichment ratio, c/f	Concentration ratio, f/c
90	5	500	20.22	126.26	57.90	72.30	2.86	3.96
	7	500	20.22	56.71	57.92	32.49	2.86	8.82
	9	500	20.22	38.61	58.09	18.74	2.87	12.95
125	5	500	20.22	42.11	56.07	23.39	2.78	11.87
	7	500	20.22	40.82	56.15	22.64	2.77	8.92
	9	500	20.22	31.25	56.07	17.33	3.22	16.00
180	5	500	20.22	15.15	65.11	9.76	2.713	33.00
	7	500	20.22	20.14	54.14	10.78	2.67	24.15
	9	500	20.22	75.76	59.11	44.29	2.92	6.60

Table 7: Metallurgical Accounting Balance for the Depressed Sample

Sieve sizes (μm)	pH	Feed weight (g)	Feed assay (%)	Concentration weight (g)	Concentration Assay (%)	Recovery Cc/Ff	Enrichment ratio, c/f	Concentration ratio, f/c
90	5	500	20.22	373.74	6.27	23.18	0.31	3.22
	7	500	20.22	463.29	6.27	28.73	0.31	3.22
	9	500	20.22	461.54	7.04	32.14	0.35	2.87
125	5	500	20.22	457.89	6.37	28.85	0.32	3.17
	7	500	20.22	459.18	6.13	27.84	0.30	3.29
	9	500	20.22	468.75	6.21	28.79	0.31	3.26
180	5	500	20.22	484.84	6.87	32.95	0.34	2.94
	7	500	20.22	479.59	6.94	32.92	0.34	2.91
	9	500	20.22	424.24	7.21	30.25	0.36	2.78

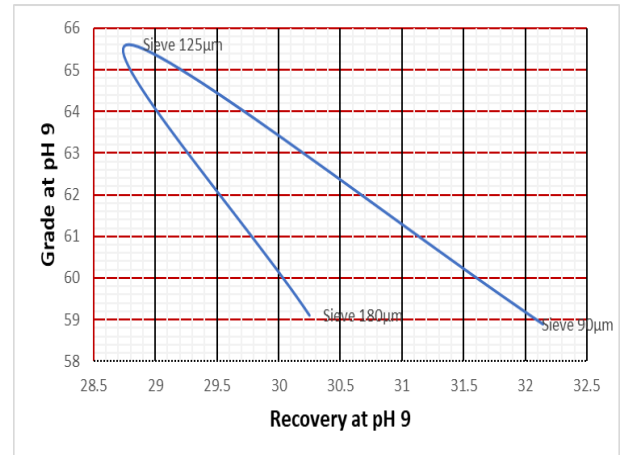
Froth flotation separation method of the Farin-Lamba cassiterite ore using Denver flotation cell

Tables 3-5 reveal the yield and the chemical characterization of the processed (Froth and Depressed) samples of the froth flotation process. From the result in Table 3, it was observed that the yield for the resulting products for the Froth was (15.15 to 126.26 g) compared to that of the yield of the resulting products of the Depressed throughout the process, ranging from 373.74 to 484.84 g. Table 4 shows the chemical composition of the Froth and the economical liberation size (-180+125 μm) has the highest assay of SnO_2 , which is 65.62% when compared to other sieve size fractions. This shows an improvement in the percentage assay of the Farin-Lamba Cassiterite ore present in this mineral. In Table 5, it was observed that there was an increase in the assay of SiO_2 for the entire sieve size fractions and there was also a decrease in the assay of SnO_2 depressed compared to the assay of the froth. Figure 4 shows the grade–recovery curves of Farin-Lamba cassiterite ore at varying pH values (9, 7, and 5, respectively).

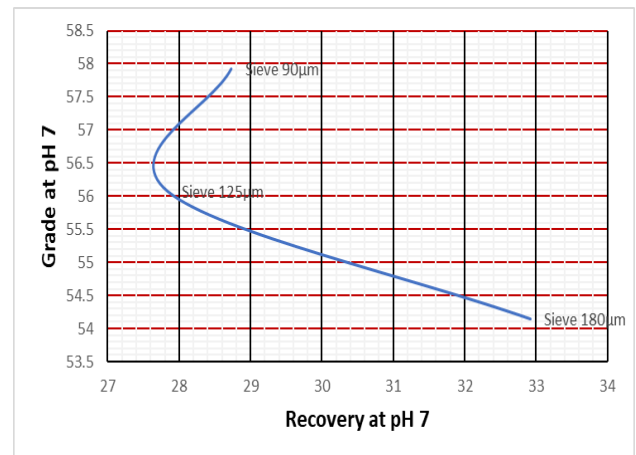
Metallurgical accounting balance and grade-recovery analysis

Tables 6 and 7 present the metallurgical accounting balance for the Froth and Depressed samples, respectively. From Table 6, it was observed that the concentration, weight, assay, and recovery of the froth samples at different sieve sizes and pH conditions varied considerably. The highest concentration assay (65.11% SnO_2) was obtained at sieve size 180 μm and pH 5.0, with an enrichment ratio of 2.713 and a concentration ratio of 33.00. This suggests optimal upgrading of SnO_2 at these conditions. Similarly, Table 7 shows that the depressed samples had very low SnO_2 assays (mostly around 6-7%) across all sieve sizes and pH values, with relatively low recovery values,

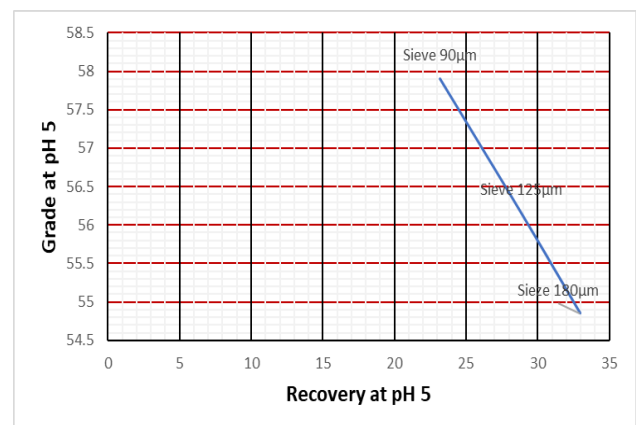
confirming that SnO_2 was effectively floated into the froth fraction during beneficiation.



(a)



(b)



(c)

Figure 4: Grade–Recovery curve of Farin-Lamba cassiterite at pH 9, 7, and 5, respectively

Metallurgical accounting balance and grade-recovery analysis

Tables 6 and 7 present the metallurgical accounting balance for the Froth and Depressed samples, respectively. From Table 6, it was observed that the concentration, weight, assay, and recovery of the froth samples at different sieve sizes and pH conditions varied considerably. The highest concentration assay (65.11% SnO₂) was obtained at sieve size 180 µm and pH 5.0, with an enrichment ratio of 2.713 and a concentration ratio of 33.00. This suggests optimal upgrading of SnO₂ at these conditions. Similarly, Table 7 shows that the depressed samples had very low SnO₂ assays (mostly around 6-7%) across all sieve sizes and pH values, with relatively low recovery values, confirming that SnO₂ was effectively floated into the froth fraction during beneficiation. Furthermore, Figure 4 presents the grade-recovery curves of Farin-Lamba Cassiterite ore at pH values 5, 7, and 9. It was observed that at pH 9, the grade of SnO₂ in the froth was highest, correlating with better metallurgical performance in terms of achieving higher-grade Tin oxide concentrate. The trend also shows that a higher pH favours a better grade-recovery balance during flotation of Farin-Lamba Cassiterite ore.

CONCLUSION

The preliminary studies on the process design of Farin-Lamba Cassiterite has helped to provide ample information about the Ore, which will greatly help in its exploitation. The following were concluded from the studies carried out. The chemical characterization of the crude cassiterite using XRF shows that SiO₂ is dominant with the present of mineral of interest to the tune of 20.221% SnO₂ and other impurities. The chemical analysis of the froth revealed that the sieve size of -180+125 µm at pH of 9 has the highest assay of 65.62% SnO₂.

Hence, this froth flotation condition is recommended.

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