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INFLUENCE OF VARIED AGITATION TIME ON UPGRADING ITAKPE IRON ORE DUMPED TAILINGS USING REVERSED FROTH FLOTATION METHOD

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ABSTRACT

The effect of varied agitation time on the upgrading of Itakpe iron ore dumped tailings via reverse froth flotation technique was investigated. Chemical and mineralogical characterization of the crude sample of Itakpe iron ore dumped tailings was carried out using Energy Dispersive X-Ray Fluorescence Spectrometry (ED-XRF), X-ray Diffraction (XRD) and Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDX). Samplesused were sourced from different tailing dump sites at National Iron Ore Mining Company (NIOMCO) in Itakpe, Kogi State, Nigeria and properly homogenized. Liberation size was determined to be -250+180 μm through fractional sieve size at a sieve range of +500 μm to -63 μm. 250 g of the prepared sample was frothed in a Denver D-12 Flotation Cell using froth floatation reagents while varying agitation time at an interval of 60 seconds, followed by chemical analysis of the froth and depressed at different agitation times. Chemical analysis of the crude shows that it contains 27.71% Fe_2O_3, 70.1% SiO_2, and other trace impurities. XRD pattern of the crude sample revealed present of Quartz (68.41 % (SiO_2), Rutile (0.79 % TiO_2), Hematite (20.05 % Fe_2O_3), Magnetite (1.21 % Fe_3O_4), Biotite-1M (1.02 % KNaFeF), and Albite (8.52 % Na ($AlSi_3O_8$)) while SEM imaging showed interlocking nature of some minerals within the crystal aggregates. The EDX shows that silicon and Iron are the major elemental constituents of the ore matrix. However, analysis of the froth revealed that optimum assay was achieved at an agitation time of 60 seconds; and as such chosen as the best agitation time.

Keywords: Itakpe Iron-Ore, Tailings, Characterization, Reversed Froth-floatation, Agitation time

1. INTRODUCTION

Nigeria is endowed with abundant iron ore deposits of which some have been investigated and some are still under investigation (Also and Yakubu, 1995). Iron ore is the major raw material in the iron and steel industry and as such tremendous development in civilization has been attributed to the utilization of these metals. Iron ore occurs as iron oxide [magnetite (Fe_3O_4) and hematite (Fe_2O_3)], hydro-oxide [goethite ($HFeO_2$) and Limonite ($2Fe_2O_3 . xH_2O$), and carbonates [siderite ($FeCO_3$)]. The Nigerian iron ore deposits fall into two (2) categories depending on their mode of occurrence; firstly, the sedimentary Oolitic/Pisolitic type in which the mineral occurs as an aggregate of rounded pellets either small or large; and secondly, deposits of cretaceous recent years age and of meta-sedimentary banded iron ore formation of the Precambrian (early years) age (Famuboni, 1990); as such Itakpe iron ore deposit falls under this category. This deposit has geological coordinates of latitude 07°36’20”N and longitude 6°18’35” E and it is located at Okehi Local Government Area of Kogi State, Nigeria. The deposit is a magnetite-hematite mineralization composed of fourteen (14) ore strata of economic value predominantly assaying 14.8% - 41% Fe with an overall average of 36% Fe (NIOMC Project Report, 1980; Gbadamosi*et al.*, 2017). The Itakpe iron ore deposit has an estimated reserve of about 200 million tonnes and as such the National Iron Ore Mining Company, Itakpe charged with the mining and processing of this mineral adopt open-pit mining exploration method, and so far, it is being processed using magnetic and gravity methods of mineral processing while the flotation plant is being constructed for future beneficiation of the iron (NIOMC Project Report, 1980). The deposit is being beneficiated at a rate of 8 million tonnes per year to yield 64% Fe concentrate as sinter material for Ajaokuta blast furnace and 68% Fe concentrate as pellet feed for Aladja Direct Reduction Iron plant while leaving out large quantities of siliceous residues called tailings (Alabi O.O., 2016; Gbadamosi*et al*., 2017). These tailings occupy large land mass and as such mitigate the number of viable lands that can be maximized for other purposes such as construction and agriculture. Researches have shown that these siliceous residues contain viable grade of iron content worth being beneficiated and extracted for profit (Adepoju and Olaleye, 2001; Alabi O.O., 2016; Gbadamosi*et al*., 2017). Thus, the need for drastic methods which can proffer solution to the land underutilization and environmental nuisance posed by these tailings cannot but be overemphasized. Therefore, a method such as reverse froth floatation is being investigated in order to recover these lost valuable minerals towards maximizing the productivity of iron concentrates required for the production of iron. Also, the more siliceous tailings gotten afterward are better utilized for construction, paving or backfilling purposes instead of constituting nuisance of viable land mass.

1.1 Froth Flotation Method

Froth flotation is a process used to separate minerals, suspended in liquids, by selectively attaching them to gas bubbles. Hence, in flotation, there exists a three-phase system. Froth flotation utilizes the differences in physicochemical surface properties (hydrophobicity or hydrophilicity) of various minerals to facilitate separation (Kawatra*et al*., 2001). After treatment with reagents, such differences in surface properties between the minerals within the flotation pulp become apparent. For flotation to take place, an air bubble must be able to attach itself to a particle and lift it to the water surface (Guardin, 1957). The process can be applied to relatively fine particles. Depending on which mineral is frothed or depressed, froth flotation can be classified as Direct or Reverse. In direct froth floatation, the iron-bearing minerals are activated and frothed while silicates are depressed; In Reverse Flotation, silicates are activated and frothed while the iron-bearing minerals are depressed (Wills, 2006; Gbadamosi*et al*., 2017).

The reagents employed in flotation selectively alter the physico-chemical properties of the mineral such as interfacial surface tension modifier and surface chemistry. Usually, these are classified as collectors, frothers and modulating agents (Wills, 2006).

1.1.2 Froth Flotation Reagents and Their Uses

i. The froth flotation reagents utilized at the course of Iron ore froth flotation are as listed:

i. Collectors: are reagents that are absorbed on the mineral surfaces to facilitate hydrophobicity and allows mineral particles to attach to air bubbles via which they are transported to the surface;

ii. Frothers: are heteropolar organic reagents capable of being adsorbed on the air-water interface and reduce the surface tension to form a stable air-bubble. Examples include: Pine oil, cresylic acid, and MIBC;

iii. Activators: are chemical compounds which attach onto the mineral surface thus altering its chemical nature to promote the adsorption of collector species;

iv. Depressants: are chemical compounds which alter the mineral surface to inhibit or retard the action of collectors. They are required to depress certain minerals to aid the selective flotation of the desired minerals;

v. pH regulators: are used to control the acidity and alkalinity level of the slurry towards selective separation of the minerals (Araujo *et al*., 2005; Filippov*et al*., 2010c).

2. MATERIALS AND METHOD

2.1 Material Sourcing

Samples of the iron ore dumped tailings were sourced from different tailing dump sites at National Iron Ore Mining Company (NIOMCO) Itakpe, Kogi State, Nigeria, homogenized and 50 kg was weighed out. 5 kg crude sample was sampled out via cone and quartering method and dried at 105 ^{0}C in a Carbolite Oven for 24 hours to reduce its moisture content 0.001% H_2O. The sample was subjected to hand picking and sieving to remove coarse aggregates and unwanted materials, crushed, properly homogenized, and prepared towards analysis.

2.2 Chemical Characterization of Crude

Energy Dispersive X-ray Fluorescence Spectrometer, model: PANanalyticalMinipal 7 was adopted to chemically characterize the crude sample. 20 g of the sample was pulverized to pass through a 200-250 mesh sieve, properly mixed with a cellulose flakes binder in the ratio of 5:1 Respectively, and pelletized at a pressure of 10-15 tons/inch^2 in a pelletizing machine. The pelletized sample(s) were stored in a desiccator for analysis. The ED-XRF machine was switched on and allowed to warm up for 2 hours. Finally, appropriate programs of various elements of interest were employed to analyze the crude sample(s) to detect their presence or absence. Result of the analysis was either reported in percentage (%) for minor and major concentrations of elements.

2.3 Mineralogical Characterization of Crude

Mineralogical characterization of the crude was carried out via XRD and SEM-EDX analysis.

2.3.1 XRD Analysis of Crude

Qualitative and Quantitative determination of the nature of the phases and the amount of the phases present in the sample were determined by a PANalytical Empyrean diffractometer with PIXcel detector and fixed slits with Fe filtered Co-Kα radiation. The material was prepared for XRD analysis using a backloading preparation method. The phases were identified using X'PertHighscore plus software. The relative phase amounts (%weight) were evaluated using the Rietveld method.

2.3.2 SEM-EDX Analysis of Crude

Morphological and qualitative analyses of the bulk ore were performed using SEM-EDX. The SEM provided information on the physical properties of minerals, while EDX provided information on their chemistry. Scanning Electron Microscopy studies for mineral analysis of representative samples were conducted in two stages using SEM model JEOL 840. Samples for analysis were cut, mounts in embedded epoxy resin, and finally polished to obtain a mirror-like surface. The polished surfaces were finally carbon coated before analysis in order to make the mineral's surface conductive. EDX detector attached to the SEM was employed to carry out qualitative chemical analyses of the sample.

2.4 Particle Size Analysis of Crude

100 g of the sample was fed into the uppermost sieve of a set of sieves arranged in conformity with $\sqrt{2}$ series ranging from 500 – 63 μm (Wills, 2006). Attached to the bottom sieve is a tight fitting pan to receive the final undersize, also a lid was placed on top of the uppermost sieve to prevent the escape of the sample. The set of sieves was placed on an Automated Endecott test sieve shaker model EFL2mk11 (5471) and agitated for 30 minutes. During agitation, undersize particles descend through successive sieves until they are retained on a sieve having aperture size slightly smaller than their diameter (Wills, 2006). The set of sieves was separated and the retained on each sieve was weighed. The sieve fractions were chemically characterized via ED-XRF.

2.5 Sample Preparation

2 kg dried crude sample was weighted out and finely ground using Bico Ball Milling Machine, Model 69012 (USA) to 100 % passing liberation size; -250+180 μm. Prior to pulverization, proper cleaning of the milling steel balls and compartment was done to safeguard against contamination. The pulverized sample was homogenized

and prepared towards reverse froth flotation process.

2.6 Reverse Froth Flotation

Reverse Froth flotation of Itakpe iron ore dumped tailings was carried using the Denver D12 Flotation Cell. Using a mixture ratio of 1:4, 250 g of the pulverized head sample was charged with 1000 ml of distilled water into the 2000 ml capacity flotation tank and kept in suspension by a rotating impeller at a speed of 1200 rpm. The resulting slurry was conditioned at a pH of 5.0 as by adding sodium hydroxide (NaOH). The following were added subsequently; 0.5g of $CuSO_4$ (Activator), 0.5g of Corn Starch (Depressant),
0.5 g of Potassium Amyl Xanthate (PAX) (Collector), and 0.5 g of Senfroth (Frother). The process was repeated at varied agitation time of 60, 120, 180, 240, and 300 seconds while keeping other parameters constant. Cleaning and re-cleaning of the products were carried out for 300 and 120 seconds respectively. The products (Froth and depressed) were decanted, dewatered at 105 ^{0}C in a carbolite oven, and weighed. Samples of the froth and depressed were chemically analyzed via ED-XRF.

The percentage recovery of the process was also evaluated using the standard equation as shown below (Wills, 2006);

%Recovery=$\frac{Cc}{Ff}\times 100$………………..2.1

- C = Mass of Concentrate
- c = Assay of valuable mineral in the concentrate
- F = Mass of Feed
- f = Assay of valuable mineral in the fee

3. RESULTS AND DISCUSSION

3.1 Chemical and Mineralogical characterization of crude

Results of chemical and mineralogical characterization obtained in the course of this research are shown in Table 1 and Figure 1.

ED-XRF analysis of the crude sample as shown in Table 1 revealed that it contains 27.71% Fe_2O_3, 70.1% SiO_2 and other constituent compounds in trace form. Base on the assertion by Yaro and Thomas (2009), that for an ore to be named an iron-bearing mineral and also viable for processing, it must certify at least the standard requirement of 1 – 5% iron; thus, Itakpe iron ore dumped tailings assaying 27.71% Fe_2O_3 is an iron-bearing mineral and also viable for re-exploitation.

The mineralogical assemblage of Itakpe iron ore dumped tailings via XRD is shown in Figure 1. The peaks of minerals present within the head sample matrix and their relative phase amounts (weight %) are contained in the diffractogram. The minerals present and their relative amounts in weight percent are as follows: Quartz (68.41 % SiO_2), Rutile (0.79 % TiO_2), Hematite (20.05 % Fe_2O_3), Magnetite (1.21 % Fe_3O_4), Biotite-1M (1.02 %) and Albite (8.52 % (Na ($AlSi_3O_8$)). Hematite and quartz are found to be predominant in the matrix. compared to other minerals, thus this affirms that quartz is the major associative gangue which needs to be liberated and separated from the iron-bearing mineral (hematite); this may be achieved via beneficiation techniques such as reversed froth floatation (Wills, 2006; Gbadamosi*et al.*, 2017). 3.2 Scanning Electron Microscopy (SEM) / Energy Dispersive Spectrometer

The result obtained at the course of performing mineralogical investigation of of the ore using Scanning Electron Microscopy (SEM) / Energy Dispersive Spectrometer are obtaine in Figure 2 (a,b, c,d) and Figure 3 (a, b, c and d) respectively

Table 1: Chemical Composition of Crude Sample of Itakpe Iron Ore Dumped Tailings

Sample / Assay (%)	SiO_2	K_2O	CaO	V_20_5	MnO	Fe_2O_3	CuO	ZnO
Crude	70.1	0.967	0.559	0.008	0.068	27.71	0.034	0.006

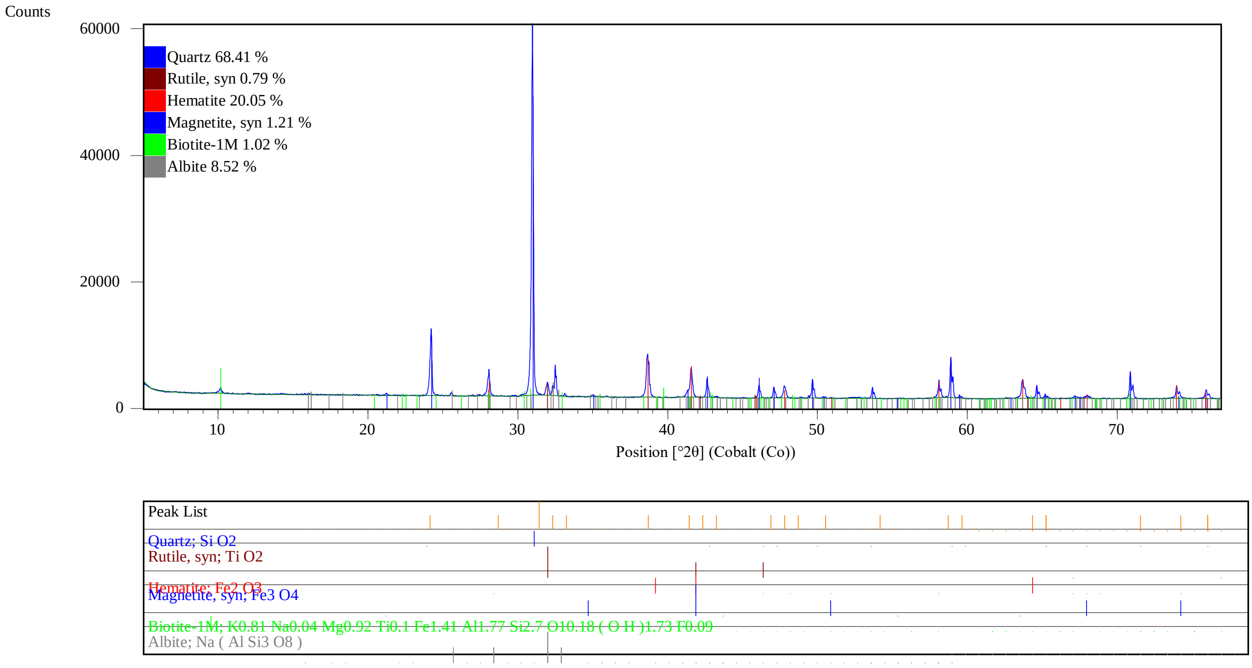
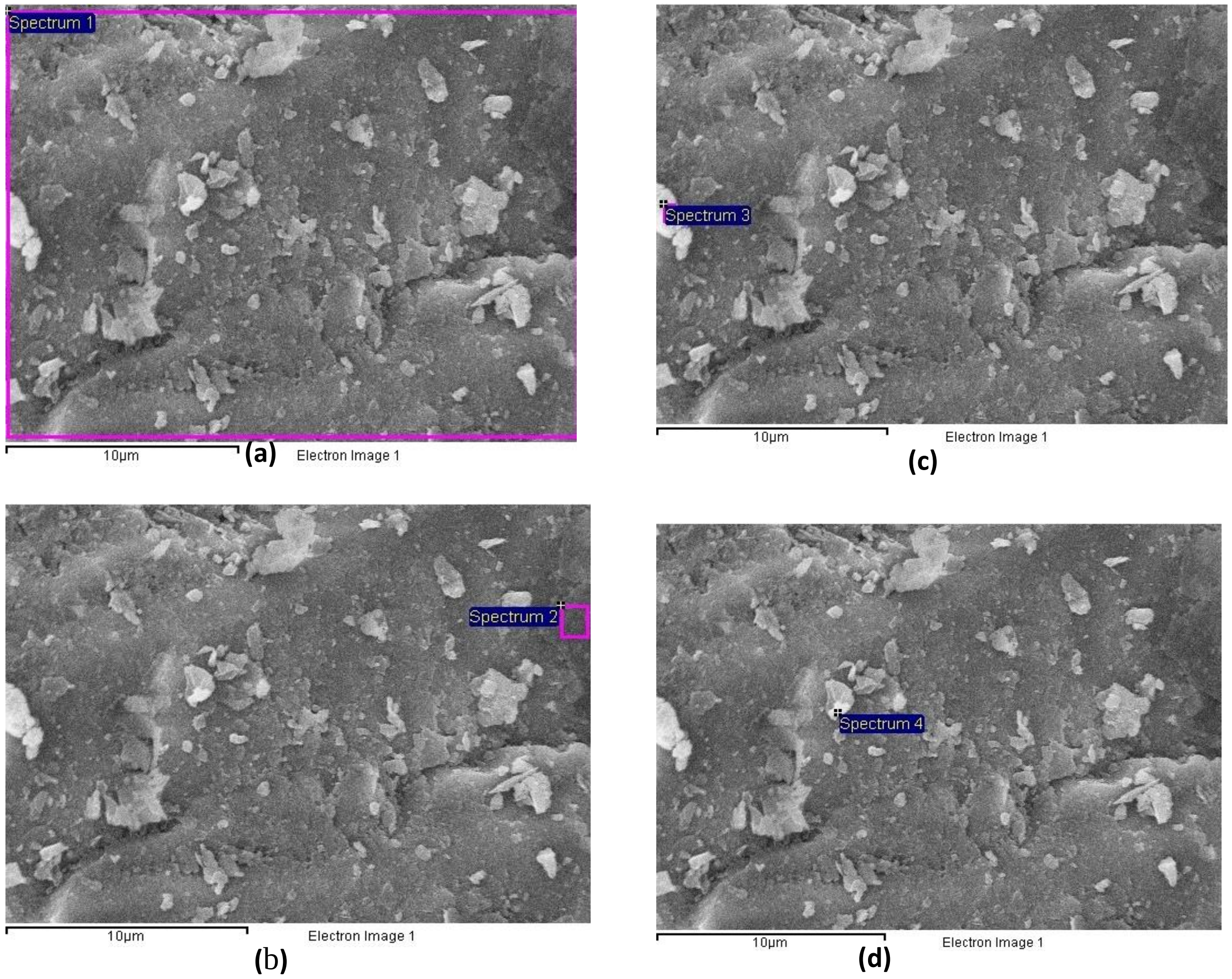
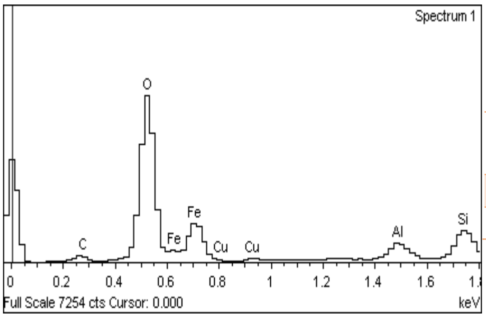


Figure 1: XRD pattern of Itakpe Iron Ore Dumped Tailings



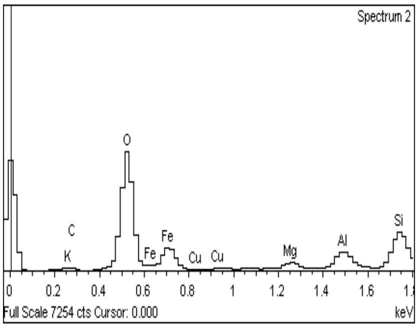
(a) **(c)** **(b)** **(d)**

Figure 2: Scanning Electron Microscopy (SEM) Micrograph of Itakpe Iron Ore Dumped Tailings at 10 µm, showing (a) Holistic analysis, (b,c,d) Point analyses. Mag.X1000



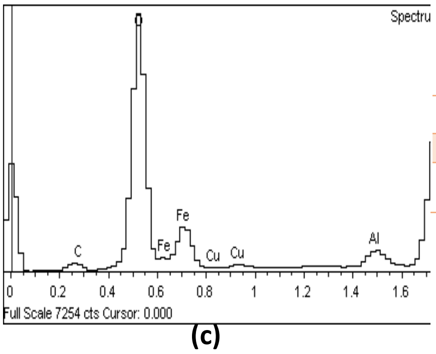
Element	C K	O K	Al K	Si K	Fe K	Cu K	Total
Weight%	6.97	43.53	2.04	3.45	43.62	0.39	100
Atomic %	13.53	63.48	1.77	2.86	18.22	0.14	

(a)



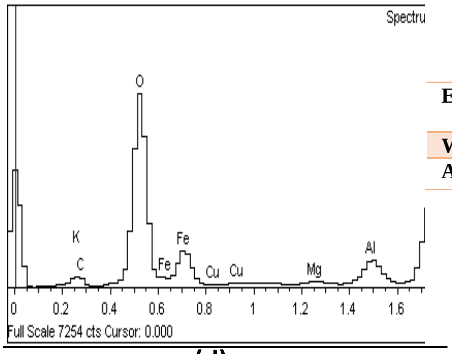
Element	C K	O K	Mg K	Al K	Si K	P K	K K	Fe K	Cu K	Total
Weight%	4.19	37.36	1.43	2.68	5.04	0.85	0.33	47.82	0.29	100
Atomic %	8.90	59.60	1.50	2.54	4.58	0.70	0.22	21.85	0.12	

(b)



Element	C K	O K	Al K	Si K	P K	Fe K	Cu K	Total
Weight%	7.25	56.06	1.29	11.98	1.18	21.89	0.35	100
Atomic %	12.03	69.83	0.95	8.50	0.76	7.81	0.11	

(c)



Element	C K	O K	Mg K	Al K	Si K	K K	Fe K	Cu K	Total
Weight%	7.79	49.16	0.36	2.22	9.22	0.42	30.39	0.44	100
Atomic %	13.78	65.26	0.31	1.75	6.97	0.23	11.56	0.15	

(d)

Figure 2: Scanning Electron Microscopy (SEM) Micrograph of Itakpe Iron Ore Dumped Tailings at 10 µm, showing (a) Holistic analysis, (b,c,d) Point analyses. Mag.X1000

Figure 2 (a,b,c,d) shows the Scanning Electron Microscopy (SEM) imaging of the head sample at 10 µm while the detailed EDX analysis of the crude sample at different spectra as represented (a,b,c,d) is presented in Figure 3 (a,b,c,d). The SEM imaging revealed that the minerals constituting the head sample are interlocked within crystal aggregates of the head sample matrix and also these minerals are observed to be coarsely packed. This consequently fosters easy liberation via communition because the more coarsely packed minerals are, the easier their liberation (Wills, 2006). Detailed EDX analysis carried out holistically and at specific regions in the ore matrix revealed the presence of C, O, Al, Si, Fe, Cu, Mg, P, and K; such that silicon and iron are the major elemental constituents of the ore matrix while other elements exist in trace form. The result obtained further compliment the analyses done on the sample using XRD and XRF and also indicates that the ore contained the mineral of interest (iron) and other associated minerals.

3.3 Particle Size Analysis of the Crude Itakpe Iron ore.

Result of the Particle size analysis of the crude iron ore dump tailing is as seen in Table 2 and Figure 4.

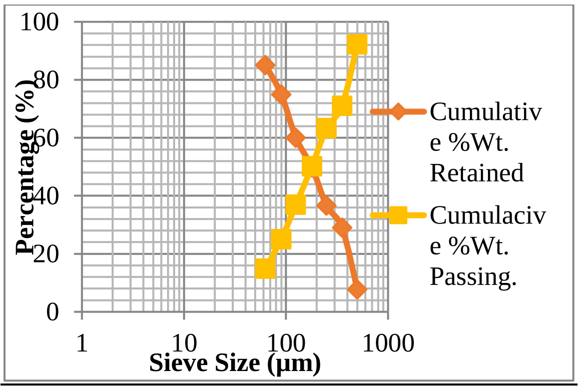


Figure 4: log-log plot of percentage cumulative retained and passing against sieve sizes (µm)

Table 2 and Figure 4 shows the fractional size analysis of Itakpe iron ore dumped tailings and the log-log graph of percentage cumulative weight retained and passing against sieve sizes respectively. The two curves as shown in the chart are mirror images of each other and they intercept at -250+180 µm which indicate the economic liberation size of the Itakpe iron ore dumped tailings. ED-XRF analysis of each sieve fractions as shown in Table 2 revealed that the actual liberation size lies at – 63 µm; however, considering the influence of size and density on separation efficiency (Wills, 2006), the economic liberation size of 63 µm passing was adopted.

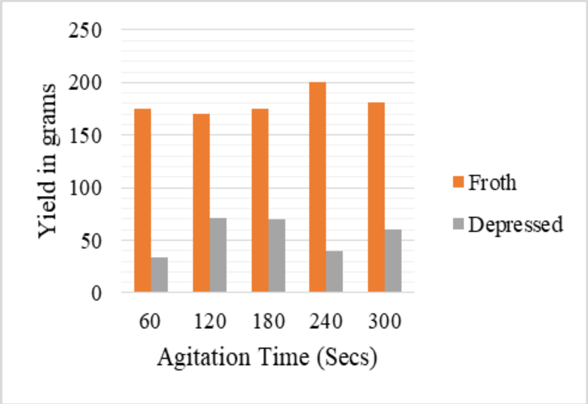


Figure 5: Yield in mass of Froth and Depressed at varying Agitation time

3.4 Reversed Froth Flotation of Crude Itakpe Iron ore

Result obtained at the course of reverse froth flotation process of crude Itakpe iron ore is presented in Figures 5 – 6 and Tables 3 – 4.

Table 2: Fractional Sieve Size Analysis of Itakpe Iron Ore Dumped

Sieve size range (µm)	Weight retained (g)	% Weight Retained	Cumulative weight retained	Cumulative weight passing	% Fe
+500	7.67	7.70	7.70	92.30	35.10
-500+355	21.18	21.28	28.98	71.02	26.34
-355+250	7.63	7.67	36.65	63.35	20.20
-250+180	13.04	13.10	49.75	50.25	15.81
-180+125	13.23	13.29	63.04	36.96	12.83
-125+90	11.82	11.88	74.92	25.08	11.06
-90+63	10.13	10.18	85.09	14.91	20.56
-63	14.84	14.91	100	0	43.91

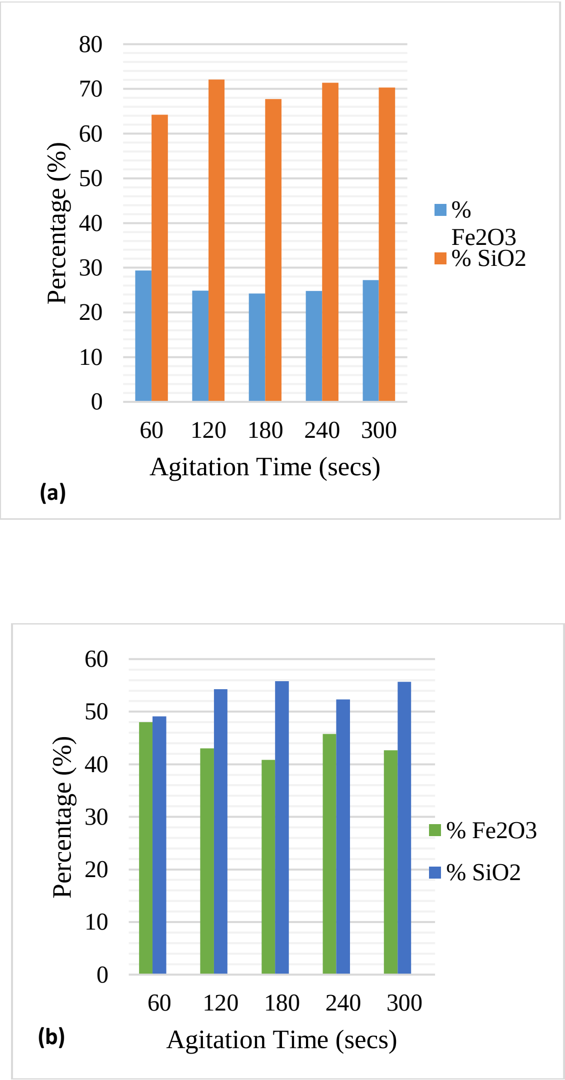


Figure 6: Histogram plot showing % Fe_2O_3, SiO_2 of (a) froth and (b) Depressed samples against Agitation Time (secs)

Table 4: Recovery of Iron oxide in Froth and Depressed Sample at Varying Agitation Time

C – Concentrate (Depressed), T – Tailings (Froth)

Table 3 - 4 show the yield in mass, assay and percentage recovery of froth and depressed samples at varying agitation time respectively. Figure 5 reveals that the yield in mass of froth realized at any agitation time was more than that of depressed, thus showing that extensive separation did occur. However, the chart revealed that the highest yield in mass of depressed (concentrate) (70.8 g) assaying 43.0% Fe_2O_3 occurred at an agitation time of 120 secs with recovery of Fe_2O_3 and %SiO_2 of the froth and depressed samples at different agitation time. This further showed that optimum % Fe_2O_3 (47.99%) was achieved in the depressed at an agitation time of 60 secs with recovery and yield of 23.41% and 33.8 g respectively, while high % SiO_2 was contained in the froth at an agitation time of 180 secs.

3.5 Metallurgical Accounting of froth flotation of Itakpe iron ore dumped tailings

Metallurgical accounting of froth flotation of Itakpe iron ore dumped tailings is presented in Table 5

Table 3: Chemical Composition of Froth and Depressed Sample at Varying Agitation Time

Time (secs)	Sample / Assay (%)	SiO_2	K_2O	CaO	V_20_5	MnO	Fe_2O_3	CuO	ZnO
60	Concentrate	49.1	1.04	0.614	0.023	0.077	47.99	0.198	0.01
	Tailing	64.2	1.22	0.685	0.02	0.063	27.39	0.848	0.027
120 secs	Concentrate	54.3	0.803	0.54	0.02	0.053	43.0	0.077	0.041
	Tailing	72.1	1.07	0.5	0.01	0.055	24.88	0.131	0.007
180 secs	Concentrate	55.8	0.882	0.52	0.02	0.049	40.83	0.047	BDL
	Tailing	67.7	1.11	0.572	BDL	0.048	24.26	0.078	0.004
240 secs	Concentrate	52.3	0.853	0.53	BDL	0.047	45.79	0.073	BDL
	Tailing	71.4	1.00	0.566	0.02	0.043	24.83	0.091	BDL
300 secs	Concentrate	55.47	0.727	0.47	0.01	0.045	42.65	0.051	BDL
	Tailing	70.3	1.08	0.576	0.02	0.054	27.22	0.10	0.008

Table 4: Recovery of Iron oxide in Froth and Depressed Sample at Varying Agitation Time
C – Concentrate (Depressed), T – Tailings (Froth)

	Charge	60 secs		120 secs		180 sec		240 secs		300 secs	
		C	T	C	T	C	T	C	T	C	T
Weight (g)	250	33.80	174.80	70.80	170.70	70.30	175.10	40.10	200.60	60.2	180.50
Assay (%)	27.71	47.99	29.39	43.00	24.88	40.83	24.26	45.79	24.83	42.65	27.22
Recovery (%)		23.41	74.15	43.95	61.31	41.43	61.32	26.51	71.90	37.06	70.92

Table 5: Metallurgical Accounting of Froth Flotation of Itakpe Iron Ore dumped Tailings

	Processed Samples	Recovery (%)	Enrichment Ratio	Concentration Ratio	Grade (%)
60 secs	Concentrate	23.41	1.73	7.40	47.99
	Tailings	74.15	1.06	1.43	29.39
120 secs	Concentrate	43.95	1.55	3.53	43.00
	Tailings	61.31	0.90	1.46	24.88
180 secs	Concentrate	41.43	1.47	3.56	40.83
	Tailings	61.32	0.88	1.43	24.26
240 secs	Concentrate	26.51	1.65	6.23	45.79
	Tailings	71.90	0.90	1.25	24.83
300 secs	Concentrate	37.06	1.54	4.15	42.65
	Tailings	70.92	1.00	1.39	27.22

Table 5 presents the metallurgical accounting of reverse froth flotation of Itakpe iron ore dumped tailings carried out at varied agitation time in terms of percentage recovery, grade, enrichment ratio, and concentration ratio. It was observed that concentration ratio, enrichment ratio, and grade are inversely related to the % recovery for all agitation time. On-premise of enrichment and concentration ratio being parameters employed to assess the efficiency of a separation method (Wills, 2006), it can be deduced that maximum separation efficiency was achieved at an agitation time of 60 secs having enrichment and concentration ratio of 1.73 and 7.40 respectively; the concentrate obtained assayed 47.99% Fe_2O_3 at a recovery of 23.41%.

4. CONCLUSION

The characterization of Itakpe Iron Ore Dumped tailings and its reverse froth flotation at varying agitation time have been considered. The crude sample having been characterized via ED-XRF, XRD, and SEM/EDX was found to contain quartz and haematite (Fe_2O_3) as the major minerals in the crude; though they are lossely packed. Results gotten from the reverse froth flotation of the crude revealed that at agitation time of 60 secs, concentrate obtained assayed 47.99% however at low % recovery, while at 120 secs, concentrate obtained assayed 43%, however at high % recovery. Finally, the crude sample has been successfully upgraded to 47.99 % Fe_2O_3 via reverse froth flotation method at an agitation time of 60 seconds, while keeping other parameters constant leaving out a silica-rich tailing of 64.2 % SiO_2.

RECOMMENDATIONS

The followings are recommended base on the findings of this research;

i. Exploitation of Itakpe Iron Ore dumped tailings via reversed froth flotation should be ventured into, towards judicious re-separation of the iron-bearing mineral which thus, optimally contributes to the quantity of iron-bearing concentrates shipped out to extraction plants or for exportation.

ii. Also, the silica-rich tailings gotten after beneficiation can be judiciously employed as backfill for mining sites land reclimation, as paving material for road construction, and also as coarse aggregates for building construction. This will consequently promote land reclamation and facilitate its judicious use for other purposes such as agriculture.

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