



GEOMETALLURGICAL EVALUATION OF AKURE SOUTH SILICA-IRON EARTH SIPPED ORE DEPOSIT FOR EFFECTIVE PROCESSING AND EXTRACTION

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Abstract:

Chemical and mineralogical characterization of Akure South Silica-Iron earth sipped mineral was investigated. Its susceptibility to magnetic and gravity methods of mineral separation was also studied. Samples of the mineral were sourced from three different deposit regions in Akure South Local Government Area, Ondo State, Nigeria. The sourced samples were crushed, homogenized, and 50 kg was weighted out. Chemical characterization of the mineral was carried out via Energy Dispersive X-ray Fluorescence Spectrometry (ED-XRF). Mineralogical characterization was carried out via X-ray Diffraction (XRD) and Scanning Electron Microscopy coupled with Energy Dispersive X-Ray Spectroscopy (SEM-EDS). Fractional sieve analysis of the crude sample was carried out using sieve range of 500 μm to -63 μm towards particle size distribution and liberation size determination. 2 kg of the crude was randomly sampled out and pulverized to 100% passing -180+125 μm . Susceptibility of the mineral to magnetic and gravity separation methods were assessed via hand induced magnetic separation and sink float technique respectively. Chemical analysis of the crude sample via Energy Dispersive X-ray Fluorescence Spectrometry (ED-XRF) revealed that the crude sample contains 17.55 % Fe_2O_3 , 63.7 % SiO_2 , 15.5 % TiO_2 , and other compounds in trace form. Mineralogical analysis via XRD revealed the mineral phases present in the crude sample as Quartz (87.07 % SiO_2), Magnetite (0.59 % Fe_3O_4), Ilmenite (9.89 % FeTiO_3) with other associated minerals such as Rutile (0.53 % TiO_2), Anatase (0.25 % TiO_2), Kaolinite (0.09% $\text{Al}_2(\text{Si}_2\text{O}_5(\text{OH})_4$) and Albite (1.57% $\text{Na}(\text{AlSi}_3\text{O}_8)$). SEM imaging revealed interlocking of some minerals within the crystal aggregates of the crude and the EDS analysis revealed the presence of Al, Mn, Cu, O, Si, C, Fe, and Ti; such that silicon, titanium, and iron are the major elemental constituents of the ore matrix. Chemical analysis of

the processed samples gotten from susceptibility studies, revealed that the crude (17.55% Fe_2O_3) has been successfully upgraded via magnetic separation to 33.01% Fe_2O_3 at a recovery of 22.78 % and via gravity separation to 18.54% Fe_2O_3 at a recovery of 54.0%. In conclusion, characterization studies carried out revealed the mineral to be an iron-bearing mineral having silica (quartz) as the major associative gangue and susceptibility studies carried out revealed that it is highly susceptible to magnetic separation method compared to gravity separation.

Keywords: Akure South, Silica-Iron, Earth Sipped, Mineral, Geometallurgical Evaluation

1. Introduction

Nigeria is richly endowed with vast metallic mineral deposits which are distributed in virtually all geopolitical zones; most of which are yet to be discovered or explored. Some of this metal bearing minerals includes; tantalite, columbite, haematite, wolframite, sphalerites, galena, ilmenite, and so on [1]. Mineralization of these minerals is mainly regulated by geological factors such as magmatic, hydrothermal, sedimentary, and weathering processes. These consequently cause the selective concentration of desirable metallic elements in a particular deposit, thus rendering such metallic deposit viable for profitable exploration and exploitation [2]. However, detailed information on the occurrence of these minerals, geology, mineralogy, and texture will facilitate effective design of beneficiation flow-sheet [3]. Likewise, having prior knowledge of these characteristics helps to determine the liberation size to which the ore body should be ground and also facilitate proper selection of the concentration techniques to be adopted to achieve optimum separation [3][4]. Furthermore, having prior knowledge of the mineralogical/chemical composition, size, morphology, and association with other minerals is expected to provide insights and information on the characteristics, type, nature and amount of minerals and elements present within the ore at different locations consequently, permits an assessment and determination of the optimal processing route(s) for its constituent minerals/metals [5].

Nigeria's geology comprises three major litho-petrological components - the basement complex, younger granites and sedimentary basins [6]. Akure South Local Government Area of Ondo State falls within the basement complex region of Nigeria having coordinates; 7.2146° N and 5.1641° E and it is underlain by granite and charnockite rocks which constitute the precambrian basement complex rocks of South Western Nigeria [7]. However, these rocks are prone to hydrothermal forces which causes weathering and as such during rainy season, these weathered rocks sip out of the earth and are conveyed by running water to be deposited as alluvial silica-iron ore in drenches, grooves, ditches and on roads present within Akure South Local Government Area of Ondo State, Nigeria. The ore is often tagged blacksand and it occurs unconsolidated on a layer of silica bed [8]; its distinct appearance and physical properties initiated a drive to carry out geometallurgical evaluation towards ascertaining its mineralogical characteristics and economic viability.

Several research reports have been published regarding the geological and metallurgical assessment of different mineral deposits across the boundaries of Nigeria towards proving their economic viability for processing and extraction [9]. Therefore, the silica-iron earth sipped deposit at Akure South Local Government; Ondo State, Nigeria is being investigated to ascertain its metallurgical characteristics and also its susceptibility to gravity and magnetic separation method towards effective processing and extraction.

2. Research Methodology

2.1 Material Sourcing

Samples were sourced from three different locations; Obanla, Orita-obele and Road block, which are all regions within the Akure South Local Government Area of Ondo State, Nigeria having geological coordinates of latitude 7.292342 and longitude 5.160443; latitude 7.306548 and longitude 5.139643; latitude 7.291196 and longitude 5.160527 respectively. The collected samples were properly mixed for homogenization and 50 kg was weighted out. 2 kg of the collected crude sample was sampled out via cone and quartering sampling method. Hand picking and sieving were carried out to remove coarse aggregates and unwanted materials like broken bottles, wood, etc. Thorough mixing was done to ensure proper homogenization of crude sample.

2.2 Chemical Characterization

Chemical characterization of the crude was carried out using Energy Dispersive X-ray Fluorescence Spectrometer (PANalytical Minipal 7). 20 g of the sample was pulverized to pass through a 200-250 mesh sieve. Thereafter, the sample was properly mixed with a binder in the ratio of 5.0 g sample(s) to 1.0 g cellulose flakes binder and pelletized at a pressure of 10-15 tons/inch² in a pelletizing machine. At this stage, the pelletized sample(s) are stored in a desiccator for analysis. The ED-XRF machine was switched on and allowed to warm up for 2 hours. Finally, appropriate programs for the various elements of interest were employed to analyze the sample material(s) for their presence or absence. The result of the analysis was either reported in parts per million (ppm) or percentage (%) for minor and major concentrations of elements.

2.3 Mineralogical Characterization via XRD

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-K α radiation. The material was prepared for XRD analysis using a backloading preparation method. The phases were identified using X'Pert Highscore plus software. The relative phase amounts (%weight) were evaluated using the Rietveld method.

2.4 Mineralogical Characterization via SEM-EDS

Morphological and qualitative analyses of the bulk ore were performed using SEM-EDS. The SEM provides information on spatial distribution of mineral phases present in the crude, while EDS provides information on their elemental composition [10]. Mineralogical analysis via SEM-EDS was conducted on representative samples in two stages using SEM (Model: JEOL 840). All the samples were carbon coated in order to make the mineral's surface conductive. Samples for analysis were cut, polished mounts in embedded epoxy resin, and finally polished to obtain a mirror-like surface. The polished surfaces were finally carbon coated before analysis. Qualitative chemical analysis of the samples was carried out using EDS detector attached to the SEM.

2.5 Particle Size Analysis

Fractional sieve analysis technique was adopted to ascertain the particle size distribution and the liberation size of the mineral. The set of sieves was properly cleaned to avoid contamination of the mineral sample and arranged in conformity with $\sqrt{2}$ series ranging from 500 – 63 μm [11]. 100 g crude sample was weighed and charged into the upper sieve (500 μm) and agitated for 30

minutes using an Automated Sieve Shaker, (Model: Endecott AS400 control). This consequently causes the undersize mineral particles to fall through successive sieves until they are retained on a sieve having an aperture lesser than the particle diameter.

2.6 Sample Preparation

1 kg of the crude mineral was weighted out and pulverized using Bico Ball Milling Machine, Model 69012 (USA) to 100 % passing liberation size; -180+125 μm . The liberation size was selected taking into consideration the effect of particle size and density on separation efficiency. The pulverized sample was properly homogenized and prepared towards susceptibility tests.

2.7 Gravity Susceptibility Test

100 g crude mineral was charged into 100 ml Pyrex measuring cylinder containing 50 ml bromoform solution. The admixture was agitated using a spatula for 60 seconds and allowed to settle into three strata; the sink, separating medium, and the float. The sink and float samples were discharged separately, rinsed properly with distilled water, oven dried and subjected to specific gravity and density test. Chemical analysis of the processed samples (sink and float) was also carried out. The concentration criterion of the process was evaluated using equation 1. The resulting quotient must satisfy the condition $CC \geq 2.5$ for the mineral to be susceptible to gravity separation method.

$$\text{Concentration Criterion (CC)} = \frac{D_h - D_f}{D_l - D_f} \geq \pm 2.5 \dots\dots\dots (1)$$

- D_h – specific gravity of heavy minerals
- D_l – specific gravity of light minerals
- D_f – specific gravity of separating medium

2.8 Magnetic Susceptibility Test

200 g head sample was weighed and evenly spread on a pyrite sample dish. Hand induced magnet of dimensions; 100 mm × 50 mm × 10 mm was passed over the spread mineral at 10 mm height. This consequently causes minerals which are magnetic in nature to get attracted leaving behind the non-magnetic minerals of the head sample. The processed samples (Magnetic and Non-magnetic) were chemically analyzed via ED-XRF.

2.9 Specific Gravity and Density Determination

The specific gravity and density of the crude and processed samples were determined.

2.9.1 Specific Gravity Determination

Specific gravity was determined on the basis of the amount of fluid displaced by a solid when immersed in a fluid medium such as water. 4 ml of water was measured into a 10 ml Pyrex measuring cylinder and recorded as the initial water volume. 10 g of the crude sample was charged into the measuring cylinder; the resulting rise in the level of water was noted and recorded as the final water volume. The results obtained were employed to evaluate the specific gravity of the head sample utilizing the expression as shown in equation 2.

$$\text{Specific Gravity (S.G.)} = \frac{M}{V_2 - V_1} \times \text{S.G}_f \dots \dots \dots (2)$$

- M – Mass of sample in grams
- V_1 – Initial volume of fluid in ml
- V_2 – Final volume of fluid in ml
- S.G_f – Specific Gravity of fluid

The above procedure was repeated for the processed samples (sink, float, magnetic and non-magnetic).

2.9.2 Density Determination

Density of the crude and processed samples was determined using a 150 ml density bottle. The weight of the corked density bottle was measured and recorded, after which it was filled with water up to the marked point, corked, and their equivalent weight measured and recorded. The density bottle was emptied and dried properly. 10 g crude sample was charged into the bottle, corked, and their equivalent mass was measured and recorded, after which it was filled with water to the marked point, corked, agitated for 30 seconds, and the equivalent mass of the entity measured and recorded.

The data obtained were employed to evaluate the specific gravity of the sample utilizing the expression as shown in equation 3.

$$\rho = \frac{M_2 - M_1}{(M_4 - M_1) - (M_3 - M_2)} \times D_F \dots\dots\dots (3)$$

- D_f – Density of fluid used
- M_1 – Mass of bottle + cork in grams
- M_2 – Mass of bottle + cork + dry sample in grams
- M_3 – Mass of bottle + cork + dry sample + water in grams
- M_4 – Mass of bottle + cork + water in grams
- ρ - Density of solid in grams per cubic meter, [12]

3. Results and Discussion

3.1 Results

Results obtained along the course of this research work are presented below.

Table 1: Chemical Analysis of Crude Sample

Sample/ Assay %	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	TiO ₂	V ₂ O ₅	MnO	Fe ₂ O ₃	ZnO
Crude	BDL	63.7	0.2	0.691	0.23	15.5	0.38	0.29	17.55	0.009

BDL – Beyond Detectable Limit

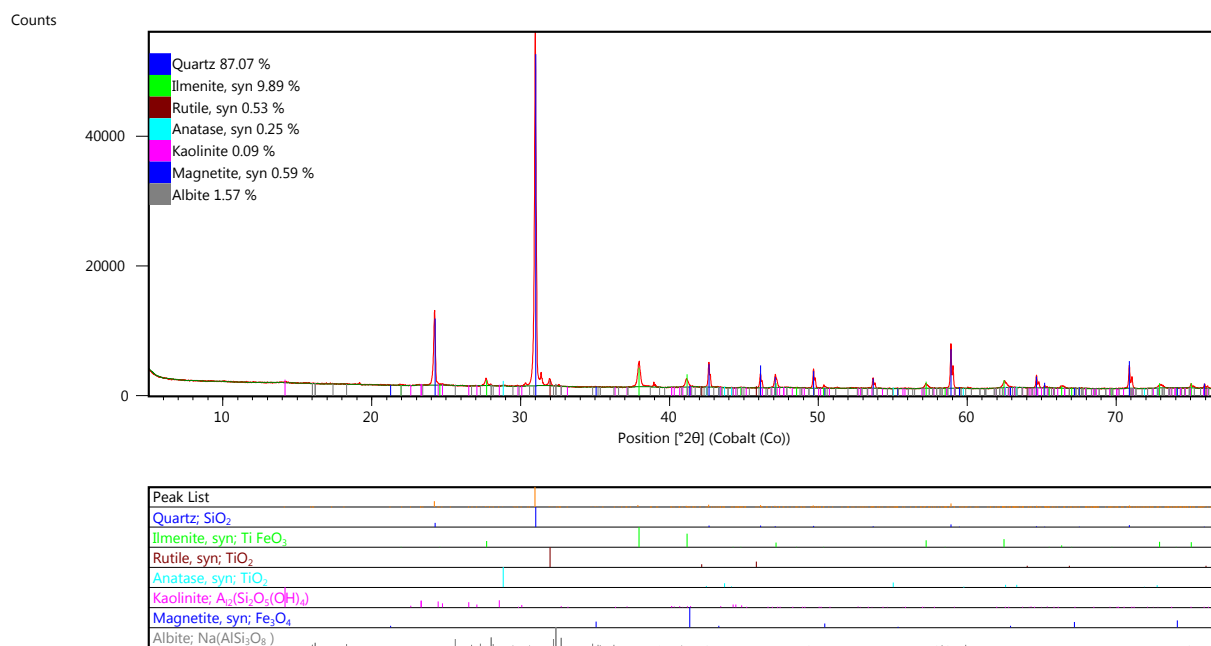


Figure 1: XRD pattern of Akure South mineral

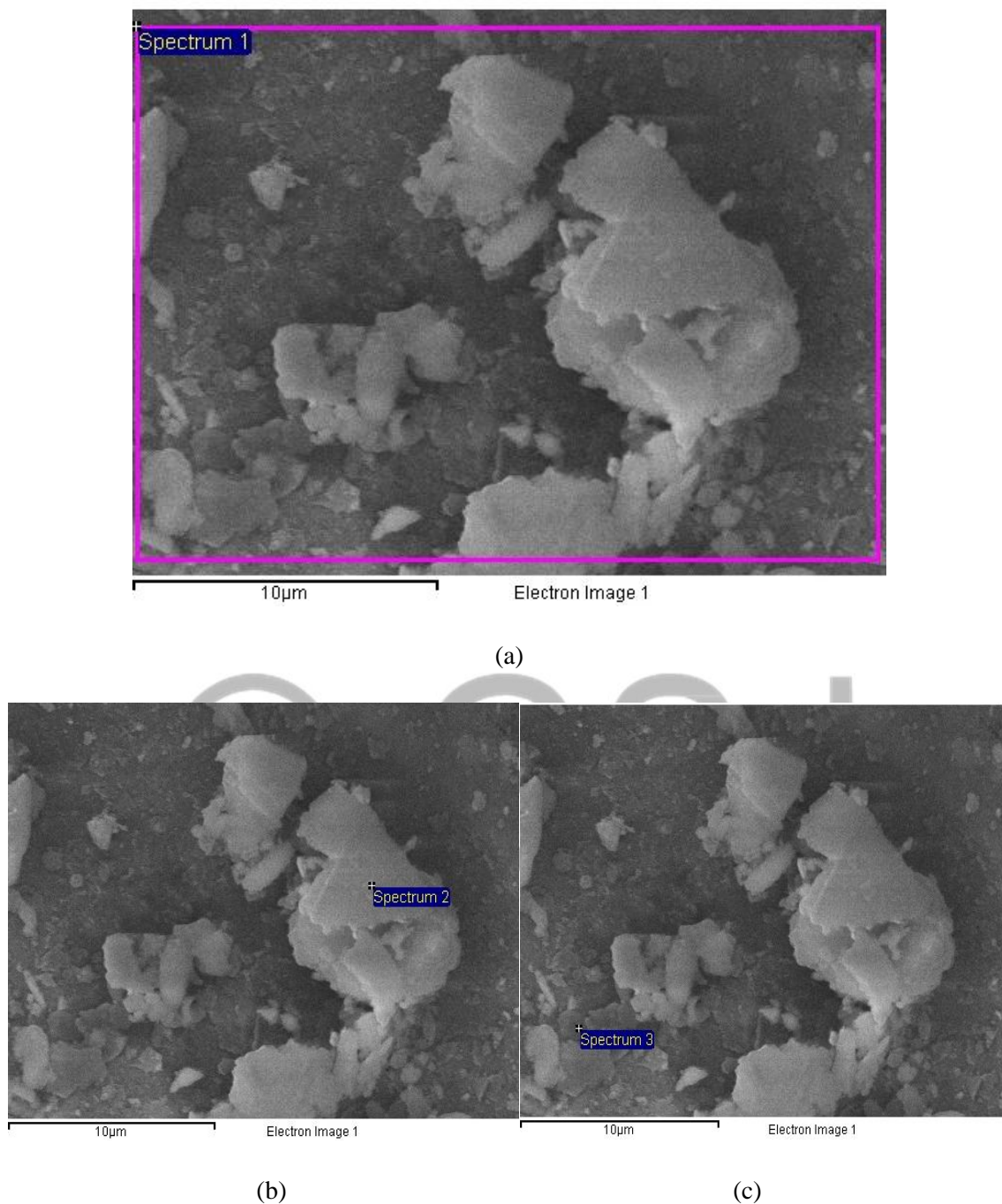
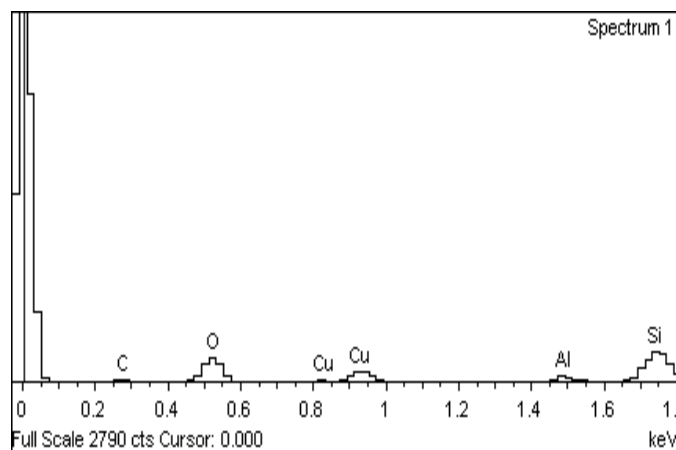
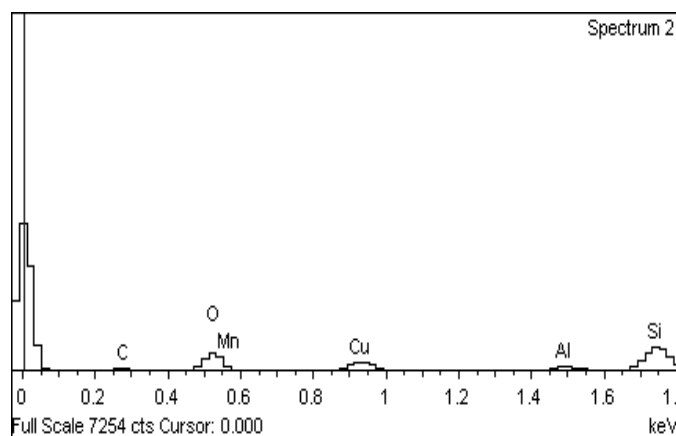


Figure 2: Scanning Electron Microscopy (SEM) Micrograph of the head sample, showing (a) Holistic analysis, (b,c) Point analysis



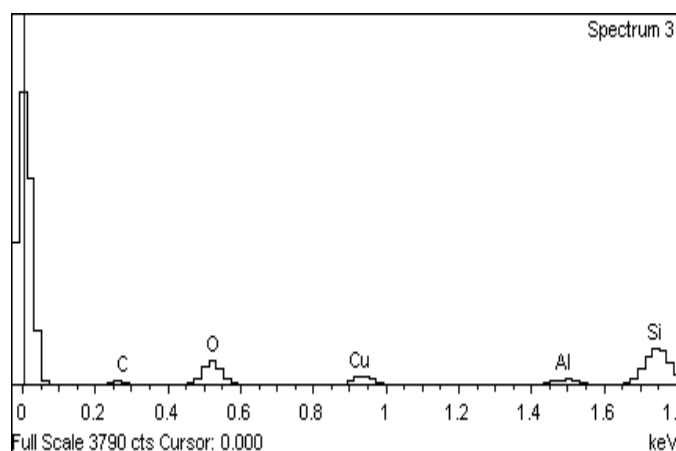
Element	Weight%	Atomic%
C K	15.50	27.67
O K	36.62	49.06
Al K	2.01	1.60
Si K	10.52	8.03
Ti K	3.83	1.71
Fe K	27.88	10.70
Cu K	3.63	1.22
Total	100	

(a)



Element	Weight%	Atomic%
C K	9.57	21.28
O K	25.51	42.57
Al K	1.59	1.57
Si K	7.62	7.24
Ti K	10.48	5.84
Mn K	0.70	0.34
Fe K	42.46	20.30
Cu K	2.07	0.87
Totals	100.00	

(b)



Element	Weight%	Atomic%
C K	18.79	28.45
O K	49.14	55.84
Al K	2.31	1.56
Si K	13.98	9.05
Ti K	1.62	0.61
Fe K	10.89	3.54
Cu K	3.27	0.94
Totals	100.00	

(c)

Figure 3: Energy Dispersive X-ray Spectroscopy (EDS) peaks for the various elements present in the head sample SEM Micrographs for (a) Holistic analysis, (b,c) Point analyses

Table 2: Particle Size Analysis Result of Akure South Mineral

Sieve Size Range (μm)	Weight Retained (g)	% Weight Retained	Nominal Aperture	Cumulative % Weight Retained	Cumulative % Weight Passing	% Fe
+500	2.35	2.36	500	2.36	97.64	6.018
-500+355	21.61	21.68	355	24.04	75.96	7.535
-355+250	10.76	10.79	250	34.83	65.17	12.19
-250+180	20.52	20.59	180	55.42	44.58	12.37
-180+125	19.90	19.96	125	75.38	24.62	19.23
-125+90	12.31	12.35	90	87.73	12.27	21.01
-90+63	5.90	5.92	63	93.65	6.35	20.40
-63	6.33	6.35	-	100	0.00	24.86

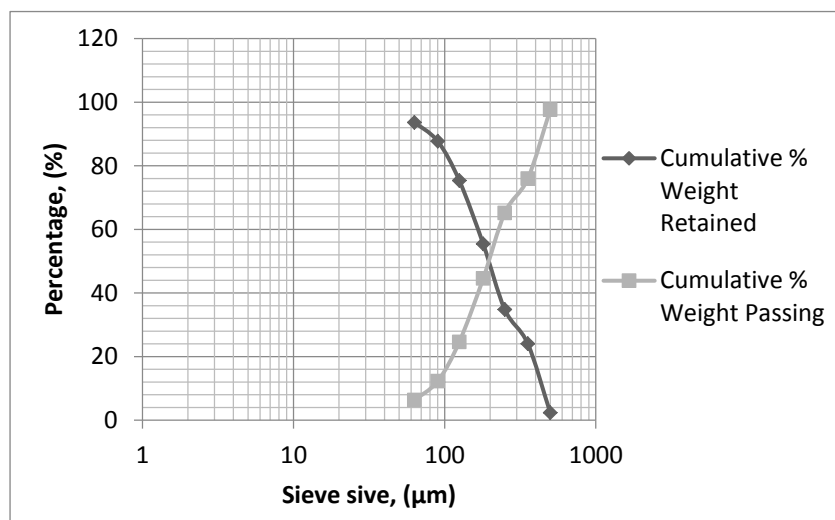


Figure 4: Log-Log plot of % Cumulative Weight retained and passing against Sieve Sizes (μm) of the Fractional sieve analysis of Akure South mineral

Table 3: Yield Results of Gravity Separation and Magnetic Separation

Method	Charge (g)	Concentrate (g)	Tailing (g)
Gravity Separation	100	51.10	48.50
Magnetic Separation	200	24.20	175.10

Table 4: Chemical Analysis of Processed Samples of Akure South Mineral

Sample/ Assay %	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	TiO ₂	V ₂ O ₅	MnO	Fe ₂ O ₃	CuO	ZnO
Sink	3.0	61.0	0.2	0.661	0.29	14.9	0.36	0.27	18.54	0.079	0.031
Float	2.0	63.9	0.2	0.637	0.23	14.0	0.36	0.27	16.47	0.090	0.036
Magnetic	5.4	33.4	0.2	0.40	0.20	25.3	0.66	0.40	33.01	0.067	0.040
Non Magnetic	BDL	67.6	0.2	0.657	0.28	13.9	0.35	0.27	15.47	0.070	0.029

BDL – Beyond Detectable Limit

Table 5: Percentage Recovery of Gravity and Magnetic Methods of Separation

Sample	Gravity Separation			Magnetic Separation		
	Charge	Sink	Float	Charge	Magnetic	Non-Magnetic
Weight (g)	100	51.10	48.50	200	24.20	175.10
Assay (%)	17.55	18.54	16.47	17.55	33.01	15.47
Recovery (%)	-	54.00	45.51	-	22.78	77.17

Table 6: Density of Crude and Processed Samples

Samples	M ₁ (g)	M ₂ (g)	M ₃ (g)	M ₄ (g)	ρ (g/cm ³)
Crude	25.20	35.35	83.90	77.20	2.94
Sink	25.20	35.25	84.85	77.20	3.13
Float	25.20	35.30	83.85	77.20	3.03
Magnetic	25.20	35.30	85.00	77.20	4.39
Non magnetic	25.20	35.30	83.70	77.20	2.81

Table 7: Specific gravity of Crude and Processed Samples

Samples	M(g)	V ₁ (mls)	V ₂ (mls)	Specific Gravity
Crude	10	4.0	7.4	2.94
Sink	10	4.0	7.0	3.33
Float	10	4.0	7.3	3.03
Magnetic	10	4.0	6.0	4.76
Non magnetic	10	4.0	7.4	2.94

Table 8: Metallurgical Accounting of Gravity and Magnetic Separation of Akure South Mineral

Sample	Recovery (%)	Enrichment Ratio	Concentration Ratio	Grade (%)
Crude	-	-	-	17.55
Sink	54.00	1.06	1.96	18.54
Float	45.51	0.94	2.06	16.47
Magnetic	22.78	1.88	8.26	33.01
Non magnetic	77.17	0.88	1.14	15.47

3.2 Discussions

3.2.1 Chemical Analysis of the Crude

Table 1 presents the chemical analysis of the head sample via ED-XRF. The crude contains 17.55 % Fe_2O_3 , 63.7 % SiO_2 , 15.5 % TiO_2 , and other trace compounds; SiO_2 being the pertinent associative gangue. This consequently certifies the standard of 1-5% iron requirement in an ore to be tagged an iron bearing mineral [13].

3.2.2 Mineralogical Characterization of the Crude via XRD

XRD pattern of the crude sample of Akure South mineral as shown in Figure 1 reveals the peaks of the various mineral compounds present, nature of occurrence, and respective phase amounts. The minerals present in the ore, their state of occurrence, and respective weight percent include; Quartz (87.07 % SiO_2), Magnetite (0.59 % Fe_3O_4), Ilmenite (9.89 % FeTiO_3), Rutile (0.53 % TiO_2), Anatase (0.25 % TiO_2), Kaolinite (0.09% $\text{Al}_2(\text{Si}_2\text{O}_5(\text{OH})_4)$ and Albite (1.57% $\text{Na}(\text{AlSi}_3\text{O}_8)$); such that Ilmenite and quartz are the major minerals in the ore matrix. The abundance of ilmenite may be ascribed to the mineralization of minerals of iron and titanium during formation of the ore deposit [14].

3.2.3 Mineralogical Characterization of the Crude via SEM-EDS

The Scanning Electron Microscopy (SEM) micrograph of the crude sample at 10 microns as shown in Figure 3 reveals the interlocking nature of minerals within the crystal aggregates in the ore matrix. These minerals possess coarse grain boundaries and as such facilitate easy liberation via comminution; the more coarsely packed the minerals the easier their liberation [15]. EDS analysis of the phases present within the crude sample matrix reveals the peaks of various elements present and their relative amounts in weight percent as shown in Figure 3. Holistic analysis of the crude revealed the presence of C, Al, Cu, O, Fe, Ti, and Si; carbon element identified is probably due to the pretreatment of the sample surface via carbon coating to enhance conductivity. However, on further point analyses iron, titanium, and silicon were found to be the major elemental constituents of the ore matrix. Thus, the result obtained conforms with and further compliment the ED-XRF and XRD analyses carried out and as such, the crude sample contains iron as the mineral of interest in association with other minerals that can hinder its processing unless they are reduced to a minimal level by separation techniques to yield a high grade concentrate while the associated minerals are discarded as tailings [16].

3.2.4 Particle Size Analysis of the Crude

Table 2 and Figure 4 shows the fractional sieve size analysis of the crude and the log-log plot of % Cumulative Weight Retained and Passing against Sieve Sizes (μm) respectively. The two curves obtained are mirror images of each other and they intercept at -250+180 μm which represent the sieve size having 50% cumulative passing and 50% cumulative retained. Furthermore, chemical analysis of the sieve fractions obtained revealed that significant increment in %Fe started at this sieve size as shown in Table 2; as such the liberation size of the mineral lies in the range -250+180 μm to -63 μm . However, taking into consideration grade requirement

coupled with the effect of size and density on separation efficiency, $-180+125\mu\text{m}$ was chosen as the liberation size.

3.2.5 Gravity and Magnetic Susceptibility Test

Table 3, 4 and 5 presents the yield in mass, assay and percentage recovery of the processed samples respectively of the processed samples obtained from the susceptibility studies carried out. The sink and float method of gravity separation yields concentrate (sink) and tailings (float) of mass 51.10 g and 48.50 g, assaying 18.54 % Fe_2O_3 and 16.47 % Fe_2O_3 at recovery of 54.0% and 45.51% respectively, while the hand induced magnet method of magnetic separation yields concentrate of 24.20 g and tailing of 175.10 g assaying 33.01% Fe_2O_3 and 15.47% Fe_2O_3 at recovery of 22.78% and 77.17% respectively. Furthermore, it can be deduced that the quantity by mass of concentrate and % recovery of both separation methods are inversely related to the assay of the concentrate, such that magnetic separation gave the highest assay of 30.01% Fe_2O_3 at low yield and % recovery compared to that of gravity separation. More so, comparison of the obtained concentrates led to the deduction that concentrate obtained via magnetic is of higher grade than that obtained via gravity separation. This consequently influence the choice of processing route(s), such that gravity separation methods can be opted for if high % recovery and yield at low grade of priority, while if high grade at low recovery and yield is of priority, magnetic separation can be opted for to beneficiate this mineral.

3.2.6 Specific Gravity and Density of Crude and Processed Samples

Table 6 – 7 presents the evaluated specific gravity and density (g/cm^3) of the crude and processed samples respectively. The resulting variation in specific gravity and density values of the crude and processed samples indicates that separation did occur. The specific gravity and density of the crude are 2.94 and 2.94 g/cm^3 respectively. The evaluated specific gravity and

density of the sink and float samples are; 3.33; 3.13 g/cm³, and 3.03; 3.03g/cm³ respectively. The slight variation in values obtained reveals that separation has occurred, although not extensively probably due to the presence of valuable and non-valuable mineral in the float and sink sample respectively. The concentration criterion using bromoform as separating medium was evaluated as 1.77; which does not meet the condition $CC \geq \pm 2.5$. This further affirms that separation efficiency via gravity method of mineral separation is low; since separation efficiency decreases with decreasing quotient [17]. The evaluated specific gravity and density of the magnetic and non-magnetic mineral are 4.94; 4.39 g/cm³ and 2.91; 2.81 g/cm³ respectively. The large variation in the obtained values depicts that separation has occurred extensively. This is due to the weak magnetic property of the valuable mineral which makes it susceptible to attraction when exposed to magnetic field from a hand-induced magnet [18], as such more of the valuable mineral report in the magnetic sample while the gangue which is nonmagnetic in nature is left behind in the sample.

3.2.7 Metallurgical Accounting of Susceptibility Tests

Table 8 presents the metallurgical assessment of the susceptibility tests carried out in terms of percentage recovery, grade, enrichment ratio, and concentration ratio. The result shows that the grade, % recovery, enrichment and concentration ratio of the concentrates obtained (Sink and Magnetic) differs extensively. The concentrate obtained from sink-float technique assaying 18.54% Fe₂O₃ at a recovery of 54.0% has enrichment and concentration ratio of 1.06 and 1.96 respectively, while the concentrate obtained from hand induced magnetic separation method assaying 33.01% Fe₂O₃ at a recovery of 22.78% has enrichment and concentration ratio of 1.88 and 8.26. Thus, it can be deduced that an inverse relationship exist between grade and % recovery, while enrichment ratio increases with increasing concentration ratio [11][18].

However, on account of the enrichment and concentration ratio being parameters used to investigate the efficiency of a separation method [18] thus, it can be said that magnetic method of separation is more efficient than gravity method of separation.

4. Conclusion

The sipped mineral having been characterized using XRF, SEM/EDS, and XRD, has been revealed to contain loosely bounded Silica and Iron, in the form of hematite. By way of adding value to this ore, susceptibility to gravity and magnetic test was carried out using sink-float technique and hand induced magnetic separation respectively; and it was found out that the mineral in the ore can be separated using both magnetic and gravity separation method though it is more susceptible to magnetic than gravity.

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