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Characterization and Beneficiation of Fanibi Laterite for Nickel Metal Recovery Using Froth Flotation Method

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Abstract: *This study investigates the characterization of Fanibi Laterite and the efficiency of froth flotation to beneficiate nickel from its host rock sourced in Akure, Ondo State, Nigeria. A sample of the laterite was obtained and characterized by the use of an X-ray fluorescence Spectrometer (XRFS), X-ray diffractometer (XRD), and Scanning electron microscope with Energy dispersive spectroscope (SEM/EDS). The ore was comminuted to its liberation size of -125 + 90 µm. The froth flotation process was done using Sodium hydroxide and Hydrogen tetraoxosulphate (VI) acid as hydrogen potential (pH) modifier within the range of 4,5,7,9 and 10. However, other reagents are sodium oleate as the collector, potassium dichromate as the depressant, and oleic acid as the frother. The froth and depressed obtained were dewatered and characterized using XRFS. The result revealed that the ore contained 35.65% Si, 8.24% Al, 10.87% Fe, and 0.07% Nickel. It also included some other associated minerals such as Zn, Mg, Ti, Co, and Mo in trace form of less than 1 %. XRD analysis reveals nickel as nickel iodate. The result reveals nickel assays of 0.14, 0.20, 0.15, 0.25, and 0.12%, while their recoveries are 70.82, 67.80, 86.70, 98.04, and 86.98%, respectively. The optimum nickel recovery of 98.04% was attained at a pH of nine (9). It was concluded that froth flotation could successfully beneficiate Fanibi laterite ore for nickel. At the same time, other minerals such as Titanium, Molybdenum, Tin, and Tungsten were present to be extracted for Metallurgical applications.*

Keywords: *Fanibi Laterite, Nickel, Froth Flotation, Recovery, Characterization.*

1. INTRODUCTION

Nigeria is a giant sitting on wealth; however, its citizens are still crying about hunger. Several mineral resources abound in the country, ranging from precious stones and solid minerals to industrial minerals. Nickel is a minor metal and mineral found in most laterite ore in Nigeria [11]. Laterite is soil produced in tropical climates, usually by prolonged weathering [17]. In the presence of oxygen and water, most water-soluble minerals are leached from their parent rock with non-soluble residues of hydroxides of aluminium, nickel, iron, magnesium and titanium [17]. Laterites that contain specific metals are mostly strip-mined as laterite ores. Laterites that occur in form of basalt rich in iron and nickel are called Ferruginous laterites [8]. Lateritic ores are excavated using large earth-moving equipment and are screened to remove boulders as they are very close to the surface and are in degraded sandy form [18]. Their closeness to the surface means they are mined significantly cheaply. Unfortunately, they require higher processing costs because they are less readily available [3].

1.1 Overview of the Occurrence of Nickel Laterite ore in Nigeria

Laterite, a reddish clay-like material, is abundant in Nigeria's market centres and most states, particularly Abia and Oyo State. It is used in construction due to its cost-effectiveness and water retention. However, mining laterite can lead to severe land degradation, erosion, loss of agricultural farmlands, and groundwater contamination. Land coverings, including grasses and plants, are removed during quarrying. Laterite deposits are primarily found in tropical African countries, and their extraction can lead to land degradation, erosion, and loss of agricultural farmlands [11, 12]. A more environmentally friendly technique is recommended to minimize environmental degradation caused by laterite mining.

Nickel formed the major valuable metal sought for in the laterite soil. Nickel ore, which occurs in laterite form in Nigeria, is locally found in Ife-Ilesha in Osun State [4]. It is also present in Fanibi area of Ondo road in Akure, Ondo State [12]. Due to the vast application of nickel due to its good mechanical properties such as strength, toughness, high melting point, corrosion and wear resistance, the demand for nickel is at increasing order [11, 12, 14]. Majority of economically mineable nickel ore is in the sulphide form, which consist just about 30% of the nickel ore deposit in the world, there is therefore need to develop economic means for extraction of the nickel from laterites.

The utilization of right mineral beneficiation methods are the foundation for innovation and adaptation technology for national development of a country. A country that neglects its mineral deposit endowments will ever remain underdeveloped and technologically backward in its national development plan [12]. The types of mineral processing methods available are classified into Magnetic, Gravity and Froth flotation [2].

Froth flotation is a method for physical separation of particles based on differences in the ability of air bubbles to selectively adhere to specific mineral surfaces in mineral/water slurry [10]. It is a process of separating and concentrating minerals based on differences in the physicochemical properties of interfaces in view. This can either be at a liquid-liquid, a liquid-solid, a liquid-gas or a solid-gas interface. Flotation is a very important process in concentration of minerals. Flotation is used in the processing of the bulk of the world's non-ferrous metals, as well as of a growing proportion of its iron, non-metallic minerals, and coal [6].

The metallic mineral resources of Nigeria have not been fully appraised due to lack of sufficient studies and technology [4]. Therefore, urgent attention is needed in the Solid Mineral sector of the country. Hence the need to characterize and beneficiate Fanibi laterite for nickel metal recovery using froth flotation method which this study is aimed at.

2. MATERIALS AND METHODS

2.1 Materials

Material used at the course of this research was Laterite ore sample sourced from Fanibi area of Akure, Ondo State, Nigeria with geological coordinate 7° 15' 2.7756" N (latitude) and 5° 12' 36.9576" E (longitude). Chemicals and reagents used for froth flotation experiment are Sodium hydroxide and Tetraoxosulphate (vi) acid ($NaHSO_4$) as pH controller, Sodium Oeate ($C_{18}H_{33}NaO_2$) used as collector, Potassium Dichromate ($K_2Cr_2O_7$) as depressant, Oleic acid ($C_{18}H_{34}O_2$) as the frother and Distilled water. The flowsheet adopted for this research is presented in Figure 1.

2.2 Methodology

The procedure adopted for this research includes Sample collection and preparation, followed by chemical and mineralogical assessment of the ore, determination of the ore's physical properties, and sieve analysis leading to liberation size determination. The froth flotation method was used to beneficiate the laterite ore at varying hydrogen potential (pH) levels.

2.2.1 Sample collection and preparation

Samples were collected from 10 different pits dug at a dimension of 4 m by 6 m by 4 m (length, breadth and height respectively). Sample from these pits (on the mine site) were homogenize by mixing thoroughly using a hand shovel. The homogenized samples collected in lumps size were crushed manually using a 5 kg sledgehammer to provide a required size (5 mm passing) acceptable to a laboratory Jaw crusher (Fritsch pulverisette, Model No. 01302) at the Mineral Processing Laboratory, Metallurgical and Materials Engineering, Federal University of Technology, Akure. To have a true representative fraction of nickel from the bulk on the mine site (Study Area), a random sampling technique was used to draw 50 kg of the crude laterite ore. Two kilograms (2 kg) were sampled from the lot using the Jones riffle splitter, followed by a random sampling method. This sample was then grind to 100% passing through a 1700 µm sieve. Chemical and mineralogical analyses were carried out using Energy Dispersive X-ray Fluorescence Spectrometer (EDXRF), X-ray Diffractometer (XRD) and Scanning electron microscope with an Electron Dispersive Spectrometer (SEM/EDS). Thereafter, 100 grams of the ground sample was used for sieve analysis to determine the liberation size. The specific gravity of Fanibi laterite ore was determined using the density bottle method. The picnometric (density) bottle was washed thoroughly to remove dust and then weighed and the weight was recorded (M_1). The ground sample was filled into the specific gravity bottle to about $\frac{1}{3}$ of the bottle and weighed (M_2), the remaining space in the bottle was filled with water and weighed (M_3), and the content of the bottle was discarded and filled afresh with water and the weight of bottle filled up with water (M_4) was determined. The experiment was repeated thrice and the average was taken. These parameters were substituted into Equation 1 [1].

$$\text{Specific Density of the Ore (S. G.)} = \frac{M_2 - M_1}{(M_4 - M_1) - (M_3 - M_2)} \quad (1)$$

Where:
Mass of empty bottle (M_1)
Mass of empty bottle + ore (M_2)
Mass of empty bottle + ore + water (M_3)
Mass of empty bottle + water (M_4)
Mass of water filling the bottle ($M_4 - M_1$)
Mass of water having equal volume as ore = $(M_4 - M_1) - (M_3 - M_2)$
One kilogram (1 kg) of the sample was used for the concentration of the laterite ore using froth flotation method. One hundred grams (100 g) of the laterite ore samples were used for each set of froth flotation experiment.

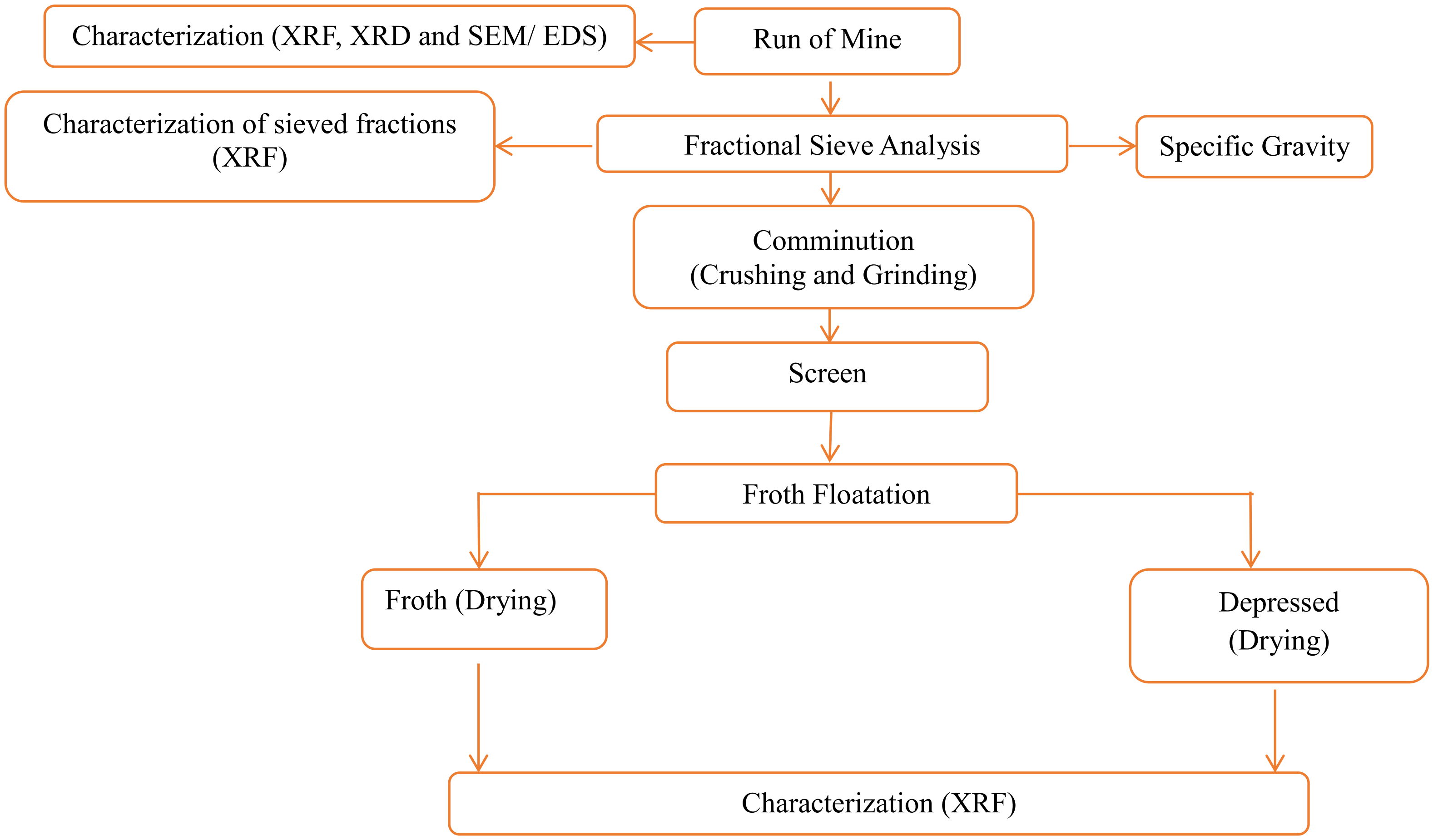

Figure 1: Process Flow Sheet

The samples were transferred to a 3 Litres Denver D-12 flotation cell and 10 % weight / Solid of the laterite samples were used as the pulp density of the slurry. Impeller sped was kept constant at 1300 rpm. Weighed laterite ore was charged into a 3 litres froth flotation cell and 1 litre of water heated to a temperature of 100ºC was added to the pulp. The machine was switched on and the initial Hydrogen Potential (pH) of the pulp was checked and left to agitate for one minute. The pulp pH was regulated first to a PH of 4, and later 5, 7, 9 and 10, using Hydrogen Tetraoxosulphate (VI) and Sodium Hydroxide, then one gram (1 g) of depressant (Sodium Dichromate) was added to the pulp and the pulp was further conditioned for 2 minutes, 0.5 gram of the collector (Sodium Oleate) was added and the pulp was further conditioned for 2 minutes. One millilitre (1 ml) of frother (Oleic acid) was then added to the pulp and another 2 minutes of further conditioning air was introduced through the air inlet valve. The air inlet was then turned on fully and scheming of the froth was continued for 3 minutes. The procedure was repeated at varying pH of 5, 7, 9 and 10 [10]. After froth flotation, each product of froth flotation recovered (froth and depressed) were allowed to settle for 24 hours before decanting, filtered, dried at a temperature of 105 °C and weighed. Energy dispersive X-ray Fluorescence (ED-XRF) was used to analyse both products of froth flotation (froth and depressed).

3. RESULT AND DISCUSSION

Table 1: Elemental composition of nickel laterite ore

Element	Al	Si	P	K	S	Cr	Co	Ni	Fe	W	Nb	Mo	Sn
% Composition	8.23	35.65	0.23	0.98	0.80	0.02	0.15	0.07	10.87	0.01	0.02	0.19	1.26

Table 2: Elemental composition of nickel laterite ore

Element	Content
Mg	BDL
Al	8.2388
Si	35.6446
P	0.2386
S	0.8014
K	0.9797
Ca	0.2044
Ti	0.0302
V	0.0108
Cr	0.0099
Mn	0.0238
Co	0.1576
Fe	10.8713
Ni	0.0702
Cu	0.1024
Zn	0.0974
As	BDL
Pb	BDL
W	0.0112
Au	BDL
Ag	BDL
Rb	0.0042
Nb	0.0158
Mo	0.1991
Cd	BDL
Sn	1.2640
Sb	1.2103

BDL – Below Detectable Level

3.1 Characterization of the Head Sample of Nickel Laterite Ore

The result of the elemental analysis of nickel laterite ore under study using ED-XRF is presented in Table 1-2. This result revealed that the ore contained 35.65% Si, 8.24% Al, 10.87% Fe and 0.07% of Ni. It also contained some associated minerals such as Ca, K, Zn, Mg, Ti, Mn, Co, and Mo in trace form - less than 1%. [7]; [9]; [5]; [16]; and [17] confirmed that the Nickel laterite ore contains Si, Fe and Al as its major composition and contaminate; and this confirmed the presence of nickel (< 1.5 %) in its oxide form. However, the study in [16] confirmed that the percentage of Nickel in a laterite ore is less than 1.5%. The studies in [7]; [5]; [15]; [16]; [9]; and [14], also confirmed Ca, K, Zn, Mg, Ti, Mn, Co, and Mo as trace elements and associating minerals of nickel laterite ore. Hence, this initiated the need for the beneficiation of the nickel laterite ore in order to upgrade its nickel content.

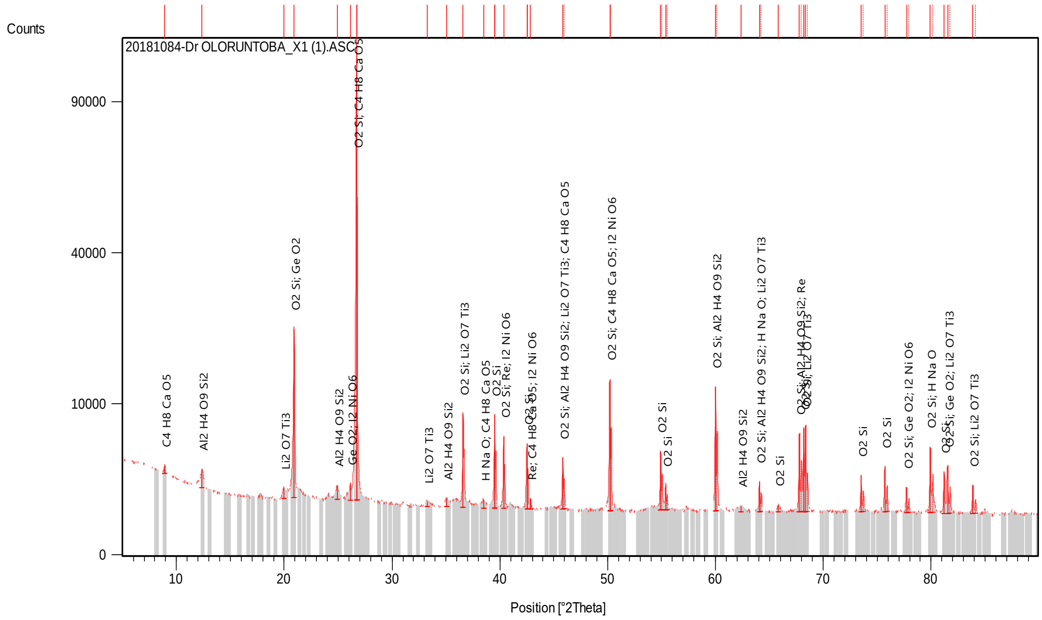

Figure 2: XRD pattern of the head sample of Fanibi laterite ore showing the major diffraction peaks of the minerals compound, appearance and corresponding chemical compound name.

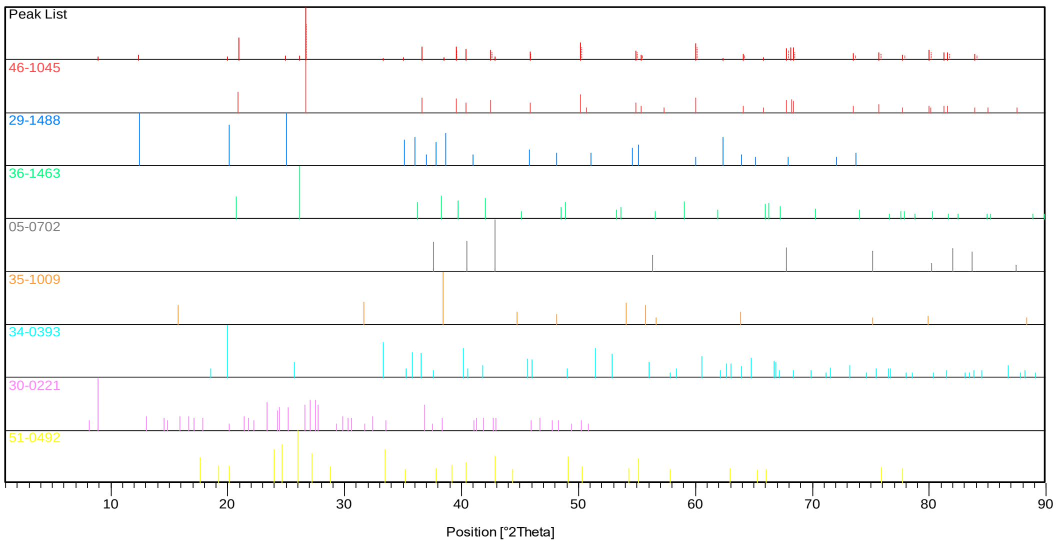

Figure 3: XRD pattern of the head sample, indicating the major diffraction peaks of the minerals in the ore and their reference codes

Table 3: The mineral phases present in nickel laterite ore with reference code

Mineral Name	Formula	Reference code
Quartz, syn	SiO_2	46-1045
Kaolinite-1\itmd\rg	$Al_2Si_2O_5(OH)_4$	29-1488
Germania	GeO_2	36-1463
Rhenium, syn [nr]	Re	05-0702
Sodium hydroxide	Na(OH)	35-1009

Lithium titanium oxide	$Li_2Ti_3O_7$	34-0393
Unnamed mineral, syn [nr]	$C_4H_6CaO_4!H_2O$	30-0221
Nickel iodate	$Ni(IO_3)_2$	51-0492

3.2 Mineralogical Characterization of Nickel Laterite Ore

i. X-Ray diffraction

The result of XRD analysis of the bulk ore sample is presented in Figures 2-3 and Table 3. The background and peak position were identified and based on the peak position a search match was performed. After a proper investigation, it was observed that Quartz, Kaolin, Germania, Rhenium, Sodium hydroxide, Lithium titanium oxide, and Nickel iodate were present in the laterite ore; the analysis reveals nickel (mineral of interest) as Nickel iodate. Therefore, the result obtained confirms the presence of the mineral of interest (Nickel) in the laterite ore.

However, other associating major constituents include silicon, aluminium, sodium, lithium, and titanium. The finding obtained is in accordance to [16] and [14] research, where it is revealed that the major constituent of nickel laterite ore may contain Iron, Silicon and Magnesium. However, the software application failed to identify the name of a mineral having $C_4H_6CaO_4!H_2O$ as its chemical formula.

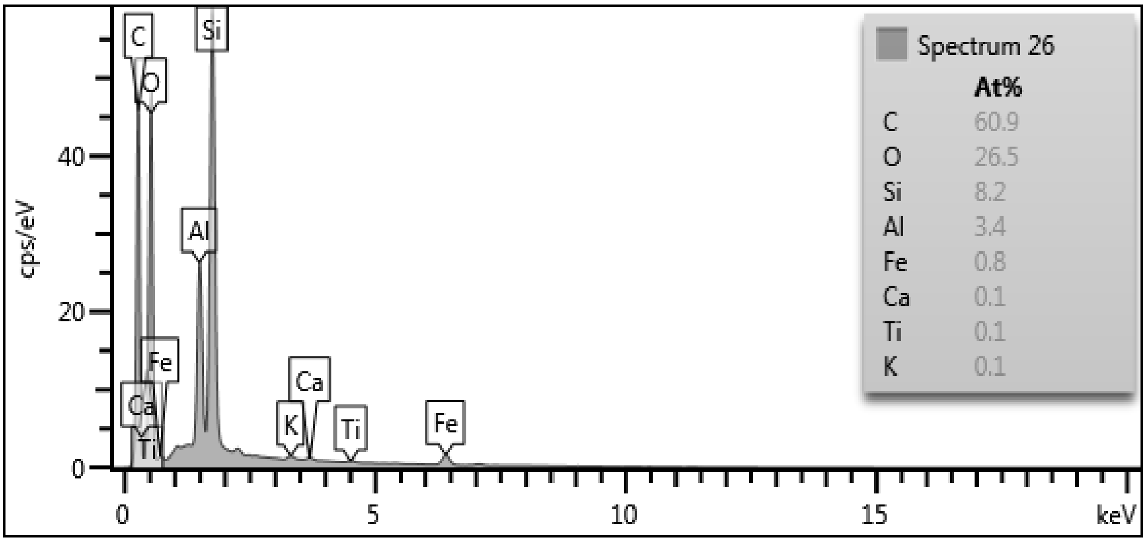

Figure 4: EDS pattern of nickel laterite ore at spectrum 26

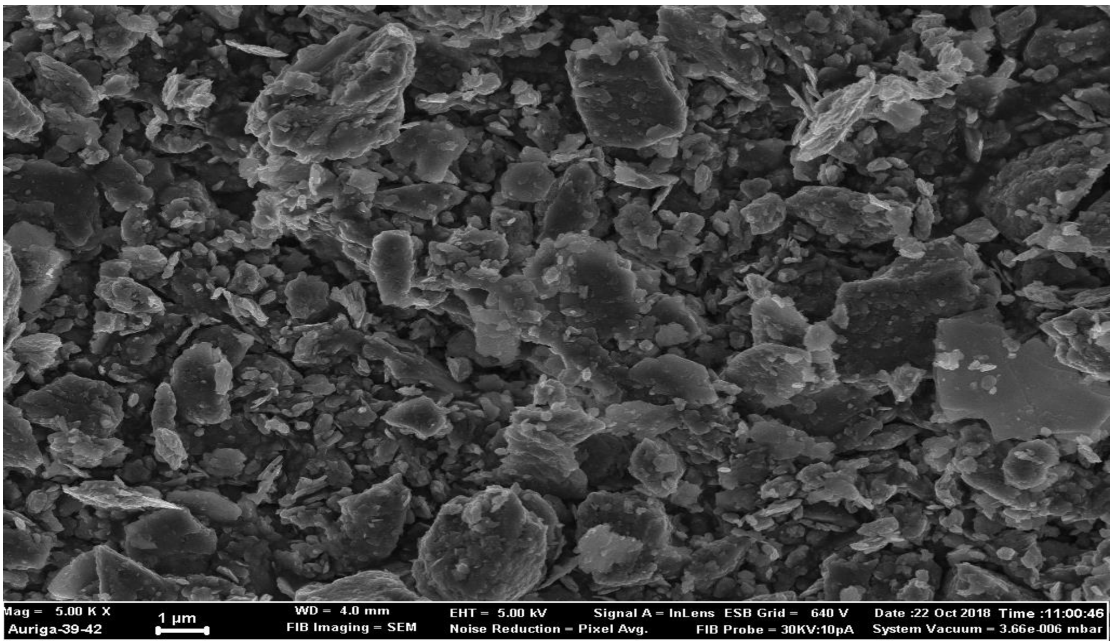

Figure 5: SEM image of nickel laterite ore (1 µm)

ii. Scanning electron microscopy / Energy dispersion spectrometer (SEM/EDS)

Figures 4 and Table 2 shows the EDS pattern-of Nickel Laterite Ore at Spectrum 26 with the presence of nickel present and the SEM images (backscattered electron images) of the Nickel Laterite Ore deposit as received at varying magnification level and the general microstructure of the particles respectively. The greyscale in the image shows the relative mean atomic number of the material, thus representing differences in compositions. In general, complex structures, such as composite, intergrowth, vein-like/plate-like, porous and dense structures, were observed in the particles in the ore feed sample. The SEM image (Figure 5) revealed the presence of aluminum, potassium, iron, titanium, silicon, oxygen, calcium, carbon and other minerals in low concentrations. Although, the analysis failed to reveal quantitative value of nickel (mineral of interest). This could be attributed to its low concentration, which is below detectable level. However, the morphology of the ore shows combination of the different particle sizes with irregular shapes with large grain boundaries and general mineral species names were reported: no attempts were made to distinguish the mineral species. This finding was found to match with, those in [16]; [1] and [14] revealing that after communition of nickel laterite ore, there are different particle sizes with irregular shapes to liberation size of -125 + 90 µm.

Furthermore, it could also be observed that the minerals are separated by grains boundaries, no interlocking of minerals and the mineral particles vary in sizes ranging from 1 to 10 µm. This phenomenon indicates that the minerals can be easily freed from each other during communition. This result is found favourable with the studies in [16] and [13] as low energy will be required for the liberation of the mineral.

Table 4: Result of specific gravity test of the nickel laterite ore

S/N	M_1 (g)	M_2 (g)	M_3 (g)	M_4 (g)	S.G.
1.	20.22	55.79	82.49	62.08	2.35
2.	20.22	57.66	84.12	62.08	2.43
3.	20.22	47.04	77.30	62.08	2.31

Average Specific Gravity = 2.36
Where:
Mass of empty bottle (M_1),
Mass of empty bottle + ore (M_2)
Mass of empty bottle + ore + water, (M_3),
Mass of empty bottle + water (M_4)

iii. Specific gravity test of nickel lateritic ore

The result of the specific gravity test carried out on the nickel laterite ore is as shown in Table 4. The average specific gravity of the nickel lateritic ore was found to be 2.36. This value was compared favourably with the specific gravity value of laterite as 2.4 cited in literatures by Wills and Napier-Munn, (2006). This specific gravity value of 2.36 obtained from the ore sample indicates that the ore is relatively dense and can also be beneficiated using gravity separation method as stated by Wills and Napier-Munn, (2006).

Table 5: The grade of nickel in the froth at varied pH from the chemical analysis

pH	4	5	7	9	10
% Ni grade	0.14	0.2	0.15	0.25	0.12

Table 6: The grade of nickel in the depressed at varied pH from the chemical analysis

pH	4	5	7	9	10
% Ni grade	0.1	0.07	0.03	0.03	0.08

iv. Concentration test

Table 5 shows the grade of nickel in the froth and depressed respectively after beneficiation using Froth flotation method. The nickel grades of the processed were obtained to be 0.14, 0.20, 0.15, 0.25 and 0.12 with pH 4, 5, 7, 9 and 10 respectively.

Table 6 is the result obtained from the XRF analysis of the froth flotation products (froth and depressed samples), showing the grade of nickel in the froth at pH 4, 5, 7, 9, and 10. Generally, the analysis reveals that nickel (mineral of interest) was gained to the froth while iron and silicon (major contaminants of the ore) were gained to the depressed. These have proven the effectiveness of both the collector and depressant.

The metallurgical accounting of the processed nickel laterite sample showing the grade, recovery, concentration ratio and enrichment ratio on the nickel recovery is presented in Table 7. The result reveals that nickel assay are 0.14, 0.20, 0.15, 0.25 and 0.12 all in percentages; while its recovery are 70.82, 67.80, 86.70, 98.04 and 86.98 % respectively. This simply means that 70.82% of the nickel present in the processed sample was recovered in the concentrate (froth), while the rest was lost in the tailing (depressed). The result also indicated an increase in recovery within the neutral to alkaline region (pH 7, 9 and 10) compare to the acidic region (pH 4, 5 and 7). The result revealed that the grade, recoveries, enrichment ratio and ratio of concentration were affected by increasing the pH of the pulp slurry. The optimum nickel recovery of the processed nickel ore samples was attained at pH of 9, having a recovery of 98.04%.

Table 7: Metallurgical accounting of nickel laterite ore

pH	Charge (g)	Froth (g)	Froth Nickel Grade (%)	Depressed (g)	Depressed Nickel Grade (%)	Recovery (%)	Enrichment Ratio	Concentration Ratio
4	100	35.41	0.14	63.07	0.1	70.82	2.824	2.000
5	100	23.73	0.20	75.38	0.08	67.80	4.214	2.857
7	100	40.46	0.15	58.37	0.07	86.70	2.470	2.143
9	100	27.45	0.25	71.82	0.03	98.04	3.642	3.571
10	100	50.74	0.12	48.14	0.08	86.98	1.970	1.714

4. CONCLUSION

The concentration and beneficiation of Fanibi Laterite ore for Nickel metal were carried out and the following conclusions were drawn:

i. Nickel laterite ore was found to contain 0.0702% Ni alongside some associated minerals such as Ca, K, Zn, Mg, Ti, Mn, Co, Mn and Mo found in trace form which is less than 1%,
ii. XRD analysis reveals nickel (mineral of interest) as Nickel iodate. The nickel bearing minerals are separated from other associated minerals in the ore by large shapeless grains boundaries and non-interlocking by another mineral,
iii. the optimum Nickel recovery of 98.04% was attained at the pH of 9.

Therefore, Froth flotation can be successfully used in the beneficiation of Fanibi laterite Ore for Nickel, while other minerals such as Titanium, Molybdenum, Tin, and Tungsten were present for Metallurgical applications.

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