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Up-Grading Of Low-Grade Wolframite Ore from Rafin-Gabas, Nasarawa State, Nigeria

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ABSTRACT
This research aimed at finding suitable methods four upgrading Wolframite from Rafin-Gabas, Nasarawa State for industrial applications. The process commenced by beneficiating it using two standard methods namely; Rapid Magnetic Separation (RMS) and Air Float Separation (AFS). The crude ore and the beneficiated samples were analysed to detect their chemical compositions using XRF analytical technique. Prior to detection, the crude ore were authenticated using Leica (DMRX) microscope, liberation size determination through sieve analysis. The sieve size analysis helped to establish the liberation size of the wolframite as +0.250mm. After beneficiation, RMS method upgraded the ore to meet industrial standard by 62%W from the crude ore which is 13%W, and AFS methods gave 50%W. From the results, RMS method was better than the AFS method, because it gave higher yield of the wolframite. This study provides a useful guide for upgrading of wolframite for further industrial usage.

Keywords: *Up-grading, Low-Grade, Wolframite Ore, Rafin-Gabas.*

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I. INTRODUCTION

Nigeria is endowed with metallic and nonmetallic mineral resources, however, owing to the low level of exploration of the mineral deposits most of the known mineral occurrences are yet to be evaluated for their reserves and grade (Jamil, 2015). Nigeria is blessed with rich human and several mineral resources that are widely distributed. There are over thirty-four mineral commodities spread across the entire country, which include large deposits of iron ore along the Kogi-Benue through, Agbaja iron ore deposits (which is the largest iron ore deposit in Nigeria with about one billion tones in reserve). Reserves of good quality coal low in sulphur and ash are about over two and half million tons (Balasubramanian, 2015).

Wolframite is the main mineral as a source of tungsten metal.This lead to a high mining rate of wolframite as a result of increasing global demand of tungsten ores, which are in huge need for alloys all over the world.Tungsten ore producing countries have increased their production by initiating steps to utilize the low-grade complex tungsten ores. Tungsten has a wide application in industries such as high temperature technology, chemical, lighting, X-ray technology, Emission Spectrometry, machine construction due to its low vapor pressure, high melting point, good electrical and thermal conductivities, high density, high elastic modulus, high wear resistance, and good X-ray performance (Ilhan et al., 2013). It is one of the strategic metals identified for stockpiling in many countries.

Wolframite is not scientifically classified as an individual mineral species by the IMA (international mineralogical association). However, it is universally recognized as a mineral series, with the Huebnerite and Ferberite being its end members. Huebnerite is the manganese rich end member, and Ferberite is the iron rich end member. The term wolframite can be used generally to describe unspecified members of this group or to describe the intermediary member of this series. Meanwhile, wolframite is often associated with other minerals such as quartz, calcite, fluorite, and apatite. Therefore, wolframite must be separated from gangue minerals (Kim et al. 2020). Wolframite is named for its tungsten content, since wolfram means tungsten in German (Wills, 2016).

Wolframite from Rafin Gabas in Nassarawa state

The most commonly used beneficiation methods for tungsten ores involves crushing-grinding to liberation size followed by gravity separation and magnetic separation, which are widely used in mineral beneficiation practices for their low-cost, easy-to-control, and eco-friendly advantages. However, since the gravity separation is uncertain, flotation becomes an attractive alternative process (Deng et al., 2015; Gabi et al., 2020).

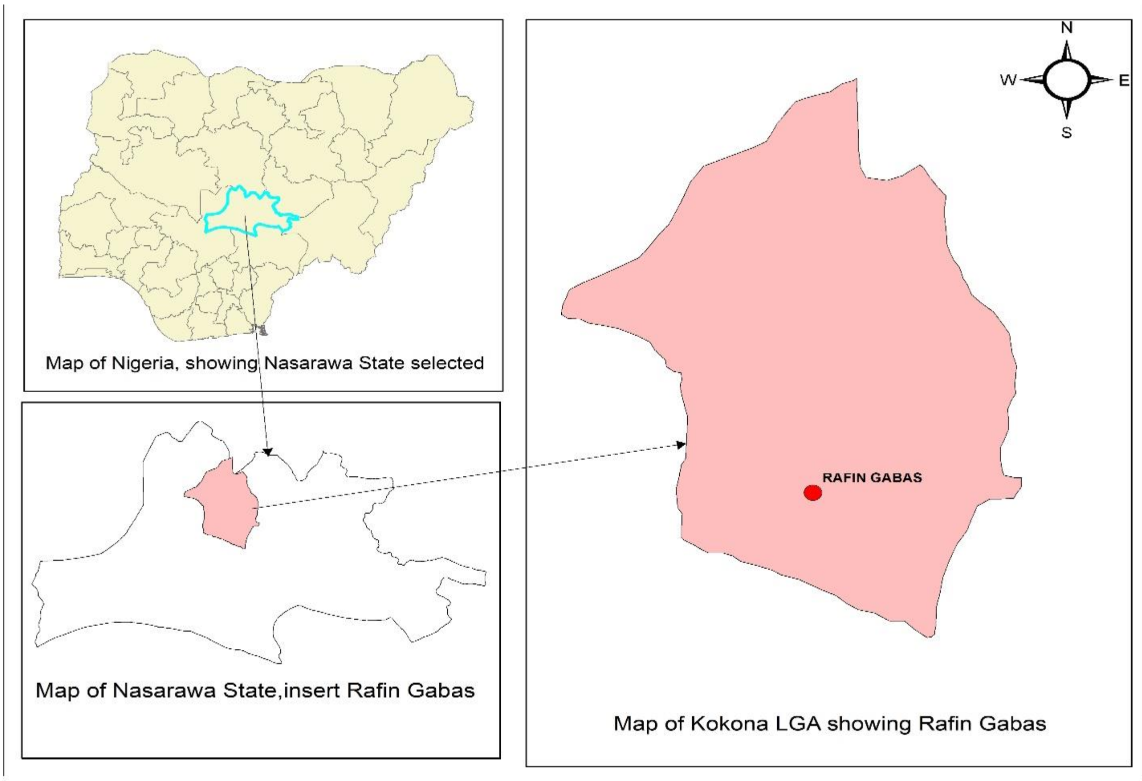
Wolframite is the principle mineral of tungsten and is often associated with other minerals such as quartz, calcite, fluorite, and apatite. Therefore, wolframite must be separated from gangue minerals (Dawei et al.,1986). The main beneficiation methods of wolframite is gravity and magnetic separation (Xian-Ping et al., 2017). However, since the gravity and magnetic separation is uncertain, flotation becomes an attractive alternative process (Deng et al., 2015). The separation method must ensure that concentrate lost in tailing is of no economic significance; it will be particularly vital to utilize high-efficiency preparation equipment, simple and feasible process and efficient beneficiation reagents to recycle fine wolframite slime (Chen et al., 2013).

For purpose of beneficiating any type of ore for its minerals or metal values the ore is subjected to some concentration processes that can separate the minerals into two or more products. Separation is usually achieved by utilizing some specific differences in physical and chemical properties between the valuable and gangue minerals in the ore. However, mineral processing operations are mainly concerned with utilizing the physiochemical methods of separation. Some of these methods are: gravity, magnetic, froth flotation. The wolframite deposit at Rafin Gabas forms part of the Pan-African mobile belt and lies between the Pan-African mobile belt and lies between the West African and Congo Cratons and south of the Tuareg shield (Burke & Dewey, 1972; Black, 1980). The rock types in Rafin Gabas include coarse grained silicified biotite granite, dark grey fine-medium grained amphibolite, granite gneiss, and coarse-grained quartz schist.

The coarse-grained silicified biotite granite (Younger granite) occurs as undulating hills trending North-South. The Rafin-Gabas village is located in the valley between two of such hills. The low-lying outcrops are usually decomposed and greissenised. The grainite is composed of quartz, microcline perthite, and mica + muscovite and ferromagnesium minerals. Tin, wolframite, Topaz chalcopyrite are disseminated or are found in pockets on this rock.

Amphibolites are dark grey medium grained rocks occurring as boulders/ low lying outcrops in contact with gneisses. Boulders of amphibolite also occur at latitude 08° 29.55'N and longitude 08° 02.52'E. Quartz schist occurs together with mica schist and quartz boulders on the crest and slopes of the ridges. It is a brown white fine-sugary grained rock consisting of mainly quartz, feldspars and sericitic mica on the surfaces.

Biotite-gneiss also form part of the rock type in the project area. They are characterized by abundant oligoclase and contain little microcline. They are well foliated. The granite gneisses are formed under low to medium grade metamorphism, though rare occurrences of high grade charnockitic rocks have be noted elsewhere. Those gneisses with high content of mafic minerals may weather to clayey soils. The wolframite mineralization of the Rafin Gabas area is confined to biotite granite and the gniess/amphibolite group which borders the grainite



Map of Study Area

II. MATERIALS AND METHOD

Materials such as GPS, camera, boots, compass, hammer, head pan, bucket were collected from the geology and mineralogical section of National Metallurgical Development Centre. Markers, sample bags, Paper tape, plastic bags were purchased from the market.

2.1.1 Equipment

In beneficiating crude Rafin-Gabas wolframite ore the following Equipment were used. Jaw Crusher (For primary crushing), Cone Crusher (For secondary crushing), Roll Crusher (For secondary crushing), Sieve Shaking Machine/Test sieve **(**For sieve analysis), Pulverizing machine, Ball Mill Machine (for grinding to liberation size), Rapid Magnetic Separator (Dry magnetic method). Kipp Kelly Air-Float Separator Dry gravity method).Kern weighing scale of 600gms capacity, MJ – Peps – 002 Electronic computing price scale 300kg/100g capacity, 8 Nos. of head pans, 8 nos. of 20 litres capacity buckets, Hammers (For breakage of boulders and fresh samples.), Sample Bags **(**for **c**ollection of Representative samples.), Camera (For photographing features of interest.), Global positioning system (GPS used to determine the elevation, finding positions and locations.), Sample Bags (For collection of Representative samples).

2.1.2Methods Sampling

Google satellite map was used as a reference map to locate the wolframite deposit in Rafin-Gabas and the villagers were also helpful. Fresh samples of wolframite ore were randomly chiseled out from the major rock exposures in Rafin-Gabas, Nassarrawa State; using geologic hammer and chisel while Monday hammer was used to break boulders where necessary from various locations within the Rafin-Gabas wolframite deposit, these were properly labeled and packed in sample bags(Barnes, 1981).

2.1.3 Sample Preparation

Fresh representative samples were thin-sectioned and some were mounted and polished for sample Identification studies. Samples were grinded for sieve analysis in order to determine the liberation size of Rafin-Gabas wolframite ore; samples were also pulverized and pressed into pellets and chemically analyzed by instrumentation method.

2.1.4 Microscopic Analysis

The application of microscopy to study the polished surface of Rafin-Gabas wolframite ore to processing is indispensable. The Rafi-Gabas sample was placed in a holder with bakelite of about 20 to 30 ml poured into it; then the sample was mounted and the mounting press is turned on. The machine now begins to heat and press the bakelite to mount the Rafin-Gabas wolframite sample. After which I have to wait for like 10minutes for the machine to cool before removing the produced mount of wolframite ore after the mounted sample was then polished with the polishing machine using grits of different texture and finally the sample was polished using the diamond slurry till the section is properly polished. Many problems can be solved or sufficient information secured by studying polished block of the representative fragments of the rafin-Gabas wolframite ore. The major parameters that determine the method of concentration of an ore are mineral composition, texture, grain, size and locking types. These parameters are determined by microscopic examination (Jamil, 2015). In order to characterize the ore and for mineralogical analysis, the samples were studied using the reflected light microscopy.

2.1.5Reflected Light Microscopy

A Leitz research microscope equipped for reflected light microscopy was used to study opaque minerals in polished sections. Using the complimentary aspects of microscopy, polished blocks of ore were studied in plane polarized light (PPL) and cross polarized light (XLP) utilizing characteristic features that allow partial or conclusive identification (Jamil & Tersoo 2012). The polished mounted Rafin-Gabas wolframite ore was placed on the stage of the microscope and the reflected light was focused on polished face of the wolframite ore with image being transmitted to the monitor where it is first analysed before being captured.

2.2 BENEFICIATION

Crushing is the first mechanical stage in the process of communition, which is the liberation of the valuable minerals from the gangue. It is usually a dry operation. The Rafin-Gabas wolframite was crushed from lump size of about 1.5m with the Jaw crusher at the mineral processing laboratory of National metallurgical Develeopment centre to a reduced size within 70-100mm; it was then moved to the Cone crusher where it was further reduced to sizes ranging from 5-10mm after the wolframite sample was transferred to the roll crusher which further reduced the ore to sizes ranging from fine to 2mm.

2.3 DETERMINATION OF LIBERATION SIZE OF THE WOLFRAMITE ORE

Sieve analysis was carried out on the crushed wolframite ore to determine the liberation size as stated below: Arrangement of sieve was done according to the formula: √2system (Wills, 2006). 100 grams of representative sample was weighed (Mettler AE 2000 Balance). The sample was then placed into a stack of arranged test sieve. The sieves used for analysis are of the following mesh sizes; 1400µm, 1000µm, 710µm, 500µm, 355µm, 250µm, 180µm, 125µm, 90µm and 63µm. The sieves were arranged in descending order and a tight-fitting pan placed at the bottom to receive the final undersize and a lid was placed on the top of the coarsest sieve to prevent loss of sample.

The weighed sample was put on the top of the coarsest sieve (1400µm) and the stack of sieves placed on an Endecott test sieve shaker and allowed to shake for 15 minutes. Each size fraction was carefully weighed., The weight was recorded and the sample stored in labeled sample bags. All the samples were chemically analyzed by instrumentation method to determine the liberation size of the ore. The size obtained was -250um+180um, which was confirmed by ED-XRF analysis result (Tables 1 and 2) before grinding.

2.4 GRINDING: The liberation size was obtained as -250um+180um of sieve fraction, therefore 50kg of the crushed ores was ground into liberation size using the Bond's test ball mill of dimension Φ317×305mm to produce materials of -0.355mm for processing.

2.5 RAPID MAGNETIC SEPARATOR

The crushed ore (5kg) was fed into the machine's conveyor belt through a hopper at a controlled rate of loading. The belt moved over sets of rollers, across three magnetic discs having magnetic field arranged in an increasing order. This will enable attraction of both magnetic and Para-magnetic materials making them fall into side collection bins, while the non-magnetic materials (gangue) go along the belt and falls into a collector at the front end.

2.6 AIR FLOAT SEPARATION TECHNIQUE

Air floatation machine, another gravity equipment was used. It is an oscillating deck, inclined longitudinally with a fined porous meshed cover cloth. Air was passed upward the deck under pressure at accurate air-controlled rate. The crushed ore (5kg) was fed by gravity from a hopper on to the table. The air going through the meshed cloth acts as a pneumatic cushion upon the settling particles. The combined pressured air and the oscillating motion makes the entire deck load becomes fluid in motion, thus effecting separation by density difference. The heavy particles (ore concentrate) fall and displaced the lighter particles (gangue) and moves across the deck end where they are discharged into receiving buckets. These are then pulverized and sent for analysis.

2.7 CHEMICAL ANALYSIS

Representative samples of the major rock units including the crude ore deposit, sieved size fraction and the processed wolframite ore (Concentrate and Tailings) were prepared for chemical analysis to determine the major, minor and trace elements composition.The rock samples were broken down into chips by hammer and subsequently reduced further by a cone crusher and later pulverized in a 3-disc Bico industrial pulverizing machine for two minutes to a fine size of 200unit mesh size. High grade of pure quartz was used for cleaning between each sample to avoid contamination. A representation sample was obtained through quartering. The prepared samples were packaged in sample envelops, labeled and analyzed at Instrumentation Laboratory of the Department of Science Laboratory Technology (SLT), University of Jos, for analysis using XRF

2.7.1 Sample Preparation for XRF

Standards and samples are ground to the same mesh size, preferably, finer than 200 mesh. Powders are pressed into pellets in a metallurgical specimen pressed orconverted into a solid solution by fusion with Borex.

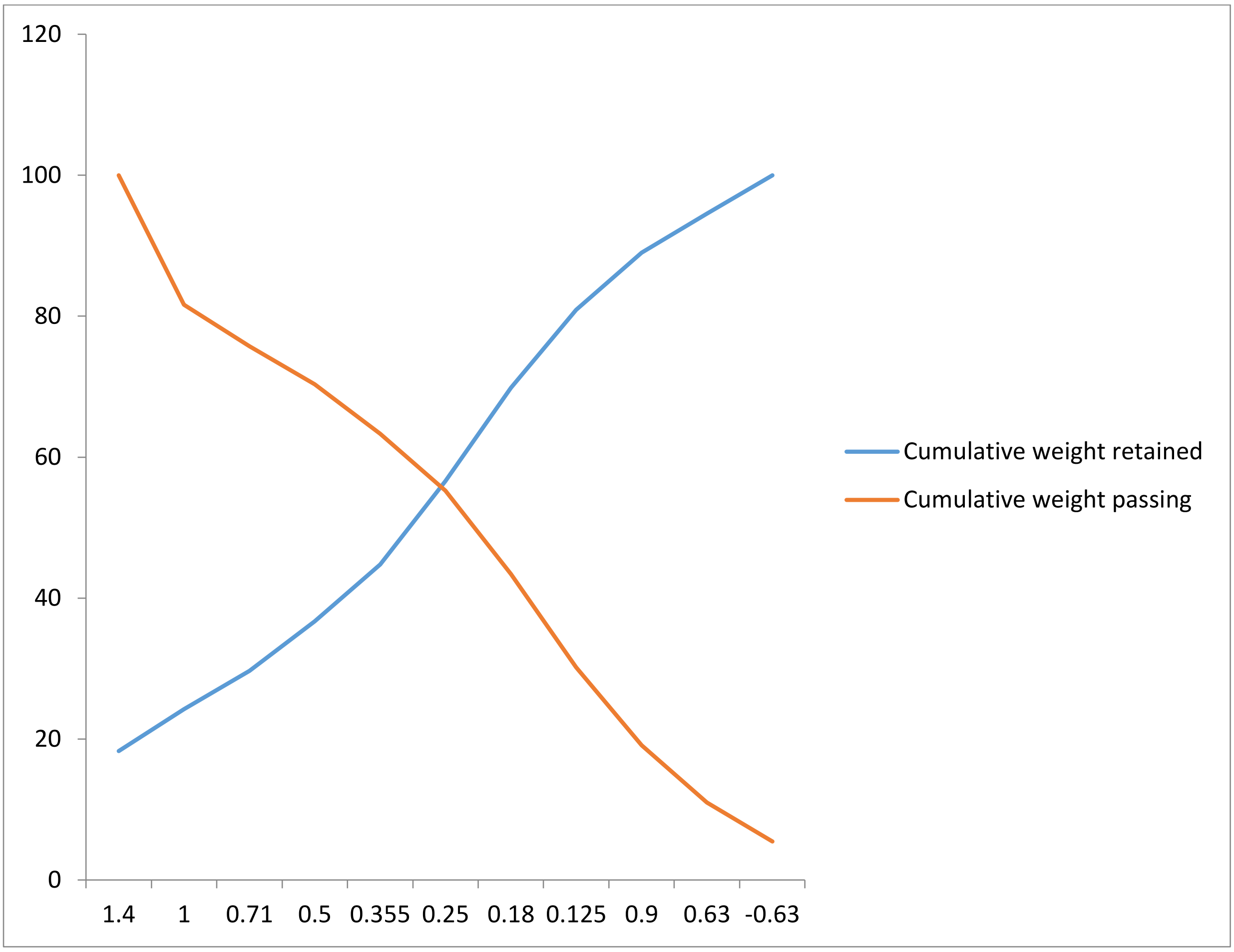
2.7.2 Analytical Technique

X-Ray Fluorescence (XRF) was used for the determination of the Major (W, Zr, Fe, Mn, Bal, Au, K, Ca and P_2O_5) minor and trace (Cr, Nb, Cu, Sr, Sn, Pb) elements in the samples. Each sample was fused with borax and mould pressed into pellets. The pellets were then placed in the machine and analyzed.

III. RESULTS AND DISCUSSION

Table 1: Sieve Analysis of Liberation Size

Sieve Size Fraction (mm)	Weight (gm)	Weight (%)	Cummulative Weight Retained	Cummulative Weight Passing
1.4	91.50	18.36	18.36	100.00
1.0	19.33	5.89	24.25	81.64
0.71	27.06	5.43	29.68	75.75
0.50	34.98	7.02	36.70	70.32
0.355	40.10	8.05	44.75	63.30
0.250	59.03	11.85	56.60	55.25
0.180	65.89	13.22	69.82	43.40
0.125	55.15	11.07	80.89	30.18
0.90	40.46	8.12	89.01	19.11
0.63	27.68	5.55	94.56	10.99
-0.63	27.13	5.44	100.00	5.44
TOTAL	498.31	100.00		



: LIBERATION SIZE GRAPH

TABLE 2ELEMENTAL COMPOSITION OF SAMPLE IN EACH SIEVE SIZE (mg/kg)

Sieve size (mm)	W	Si	Ca	Au	K	Se	As	Fe	Mn	Sn	Bal	Nb	Bi	Ti	Sc
+ .1.4	436800. 125	79.532	1656.307	3197.650	1799.075	1149.649	254.722	95087.648	10444.952	67926.516	372688.594	4333.976	1041.106	2358.246	56,286
- 1.4+1.0	481658. 781	149.230	1375.234	3888.906	1256.298	1281.218	280.687	96951.000	11769.269	76566.570	315675.188	4473.663	513.152	2919.094	90.943
- 1.0+0.71	437547. 406	291.029	2352.872	3224.682	1564.761	1214.762	266.002	87983.563	11152.112	112680.961	329614.844	4270.231	1010.091	4791.516	59.448
- 0.500+ 0.355	508714. 438	416.364	1922.911	3468.136	927.658	1515.039	257.620	102346.109	12658.571	30103.076	325079.156	4573.512	1262.977	5586.173	83.910
- 0.355+ 0.25	494711. 344	239.105	1295.731	3170.976	807.938	1409.606	276.578	98203.414	12396.939	63675.586	3141.177.250	4627.333	1131.734	2917.752	70.917
- 25+ 0.180	475521. 063	569.818	ND	4006.360	632.007	1609.950	376.607	140208.000	32420.613	34560.020	244429.281	8656.966	823.998	53448.297	ND
- 0.180+ 0.125	385455. 844	342. 903	12354.563	2333.747	7415.079	1006.873	161.138	86032.719	10793.565	16923.502	449668.344	3770.646	2517.328	17182.586	37.819
- 0.125+ 0.09	421183. 844	430.695	4762.356	2569,622	6446.694	1174.262	216.060	90349.586	10778.807	32050.650	411078.563	4236.901	2401.744	7218.983	37.937
- 0.09+ 0.063	422355. 219	124. 483	4218.177	2642.093	8587.208	1169.877	212.815	86902.820	10317.821	21212.711	431436.375	4556.667	1888.483	2123.450	28.222
- 0.063	437029. 750	197. 027	6491.607	2783.676	13668. 770	1135.107	179.532	91828.555	10702.769	28175.570	391326.969	4483.591	1616.586	8205.962	52.818

TABLE 3: ELEMENTAL COMPOSITION OF CRUDE ORE AND BENEFICIATED WOLFRAMITE. (mg/kg)

Conc (PPM)	W	Zr	Ca	Au	K	Se	As	Fe	Mn	Sn	Bal	Nb	Bi	Ti	Sc
Crude Ore	139251.656	15.478	2531.692	1898.292	655.461	691.074	1790.972	64296.402	4547.259	143.334	771967.250	11.581	-	270.756	17.278
Concentrate from Air Float	501623.375	289.901	3373.630	3162.333	8396.362	1288.644	296.075	102173.188	11867.805	25444.855	331496.813	5344. 401	1437.574	2306.052	36.880
Concentrate with Rapid Magnetic separator	625365.750	242.057	1436.219	3617.290	439.969	1863.118	305.467	114761.305	15041.225	4907.749	224539.625	6060. 299	523. 634	896.332	15.995
Tailing from Magnetic separator	1984.582	105.837	6518.307	-	1965.653	-	-	593290.250	1742.111	154.297	389713.438	14.738	-	1871.955	-
Middling from Magnetic separator	407016.375	477.270	918.126	3474.847	601.735	1388.719	788.245	123551.008	25359.176	25768,096	360230.094	6525. 742	701.390	39581.254	28.103

IV. DISCUSSION

3.2Petrography and Characterization of the Wolframite ore

The ore microscope is an indispensable tool in the determination of the mineral constituents of an ore, texture, intergrowth of ore minerals and gangue and the determination of grain size. These parameters are important to the mineral processing engineer in selecting process equipment, method and reagents for processing of ores and minerals (Rudolf et al.,2014)**.**

Wolframite ore was characterized prior to beneficiation using ore microscopy, to identify the constituent minerals, textures and grain size analysis, to determine the optimum size of liberation. Polished sections of rock were studied in the mineral processing department of National Metallurgical Development Centre. The results obtained are presented below.

Wolframite polished Section

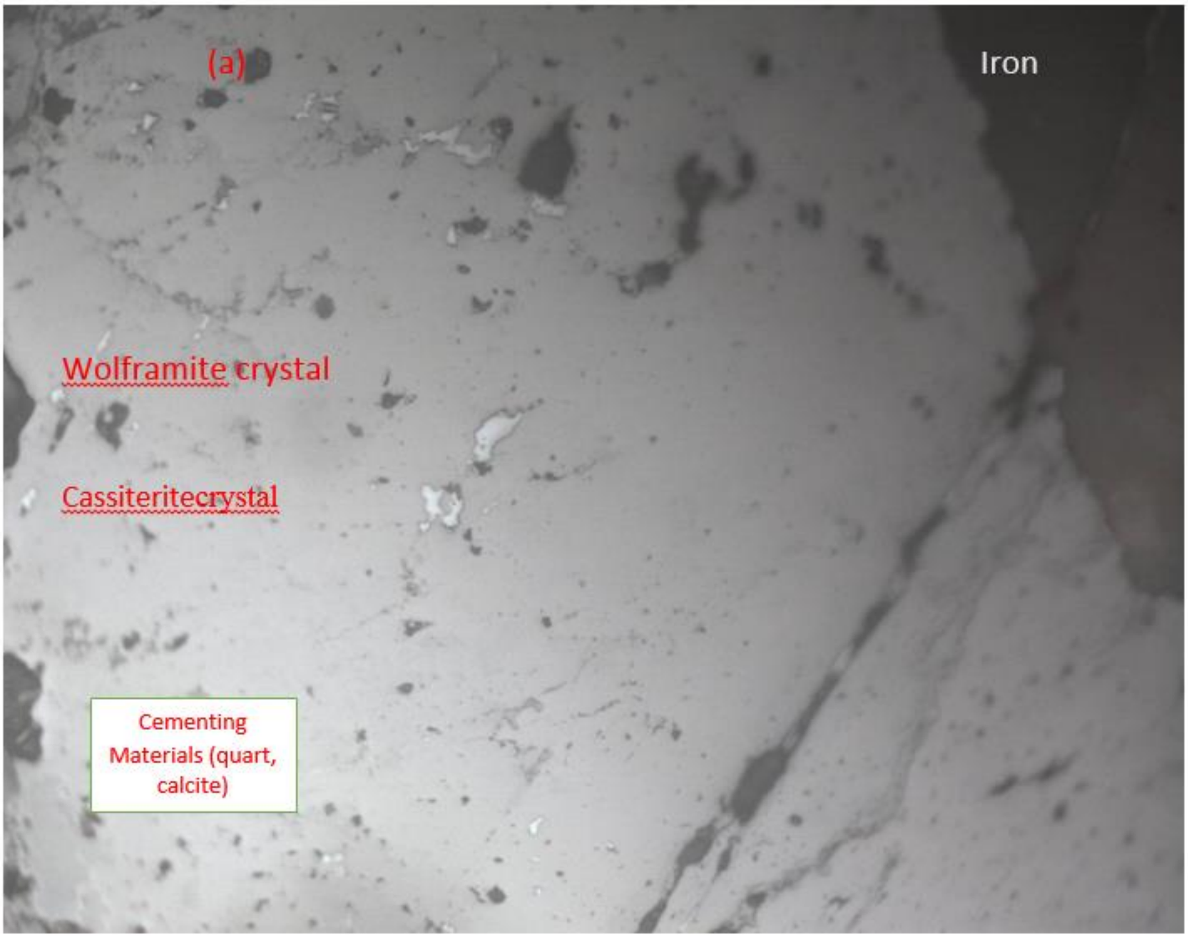


Plate a:Microscopic Image of Wolframite Ore

From plate (a) the sample under microscope. It shows Wolframite crystal, cassiterite crystal, iron crystal and the cementing material which include quartz and calcite. From the image, it was observed that wolframite crystal is in a heterogenous state because of the inclusion of other mineral crystals within it. The dark spots within the wolframite crystal are iron and some crystals of the cementing materials which are quartz and calcite. This shows that the wolframite must be dressed with the proper mineral processing separation method in order to recover the pure form of the wolframite ore.

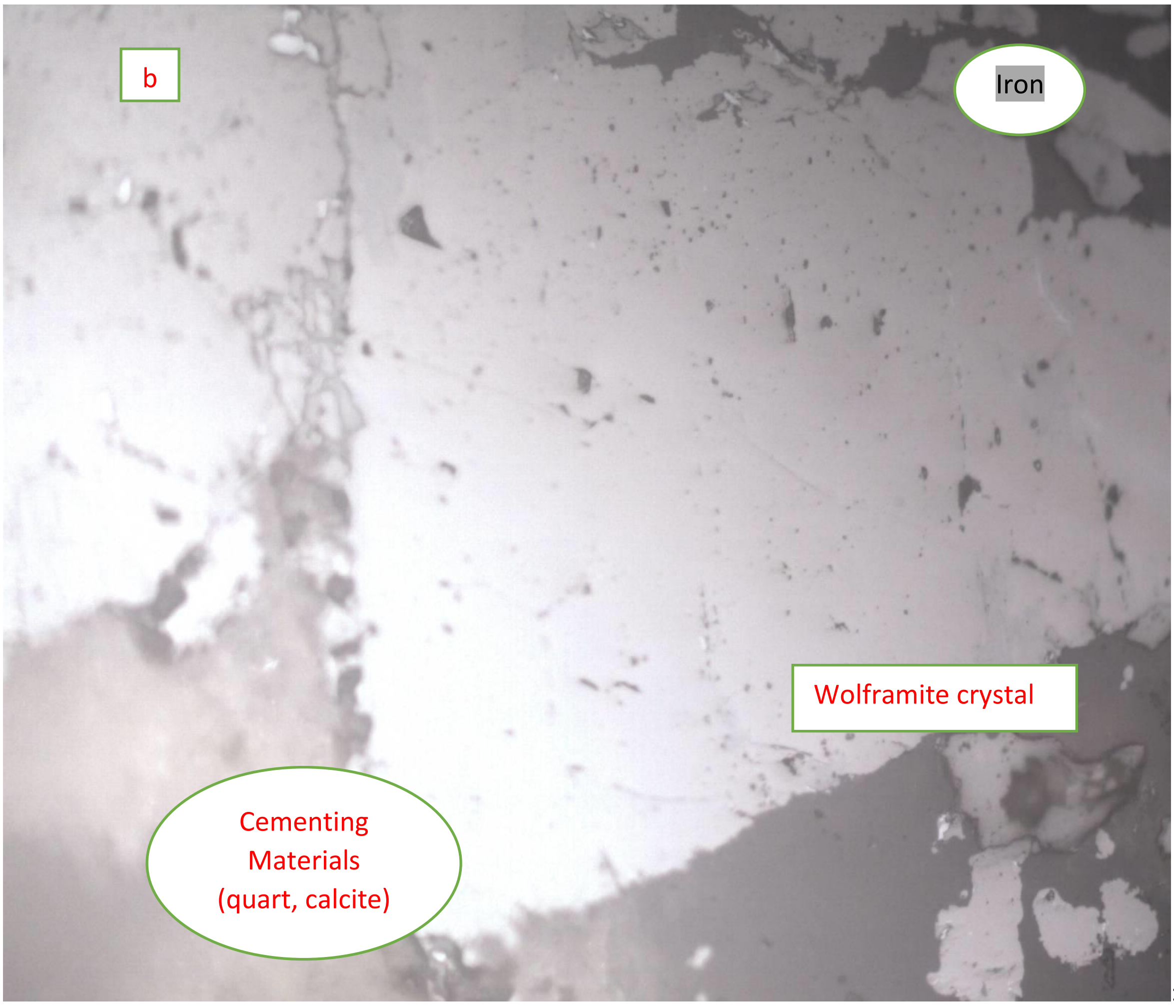


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Plate b: Microscopic Image of Wolframite Ore

Plate(b) shows that the ore contain grain sizes that are small to medium in size and this will help to guide in deciding the separation method that will be suitable for separating the concentrate from the gangue. It was also observed that the wolframite ore present have significant economic volume which will result in high recovery rate during ore beneficiation.

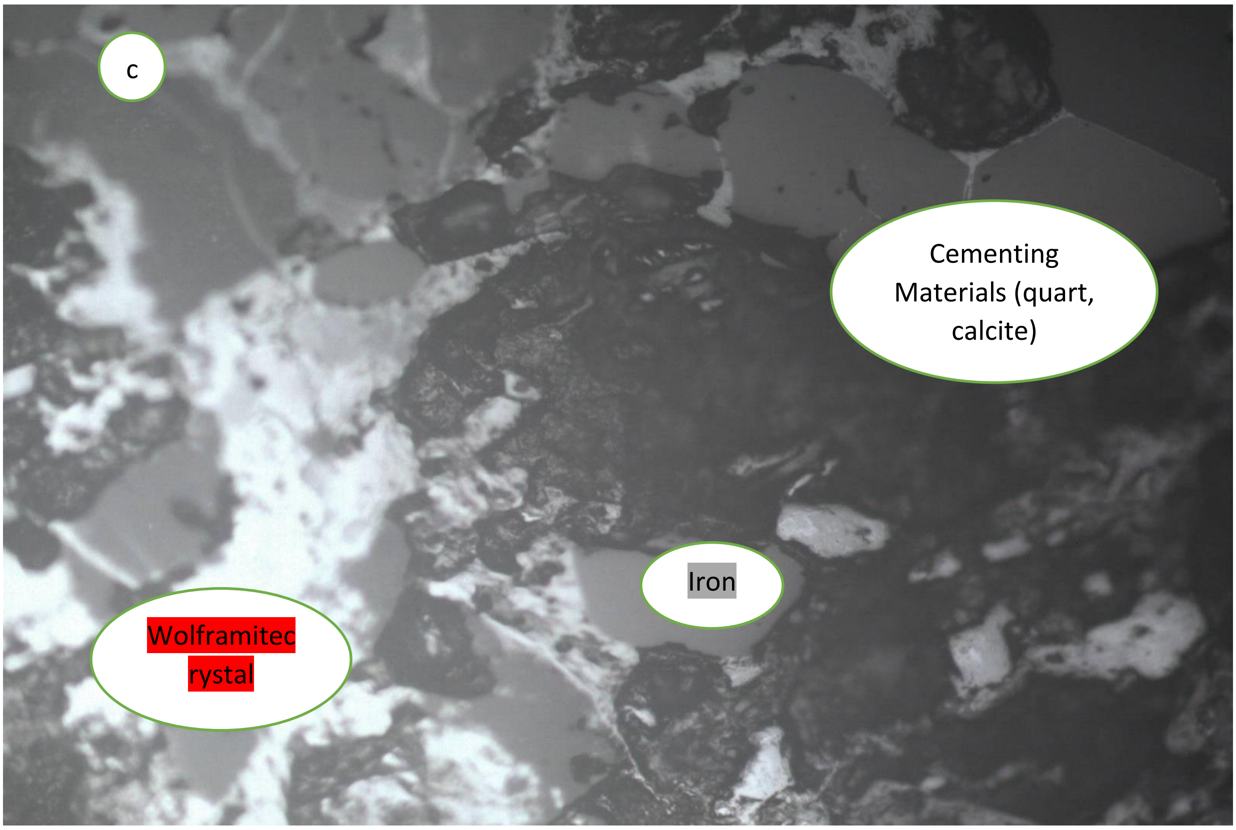


plate c: Microscopic Image of Wolframite Ore

Plate c: shows the complex matrix of the various mineral crystal constituent of the ore. This shows that the separation must be done carefully to avoid contamination during the separation process.

3.2.1 Determination of Liberation Size.

From the result obtained from the sieve analysis (Table 1) the amount of sample retained in each sieve indicated that 1.4mm sieve size retained 91.5g, 1mm sieve size retained 19.33g, 0.71mm sieve size retained 27.06g, 0.5mm sieve size retained 34.98g, 0.355mm sieve retained 40.1g, 0.25mm sieve retained 59.03g, 0.18mm sieve retained 65.89g, 0.125mm sieve retained 55.15g, 0.09mm sieve retained 40.46g, 0.063mm sieve retained 27.68g, -0.063mm sieve retained 27.13g. The total weight retained was 498.31g; which shows minimal loss in sample weight of 500g measured for the analysis which signifies that the analysis was carried out in strict adherence to professional standard.

The liberation size can be derived from the liberation size graph in Figure 15. The liberation size graph is a plot of cumulative weight percentage against the sieve size.The cumulative weight percentage is the cumulative weight retained (red coloured line) and the cumulative weight passing (blue coloured line). The liberation size of the wolframite is the point of intercession of the cumulative weight retained and the cumulative weight passing on the graph. From the point of intercession in the graph, the liberation size is 0.25mm.This information will help to guide researchers that are intending to carry out further investigations on Rafin Gabas wolframite ore.

3.2.2Determine Elemental Chemical Composition of Sample in each Sieve Size through Chemical Analysis using XRF

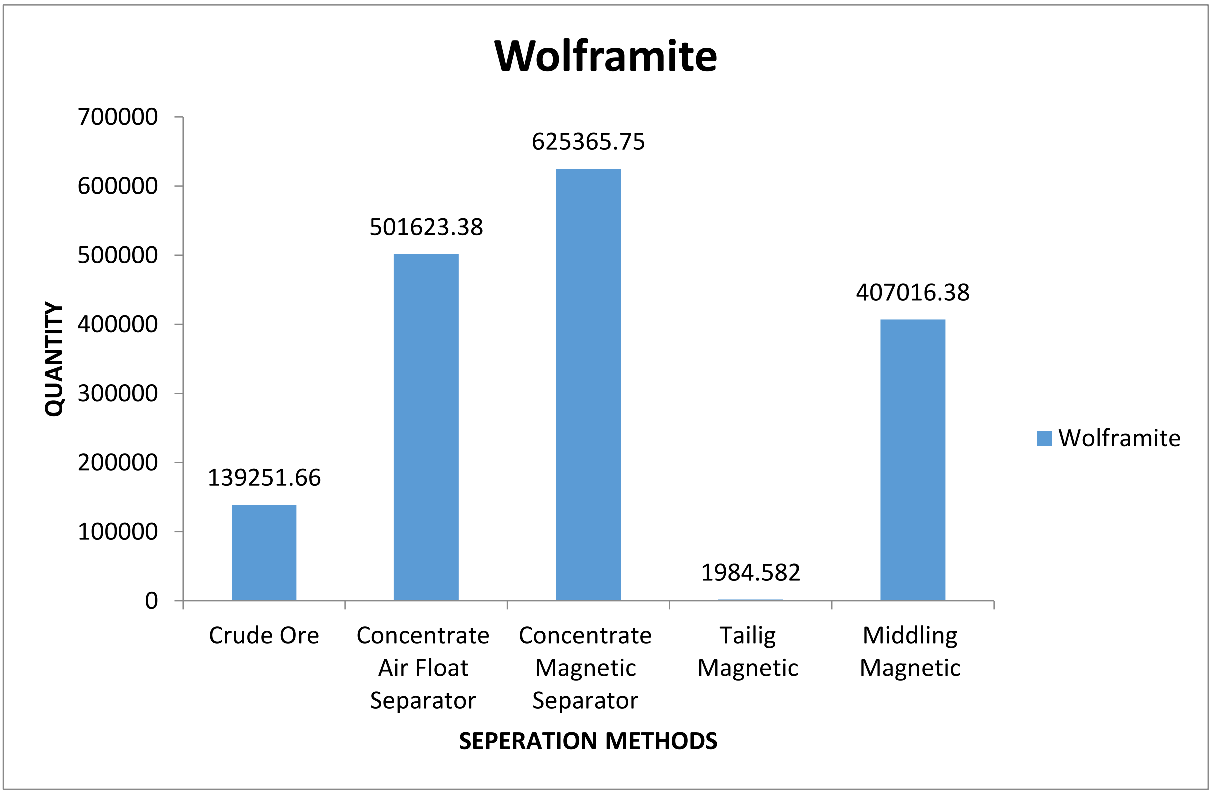
Table 2 shows the chemical composition of the various sieve sizes of the Rafin Gabaswolframite ore as revealed by XRF. The result showed that the wolframite ore contains W, Si, Ca, Au, K, Se, As, Fe, Mn, Sn, Bal, Nb, Bi, Ti, and Sc. The negative (-) sign in Table 2 is the underflow. while the positive sign (+) is the overflow. Both the valuable mineral W and the major gangue mineral Si were averagely distributed in all sieve sizes. This phenomenon indicates that W and Si were normally distributed and the weight of the minerals varies in the sieves, but still within a normal range to each other except for +0.125mm, which have the lowest weight for W and the lowest weight of Si is in sieve size +1.4mm because there was no liberation; while the highest weight for W is observed in sieve size +0.355mm and the highest weight for Si is in sieve size +0.09mm. Since the major gangue which is Si is lowest in +0.250mm with a very high amount of W. Based on this, the liberation size of the major gangue mineral, Si is +0.250mm, since it has the lowest weight of Si and very high weight of W at +0.250mm. This trend compares favorably with works done by Weiss (1985), where they also observed similar trends.

3.2.3 Determining the most Suitable Beneficiation Method for Separation of Rafin-Gabas Wolframite through Chemical Analysis using XRF.

From Table 3, it is observed that the weight of Wolframite(W) in the crude is 139251.656 and the gangue with the highest weight is Fe having 64296.402. The major aim of beneficiation of the crude ore of wolframite is to increase the quality of the wolframite ore for industrial application. Two major beneficiation methods were employed in this research – they are Air floatation method and Magnetic separation method. The beneficiation was done in the beneficiation pilot plant of National Metallurgical Development Centre, Jos with strict adherence to standard processing procedures which makes the result obtained from the research very reliable.

It can be observed from 3that the air floatation process upgraded the crude wolframite ore from 139251.656 to 501623.375 unit which shows a significant upgrade in the concentration of the wolframite ore (W) but in comparison to the other method used in this work to beneficiate the Wolframite Ore which is magnetic separation method. It is observed from Table 3 that the magnetic separation method upgraded the crude wolframite ore from 139251.656 to 625365.750 unit which is significantly higher than the upgrade value obtained from the use of air floatation method. It can also be observed from the result obtained from the further analysis of the tailing and middling that some upgraded wolframite ore can still be recovered mostly from the middling.

It is observed from the results in the Table 3 above that the amount of gangue (Fe) is very high when compared to the Wolframite concentrate recovered from the tailing - this shows that the liberation of wolframite from the gangue is very successful. Since the concentrate of wolframite recovered with magnetic separation method is higher than that from the air floatation method, it can be stated that magnetic separation method is better used for the beneficiation of wolframite ore for maximum recovery of the ore.



: Graph of Wolframite

V. CONCLUSION

The occurrence of wolframite ore in Rafin-Gabas, Nassarawa state of Nigeria as confirmed by this research is a blessing to this nation. This work as been able to established that the liberationsize of the wolframite ore is +0.250mm and the beneficiation method that suites the concentration of Rafin-Gabas wolframite is magnetic method.

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