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Characterization of Graphite Mineral of Ningi in North-Eastern Nigeria

B. Osasona [a*], O. O. Alabi [a] and F. O. Aramide [a]

[a] *Metallurgical and Materials Engineering Department, School of Engineering and Engineering Technology, Federal University of Technology, P.M.B. 704, Akure Ondo State, Nigeria.*

Authors' contributions

This work is a collaborative effort of all authors. Authors BO and OOA designed the study, performed the statistical analyses and wrote the protocol. Author BO managed the Literature searches and wrote the first draft of the manuscript. Authors OOA and FOA managed the analyses of the study. All authors read and approved the final manuscript.

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ABSTRACT

The future value of graphite due to its use in lithium-ion batteries, and the scientific possibilities of graphene has markedly increased both the demand and consequently the price of natural flake graphite amidst flat global supply in the last decade. Despite the boom, most graphite mineral deposits found in Nigeria remain unexploited due to lack of technical information thereby denying the country the opportunity to create wealth through mineral development. This study aims to characterize the graphite mineral deposit from Ningi, North-eastern Nigeria. Energy dispersive fluorescence (XRF), Bomb calorimetry, proximate analysis, X-ray diffraction (XRD), and Optical microscopy techniques were employed in the investigation. XRF results shows that Ningi graphite mineral contains 81.5 wt.% SiO_2. It also confirms the presence of ZnO, MnO, and Ni up to 0.055 wt. %. Proximate compositional analysis indicated that Ningi graphite is a low-grade deposit as it analyzed 0.4 wt.% C and 88.51 wt.% ash. XRD studies indicate that the graphite mineral consists of quartz, mica, and graphite as major mineral phase and the graphite flake size as reported by optical microscopy ranges widely between 10 µm – 350 µm. Even as the probable reserve of Ningi

**Corresponding author: Email: tolaosasona@gmail.com;*

graphite remain un-estimated, this study shows that it is technically feasible to exploit Ningi graphite towards concentration. However, further structural and beneficiation studies are necessary to transform Ningi graphite resource into economic opportunities.

Keywords: Graphite mineral; mineral economics; mineralogical properties; sustainable beneficiation.

1. INTRODUCTION

The global population is expected to grow to around 8.5 billion in 2030, 9.7 billion in 2050, and 10.9 billion in 2100. This presents challenges for sustainable development putting pressure on existing resources and challenging policies aimed at improving the welfare of global citizens [1,2]. To offset some of the negative effect of population on the economy and environment, there is need to derive the greatest value from non-renewable mineral assets. Thus, a proper mineral evaluation is necessary [3,4].

Graphite are the most common polymorphs of crystalline carbon, they are end-member products of the continuous transformation of organic matter (coalification). They, therefore, represent a high metamorphic grade [5]. Natural graphite resources are classified based on a multitude of character, including not only grade (fixed carbon content) but also flake size, as well as crystallinity. The excellent and unique properties of graphite make them a critical resource used in 4th industrial revolution [6,7]. On the basis of the above, the demand for graphite has been projected to increase markedly by over 1000% in the next decade due to the diverse use in new and green technologies-especially as the anode of lithium-ion batteries that powers electric cars, and for the scientific possibilities of graphene [7,8]. However, the supply risk is high since only in few applications can graphite be recycled [9]. Thus, the need to find unexploited reserves of natural graphite as a means to help meet the rapidly growing global demand.

In Nigeria, graphite deposit exists within the migmatite-gneiss complex (MGC) of the north-central and north-eastern part of the basement complex of older granites. Such complex consists mainly of pegmatites, gneisses and schists that have been intruded by basic and ultrabasic rocks [10,11]. However, the probable reserve of this mineral block (northern) has not been estimated and few characterization studies has been documented for the explored occurrences [11,12]. To this end, this paper presents a summary of the chemical and mineralogical characterization study on Ningi graphite as a means to assess the technical viability of the graphite deposit for beneficiation.

2. EXPERIMENTAL DETAILS

2.1 Sample Collection

Graphite mineral samples were obtained in lump form from Ningi Graphite deposit *(approximately 11º08'N, and 9º41'E)* west bank of Dilimi (Bonga) river in Ningi town, Bauchi state, North-Eastern Nigeria (see Fig. 1). All samples were broken manually using a geological hammer, and mixed together to represent the properties of the mineral which can affect the metallurgical process. The samples underwent dry grounding in stages using laboratory size Fritsch pulverisette (FAM8034 - AK33) and planetary ball mill. They were dry-sieved to 100% passing of 180 µm using vertically vibrated automatic sieve shaker and stored in desiccator. For chemical and mineralogical analysis, a representative sample was drawn through Jones Rifle sampler.

2.2 Procedure

2.2.1 Chemical analysis

The heating value, compositional, and chemical analysis of Ningi graphite was obtained by Bomb calorimetry, proximate analysis using ASTM D3172-13 testing specification [13], and Energy Dispersive X-ray Fluorescence (ED-XRF, SHIMADZU EDX-800 spectrometer) respectively. For ED-XRF spectroscopy, Ningi graphite sample was milled to -75 µm, pressed into powder briquettes for proper mineral count and placed on the sample cell for a total of one hour thirty minutes (thirty minutes for major elements, and one hour for trace elements. However, for trace elements analyses, sample was mixed with polyvinyl alcohol (PVA) binder. The result generated was analysed using the Mini Pal-4, WinXRF software for all elements in the periodic table between sodium (Na), and Uranium (U). The detection limit of this instrument is 1 to 10^4 ppm and only element found above the detection limits was reported. All major elements were expressed as oxides and analysed in percentage (%) composition.

Fig. 1. Scalar Position of Ningi graphite deposit with respect to the Map of Africa

2.2.2 Mineral phase analysis

The phase analysis of Ningi graphite was done by X-ray diffractometer (SHIMADZU XDS-2400H, Cu K^{α}). Pulverized Ningi graphite was prepared and mounted on the sample stage in the diffractometer cabinet. The diffractometer was set to a tube current, voltage rating, and goniometer radius of 40mA, 45VA and 240mm respectively. The Theta-Theta scanning angle was ranged from 0 to 150° with a two-theta step of 0.026 at 13.7700 seconds per step. The intensity of the diffracted X-rays is automatically and continuously recorded on a chart and d (111) spacing (Ǻ) was determined (see Table 3). The background and peak-positions were identified and based on the peak positions and intensities; a search-match routine was performed.

2.2.3 Petrography

The mineralogical study was carried out using optical microscope (LEICA DMEX). This involve the polishing of Ningi graphite rocks of size range 4-6 mm with different emery papers to expose the smooth surface of the graphite rock. The polished rocks were mounted on glass slide and ground to 0.03 mm. The samples were mounted on the microscope with reflected and transmitted light at different magnifications to reveal the distribution of the various elements present in the mineral and their approximate particle size. Few of them, were selected for colour micrographs.

3. RESULTS AND DISCUSSIONS

3.1 Chemical and Mineralogical Analysis

The study covers the elemental and proximate compositional analysis, phase analysis, mineralogical association, microstructure, and grain size of Ningi graphite towards an efficient beneficiation.

3.1.1 Chemical analysis

Chemical analysis of raw sample was done using ED-XRFS and reported in Table 1. The data indicates that the major inorganic constituents of Ningi graphite mineral are SiO_2, K_2O, Fe_2O_3, RuO_2, and trace elements like Ni, MnO, and ZnO were also found present in the mineral deposit.

Table 1. Chemical composition of Crude Ningi graphite mineral (wt. %)

Radical	**SiO_2**	**K_2O**	**Fe_2O_3**	**RuO_2**	**V_2O_5**	**SO_3**	**BaO**	**Cr_2O_3**	
Amount (wt. %)	81.5	8.47	6.92	0.58	0.32	0.2	0.16	0.15	
Radical	**PbO**	**MnO**	**Ni**	**CuO**	**ZnO**	**Rb_2O**	**SrO**	**ZrO_2**	**Total Oxide**
Amount (wt.%)	0.052	0.055	0.022	0.091	0.018	0.042	0.022	0.096	98.698

Table 2. Proximate analysis of Crude Ningi Graphite

Graphite (r.o.m)	Moisture	Volatile Matter	Ash	Fixed Carbon	Calorific Value, (MJ/Kg)
Wt. %	2.77	8.32	88.51	0.4	6.53

Table 3. The XRD Figure of Merit for Ningi Graphite

Peak	2θ/degree	Plane	Intensity	d-Valve (A˚)	Mineral Phase
1	38.21	100	50.30	2.3533	Knebelite
2	44.86	002	69.30	1.0187	Umohoite
3	55.91	004	308.60	1.6430	Umohoite
4	65.31	105	75.60	1.4274	Irarsite
5	67.21	006	94.50	1.3917	Umohoite
6	84.23	103	61.20	1.1486	Pseudobrookite
7	87.80	008	81.70	1.1108	Pegmatite
8	88.51	112	48.10	1.0979	Tritomite
9	102.40	102	50.30	0.9707	Graphite oxide
10	119.21	110	42.80	0.8930	Tritomite
11	127.41	003	48.10	0.8591	Irarsite
12	128.30	104	42.80	0.8558	Knebelite
13	133.41	218	29.60	0.8386	Pseudobrookite

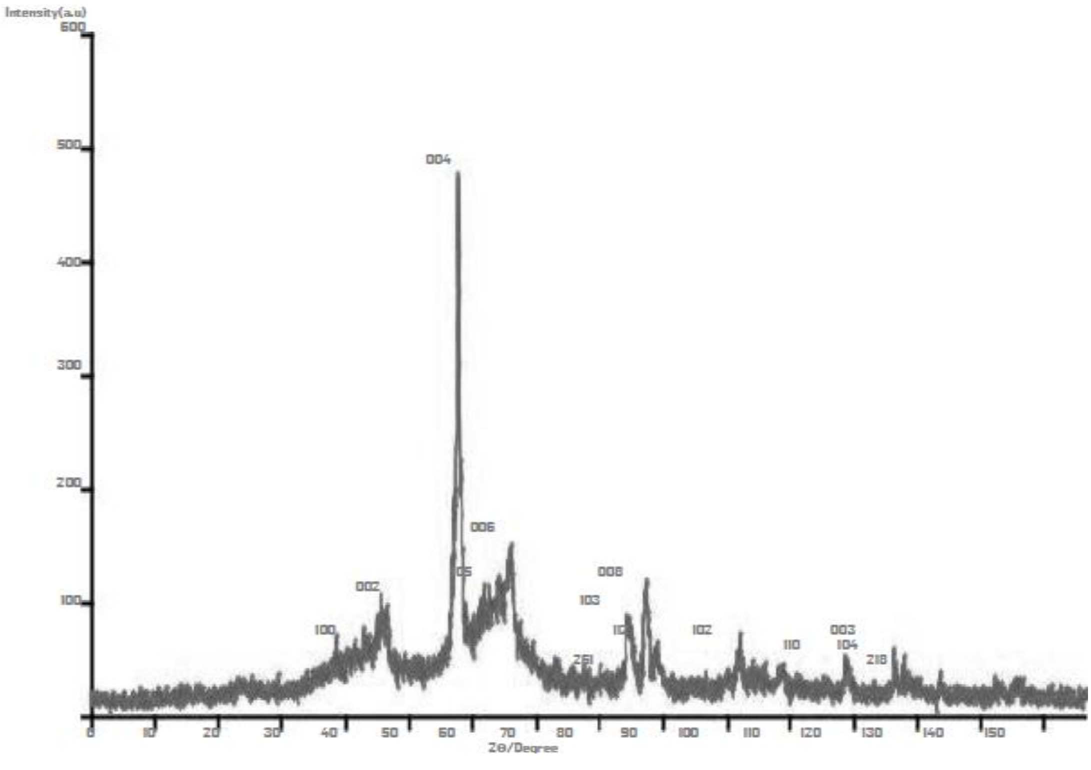

Fig. 2. The open circles and the solid line X-ray diffraction patterns of Ningi Graphite

Table 2 presents the average heating/calorific value and the proximate analysis test carried out on twenty different Ningi graphite samples. It is shown that the raw sample contains high weight percentage composition of ash (88.51%) and low graphitic carbon content (0.4%). Therefore, the chemical analysis summarily reveals that Ningi graphite is a low-grade graphite with high silica content.

3.1.2 Phase analysis

Phase analysis of Ningi graphite powder is given in Fig. 2. The predominant phases present in

Ningi graphite mineral as indicated by XRD data presented in Table 3 are Graphite Oxide, Tritomite, pegmatite, Knebelite and Pseudobrookite. This suggests that the sample ore contain Quartz-sillica, Feldspar, and Mica, as a dominant inclusion while Elements like manganese, molybdenum, potassium, iron, iridium, and titanium are suggested to be present.

3.1.3 Petrography

The phase distribution, microstructure, and graphite flake size within the matrix of Ningi graphite mineral were investigated and analysed using optical microscopy and thin-section modal mineral analysis. Fig. 3 shows one of the few micrographs (taken at a magnification of 100) of the graphite mineral. The modal analysis of the optical micrographs reveals that Ningi graphite rock contains primarily of opaque graphite constituents (15.9% modal composition of the specimen) of different morphology dispersed in greyish quartz matrix (73%% modal composition), with minor quantity of yellowish biotite. The graphite mineralization and size distribution as observed from Fig. 3 shows a fractured, finely distributed, mostly angular, and bladed graphite crystals. The flake size of graphite in Ningi graphite mineral is found to range between 0.01 mm to 0.35mm (fine to large flake size). Furthermore, from Fig. 3, it is observed that the constituent minerals of the Ningi graphite sample were separated by semi-coarse grains boundaries, with weak interlocking of minerals.

3.2 Discussion

This research addresses a single question: Does the chemical and mineralogical character of Ningi graphite deposit reveals a possibility for beneficiation? To answer the above, two dimensions were considered which are the technical feasibility of exploitation and the prevailing economic reality.

From a technical point, we found that the chemical and mineralogical character of Ningi graphite compares favourably with previous research on other low-grade graphite deposits of Northern Nigeria. In a beneficiation study carried out Nwoke et al., 1997 on low-grade graphite of Birnin Gwari, and Alawa, the chemical analysis of the deposits showed high $Si0_2$ (66.7% and 67.8 %), high Fe_2O_3 +Fe_30_4, (8.2% and 6.7%) and high Al_2O_3 (15.2%and 13.6%) contents for the Birnin Gwari and Alawa samples, respectively and the mineralogical evaluation showed the presence of quartz, magnetite, aluminosilicates, feldspar, biotite, diopside and rutile-a characterization similar to Ningi graphite (see Table 1 and Table 3). Nevertheless, both graphite deposits of Birnin Gwari and Alawa were successfully concentrated from 5.2% and 3% Cg to grades of 90.2%and 88.0% Cg, respectively [11].

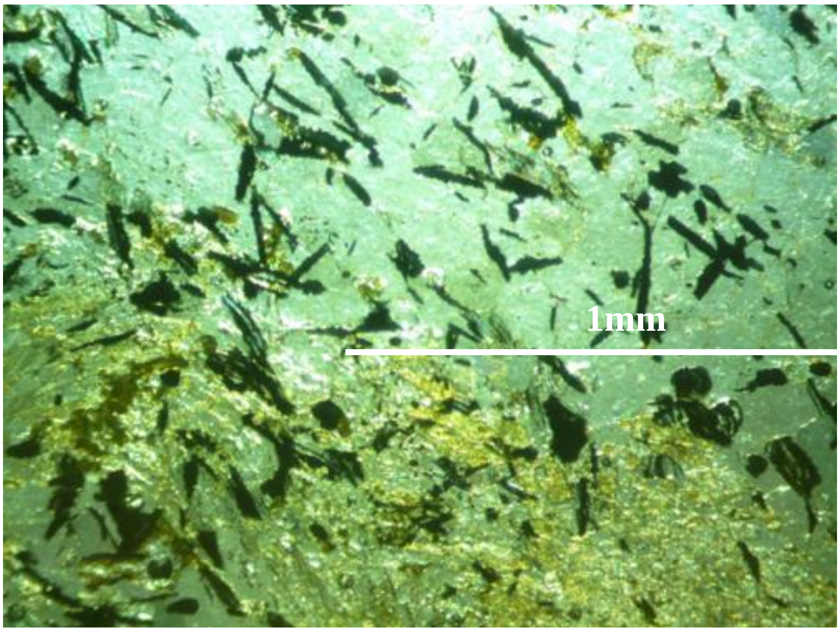

Fig. 3. Photo-Micrograph of Ningi graphite mineral showing graphite flakes in silica rich matrix

Furthermore, the proximate analysis of Ningi graphite reveals that its ash content (88.51 wt.%) compares favorably with the low-grade graphite of eastern India (89.89% wt.% of ash content), yet Vasumathi et al., 2014 were able to beneficiate such low-grade, high ash content graphite of eastern India to a final concentrate of 8.97% weight recovery with 5.80% ash and 92.13% fixed carbon [6]. Finally, it is observed that the constituent minerals of the Ningi graphite sample are separated by semi-coarse grains boundaries, with weak interlocking of minerals (Fig. 3). The implication of which is that the constituent minerals of Ningi graphite deposit can be easily liberated via comminution and as such, the separation of graphite mineral from the high-silica quartz matrix is technically feasible [14]. Finally, the feasibility of extracting Ningi graphite deposit is further asserted by the prevailing global economy as a rising commodity price and a declining supply trend has over the ages permitted the extraction of several low-grade deposit [2, 15].

4. CONCLUSION

- ED-XRFS of Ningi graphite mineral confirms 81.5 wt.% of SiO_2 and 8.47 wt.% of K_2O. It also confirms the presence of Ni, MnO and ZnO up to 0.055 wt.%
- Proximate compositional analysis revealed that Ningi graphite deposit is a low-grade deposit as evident in 0.4 wt.% of Fixed carbon(graphitic) content.
- XRD Studies revealed graphite Oxide, tritomite, pegmatite, knebelite and pseudobrookite as significant mineral phase present in the deposit.
- Microscopic studies show that the flake size of Ningi graphite varies widely between 10 µm– 350 µm and are finely distributed in quartz-silicate matrix.

As a whole, we conclude that Ningi graphite deposit is of a low-grade graphite mineral with wide range of flake size, and weak interlocking of graphite in high silicate matrix. Thus, the liberation of graphite from the Ningi graphite mineral rock is technically feasible by comminution towards concentration [6,14]. In addition, the prevailing global economic conditions of a rising commodity price and a declining supply trend of graphite commodity makes the extraction of a low-grade Ningi deposit more viable [2]. Future work will entail a structural characterization (Raman microspectroscopy) and beneficiation studies of Ningi graphite as well as the evaluation of the probable reserve estimate to determine that the low-grade Ningi graphite will be able to balance the costs associated with mining and processing [3].

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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