Original Research Article

Characterization of Graphite Mineral of Ningi in North-Eastern Nigeria

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. Wavelength dispersive fluorescence (XRF), Bomb calorimetry, 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 Zn, Mn, and Ni up to 0.055 wt. %. Bomb calorimetry 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 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 – 350 µm.

Keywords*: Graphite mineral, Mineral economics, Mineralogical properties, Sustainable beneficiation.*

1 INTRODUCTION

Graphite is a lustrous black mineral, relatively soft, greasy with a hardness of 0.5 - 1.0 on Moh's scale. It is the most common natural polymorphs of crystalline carbon – another one being diamond. Natural graphite resources are classified based on a multitude of properties, including not only grade (fixed

carbon content) but also crystallinity as well as grain (flake) size and shape [1, 2]. Flake graphite are commercially classified into coarse (+150– 850 µm in diameter) and fine flakes (+45–150 µm in diameter) [3].

Graphite are principally formed by graphitization and, essentially, they are end-member products of the continuous transformation of organic matter (coalification). They, therefore, represent a high metamorphic grade [4]. Associated gangue minerals with graphite occur as quartz, rock-forming silicates such as amphiboles and feldspars, also calcite, mica, sulfides, magnetite and carbonates. These gangue minerals are finely disseminated [3, 6]. The most problematic graphite deposit is of calcareous origin because the calcite often redeposits even in the flakes of graphite [7].

Graphite assumes different morphological forms and its structure consists of six-membered rings in which each carbon atom has three near neighbors at the apices of an equilateral triangle. The layers in graphite crystal are held together by van der Waals bonding forces of energy of 0.2 eV/atom [4]. Graphite has both metallic and nonmetallic properties, some of its properties are refractoriness, high-heat and electrical conductivity, negative temperature coefficient of resistivity, inertness, and high solubility in iron [2, 4].

These unique properties give rise to the major uses of graphite in welding rods, desulfurizing agents, facings, lubrication, refractories, marking instruments, electrical products, batteries, brake linings, bearings, conductive coatings, crucibles, electrodes, and paints [2, 5]. The interesting thing about graphite is its futuristic value in Lithium-Ion Batteries used in smart devices and electric cars, and the scientific possibilities of graphene, and as such, they are currently being subjected to intensive exploration to help meet rapidly growing global demand [8, 9, 10].

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 [11, 12]. This makes the exploitation and beneficiation of graphite resources feasible as a way to support the import-dependent, and undiversified mono-economy [10, 13]. However, the probable reserve of this mineral block (northern) has not been estimated and no characterization study has been documented for Ningi graphite. To this end, this paper presents a summary of the characterization study on Ningi graphite as a means to assess the technical and economic viability of the deposit.

2 EXPERIMENTAL DETAILS

2.1 Sample Collection

Graphite mineral samples were obtained in lump form from Ningi Graphite deposit *(approximately 11°08'N, and 9°41'E)* west bank of Dilimi (Bonga) river in Ningi town, Bauchi state, North-Eastern Nigeria (see Figure 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.

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

2.2 Procedure

2.2.1 Chemical analysis

The chemical composition of Ningi graphite was obtained by proximate analysis (Bomb calorimeter) using ASTM D3172-13 testing specification [14], and Energy Dispersive X-ray Fluorescence (ED-XRF, SHIMADZU EDX-800 spectrometer). 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.

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 (Á) 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 DISCUSSION

3.1 Chemical and Mineralogical analysis

The study covers both ultimate and proximate 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, Mg, and Zn 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 Presents the average value for 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.

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

3.1.2 Phase analysis

Phase analysis of Ningi graphite powder is given in Figure 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. This finding compared favourably with previous research on natural graphite [1,15].

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

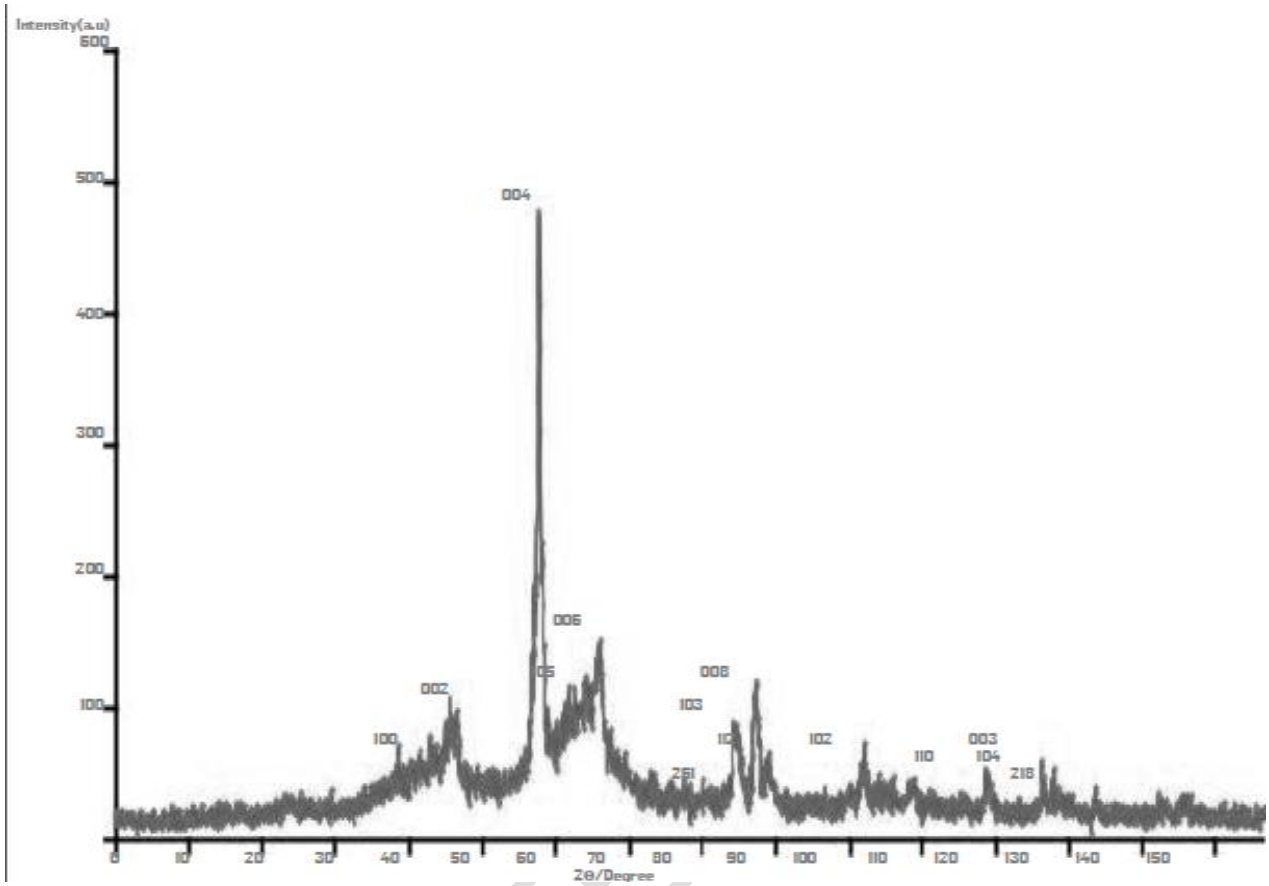

Figure 2 The Open Circles and the Solid line X-ray Diffraction patterns of Ningi Graphite

3.1.3 Petrography

The phase distribution and microstructure within the matrix were investigated using optical microscopy and thin-section modal mineral analysis. Figure 3 shows the micrograph (taken at a magnification of 100) of the graphite mineral and 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 is also observed as fractured, finely distributed, mostly angular, and bladed, with a flake size ranging from fine to large flake (approximately between 0.01-0.35 mm). Furthermore, it is observed that the constituent minerals of the Ningi graphite sample are separated by semi-coarse grains boundaries, with weak interlocking of minerals. The implication of which is that the constituent minerals of the sample can be easily liberated via comminution and as such, the separation of mineral of interest (graphite) from the gangue should be technically feasible.

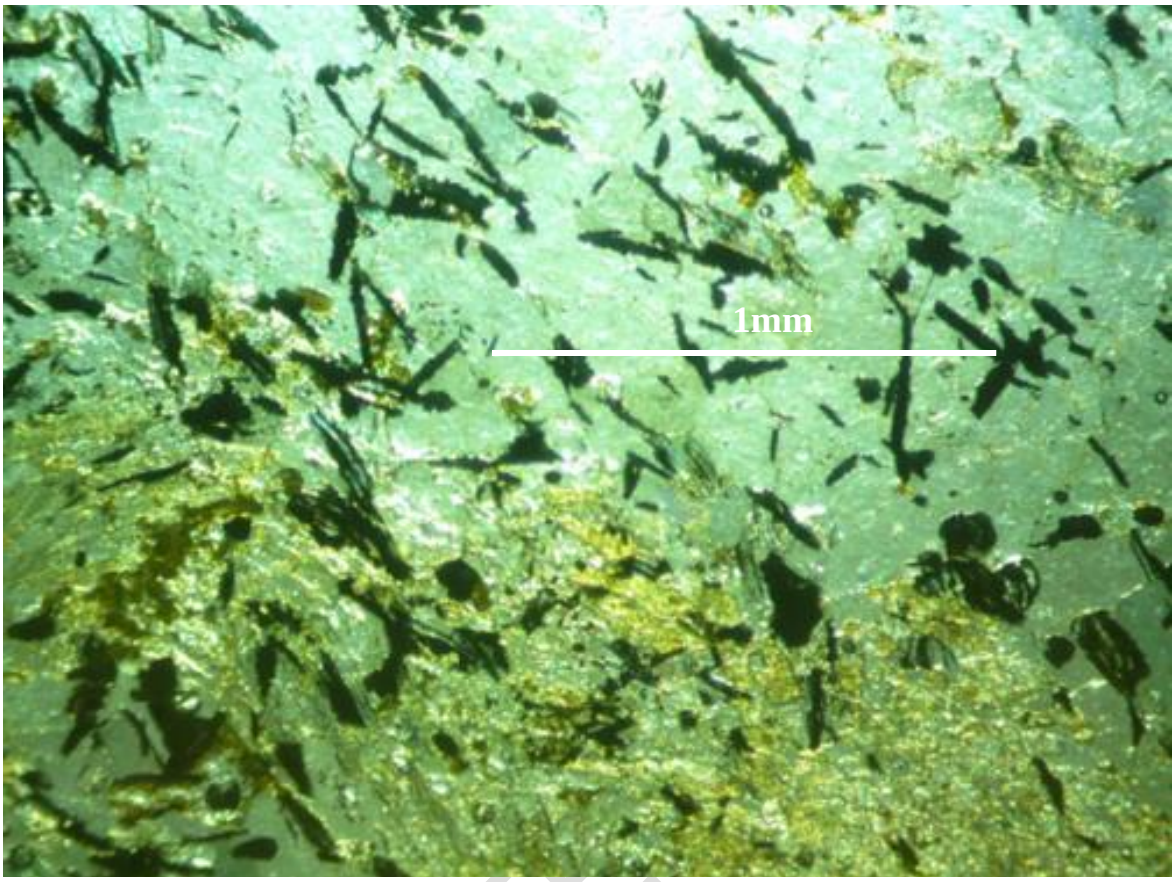

Figure 3 Photo-Micrograph of Ningi graphite mineral showing graphite flakes in silica rich matrix

4. CONCLUSION

- EDXRF confirms 81.5 wt.% of SiO_2 and 8.47 wt.% of K_2O. It also confirms the presence of Ni, Mg and Zn up to 0.055 wt.%
- Bomb calorimetry 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 – 350 µm and are finely distributed in quartz-silicate matrix.

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