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Characterization of Graphite from Sama-Borkono Area of Bauchi State-Nigeria

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***Abstract*— Nigeria is blessed with so many minerals but there is little or no attention paid in explorations of these minerals. The only resources that attract the attention of Nigerians and Nigerian government is crude oil which is depleting fast and its exploration has advent effect on the environment. Many States that are none oil producing suffered from low income despite the abundant minerals deposit in their states. Bauchi State is one of these states that considered to be poor but is blessed with more than ten solid minerals among them graphite. In this research raw graphite was collected from *Sama-Borkono* (SB) in Warji Local Government Area, Bauchi State, Nigeria. It was beneficiated, using froth floatation method, and characterized and found to be of high grade and purity. The carbon contents were found to be 46.58 and 95.42.wt% before and after beneficiation respectively. This purity is acceptable for many end uses such production of lubricants, fuel cell bipolar plates, refractories atc. TGA and SEM were used to investigate the thermal stability and micro structure of the mineral. The impurities removed from the graphite were characterized and found to have high percentage silica (81.2% SiO_2) which is very useful in the production of Portland cement. Both the pure graphite and its byproducts are very useful hence the graphite can be very viable for exploration and commercialization.**

***Keywords*— Graphite, Sama-Borkono, Warji, Bauchi State, Beneficiation, Froth floatation, Tailings, Fixed carbon content.**

I. INTRODUCTION

Sama-Borkono is a village in Warji Local Government Area in Bauchi State Nigeria. Bauchi state is one of the six states in Nigerian north-eastern region. The state is on Latitude $10^0 30$'N and longitude $10^0 00$' E and has twenty local government areas. Being one of the north eastern states, the state is considered to be one of the poor states in Nigeria, however, it is blessed with so many minerals.

These minerals include; kaolin, Graphite, tantalite, sesame, gum Arabic, gem stones, tin, columbite. Others are Lead, Zinc, Silver, Molybdenum, Uranium and many that are yet to be discovered [2]. The minerals are mostly unexploited and were only mined by the locals. Many researchers and miners pay little or no attention to graphite deposit in Ningi and warji local governments undermining the usage and potentials of that mineral.

Although graphite deposits could be found in many states in Nigeria, official information has not been gathered to ascertain their locations and or determine their commercial value

Graphite, a natural form of carbon, is characterized by its hexagonal crystalline structure as shown in Figure 1. It occurs naturally in metamorphic rocks as a result of the reduction of sedimentary carbon compounds during metamorphism such as marble, schist and gneiss. It also occurs in igneous rock and in meteorites. The same element crystallizing in an octahedral system becomes a diamond. It is a lustrous black carbon mineral, unctuous, and relatively soft with a hardness of 1-2 on the Mohr's scale [6]. It occurs naturally in the earth's crust and is the most abundant form of pure carbon .Graphite exhibits the properties of metal and non- metal which make it suitable for many industrial applications. The metallic property include thermal and electrical conductivity while non- metallic properties are inertness, high thermal resistance and lubricity and has a high melting temperature of 3,500 degrees Celsius[9]. Graphite, being one of the important non-ferrous minerals and having applications in various important industries, will find increased consumption in the coming years [2].

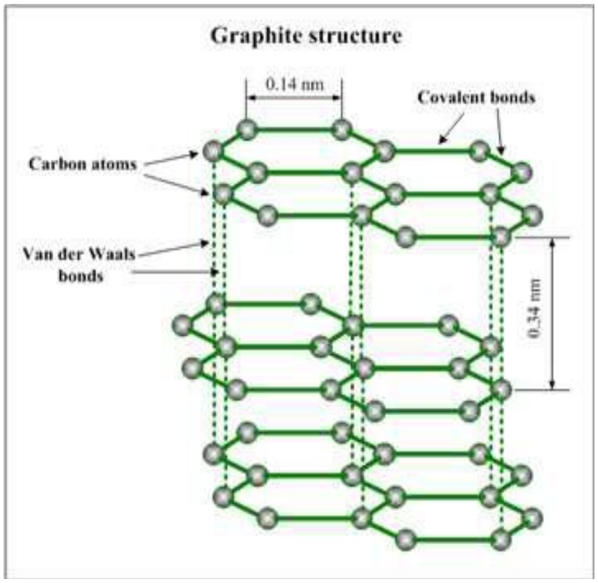

Figure 1: Graphite crystal structure [8]

The aim of this research is to investigate the characteristic of *Sama-Borkono* graphite and establish a report that shows it quality for onward research and exploration.

II. Methodology

In this paper, the potential and quality of *Sama-Borkono* graphite, in terms of purity, was verified. The raw graphite was collected from the mining site and characterized. Beneficiation using froth floatation was employed to achieve further purity. Detailed of the methods are as follows:

A. Pre beneficiation Characterization

The natural graphite collected from *Sama-Borkono* mining site was characterized to ascertain the purity of the mineral and its associated minerals and/or oxides that constituted the impurities.

A physical property such as surface morphology of the minerals was observed using field emission scanning electron microscope (Model: VPFESEM SUPRA 35VP). Element present and the weight or percentage composition of such elements were also determined using EDX elemental analysis. Graphite being an allotrope of carbon, it is expected to be pure carbon. Therefore, any initial percentage of carbon will be considered as the initial purity of the mineral. Thermal decomposition properties were also verified using TGA analysis with TGA machine (Model: METTLER TOLEDO TGA/DSC1).

Thermogravimetric Analysis (TGA) measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Measurements were used primarily to determine the composition of a material and to predict their thermal stability at temperatures up to 1000°C. The technique can characterize materials that exhibit weight loss or gain due to decomposition, oxidation, or dehydration. Figure 2 show the TGA result.

B. Beneficiation:

Graphite ore is usually found to be associated with many impurities that make its carbon content low, hence, it often requires beneficiation to obtain a desired grade for various end-uses. Processes for graphite beneficiation depend upon nature and association of gangue minerals present. In this research froth floatation is used to beneficiate the raw graphite. The stages of the beneficiation includes: Crushing; Milling; Sieving; Froth floatation; and Drying.

The raw graphite which is rocky was first crushed to a lower particle size using Jaw crusher and then grinded using ball milling machine to a size lower than 5mm. As the name implies, the ball milling machine has balls of different sizes contained in a cylinder. The sample was poured into the cylinder which is rotated by the machine. The rotation resulted into particle size reduction

A sieve of size 90µm and 50µm were used to sieve the sample to a fine powder. The sample is put into the sieve and mounted on an electric sieve shaker machine which vibrates for 20 minutes. The fine powder was collected at the bottom attachment.

The powder was then taken to the floatation machine for separation. For each run, 200g of the graphite powder is dissolved in 300ml of distil water. Pine oil and xanthate were added (both obtained from Philco chemicals Ltd, B/Kura Street, Bauchi), to stabilize the mixture and make it more aerophilic, for easy floating. Air is been sent through the manifold and the mixture is agitated to form a froth. Graphite been aerophilic (has high affinity to air) attached itself to the air bubble and float as froth. The pure concentrate was collected using skimmer to skim the froth to a container while the impurities (Tailings) that retained and remain wetted in the water were collected by decanting the water. Both the pure concentrate and the tailings were dried in an Oven and obtained in a powdered form.

III. Results And Discussion

The results obtained for pre and post beneficiation characterization are presented and discussed as follows:

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A. TGA Result

Following the procedure described by the manufacturer's manual, the Thermogravimetric analysis on the graphite sample was performed using 61.5mg weight of the sample in an air atmosphere charged at 0.01 l/min and at heating rate of 10^0C/min. At 59.1^0C temperature the first reaction occur and weight of about 1.0 mg at the rate of 0.3mg/min is lost which may be due to lost of moisture in the graphite. After the first point there is a steady and slower change in mass of 0.04mg/min up to temperature of 505.2^0C and no significant change until above 550^0C.This shows that graphite is thermally stable up to 550^0C. There was shaft decrease in mass between temperature 550^0C to 996.4^0C which is the range of combustion temperature and the carbon in the graphite is said to have reacted with the air to form CO and CO_2. This brought the total weight lost to be 10.67mg from the original 61.5mg and accounted for about 17.5% of the mass. The remaining mass of 50.34mg is observed at temperature 1000.1^0C. The residue is basically the impurities like the metal oxides (Fe_2O_3, Al_2O_3 SiO_2 etc) which sometimes serve as catalyst for the combustion reaction.

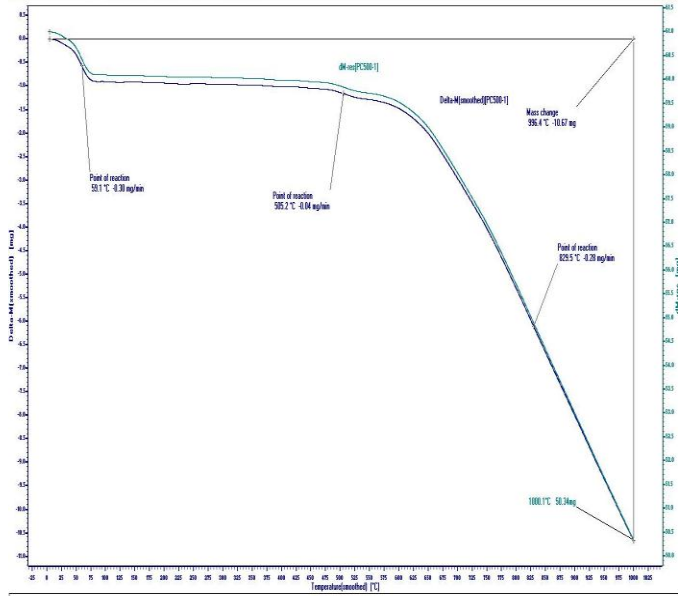

Figure 2: TGA result of raw graphite

B. SEM Result

The scanned image of the raw graphite at 10μm magnification shows some different colour spots. These spots indicate the porous point in the mineral crystal structure which signifies impurities presence. The difference colours also indicate the presences of many associated minerals which are attached with the gangue as shown in Figure 3.

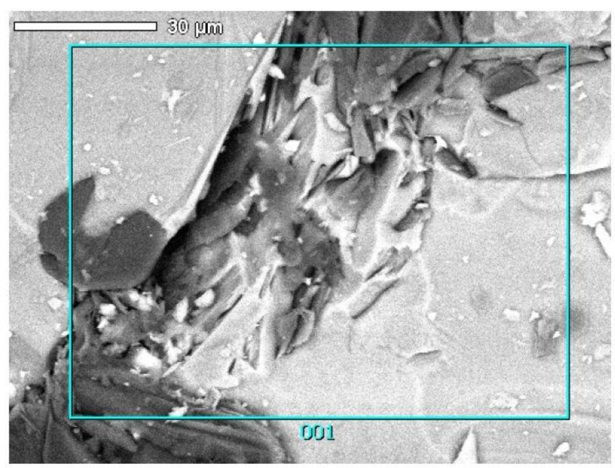

Figure 3: SEM Image at 10μm magnification

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C. EDS Elemental Analysis

The amount of energy to bombard each element for this analysis is the characteristics of that element. That is, each element requires a certain amount of energy for its bombardment. It is evident that C, O, K, and Si require 0.277, 0.525, 3.312 and 1.739KeV respectively. The mass% and the atom% show the amount of element present in the sample in question. The first analysis using this equipment in this research was that of raw graphite and the results shows that it contains 46.58% Carbon by mass with an error of only 0.26%. Figure 4 shows the peaks corresponding to each element. The vertical axis is the intensity of the X-ray which is related to number of counts of the elements present, and the horizontal axis is the energy (KeV) of bombardment. The results are as shown in Table I. The beneficiated graphite concentrate was also analysed using this method.

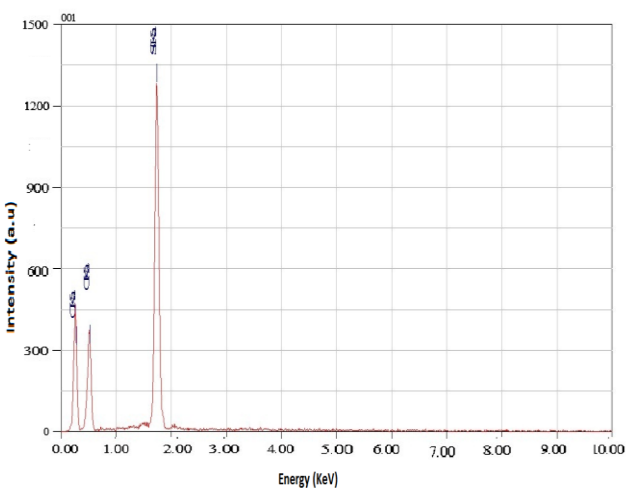

Figure 4: EDS elemental analysis of raw graphite

TABLE I
EDS RESULT OF THE RAW GRAPHITE

Element	Energy (KeV)	Mass %	Error %	At%
C	0.277	46.58	0.26	58.01
O	0.525	33.64	0.48	31.45
Si	1.739	19.78	0.14	10.54
Total		100		100

D. Post Beneficiation Characterization

Froth Floatation is one of the efficient methods of purification; it was used to beneficiate the raw graphite. After floatation and drying, it was discovered that for one run and feed of 200g, 92.70% of the solid was recovered with a loss of only 7.3%. This portrays a minimal lost and high efficiency of the system.

The characterization of the beneficiated graphite yielded 95.42% and 91.13% purity for 50µm and 90µm sizes, respectively, using SEM-EDX analysis as shown in Figure 6 and Figure 7. These purities fall within the range of high-carbon graphite (94-99.9%) and medium-carbon graphite (80-94%) respectively [13]. This method is no doubt efficient and the purity obtained is acceptable for many end uses. The purity is dependent on the particle size to some extent, the smaller the size the more efficient the beneficiation. However, a coarse size of 300µm or above and a finest size of 5µm or below did not obey this rule [1].

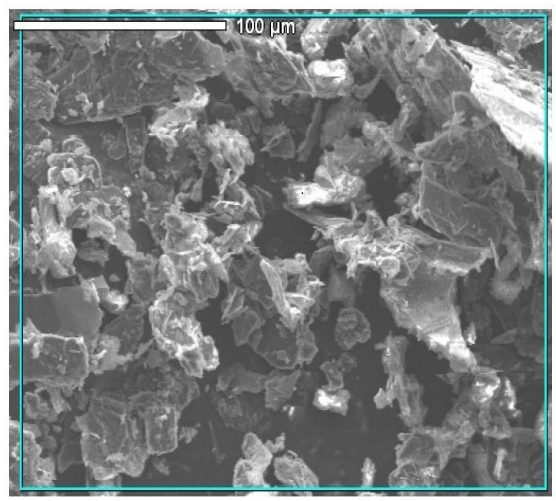

Figure 5: SEM image of beneficiated graphite powder

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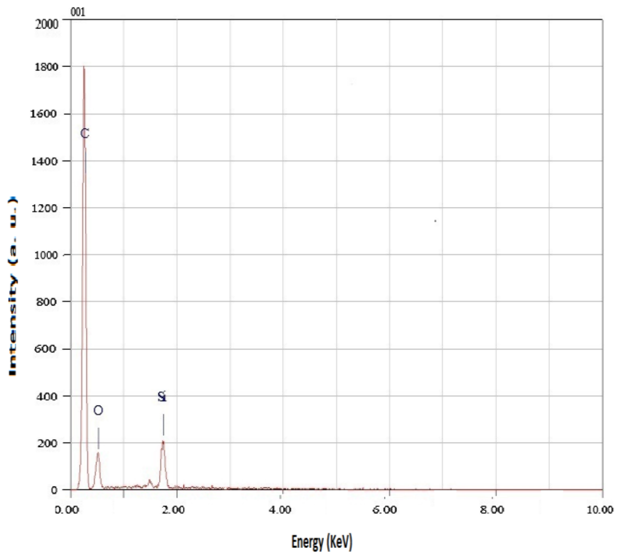

Figure 6: EDS elemental analysis of beneficiated graphite concentrate for 90µm size

Table III
EDS ELEMENTAL ANALYSIS OF BENEFICIATED GRAPHITE CONCENTRATE (90µm size)

Element	Energy (KeV)	Counts	Mass %	Error %	At%
C	0.277	10862.96	91.13	0.00	94.96
O	0.525	975.53	3.25	0.07	2.54
Si	1.739	1890.80	5.62	0.09	2.50
Total			100		100

The impurities removed (Tailings), after decantation, contained high percentage of silicon oxide (81.2%) with other oxides such as Fe_2O_3, Al_2O_3, MnO etc. The XRF oxide result for the tailings is shown in table 4.

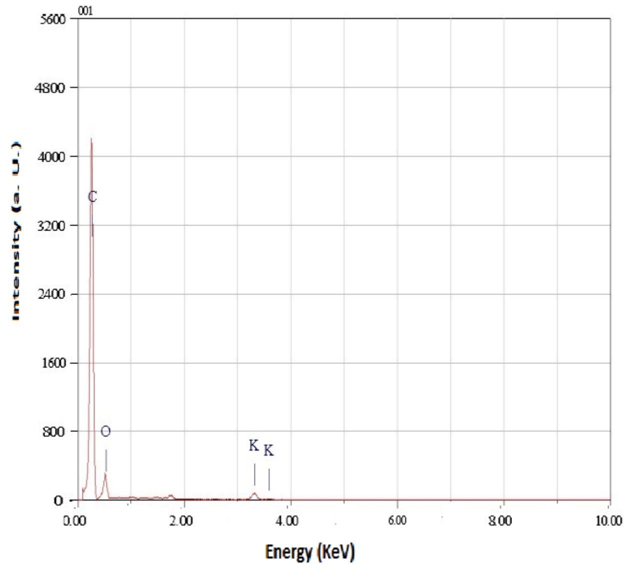

Figure 7: EDS elemental analysis of beneficiated graphite concentrate for 50µm size

Table III
EDX ELEMENTAL ANALYSIS OF BENEFICIATED GRAPHITE CONCENTRATE (50µm sieve size)

Element	Energy (KeV)	Counts	Mass %	Error %	At%
C	0.277	24840.59	95.42	0.00	97.41
O	0.525	1669.89	2.55	0.10	1.95
K	3.312	831.25	2.03	0.49	0.64
Total			100		100

The percentage carbon (46.58%) in the raw graphite confirmed that *Sama-Borkono* graphite has high initial purity and is viable for commercial exploration.

The recovery of graphite from froth floatation indicates that the floatation method of beneficiation is efficient and also viable for large scale production of pure graphite concentrate. Tailing recovered from the mineral gangue, will be a source of producing large amount of silica since it constituted 81.2% of the entire tailings.

The purity obtained is considered to be within the range of high purity graphite. Graphite containing 80 to 85% carbon is used for crucible manufacture; 93% carbon and above is preferred for the manufacture of lubricants, and graphite with 40 to 70% carbon is utilized for foundry facings. Natural graphite, refined or otherwise pure, having carbon content not less than 95% is used in the manufacture of carbon rods for dry battery cells and fuel cell bipolar plates [11, 12]. The impurities removed from the graphite contained high silica (81.2%) which can be used in the production of Portland cement, as an additive, glass for windows, and optical fibres for telecommunications. It can also be used for food and pharmaceutical application to mention but few.

TABLE IV
XRF OXIDE ANALYSIS OF TAILINGS

Sample	Tailings %
SiO_2	81.2
Al_2O_3	5.10
K_2O	2.59
CaO	1.24
TiO	0.944
MnO	0.17
Fe_2O_3	4.92
BaO	0.33
Na_2O	0.73
MgO	0.84
LOl	1.93

IV. CONCLUSION

From the results obtained for pre and post beneficiation characterization, it can be concluded that the graphite is of good grade since its purity before and after beneficiation falls within the range of high purity graphite world wide. This, no doubt, makes the *Sama-Borkono* graphite viable for commercialization. The beneficiation of the graphite using froth floatation upgraded the purity to 95.46%, which is among the range of high purity graphite concentrate as stated by Asbury Carbon [12]. Other oxides, like Al_2O_3 and Fe_2O_3 which constitute about 5% of the impurities are also very useful. Therefore, it can be concluded that the graphite is generally good and can be commercialized if exploited.

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