## CHARACTERIZATION OF MAYO-BUTALE AND WALOL-KOLEL GRAPHITE OF ADAMAWA STATE

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**Abstract.** Graphite sample from Mayo-Butale and Walol-Kolel were beneficiated by froth floatation and a yield of 52.1% and 62.65% were obtained for the two samples respectively on a second float. On aching the carbon content was improved from 36.6% and 33.6% for the raw samples to 85.0% and 83.4% for the beneficiated samples. Chemical analysis of the ash using X-ray fluorescence (XRF) showed that SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and Al<sub>2</sub>O<sub>3</sub> were present in high quantity. Others present in low quantity were K<sub>2</sub>O, CaO and TiO<sub>2</sub>. On determining the powdered density of 2280Kg/m<sup>3</sup> and 2360Kg/m<sup>3</sup> were obtained for the two samples which compares favorably to 2260Kg/m<sup>3</sup> for pure graphite. Micro structural studies revealed that the two graphite samples had a flake size of 0.5mm.

Keywords: Graphite, Mayo-Butale, Walol-Kolel, Adamawa State Received for Publication on 2 December 2017 and Accepted in Final Form 5 December 2017

#### INTRODUCTION

Graphite in its natural formation consists of carbon. Graphite has several uses, the most popular one for making pencil. Graphite is also useful as a refractory material. It is used in the production of refractory bricks, manufacture of crucibles, ladles and mould for containing molten metal. Graphite also finds application in chemical, nuclear and metallurgical industries (Cooper, 1980). Only few main deposits of graphite are found in Nigeria as revealed by the geological survey agency (Nwobi*et al*, 2002). These deposits are found in Birnin Gwari (Kaduna State), Mayo-Butale

(Adamawa State), and Gayama, Hosere Nuwa, Jauro Jalo (Taraba State). Others are Dutsen Haiya and Sama - Borkono (Bauchi State) and Alawa (Niger State). None of these deposits are being exploited to date. Investigation of the quality of the graphite in these deposits could help in this much needed utilization, Most graphite is mined with unwanted impurities. Subsequent to mining, the graphite requires a considerable amount of mineral processing such as froth floatation to concentrate the graphite and make suitable for its various applications (Mohammed et al, 2014). This work preferred froth floatation due to its versatility, selectivity and ease of setting up. The objective of this work is to study the characteristic of graphite of Mayo-Butale and Walol-Kolel of Tongo local Government area of

Adamawa state and to identify the most superior quality for exploration.

#### METHODOLOGY

In this paper, the potential and quality of Mayo-Butale and Walol-Kolel graphite, in terms of purity, was verified. The raw graphite was collected from Mayo-Butale and Walol-Kolel hill and characterized. Beneficiation using froth floatation was employed to achieve further purity.

#### 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 for various grade 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 include. 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, 500g of each of the samples were mixed in a required quantity of water and resulting pulp was continuously mixed for 10 minutes to ensure intimate mixing. Small quantities of kerosene and tunpentire oil were added in drops. The pH of the pulp was made alkaline by adding four drops of aqueous sodium carbonate to pH of 8. The pulp was again conditioned for 10 minutes again to

ensure proper effect of chemicals. Air was then introduced in to the pulp leading to the formation of froth at the surface of the pulp. The concentrate was skimmed off from the surface. collected in a container and dried in an oven at а temperature of 100°C overnight, after which the weight was taken. The water in the container was decanted leaving the tailings. The tailings were also dried in the oven overnight at 100°C and weight. From this, the yield of graphite was calculated. For further quality improvement of graphite, a second beneficiation was carried out on the beneficiated sample using the same procedure.

# CHARACTERIZATION OF GRAPHITE

#### Ash analysis (carbon content)

Five grams of powdered graphite was taken in a sintered glass crucible and placed in a furnace at a temperature of 1200°Cfor 48hours, and thereafter cooled in a desiccator. The weights of the samples were taken after cooling and the same procedure repeated until a standard weight was obtained. From the weight of the burnt graphite and the weight of the initial sample, the percentage purity of the graphite or the carbon content was obtained.

#### Powdered Density Determination

The powdered density of the pure sample was determined using a 50ml specific gravity bottle with acetone as the weighting fluid, the sample was ground to pass through 200 meshes tyler sieve. This was considered fine enough to open up all the closed pores of the particles. To a pre-weighed 50ml specific gravity bottle, 1gram of powder ore sample was added and the bottle reweighed. Acetone was then added in sufficient quantity to fill the specific gravity bottle to the brim. The weight of the bottle and its content was taken; the bottle was later filled to the brim with only acetone and weighed. Using these weight differences, the powdered density of the samples was calculated.

#### X-ray Fluorescence Studies

The X-ray fluorescence (XRF) analysis was conducted at the of the laboratory National Metallurgical Development Centre, Jos with a Philips P W1480 x-ray spectrometer using a voltage of 50KVand a current of 50Ma. Lithium fluoride 200(LIF) and pentaerythritol (PE) crystals were used. The sample was first scanned for quantitative element analysis. A tea spoon of powdered graphite was used to fill a standard specimen cell. Qualitative analysis was then carried out for the component required. The dilution technique was used and the sample was fused with lithium Meta borate (calcined state) was used. The mixture was taken in a platinum crucible, heated in electric furnace an at а temperature of 1050°C for one hour, melted over a Bunsen burner and cast into platinum dishes.

#### Micro Structural Studies

The micro-structural study was carried out at the research laboratory of the Department of Chemical Engineering, Ahmadu Bello University, Zaria. In this study, the graphite was formed, polished and examined using polarized light microscope (AXIOSCOP 40) fitted with a digital camera. Photographic records of the micro structures were made for each parallel and perpendicular sample.

#### **RESULTS AND DISCUSSIONS**

In figure 1, subscript 1 represent first float, while subscript 2 represents second float. The effects of refloating the samples are clearly observed in figure 1. The Mayo-Butale sample improved from 38.40% yield for the first float to 57.12%, while the Walol-Kolel sample improved from 34.40% yield for the first float to 62.65%. This result showed that there are more impurities in the Mayo-Butale than sample the Walol-Kolel sample. Figure 2 shows the carbon contain of 39.60% and on first float it contained 69.60%, while on second float it contained 85.0% carbon. The Walol-Kolel raw sample contained 33.60% carbon; the first float had 66.40%, while the third float had 83.40%. The MayoButale sample with 85.0% carbon is of better grade than the Walol-Kolel sample. The result of the powdered density determination is presented in Figure 3. The result showed that the powdered density of the Mayo-Butale final float sample of 2280kg/m<sup>3</sup> was closer to that of the pure graphite with density of  $2260 \text{kg/m}^3$  than that of Walol-Kolel the sample of 2360kg/m<sup>3</sup>. The result of the chemical analysis of Adamawa State Graphite using x-ray fluorescence is shown in the Table 1. The result showed that the Mayo-Butale sample was dominated with  $SiO_2$ (71.40%), Al2O3 (8.00%), CaO (3.50%), and MgO (5.40%). While Walol-Kolel sample was dominated with SiO<sub>2</sub> (68.50%),  $Al_2O_3$ (11.38%), K<sub>2</sub>O (7.40%), and CaO (4.10%). the presence of  $Al_2O_3$  and  $SiO_2$  in high quantity indicates that the graphite is of good for ceramic purposes since aluminum and silicon possess less thermal shock resistance in ceramics. The micro structural studies carried out for the and beneficiated samples raw revealed that the photographic

records of the raw sample had thin lines when viewed from the sides and had circular structures when viewed from the top. In the beneficiated samples, the thin lines and circles became more prominent in comparison with the raw samples.

Figure 1. Yield Percentage of Graphite First and Second Float



Figure 2: Carbon Percentage of Graphite Float



Figure 3. Powdered Densities



Figure 4: Microstructure of Mayo – Butale Graphite



Figure 5. Microstructure of Walol- Kolel Graphite



Table 1.XRF Graphite Analysis

Constituent	Mayo – Butale (%)	Walol – Kolel(%)	
$SiO_2$	71.40	68.50	
$Al_2O_3$	8.00	11.38	
$Fe_2O_3$	2.00	1.40	
$P_2O_3$	1.52	1.52	
Na <sub>2</sub> O	0.55	0.20	
K <sub>2</sub> O	0.28	0.04	
CaO	3.50	4.10	
TiO	0.24	0.27	
MgO	5.40	0.22	
V <sub>2</sub> O <sub>5</sub>	0.002	0.002	

#### CONCLUSION

From the investigations carried out, the following conclusions could be drawn.

- The raw Mayo- Butale sample had a higher yield of 38.40% than the Walol-Kolel sample with a yield of 34.40%.
- 2. Beneficiation improved the of vield Mayo-Butale from 38.40% to graphite 57.12% on first float, and to 85.00% on second float while the Walol-Kolel graphite improved from 34.40% for the raw sample to 62.65% on the first float and 83.40% on second float.
- 3. Both the Mayo-Butale and Walol-Kolel final float graphite with 85.00% and 83.40% carbon respectively are good for refractory purpose.
- 4. The micro structural studies revealed that the Adamawa State graphite is of the flake type with size of 0.5mm.
- 5. Therefore, it can be concluded that the graphite is

generally good and can be commercialized if exploited.

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Characterization of Mayo-Butale and Walol-Kolel Graphite of Adamawa State

Proceedings	of	the	XI	Mineral Processing
International	Seminar		on	Technology (MPT) Pp 9-15

**Reference** to this paper should be made as follows: Taru Tizhe Tatas<sup>1</sup>, et al (2017), Characterization of mayo-Butale and Walol-Kolel Graphite of Adamawa State. *J. of Engineering and Applied Scientific Research*, Vol. 9, No. 4, Pp. 38-46