

## FROTH FLOTATION UPGRADING OF A LOW GRADE COAL

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### Abstract

Nigeria is endowed with a large deposit of non-coking coal found in Okaba, Kogi State. This vast reserve of coal has not been exploited for the production of useful carbon products such as graphite and activated carbon. The coal sample was subjected to proximate/particle size analyses and froth flotation mineral processing to reduce the ash and sulphur contents. The coal as-received and as-floated was then analyzed for ash and sulphur contents. The results obtained showed that the ash and sulphur contents of the coal were reduced by 32% and 90%, respectively.

**Keywords:** Coal, carbon products, analyses, froth flotation, ash, sulphur.

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### 1. Introduction

Coal is a fossil fuel. It is a combustible, sedimentary, organic rock, which is composed mainly of carbon, hydrogen and oxygen. It is formed from vegetation, which has been consolidated between other rock strata and altered by the combined effects of pressure and heat over millions of years to form coal seams [1]. It has been estimated that there are over 909 billion tons of proven coal reserve in the world. This means that there is enough coal to last us for over 155 years. Coal can be found on every continent, with the biggest reserves in the USA, Russia, China and India [1]. In Nigeria, coal is found in Enugu, Onyeama, Okaba, Lafia, Ezimo, Akpunje and some other locations with an approximated estimate of proven reserve of over 257 million tons and inferred reserve of over 820 million tons [2-4].

Coal has found applications in different areas of lives, both domestically and industrially. The most significant uses are in electricity generation, steel production, cement manufacturing, production of activated carbon and other processes and as a liquid fuel [5]. The presence of high ash and sulphur content adversely affect the application of coal. Ash is an impurity that will not burn and it reduces the handling and burning capacity of coal. Ash increases handling costs, affects combustion and boiler efficiencies, causes clinkering and slagging while sulphur affects clinkering and slagging tendencies, corrodes chimney and other equipment such as air heaters and economizers and limits exit flue gas temperature [6].

Froth flotation is a process for selectively separating hydrophobic materials from hydrophilic by taking advantage of differences in their hydrophobicity. The flotation process is used for the separation of a large range of sulfides, carbonates and oxides prior to further refinement. Phosphates and coal are also processed and upgraded by flotation technology [7]. Flotation is a chemical process that depends on the selective adhesion to air of some solids and the simultaneous adhesion to water of other solids. It utilizes the difference in physio-chemical surface properties of particles of various minerals. After treatment with reagents, such differences in surface properties become apparent and flotation finally occurs after an air bubble attaches itself to a particle and lifts it to the water surface. It is applicable to relatively fine particles since for the coarse particles the adhesions between the particles and the bubble will be less

than the weight and the bubble will drop its load. Froth flotation is only applicable to very fine coals and coal is naturally floatable [8,9].

In view of the large deposit of coal in Okaba, Kogi State and its potential applications, this research is aimed at developing process parameters for the froth flotation of the coal.

## **2. Materials and methods**

### **2.1 Materials**

The basic material used in this work was lumps of coal obtained from Kogi State Nigeria.

#### **2.1.1 Sample collection**

About 25 kg of Okaba bulk sample was obtained from the open pit mine opened by the Nigerian Mining Corporation (NMC) from seams number 2 and 3 at depths varying between about 10 and 15 m. About 2 kg of the bulk sample was used in this work.

### **2.2 Methods**

The coal sample was subjected to particle/proximate analyses as described below:

#### **2.2.1 Particle size analysis**

The coal sample was crushed and pulverized. The sizing of the crushed coal sample was carried out by employing sieves and an automated sieve shaker (vibrating type). The sieve surface was made up of parallel bars. The sieves were arranged in the order of decreasing mesh sizes with the sieve with largest mesh size at the top. The mesh sizes employed were 1.4mm, 1mm, 850, 600, 500, 300, 250, 125, 106 and 63  $\mu\text{m}$ . A representative sample of about 172 g was measured from the crushed coal sample and was poured into the 1.4mm mesh size. The stack of sieves was subjected to the vibratory action of the sieve shaker for about an hour. The coal sample retained on each sieve was then weighed in order to determine the particle size distribution of the coal.

#### **2.2.2 Equilibrium moisture content**

Both the equilibrium and the inherent moisture content of the representative sample were determined. About 5 g of the -250 + 125  $\mu\text{m}$  was measured and placed in open air over night. About 1 g was taken each, placed in air for another 6 hours and the weight was measured, this was repeated at an interval of 30 minutes until a constant weight was maintained. The change in weight represents the equilibrium moisture content.

#### **2.2.3 Total moisture content**

For the total moisture content, the crucibles were pre-heated at a temperature of 110°C in the muffle furnace for a period of an hour, cooled and weighed. About 1 g of the -250 + 125 $\mu\text{m}$  was then poured into the pre-heated crucible and placed in the muffle furnace at a temperature of 110°C for an hour. The crucible was then removed from the muffle furnace with the aid of a tong and placed in the desiccators to cool and then weighed. This was repeated until a constant weight was maintained. The loss in weight accounts for the total moisture content.

#### **2.2.4 Ash content**

About 0.5 g of the sample was measured and poured in the silica crucible for holding the sample. The crucible and the samples were heated from ambient temperature to 815°C until the samples was fully ashed. The crucible was then removed from the furnace with the aid of a tong and placed in the desiccators prior to weighing. The incombustible residue constitutes the ash content. The result of the ash content carried out on a paired sample of Okaba coal is calculated thus:

$$\% \text{ Ash Content} = \frac{\text{final weight (g)}}{\text{initial weight (g)}} \times 100 \quad (1)$$

### 2.2.5 Sulphur content determination

About 2 g of the sample was weighed into a beaker with 10 ml of concentrated HCl and 20 ml of distilled water. The solution was continuously stirred for a period of an hour. The mixture was filtered by pouring into a conical flask which had a filter paper and a funnel at the top. After the filtrate had been completely collected in the conical flasks, about 15 ml of each of the filtrate was taken for further UV spectrophotometer analysis to determine their sulphur contents. The procedure was repeated for all the size ranges.

### 2.2.6 Ultra-violet spectrophotometry

About 5 ml of the filtrate was pipetted and taken into a test tube and 15 ml of distilled water was added. About 2 ml of conditioning reagent was also added. The content was covered and allowed to stand for few hours. A spatula full of barium chloride was then added. The turbidity was finally read with a UV spectrophotometer at 344 and 348 nm. A calibration curve was constructed from a stock solution containing 1 g of sulphur in 1000 ml. The sulphur content was determined with Equations 2, 3 and 4.

$$\text{Absorbance per gram of coal} = \frac{\text{absorbance value}}{2} \quad (2)$$

$$\text{Weight of sulphur is} = \frac{\text{Absorbance value} \times \text{Volume of filtrate}}{2.6265} \quad (3)$$

$$\% \text{ Sulphur Content} = \frac{\text{weight of sulphur}}{\text{weight of coal}} \times 100 \quad (4)$$

### 2.2.7 Volatile matter of the coal

The silica crucible was pre-heated in the furnace at a temperature of 900°C for 7 minutes, cooled and weighed. About 0.5 g of the sample coal was weighed into the silica crucible. Two drops of benzene was added to the coal and the crucible was covered. It was placed in the furnace at a temperature of 900°C for 7 minutes. The crucible was then removed from the furnace and placed in the desiccators prior weighing. The loss in weight accounts for the volatile matter of the coal sample.

$$\text{percentage volatile matter} = \frac{\text{loss in weight of moisture free coal}}{\text{weight of moisture free coal}} \times 100 \quad (5)$$

### 2.2.8 Fixed carbon content

The fixed carbon content of the coal was determined by calculation with the relation below:

$$\% \text{ Fixed Carbon Content} = 100 - \% \text{ moisture} - \% \text{ ash content} - \% \text{ volatile matter} \quad (6)$$

### 2.2.9 Froth flotation

The flotation cell was filled to one third of its volume with water and about 200 g of the -300+250µm sample was added into it. About 5 ml of kerosene was added as collector and the pulp was conditioned for about five minutes by agitating it with the impeller. Then, about 2 ml of turpentine was added as the frother and the pulp was aerated. The stable froth on the surface of the flotation cell was collected, dried and taken for further laboratory analysis. The procedure described was repeated but with -250+125 and -125+106 µm size fractions. The reduction percentages in ash and sulphur are calculated with the Equations below

$$\% \text{ Reduction in Ash} = \frac{\text{wt of ash in raw materials} - \text{wt of ash in froth sample}}{\text{wt of ash in the raw sample}} \times 100 \quad (7)$$

$$\% \text{ Reduction in Sulphur} = \frac{\text{wt of S in raw sample} - \text{wt of S in froth sample}}{\text{wt of S in the raw sample}} \times 100 \quad (8)$$

## 3. Results and discussion

Figure 1 shows the particle size distribution analysis of Okaba coal at A to K size ranges corresponding to +1.4, -1.4+1 mm, -850+600, -600+500, -500+300, -300+250, -250+125, -125+106, -106+63 and -63 µm, respectively. The results obtained shows that after primary and secondary crushing the mesh size H (-250+125 µm) has the highest weight fraction of 28.8% of the total fraction, followed by the mesh size F (-500+300) with 22.4% of the total weight. The results thus show that about half of the coal material is contained in the two

relatively coarse size ranges. The results suggest that Okaba coal has a low grindability being somewhat resistant to crushing and grinding.

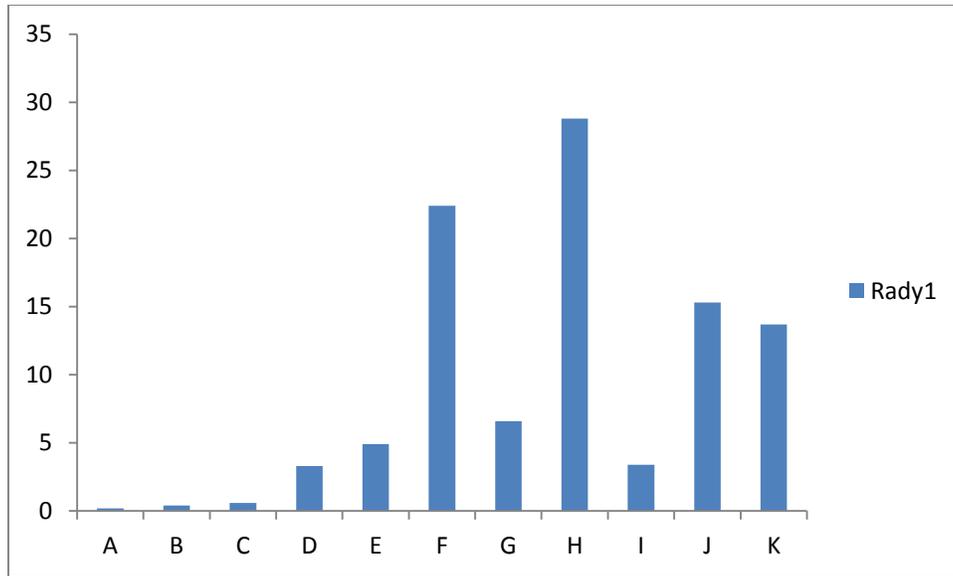


Fig. 1 Particle size distribution of Okaba coal as-received

Figure 2 shows the ash content and the mg of sulphur dissolved in the size fractions C to J. The results show that the highest ash content of 13.6% was obtained for the mesh size F followed by the mesh G with ash content of about 9.4%. The lowest ash content of 8.4% was found in the mesh size I. The average ash content was calculated as about 9.6%. The results showed that the ash contents of the finer coal fractions are generally lower than those of the coarser fractions. The observation made in the distribution of the ash contents seems to agree with the lower response of the coarser fractions to crushing and grinding. The figure further shows that the highest sulphur was dissolved from the size fraction F and the lowest from the size fraction D.

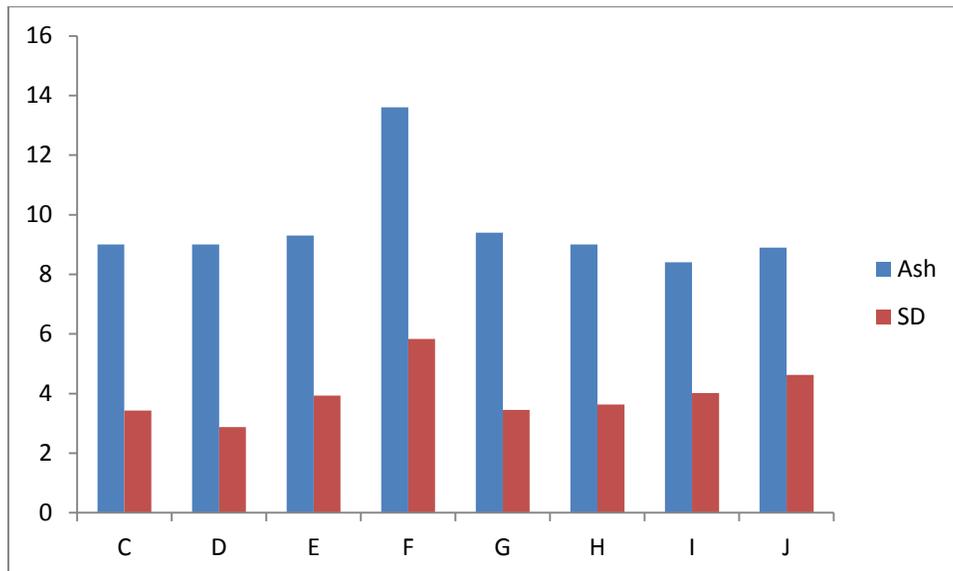


Fig. 2 The ash content and mass of sulphur dissolved (SD, mg) in the floated coal samples

Figure 3 shows the effects of the froth flotation on the ash and sulphur contents of the coal. The results obtained show that the ash content of the floated concentrate was reduced by about 43.25%, while the sulphur content was estimated to have been reduced by about 90%. The high reduction in the ash and sulphur content is very significant as it renders Okaba coal more suitable for inclusion in coal blending for cokemaking, coking coal and for other engineering

applications [10,11]. With the upgraded Okaba coal having an ash content of less than 7% and sulphur content of less than 0.1% it makes it suitable for the production of activated carbon [12,13]. The results obtained have shown that froth flotation is an efficient method to de-mineralize the Okaba coal.

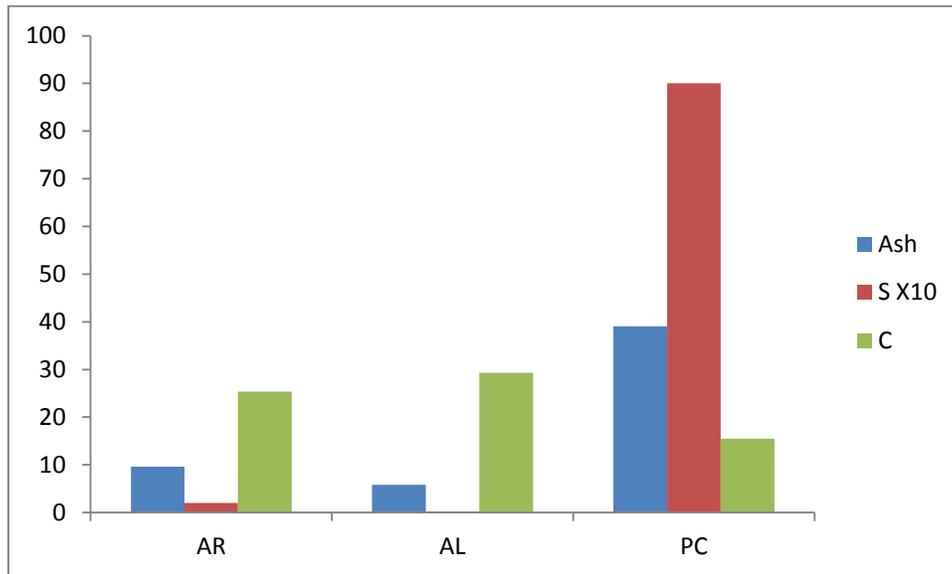


Figure 3 shows the effects of the froth flotation on the ash and sulphur contents of the coal

#### 4. Conclusions

This research have revealed that upgraded Okaba coal has a total moisture content, volatile matter and fixed carbon content of 1.0%, 58.4% and 29.28% respectively. The volatile matter content places Okaba coal as high-volatile bituminous rank [11]. It can be deduced from figure 3 that froth flotation is an effective way of upgrading Okaba coal as it was able to reduce the ash content of Okaba coal from an average of 9.6% to an average of 5.85% representing 43.25% reduction in ash content, it was also able to reduce the sulphur content greatly from an average value of 0.2% to an average value of 0.02% representing about 90% reduction in sulphur content. With this upgrade, Okaba coal can be effectively utilized in metallurgical coke making and in the production of carbon useful products such as activated carbon.

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