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BENEFICIATION OF OVBIOMU LOW GRADE COAL USING FROTH FLOTATION FOR INDUSTRIAL APPLICATIONS

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ABSTRACT: *Coal flotation is a highly effective separation method utilizing the differences in surface properties of coal and gangue minerals. This research entailed the beneficiation by froth flotation of low-grade coal from Obviomu Coal Mines, Edo State, Nigeria for improvement of the fixed carbon content and calorific value for industrial application. 50 kg of coal samples were collected from five different pits and were reduced using a jaw crusher to a size of 1400 µm, pulverized to a size of 1100µm. A 500-gram sample of coal was weighed and ground in a ball mill until 100% of it passed through a 250-micrometer sieve; these steps were then repeated for sieve sizes of 180, 125, 90, and 63 micrometers, respectively. A DENVER flotation cell was charged with 500 grams of well-ground coal sample of 250 µm sieve size. The process was then repeated with sieve sizes of 180 µm, 125 µm, 90 µm, and 63 µm, in that order, the results of the froth flotation beneficiation method revealed that, the weight in mass of the concentrate at sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm was found to be 319.209 grams, 119.227 grams, 113.028 grams, 237.657 grams, and 141.343 grams, respectively, while the weight in mass of the tailings was found to be 180.791 grams, 380.773 grams, 386.972 grams, 262.343 grams, and 358.657 grams, respectively. The calorific values of the concentrate and tailings were measured using a CalK2 bomb calorimeter. At sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm, the concentrate's values were found to be 44.45 MJ/Kg, 43.84 MJ/Kg, 43.66 MJ/Kg, 46.02 MJ/Kg, and 43.48 MJ/Kg, respectively, while the calorific values of the tailings were found to be 3.13 MJ/Kg, 3.31 MJ/Kg, 0.79 MJ/Kg, 3.25 MJ/Kg, and 0.18 MJ/Kg respectively. The fixed carbon content at sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm was found to be 74.00%, 73.000%, 72.70%, 76.60%, and 72.40%, respectively, in the concentrate, while 5.20%, 5.50 %, 1.30%, 5.40%, and 0.30 % were found in the tailings. This information was obtained through proximate analysis of the concentrate and tailings at different sieve sizes. At 90 µm, the optimum sieve size, a rise in the fixed carbon content of the coal samples was recorded, accounting for 88.53% increase of the calorific value. The study concludes that direct froth flotation is a viable and efficient method for improving the quality of low-grade coal for industrial use.*

KEYWORDS: Coal beneficiation, Froth flotation, Proximate analysis, Calorific value.

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INTRODUCTION

Alabi et al. (2024) revealed that the actual liberation size of Ovbiomu coal is 125 µm, whereas the economic liberation size is 180 µm, indicating that the coal is of low grade. The coal deposit has a low fixed carbon content of 40.65% and a low calorific value of 24.41 MJ/kg, according to the results and data acquired. The actual liberation size has a fixed carbon content of 63.40% and a calorific value of 38.07 MJ/kg, indicating that the coal should be upgraded to a fairly high grade quality product that can be used for home, metallurgical, and industrial purposes. The economic liberation size has a fixed carbon content of 30.93% and a calorific value of 18.57 MJ/kg, the ultimate analysis revealed the following elements Oxygen (29.56%), Carbon (27.68%), Hydrogen (4.8 %), Nitrogen (1.15%), and Sulphur (0.13%).

According to several studies by Smith et al. (2018) and Jones and Brown (2019), the process of treating the coal slurry with particular chemicals to make the coal particles hydrophobic is the first step in the froth flotation method of lignite coal beneficiation. This is a critical stage because it enables the precious coal particles to adhere to air bubbles that have been added to the slurry. The required coal particles are then present in a froth layer that forms as a result of stirring the aerated slurry in a flotation cell.

Furthermore, in order to attain the best separation efficiency, researchers like White and Green (2020) and Davis (2021) have stressed the need of regulating a number of variables, including pH, temperature, and the kind and dose of chemicals used in the froth flotation process. After skimming out the froth containing the hydrophobic coal particles, additional processing is performed to produce a concentrate that is high in lignite coal.

According to Smith and Doe (2018), the process of froth flotation for coal beneficiation has been extensively researched in the literature. Using the variations in the surface characteristics of coal and related contaminants, froth flotation is a very successful method of separation. In order to lift the hydrophobic coal particles to the surface and remove the hydrophilic impurities, the procedure includes creating a froth layer on top of a slurry mixture using air bubbles.

Furthermore, Jones et al. (2017) emphasized the significance of a number of variables in maximizing the froth flotation process for coal beneficiation, including pH levels, reagent dose, pulp density, and particle size. Researchers have been able to significantly enhance coal quality and recovery rates by adjusting these variables, which suggests that froth flotation is a viable method for increasing the value of coal reserves.

Compared to other coal varieties, there has been relatively little research on the froth flotation method of anthracite coal beneficiation; however, some studies have looked at the possible uses of this procedure. Because anthracite coal is hydrophobic, it poses special challenges in beneficiation despite its high carbon content and low volatile matter (Cao *et al.*, 2017).

Froth flotation is one technique that can be used in the anthracite coal beneficiation process to separate the hydrophobic anthracite particles from the hydrophilic gangue minerals. By preparing the coal slurry with the right chemicals to encourage the selective attachment of air bubbles to the hydrophobic coal surfaces, this separation is accomplished. Next, the anthracite concentrate-containing foam is collected for additional processing (Cao *et al.,* 2017).

To accomplish effective separation and optimum anthracite recovery, froth flotation parameters, such as reagent dosage, pH level, and flotation period, must be optimized. To

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further improve flotation performance, appropriate frothers and collectors that are matched to the special surface characteristics of anthracite coal must be used (Cao *et al.*, 2017).

Coal beneficiation helps to eliminate impurities that lead to the production of ash and sulfur when coal is burned by promoting uniformity in the size of the pulverized coal following commuting. One method of beneficiation is gravity separation, which entails passing coal particles through sieves with progressively decreasing mesh sizes. A method of separating tiny particles based on the variations in surface hydrophobicity of various constituents is called foam flotation. Generally speaking, coal particles less than 0.5 mm are beneficiated by froth flotation. The wetting abilities of the coal-rich and mineral-rich particles in an aqueous solution are what determine the fine coal separation in this technique (Felici, 1996; Saleh *et al.*, 2007). The stable bond between air bubbles and the coal surface forms the basis of this mechanism. Numerous physical and chemical variables affect this connection's durability (Cebeci, 2002). Because minerals are hydrophilic by nature and coal is hydrophobic by nature, flotation is frequently a very successful method of cleaning coal (Tao *et al.*, 2002). According to Eroġl *et al.* (2003), froth flotation has several benefits in the coal processing industry, including comparatively low capital and space needs and a comparatively high recovery that may be achieved under a variety of operating conditions. The three objectives of the traditional froth flotation method are to produce coking coal, reduce environmental pollution by cleaning the process water, also known as black water, which is removed from the coal preparation plant, and obtain a product with low sulfur and ash content by recovering the coal in slime (Demirdas, 2002). Desulphurization using physical, chemical, and biological techniques can improve high sulfur coals. Froth flotation is one of the best physicochemical techniques for removing pyritic sulfur from coal (Demirdas, 2002).

Ehsani *et al.* (2007) declared that the ash and sulfur content of coal has been reduced more than with conventional collectors and frothers when kerosene and pine oil are used. The presence of kerosene and methanol collector increases flotation yield, which in turn leads to high recoveries of coal and ash of the concentrates by floating ash-forming materials. However, the use of sodium polyacrylic acid as a pyrite depressant improved the overall recovery of coal concentrate but did not enhance the reduction of sulfur.

Coal preparation includes size reduction, grinding, screening, and handling (Sujeet *et al.*, 2019). Compared to coal, which varies greatly based on the type of coal, moisture content in the ore, and ash content, fuel oils have a far more consistent calorific value. Proximate analysis has long been used to distinguish between volatile, fixed carbon and inert components in order to evaluate the rank of coals (Misra, 1992).

Aina *et al.* (2009) and Mahapatra (2016) stated that a substance's heating value, also known as its calorific value, is the amount of heat released when a certain amount of that substance—typically a fuel or food—is burned. The calorific value is a characteristic shared by all substances. Quantification of energy per unit of substance, usually mass, is done in measures such as Kcal/kg, KJ/kg, J/mol, and Btu/m3. According to Mahamudul *et al.* (2013), proximate analysis comprises the following elements: moisture content, volatile matter, ash content, and carbon content.

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METHODOLOGY

Sample Collection and Preparation

Fifty (50) kilograms of the coal samples were collected from five (5) different pits each at Ovbiomu coal mines Owan East Local Government Area, Edo State, Nigeria using a random sampling method. The samples were thoroughly mixed and homogenized to obtain a uniform sample using the cut and quartering method. The lumpy coal was crushed using a jaw crusher. For the froth flotation concentration processes, a sub-sample of 500 g was weighed and pulverized using and further ground using a ball mill and sieved through 250 µm until 100% passing was achieved. These procedures were repeated for 180 µm, 125 µm, 90 µm, and 63 µm mesh sizes respectively.

Froth Flotation

Five hundred (500) grams of the ground coal sample was charged into a Denver flotation cell (model BPAE22449) and mixed with water to form slurry in a ratio of 1:4 (500 grams of coal sample and 2000 ml of water). The slurry was agitated for one (1) minute and checked using a pH meter, and the pH was adjusted to 9.00 by adding sodium hydroxide (regulator) for all sieve sizes. The slurry was further agitated for one (1) minute, and 0.2 grams of zinc sulfate activator was added to the slurry and re-agitated for another one (1) minute. Consequently, 0.2 grams of copper sulfate (depressant) was added to the slurry and re-agitated for one (1) minute, and 0.2 ml of a mixture of kerosene and pine oil was added to the slurry. The slurry was re-agitated again for one (1) minute, 0.2gram of potassium ethyl xanthate was added to the slurry and agitated again for one (1) minute, and finally, 0.2 ml of sen –froth 516 was added to the slurry and re-agitated again for one (1) minute. The air valve was opened after one (1) minute to introduce air into the slurry for frothing to take place. The froth was collected for three (3) minutes until the slurry became clear. The air valve was closed. The froth (concentrate) and depressed (tailings) collected were dewatered, filtered, dried, weighed and recorded. These procedures were repeated for 180 µm, 125 µm, 90 µm, and 63 µm sample sizes respectively.

Proximate Analysis

The proximate analyses involved the determination of moisture content, volatile matter, ash content, and fixed carbon content as per ASTM Standard procedures D-3172 for the varied sieve size using Equation 1-7.

a. Moisture content

One (1) gram of the 'concentrate' and 'tailings' of the beneficiated coal of sieve sizes 250 µm was measured and put inside a porcelain crucible, heated in an electrical hot air oven at a temperature of 100 -110˚C for one hour, cooled in a desiccator, weighed and recorded. This process was repeated until the weight of the crucible containing anhydrous coal became constant. Loss of weight was reported as moisture content, as shown in equation one (1). These procedures were repeated for the beneficiated products of 180 µm, 125 µm, 90 µm, and 63 µm sieve mesh respectively.

$$\text{Moisture content (\%)} = \frac{loss\ of\ weight\ in\ gram}{initial\ weight\ of\ sample\ in\ gram} \times 100\% \qquad 1$$

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Moisture content (%) = $\frac{M_1 - M_2}{M_1} \times 100\%$ 2

Where M1 is the initial mass of the coal sample, and M2 is the final mass of the coal sample.

b. Volatile matter

One (1) gram of moisture free 'concentrate' and 'tailings' of the beneficiated coal of sieve sizes 250 µm was measured and put inside a crucible covered with a lid, placed in a muffle furnace pre-heated at temperature 950˚C for seven minutes, cooled first in the air, then inside the desiccator, weighed and recorded. This process was repeated until the weight of the crucible containing coal became constant. Loss of weight was reported as volatile matter (%) and calculated using equation three (3). These procedures were repeated for the beneficiated products of 180 µm, 125 µm, 90 µm, and 63 µm sieve mesh respectively.

Volatile matter (%) = $\frac{loss\ of\ weight\ in\ gram}{initial\ weight\ of\ sample\ in\ gram} \times 100\%$ 3

Volatile matter (%) = $\frac{M_1 - M_2}{M_1} \times 100\%$ 4

Where, M_1 is the initial mass of the coal sample and M_2 final mass of the coal sample.

c. Ash content

One (1) gram of the 'concentrate' and 'tailings' of the beneficiated coal of sieve sizes 250 µm was measured, put inside an open crucible, placed in a muffle furnace heated at a temperature of 750˚C for one and a half hours (90 minutes), crucible was taken out, cooled first in the air, then inside the desiccator, weighed and recorded. This process was repeated until the weight of the crucible containing coal became constant. Loss of weight was reported as ash content (%) and calculated using equation five (5). These procedures were repeated for the beneficiated products of 180 µm, 125 µm, 90 µm, and 63 µm sieve mesh respectively.

Volatile matter (%) = $\frac{loss\ of\ weight\ in\ gram}{initial\ weight\ of\ sample\ in\ gram} \times 100\%$ 5

Volatile matter (%) = $\frac{M_1 - M_2}{M_1} \times 100\%$ 6

Where, M_1 is the initial mass of the coal sample and M_2 final mass of the coal sample.

d. Fixed carbon content

Fixed carbon content was calculated using Equation 7

Carbon (%) is reported as (100% - (Moisture Content (%) + Volatile Matter (%) + Ash Content (%)) ………………………………………………………………………..…… 7

Calorific value of Ovbiomu coal determinations using (CAL 2k model 73/23/EEC)

bomb calorimeter

One (1) gram of the 'concentrate' and 'tailings' of the beneficiated coal of sieve sizes 250 µm was weighed, placed inside the vessels, the firing wire was placed on the sample inside the

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vessel and closed with the cap thread, the vessel was filled with 3000 kpa oxygen gas from the filling station, the bomb calorimeter lid was opened, the prepared vessel containing the coal sample was inserted into the well of already warmed bomb calorimeter; the calorimeter's lid was closed, drift and time was displayed, the vessel was fired. Immediately after firing the final status and result was displayed as it developed during the final period. The final period criteria were met then the determination ended and the values were recorded. These procedures were repeated for the beneficiated products of 180 µm, 125 µm, 90 µm, and 63 µm sieve sizes respectively.

RESULTS AND DISCUSSION

Results of Froth Flotation Concentration Processes of Ovbiomu Coal

Table 1: Weight in Mass of Concentrate and Tailings from Froth Flotation Process of Ovbiomu Coal

Sieve sizes (µm)	Weight of concentrate (g)	Weight of tailings(g)	Total weight (g)
250	319.209	180.791	500.00
180	119.227	380.773	500.00
125	113.028	386.972	500.00
90	237.657	262.343	500.00
63	141.343	358.657	500.00

Table 1 showed the results from the froth flotation process; it is evident that the weight in mass of concentrate varies across different sieve sizes. The highest weight in mass of concentrate was obtained at a sieve size of 250 µm, with a value of 319.209 grams. However, as the sieve size decreases, the weight in mass of concentrate decreases as well. This trend can be attributed to the fact that smaller sieve sizes allow for more efficient separation of the coal particles from the gangue minerals, resulting in a higher yield of the desired concentrate. In contrast, the weight in mass of the tailings product, which consists of gangue minerals, increases as the sieve size decreases. This is expected since smaller sieve sizes allow for better separation of the gangue minerals from the coal particles. The highest weight in mass of the tailings was obtained at a sieve size of 125 µm, with a value of 386.972 grams. These results indicate that the froth flotation process is effective in separating the coal particles from the gangue minerals, resulting in a higher yield of the desired product.

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Results of Proximate Analysis

Table 2: Proximate Analysis of the Concentrate

Sieve sizes (µm)	Fixed carbon content (%)	Moisture content (%)	Volatile matter (%)	Ash content (%)
250	74.00	9.50	3.20	13.30
180	73.00	9.30	1.60	16.10
125	72.70	8.20	1.10	18.00
90	76.60	8.60	1.40	13.40
63	72.40	8.80	4.40	14.40

Table 3: Proximate Analysis of the Tailings

Sieve sizes (µm)	Fixed carbon content (%)	Moisture content (%)	Volatile matter (%)	Ash content (%)
250	5.20	18.40	56.70	19.70
180	5.50	18.80	61.40	14.30
125	1.30	18.60	67.20	12.90
90	5.40	17.30	65.60	13.70
63	0.30	13.40	68.70	17.60

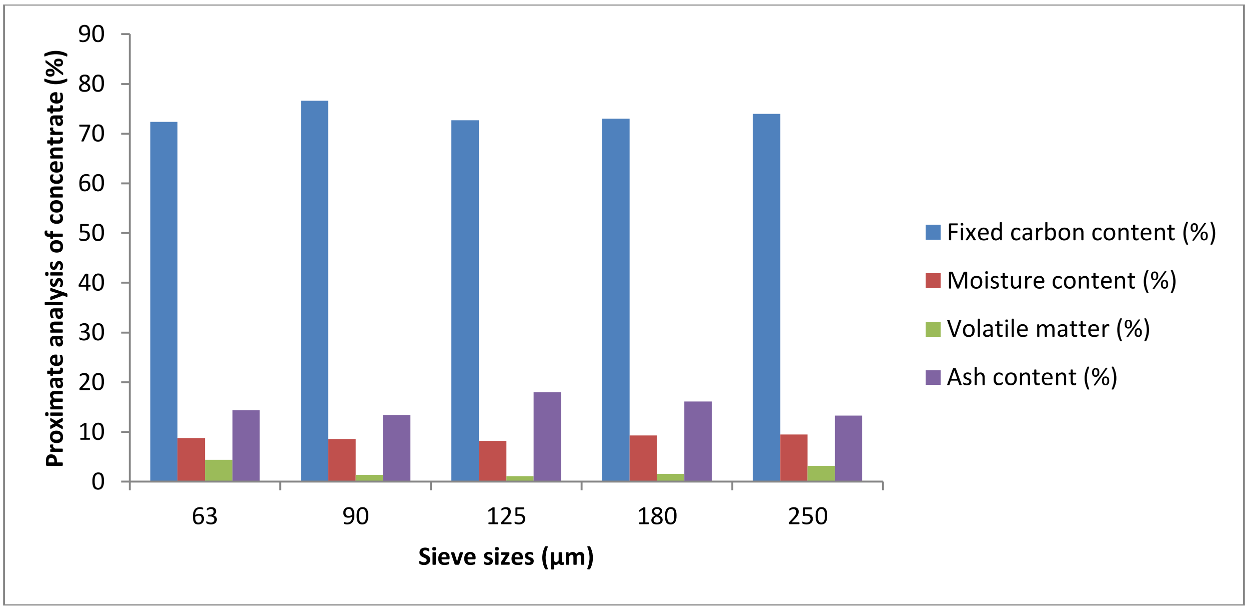

Figure 1: Plot of proximate analysis (%) of concentrate and against sieve sizes (µm) of Ovbiomu coal

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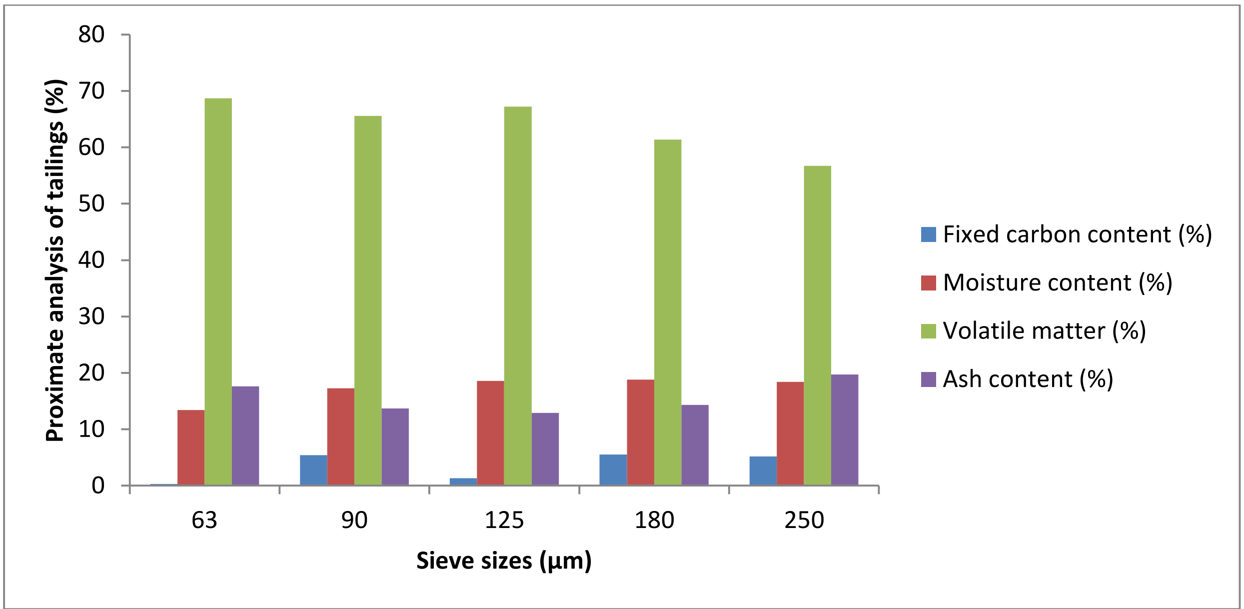

Figure 2: Plot of proximate analysis (%) of tailings and against sieve sizes (µm) of Ovbiomu coal

Figure 1 revealed that the highest percentage fixed carbon content of the concentrate was obtained at a sieve size of 90 µm, with a value of 76.60%. As the sieve size increases or decreases from this value, the percentage fixed carbon content of the concentrate decreases. This trend can be attributed to the fact that the coal particles with higher fixed carbon content are more efficiently separated at the optimal sieve size, resulting in a higher percentage fixed carbon content of the concentrate, these values of the concentrate falls within the range of 60% and 90% fixed carbon content which suitable for coke production (International Energy Agency, 2018). In contrast as revealed in Figure 2, the percentage fixed carbon content of the tailings is significantly lower compared to that of the concentrate, indicating that the gangue minerals have lower fixed carbon content. The lowest percentage fixed carbon content of the tailings was obtained at a sieve size of 125 µm, with a value of 1.30%. These results further support the effectiveness of the froth flotation process in separating the coal particles from the gangue minerals, resulting in a higher percentage fixed carbon content of the desired product.

Figure 1 also revealed that the moisture content of the concentrate to be 9.50%, 9.30%, 8.20%, 8.60%, and 8.80%, for sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm respectively. The highest moisture of concentrate was obtained at a sieve size of 250 µm, with a value of 9.50%. However, as the sieve size decreases, the moisture content of concentrate decreases as well. This trend can be attributed to the fact that smaller sieve sizes absorbed less moisture during beneficiation, while Figure 2 revealed that the tailings have the moisture content of 18.40%, 18.80%, 18.60%, 17.30% and 13.40% for the following sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm respectively. The highest moisture of the tailings was obtained at a sieve size of 180 µm, with a value of 18.80 %. However, as the sieve size decreases the moisture content of the tailings varies and decreases as well. This trend also contributes to the fact that smaller sieve sizes absorb less moisture.

Figure 1 shows that the volatile matter of the concentrate to be 3.20%, 1.60%, 1.10%, 1.40% and 4.40% for sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm respectively. The highest volatile matter of the concentrate was obtained at a sieve size of 63 µm, with a value

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of 4.40%. However, as the sieve size decreases, the volatile matter of the concentrate varies and increases. This trend can be attributed to the fact that smaller sieve sizes contain more volatile matter, while Figure 2 revealed the tailings have volatile matter of 56.70%, 61.40%, 67.20%, 65.60% and 68.70%. Most of the volatile matter was depressed using depressants into the tailings across the sieve mesh which is in contrast to that of the concentrate which revealed the presence of a small amount of volatile matter. The highest volatile matter of the tailing was obtained at a sieve size of 63 µm, with a value of 68.70%. However, as the sieve size decreases, the volatile matter of the tailings varies and increases. This trend can be attributed to the fact that smaller sieve sizes contain more volatile matter. These values are of the concentrate that falls within the range of 60% and 90% carbon content for coke production (International Energy Agency, 2018).

Figure 1 revealed the ash content of the concentrate to be 13.30%, 16.10%, 18.00%, 13.40% and 14.40% for sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm respectively. The highest ash content of the concentrate was obtained at a sieve size of 125 µm, with a value of 18.00%. However, as the sieve size decreases, the ash content of the concentrate varies, while Figure 2 revealed the tailing have ash content of 19.70%, 14.30%, 12.90%, 13.70% and 17.60% for sieve sizes of 250 µm, 180 µm, 125 µm, 90 µm, and 63 µm respectively. The highest ash content of the tailing was obtained at a sieve size of 250 µm, with a value of 19.70 %. However, as the sieve size decreases, the volatile matter of the tailings also varies. The beneficiated coal products contain good ash percentages that a clean coal should have.

Calorific Value of the products from concentration processes of Ovbiomu Coal

Table 4: Calorific Value of the Concentrate and Tailings from Froth Flotation Process of Ovbiomu Coal

Sieve sizes (µm)	Calorific Value (MJ/kg)	
	Concentrate	Tailings
250	44.45	3.13
180	43.84	3.31
125	43.66	0.79
90	46.02	3.25
63	43.48	0.18

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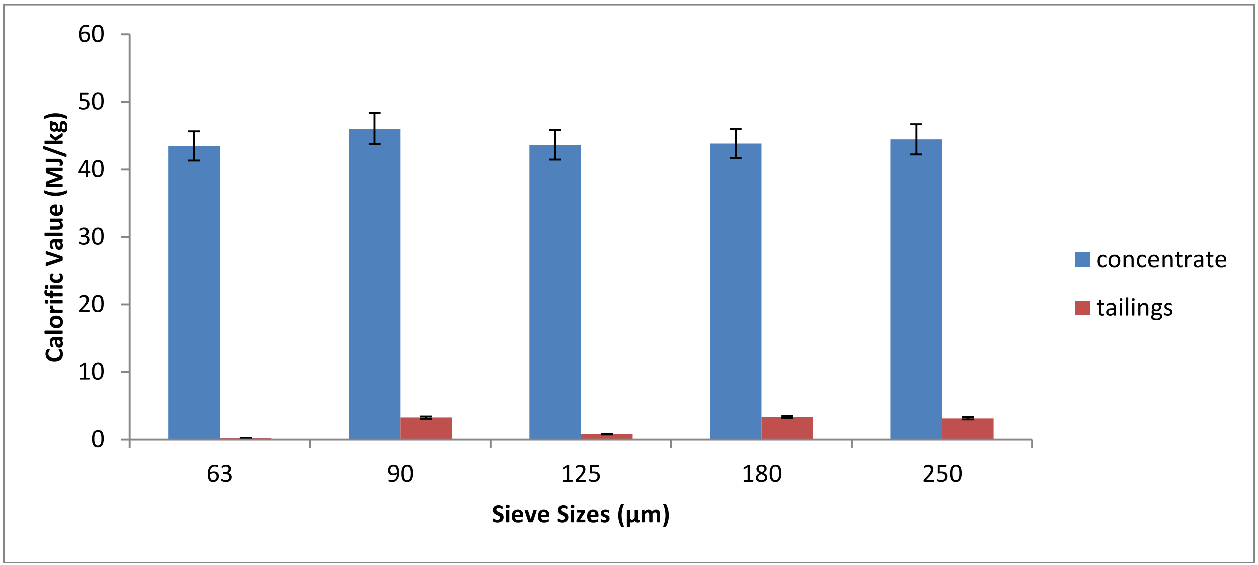

Figure 3: Plot of calorific value (MJ/kg) of concentrate and tailings against sieve sizes (µm) of Ovbiomu coal

Moving on to the calorific values of the concentrate and tailing, as shown in Figure 3 it was observed that the calorific values of both products varied across different sieve sizes. The highest calorific value of the concentrate was obtained at a sieve size of 90 µm, with a value of 46.02 MJ/kg, these values of the froth products meet the standard calorific value of 28 MJ/kg to 31 MJ/kg for coke production (World Coal Association, 2022). As the sieve size increases or decreases from this value, the calorific value of the concentrate decreases. This trend can be attributed to the fact that the coal particles with higher calorific values are more efficiently separated at the optimal sieve size, resulting in a higher calorific value of the concentrate.

In contrast, the calorific value of the tailings is significantly lower compared to that of the concentrate, indicating that the gangue minerals have a lower calorific value. The lowest calorific value of the tailings was obtained at a sieve size of 125 µm, with a value of 0.79 MJ/Kg. These results further support the effectiveness of the froth flotation process in separating the coal particles from the gangue minerals, resulting in a higher calorific value of the desired product.

Finally, the percentage fixed carbon content of the concentrate and tailings, as shown in Figure 1 and 2, reveals a similar trend to that of the calorific values. As the sieve size increases or decreases from this value, the percentage carbon content of the concentrate decreases.

CONCLUSIONS AND RECOMMENDATIONS

Conclusion

This study provides a comprehensive beneficiation of Ovbiomu low grade coal using froth flotation for quality improvement towards metallurgical industries utilization. The beneficiation of the coal was done across five sieve sizes using froth flotation, it can then be concluded that:

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i. The data offer insightful information about the weight in mass of Ovbiomu coal and it shows that, as the sieve size decreases, the weight in mass of the concentrate decreases as well.

ii. The weight in mass of the tailings which consists of gangue minerals increases as the sieve size decreases.

iii. 90 µm sieve size is the optimum after the froth flotation concentration process; the calorific value of Ovbiomu coal increased from 24.41 MJ/kg to 46.02 MJ/kg and the percentage fixed carbon content increased from 40.65% to 76.60%.

iv. These findings suggest that the percentage fixed carbon content of the concentrate and tailings is influenced by the proximate analysis conducted after the concentration process. The disclosed concentration process seems to have a varying impact on the percentage fixed carbon content of the concentrate and tailings at different sieve sizes.

v. Generally, the froth flotation process was found to be more effective in improving the fixed carbon content and calorific value of the coal, as indicated by the higher weight in mass, calorific value, and percentage fixed carbon content of the concentrate compared to the tailings.

vi. Finally, the findings highlight the effectiveness of the froth flotation concentration process in improving the quality of the coal for industrial utilization. The results contribute to the understanding of coal beneficiation and its potential for enhancing the utilization of low-grade coal resources.

Recommendation

Other concentration methods should be used to beneficiate Ovbiomu coal to improve its calorific value and carbon content and ascertain a better beneficiated process and products which can be used for domestic, metallurgical and industrial purposes.

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CONFLICTS OF INTEREST

The authors declare that there exist no known competing interests that could have appeared to influence the work reported in this paper.

AUTHORS CONTRIBUTION

All the authors contributed to formulating, developing, and drafting this research article.

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