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Proximate and Ultimate Analyses of Biocoal Briquettes of Nigerian's Ogboyaga and Okaba Sub-bituminous Coal

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Authors' contributions

This work was carried out in collaboration between all authors. Author JOA designed the study, wrote the protocol, and wrote the first draft of the manuscript. Author JSI managed the analyses of the study while author EIK managed literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Proximate and Ultimate analyses of biocoal briquettes were undertaken with the aim of presenting the analytical results and ascertaining the optimum biomass composition for use as composite domestic fuel. Coal samples from 2 coal mines of Ogboyaga and Okaba in Kogi state of Nigeria were collected. The samples were pulverized and blended with sawdust at various mixing proportions of 0:100, 10:90, 20:80, 30:70, 40:60, 50:50 and 100:0 sawdust: Coal. Cassava starch was used as binding agent while calcium hydroxide was used as a desulphurizer for the briquettes. Briquettes of various blends were produced. Proximate and ultimate analyses were carried out to ascertain the elemental composition of the raw materials and the biocoal briquettes. Experimental tests, which involved the determination of calorific value was also determined. This study revealed that biocoal briquettes from Okaba and Ogboyaga coal mines are suitable for the production of environmentally solid fuel that can be used for domestic heat applications. It showed further that all

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the 50/50% biocoal briquettes of the two coal samples are good as fuel. All the 50/50% biocoal briquettes compositions have better characteristics than other blends while Ogboyaga, $OG_{90/10}$ biocoal briquette exhibits the best quality with the highest calorific value of 29.55 MJ/kg.

Keywords: Sawdust; coal; biocoal; moisture; volatile; carbon; hydrogen; sulphur.

1. INTRODUCTION

Coal occurs in several areas in Nigeria and ranges from bituminous to lignite; it is one of the abundant fossil fuel resources. There are many coal deposits in Nigeria whose fuel characteristics are unknown. Coals of different varieties and their characterization are very vital in deciding their suitable applications. The quest by nations to produce efficient and reliable energy for manufacturing and domestic uses has reawakened interest in coal and how it can be used without its harmful environmental impact and its accompanying health hazards. Densification of coal for making solid fuels has been in practice in many countries [1-3], however, with the advancement in technology, it has been proved that blending coal with biomass gives rise to briquettes which have better fuel properties and less harmful emissions compared to raw coal briquettes [1]. Nigeria coal deposits have not been maximally utilized due to availability of other sources of energy resources such as petroleum and natural gas but attention is now being shifted to some of these deposits which are now being investigated for conversion into coal briquettes as well as biocoal briquettes [4].

A Biocoal briquette is a product of the densification of biomass with coal and the addition of desulphurizer; it is agglomerated by compacting pulverized coal, biomass, binder and a desulphurizer [5]. The high pressure involved in the process ensures that the coal and the biomass materials bind together which eases transportation and storage. Biocoal briquette technology combines irrenewability in coal and renewable biomass as synthetic energy source [4]. The presence of desulphurizer ensures that most of the sulphur content of the coal is fixed into the ash instead of being liberated into the atmosphere as sulphur dioxide [6]. The desulphurizer reacts with the sulphur content of coal to fix its larger percentage between 60-80% into ash [6]. With this technology, the pollution problems associated with coal burning is reduced considerably. Nigeria has the ability to use biocoal technology in addressing some of its energy problem especially the rural household energy requirements by blending coal with sawdust [7].

This research is aimed at investigating the properties of biocoal briquettes for varying proportions of sawdust with the aim of determining the optimum composition using Nigeria's Ogboyaga and Okaba coal deposits.

2. MATERIALS AND METHODS

2.1 Sources and Preparation of Materials

Coal samples were collected from coal mine site s of Ogboyoga - Odu and Okaba in Kogi State in North Central Nigeria. Sawdust was collected from a saw mill in Akure, South-west Nigeria while starch was purchased from an open market. Calcium hydroxide was bought and used as desulphurizer. The samples were dried and the coal samples were pulverized and sieved using 0.25 mm mesh size.

2.2 Proximate and Ultimate Analysis of Materials

Proximate and ultimate analyses of the coal, sawdust and biocoal briquettes were conducted in accordance with American Society of Testing and Materials (ASTM). The calorific value was determined using Ballistic Bomb Calorimeter.

2.2.1 Moisture content

The moisture content was determined based on American Society for Testing and Materials Standards (ASTM- D3173). 1 g of the sample was weighed into a previously weighed crucible. The crucible plus sample was placed in cold muffle furnace and heated to 104°C for one hour to evaporate the moisture. The crucible was then cooled in a desiccator and weighed. The percentage moisture was calculated as percentage weight loss moisture content.

2.2.2 Volatile matter

The volatile matter was determined based on American Society for Testing and Materials

Standards (ASTM- D3175). 1 g moisture free sample was weighed into a tarred crucible, covered with the lid; and the crucible and its content placed in the muffled furnace and heated to 925°C. It was removed after exactly 7 minutes residence in the hot zone of the furnace just before attaining the ignition temperature and was then cooled in a desiccator. The crucible with its content was weighed and expressed as the percentage weight loss volatile matter.

2.2.3 Ash content

The ash content was determined based on American Society for Testing and Materials Standards (ASTM- D3174). 1 g of the sample was weighed into a clean crucible. The uncovered crucible was placed in cold muffle furnace and heated gradually so that the temperature increased to 500°C in one hour and 750°C in two hours. The crucible was thereafter cooled and covered in a desiccator and weighed. The percentage ash content was then calculated.

2.2.4 Fixed carbon

After the ash was separated, what was left was carbon; percentage fixed carbon was then calculated by subtracting the sum of volatile matter, moisture content and ash from 100 [8].

2.3 Preparation of the Briquette Samples

The two coal samples were milled preparatory to briquetting and labelled while the sawdust sample was screened of impurities like sand, metallic objects and chips of wood by passing through 10 mm sieve size. Six samples each from the two coal samples were blended with sawdust at various mixing proportions of 0:100, 10:90, 20:80, 30:70, 40:60 and 50:50 and 100:0 sawdust, 5% $Ca(OH)_2$ based on the mass of the coal was added for desulphurization and 10% cassava starch gel based on the entire mass of the mixture was used as binder for all the samples as adopted by [4]. The samples were weighed using digital weighing balance with maximum load of 600 g and accuracy of 0.1 g. The different concentrations were loaded into the mould compartment of the manually operated hydraulic briquetting machine. A maximum of 16 briquettes were obtained at each operation of the machine under a total load of 57.4 N. Biocoal briquettes of different mixing proportion as well as pure coal and sawdust briquettes were produced under this condition while maintaining the pressure at 6.89 MPa throughout production.

2.4 Proximate Analysis

The proximate analysis which involved the determination of moisture content, volatile matter and ash were determined based on the American Society of Testing and Materials (ASTM) D3173, D3175 and D3174 standards. The fixed carbon was obtained by subtracting the percentage of ash, moisture content and volatile matter from 100, Nag, [9].

2.5 Calorific Value Determination

The calorific value was determined based on ASTM D5865 standards. The Ballistic Bomb Calorimeter was first calibrated using a standard sample of benzoic acid whose known calorific value is 6.32 kCal/g. A known mass of sample of small quantity, 0.5 g of the different samples were placed in the crucibles. The bomb body was screwed in position and the thermocouple wire was plugged into the bomb body. The pressure release valve was closed and oxygen was admitted into the bomb until the pressure rose to 25 bars. The firing knob was depressed and released to fire the bomb. Heat was released and the maximum deflection of galvanometer scale was recorded. The maximum deflection obtained in the galvanometer was converted to energy value of the sample material by comparing the rise in galvanometer deflection with that obtained when a sample of known calorific value of benzoic acid is combusted as shown in equation:

Heat released from sample,

$$Q = \frac{G.meter\ deflection \times calibration}{Original\ weight\ of\ sample}$$

This is equal to:

$$Q = \frac{(\theta_3 - \theta_1)\,\gamma}{Z}\ \text{kcal/g} \tag{1}$$

Galvanometer deflection without sample, θ_1
Galvanometer deflection with sample, = θ_3
Mass of sample = Z g
Calibration constant = γ

The whole experiment was repeated for all the different samples of coal, biocoal and sawdust briquette samples.

2.6 Ultimate Analysis

Ultimate analysis which involves the determination of carbon and hydrogen, nitrogen, sulphur and ash were also determined based on the American Society of Testing and Materials (ASTM) D3178, D3179 and D3177 standards. The oxygen was obtained by subtracting the percentage of ash, carbon, hydrogen, nitrogen and sulphur from 100(2002).

2.7 Determination of Carbon and Hydrogen by Liebig Method

The carbon and hydrogen contents value were determined based on standards (ASTM- D3178) standards. 2 g of sample was placed in a platinum crucible and put into the combustion tube where it was burnt in oxygen at the temperature of 1300°C. The combustion product flow over a heated copper oxide and lead chromate and into the absorption train. The copper oxide ensures the complete combustion of the carbon and hydrogen in the coal, whereas the lead chromate absorbs the oxides of sulphur. The pre weighed absorbers in the absorption train absorbed water and carbon dioxide, and the percentage of carbon and hydrogen in the sample was calculated from the gain in weight of absorbers. The carbon as carbon dioxide and hydrogen as water were calculated from the increase in weight of the absorbents which was used to collect the water and carbon dioxide. An oxide of sulphur which was released in significant amount was removed from the combustion gases by passage over silver at about 600°C while nitrogen dioxide was removed by lead chromate.

2.8 Determination of Nitrogen Content by Kjeldahl Method

The nitrogen content was determined using ASTM- D3179 standard. 2 g of sample was weighed into a Kjeldahl digestion flask. 20 ml sulphuric acid and 1 g each of copper sulphate and potassium sulphate as catalysts were added into the flask. The flask was heated gently until boiling; the mixture was then diluted with 100 ml of distilled water and allowed to cool. The flask was then connected to the Kjeldahl distillation apparatus and sodium hydroxide solution was added to the mixture and then heated to boiling. The ammonia gas was condensed into the receiving flask containing 2% boric acid. Bromocresol green and methyl red indicators were added dropwise and alkaline distillate was titrated against 0.1 M hydrochloric acid. The procedure was repeated for the 13 samples and the percentage of nitrogen was calculated as shown in equation (2):

Percentage Nitrogen =

$$\frac{[(VH_2SO_4 \times NH_2SO_4) - (VBK \times NNaOH) - (VNaOH \times NNaOH)]}{1.4007 \times W} \times 100 \quad (2)$$

where:

VH_2SO_4 = mL standard H_2SO_4 pipetted into flask for sample
VNaOH = mL standard NaOH used to titrate sample
N H_2SO_4 = Normality of H_2SO_4
N NaOH = Normality of NaOH
VBK = mL standard NaOH used to titrate 1ml standard H_2SO_4 minus B
B = mL standard NaOH used to titrate reagent blank distilled into H_2SO_4
1.4007 = milliequivalent weight of nitrogen x 100
W = sample weight

2.9 Determination of Sulphur by the Eschka Method

Sulphur content was determined using ASTM-D 3177 standard. 1.00 g weight of coal sample was put into a porcelain crucible and mixed with 3.00 g of Eschka mixture. The mixture was then covered with 1.00 g of Eschka mixture. The crucibles were then put in a cold muffle furnace and heated gradually to 800°C for 60 minutes. Digestion was carried out in hot water for 45 minutes with intermittent stirring. The solution in each beaker was then decanted through a no. 540 watman filter paper into a 400 ml beaker. Three drops of methyl orange indicator were added dropwise until colour turned just neutral. Then, 1 ml of hydrochloric acid was added, after which 25 ml of potassium sulphate solution was also added.

The sample was thereafter heated to boiling and 10 ml of 10% barium chloride solution was gradually added while stirring. The solution was boiled for 30 minutes and filtered with no. 42 Watman filter paper after it has cooled down. The trapped residue was washed thoroughly with hot water. The total sulphur content was calculated using the formula shown in equation below:

$$\text{Total sulphur} = \frac{A - B \times 13.738}{C} \quad (3)$$

where:

A = the mass of barium sulphate from the sample

B = The mass of barium sulphate from the blank
C = The mass of sample

2.10 Determination of Oxygen Content

In practice, in the expression of the ultimate analysis is done by deducting from hundred the sum of the percentages of ash, carbon, hydrogen, nitrogen, sulphur, moisture and ash, hence, the oxygen was determined by the difference [8] as shown in equation below:

$$\%Oxygen = (Carbon + Hydrogen + Nitogen + Sulphur + Moisture + Ash)\ \% \quad (4)$$

3. RESULTS AND DISCUSSION

3.1 Result

The results from the proximate and ultimate analyses of the raw materials are presented in Tables 1 and 2. Figs. 1 and 2 show the relation ship between blending and briquetting on the proximate analysis of coal and biocoal briquettes respectively. Fig. 3 shows the effect of briquetting on the proximate analysis of sawdust and Figs. 4 and 5 show the relationship between blending and briquetting on the ultimate analysis of pure coal and the biocoal of Ogboyaga and Okaba coal deposits. Fig. 6 shows the effect of briquetting on the ultimate analysis of sawdust, Figs. 7 and 8 show the effect of blending and briquetting on the calorific value of coal and biocoal briquettes.

3.2 Discussion

3.2.1 Effect of proximate analysis on the samples

The proximate analysis of the raw materials is shown in Table 1. It was observed that Okaba coal has higher fixed carbon content of 58.13% as compared to 54.33% for Ogboyaga. In Figs. 1 and 2, the fixed carbon of the samples decreased from 100% coal briquettes compositions to 50/50% biocoal briquettes compositions. The fixed carbons are substantially higher than that of 21% sawdust briquette in Fig. 3. The reduction in fixed carbon may be due to the addition of biomass material which is lower in fixed carbon compared to coal. Fixed carbon is a measure of the solid combustible material in solid fuel after the expulsion of volatile matter; its content is used as an estimate of the amount of coke that will be obtained on carbonization [10]. The fixed carbon content of all the biocoal briquettes are higher than that of sawdust briquette which makes them better as solid fuel in comparison to sawdust.

In the case of moisture content, Okaba has 5.99% as compared to 6.93% Ogboyaga while sawdust has 8.28% as compared to the two samples. In Figs. 1 and 2, the moisture contents range between 6.07% and 8.73% with a marginal increment amongst the blends which progressed from the 100% coal briquettes to 50/50% biocoal in all the compositions. The marginal increment may be due to the blending of biomass and addition of starch as binder which added to the moisture content of the biocoal briquettes. Moisture content is undesirable constituent because it reduces calorific value and its weight adds to the transportation cost of solid fuel. However, the percentages present in these biocoal briquettes are low enough not to have serious negative impact on the combustibility of the samples when used for domestic heat applications.

Table 1 shows Ogboyaga coal has higher ash content of 8.63% as compared to Okaba with 3.32%. In Figs. 1 and 2, it was observed that the ash content increased from the 100% coal briquettes to the 50/50% biocoal briquettes. The progressive increment may be due to the addition of biomass, binder and desulpurizer which fixed some of the sulphur to ash. It may also be due to the loss of water from the carbonate mineral and oxidation of iron pyrite to oxides [8], a fuel with low ash is desirable as in these biocoal briquette samples.

Okaba has 32.56% volatile matter as compared to 30.41% of Ogboyaga. In Fig. 1, the volatile matter decreased relatively from the 100% coal briquettes to 50/50% bicoal briquettes in the two deposits. The quantity of volatile matter determines whether the coal will burn with good flame and whether it will produce smoke, a coal with high volatility will produce more smoke. Hence, the biocoal briquettes are all expected to generate less smoke and burn with high flame since they have less volatile matters than 100% coal briquettes.

3.2.2 Effect of ultimate analysis on the samples

Table 2 shows the ultimate analysis of the raw materials. It can be seen that the carbon contents are substantially high in all the two deposits with 82.8% of Okaba and 78.9% of Ogboyaga while sawdust has 1.48%. This is in agreement with Nag [9] that a good coal sample should have high amount of carbon. The higher the carbon content, the higher is the calorific value and the better is the quality of the coal. In Figs. 4 and 5, the carbon content decreased relatively from 100% coal briquettes to 50/50% biocoal in the samples while sawdust has 10.5% as shown in Fig. 6. Since carbon is one of the major combustible constituents of coal, it then follows that these biocoal briquettes have higher combustible carbon constituents than sawdust briquette although lower in comparison to coal briquettes. The carbon contents of the biocoal briquettes are high enough to be good fuel for domestic heat applications.

In Table 2, Okaba deposit has 4.3% hydrogen content while Ogboyaga has 4.1%. In Figs. 4 and 5, it is shown that the hydrogen decreased in all the biocoal briquettes as compared to 100% coal briquettes with Ogboyaga, $OG_{60/40}$ having 3.80% hydrogen and $OG_{90/10}$ having 3.81% while sawdust briquettes has 2.13%. Hydrogen is mos tly associated with volatile matter [11], hence, a good coal sample should have low amount of hydrogen as in these biocoal briquettes hence, the available low hydrogen in the right proportion will enhance the combustibility of the samples, this makes the biocoal briquettes a good fuel.

Table 1. Proximate analysis of the raw materials

Samples	Percentage moisture content	Percentage volatile matter	Percentage fixed carbon	Percentage ash content	Calorific value (MJ/kg)	Coal rank
Ogboyaga (Odu) coal	6.93	30.41	54.33	8.63	32.51	Sub- bituminous
Okaba coal	5.99	32.56	58.13	3.32	32.93	Sub- bituminous
Sawdust	8.26	70.1	21.03	0.68	16.68	N/A

Table 2. Ultimate analysis of the raw materials

Samples	Percentage hydrogen	Percentage carbon	Percentage nitrogen	Percentage oxygen	Percentage sulphur	Percentage ash
Ogboyaga - Odu coal	4.1	78.9	1.2	6.6	0.6	8.63
Okaba coal	4.3	82.8	2.4	7.3	0.6	3.32
Sawdust	2.13	1.48	13.2	82.51	0	0.68

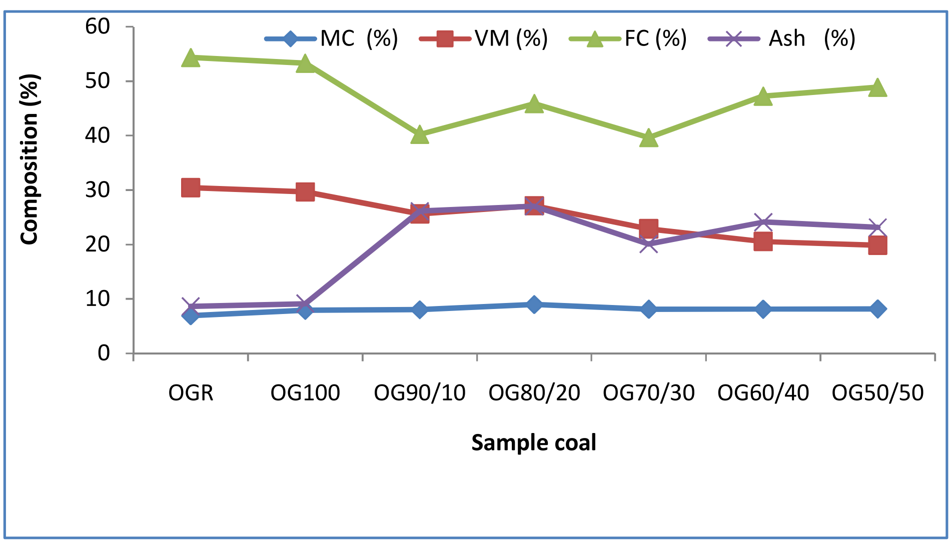

Fig. 1. The relationship between blending and briquetting on the proximate analysis of Ogboyaga deposit

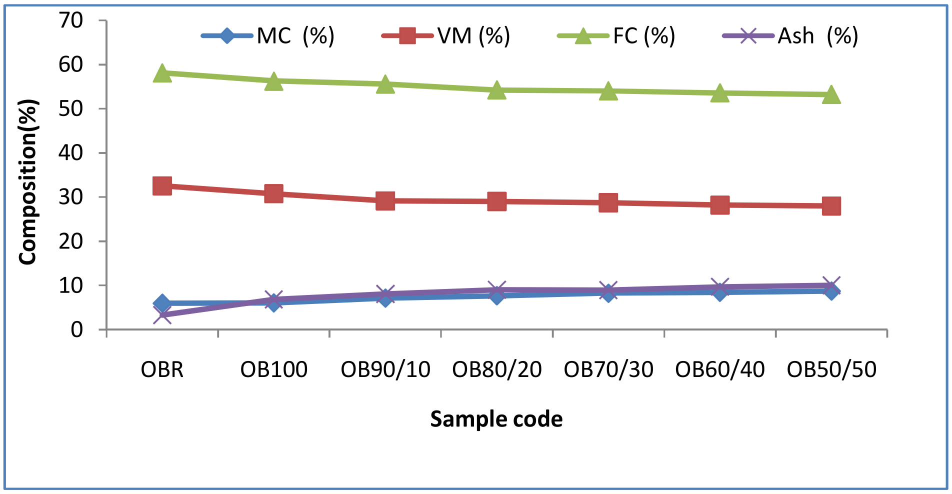

Fig. 2. The relationship between blending and briquetting on the proximate analysis of Okaba deposit

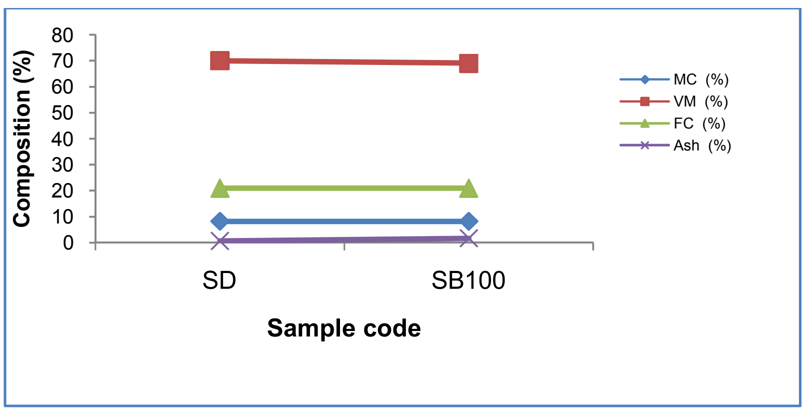

Fig. 3. The relationship of briquetting on the proximate analysis of sawdust briquette

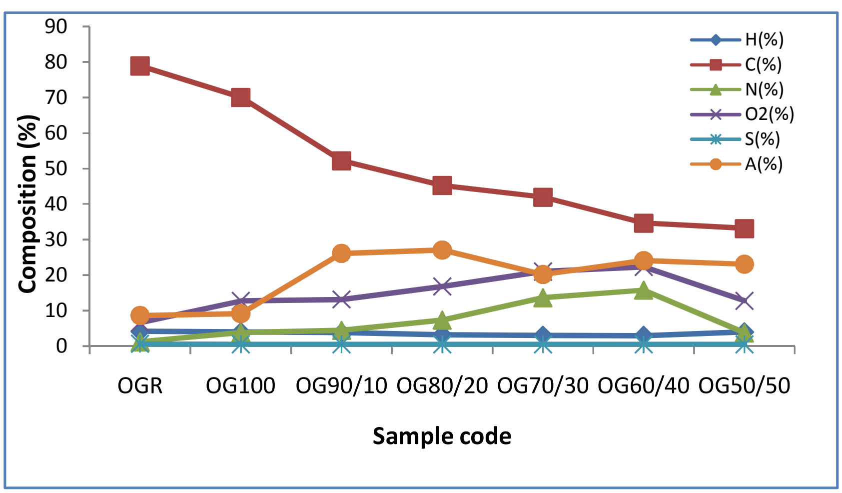

Fig. 4. The relationship between blending and briquetting on the ultimate analysis of Ogboyaga (Odu) deposit

In Table 2, Okaba deposit has 2.4% nitrogen and 1.3% for Ogboyaga. Figs. 4 and 5 show that the nitrogen content increased relatively in the all biocoal briquettes while the $OB_{50/50}$ biocoal briquette has the highest nitrogen content of 25% while the OG_{100} and $OG_{50/50}$ have 3.74% each. The slight increase in nitrogen content may be due to the biomass blend which contains more plant materials that have plant alkaloids, chlorophyll and other porphyrins containing nitrogen in cyclic structures. It can be said that it also draws nitrogen from the air in which it burns during combustion [8]. Nag [9] affirmed that nitrogen has no calorific value but low nitrogen is required in coal because it reduces oxidation, hence the low nitrogen in the biocoal as compared to 100% coal briquettes will reduce oxidation.

Table 2 shows that the two coal deposits have low oxygen content with Ogboyaga having 6.6% while Okaba has 7.30% and 82.5% of sawdust. In Figs. 4 and 5, it is shown that the oxygen content increased relatively in 100% coal briquettes to 50/50% biocoal briquettes in all the compositions with increase in sawdust concentration. The oxygen content of 28.45% for $OB_{50/50}$ coal briquette is the highest as compared with that of $OG_{50/50}$ biocoal briquettes put at 12.72%. The increase may be due to the blend of sawdust which is a plant material that has higher oxygen content than coal. This is significant because the more the oxygen content in the solid fuel, the better is its combustibility [9]. Hence, the biocoal will be easier to burn than pure coal briquettes.

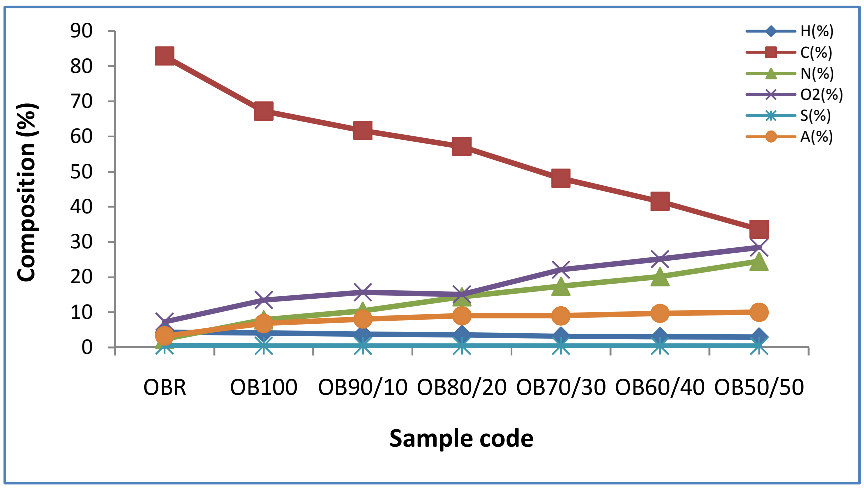

Fig. 5. The relationship between blending/briquetting on the ultimate analysis of Okaba deposit

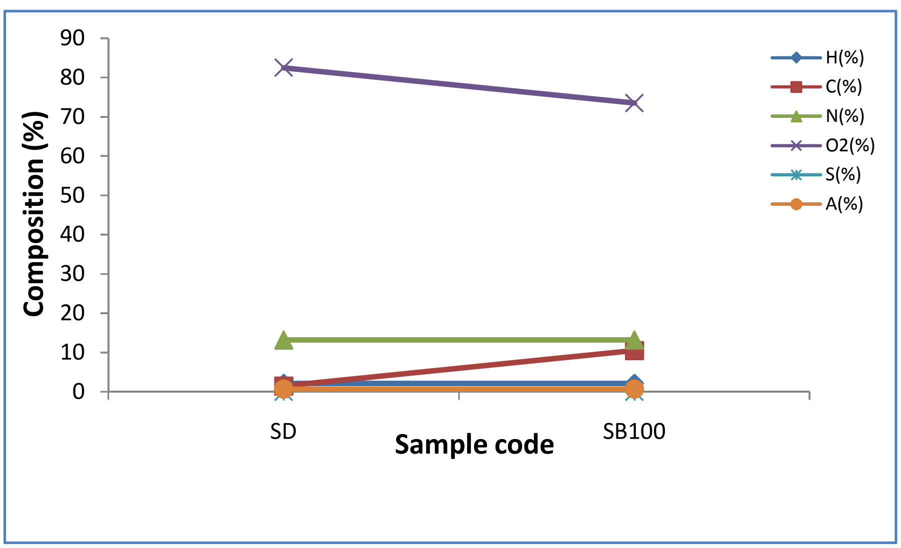

Fig. 6. The relationship of briquetting on the ultimate analysis of sawdust briquette

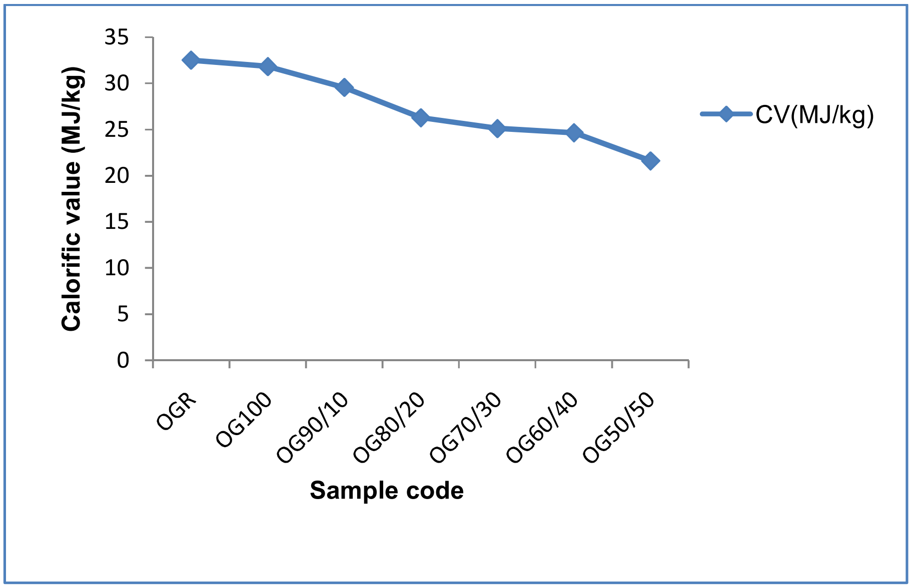

Fig. 7. The effect of blending/briquetting on the calorific value of Ogboyaga deposit

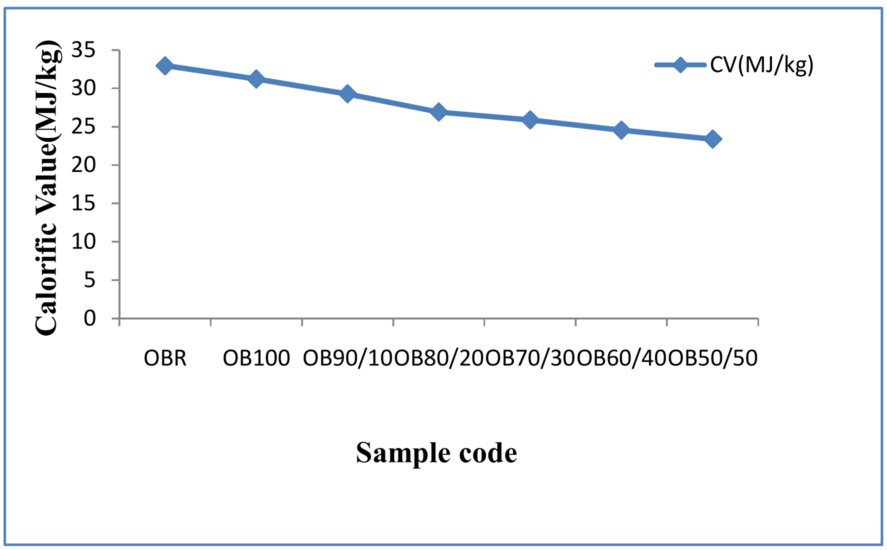

Fig. 8. The effect of blending/briquetting on the calorific value of Okaba deposit

In Table 2, the sulphur content of Ogboyaga and Okaba deposits are put at 0.6% each. In Figs. 4 and 5, the sulphur content decreased relatively with increase in sawdust concentration in all the compositions. The addition of calcium hydroxide used as a desulphurizer further reduced the sulphur content in all the coal and biocoal briquettes in Figs. 4 and 5 to between 0.47 and 0.48 on one hand, Ogboyaga, 0.6% to 0.46% and Okaba, 0.6% to 0.47% on the other hand. Sulphur is one of the major undesirable elements in coal even though it contributes to the heating value on combustion, it produces acids of sulphur dioxide and sulphur trioxide which corrodes combustion equipment and also cause atmospheric pollution. Since the sulphur in the biocoal briquettes are low as compared to the raw coal and coal briquettes, it will be better as fuel because it will emit less sulphur dioxide to the atmosphere.

3.2.3 Effect of blending and briquetting on the calorific values of biocoal briquettes

In Table 1, Okaba deposit has calorific value of 32.93 MJ/kg while Ogboyaga has 32.51. MJ/kg.

In Figs. 7 and 8, it can be observed that the calorific value decreased with increase in sawdust concentration from 100% coal briquettes to 50/50% biocoal briquettes in the two samples. The $OG_{90/10}$ biocoal has the highest calorific value of 29.55 MJ/kg while $OG_{50/50}$ has the lowest at 21.61 MJ/kg among the compositions. Even though the calorific value of the biocoal decreased with increase in sawdust concentration, they are still substantially higher compared to 17.68 MJ/kg for SB_{100} sawdust briquette, with 17.68 MJ/kg and comparable with net calorific value of kerosene put at 37 MJ/kg and wood at 14.6 MJ/kg [7] used for domestic heat applications. The decrease in calorific value may be due to the blending of biomass and the addition of calcium hydroxide, a non-combustible substance and the starch used as binder which does not contribute to the total heat value released.

4. CONCLUSION

This study revealed that biocoal briquettes from Okaba and Ogboyaga coal mines are suitable for the production of environmentally solid fuel that can be used for domestic heat applications. It showed further that all the 50/50% coal and sawdust blends of the two coal samples are good as fuel while Ogboyaga with 90/10% coal and sawdust blend may be better in terms of having the highest calorific value.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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