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**6 authors**, including:

Mohammed UMAR Garba
Federal University of Technology Minna


Umaru Musa
University of Nottingham


Kariim Ishaq
Federal University of Technology Minna


Usman Shehu Onoduku
Federal University of Technology Minna

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# CHARACTERIZATION AND ASH CHEMISTRY OF SELECTED NIGERIAN COALS FOR SOLID FUEL COMBUSTION

*Mohammed. U. Garba[1*], Umaru Musa[1], Priscilla. E. Azare[1], Kariim Ishaq[2], Usman. S. Onoduku[3] and Yahaya S. Mohammad[4]*

[1] *Chemical Engineering Department, Federal University of Technology, Minna, Niger State, P.M.B. 65, Nigeria*
[2] *Center for Genetic Engineering and Biotechnology, Federal University of Technology Minna, P.M.B. 65, Niger State, Nigeria*
[3] *Geology Department Federal University of Technology, Minna, Niger State, P. M. B. 65, Nigeria*
[4] *Water Resources and Environmental Engineering Ahmadu Bello University, Zaria, P. M. B. 65, Nigeria*

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**Abstract**

In this paper, characterization and ash chemistry of selected Nigeria coal samples were investigated to determine its suitability as a solid fuel. The three coal samples considered originated from Northern Benue trough, Central Benue trough and Anambra Basin of Nigeria where proven reserve deposits are found. The coal samples were analysed using various analytical methods such as thermosgravimetric analysis (TGA) and X-ray Fluorescence spectroscopy (XRF) analyses. The ash chemistry indices used in predicting the performance of the coal samples includes basic to acidic oxide (B/A), silica ratio, iron index and sulphur slagging index (Rs). The TGA profile suggests that Enugu coal showed high thermal stability than that of Okaba and Maiganga coals but had an ash content higher by a factor of 2.0 when compared with Okaba and Maiganga coals. Maiganga coal however has the highest heating value of 27.40 MJ/kg when compared to Okaba coal (25.74 MJ/kg) and Enugu coal (22.92 MJ/kg). The silica ratio indicated that Enugu coal has high slag volume, while the B/A suggest that all the coals were found to be less than 0.6 indicating low slagging potential. The comparison of the ash chemistry suggests that Maiganga coal has lowest slagging potential while Enugu coal highest slagging potential.

***Keywords:*** Coal; fuel; thermogravimetric; combustion; ash chemistry; slagging.

## 1. Introduction

Coal is expected to be the dominant source for power generation globally and shall remain relevant in next few decades to come [1]. Nigeria is endowed with a large coal deposits most of which are reported to be within the Anambra Basin and Benue trough [2-3]. Despite the abundant coal reserve of over 2.6 billion tonnes predominantly sub-bituminous coal type [1]. Nigerian current power generation of about 5000 MW of electricity is grossly inadequate. This situation has adversely affected the domestic and industrial electricity supply thereby hindering socio-growth of the nation [1]. This situation account for why only about 40 % of the estimated population (approximate 170 million people) of the Nigerians are connected to the national grid [1].

Nigeria is endowed with a large coal deposits most of which are reported to be within the Anambra Basin and Benue trough [2-3]. Anambra Basin is the largest coal producing basin where intensive exploration and exploitation activities have been reported while new discovered coal deposit exist. Coals are of divers in nature, and the individual characterizations are very important in deciding their suitable applications [4-5].The contemporary global concern for a clean source of energy has rekindle interest in coal. Large coal deposits which have been abandoned due to the availability of petroleum are now being investigated for their clean coal technology [6].

Coals are of divers in nature, and the individual characterizations are very important in establishing their relevant domestic and industrial applications [4-5]. The contemporary global concern for a clean source of energy has rekindle interest in coal. Large coal deposits which have been abandoned due to the availability of petroleum are now being investigated for their clean coal technology [6]. Hence, the undeniable need to utilize the abundant and neglected coal resources in Nigeria for massive power generation to cater for the nations populace demand. According to Garba *et al.* [7] coal power plants are characterized with high energy capacity and pose minimal threat to environment if properly managed. A number of literatures have been documented on coal combustion [7], coal liquefaction [8,11] and coke making [9]. Most Nigerian coal has been reported to be non-coking except for Obi-Lafia coal [10]. Nearly in all studied reported on Nigeria coals, the evaluation of combustion properties have been limited to the determination of proximate, ultimate and calorific values [12]. Research has shown that coal combustibility, calorific values and ash chemistry are genetically linked to one another and principally depends on the coal rank and maceral composition.

Over the years, ash chemistry has been used to predict the performance of coals prior to actual combustion. Majority of the studies on ash chemistries relate the chemical composition of the coal ash to the slagging tendency (B/A). The slagging tendency is describe by numerical indices which are capable of predicting the severity of slagging. These indices include the ratio of the basic metal oxides to the acidic metal oxides (B/A), sulphur slagging index (Rs), iron index and silica ratio. B/A index is an important index for determining the melting behaviour of coal ash systems and distinguishing between good and bad coals prior to actual combustion. Bad coals are said to have a low silicon oxide level of equal and less than 87 % or a high level of iron oxide, above 6% [13-14]. Lawrence *et al*. [14] reported that a decrease in the B/A of coal raise its fusibility and hence decreases its slagging potential, the author stated that good coals have B/A ≤ 0.11. An empirical slagging index (product of B/A and the dry sulphur content) of coal was developed and tested on United State coals due to the presence of iron sulphide ($FeS_2$) as pyrite in the coal samples. But in low sulphur coals, where there is strong correlation between iron and carbonate ($FeCO_3$) as in the case of Australian coals, slagging index is better defined as the product of B/A and $Fe_2O_3$. According to McLennan *et al.* [13], if the sulphur slagging index (Rs) of a coal, assessed as the product of B/A and dry S is greater than 0.6, the coal is said to be of high slagging potential. Other slagging indices include the silica ratio and the iron oxide. However, these indices have been established based on particular coal properties, and therefore, can be used to assess Nigerian coals.

There are only a few experimental investigations on the combustion profile of Nigerian coals and investigation their slagging potentials are even rarer. An in-depth understanding of the characterization and ash chemistry of the Nigerian coals is necessary in order to assess indigenous coal combustion efficiency. In this paper, characterization and ash chemistry of selected Nigerian coals were carried out. The selection of coal sample was done in view of disparity of coal properties from different geographical locations as coal formation largely depends on constituent's formation, compression temperature and pressure.

## 2. Materials and method

*Coal Samples preparation*: The coal samples used were sourced from Maiganga (Northern Benue trough), Okaba (Central Benue trough) and Enugu (Anambra Basin) respectively. All samples were initially crushed and pulverized. The coal samples were sieved to a particle size of 150 micrometer in preparation for further analysis.

### 2.1. Proximate and ultimate analysis

The moisture, volatile matter and ash contents were determined based on American Society for Testing and Materials D3172-13. The fixed carbon (dry basis) in the coal samples was obtained by subtracting the percentages of moisture, volatile matter and ash from 100. The ultimate analysis (sulphur, nitrogen, carbon and hydrogen) were also based on D3176-15. The High heating value (HHV) of coal was calculated using the correlation by Sami *et al.* [15].

$$HHV = 0.3491C + 1.1783H + 0.1005S - 0.1034O - 0.0151N - 0.0211A \text{ (MJ/Kg)} \quad (1)$$

where; C = carbon, H = hydrogen, S = sulphur, O = oxygen N = nitrogen and A = ash

## 2.2. Thermal and chemical characterization

*Thermal Characterization*: The thermal degradation of the coal samples was performed using thermogravimetric analyser (Perkin ElmerTGA-4000). The samples were analyzed in pure nitrogen environment with a nitrogen flow rate of 20 ml/min, pressure of about 2.5 bars and a constant heating rate at 10ºC/min for a temperature range of 23ºC to 950ºC. Five (5) gram of the coal samples each of particle size less than 150µm was accurately weighed and placed on to a clean crucible. The coal sample was heated until a constant weight was observed. The Pyris manager software was used to plot the degradation profile.

*Chemical Characterization:* The ash compositions of the samples were carried out using X-ray fluorescence (XRF). The sample was placed in a sample holder contained in the XRF equipment and oriented to an angle of 45°. The X-ray machine was closed and the window of the X-ray tube was opened via the shutter. The filament voltage was then set progressively to 40 kV and the current to 20 mA. A silicon drift detector was used to detect the secondary X-rays (X-ray detector) and to record the spectrum (acquisition and processing system). The each element was displayed in their oxide forms.

## 2.3. Ash chemistry prediction

Following ASTM D3174-12, the samples were heated at 800ºC for 5 hours and cooled. The oxide composition of the ash samples were analyzed by XRF and were then used as input into silica ratio, basic to acidic oxide [13-14], iron percentage of iron index [14] and sulphur index in Equations (2), (3), (4) and (5) respectively.

$$S_R = 100\left(\frac{SiO_2}{SiO_2 + Fe_2O_3 + CaO + MgO}\right) \quad (2)$$

$$B/A = \frac{Fe_2O_3 + CaO + MgO + NaO + K_2O}{SiO + Al_2O_3 + TiO_2} \quad (3)$$

$$Iron\,Index = \%Fe_2O_3 \quad (4)$$

$$R_S = \left(\frac{B}{A}\right) \times S \quad (5)$$

# 3. Results and discussion

## 3.1. Proximate and ultimate analyses

Table 1 displays the result obtained from the proximate analysis carried out on the three (3) coal samples (Okaba, Maiganga and Enugu samples).

*Moisture content:* The result of moisture content of coal showed that the moisture contents ranges from 5.17 % to 6.200 %. Enugu coal has the highest moisture content (6.2%) followed by Okaba coal (5.44%) and then Maiganga coal sample (5.17%) respectively. These values revealed that the three coal samples are of low moisture content. The low values obtained are within the range considered for good coking coals [9]. Low moisture content is an indication that the coal is of a high rank and good quality, possibly the rank of bituminous grade [15]. Low moisture content also represents a significant improvement in coal's quality because moisture affects the calorific value, the concentration of other constituents, decreases system capacity and increases operational cost. The moisture content of coal depends on the degree of maturity [15]. Therefore Maiganga coal sample with the lowest moisture content (5.171%) may be the most matured, followed by Okaba and then Enugu coal samples.

*Ash Content:* The ash content of Enugu coal was 20.02 %, while ash contents of Maiganga and Okaba coals were 13.981 % and 10.719% respectively. These results agree with report of Fatoye *et al.* [12] who found that typical ash content of Nigerian coals are always within the range of 5-40 %. High ash content in coal are undesirable as its leads into lower fusion temperature which result to high slag volume and low blast furnace efficiency. The high ash content is also an indication of low degree of coalification and hence immaturity of the coal [16].

*Volatile Matter:* The analysis shows that the volatile matter of the coal samples was within 19.19-28.98 %. Maiganga, Okaba and Enugu coals are 19.19%, 21.54% and 28.98 % respectively. Enugu coal has the highest amount of volatile matter (28.98%) while Maiganga coal has the lowest amount (19.19 %). The volatile matter contents of coal one of the key parameters used in determining its applications [17]. The higher the volatile matter contents of a coal, the faster the ignition rate of such coal. High volatile matter contents coals are characterize with low ignition temperature and long smoky yellow flame during combustion [18]. Typically the percentage of volatiles matter contents of high-rank coals at 1300 K is less than 30% [15]. The values obtained in this study were appreciably within this reported value. The higher the volatile matter contents of a coal, the lower the rank of the coal. Although high volatile matter could generates high pressure during combustion which is detrimental to the boiler walls. The coals investigated in this study do not have these characteristics.

*Fixed Carbon Content (FC):* The fixed carbon content of a bituminous material is the solid residue other than ash obtained by destructive distillation. It is the carbon found in organic materials after volatile materials have been driven off [18-19]. The Fixed carbon content of coal also determines the rank and quality of a coal sample. The Fixed carbon contents of Maiganga, Okaba and Enugu coals were 61.66%, 54.86% and 52.65% respectively. The results indicate that the three coal samples are high carbon coals. High carbon content in coal is essential for coke making [18]. This finding suggests that Maiganga coal sample with the highest carbon content (61.66%) has more potential for coke formation, than Okaba followed by Enugu coal samples.

*Ultimate Analysis:* From the result of this analysis showed in Table 1. Enugu coal has the highest sulphur content of 1.82% while Okaba and Maiganga have a sulphur content of 0.89% and 0.58% respectively. Low sulphur content signifies that the solid fuel can be used to generate power with reduced emissions to the environment. The elemental carbon content of the three coal samples ranges between 53.27-59.23 % with Maiganga coal having the highest carbon content of 59.23 % while Okaba and Enugu coals have carbon content of 57.94% and 53.27% respectively. The high carbon content associated with these coal samples is an indication of high heating value. In terms of oxygen content in Table 1, Okaba coal had the highest oxygen content of 13.76% while Maiganga and Enugu coal had oxygen content of 12.07% and 12.43% respectively while the hydrogen content is within the range of 4.97 – 7.10 % with Maiganga coal having the highest (7.10%), followed by Enugu (4.97%) and Okaba (6.02%).

Table 1. Data obtained from the proximate and ultimate analyses of some Nigerian coal samples.

| Coal sample | Maiganga coal | Okaba coal | Enugu coal |
|---|---|---|---|
| | Proximate analysis | | |
| Moisture Content (%) | 5.17 | 5.442 | 6.200 |
| Ash Content (%) | 13.98 | 10.72 | 20.02 |
| Volatile Matter (%) | 19.19 | 21.54 | 28.98 |
| Fixed Carbon (%) | 61.66 | 54.86 | 52.65 |
| | Ultimate analysis, % | | |
| Nitrogen | 1.37 | 2.20 | 2.67 |
| Carbon | 59.23 | 57.94 | 53.27 |
| Hydrogen | 7.10 | 6.02 | 4.97 |
| Sulphur | 0.58 | 0.98 | 1.82 |
| Oxygen | 13.53 | 13.76 | 12.43 |
| | | | |
| HHV( MJ/kg) | 27.40 | 25.74 | 22.92 |

From the result of the calculated higher heating values (HHV) shown on Table 1, it was observed that Enugu coal had the lowest heating value of 22.92 MJ/kg. Maiganga coal showed the highest heating value of 27.40 MJ/kg then followed by Okaba coal with 25.74MJ/kg. The HHV of a good coal must not be less than 26 MJ/kg [15]. The results of investigation point out that Maiganga coal is considered a good coal and most appropriate coal for solid fuel combustion when compared to Okaba and Enugu coal.

### 3.2. Thermal analysis

The thermogram (TG) curves of Enugu, Okaba and Maiganga coals are shown in Figure 1. For Maiganga coal, about 53.89 % of the sample was left undecomposed at 823.55°C which is an indication that only about 46.109 % of the coal sample was decomposed. This further implied that 53.89 % of the sample that was left undecomposed consisted of certain materials such as clay, silt, and sand stone. For Okaba and Maiganga coals, about 46.923 % and 84.647 % of the sample were left undecomposed at 825.31 and 845.06°C indicating that only about 53.077% and 15.353% of the coal were decomposable respectively.

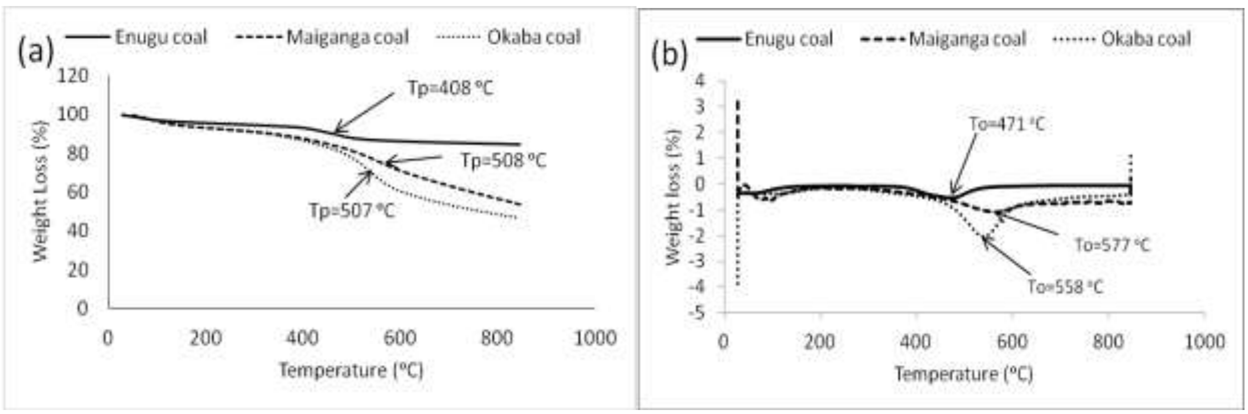


Figure 1. Thermal profile of the coal samples showing (a) TG and (b) DTG curves

In this first stage, Maiganga and Okaba TG curves showed visible steep fall approximately in the range of 28.26 -187.29°C (extracted from TGA and shown in Table 2) which can be described as a region where the drying of surface water occurred due to heat treatment. The process is endothermic in nature (heat is absorbed). During stage Maganga coal shows visible peak at a temperature of 96.96ºC due to the loss of surface water. In the case of Enugu coal a visible steep fall was observed between 28.99°C and 117.77ºC during drying of surface water.

Table 2. Thermal decomposition characteristics extracted from TGA

| Coal sample | Degradation temperature range (°C) | To (°C) | Tp (°C) |
|---|---|---|---|
| Maiganga | 432.53-643.33 | 508.22 | 577.28 |
| Okaba | 417.65-641.74 | 506.87 | 558.27 |
| Enugu | 239.75-686.75 | 407.57 | 470.83 |

During dehydration, the second stage is called the fairly constant region observed between 178.88°C and 417.65°C as shown in the TG in Figure 1(b). This probably depicts that no volatiles was released, hence the absence of decomposition peak in this region. The third stage occurs between degradation temperatures of 417.65ºC - 641.741°C. In this stage volatiles were released. From the DTG in Figure 1(b), decomposition peak describing mass loss was observed at a peak temperature of 558.27°C with a visible downward major peak, indicating devolatilization process where compounds containing carbon, hydrogen and oxygen were released. According to Crelling *et al.* [20] this stage is identified as a primary decomposition process which is followed by a downward fall. The onset temperature is the temperature that marks beginning of degradation. The onset temperature of Maiganga coal was observed to be the highest onset temperature (508.22°C) in contrast with Okaba and Enugu coal which were 506.87 ºC and 407.570 ºC respectively. These results show that Enugu coal was the earliest to decompose followed by Okaba coal and then Maiganga coal. The early response of Enugu coal to degradation can be attributed to presence of high volatile matter which are easily released at a low temperature of about 407.570°C, as against about 508.22°C and 502.27°C for Maiganga and Okaba coals. It can also be seen in Figure 1 that Enugu coal showed more thermal stability than either Okaba or Maiganga coal. The results of thermal degradation at 845.06°C revealed that Enugu coal had the highest residual matter after decomposition (84.647%) as against 53.891% and 46.923% for Maiganga and Okaba respectively.

Figure 1(b) shows the derivative of thermogravimetric (DTG) of coal samples. The result on this figure shows that Enugu has the highest the peak temperature follows a pattern as a function of carbon content [17]. The peak temperature is defined as the temperature at which nearly all the volatiles were removed. Amongst the coal samples, Maiganga coal had the highest peak temperature of 577.28°C indicating that it's the highest in rank between the three samples followed by Okaba and then Enugu coal with peak temperature of 558.27°C and 470.830°C respectively.

### 3.3. Chemical analysis

The results of the elemental oxide analysis of the coal ash deduced with the aid of XRF (X-ray fluorescence) are given in Table 3. It is evident from the results that there was variation in the distribution of various inorganic elemental oxide in the coal samples (Maiganga, Enugu and Okaba) analyzed. XRF analysis indicated that sulfur was present as compound $SO_3$. Contrary to what was expected, the analysis did not detect any presence of pyrite/marcasite ($FeS_2$). Those compounds are generally present in coals in variable amounts. Sulphur being the element of interest was seen to be occurring in large amount of 5.91% in Enugu coal then followed by 2.78% in Okaba coal and very little amount of 2.31% in Maiganga coal. Maiganga showed very acceptable level of sulphur content which is occurring in a minimal amount.

Table 3. Ash chemical composition of Maiganga, Okaba and Enugu coal samples.

| Oxides | Maiganga Coal | Okaba Coal | Enugu coal | Oxides | Maiganga Coal | Okaba Coal | Enugu coal |
|---|---|---|---|---|---|---|---|
| $TiO_2$ | 4.33 | 4.11 | 3.85 | $SO_3$ | 2.31 | 2.78 | 5.91 |
| $Al_2O_3$ | 23.6 | 26.02 | 28.12 | MnO | 1.72 | 2.58 | 2.08 |
| $SiO_2$ | 49.4 | 44.8 | 39.0 | $P_2O_5$ | 0.57 | 0.44 | 0.83 |
| $Fe_2O_3$ | 5.52 | 5.68 | 7.2 | CuO | 0.019 | 0.07 | 0.02 |
| CaO | 8.06 | 10.34 | 12.01 | ZnO | 0.210 | 0.95 | 0.10 |
| MgO | 2.4 | 2.7 | 3.4 | $Ag_2O$ | 0.550 | 0.39 | 0.45 |
| $Na_2O$ | 0.73 | 0.85 | 1.03 | BaO | 0.210 | 0.19 | 0.38 |
| $K_2O$ | 1.35 | 1.70 | 2.93 | | | | |

Silicon oxide occurred as the major element predominantly in all the coal samples. Maiganga coal had the highest amount of silicon oxide. More so, Fe, Ca, K and P in their oxide forms were found occurring as minor elements. These elements do not evaporate easily on combustion and share similar characteristics. Furthermore, CuO, ZnO, AgO and BaO occurred in only in minute traces in all the coal samples.

### 3.4. Ash chemistry of coal samples

The ash chemistry of the coal samples in Table 2 showed high levels of silicon and aluminium. The oxides of these elements constitute more than seventy percent except for Enugu coal. Iron oxide and calcium oxide are the dominate basic oxide in the ash of the coal samples. When titanium dioxide is added silicon and aluminium it is reported as acidic oxides. The basic oxides equal the sum of the percentages of iron, calcium, sodium, magnesium and potassium components. High content of silica results in a higher degree of covalence, and this behaviour yields a high temperature melting phase and hence a low deposition potential [21-22]. From Table 4 coal Maiganga coal has the silica ratio, followed by Okaba coal, the least is Enugu coal. Guided by the silica ratio, Maiganga and Okaba coals are expected to show low slagging propensities, while Enugu coal show high slagging propensities. Enugu coal has the highest percentage of alumina. It is believed that silicon combine with alumina alumina-silicate is formed which is known to increase slag volume [22]. A significantly higher potassium content in Enugu coal than the other of the samples ashes (Table 4) could be the reason for severe slagging [23]. During combustion potassium is released to the gas phase KOH and KCl. Transformation of KOH and $K_2SO_4$ can be carried out through the following global reactions [24].

Table 4. Summary of existing coal slagging indices employed and this study

| Expression | Slagging potential | | | | Coal samples in this study | | |
|---|---|---|---|---|---|---|---|
| | Low | Medium | High | Severe | Maiganga | Okaba | Enugu |
| *B/A* | <0.5 | 0.5< *B/A* <1 | 1 | ≥1.75 | 0.24 | 0.31 | 0.42 |
| $S_R$ | 72–80 | 65–72 | 50–65 | – | 76 | 69 | 59 |
| Iron Index | < 6 % | 6–7 % | > 7 % | – | 5.52 | 5.68 | 7.2 |
| *Rs*=(*B/A*)×*S* | < 0.6 | 0.6–2.0 | 2.0–2.6 | > 2.6 | 0.13 | 0.30 | 0.76 |

The content of iron is another important parameter in determining the ash slagging potential. High iron content usually lowers the melting point of slag [22], which results in a high slagging potential. Iron content lower than 6% in coal ash translate to a low slagging tendency [13]. The percentage of iron oxides for the three coal samples has been computed and the results obtained are also presented in Table 4. The results indicate that coal Maiganga coal has the lowest $Fe_2O_3$ content, followed by Okaba coal, then Enugu coal coal, and therefore, we expect their deposition potential to be in the same ascending order. Iron oxide is a low melting phase compound and act as a strong fluxing agent [13,22]. Where the iron index is high slagging of heating surface is controlled by pyrite behaviour which may react with clay and quartz to form alumino-silicate slags. The dissolution of iron into alumina-silicate reduces the melting point of fly ash in the combustion system [25].

The basic to acidic oxides ratio (B/A) of the coals also aids in describing good and bad coals prior to combustion. An decrease in the B/A of the coal will raise its fusion temperature. The calculated values for the B/A for three coals been investigated, are also shown Table 4. The results indicate that the slagging potential of Enugu coal is expected to be the worst, then Okaba coal, and Maiganga coal, in decreasing order. If a portion of the sulfur in coal exists with more pyrite than siderite B/A. B/A is multiplied by the percentage of dry sulphur in the coal in order to obtain Rs shown in Table 4. Coal with Rs > 0.6 is said to have high slagging propensities [22,26,27]. The slagging indices for coal samples are also presented in Table 4. The calculated results suggest that Maganga and Okaba have lower slagging potential than Enugu coal.

## 4. Conclusion

The different geographical locations as well as mode of occurrence coal constituents play a major role in coal formation. Hence three coals were selected from Northern Benue trough, Central Benue trough and Anambra Basin. The coal samples were first characterized and then combusted to ash samples. The thermal profiles suggest that Enugu has higher thermal stability and lower calorific value than Maiganga and Okaba coals. The ash chemistry indices used to predict the performance of the coal samples prior to combustion shows that silica ratio indicated that Enugu coal high slagging tendency, B/A suggest that all the coals were found to be less than 0.6 indicating low slagging potential. The ash chemistry suggests that Maiganga coal has low slagging potential while Enugu coal has severe slagging potential.

***Nomenclatures***

| | | | |
|---|---|---|---|
| *HHV* | *high heating value* | *B/A* | *basic to acidic oxides ratio* |
| *TGA* | *thermogravimetric analysis* | *Rs* | *sulphur slagging index* |
| *XRF* | *X-ray Fluorescence spectroscopy* | $S_R$ | *silica index* |

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** Corresponding author: umar.garba@futminna.edu.ng*