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PHYSICOCHEMICAL, MINERALOGICAL, AND THERMOGRAVIMETRIC PROPERTIES OF NEWLY DISCOVERED NIGERIAN COALS

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Abstract

Coal is an abundant and accessible energy source despite growing environmental concerns about its use. It is a critical driver of socio-economic growth and development particularly in nations with vast proven reserves. The discovery of new coal deposits in Nigeria has renewed interest in its utilisation. However, the scarcity of data has limited the energy utilization. Therefore, this study examines and reports data for the physicochemical, mineralogical, and morphological fuel properties of coals from Obomkpa (BMK), Ihioma (IHM) and Ogboligbo (OGB). The results revealed BMK contains the highest C and H, but OGB has the highest N, S, and O content. Morphology revealed coarse-grained coal particles with distinct lustre comprised of C, O, Si, Al, S, Fe, Ti, K, and Ca. Thermal analysis revealed IHM is the most reactive coal, whereas OGB is the least reactive. The DTG revealed two endothermic peaks for drying (30 – 200°C) and devolatilization (200°C to 600°C). The highest mass loss was observed in IHM, which confirmed its higher reactivity compared to BMK and OGB. The heating values were: 15.55 MJ/kg, 19.40 MJ/kg, and 19.66 MJ/kg for OGB, IHM, and BMK, respectively. Based on the findings, the coals are classified as lignite with potential for thermal energy recovery.

Keywords*: Fuel Characterization; Flash Pyrolysis; Lignite; Coal; Nigeria.*

1. Introduction

Despite global concerns about greenhouse gas emissions, coal remains the dominant fuel for electric power generation [1]. Currently, coal accounts for 40% of global power generation or about 64% of economically recoverable resources [2]. It provides an economical, reliable, and efficient source of energy for various industries around the globe [3]. Furthermore, the global importance of coal is augmented by its widespread accessibility and abundance [4]. As a result, coal plays a critical role in the socio-economic growth and sustainable development in emerging nations such as China, India and South Africa [5]. Therefore, the discovery of large coal deposits in Nigeria – Africa's largest economy and the most populous nation has renewed interest in the coal sector. However, coal does not currently contribute to the electric power production despite the nation's large coal deposits currently estimated at 2.8 billion tonnes [3].

Over the years, successive governments have made initiatives to revive the nation's moribund coal sector through policies reforms and strategic investments aimed at generating 30% of the nation's electricity from coal [6-7]. According to experts, coal utilisation for electric power generation is hampered by numerous socio-economic, environmental, and technological challenges [8-10]. The most notable technological challenges include lack of comprehensive data on coal such as its elemental, mineralogical and calorific properties [11-12]. The outlined coal properties are critical to the design, optimisation and scale-up of efficient conversion processes [13]. Furthermore, the assessment of coal properties is an important step in defining its rank, energy application, or potential emissions [14-16]. Such information is a crucial dynamic in debates on the future coal and its applications particularly in the age of global warming and climate change.

With this in focus, several studies have examined the fuel properties of various Nigerian coals. The study by Ryemshak and Jauro [17] examined the proximate, rheological properties and potential applications of selected coals from Garin Maiganga, Shankodi-Jangwa and Tai environs in Nigeria. Chukwu, Folayan [3] examined the fuel characteristics of selected Nigerian coals for power generation. The study examined the ultimate-proximate, calorific, petrograp-hic and thermal properties of Odagbo, Owukpa, Amansiodo, Ezimo, Inyi, Odagbo, and Owukpa coals from Nigeria. Similarly, Sonibare, Ehinola [18], examined the thermal and kinetic proper-ties of Agbogugu, Enugu, Chikila and Lamja coals under oxidative and non-oxidative thermos-gravimetry. Other studies have also examined the mineralogical, morphological and micro-structural properties of Nigerian coals in the literature [19-21]. However, there is an absence of comprehensive data on the fuel properties of many newly discovered Nigerian coals in the literature. These include the recent discoveries of coal deposits in Obomkpa, Ihioma, and Ogboligbo in Delta, Imo and Kogi States, respectively, in Nigeria. The new deposits are strategically located in three out of the six geopolitical zones in the country. This revelation presents significant prospects for electric power generation required for socio-economic growth and development in the country.

To the best of the authors' knowledge, there is limited knowledge on the fuel properties of the Obomkpa (BMK), Ihioma (IHM) and Ogboligbo (OGB) coals. Therefore, this study will examine the physicochemical, mineralogical, and thermogravimetric characteristics of the newly discovered coals.

2. Experimental

The three (3) coal samples; Obomkpa (BMK), Ihioma (IHM) and Ogboligbo (OGB) were acquired from the National Metallurgical Research and Development Centre (NMRDC) Jos in Plateau State Nigeria. The coal samples were originally sourced from the following coal fields: Obomkpa (BMK) in Aniocha-North Local Government Area of Delta State; Ihioma (IHM) in Orlu Local Government Area of Imo State; and Ogboligbo (OGB) in Igalamela-Odolu Local Government Area of Kogi State. The coal samples were crushed and pulverised in a dry miller and then sieved through the 250 μm analytic test sieve based on the procedure described in a previous study [22].

The pulverised coal samples were subjected to the physicochemical analyses; ultimate, proximate, and bomb calorimetry to determine the elemental composition, fuel properties and higher heating values. The ultimate analysis was done using the vario MICRO Cube™ Elemental Analyzer (Germany) based on ASTM D5291-16. The proximate analysis was performed through thermogravimetric analysis (TGA) according to the procedure described in the literature [23-24]. Lastly, the higher heating value (MJ/kg) was determined by bomb calorimetry using the IKA C2000 (USA) combustion calorimeter based on ASTM standard D2015. Each test was performed in duplicate to ensure the accuracy and reliability of the measurements.

The mineralogical, morphological and microstructural characterisation of the coal samples were done by Scanning Electron Microscopy/Electron Dispersive X-ray (SEM/EDX) analysis. The tests were performed on the JEOL-JSM IT 300 LV (Germany) Scanning Electron Microscope (SEM) equipped with an Energy Dispersive X-Ray (EDX) detector. For each test, microscopic amounts of the samples were sprinkled on the SEM/EDX carbon stubs. Next, the samples were sputter coated with platinum using the Quorum Q150R S Sputter coater for 10 minutes before SEM/EDX analysis. The analysis was performed at 20 kV and 5 mm working distance to obtain micrographs at magnifications of ×1000. The AZTEC EDX software from Oxford Instruments, UK was used to analyse the captured SEM images. The elemental composition of the samples was quantified in wt. % based on a charge balance and the EDX peaks acquired.

The thermal degradation characteristics of the coals were examined through non-isothermal thermogravimetric analysis (TGA) under flash heating conditions. The rapid heating (flash) test conditions were selected to simulate the conditions for pulverised coke production. For the TGA runs, about 15 mg of each coal sample was placed in an alumina crucible and heated at 50°C/min from 30°C to 900°C. The TG runs were performed on the Shimadzu TG-50 analyser. The TGA was continuously purged with ultra-pure nitrogen gas (99.99%, Air Liquide, Malaysia) at a flow rate of 20 mL/min during the experiments. On completion, the TGA was cooled to RT, and the data files were retrieved and processed on the Shimadzu TA-60WS Workstation.

Based on the recovered TA ASCII data, the mass loss (TG), and derivative degradation (DTG) curves were plotted against temperature to determine the thermogravimetric characteristics and temperature profiles (TPC) of each coal. The TPCs deduced included; ignition (T_{on}), midpoint (T_{mid}), and maximum decomposition (T_{max}), and burnout (T_{off}) temperatures along with the mass loss rate (MLR, %/min) and residual mass (R_M, %). The T_{on} is the temperature at which the sample begins to decompose after drying is completed during TGA. The midpoint temperature or T_{mid} is the half-way point on the TG plot. However, the T_{max} is the temperature at which maximum decomposition or mass loss rate (M_{LR}, %/min) of the sample occurs, as denoted by a large peak (DTG) during TGA. The burnout temperature (T_{off}) is the temperature at which thermal degradation or devolatilization of the sample is completed. Lastly, the residual mass (R_M, %) is the final mass of the sample at the final TGA temperature. It is a measure of the coke or ash yield of thermally decomposing materials.

3. Results and discussion

3.1. Physicochemical analysis

The physicochemical fuel properties; ultimate (elemental), proximate and calorific values of Obomkpa (BMK), Ihioma (IHM) and Ogboligbo (OGB) coals are presented in Table 1 in as received (*a.r*) basis.

Table 1. Physicochemical properties of BMK, IHM, and OGB

Analyses	Element	Symbol	BMK	IHM	OGB
Ultimate	Carbon	C (wt. %)	50.38	46.80	37.48
	Hydrogen	H (wt. %)	5.62	5.39	3.51
	Nitrogen	N (wt. %)	0.59	0.64	0.80
	Sulphur	S (wt. %)	0.96	1.52	2.33
	Oxygen	O (wt. %)	42.45	45.64	55.88
Proximate	Moisture	M (wt. %)	3.63	4.75	3.12
	Volatiles	VM (wt. %)	58.05	69.52	51.43
	Ash	A (wt. %)	11.73	2.43	1.03
	Fixed Carbon	FC (wt. %)	26.61	23.30	44.41
	Fuel Ratio	F_R	0.46	0.34	0.86
	Mineral Matter	M_m	13.20	3.46	2.39
Heating Value	Higher Heating Value	HHV (MJ/kg)	19.66	19.40	15.55
	Lower Heating Value	LHV (MJ/kg)	18.64	18.41	14.75

The ultimate analysis indicates the coal samples contain high proportions of the combustible elements C, H, O along with the pollutant precursor elements N and S. BMK coal sample has the highest C and H content along with the lowest N, S, and O content (Table 1). Conversely, OGB contains the highest N, S, and O, indicating its potential for generating significant amounts of NOx, SOx and other greenhouse gases (GHG). The OGB coal also exhibited the lowest C and H content which accounts for its poor heating values ranging from 14.75 to 15.55 MJ/kg compared to IHM (18.41 to 19.40 MJ/kg) and BMK (18.64 to 19.66 MJ/kg).

The proximate analysis of the coal samples revealed high VM and FC along with low moisture and ash contents. IHM coal contains the highest M, and VM whereas BMK and OGB contain the highest Ash and FC, respectively (Table 1). Conversely, the lowest M, VM and Ash were observed in OGB, whereas IHM contains the lowest FC. Based on the findings, the high M and VM properties of IHM indicate that it may be suitable as a feedstock for coal gasification into syngas or flue gases for energy production. Conversely, the low M, VM, A and high FC indicate that OGB is best suited for pyrolysis or coke production.

The fuel ratio (F_R) is a ratio of the fixed carbon to the volatile matter [2]. At high F_R, coal combustion will result in high residues of unburnt coal and ultimately poor boiler efficiency [25]. Therefore, F_R is a good measure of the reactivity or the combustibility of coals. The F_R for the coals are: 0.46 (BMK), 0.34 (IHM) and 0.86 (OGB) (Table 1). This indicates that the conversion of OGB coal will result in high deposits (ash or coke), compared to IHM and BMK. Hence, the most reactive coal is IHM, followed by BMK whereas OGB is the least reactive coal. Lastly, the physicochemical properties and calorific values of the coals, indicate that all the coal samples (BMK, IHM and OGB) are lignite (brown) low-rank coals (LRC) with potentials for power generation and cement manufacture.

3.2. SEM/EDX Analysis

The SEM/EDX micrographs for BMK, IHM and OGB are presented in Figures 1-3 (a). The EDX plots showing the mineralogical composition of the coals are presented in Figures 1-3(b) and Table 2.

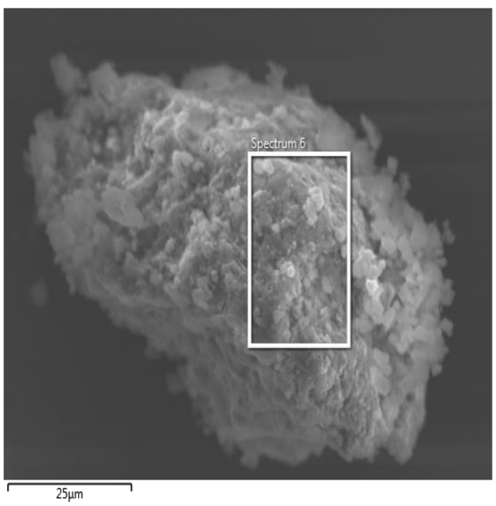

Figure 1(a). BMK Coal

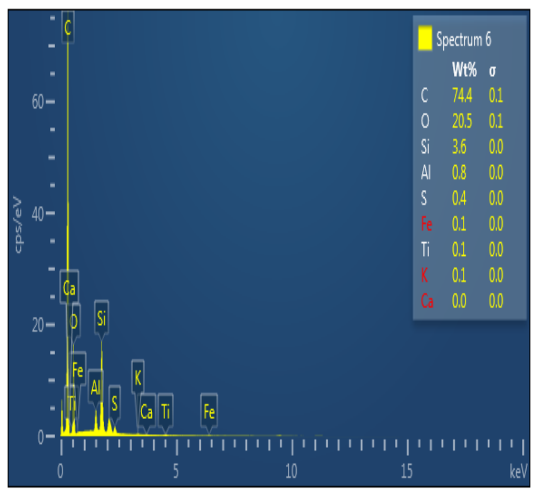

Figure 1(b). BMK Coal

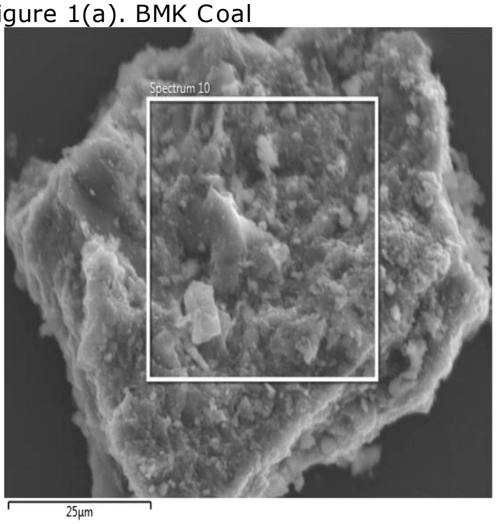

Figure 2(a). IHM Coal

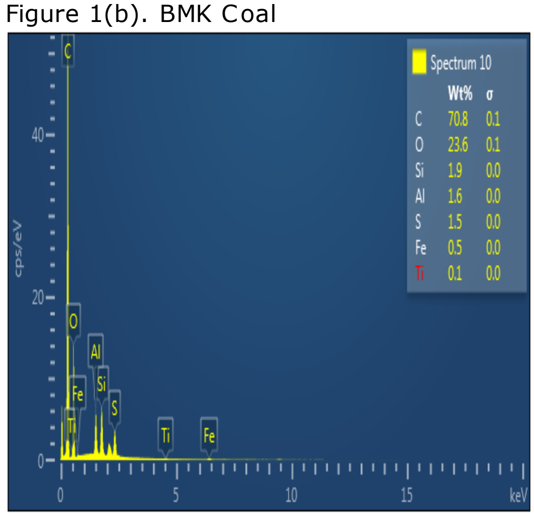

Figure 2(b). IHM Coal

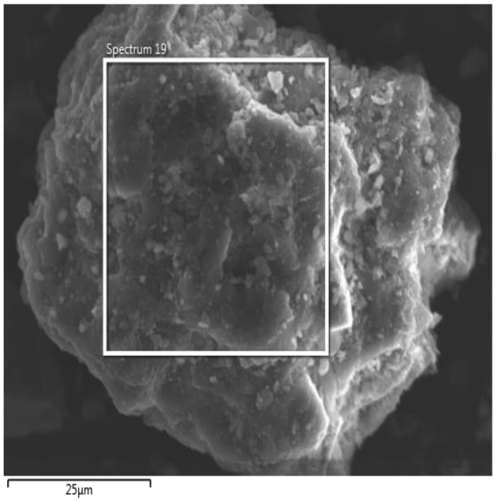

Figure 3(a) OGB Coal

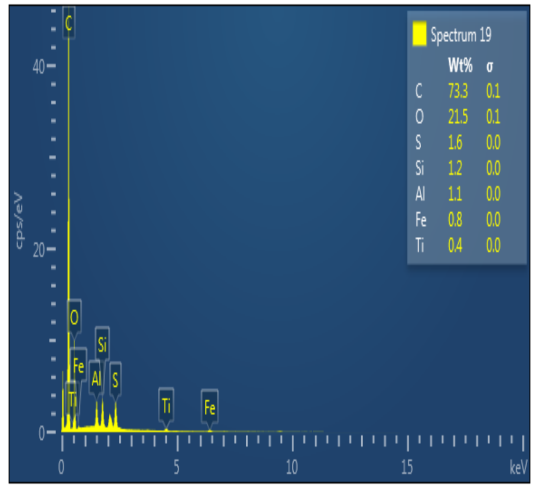

Figure 3(b). OGB Coal

The EDX plots revealed that the coals comprise of C, O, Si, Al, S, Fe, Ti, K, and Ca, although K and Ca were not detected (ND) in the IHM and OGB samples. However, for all samples, the highest elements observed were C, O, Si, and Al whereas the lowest were Ca, K, Ti, and K (Figures 1-3(b) and Table 2.

Table 2. EDX Elemental composition of BMK, IHM and OGB coals

Elements	Symbol	BMK (wt. %)	IHM (wt. %)	OGB (wt. %)
Carbon	C	74.38	70.85	73.34
Oxygen	O	20.46	23.62	21.54
Silicon	Si	3.59	1.91	1.23
Aluminum	Al	0.85	1.61	1.11
Sulfur	S	0.37	1.49	1.63
Iron	Fe	0.14	0.45	0.75
Titanium	Ti	0.12	0.08	0.41
Potassium	K	0.05	ND	ND
Calcium	Ca	0.04	ND	ND

In comparison, BMK exhibited the highest composition of C and S but the lowest O content. This may somewhat be responsible for its porosity and grindability along with its high heating value (Table 1). However, the lowest C composition and highest O content were observed in the IHM coal sample. It is widely reported that the high O content in carbonaceous fuels can result in over oxidation during thermal conversion resulting in high NO_x, SO_x and other greenhouse gases (GHG). Hence, the IHM has the potential to be highly reactive during thermos-chemical conversion. Furthermore, the high concentration of metallic elements such as Al and Fe in IHM can potentially catalyse thermal conversion processes for energy recovery.

3.3. Thermogravimetric analysis

The TG-DTG plots for the flash pyrolysis and thermal degradation of the BMK, IHM and OGB coals are presented in Figures 4 and 5, respectively.

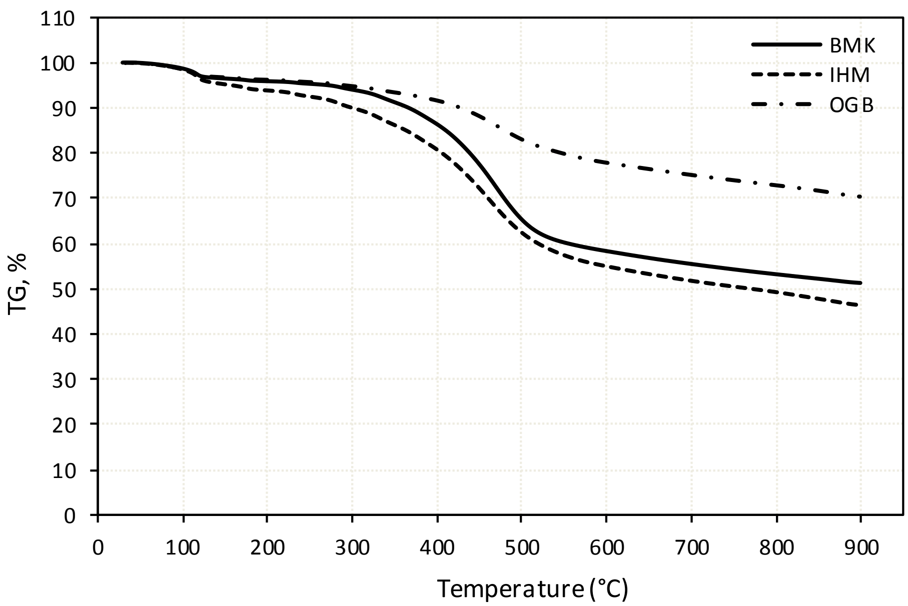

Figure 4. TG Plots for Flash Pyrolysis of Nigerian Lignite Coals

As observed in Figure 4, the TG curves exhibited downward sloping trends from left to the right of the plot. The trends indicate an increase in temperature from 30 to 900°C resulted in a progressive loss of mass during thermal degradation of the samples. However, the thermal degradation and mass loss observed in BMK and IHM were more significant compared to OGB coal. This could be ascribed to the higher contents of C, H, VM and HHV in BMK and IHM compared with OGB (Table 1). The findings indicate that BMK and IHM are more reactive than OGB coal. To confirm this, the reactivity of the coals was further examined by its characteristic temperature profiles (TPC) from the TG plots, as presented in Table 3.

Table 3. TPCs for Flash TG-Pyrolysis of BMK, IHM, and OGB

Characteristic Profiles	Symbol	BMK (°C)	IHM (°C)	OGB (°C)
Onset Temperature	T_{on}	364.94	319.36	340.69
Midpoint Temperature	T_{mid}	453.34	437.41	465.66
Burnout Temperature	T_{off}	543.36	556.86	592.62
Residual Mass (%)	R_M	51.27	46.43	70.35

As observed in Table 3, the ignition temperatures (T_{on}) for the coal samples ranged from 319.36 °C to 364.94°C or an average of 341.66°C. The lowest and highest values of T_{on} were observed for IHM and BMK coals, respectively. The midpoint (T_{mid}) temperatures ranged from 437.41°C to 465.66°C or an average of 452.14°C. The lowest and highest values of T_{mid} were observed for IHM and OGB, respectively. However, the burnout (T_{off}) temperatures ranged from 543.36°C to 592.62°C or an average of 564.28°C. The lowest and highest values of T_{off} was observed for BMK and OGB, respectively. Lastly, the residual mass (R_M, %) for the coals ranged from 46.43% to 70.35%. The lowest and highest values of R_M was observed for IHM and OGB, respectively. Based on the TPC values derived from the TG plots, IHM is the most reactive of the coals whereas OGB is the least reactive.

The reactivity of the coal samples was examined from the DTG plots presented in Figure 5. As observed, the thermal decomposition of the samples resulted in two sets of endothermic peaks. The first peak was observed from RT to 200°C, whereas the second larger peak was from 200°C to 600°C. The mass loss in the temperature range of the first peak was 4.11% for BMK, 6.09% for IHM and 3.82% for OGB. The values are marginally higher than the moisture content of BMK (3.63%), IHM (4.75%) and OGB (3.12%) reported in Table 1. This indicates the mass loss in this range can be ascribed not only to drying but also the loss of low molecular weight compounds during thermal decomposition of the coals.

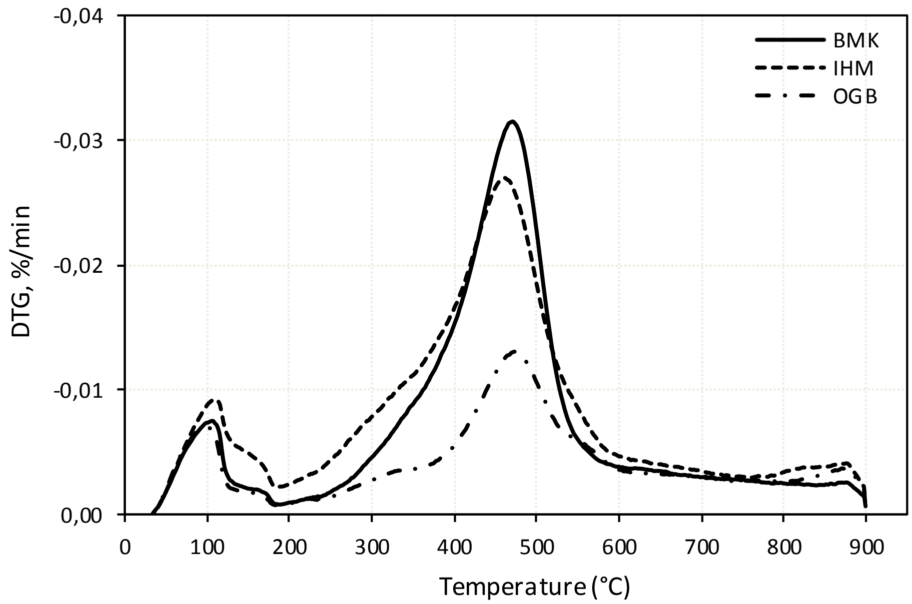

Figure 5. DTG Plots for Flash Pyrolysis of Nigerian Lignite Coals

The second peaks observed from 200°C to 600°C resulted in mass losses of 37.59% for BMK; 38.90% for IHM; and 18.35% for OGB coals. The highest mass loss and thermal decomposition was observed for IHM, followed closely by BMK and finally OGB coal. This confirms that IHM is the most reactive whereas OGB is the least reactive of the coals examined. Furthermore, the mass loss observed is much lower than volatile matter content for BMK (58.05%); IHM (69.52%); and OGB (51.43%). Therefore, the complete or higher decomposition of the coals may require higher temperatures and slower heating rates in future energy conversion systems. These conditions will potentially improve the TPCs and energy recovery potential of the coals. The TPCs deduced from the DTG plots for flash pyrolysis of the coals are presented in Table 4.

Table 4. DTG TPCs for flash pyrolysis of BMK, IHM, and OGB

Characteristic Profiles	Symbol /Unit	BMK (°C)	IHM (°C)	OGB (°C)
Drying peak	T_{dry}	166.90	32.20	98.32
Decomposition peak	T_{max}	470.59	460.98	473.07
Mass Loss	M_L, %	48.73	53.58	29.65

As observed, the results show that IHM exhibited the lowest value of drying peak temperature whereas the BMK had the highest at 166.90°C. This indicates that the process of drying BMK requires more energy input and reaction time compared to OGB and IHM. In contrast, the highest maximum peak decomposition temperature (T_{max}) was observed for OGB, followed by BMK and IHM which has the lowest. These results indicate that based on T_{max}, the IHM coal is the most reactive as its maximum decomposition occurs at 460.98°C which is lower than BMK (470.59 °C) and OGB (473.07°C).

Lastly, the mass loss (M_L, %) of the coals was examined to highlight the potential solid product formed from the decomposition process. As observed in Table 4, the highest M_L (%) was observed for the IHM coal sample whereas the lowest was observed for OGB. Since the product of coal pyrolysis is typically coke, the results demonstrate that OGB has highest coke forming potential (based on the residual mass R_M values in Table 3), followed by BMK and lastly IHM. However, further tests are required to verify this inference.

4. Conclusions

The coal samples from Obomkpa (BMK), Ihioma (IHM) and Ogboligbo (OGB) contain high proportions of C, H, O along with N and S elements. The BMK coal sample has the highest C and H content along with the lowest N, S, and O. However, OGB contained the highest N, S, and O indicating a high potential for NOx, SOx and other GHG emissions. OGB has the lowest C and H content and the corresponding least HHV (15.55 MJ/kg) compared to IHM (19.40 MJ/kg) and BMK (19.66 MJ/kg). Hence, the coals BMK, IHM and OGB coals are classified as lignite (brown) or low-rank coals (LRC) with potential for thermal energy recovery. The morphological analysis revealed that BMK, IHM and OGB coals are comprised of particles of various shapes and sizes. The mineralogical analyses revealed the coal composition consists of C, O, Si, Al, S, Fe, Ti, although K and Ca were not detected in IHM and OGB.

The thermal degradation behaviour and TPCs were also examined. The findings show that IHM and BMK are more reactive than OGB coal. Based on the TGA derived TPCs, IHM is the most reactive coal whereas OGB is the least reactive under the conditions examined in this study. The DTG revealed two endothermic peaks for drying (RT – 200°C) and devolatilization (200°C to 600°C). The highest mass loss and thermal decomposition were observed in IHM, followed by BMK and finally OGB coal. This confirms that IHM is the most reactive whereas OGB is the least reactive of the coals examined. In conclusion, the Obomkpa (BMK), Ihioma (IHM) and Ogboligbo (OGB) coal samples possess the requisite physicochemical, mineralogical, morphological fuel properties for future energy conversion and recovery systems in Nigeria.

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