Petroleum and Coal

PHYSICOCHEMICAL AND OXIDATIVE THERMAL ANALYSIS OF NIGERIAN LIGNITE COALS

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Abstract

This paper presents the physicochemical and oxidative thermal properties of new lignite coals from Obomkpa (BMK), Ihioma (IHM), and Ogboligbo (OGB) in Nigeria. Furthermore, the microstructural and mineralogical properties of the coals were examined in the study. The results revealed high compositions of carbon (C), hydrogen (H), oxygen (O) and sulphur (S) but low nitrogen (N) for all samples. The highest values of C (50.4 wt.%), H (5.62 wt.%), HHV (19.66 MJ/kg) and the lowest O (42.45 wt.%) were observed in BMK. However, IHM exhibited the highest volatile matter (69.5 wt.%) and lowest fixed carbon (23.3 wt.%); whereas the lowest ash (1.03 wt.%) and moisture (3.12 wt.%) were observed in OGB. The microstructure analysis revealed Al, C, Ca, Fe, K, O, S, Si, and Ti although K and Ca were undetected in IHM and OGB. Thermal analysis revealed a mass loss of 75.7% for BMK, and 79.4% for IHM, and 56.2% for OGB. The DTG plots revealed two distinct regions for drying and devolatilization during coal thermal degradation. The temperature profile characteristics (TPC) revealed IHM is the most reactive compared to BMK and OGB. Overall, the results indicated that IHM is suited for gasification, BMK for combustion, and OGB for pyrolysis.

Keywords: *Fuel Characterization; Combustion Characteristics; Lignite; Coal; Nigeria.*

1. Introduction

Coal is one of the most abundant and accessible fossil-based fuels worldwide [1]. Currently, coal accounts for over 35% of global electricity generation and by extension the world's energy mix. Over the years, this widely distributed resource has contributed significantly to socio-economic growth and development as witnessed in India and China. Against this backdrop, many analysts posit that coal has the potential to supply most of the world's growing energy demand particularly in developing countries with large deposits. In recent years, various studies have reported on the discovery of large deposits and the potential of various low-rank coals (LRC) in Nigeria [2-6]. The findings indicate that despite growing attention, coal mining in Nigeria has dwindled from its peak of 925,000 tonnes in 1958 to below 20,000 tonnes today [3]. Furthermore, the studies report that Nigeria holds 640 million tonnes of proven reserves and 2.8 billion tonnes of provisional reserves located across the nation's sedimentary basin [7]. The rank classification of Nigerian coals consists of 39% bituminous, 49% subbituminous, and 12% lignite [5], currently utilized in cement, iron and steel manufacturing.

However, the contribution of coal to Nigeria's electricity generation remains insignificant [3]. As a result, Nigeria's drive for industrialisation has been stunted by the current energy crises

and inability to exploit the large coal deposits for electricity generation [5-6]. This is partly due to limited data on the thermochemical fuel characteristics of lignites as required for the design and operation of coal-fired power plants. Even so, various studies have examined the geological [8-10], petrographic [11-13], and mineralogical [14-16] properties of selected Nigerian coals. However, the reviewed literature is limited to the geochemical and mineralogical properties of Nigerian coals and its potential environmental impacts. Other studies including Sonibare *et al.,* [17], Ryemshak and Jauro [2], and Chukwu *et al.* [5], have examined the thermal and power generation properties of sub-bituminous and bituminous Nigerian coals. However, there are limited studies on the thermal, kinetic, thermodynamic and rheological fuel properties of lignites, typically utilised for electricity generation through pulverised coal combustion [18]. This shows there is an urgent need to examine and highlight the fuel properties of Nigeria's abundant lignite reserves for future power generation.

Therefore, this study will present an in-depth analysis on the physicochemical, microstructural, mineralogical, and thermal properties of Obomkpa (BMK), Ihioma (IHM), and Ogboligbo (OGB) lignites from Delta, Imo, and Kogi States, respectively. It is envisaged that the findings will avail engineers and policymakers with empirical data for power generation, greenhouse gas (GHG) emissions and life cycle analyses.

2. Experimental

The lignite coals examined in this study were acquired in rock form from Obomkpa in Aniocha-North, Ihioma in Orlu, and Ogboligbo in Igalamela-Odolu located in Delta, Imo, and Kogi States of Nigeria, respectively. The samples were subsequently labelled; BMK (Obomkpa), IHM (Ihioma) and OGB (Ogboligbo) before pulverisation and sieving into 250 μm sized particles for characterisation. The coal preparation procedures are described in our previous study [19]. Next, the fuel characteristics of the pulverised coals were characterised by physicochemical, functional group, microstructural, mineralogical, and thermal analyses.

The physicochemical analyses were performed to determine the elemental, proximate, and calorific properties of the coals. The elemental analysis was performed using the CHNS elemental analyser (Model: vario MICRO Cube, Germany) according to the ASTM standard D5291-16. The proximate analysis was performed by thermogravimetric analysis (TGA) according to the procedures described in the literature [20]. Lastly, the calorific analysis was determined by bomb calorimetry based on the procedures described in ASTM D2015 for the IKA C2000 (Bomb calorimeter, USA).

The functional group compositions of the samples were determined by Fourier transform infrared-attenuated transform reflectance (FTIR-ATR) spectroscopy (Model: Shimadzu Prestige 21, Japan). During each test, a small amount of each coal sample was placed in the ZnSe prism plate and scanned with the detector to acquire FTIR spectra from 4000 – 600 cm^{-1} based on Happ-Genzel Apodization. Each sample was subjected to 20 scans at 8 cm^{-1} resolution for a total runtime of five (5) seconds. After the analysis was completed, ATR correction was applied to the spectra before plotting the raw data in transmittance (T %).

The microstructural and mineralogical analyses were performed by SEM/EDX (scanning electron microscopy-energy dispersive x-ray) microscopy. The SEM microscope (Model: JEOL-JSM IT 300 LV, Germany) equipped with an EDX detector was used to examine the microstructure and mineral composition of the coals. Before each test, the samples were sputter coated with platinum using the Quorum Q150R S apparatus. Next, the samples were placed in the SEM/EDX analyser operating at 20 kV, the working distance of 5 mm, and magnification of ×1000. The SEM images were subsequently examined on the AZTEC EDX software (Oxford Instruments, UK) and the mineral composition determined by point ID and mapping. The resulting mineral compositions were reported in weight per cent (wt.%).

The thermal analysis was determined by thermogravimetric analysis (TGA) under non-isothermal oxidative conditions. The TG runs involved heating approximately 18 mg of sample in an alumina crucible from 30°C to 900°C by employing a dynamic heating rate of 50°C/min to simulate flash combustion. During each test, the TG analyser (Model: Shimadzu TG-50, Japan) was

purged with air at a flow rate of 20 mL/min to ensure an oxidative atmosphere. At the end of the process, the raw (.tad) data was retrieved and analysed to determine the mass loss (%) and derivative mass loss (%/min) for the samples which were plotted as TG and DTG, respectively.

Based on the TG-DTG data, the temperature profile characteristics (TPC) for the flash combustion of the coals were examined. The examined TPCs were onset or ignition (T_{ons}), midpoint (T_{mid}), maximum decomposition (T_{max}), and burnout (T_{off}) temperatures, along with the mass loss (M_L, %), mass loss rate (M_{LR}, %/min) and residual mass (R_M, %). The descriptions of the TPC and the procedures for determining the terms are presented in our previous study [6].

3. Results and discussion

This section presents the physicochemical properties, functional group analysis (FTIR), microstructural and mineralogical properties (SEM/EDX), thermal degradation properties (TGA-DTG), and temperature profile characteristics (TPCs) of the coal samples.

3.1. Physicochemical fuel properties

Table 1 presents the physicochemical fuel properties of the coals based on ultimate, proximate and calorific analyses. As observed, the ultimate (elemental) analysis revealed high compositions of C, H, O and S but low N content. BMK exhibited the highest composition of C, and H but the lowest O whereas OGB exhibited the lowest composition of C and H. The proximate analysis revealed high compositions of VM and FC but low M and ash as observed in Table 1. The highest and lowest compositions of VM was observed in IHM and OGB, respectively. However, OGB contained the highest FC and the lowest was observed in IHM. The highest ash content was observed in BMK and the lowest in OGB.

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

Analyses	Element	Symbol	BMK	IHM	OGB
	Carbon	C (wt. %)	50.38	46.80	37.48
	Hydrogen	H (wt. %)	5.62	5.39	3.51
Ultimate	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
	Moisture	M (wt. %)	3.63	4.75	3.12
	Volatiles	VM (wt. %)	58.05	69.52	51.43
Proximate	Ash	A (wt. %)	11.73	2.43	1.03
	Fixed Carbon	FC (wt. %)	26.61	23.30	44.41
Heating Value	Higher Value	HHV (MJ/kg)	19.66	19.40	15.55

Lastly, the calorific analysis revealed BMK has the highest energy content based on the HHV of 19.66 MJ/kg compared to 19.40 MJ/kg for IHM and 15.55 MJ/kg of OGB. The HHV variation is due to the high compositions of C, H and low O in the BMK coal compared to the other samples. The findings also indicate that BMK has the highest energy recovery potential compared to IHM and OGB coals. Hence, the coals can be classified as brown or lignite, low ranked coals (LRC) with potential for electric power generation or cement manufacture.

3.2. Functional group properties

The functional group composition of the coal samples was determined by FTIR-ATR spectroscopy as presented in the spectra in Figure 1. As observed, the spectra revealed several peaks for stretching vibrational intensities which ranged from 4000 – 600 cm^{-1} during the analyses. The results reveal that the largest stretching vibrations were in the high-frequency region (4000 – 1450 cm^{-1}), compared to the fingerprint region (1450 – 600 cm^{-1}). Based on the spectra, the stretching vibrations in the region 4000 – 3800 cm^{-1} can be ascribed to the rotational vibrations of O–H groups present in water vapour. This confirms the presence of moisture in the structure of the coals examined in this study. However, the sharp peaks observed in the region

3800 – 3500 cm^{-1} and the medium to weak peaks in the regions 1300 – 1400 cm^{-1} and 1200 – 1000 cm^{-1} are either due to free or H bonded O–H groups.
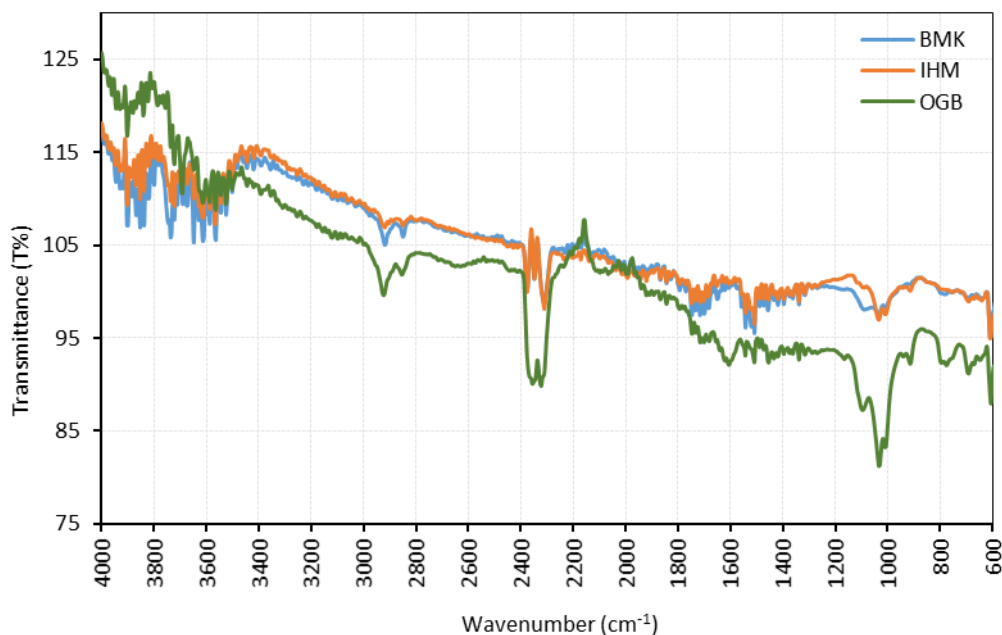

Figure 1. FTIR spectra of Nigerian lignite coals

Furthermore, the pair of medium to weak peaks the region 3000 – 2800 cm^{-1} are due to the deformation or rocking effects of –CH$_3$, –CH$_2$ and C–H aliphatic groups of alkanes. However, the strong peaks in the range 2300 – 2200 cm^{-1} can be due to the –C≡C– or –C≡N– groups in alkynes. However, the medium peaks in the same region may be due to –N=C=O, –N=C=S or N=C=N groups. The medium to strong peaks at 1500 cm^{-1} and 1600 cm^{-1} could be due to –C=C– groups found in arenes.

In general, the FTIR-ATR results indicate OGB coal has the most intense peaks followed by IHM and BMK. Lastly, the findings indicate that the chemical structure of the coals is complex and could be due to the presence of alkane, alkene, arene, and aromatic functional groups. However, further analyses are required to elucidate the chemical structure of the coals.

3.3. Microstructural and mineralogical properties

The microstructural and mineralogical properties of the coals were determined by SEM/EDX analyses, as presented in Figures 2-4(a) and 2-4(b), respectively.
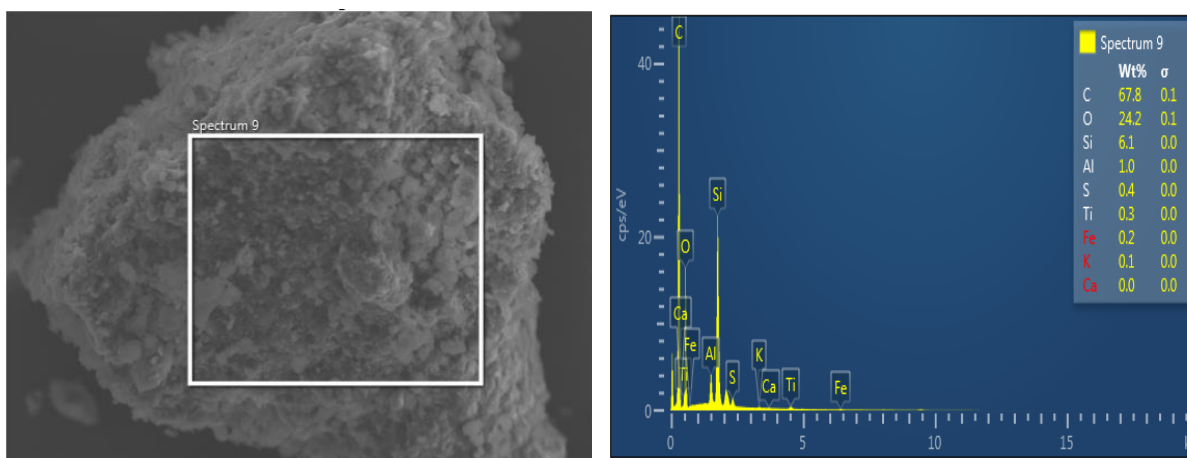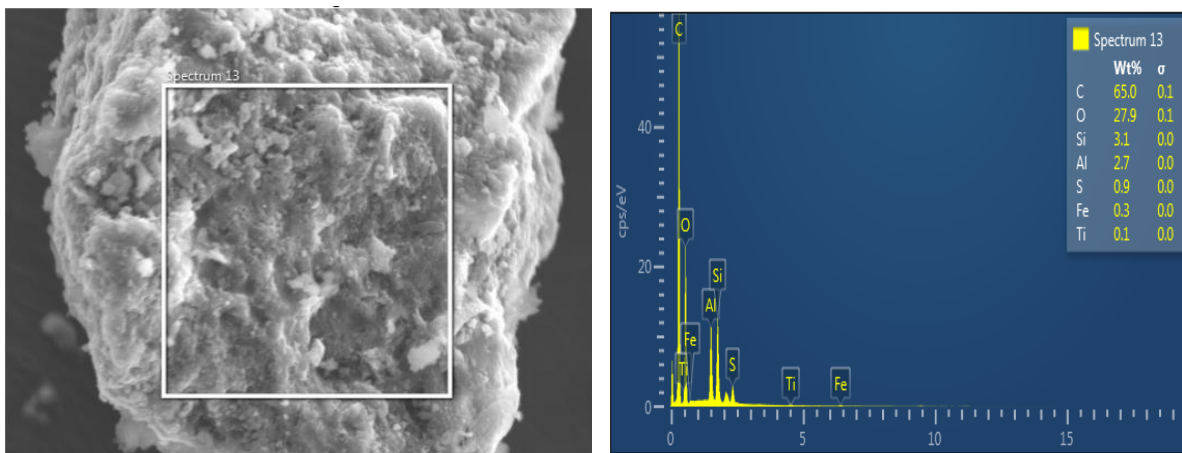

Figure 2 (a) (b). SEM/EDX Spectra for BMK
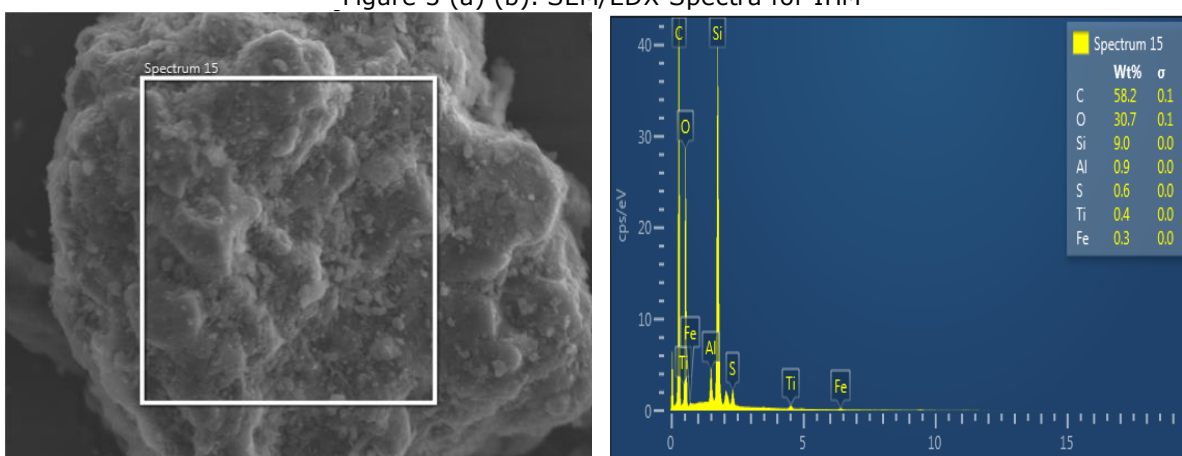
Figure 3 (a) (b). SEM/EDX Spectra for IHM

Figure 4 (a) (b). SEM/EDX Spectra for OGB

The results indicate that BMK, IHM, and OGB contain a range of fine to coarse-grained particles in their microstructures. In particular, the morphology of BMK is characterised by small-sized particles which appear scattered in clusters or agglomerates on the sample surface. However, OGB is characterised by layers of small particles in its microstructure. Lastly, IHM is comprised of coarse-grained particles characterised by a distinct lustre which may be due to the metal elements present in its structure. To verify this, the mineral composition of the coals was examined by EDX as presented in weight percentages (wt.%) in Table 2.

Table 2. Mineral composition of Nigerian lignite coals BMK, IHM, and OGB

Element	Symbol	BMK (wt. %)	IHM (wt. %)	OGB (wt. %)
Carbon	C	67.79	65.01	58.15
Oxygen	O	24.17	27.88	30.66
Aluminium	Al	0.97	2.73	0.88
Silicon	Si	6.10	3.08	9.01
Sulphur	S	0.38	0.88	0.60
Potassium	K	0.07	ND	ND
Calcium	Ca	0.04	ND	ND
Titanium	Ti	0.30	0.13	0.36
Iron	Fe	0.18	0.28	0.34

As observed in Table 2, the coal samples examined in this study contain both metal and non-metal elements; C, O, Al, Si, S, K, Ca, Ti, and Fe, although K and Ca were not detected (ND)

in IHM and OGB. In comparison, the most abundant elements present in the coals were C, O, Si whereas the least abundant elements were Ti and Fe. Based on the results, the highest C was observed in BMK, followed by IHM and OGB. However, the presence of O was in the order BMK < IHM < OGB which accounts for the higher heating value (HHV) of BMK compared to IHM and OGB as presented in Table 1. The Si was in the order IHM < BMK < OGB. The presence of Si typically in free crystalline form is indicative of the presence of SiO_2 (quartz) in the structure of the coals. Lastly, the elements S and Al are also present in the coals with the highest compositions in IHM.

3.4. Thermal and degradation behaviour

The thermal degradation behaviour and temperature profile characteristics (TPCs) of the coals were also examined as presented in the TG-DTG plots in Figures 5 and 6.
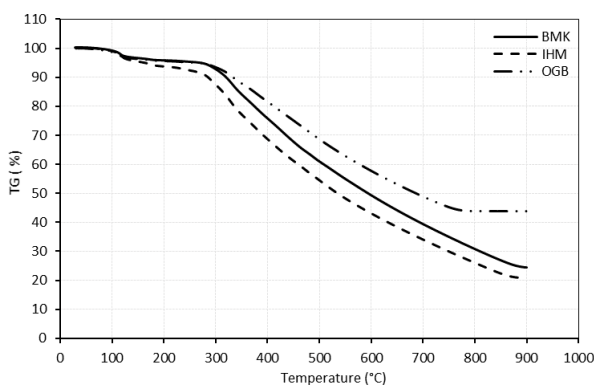

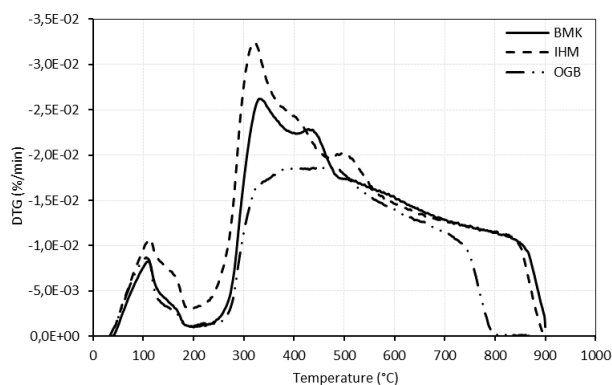Figure 5. TG Plots for flash combustion of BMK, IHM, OGB

Figure 6. DTG Plots for flash combustion of BMK, IHM, OGB

As observed in Figure 5, the thermal analysis resulted in a significant mass loss or thermal degradation as evident in the downward sloping plots in Figure 5. Furthermore, the TG plots revealed that the highest degradation occurred in IHM compared to BMK and OGB. Hence, IHM is more reactive compared to BMK and OGB, under the conditions examined in this study. However, the lowest thermal reactivity or degradation was observed for OGB as evident in its TG plot which plateaus at 43.80%. The high reactivity of IHM may be ascribed to its higher volatiles (VM) and fixed carbon (FC) contents compared to OGB as reported in Table 1. Therefore, the VM and FC are responsible for the temperature profile characteristics (TPC) and thermal reactivity during TGA. To further examine this, the TPCs were deduced from the TG-DTG plots using the Shimadzu TA-60 Workstation, as presented in Tables 3 and 4.

The degradation mechanism for the flash combustion of the coals was further examined from the DTG plots in Figure 6. As observed, the TG analysis resulted in two distinct regions of thermal degradation. The first region, characterised by the small peaks observed below 200°C, can be ascribed to the loss of moisture and low molecular weight volatile compounds. However, the second region occurred at 200°C – 750°C for OGB and 200°C – 850°C for both BMK and IHM during TG analysis. The mass loss (M_L, %) in this stage can be ascribed to the loss of high molecular weight or volatile compounds resulting in ash formation due to the oxidative nature of the process. As a result, the final or residual mass (R_M, %) of the process are representative of the ash that could potentially result from the flash combustion of BMK, IHM, and OGB under flash conditions. The resulting values for M_L, % and the R_M, % for each coal is presented in Table 3.

3.5. Temperature profile characteristics (TPC)

The TPCs for the TG is presented in Table 3, and Table 4 presents the TPCs from the DTG plots. The TPCs examined in this study are; onset or ignition (T_{ons}), midpoint (T_{mid}), burnout (T_{off}) temperatures, along with the mass loss (M_L, %) and residual mass (R_M, %). The results

revealed that the ignition (T_{ons}) temperatures ranged from 246.18°C (IHM) to 269.69°C (BMK) or an average of 256.68°C. This indicates that IHM undergoes thermal degradation or devolatilization earlier than BMK and OGB (254.16°C). This is due to the high VM content of IHM (69.52 wt.%) compared to BMK (58.05 wt.%) and OGB (51.43 wt.%).

Table 3. TG Temperature Profiles Characteristics of BMK, IHM, OGB

Coal Sample	Onset temperature (T_{ons}, °C)	Midpoint temperature (T_{mid}, °C)	Burnout temperature (T_{off}, °C)	Mass loss (M_L, %)	Residual mass (R_M, %)
BMK	269.69	479.68	614.35	75.67	24.33
IHM	246.18	453.98	587.86	79.37	20.63
OGB	254.16	474.17	689.14	56.19	43.81

The midpoint (T_{mid}) temperatures ranged from 453.98°C to 479.68°C for IHM and BMK, respectively. However, the burnout (T_{off}) temperatures were between 587.86°C and 689.14°C for IHM and OGB, respectively. Lastly, the mass loss (M_L, %) and residual mass (R_M, %) for the coals were examined. The mass loss (M_L, %) was between 56.19% and 79.37% for OGB and IHM. Therefore, OGB experienced the lowest mass loss whereas the highest (and most reactive) was IHM. However, the residual mass was from 20.63% to 43.81% for IHM and OGB, respectively. This indicates that OGB is thermally stable during TG compared to IHM and BMK.

The TPCs for the DTG plots are; the drying peak (D_{max}), maximum decomposition (T_{max}), and mass loss rates (M_{LR}, %/min) at D_{max} and T_{max} were examined as presented in Table 4.

Table 4. DTG temperature profiles characteristics of BMK, IHM, OGB

Coal Sample	Maximum drying peak temperature (D_{max}, °C)	Rate (%/min)	Maximum decomposition peak temperature (T_{max}, °C)	Rate (%/min)
BMK	109.29	2.82	330.03	7.25
IHM	113.65	3.09	322.00	8.56
OGB	105.82	2.88	452.94	3.28

As observed, the maximum drying peak (D_{max}) temperatures ranged from 105.82°C (OGB) to 113.65°C (IHM). However, the maximum decomposition (T_{max}) temperatures occurred between 322.00°C (IHM) and 452.94°C (OGB). Further examination of the M_{LR} (%) at D_{max} showed that the lowest and the highest mass values were observed for BMK (2.82 %/min) and IHM (3.09 %/min). Conversely, the lowest and the highest M_{LR} (%) at T_{max} was from 3.28 %/min (OGB) to 8.56 %/min (IHM). In summary, the temperatures D_{max}, T_{max} and mass loss rates (M_{LR}, %) confirm that IHM is more reactive than BMK and OGB.

4. Conclusion

The study examined physicochemical and oxidative thermal analysis of newly discovered brown coals from Obomkpa (BMK) in Aniocha-North, Ihioma (IHM) in Orlu, and Ogboligbo (OGB) in Igalamela-Odolu in Nigeria. The study investigated the elemental, proximate, calorific and functional group properties of the coals. Furthermore, the study examined the microstructural, mineralogical and thermal analyses. The results revealed high compositions of C, H, O and S but low N. BMK exhibited the highest composition of C, and H but the lowest O whereas the lowest values were observed in OGB. The highest and lowest compositions of VM was observed in IHM and OGB, respectively. The calorific analysis revealed an HHV of 19.7 MJ/kg for BMK, 19.4 MJ/kg for IHM and 15.6 MJ/kg for OGB.

The functional group analysis revealed the complex coal structure is due to the presence of alkane, alkene, arene, and aromatic functional groups. The microstructure analysis also revealed fine to coarse-grained particles characterised by metal and non-metal elements such as C, O, Al, Si, S, K, Ca, Ti, and Fe. The thermal analysis revealed the highest degradation

occurred in IHM which is more reactive than BMK and OGB. The DTG plots revealed two distinct regions of thermal degradation ascribed to drying and devolatilization. The TPC analysis of ignition (T_{ons}), midpoint (T_{mid}), burnout (T_{off}) temperatures, and the mass loss (M_L, %) and residual mass (R_M, %) revealed IHM is the most reactive coal compared to BMK and OGB.

Acknowledgement

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