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Petrology, Physicochemical and Thermal Analyses of Selected Cretaceous Coals from the Benue Trough Basin in Nigeria

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Petrology, physicochemical and thermal analyses of selected cretaceous coals from the Benue Trough Basin in Nigeria

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Abstract Abundant coal resources that were previously neglected due to a crude oil boom need revitalisation and integration into the national electricity mix to address the energy demands of the Nigerian population. Selected coal samples from the Benue Trough sedimentary basin in Nigeria were examined by various techniques, including proximate and ultimate analyses, organic petrography, Fourier transform infrared ray spectroscopy, and thermogravimetric analysis. Based on vitrinite reflectance, the Lafia-Obi (OLB), Garin Maiganga (GMG), Imiegba (IMG), and Okaba (OKB) coals are classified as subbituminous, while the Lamja1 (LMJ1), Lamja2 (LMJ2) and Chikila (CHK) coals are high volatile B bituminous. The Enugu (ENG) coal is on the boundary between subbituminous and high volatile C bituminous. Organic petrographic results indicate vitrinite and fusinite contents steadily increase from the Lower Benue Trough coals to the Upper Benue Trough coals, while semifusinite and total mineral contents follow a reverse pattern. Thermal decomposition occurred in three stages, i.e., drying, devolatilization, and coke formation above 700 °C; and the coal reactivity follows the following order, ENG>IMG>IGH>CHK>LMJ>OKB>GMG>LFB. The higher temperatures (above 900 °C) are required to decompose the coals for efficient energy recovery. The LMJ1, LMJ2, OLB, CHK, GMG, and OKB coals can be exploited for electricity power generation. However, the Imeagha and Enugu coals are best suitable for both cement and power generation.

Keywords Petrology · Thermal analysis · Cretaceous coal · Benue Trough, Nigeria

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1 Introduction

The inadequate supply of electricity in Nigeria is hindering socio-economic growth and sustainable development, despite the nation's abundant energy resources. There exists a robust relationship between socio-economic development and access to electricity in the nations around the world (Kanagawa and Nakata 2008). In spite of this, the electricity generation capacity of Nigeria has declined by 60% owing to years of negligence by successive regimes (Aliyu et al. 2013; Oodo and Zou 2009). As such, only 40% of Nigeria's citizens have access to electricity, with varying degree of power failure, systematic power cuts, and low voltage (Aniefiok et al. 2013; Sambo 2009; Vincent-Akpu 2012). Moreover, Nigeria's gas-dominated electricity grid consistently breaks down, primarily due to insufficient gas supply. This has been exacerbated by gas pipeline vandalism linked with resource-control militancy in the oil-producing Niger Delta region (Iwayemi 2008). Therefore, there is an urgent need to diversify the national energy mix and protect power-generation infrastructure.

Coal utilization for electric power generation can potentially address the problems of energy diversification and self-sufficiency in Nigeria. As a primary source of energy, coal is an essential part of the global energy mix, currently accounting for 38% of global electricity generation (Khan et al. 2018). Coal has significantly contributed to the socio-economic growth and sustainable development of many countries across the globe (Kettanah and Eble 2017), despite growing concerns about environmental degradation, global warming, and climate change (McGlade and Ekins 2015; Meinshausen et al. 2009). According to the United States Energy Information Administration (IEA-OECD 2002), the world's total coal reserves are estimated at 926 Gt (billion tonnes), with the significant deposits located in China, USA, India, Australia, Indonesia, Russia, and Germany (Speight 2012).

In Nigeria, the proven reserves of coal are about 639 Mt (million tonnes), whereas the inferred reserves are 2.75 Gt (Adedosu et al. 2007; Chukwu et al. 2016; Ohimain 2014). According to Chukwu et al. (2016), Nigerian coals consist of 12% lignite, 49% subbituminous, and 39% bituminous, which are primarily located in the Benue Trough sedimentary basin. However, recent exploration efforts have revealed the discovery of commercial quantities of coal in 14 of the 36 Nigerian States, including Abia, Adamawa, Anambra, Bauchi, Benue, Cross-River, Delta, Ebonyi, Edo, Gombe, Imo, Kogi, Nasarawa, and Plateau States of Nigeria (Ohimain 2014). There are 22 known coalfields in Nigeria, and only Okaba, Okpara, Onyeama, and Orukpa are currently operational as depicted in Fig. 1. Obwette, Ribadu, and Ogbete were historically mined (Oboirien et al. 2018; Ohimain 2014; Sambo 2009).

The latest discovery of new deposits has renewed interest in coal utilization for electricity power generation in Nigeria (Nyakuma et al. 2018; Ohimain 2014; Ryemshak and Jauro 2013). However, there is limited research on the assessment of the power generation potential of some newly discovered coal deposits, and also on their organic petrology and fuel characteristics. Earlier research on Nigerian coals has centred on their metallurgical applications (Akpabio et al. 2008; Jauro and Chukwu 2011) along with its rheological and proximate (Ryemshak and Jauro 2013), mineralogical (Adedosu et al. 2007; Ogala et al. 2012), and geochemical characteristics (Jauro et al. 2007; Olajire et al. 2007). However, more information is required for the efficient design, operation, and maintenance of coal-fired power plants in Nigeria. Hence, extensive research is needed to examine the fuel characteristics of Nigerian coals. The aim of this research is to investigate the petrography, rank classification, and thermal fuel characteristics of some selected coal deposits from the Benue Trough Basin, with a view to appraise their energy recovery and electricity generation potentials in Nigeria.

2 Geological setting of Benue Trough

The Benue Trough is a linear, intracratonic and graben basin, 800 km long and 90 km wide on the average in eastern Nigeria (Ogungbesan and Akaegbobi 2011; Fatoye and Gideon 2013). The Benue Trough is a linear NE-SW trending Anticlinorium stretching from the Chad Basin in the North to the Gulf of Guinea through Niger Delta in the South (Avbovbo 1980). The Benue Trough basin is filled by approximately 6000 m thick alternating marine, paralic, and continental sediments of Cretaceous-Tertiary ages predating the mid-Santonian age. The sediments were compressed, deformed, folded, faulted, and uplifted in several places, and produced over 100 anticlines and synclines. The Benue Trough is geographically subdivided into Lower, Middle, and Upper Benue Trough (Whiteman 1982) (Fig. 2). These subdivisions are broadly based on differences in the physiographic and lithostratigraphic nomenclature (Sonibare et al. 2008).

The comprehensive reports of the geological and stratigraphic progressions in the Benue Trough are highlighted in the literature (Ayinla et al. 2017; Obaje et al. 2004; Offodile 1976; Petters 1982; Yandoka et al. 2016). The upper section of the Benue trough is split at the north-eastern side into the basins around Gongola and Yola (Fig. 2). In these basins, the Albian Bima sandstone unconformably overlies the basement and is conformably overlain by the Cenomanian transitional or coastal Yolde Formation that characterises the start of marine incursion into the Upper Benue Trough (UBT). In the Gongola basin, the laterally equivalent Gongila and Pindiga Formations correlate with the Yolde Formation. The formations were intruded by marine infiltration into the Upper Benue Trough during the later Cenomanian–Turonian-Santonian periods. In the Yola Basin, the Dukul, Jessu, and Sekuliye Formations exist along with carbonaceous Lamja Sandstones and Numanha shales. The Upper Cenomanian–Turonian-Santonian (CTS) equivalents are the Gongila and Pindiga Formations. The Upper CTS deposits in the Yola Basin are lithologically and paleoenvironmentally similar to the Gongola Basin, except for the Lamja Sandstone, which is dominated by marine sandstone lithology. The post-folding sediments are represented by the continental coal-bearing Campano-Maastrichtian-age Gombe sandstone and the Paleogene Kerri–Kerri Formation.

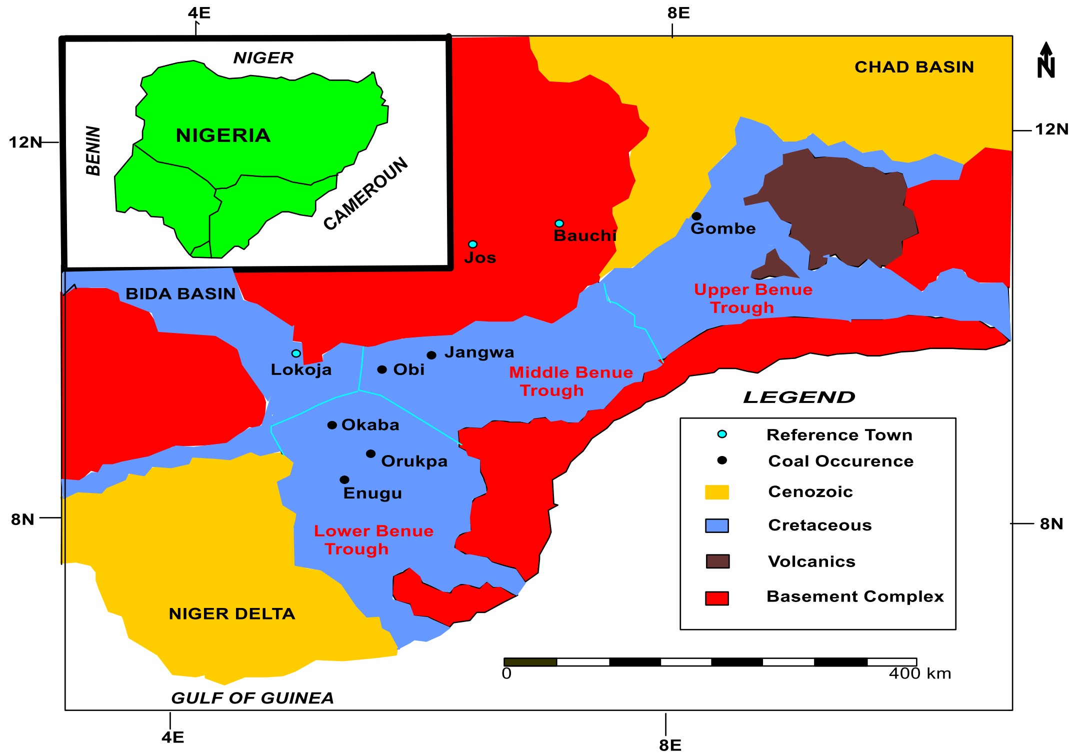

Fig. 1 Location of major coal occurrences in the Benue Trough of Nigeria (Obaje et al. 1999)

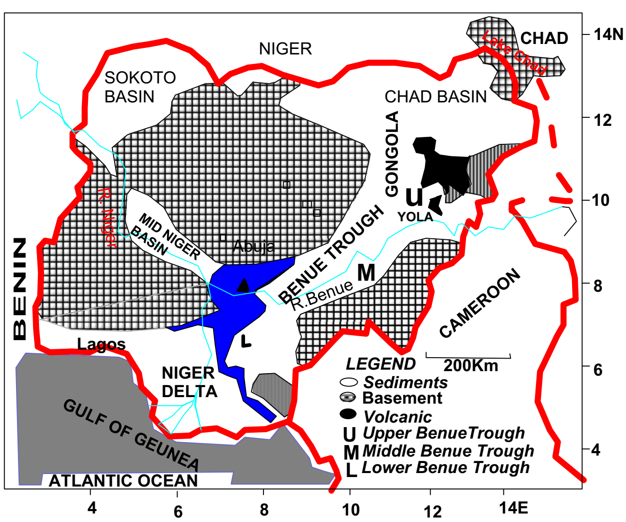

Fig. 2 Geological map of Nigeria showing the Benue Trough (Jauro et al. 2007)

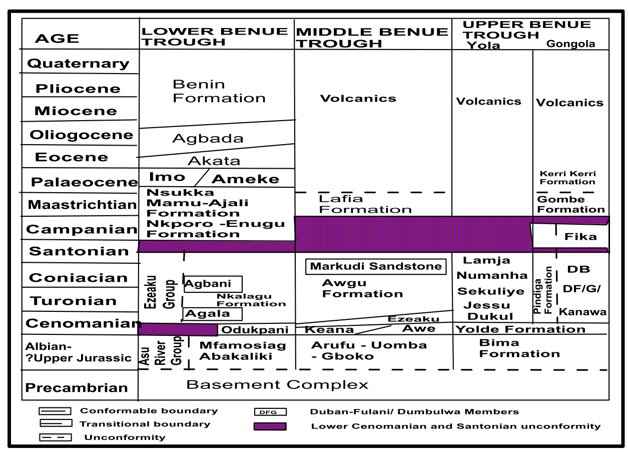

Fig. 3 Stratigraphic succession of Benue Trough (Abubakar et al. 2006)

In the Middle Benue Trough (MBT), around Lafia-Obi, six Upper Cretaceous lithogenic formations consists of stratigraphic successions (Fig. 3), including Albian Arufu, Uomba, and the Gboko Formations commonly called the Asu River Group (Offodile 1976). In turn, these were overlain by the Cenomanian Keana, Awe, and the Cenomanian–Turonian Ezeaku Formations. The late Turonian/early Santonian coal-bearing Awgu Formation lies conformably over the Ezeaku Formation. The post-folding Campanian–Maastrichtian Lafia Formation terminated the sedimentation in the Middle Benue Trough and was followed by Paleogene volcanic activity.

In the Lower Benue Trough (LBT), the oldest geological formation overlying the basement is the Albian Asu River Group. This marine-influenced formation consists of dark coloured fine-grained sandstones and siltstones, overlain by shales and limestones (Offodile 1980). Next in succession is the Cross River Group, which comprises the Odukpani, Agala, Nkalagu, and Agbani Formations. However, the only acknowledged marine Cenomanian rocks in the margins of the basin occurred in the Odukpani Formation, which consists of limestones and calcareous sandstones. The Cross River Group was succeeded by the Santonian-age major folds concurrent with depositional episodes. The Nkporo shales and deposition of Maastrichtian Mamu Formation coal measure are evidence of marine incursion in the Basin. This also includes the enigmatically bedded sandstones and shales of the Ajali and Nsukka Formations, respectively. Palaeocene deposits overlie on the older formations.

The Benue Trough is believed to have been initiated in the early Cretaceous time forming as a split from the Central West African basement during the separation of African and South American continents (break up of Gondwanaland). This break up was followed by the separation of these continents, opening of the south Atlantic, and growth of the Mid Atlantic Ridge (King 1950; Olade, 1975; Benkhelil 1989). Recent evidence from petrographic studies and significant element abundance suggest a passive margin tectonic environment for Benue Trough (Ogungbesan and Akaegbobi 2011).

Within the stratigraphic column of the Benue Trough basin, lie the coal beds and coal-bearing strata that are laterally unstable and range from a few centimetres to about 4 m (Agagu et al. 1985; Sonibare et al. 2008). In the Lower Benue Trough, the coal-bearing Mamu formation is exposed at Okoba, Enugu, Imiegba, Ezimo, Ogboyoga, Orukpa, Udi, on the Enugu—Onitsha Expressway and Agwu—Enugu Escarpment (Ogala 2018). In the Middle Benue Trough, the late Turonian/early Santonian coal-bearing Awgu Formation was exposed at Lafia-Obi. Lastly, the coal-bearing Coniacian—Lower Santonian Lamja Formation in the Upper Benue Trough was exposed at Lamja and Chikila villages while Campanian–Maastrichtian Gombe Formation was exposed at Garin-Maiganga.

3 Materials and methods

3.1 Sampling locations

Representative fresh and unweathered channel coal samples were collected from coal beds in a mine from a broad range of locations. The coals are brownish to black in colour, impermeable and occasionally showed lamination. The coals are sometimes fractured and the thickness ranges from about 0.2 to 1.5 m. At each sampling location, the weathered surfaces of the coal beds were removed before the lithological description and collection of channel samples. The channel samples were placed in zip-lock plastic bags and labelled adequately with a felt-tipped marker pen. The samples were taken to the laboratory within a few days after collection to avoid contamination.

3.2 Sample preparation

The coal samples were prepared according to the procedures described in the ASTM D-2013 standard (ASTM International 2012b). The coal samples were ground using a hammer mill crusher to reduce the particle size below 2.36 mm (mesh size—8), before splitting with a sample riffler. Next, the split was further pulverised and sieved into particle size <850 microns (−20 mesh) using a plate grind mill for petrographic analysis. The remainder of the split was further pulverised (using a pulverizer) and sieved (mesh size—60) into ≤250-micron sized particles for geochemical analysis.

3.3 Proximate, ultimate and calorific analyses

Proximate analysis (moisture, volatile matter, ash yield, and fixed carbon) was determined by thermogravimetric analysis (TGA Model, LECO thermogravimetric analyser, USA). The analyses were performed at the Center for Applied Energy Research, University of Kentucky (CAER, USA) based on procedures outlined in ASTM Standard D7582-12 (ASTM International 2012c). The total carbon and sulfur analyses were performed at the CAER in accordance with the ASTM D4239-12 standard, using a LECO carbon/sulfur analyser (ASTM International 2012b). The ultimate analysis (C, H, N, S, and O) was determined based on ASTM standard D3176-15 test procedures using a LECO CHN analyser (ASTM International 2015). Triplicate analyses were run on the coal samples. The equations for calculating the total mineral matter (MM) (Speight 2012), HHV (Kieseler et al. 2013), and LHV (Basu 2010) are presented in Eqs. (1)–(3).

$$MM = 1.08A + 0.55S \tag{1}$$

$$HHV = 0.4108 \cdot FC + 0.1934 \cdot VM - 0.0211 \cdot Ash \tag{2}$$

$$LHV = HHV - hg(9H/100 + M/100) \tag{3}$$

where, the LHV denotes the lower heating value (MJ/kg); HHV denotes higher heating value (MJ/kg); hg denotes specific heating value of water (2.26 MJ/kg); N is Nitrogen content; H is hydrogen content; M is moisture content; FC is fixed carbon; VM is volatile matter; TS is total sulphur; MM is total mineral matter; A is ash yield.

3.4 Organic petrography

The petrographic composition was determined on the epoxy-bound particulate pellets, prepared to a final 0.05-

Table 1 Proximate and ultimate analyses of Cretaceous coals from Benue Trough Basin (n=3)

Parameter	LBT		MBT		UBT			
	Enugu	Imiegba	Okaba	Lafia-Obi	Chikila	Lamja1	Lamja2	Garin-Maiganga
Proximate analysis								
Moisture (%)	3.17	3.77	8.63	11.59	3.07	2.98	12.91	11.35
Volatile matter. dry (%)	22.45	25.04	43.81	52.08	36.39	36.23	36.79	44.15
Volatile matter. daf (%)	52.65	63.44	52.36	56.70	39.60	39.53	48.95	46.30
Fixed carbon. dry (%)	20.19	15.98	47.77	39.77	55.50	55.41	49.50	51.20
Fixed carbon. daf (%)	47.35	36.56	47.64	43.30	60.40	60.47	51.05	53.70
Ash yield. dry (%)	57.36	58.97	8.42	8.16	8.11	8.36	13.71	4.65
Total sulfur. dry (%)	0.21	0.71	0.81	1.97	0.69	0.69	0.56	0.42
Ultimate analysis (*dry basis)								
Hydrogen (%)	3.15	3.45	5.88	6.98	5.65	5.72	5.73	5.09
Carbon (%)	33.23	27.40	68.65	69.90	76.39	76.48	66.04	63.07
Nitrogen (%)	0.44	0.28	1.39	1.03	1.55	1.53	1.30	0.77
Oxygen (%)	8.88	13.10	24.30	25.08	10.78	10.30	27.49	26.58
O/C	0.27	0.48	0.35	0.36	0.14	0.13	0.42	0.42
H/C	0.09	0.13	0.09	0.10	0.07	0.07	0.09	0.08
VM/FC	1.11	1.57	0.92	1.31	0.66	0.65	0.74	0.86
MM	62.06	64.08	9.54	9.89	9.14	9.41	15.12	5.25
HHV	11.43	10.16	27.92	26.24	29.67	29.59	27.16	29.47
LHV	10.71	9.38	26.53	24.56	28.45	28.36	25.70	28.18

LBT Lower Benue Trough, *MBT* Middle Benue Trough, *UBT* Upper Benue Trough

*dry basis = ultimate analysis were done dry basis

µm alumina polish, using a 50 × reflected-light and oil-immersion objective. Maceral descriptions are based on bituminous nomenclature. Vitrinite reflectance was determined using a photometer system with the incident light polarized at 45° and the light passing across a 546-nm grating.

3.5 Fourier transform infrared-attenuated total reflectance spectroscopic analysis (FTIR-ATR)

The functional group chemistry of the selected Nigerian coals was examined by FTIR-ATR spectroscopy (Shimadzu Prestige-21, Japan). The powdered coal samples from the Lower Benue Trough (LBT), Middle Benue Trough (MBT), and Upper Benue Trough (UBT) were sieved to obtain particles below 250 µm using an analytical laboratory sieve (Retsch™, Germany). Next, precisely 5 mg of the selected coal sample was placed on ZnSe prism plate and scanned to acquire spectra from 4000 to 600 cm^{-1} based on Happ-Genzel Apodization. Each coal sample was scanned 20 times at a resolution of 8 cm^{-1} for a duration of 5 s. After each scan, an ATR correction was applied to obtain the FTIR-ATR spectra. Lastly, the raw data was plotted to obtain the FTIR-ATR spectra by Microsoft Excel.

3.6 Thermogravimetric analysis (TGA)

The thermal decomposition and non-isothermal degradation of the selected coal samples from the Lower Benue Trough (LBT), Middle Benue Trough (MBT), and Upper Benue Trough (UBT) in Nigeria were conducted by thermogravimetric analysis (TGA). About 23–25 mg of sample was placed in an alumina receptacle before heating from room temperature (RT) to 900 °C at a heating rate of 30 °C/min using a Shimadzu TG-50 thermogravimetric analyser (Japan). The TG system was purged with ultra-pure nitrogen (N_2) gas at a flow rate of 20 mL/min to maintain inert conditions and to flush the coal gases evolved during analysis. Upon completion, the TG analyser was cooled to RT, and the raw TGA data files were analysed on a Shimadzu TA-60WS thermal analysis workstation. Based on the recovered ASCII files, the mass loss TG and derivative TG (DTG) data were plotted against temperature to determine the non-isothermal thermal decomposition behaviour and temperature profile characteristics (TPC) of the coals under inert conditions (pyrolysis). The TPCs determined were ignition temperature (T_{onset}), midpoint temperature (T_{mid}), maximum peak decomposition temperature (T_{max}), burnout or offset temperature (T_{off}), mass loss and residual mass (R_M, %).

4 Results and discussion

4.1 Proximate, ultimate, and calorific analyses

The results of the ultimate and proximate analyses of the coal samples are given in Table 1. The average moisture content of coals from Lower Benue Trough (LBT) varies between 3.17% and 8.63% and for the coals from Upper Benue Trough (UBT) it ranges from 2.98% to 12.97%. The Middle Benue Trough (UBT) coal has an average moisture content of 11.59%. The ash yield of LBT coal samples varies between 7.67% and 56.75%, while for the UBT coal samples it ranges from 4.14% to 11.94% (or 7.21% on average). The ash yields of Enugu and Imiegba coal samples are remarkably high. Furthermore, the moisture and ash yield of the studied coals is higher than Polish coals (0.58 wt%, 4.79 wt%) and American coals (1.07 wt%, 5.77 wt%) (Nasirudeen and Jauro 2011). In contrast, the ash yield of Garin Maiganga coal is significantly lower than most Polish or American coals (Nasirudeen and Jauro 2011). The moisture contents of analyzed coals are considerably higher than the South African coal, but lower in ash yield than the South African coal (dry basis=94.43 wt % and wet basis=93.67 wt%) (Akinyemi et al. 2012).

With the exception of the Enugu, Imiegba and Lamja2 coals, the ash contents in the other studied coals are relatively low. The higher ash contents in the Enugu, Imiegba and Lamja2 coals could increase the amount of slag. Low ash yield, typically below 10 wt%, is an essential requirement for good coking coal (Bustin et al. 1985; IEA-OECD 2002). The volatile matter (VM) contents of the LBT coal samples varies between 22.45% and 43.81% (on dry basis), and 52.08% for the MBT coal sample. The VM of the UBT coal samples ranges from 36.23% to 44.15%. The highest VM was observed in the Lafia-Obi coal, followed by Garin-Maiganga coal, and lowest VM of the Enugu coal. The VM of the analyzed coal samples (Except for the Enugu and Imiegba coals) is considerably higher than other Nigerian coals, such as Onyeama (35 wt%), Ogwashi (27.8 wt%), Ezimo (32 wt%), Inyi (30 wt%), and Ogboyoga (30.4 wt%) (Ogala et al. 2012; Sambo 2009; Sonibare et al. 2010). Moreover, the VM of the analyzed coal samples except that of Enugu and Imiegba coals is significantly higher than South African coal (dry basis= 5.6 wt% and wet basis=6.3 wt%) (Akinyemi et al. 2012) and Polish coals (32.61 wt%) (Nasirudeen and Jauro 2011). Conversely, the VM of the studied coals is significantly lower than Hemrin coal seam (60.58 wt%) (Kettanah and Eble 2017).

The contents of C, H, N are consistent with some selected Nigerian coals (Nyakuma et al. 2017; Vassilev et al. 2015). The highest carbon content was observed in the Lamja1 sample, followed by the Chikila coal sample. However, the lowest carbon content coal was obtained from Imiegba. The highest nitrogen content was recorded in the Chikila sample, whereas the lowest was in the Imiegba sample. Hydrogen content is highest in the Lafia-Obi coal, lowest in the Enugu coal. The total sulphur content is highest in the Lafia-Obi coal and lowest in the Enugu coal. The oxygen and hydrogen contents are relatively high compared to nitrogen and total sulphur contents. This observed pattern is in agreement with available data in the literature (Ayinla et al. 2017).

The Okaba, Lafia-Obi, Chikila, Lamja1, Lamja2, and Garin-Maiganga coals with VM greater than 31 wt% have correspondingly high contents of C and H with low mineral matter contents (Table 1). This trend is consistent with high HHV and LHV values for such coal samples. On the other hand, Enugu and Imiegba coals with VM less than 31 wt% show correspondingly low C and H contents. This trend is consistent with high mineral matter content and low HHV and LHV contents of Enugu and Imiegba coals.

Coal rank classification was determined by proximate analysis according to ASTM standard D388 (ASTM International 2012a). Coals with volatile matter (VM) contents higher than 31 wt% are typically classified according to calorific or higher heating value (HHV) (Speight 2012). Based on this, the Lamja1, Lamja2, and Chikila coals could be classified as high volatile B (HVB) bituminous coal and the Garin-Maiganga, Lafia-Obi, and Okaba coals as sub-bituminous coals. However, an earlier study by Jauro et al. (2008a, b) has classified the Lafia Obi coal as high volatile A bituminous coal. Consequently, the Lamja1 and Chikila coals can be possibly exploited for various energy applications. Nonetheless, the Garin-Maiganga, Lamja2, Lafia-Obi, and Okaba coals are suitable for cement production and electric power generation.

4.2 Organic petrography

The vitrinite reflectance of Lafia-Obi coal (≈0.39%R_{max}) and Garin-Maiganga coal (≈0.39%R_{max}) classifies the samples in the subbituminous rank. The vitrinite reflectance obtained from Lamja1 (≈0.73%R_{max}), Lamja2 (≈ 0.72%R_{max}), and Chikila (≈0.71%R_{max}) coals fall within the high volatile B bituminous rank (Table 2). The vitrinite reflectance of Imiegba (≈0.45%R_{max}) and Okaba (≈0.49% R_{max}) coals classified the samples in the subbituminous rank. The vitrinite reflectance of Enugu coal (≈0.55% R_{max}), falls on the boundary between subbituminous and high volatile C bituminous.

The macerals in Cretaceous coals from the Benue Trough Basin (Figs. 10, 11, 12, 13, 14) are mainly vitrinite and inertinite. The vitrinite content is higher than inertinite, despite the small quantities of liptinite and mineral matter

Table 2 Maceral composition (mineral matter free basis, mmf) and mean reflectance of Cretaceous coals from the Benue Trough

Macerals (mmf)	LBT		MBT		UBT			
	Enugu	Imiegba	Okaba	Lafia-Obi	Chikila	Garin-Maiganga	Lamja1	Lamja2
Telinite	18.06	31.96	8.27	9.20	10.40	15.81	8.77	23.74
Collotelinite	8.80	10.65	19.55	22.24	64.83	8.09	71.72	47.49
Total telovitrinite	26.86	42.62	27.82	31.45	75.23	23.90	80.48	71.23
Vitrodetrinite	21.30	9.77	25.19	26.08	11.44	16.91	6.77	10.06
Collodetrinite	0.00	0.00	0.38	0.00	0.00	0.00	0.00	0.00
Total detrovitrinite	21.30	9.77	25.56	26.08	11.44	16.91	6.77	10.06
Corpogelinite	5.09	4.44	3.76	3.84	3.12	2.21	0.40	3.22
Gelinite	0.00	0.00	1.50	3.07	0.35	0.74	0.00	0.00
Total gelovitrinite	5.09	4.44	5.26	6.90	3.47	2.94	0.40	3.22
Total vitrinite	53.26	56.83	58.65	64.43	90.13	43.75	87.65	84.51
Fusinite	7.87	2.66	23.68	12.66	4.51	37.50	5.98	10.06
Semifusinite	4.17	2.66	9.02	2.30	0.69	15.07	0.40	0.40
Micrinite	0.00	0.00	0.00	0.38	0.00	1.10	0.00	0.00
Macrinite	0.46	0.00	0.00	0.38	0.69	0.00	0.80	0.00
Funginite	0.00	0.00	0.38	0.38	0.35	0.00	0.40	2.01
Inertodetrinite	0.00	0.00	0.38	1.15	0.00	0.00	0.00	0.00
Total inertinite	12.50	5.33	33.46	17.26	6.24	53.68	7.57	12.48
Sporinite	6.48	0.00	3.01	4.99	0.69	1.10	2.39	0.80
Cutinite	0.93	0.00	1.50	0.00	1.73	0.74	1.59	1.21
Resinite	6.48	0.00	2.63	10.35	0.00	0.74	0.00	0.40
Suberinite	0.00	0.00	0.38	0.00	0.00	0.00	0.00	0.00
Total liptinite	13.89	0.00	7.52	15.34	2.43	2.57	3.98	2.41
Silicate	37.24	75.00	0.75	3.35	2.40	0.00	1.58	0.80
Sulfide	0.34	0.35	0.00	1.86	0.00	0.00	0.00	0.40
Other	3.10	0.35	0.00	0.74	0.00	0.00	0.00	0.00
Total mineral	40.69	75.69	0.75	5.95	2.40	0.00	1.58	1.20
$R°_{max}$ (%)	0.55	0.45	0.49	0.39	0.71	0.39	0.73	0.72
Stdev.	0.05	0.05	0.03	0.02	0.04	0.03	0.03	0.03
$R°_{random}$ (%)	0.52	0.42	0.45	0.35	0.66	0.35	0.67	0.69
Stdev.	0.04	0.04	0.04	0.03	0.05	0.03	0.05	0.04

(Table 2). The total vitrinite content shows the highest abundance in the UBT coals followed by the LBT coals and lowest content for the MBT coal. The vitrinite group (Fig. 4) signifies woody plant material (e.g., stems, trunks, roots, and branches), resulting from lignin and cellulose of plant tissues. The woody and herbaceous cell tissue (lignin and cellulose) form macerals of the vitrinite group. The vitrinite macerals were formed under anaerobic (high hydrogen content) and mostly reducing conditions. The vitrinite group are dominated by telinite, collotelinite (Fig. 4), and detrovitrinite, which are smaller fragments, showing a higher degree of degradation and generally forming the groundmass for other macerals. The vitrinite in the Lamja2 and Imiegba coals are dominated by telinite and collotelinite (telovitrinite), which are intact compressed fragments of woody material of varying degree of compaction. Vitrinite is fairly abundant in all the investigated coal samples and shows the highest content in the Chikila coal, followed by the Lamja1 coal, and least in the Garin-Maiganga coal. The mineral contents in the Enugu and Imiegba (LBT) coals are high compared to other studied coal samples. The argillaceous minerals matter and other mineral groups mostly observed are; silicates, clay minerals, pyrites and jarosite formed by chemical precipitation (Diessel 1992; Singh et al. 2013, 2015; Gangadharan et al. Gangadharan et al. 2019).

Inertinites are mainly oxidation products of other macerals and are consequently richer in carbon than liptinites or vitrinites. The highest concentration of inertinite is recorded in coals from the Upper Benue Trough (UBT)

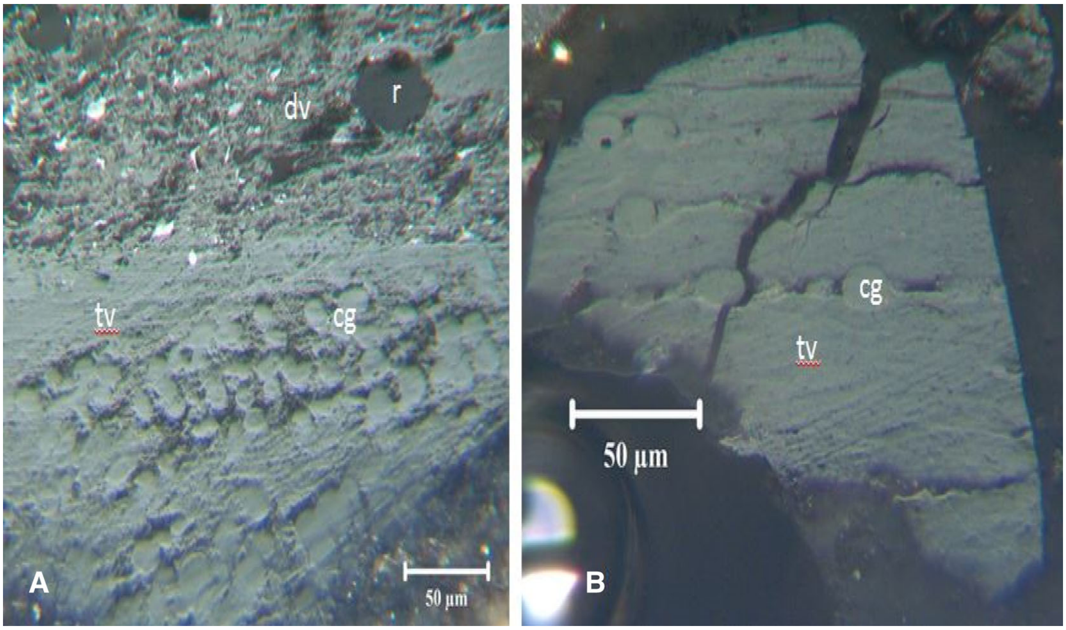

Fig. 4 Vitrinite (a) Corpogelinite (cg) with telovitrinite (tv) and a mix of resinite (r), detrovitrinite, and other detrital macerals (b) Corpogelinite (cg) with telovitrinite (tv)

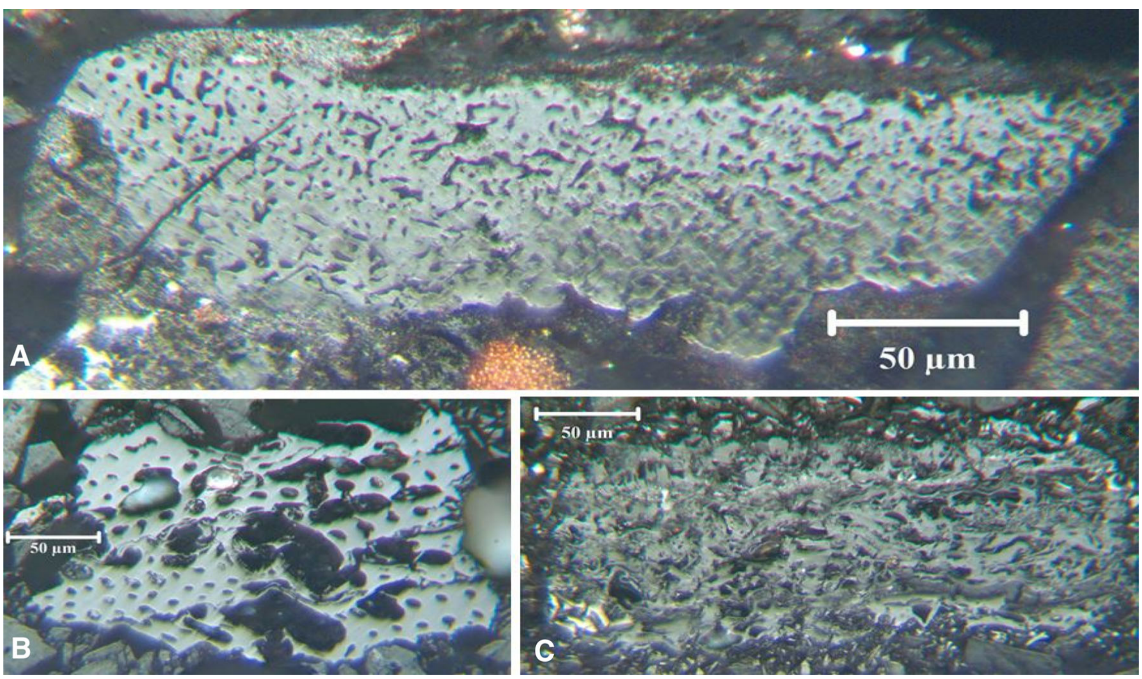

Fig. 5 Fusinite and semifusinite **a** degraded fusinite showing "shotgun" pattern suggestive of fungal attack **b** degrade fusinite showing swollen cell walls **c** broken semifusinite walls with some brighter fusinite (lower left)

followed by the coals from Lower Benue Trough (LBT), and lowest inertinite contents are obtained in the coal from Middle Benue Trough (MBT). The highest inertinite content is recorded in the Garin-Maiganga coal (53.68%), followed by the Lafia-Obi coal, and minor quantities occur in other coal samples. The inertinite macerals are dominated by fusinite and semifusinite (Fig. 5). Fusinite and semifusinite are, in part, derived from ancient fires in the coal-forming peat (Scott 1998). Macrinite is obviously present in the Enugu, Lafia-Obi, Chikila, and Lamja1 coals. Macrinite proved to be a crucial diagnostic maceral in a previous study of Nigerian Cretaceous coals (Hower et al. 1998). Macrinite originated from the biological decomposition and decay of plant material. Micrinite characterised

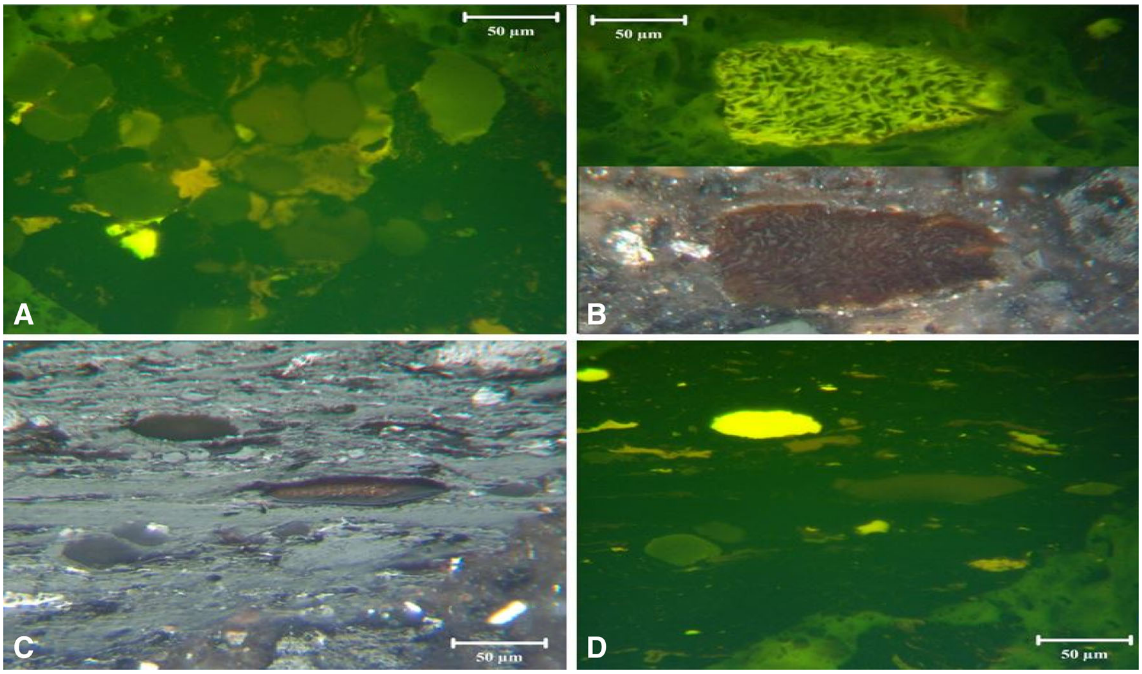

Fig. 6 Resinite **a** blue-light image of resinite **b** blue-light/white-light images of resinite **c**, **d** blue-light and white-light images of resinite

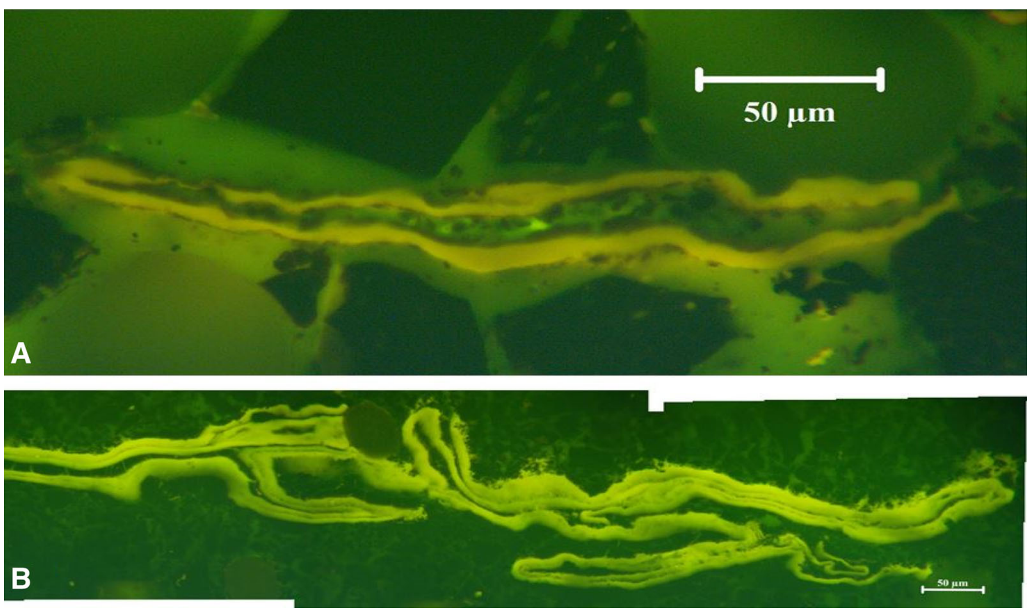

Fig. 7 Sporinite **a** blue-light images of sporinite **b** sporinite

the Lafia-Obi and Garin Maiganga coals, which are formed from thermal maturation of peat. The relatively high abundance of inertinite maceral group content suggests that the coal deposition occurred at shallow depths under aerobic condition with frequent exposure of plant debris (Chaudhuri et al. 2001).

The highest abundance of liptinites is recorded in the LBT coals, followed by MBT coal, and the least concentration is observed in the UBT coals. Liptinite shows the highest content in the Lafia-Obi coal, followed by the Enugu coal, with the least abundance being recorded in the Lamja2 coal samples. Liptinites are derived from spores, pollens, cuticles, and resins in the original plant material. The liptinite group of macerals is enriched in hydrogen compared to the vitrinite and inertinite groups. Liptinite macerals are also highly reactive due to the higher volatile content, which contribute more to the by-products. Liptinites (Figs. 6, 7, 8) occur in variable amounts, rising above 10% in the Lafia-Obi and Enugu coals. Resinite largely accounts for the liptinite in the Lafia-Obi coal, is co-equal with sporinite in the Enugu coal and nearly equal to sporinite in the Okaba coal (total liptinite of 7.5%, on the mineral-free basis). The liptinite distribution pattern could be attributed to changes in vegetation and freshwater conditions in the depositional environment (Ao et al. 2012). The relatively high vitrinite and low inertinite in the coal seam from Benue Trough indicates that anaerobic condition of deposition i.e. high water content or anoxic condition prevailed during early peat stage development of the peat swamp. Maceral of the studied coal samples trends from fluvial to lacustrine condition during peat swamp development, which indicates that the coals were deposited under the fluvio-lacustrine environment.

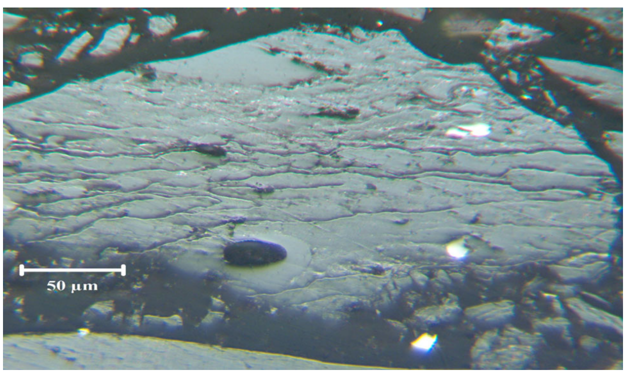

Fig. 8 Cutinite, thin cell wall, fine and bent as a wire, dispersed in degradinite

4.3 Depositional environment

The petrographic composition of studied coal seams can be used to reconstruct the paleoenvironment of the coal formation in the Benue Trough Basin. Some authors (Grady et al. 1993; Obaje et al. 1994; Shearer and Clarkson 1998) have demonstrated the influence of the paleodepositional environment on the quantifiable dissemination of macerals in the coal seams, predominantly in relation to time. The abundance of vitrinite (telinite and Collotelinite) and inertinite (fusinite and semifusinite) in the studied coal samples particularly suggests deposition in wet forest swamp sub-environments along within lagoons. The observed relative abundance of inertinite maceral in the Okaba and Garin-Maiganga coals could also be attributed to dryness experienced by these sections of the basin (Singh et al. 2013; Rajak et al. 2019). This dryness as indicated by elevated inertinite content is ascribed to regression episode that occurred during the first tectonic phase or local fluctuation in the water level. The relative abundance of mineral matter in Enugu and Imiegha (LBT) coals suggests that the Lower Benue Trough section of the basin was inundated with clastic marsh depositional environment.

4.4 FTIR-ATR analysis

FTIR-ATR technique is used in this study to determine the chemical structure and composition of the coal samples. The FTIR spectra for the coal samples from the Upper Benue Trough (UBT), i.e., Chikila (CHK), Garin Maiganga

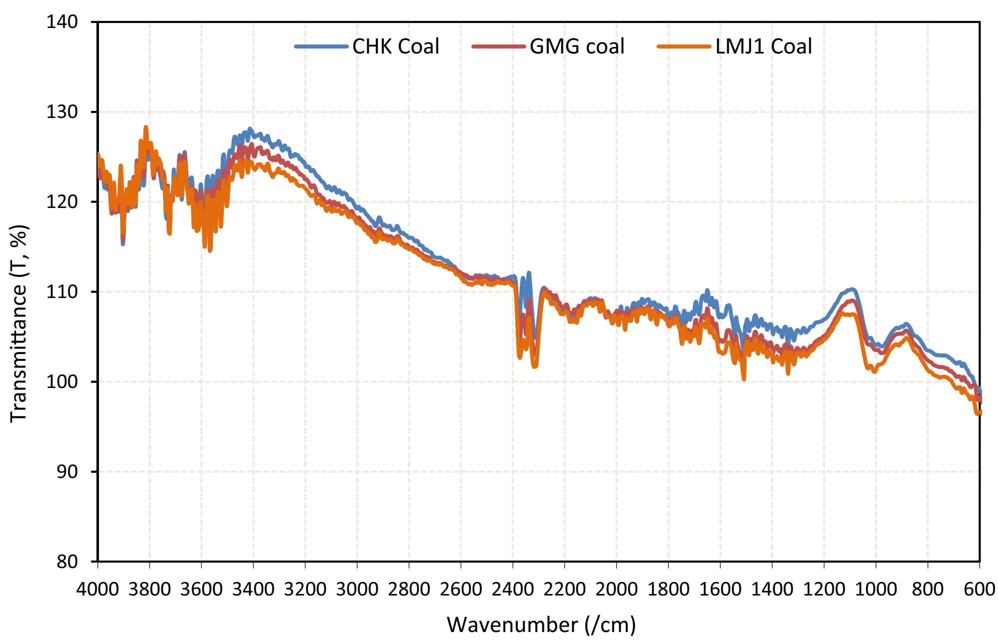

Fig. 9 FTIR of Upper Benue Trough (UBT) coals

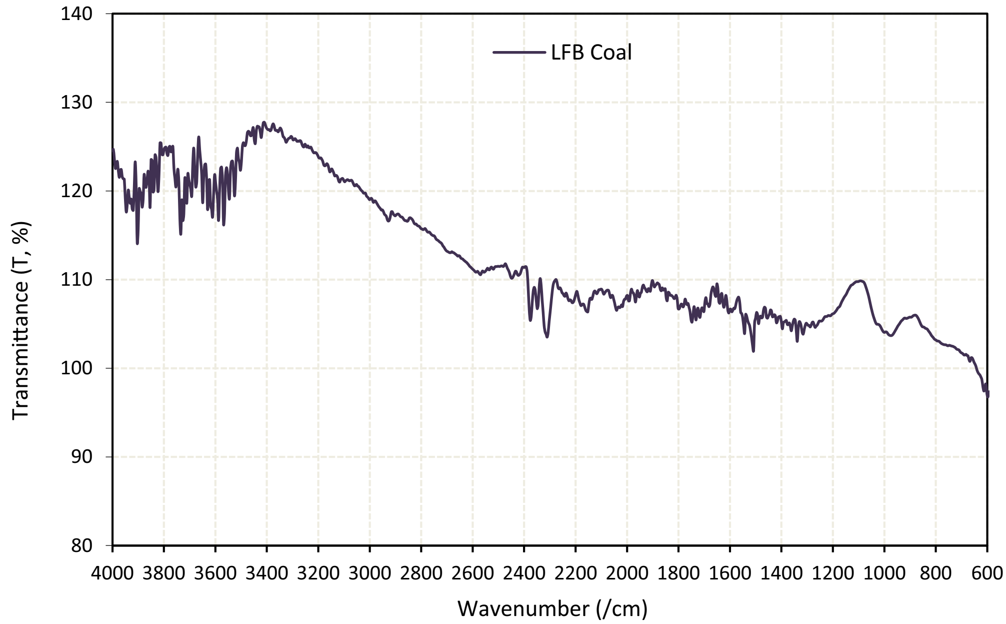

Fig. 10 FTIR of Middle Benue Trough (MBT) coal

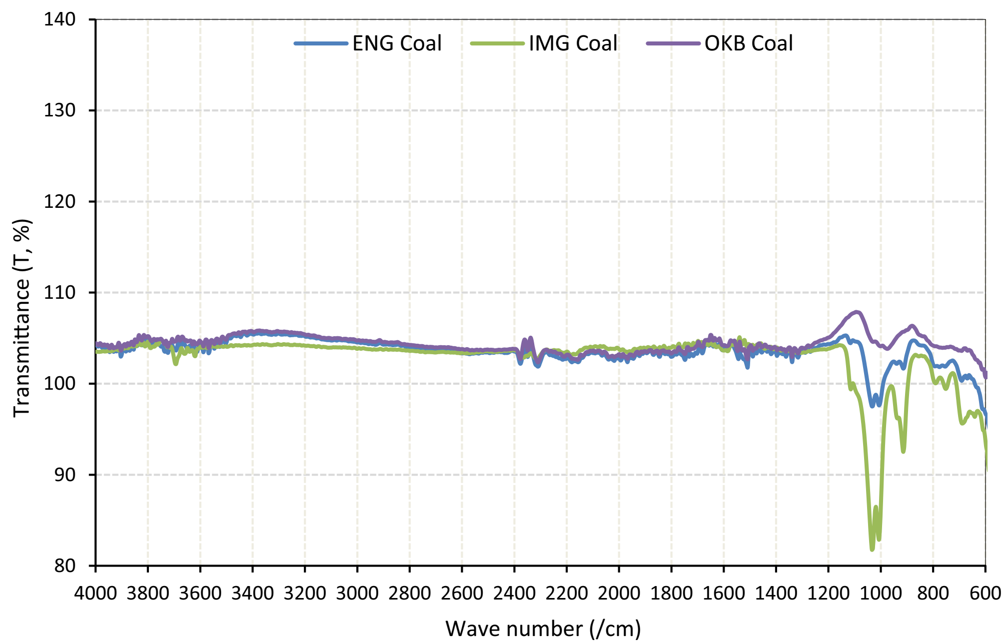

Fig. 11 FTIR of Lower Benue Trough (LBT) coal

(GMG), and Lamja1 (LMJ) are presented in Fig. 9, the Lafia-Obi (LFB) coal from the Middle Benue trough (MBT) in Fig. 10, and the Enugu (ENG), Imiegba (IMG) and Okaba (OKB) from the Lower Benue Trough (LBT) in Fig. 11. Several FT-IR peaks of variable intensities occur that can be ascribed to various organic functional groups.

The medium to sharp peaks observed between 2400 and 2200 cm^{-1} can be ascribed to the asymmetric stretching vibrations of the isocyanate (C=N=O) group. The peaks were more prominent for the UBT coals, i.e., the CHK, GMG, and LMJ1. However, the intensities declined for ENG, IMG, and OKB coals (LBT coals). The medium peak at 1600 cm^{-1} can be assigned to hydrogen-bonded C=O or non-chelated quinoid carbonyl groups (Basaran et al. 2003). Other studies have assigned the peak to the aromatic C=C stretching (Georgakopoulos et al. 2003; Balachandran 2014), which is caused by strong absorptions due to the high carbon content in coals (Balachandran 2014). The

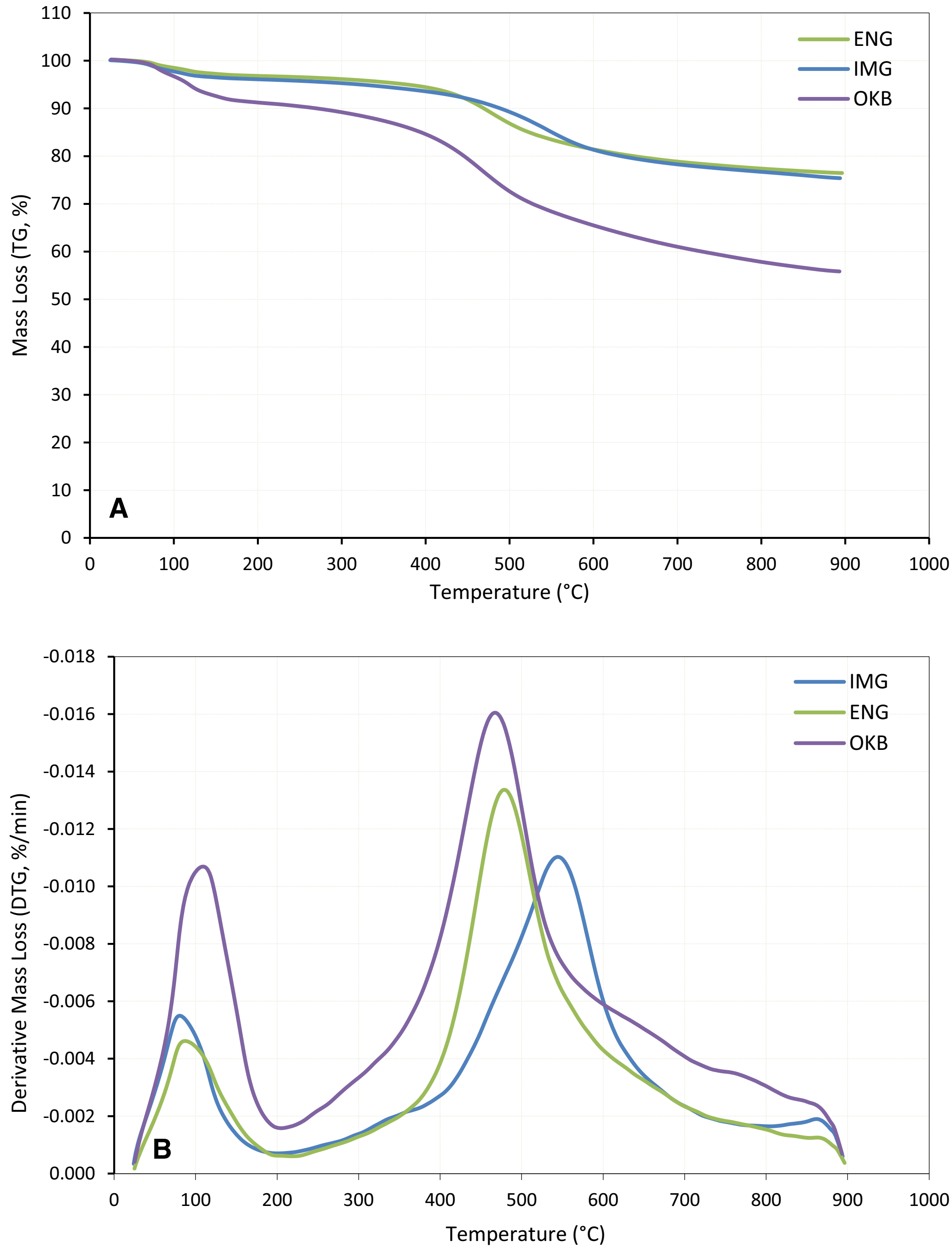

Fig. 12 **a** Pyrolysis—TG plots of LBT coals. **b** Pyrolysis—DTG plots for LBT coals

intensity of this peak is an indicator of the coal rank (Speight 2012). The peaks or bands observed at 1500 cm^{-1}, typically present in low-rank coals (Balachandran 2014), can be ascribed to condensed aromatic rings C=C.

However, the peaks at 1400 cm^{-1} and 1300 cm^{-1} are due to aliphatic and cyclic CH_2 and CH_3 groups. The absorption bands between 800 and 600 cm^{-1} are due to the hydrogen atoms on the substituted aromatic rings. This could also be due to the out-of-plane vibrations of aromatic CH groups, which are an indication of the degree of condensation of aromatic clusters in coals (Speight 2012). The sharp peaks observed between 4000 and 3400 cm^{-1} are due to the rota-vibrational bands of water vapour (Georgakopoulos et al. 2003; Zhang et al. 2015), indicating that inherent water molecules or characteristic residual moisture were included in the coals.

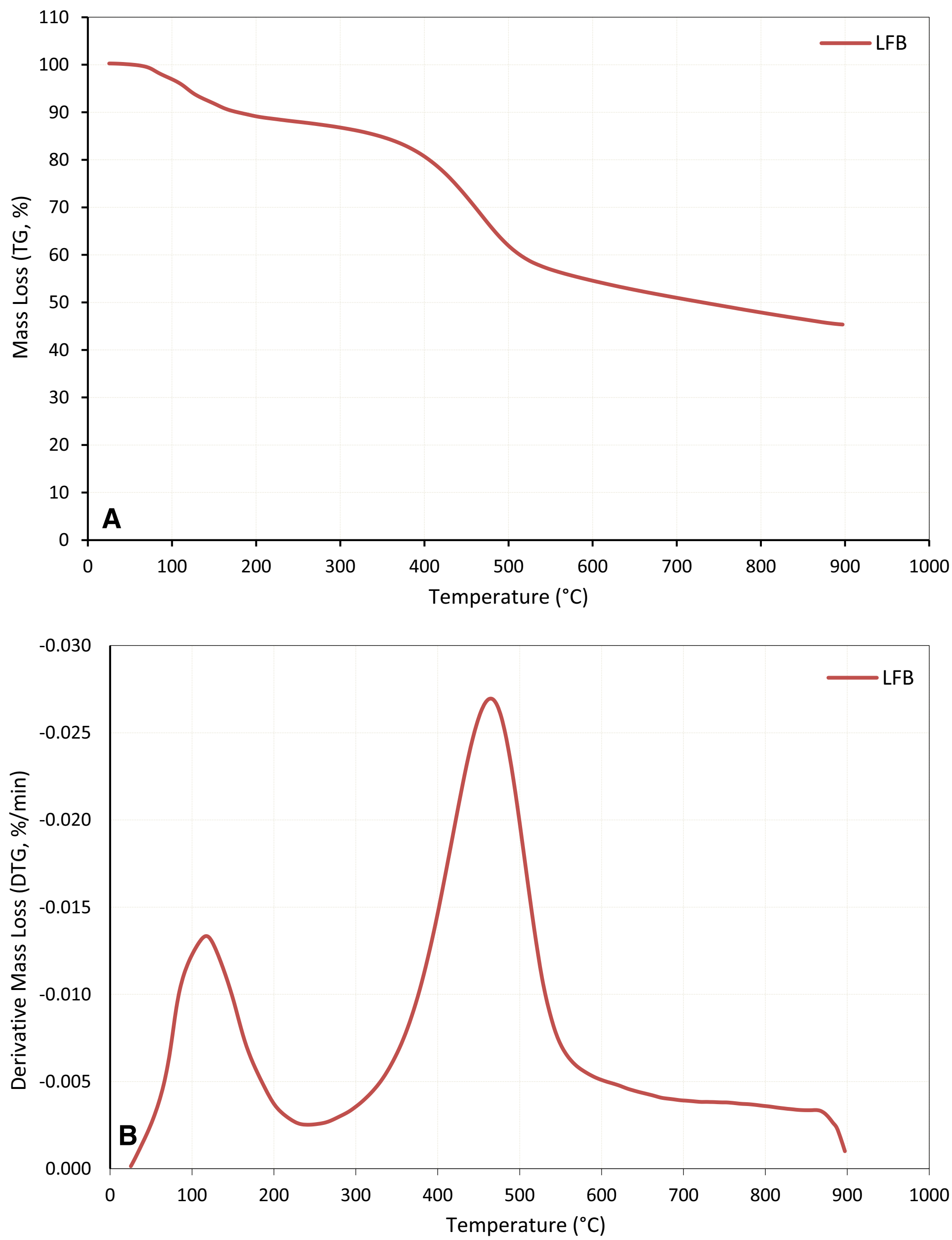

Fig. 13 **a** Pyrolysis—TG plots for MBT coal. **b** Pyrolysis—DTG plots for MBT coal

4.5 TGA pyrolysis

The TG-DTG plots for the non-isothermal thermal degradation of the selected LBT, MBT and UBT coals in Nigeria are presented in Figs. 12, 13 and 14. Figure 12a presents the TG plots for the LBT coals; Enugu (ENG), Imiegba (IMG), and Okaba (OKB). Figure 13a presents the TG plots for the MBT coal; Lafia-Obi (LFB). Lastly, the TG plots for the thermal decomposition of the UBT coals; Chikila (CHK), Garin Maiganga (GMG), and Lamja1 (LMJ) are presented in Fig. 9a. The TG plots exhibited downward sloping curves, indicating the increase in temperature from RT to 900 °C resulting in a steady thermal degradation as denoted by the mass loss (M_L, %), residual mass (R_M, %), and temperature profile characteristics (TPC) as presented in Table 3.

The mass loss for the UBT coals, i.e., CHK, GMG, and LMJ, was from 32.39% to 49.32%. The highest mass loss (49.32%) was observed for the GMG, indicating that it is the most reactive coal sample. The CHK coal is the least reactive with a mass loss of 32.39%. Consequently, the residual mass of the coals were 67.61% for CHK, 50.68%

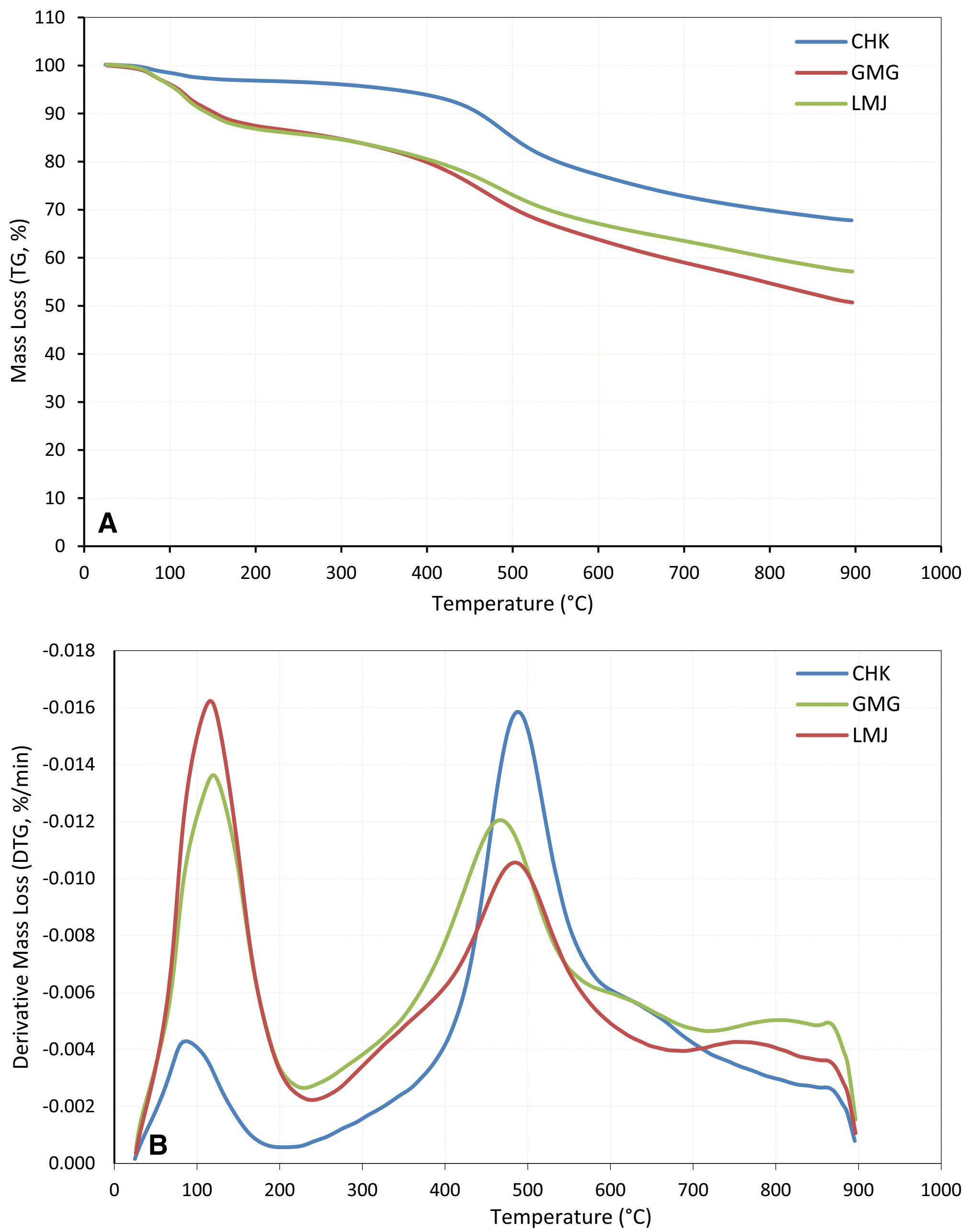

Fig. 14 **a** Pyrolysis—TG plots for the UBT coals. **b** Pyrolysis—DTG plots for UBT coals

for GMG, and 56.98% for LMJ1. The M_L and R_M for LFB were 54.93% and 45.07%, respectively. This indicates that LFB is less thermally stable and, hence, more reactive than the UBT coals. The M_L and R_M for the LBT coals, i.e., ENG, IMG, and OKB ranged from 23.72% to 44.42%, and 55.58% to 76.28%, respectively.

The highest mass loss (44.42%) was observed for the OKB coal, whereas the lowest mass loss (23.72%) was observed for the ENG coal. The results indicate that the OKB coal is most reactive, or least thermally stable coal, examined whereas the ENG coal is the least reactive, or most thermally stable. In general, the results demonstrate that, on average, the mass loss and residual mass for the coals examined are 37.31% and 62.69%, respectively. Lastly, the results indicate that higher temperatures above 900 °C are required to decompose the coals for efficient energy recovery.

The thermal degradation and chemical reactivity of the coals during the pyrolysis thermal decomposition was also examined through the TPCs. The T_{onset}, for the UBT coals, ranged from 77.86 °C (LMJ) to 393.67 °C (CHK), the T_{mid} from 362.60 °C (LMJ) to 498.25 °C (CHK), and the T_{off}

Table 3 Thermogravimetric plot data

Coal samples	Region of origin	Onset temp (T_{onset}, °C)	Midpoint temp. (T_{mid}, °C)	Offset temp. (T_{off}, °C)	Mass loss (M_L, %)	Residual mass (R_M, %)
CHK	UBT	393.67	498.25	612.43	32.39	67.61
GMG	UBT	81.38	401.95	421.50	49.32	50.68
LMJ[1]	UBT	77.86	362.60	310.70	43.03	56.98
LFB	MBT	327.15	437.97	558.05	54.93	45.07
ENG	LBT	381.78	479.44	589.90	23.72	76.28
IMG	LBT	408.88	509.27	638.39	24.72	75.28
OKB	LBT	317.71	451.23	604.54	44.42	55.58

Table 4 Derivative Thermogravimetric Data

Coal samples	Region of origin	Drying peak T_{dry} (°C)	Decomposition peak T_{max} (°C)
CHK	UBT	88.32	485.94
GMG	UBT	119.85	467.26
LMJ[1]	UBT	115.72	482.09
LFB	MBT	116.62	462.72
ENG	LBT	89.13	476.87
IMG	LBT	81.62	544.16
OKB	LBT	109.21	468.93

from 310.70 °C (LMJ[1]) to 612.43 °C (CHK) (Table 2), confirming that the CHK is the least reactive UBT coal under the conditions examined in this study. The characteristic temperature profiles for the MBT coal LFB were T_{onset}, (327.15 °C), T_{mid} (437.97 °C), and T_{off} (558.05 °C). The values are higher than reported for the UBT coals GMG and LMJ[1] but somewhat comparable to the CHK.

The characteristic temperature profiles, e.g., T_{onset}, T_{mid}, and T_{off} for LBT coals, i.e., ENG, IMG, and OKB, were also examined. The values for T_{onset}, ranged from 317.71 to 408.88 °C, T_{mid} from 451.23 to 509.27 °C, and T_{off} from 589.90 to 638.39 °C. The lowest T_{onset}, was observed for the OKB coal whereas the highest was for the IMG coal. Likewise, for T_{mid}, the lowest value was observed for the OKB coal and the highest was observed for the IMG coal. The lowest T_{off} was observed for the ENG coal and the highest was for the IMG coal.

The DTG plots for the pyrolytic decomposition of the UBT, MBT, and LBT coals are presented in Figs. 12b, 13b, and 14b, respectively. The plots are characterised by two sets of endothermic peaks in the regions; RT—250 °C and 250–700 °C, indicating that the thermal decomposition of the coals occurs in three stages: I—drying, II—devolatilization, and III—coke formation above 700 °C.

Stage I is typically characterised by the loss of moisture and low molecular weight volatile compounds in the coal. However, stage II is due to the loss of high molecular weight constituents of coal, resulting in the production of condensable and non-condensable products. The last stage III denoted by the tailing observed after 700 °C for the coals may be ascribed to coke formation and other gas–solid products. The weight loss during stages I and II are characterised by maximum decomposition peaks denoted as the Drying Peak (T_{dry}) and Decomposition Peak (T_{max}) as presented in Table 4.

As observed in Table 3, the T_{dry} and T_{max} values for the UBT coals ranged from 88.32 to 119.85 °C and from 467.26 to 485.94 °C, respectively. Based on the results, the highest T_{dry} peak was observed for the GMG coal, corresponding to its high moisture content compared to the CHK coal, which has the lowest T_{dry} peak value (Table 1). The highest T_{max} value was observed for the CHK coal whereas the lowest was for the GMG coal. The T_{dry} and T_{max} values for the MBT and LFB coals are 116.62 °C and 462.72 °C, respectively, within the range observed for the UBT coals. Lastly, the T_{dry} and T_{max} for the LBT coals ranged from 81.62 to 109.21 °C and 468.93 to 544.16 °C.

5 Conclusions

This study conducted the physicochemical analysis, maceral analysis, FTIR-ATR analysis, and thermogravimetric analysis of the Cretaceous coal from Benue Trough sedimentary basin, Nigeria. The study revealed findings on the rank classification, deposition environment, thermal decomposition and reactivity. The following conclusions can be made:

(1) Based on vitrinite reflectance, GMG, IMG, and OKB coals are categorised as subbituminous rank, while LMJ1, LMJ2 and CHK coals are identified as high volatile B bituminous. The Enugu (ENG) coal is classified as high volatile C bituminous.

(2) The vitrinite and fusinite contents show a gradual increase from LBT coals to the UBT coals. Semi-fusinite and total mineral matter follow a contrary pattern.

(3) Relatively high vitrinite and low inertinite contents indicate the anaerobic condition of deposition and maceral trend show deposition under fluvio lacustrine environment.

(4) Thermal decomposition occurred in three stages, i.e., drying, devolatilization, and coke formation (above 700 °C). Therefore, high temperature above 900 °C is required to decompose the coals for efficient energy recovery.

(5) The order of reactivity of studied coals is as follows: LFB > GMG > OKB > LMJ > CHK > IMG > ENG.
(6) Based on the volatile matter and higher heating value, the Garin-Maiganga, Lamja2, Lafia-Obi Okaba, Lamja1, and Chikila coals can be possibly exploited for various energy applications. However, the Enugu and Imiegba coals would be best suited for cement and power electricity generation. Nevertheless, extensive tests are required to confirm their intrinsic potentials for application in the abovementioned technologies.

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