Organic petrography, Rock–Eval pyrolysis and biomarker geochemistry of Maastrichtian Gombe Formation, Gongola Basin, Nigeria

Ayoola Y. Jimoh1 · Olusola J. Ojo2 · Samuel O. Akande3

Received: 18 February 2019 / Accepted: 23 August 2019 / Published online: 31 August 2019
© The Author(s) 2019

Abstract
The Maastrichtian Gombe Formation is located within one of the inland basins of Nigeria (Gongola Basin). Surface and borehole samples of coal and interbedded shale of this formation were evaluated to determine the organic matter richness, quality, maturity, source and depositional environment through organic geochemical techniques such as total organic carbon (TOC) determination, Rock–Eval pyrolysis, chromatography and organic petrography. The genetic potential and average TOC value of the coals, shales and coaly shales are 38.03 wt%, 3.14 wt% and 12.1 wt%, respectively, indicating moderate-to-good source potential. HIs are less than 200 mgHC/gTOC for all the samples indicating Type III kerogen for most of the samples (gas prone) and plant contributions from terrestrial sources. The source rocks were characterized by high molecular weight n-alkanes which range from \( \text{C}_{10} \text{ to } \text{C}_{41} \) in the coals and \( \text{C}_{9} \text{ to } \text{C}_{41} \) in the shales. The n-alkanes distributions in the coals maximize at \( \text{C}_{29} \) indicating terrestrial sourced organic matter, while the shales maximize at \( \text{C}_{16} \text{ and } \text{C}_{29} \) indicating mixed sources. \( \text{Pr/Ph} \) of the coals and shales ranges from (2.15–3.51) to (0.95–2.37), respectively, suggesting fluvio-deltaic deltaic and coastal swamp depositional environments. The predominance of pristane over phytane suggests humic origin of the organic matter. The sterane/hopane values (< 0.6) indicate incorporation of bacterial inputs commonly associated with terrigenous organic matter in the coals. The CPI is greater than unity, \( T_{\text{max}} \) is generally < 435 °C and vitrinite reflectance less than 0.55 Ro for most of the samples and suggest the samples are of low maturity status to expel liquid hydrocarbon.

Keywords Humic · Phytane · Maastrichtian · Kerogen

Introduction
The assessments of hydrocarbon accumulation in a petroleum system are subject to accurate characterization of oil source potential through various analytical techniques such as pyrolysis, elemental, chromatography and spectrosopy. Hydrocarbon prospectivity and research are still very active in the all the Cretaceous basins in Nigeria including the Northern Benue Trough (Fig. 1) in line with the Government extant policy of increasing the hydrocarbon reserve in the country. The investigated source rock interval of the Maastrichtian Gombe Formation is located at the Maiganga coal mine, near Gombe, Gongola Basin, Nigeria. Previous workers in the study area (Onoduku 2013; Jimoh and Ojo 2016) have applied preliminary organic geochemical screening methods such as TOC and Rock–Eval pyrolysis to characterize the source rock potential in the study area, and due to limitation of these techniques, a further probe was considered necessary. Also, there are no detailed organic geochemical investigations on the coal and shaly source rocks in respect of biomarker characterization. Hence, the palaeodepositional environments, source, type and maturity of the organic matter in the source beds were investigated in this study based on Rock–Eval pyrolysis and biomarker techniques.
Geological setting and stratigraphy

The Benue Trough is one of the inland sedimentary basins of Nigeria formed by a rifting process (Olade 1975). The trough trends structurally in NNE-SSW direction and extends up to 800 km in length and 150 km in width (Fig. 1). The sediment fill is more than 6000 m of Cretaceous–Tertiary age, and those predating the mid-Santonian have been deformed, faulted and uplifted in several places (Benkhelil 1989). The Benue Trough and its peculiar structural styles (Fig. 2) are believed to be vestiges of the fragmentations and the separation of South America from Africa in the Carboniferous and opening of the South Atlantic through the process of Sea floor spreading consequent to continental up-doming and rifting (Wright 1968; Olade 1975). The Trough is partitioned into southern, central and northern section (Fig. 1). The northern section is further subdivided into Gongola and Yola basins. The stratigraphy of the Gongola Basin consists of the basement rocks, Albian Bima Formation (feldspathic sandstones), Cenomanian Yolde Formation (sandstones, shales and limestones), Turonian Pindiga Formation (shales and fossiliferous limestones), Maastrichtian Gombe Formation (sandstones, siltstones, shales, ironstones and coal seams), Tertiary Kerri–Kerri Formation (sandstones) and the alluvium (Whiteman 1982) (Fig. 3). At Maiganga, the exposed part of the mine was logged and sampled. The lithologic succession consists of about 35 m thick coarsening upward section with the basal part made up of coal and shale interbeds, while the upper part consists of siltstone and sandstone (Figs. 3, 4). The coal and interbedded shale at phases I and II of the mine appear most prospective source rock facies in the formation and were evaluated with respect to their hydrocarbon source rock potential.
Methodology

Sampling and logging was carried out at phases I and II of the exposed part of the mine (Figs. 4, 5) based on vertical and lateral variation of the facies. A borehole was also sampled and logged to determine any variation in organic matter preservation with respect to TOC and S1 with the surface shale samples and to also confirm if there is weathering or impurities compared to the surface cuttings (Fig. 6). The coal seams were divided into seams A and B. Seam A was further divided into A1, A2 and A3. The coals seams and shaly intervals have varying
thickness in the two phases. At phase I, the coal seam B was sampled at 0.5 m, the shale at 0.3 m in order to have a representative samples across the section and to acquire all the geologic details in the section. At phase II, seam B coal and shale was sampled at 0.4 m and 0.3 m, respectively. Only three shale samples from the borehole were analysed. The samples were selected and subjected to various organic geochemical techniques such as TOC, Rock–Eval pyrolysis, Soxhlet extraction, gas chromatography, medium performance liquid chromatography and gas chromatography–mass spectroscopy. All the analyses were carried out at Stratochem Laboratory, Canada.

A total of twenty-nine samples (26 surface cuttings and 3 borehole samples) consisting of coal, coaly shale and shale were pulverized and weighed. Hydrochloric acid was added to the weighed samples to remove carbonates and subjected to TOC screening using LECO C230 analyser. The free hydrocarbon ($S_1$), hydrocarbon generated by thermal cracking of the kerogen ($S_2$) and the maximum temperature of the kerogen decomposition ($T_{\text{max}}$) were determined by Rock–Eval 6 instrument. Other parameters such as production index (PI), hydrogen index (HI) and genetic potential (GP) were derived from Rock to Eval pyrolysis data. Seven samples were extracted by Soxhlet method to obtain the extractable organic matter (EOM). Fresh samples were pulverized and weighed; each sample was placed in a thimble, dichloromethane and methanol was added to a conical flask in ratio 93:7. The thimble was placed in the Soxhlet apparatus and the samples extracted for seventy-two hours (72 h) after which the solvent was evaporated.

The extracts were separated into saturate, aromatic and the polar fraction by medium performance liquid chromatography containing thermally deactivated silica, in which the main column of activated silica is the stationary phase.
with n-hexane as the mobile phase. The extracts were first deasphalted by adding n-pentane to the extract. The n-pentane dissolves the maltene fractions (saturates, aromatics and NSO) and then agitated and centrifuged to make sure all the maltene is dissolved in the n-pentane and the asphaltenes precipitated. The saturated and aromatic hydrocarbon fraction were analysed by gas chromatography–mass spectroscopy by trace 1310 gas chromatograph with ISQ

Fig. 4 Lithologic succession of Gombe Formation at the Maiganga mine (Phase I). The succession coarsens upward from coal, shale, siltstone to sandstone. The arrows indicate the selected and analysed samples. Note the coordinates: 10°02′39″N, 11°12′17″E
LT single-quadrupole mass spectrometer and Agilent 7890 B gas chromatograph with a 5977 A mass spectrometer detector. The saturate fraction was weighed into a 2 ml sample vial, choline and cyclohexane was added and injected into the GC–MS machine, while the same procedure was repeated for aromatic except that orthoterphenyl (OTP) was added to serve as internal standard for the GC–MS analysis.

The organic petrography of the samples was determined by reflected light microscopy on polished specimens made from crushed samples mounted in epoxy resin. The polished briquettes were prepared from crushed 1-mm size samples (coal and shale) and mounted in epoxy resin. Ordinary white reflected light from a tungsten lamp and violet-blue light from a high-pressure mercury lamp were used to initiate...

**Fig. 5** Lithologic succession of Gombe Formation at Maiganga mine (Phase II). The succession coarsens upward from coal, shale, coaly shale. The arrows indicate the selected and analysed samples. Coordinates: (10°04′46″N, 11°12′23″E)
fluorescence. Maceral observation was carried out on a Leitz MPV-2 photomicroscope. The composition of maceral was determined by means of a swift automatic point counter; each composition was based on at least 500 counts.

**Results and discussion**

**Organic matter richness**

The organic carbon content of the coals ranges between 32.40 and 65.29 wt% with an average of 38.03 wt%, shales from 0.64 to 4.84 wt% with an average of 3.14 wt%, while coaly shales have an average of 12.1 wt% (Table 1). The minimum TOC value defining a good source rock is 0.5 wt% for shale, 0.3 wt% for carbonate and 1 wt% for clastic rock (Killops and Killops 1993). Generally, the range of 1.5–2 wt% has been generally accepted for good source rocks (Killops and Killops 1993; Hunt 1996a, b). However, the TOC values in the coal, shale and shaly coal samples exceeded the acceptable minimum values required for a potential source rock (Hunt 1996a, b) and thus indicate good source potential. This is corroborated by the average genetic potential (GP) value for the coals, shales and coaly shales which are 70.7 mg/g, 5.04 mg/g and 12.6 mg/g, respectively. The genetic potential values are greater than 2 mg/g required for a potential source rock (Hunt 1996a, b; Peters et al. 2005) (Table 1).

![Fig. 6 Litholog of borehole sampled at Maiganga. GPS: x = 734,251, y = 1,105,749, z = 458. The log shows shale, siltstone and ironstone intercalation. Arrow indicates selected samples.](image-url)
Table 1 TOC, Rock–Eval pyrolysis and vitrinite reflectance data of Maiganga coal and shale sample of Gombe Formation

| S/n | Sample No./code | Lithology | Sample type | TOC (wt%) | S1 | S2 | S3 | T_{max} (°C) | HI | OI | S2/S3 | PI | S1/TOC*100 | GP (S1+S2) | VR (Ro) |
|-----|-----------------|-----------|-------------|-----------|----|----|----|-------------|----|----|--------|----|------------|-----------|--------|
| 1   | MG1A            | Coal      | Surface    | 63.5      | 2.89 | 102.39 | 16.47 | 418         | 162 | 26 | 6.2    | 0.03 | 5         | 105.28    | 77.9   |
| 2   | MG1D            | Coal      | Surface    | 63.39     | 1.49 | 57.96  | 12.12 | 416         | 90  | 19 | 4.8    | 0.03 | 2         | 59.45     | 0.55   |
| 3   | MG1F            | Coal      | Surface    | 60.67     | 2.31 | 75.59  | 11.40 | 421         | 125 | 19 | 6.6    | 0.03 | 4         | 77.9      |        |
| 4   | MG1G            | Coal      | Surface    | 65.29     | 2.09 | 71.02  | 12.87 | 413         | 109 | 20 | 5.5    | 0.03 | 3         | 73.11     |        |
| 5   | MG1H            | Coal      | Surface    | 62.46     | 3.81 | 103.21 | 15.35 | 420         | 165 | 25 | 6.7    | 0.04 | 6         | 107.02    |        |
| 6   | MG1W            | Coal      | Surface    | 64.22     | 3.51 | 88.63  | 20.99 | 418         | 138 | 33 | 4.2    | 0.04 | 5         | 92.14     | 0.49   |
| 7   | MG1X            | Coal      | Surface    | 59.30     | 3.34 | 91.36  | 18.84 | 419         | 154 | 32 | 4.8    | 0.04 | 6         | 94.7      |        |
| 8   | MG2H            | Coal      | Surface    | 62.75     | 3.11 | 87.59  | 16.47 | 422         | 140 | 26 | 5.3    | 0.03 | 5         | 90.7      | 0.48   |
| 9   | MG2O            | Coal      | Surface    | 37.71     | 4.11 | 71.16  | 20.42 | 417         | 189 | 54 | 3.5    | 0.05 | 11        | 75.27     |        |
| 10  | MG3D            | Coal      | Surface    | 53.4      | 0.49 | 30.58  | 17.56 | 429         | 57  | 33 | 1.7    | 0.02 | 1         | 31.07     | 0.50   |
| 11  | MG3Z            | Coal      | Surface    | 55.80     | 0.97 | 73.15  | 27.26 | 435         | 131 | 49 | 2.7    | 0.01 | 2         | 74.12     | 0.46   |
| 12  | MG4K            | Coal      | Surface    | 32.4      | 0.67 | 55.29  | 21.68 | 439         | 171 | 67 | 2.6    | 0.01 | 2         | 55.96     |        |
| 13  | MG2K            | C. Shale  | Surface    | 16.35     | 0.27 | 13.72  | 1.85  | 426         | 84  | 11 | 7.4    | 0.02 | 2         | 13.99     |        |
| 14  | MG3S            | C. Shale  | Surface    | 7.90      | 0.26 | 11.03  | 5.96  | 428         | 140 | 75 | 1.9    | 0.02 | 3         | 11.29     |        |
| 15  | MG1I            | Shale     | Surface    | 2.11      | 0.05 | 1.35   | 1.29  | 430         | 64  | 61 | 1.0    | 0.04 | 2         | 1.4       |        |
| 16  | MG1J            | Shale     | Surface    | 2.13      | 0.05 | 0.72   | 1.34  | 419         | 34  | 63 | 0.5    | 0.06 | 2         | 0.77      |        |
| 17  | MG1L            | Shale     | Surface    | 1.78      | 0.05 | 0.51   | 0.83  | 425         | 29  | 47 | 0.6    | 0.09 | 3         | 0.56      |        |
| 18  | MG1O            | Shale     | Surface    | 1.85      | 0.01 | 0.84   | 1.18  | 431         | 45  | 64 | 0.7    | 0.01 | 0.5       | 0.85      |        |
| 19  | MG1Q            | Shale     | Surface    | 1.19      | 0.05 | 0.92   | 0.63  | 430         | 77  | 53 | 1.5    | 0.05 | 4         | 0.97      |        |
| 20  | MG1R            | Shale     | Surface    | 2.15      | 0.06 | 2.48   | 1.03  | 426         | 115 | 48 | 2.4    | 0.02 | 3         | 2.54      |        |
| 21  | MG1Z            | Shale     | Surface    | 2.95      | 0.11 | 3.31   | 2.07  | 428         | 112 | 70 | 1.6    | 0.03 | 4         | 3.42      |        |
| 22  | MG2C            | Shale     | Surface    | 2.99      | 0.05 | 2.32   | 1.65  | 428         | 78  | 55 | 1.4    | 0.02 | 2         | 2.37      |        |
| 23  | MG2F            | Shale     | Surface    | 3.91      | 0.10 | 3.58   | 2.06  | 425         | 92  | 53 | 1.7    | 0.03 | 3         | 3.68      |        |
| 24  | MG2L            | Shale     | Surface    | 4.81      | 0.14 | 5.14   | 2.52  | 429         | 107 | 52 | 2.0    | 0.03 | 3         | 5.28      | 0.49   |
| 25  | MG2R            | Shale     | Surface    | 3.84      | 0.17 | 6.22   | 1.79  | 430         | 162 | 47 | 3.5    | 0.03 | 4         | 6.39      | 0.50   |
| 26  | MG4D            | Shale     | Surface    | 4.64      | 0.09 | 4.84   | 3.12  | 428         | 104 | 67 | 1.6    | 0.02 | 2         | 4.93      | 0.52   |
| 27  | BH15A           | Shale     | Borehole   | 1.95      | 0.16 | 2.97   | 1.19  | 428         | 152 | 61 | 2.5    | 0.05 | 8         | 3.13      | 0.49   |
| 28  | BH15B           | Shale     | Borehole   | 2.96      | 0.09 | 5.09   | 1.99  | 428         | 172 | 67 | 2.6    | 0.02 | 3         | 5.18      |        |
| 29  | BH15C           | Shale     | Borehole   | 4.84      | 0.09 | 6.89   | 3.93  | 427         | 142 | 81 | 1.8    | 0.01 | 12        | 6.98      |        |

TOC: Total organic carbon (wt%). S1: hydrocarbons already present at temperatures about 300 °C. S2: Hydrocarbons generated through thermal cracking of kerogen at temperatures in the range of 300-650 °C. S3: CO2 that was generated from the kerogen at the same time the S2 hydrocarbons were generated. T_{max}: Temperature of maximum generation of S2 peak. HI: (Hydrogen index). An important indicator of kerogen type—oil/gas proneness measured in (mgHC/gTOC). OI: (Oxygen index). An indicator of kerogen type measured in (mgCO2/gTOC). PI: (Production index) (S1/S1+S2), indicative of free petroleum content. GP: genetic potential. VR: vitrinite reflectance.
Kerogen quality

The quality of the organic matter contained in the samples was evaluated from their hydrogen index (HI). The hydrogen index for the coal ranges from 57 to 189 mg/gTOC, shale between (29–172 mg/gTOC) and (84–140 mg/gTOC) for coaly shale. HIs in all the samples are less than 200 mgHC/gTOC (Table 1) suggesting type III organic matter (Hunt 1996a, b). The plots of S2 versus TOC (Fig. 7) indicated gas prone kerogen, i.e. type III organic matter. Plot of HI versus OI (Van Krevelen diagram) (Fig. 8) also confirms type III kerogen for the coal, shale and coaly shale samples. However, there are few samples of shale that fall under type IV kerogen. This is corroborated by plot of HI versus \( T_{\text{max}} \) (Fig. 9) and HI versus VR (Fig. 10) which indicates the organic matter is immature and mainly type III capable of generating mainly gas. Similarly, some shale samples fall under type IV kerogen which is inert. The \( S_2/S_3 \) value ranges from (1.7–6.7), (1.9–7.4) to (0.5–3.5) for the coal, coaly shale and shale, respectively, indicating type III kerogen (Hunt 1996a, b).

Hydrocarbon potential

The \( S_2 \) yield ranges from 30.6 to 91.4 mg/g for the coals, (2.97–6.89 mg/g) for the shales and (11.03–13.73 mg/g) for the coaly shale (Table 1). The average values of the \( S_2 \) are (68.7 mg/g), (4.92 mg/g) and (12.4 mg/g), respectively. This is further corroborated by the genetic potential value greater than 2 mg/g for most of the samples (Table 1). These values indicate moderate-to-good source rock potential (Hunt 1996a, b), and it is comparable to what
was observed in other hydrocarbon generating coal basins in the world such as Cooper–Eromanga Basin in Australia and Taranaki basin in New Zealand. The results showed that all the samples have low production index (PI) (less than 0.1) indicating low level conversion (Figs. 11, 12).

**Source of the organic matter and palaeodepositional environments**

To infer the environments of deposition of the source rocks, saturate, aromatic biomarker ratios and parameters were used (Tables 2, 3). The extract from the gas
chromatography was resolved into normal paraffin, isoprenoids. The samples were characterized by high molecular weight \(n\)-alkanes. The \(n\)-alkanes are the dominant component of the aliphatic hydrocarbon fraction and show variation in quantity and distribution in the samples analysed. The ion fragmentograms of the saturate fractions analysed by gas chromatography and mass spectrometry were recorded in \(m/z\ 191, m/z\ 217, m/z\ 218\) and \(m/z\ 259\) corresponding to tricyclic and pentacyclic terpanes, steranes, diasteranes and tetracyclic polyprenoids, while the aromatic fractions were recorded on the \(m/z\ 253, m/z\ 251\) and \(m/z\ 245\) corresponding to monoaromatic steroids, triaromatic steroids and triaromatic methyl steroids, respectively (Appendix). The \(n\)-alkanes distribution in the coal source rocks ranges from \(C_{10–C_{41}}\) maximizing at \(C_{29}\) (Table 2) (Fig. 13a–d). This pattern indicates organic matter derived mainly from terrestrial organic matter. High proportions of long-chain \(C_{27–C_{35}}\) members relative to the total \(n\)-alkanes especially are typical of terrestrial higher plants (Miranda et al. 1999). The shale source rocks range from \(C_{9–C_{41}}\) also maximizes at \(C_{16}\) and \(C_{29}\) (Table 2) (Fig. 13e–g) indicating mixed sources.

The predominance of pristane (\(\text{n-C}_{17}\)) over phytane (\(\text{n-C}_{18}\)) supports humic origin of the organic matter for the coal source rocks deposited in a mixed/transitional environment and less reducing environmental condition (Peters et al. 2005). Pr/Ph ratio (Table 2) for the coal source rocks ranges from 2.15 to 3.51 (average 2.79),
while the shale source rocks range between 0.95 and 2.37 (average 1.82) indicating coastal swamp and fluvio-deltaic depositional environment in sub-oxic to oxic setting (Peters et al. 2005).

The plot of Pr/n-C_{17} versus Ph/n-C_{18} (Fig. 14) also confirms terrestrial and mixed sources for the organofacies corroborated by the high values of nC_{17}/nC_{29} over nC_{18}/nC_{19}. The coals are rich in C_{29} αββS steranes (> 50%) and C_{29} αααR sterane (~ 40%). This is an indication of significant land plants contribution as evidenced from the very low C_{27} steranes (Table 3). The shale also has high C_{29} steranes but with appreciable quantity of C_{27} and C_{28} αββS sterane (26% and 29%) and (34% and 24%) in C_{25}αααR and C_{28}αααR sterane confirming possible marine contribution for the source (Table 3) and (Fig. 15). The appreciable quantity of C_{27} and C_{28} in the shale sample BH15B also reflects contributions from phytoplankton; algae, diatoms, dinoflagellates (Peters et al. 2005). This is in agreement with plot of Pr/n-C_{17} versus Ph/n-C_{18}, though there is absence of C_{30}, 24 n-propyl cholestane (biomarker of chrysophyte algae) to strengthen the marine algae contribution. However, the presences of C_{30} tetracyclic polyprenoids (TPP) a marker of lacustrine algae support fresh water lacustrine depositional conditions. The sterane/hopane ratio values range from 0.06 to 0.35 (Table 3). This ratio is (< 0.6) which suggest incorporation of high level of bacterial inputs commonly associated with terrigenous organic matter in coals (non-marine organic matter) (Norgate et al. 1999).

The appreciable quantity of homohopanes (C_{31}–C_{35}) in all the samples suggests that bacteriohopanetetrol and other polyfunctional C_{35} hopanoids (Wang et al. 1996) commonly found in prokaryotic micro-organisms were significant contributors to the biomass.

The predominance of C_{29} triaromatic steroid (TAS) indicates a terrestrial source contribution for the organic matter as evident from the ternary plot of C_{27}, C_{28}, C_{29} monoaromatic steroid (MAS) (Fig. 16). The cross plot of (9MP/(9MP + 1MP) versus Paq (Fig. 17) further confirms terrestrial land plants contribution for the organic matter of the coals and shales. The low amounts of dibenzoanthiophene to phenanthrene (DBT/PH) (Table 4) and cross plots of DBT/PH versus Pr/Ph (Fig. 18) and sulphur versus DBT/PHEN (Fig. 19) indicate deposition of the organic matter predominantly in lacustrine and/or fluvial/deltaic environments (Hughes et al. 1995). There is an appreciable amount of C_{27}, C_{29} MAS in the shale sample BH15B which may suggest mixed sources. The presence of 1,2,5- and 1,2,7-trimethylanthalene (TMN) in the coal samples indicates both angiosperm and gymnosperms material contribution to the organic matter that formed the coals (Killops and Killops 2005).
Fig. 13 Ion chromatograms of samples showing the distribution of n-alkanes. a MG1G, b MG2H, c MG2O, d MG4K, e MG3S, f MG4D, g BH15B
Fig. 13 (continued)
Fig. 13 (continued)
Fig. 13 (continued)

Fig. 14  Plot of Pr/n-C\textsubscript{17} versus Ph/n-C\textsubscript{18} of coal and shale samples of Maiganga, Gombe formation. The coals fall within the terrestrial sources in oxic depositional settings, shales plotted within the mixed sources. after Peters et al. (2005)
Thermal maturity and hydrocarbon potential

The thermal maturity of all the samples is represented by $T_{\text{max}}$ range of 413–439 °C. The $T_{\text{max}}$ of the coals ranges between 427 and 430 °C with an average of 428 °C, shales between 419 and 431 °C with average value of 427 °C, while the coaly shale is between 426 and 428 °C, averaging 427 °C. Table 1 shows that majority of the samples have $T_{\text{max}}$ less than 435 °C with exception of two coal samples having 435 °C and 439 °C. The spiked $T_{\text{max}}$ of these two samples (MG3Z and MG4K) having the threshold values for maturity could probably arise from impurities or weathering of the samples or possibly human error as there are no conditions that could possibly increase the maturity at that stratigraphic interval. Consequently, Clayton and King (1987) and Lo and Cardott (1995)) have shown that weathering in outcropping source rocks has significant effect on the amount and quality of organic matter obtained by Rock–Eval pyrolysis. This is also noted in the OI values for the two samples 49 mg CO$_2$/gTOC in MG3Z and 67 mg CO$_2$/gTOC for MG4K indicating the highest level of weathering in the sample package. The low production index (PI) recorded for all the samples (<0.1) also indicates that they are thermally immature.

The vitrinite reflectance of the coals ranges from 0.46 to 0.55 Ro averaging 0.5 Ro, and shales range between 0.49 and 0.53Ro with an average of 0.51Ro (Tables 1, 5). These average values are less than 0.55Ro and thus confirm the samples have low to marginal maturity and late diagenetic stage for oil generation (Killops and Killops 2005). The plot of HI versus $T_{\text{max}}$ (Fig. 9), HI versus VR (Fig. 10), PI versus $T_{\text{max}}$ (Fig. 11) and PI versus VR (Fig. 12) all indicated that the samples are immature. The carbon preference index (CPI) which measures the odd/even predominance of the $n$-alkanes showed the samples to range from 2.74 to 3.76 (Table 2). These values are essentially greater than unity, qualifying them as immature (Peters and Moldowan 1993). The predominance of asphaltenes and NSO in the extracts (Fig. 20) also confirms that the bitumen originates from low maturity level of kerogen cracking (Clayton and Law 1993).

The moretane/hopane and $T_s/T_s + T_m$ ratios in the samples range from 0.42–0.68 to 0.07–0.12, respectively (Table 6). These values suggest low maturity status (Peters et al. 2005). According to (Seifert and Moldowan 1986), C$_{31}$- or C$_{32}$-homohopanes can be used to calculate 22S/(22S + 22R) ratio which is also a maturity parameter and is always close to its thermal equilibrium value (TEV). In this study, considering the C$_{32}$ homohopane ratios of the coals ranging from 0.28 to 0.53 and C$_{32}$ homohopane thermal equilibrium value of 0.6, the C$_{32}$ homohopane ratios fall below the equilibrium range and thus indicate thermal maturity level has not been reached. Most of the saturate maturity parameters are lower compared to their corresponding thermal equilibrium value.
indicating low maturity status (Table 7) and hence have not reached onset of oil generation. The moretane/hopane and \( T_{1}/(T_{1}+T_{m}) \) ratios in the samples range from 0.42–0.68 to 0.07–0.12, respectively, also indicate low maturity (Table 6).

The aromatic maturity indices were used to infer the maturity status of the organofacies. The calculated methyl phenanthrene index (MPI-1) and (MPI-2) are in close agreement with each other (Table 8). The MPI-1 and MPI-2 of the coal range from 0.23–0.31 to 0.31–0.50, respectively, while the shaly source rocks range from 0.33–0.51 to 0.45–0.56, respectively. This suggests immature to marginally mature source rocks.

The proportion of vitrinite is the most abundant in the samples, and the vitrinite maceral in the coal ranges from 46.6 to 70%, while the shale ranges from 35 to 45%. The inertinite content ranges from 25 to 35% for the shale, while the coal ranges from 28.7 to 53% (Table 5). The high percentage of the vitrinite indicates gas prone and suggests they were deposited in close proximity to the source. The high percentages of the gas prone kerogenaceous debris and their low corresponding hydrogen index (HI) may be due to pre-depositional or post-depositional oxidation from the bulk of the sediments in the stratigraphic section or reworking of the organics.

According to (Hunt 1996a, b; Petersen and Nytoft 2006), the generation of petroleum from coal source rocks and its type are basically dependent on the hydrogen content. Many authors have suggested that liptinite content is essential in estimating the potential of the coal to generate liquid hydrocarbon. (Hunt 1991) has suggested 15–20% liptinite, (Snowdon 1991) proposed 10% hydrogen rich maceral, while Mukophadhyay and Hatcher (1993) suggest 15–20% liptinite content for a coal to generate oil. As indicated in (Table 5), the percentage oil prone for the shale is about 30–40% with respect to the liptinite content up to 40%, whereas the coals have low liptinite of about (5%) maximum, indicating that the tendency to generate liquid hydrocarbon is minimal. Peterson and Nytoft (2006) have proven that
Table 4  Selected aromatic biomarker source and depositional indices of coal and shale of Maiganga, Gombe Formation

| S/n | Sample code | %26TAS | %27TAS | %28TAS | %29TAS | %27MAS | %28MAS | %29MAS | DBT/PH | MDBT/MPHEN | 9MP/ (9MP+1MP) | Paq |
|-----|-------------|--------|--------|--------|--------|--------|--------|--------|--------|----------|----------------|--------|
| 1   | MG1G        | 2      | 7.3    | 3.9    | 86.8   | 23.8   | 20.1   | 56.1   | 0.03   | 0.10     | 0.67            | 0.15   |
| 2   | MG2H        | 1.3    | 21.5   | 4.6    | 72.6   | 17.3   | 25.3   | 57.4   | 0.04   | 0.09     | 0.40            | 0.06   |
| 3   | MG2O        | 1.9    | 12.2   | 6.4    | 79.4   | 9.9    | 23.3   | 66.8   | 0.10   | 0.38     | 0.55            | 0.12   |
| 4   | MG4K        | –      | –      | –      | –      | 10     | 19.3   | 70.7   | 0.19   | 0.33     | 0.49            | 0.10   |
| 5   | MG35        | –      | –      | –      | –      | –      | 16.6   | 19.4   | 63.9   | 0.11     | 0.19            | 0.50   | 0.11   |
| 6   | MG4D        | –      | –      | –      | –      | –      | 14.2   | 25.8   | 60.0   | 0.08     | 0.25            | 0.50   | 0.16   |
| 7   | BH15B       | 10     | 28.1   | 18.5   | 43.4   | 21.4   | 23.9   | 54.6   | 0.14   | 0.57     | 0.50            | 0.19   |

TAS triaromatic steroid, MAS monoaromatic steroid, DBT dibenzothiophene, PH phenanthrene, MPHEN methylphenanthrene, MDBT methyl dibenzothiophene, 9MP 9-methylphenanthrene, 1MP 1-methylphenanthrene, Paq = nC23 + nC25/nC23 + nC25 + nC29 + nC31—alkanes
Table 5  Maceral and vitrinite reflectance of coal and shale samples of Maiganga Gombe Formation

| S/n | Sample code | Lithology | Liptinite (%) | Vitrinite (%) | Inertinite (%) | VR  |
|-----|-------------|-----------|---------------|---------------|----------------|-----|
| 1   | MG1D        | Coal      | 0.4           | 46.6          | 53             | 0.55|
| 2   | MG1W        | Coal      | 1.9           | 59            | 39.1           | 0.49|
| 3   | MG2H        | Coal      | 1.3           | 70            | 28.7           | 0.48|
| 4   | MG3D        | Coal      | 5             | 50            | 45             | 0.50|
| 5   | MG3Z        | Coal      | 5             | 55            | 40             | 0.46|
| 6   | MG2L        | Shale     | 40            | 35            | 25             | 0.49|
| 7   | MG2R        | Shale     | 30            | 35            | 35             | 0.50|
| 8   | MG4D        | Shale     | 30            | 45            | 25             | 0.53|
| 9   | BH15A       | Shale     | 40            | 35            | 25             | 0.50|

VR vitrinite reflectance

Fig. 20  Ternary diagram showing the proportion of saturated hydrocarbons, aromatic hydrocarbons and polar fraction (NSO + asphaltenes) in extracts of coal, shale and coaly shale source rocks of Maiganga, Gombe Formation. The samples have higher percentage of heterocompounds indicated by the arrow. After Clayton and Law (1993)

Table 6  Selected saturate biomarker maturity indices of coal and shale sample of Maiganga, Gombe Formation

| S/n | Sample no/code | Depth (ft) | Lithology/Rank | S/(S + R) (C_{29} ααα) | C_{29}[ββS/(ββS + ααR)] | T_{f}/T_{f} + T_{m} | Mor/Hop | H_{32}S/(R + S) | TT/Hop |
|-----|----------------|------------|----------------|------------------------|--------------------------|-------------------|---------|----------------|--------|
| 1   | MG1G           | 105        | Coal(SB)       | 0.28                   | 0.26                     | 0.12              | 0.54    | 0.53           | 0.04   |
| 2   | MG2H           | 62         | Coal(SB)       | 0.28                   | 0.61                     | 0.08              | 0.62    | 0.40           | 0.03   |
| 3   | MG2O           | 59         | Coal(SB)       | 0.12                   | 0.37                     | 0.07              | 0.61    | 0.28           | 0.08   |
| 4   | MG4K           | 14         | Coal(SB)       | –                      | –                        | –                 | 0.63    | –              | –      |
| 5   | MG3S           | 38         | C. Shale       | –                      | –                        | –                 | 0.55    | –              | –      |
| 6   | MG4D           | 23         | Shale          | –                      | –                        | –                 | 0.68    | –              | 0.09   |
| 7   | BH15B          | 157        | Shale          | 0.1                    | 0.04                     | –                 | 0.42    | –              | 0.38   |

T_{f}/T_{f} + T_{m} = Trisnorhopane, Mor/Hop = moretane/hopane, TT/Hop = tricyclic terpane/hopane, H_{32}S/(R + S) = homohopanes, S/(S + R)(C_{29} ααα), C_{29}[ββS/(ββS + ααR)], SB (sub-bituminous)

Table 7  Saturate biomarker ratio, thermal equilibrium value (TEV) and vitrinite reflectance of the samples

| S/n | Saturate biomarker | Range       | TEV | VR (%) | Maturity |
|-----|--------------------|-------------|-----|--------|----------|
| 1   | S/(S + R) (C_{29} ααα) | 0.1–0.28 | 0.55 | 0.80 | IM–LM    |
| 2   | (C_{29}) ββS/(ββS + ααR) | 0.04–0.61 | 0.70 | 0.90 | IM–LM    |
| 3   | Diasterane/C_{29} ααα sterane | 0.17–1.01 | 1.00 | 1.40 | IM–LM    |
| 4   | Moretane/hopane | 0.42–0.68 | 0.05 | 0.70 | IM–LM    |
| 5   | T_{f}/(T_{f} + T_{m}) | 0.07–0.12 | 1.00 | 1.40 | IM       |
| 6   | C_{32} Homohopane | 0.28–0.53 | 0.60 | 0.60 | IM–LM    |

TEV thermal equilibrium value, IM immature, LM low maturity, VR vitrinite reflectance
humic coals dominated by huminite could generate oil, though liptinite macerals are more oil prone than huminite because of its high paraffinicity in structure and its ability to generate long-chain aliphatic component (> C20–C25) (Isaksen et al. 1998).

The ternary plot of vitrinite, liptinite and inertinite revealed the type of hydrocarbon the source beds can generate (Fig. 21). Since vitrinite is the most abundant maceral in the coal samples analysed, it is logical to say that the coal has the potential to generate gaseous hydrocarbon (dry gas), while the shale has potential to generate wet gas (Fig. 21). All the samples are characterized by the absence of alginate and amorphous kerogen eliminating the presence of Type I kerogen typical of algae. The shales and coals are said to be dominated by Type III kerogen. The photomicrograph of the coal macerals is shown in Fig. 22.

### Table 8  Selected Aromatic biomarker maturity indices of coal and shale sample of Maiganga, Gombe Formation

| S/n | Sample No./code | Depth (ft) | Lithology/rank | TAS/ (TAS + MAS) | MPI-1 | MPI-2 | DNR-1 | DNR-2 | MDR | MDR-1 | TDE-1 | TDE-2 | Rc |
|-----|-----------------|------------|----------------|------------------|--------|--------|-------|-------|-----|-------|-------|-------|----|
| 1   | MG1G            | 105        | Coal (SB)      | 0.27             | 0.31   | 0.50   | 2.70  | 1.31  | 0.66 | 1.15  | 5.10  | 0.27  | 0.56 |
| 2   | MG2H            | 62         | Coal (SB)      | 0.39             | 0.23   | 0.32   | 4.12  | 1.09  | 0.77 | 0.90  | 7.55  | 0.19  | 0.51 |
| 3   | MG2O            | 59         | Coal (SB)      | 0.19             | 0.23   | 0.31   | 4.11  | 1.88  | 0.42 | 1.64  | 9.44  | 0.40  | 0.51 |
| 4   | MG4K            | 14         | Coal (SB)      | –                | 0.34   | 0.45   | 4.51  | 1.81  | 0.38 | 2.64  | 5.43  | 0.37  | 0.58 |
| 5   | MG3S            | 38         | C. Shale       | –                | 0.37   | 0.50   | 2.99  | 1.55  | 0.67 | 1.12  | 8.21  | 0.26  | 0.59 |
| 6   | MG4D            | 23         | Shale          | –                | 0.32   | 0.45   | 2.88  | 1.18  | 0.39 | 2.01  | 8.42  | 0.30  | 0.57 |
| 7   | BH15B           | 157        | Shale          | 0.20             | 0.51   | 0.56   | 3.40  | 1.94  | 0.81 | 0.83  | 4.79  | 0.38  | 0.68 |

Methyl phenanthrene index = (MPI-1) and (MPI-2), Mono = (MAS) and triaromatic steroids (TAS), DNR = dimethylnaphthalene ratio, MDR = methylbenzothiophene ratio, Rc = calculated vitrinite reflectance of the aromatic biomarker, MPI-1 = 1.5(2MP + 3MP)/(P + 1MP + 9MP), MPI-2 = 3(2MP)/(P + 1MP + 9MP), MDR = 4MDBT/1MDBT, MDR-1 = 1MDBT/DBT, DNR_1 = (2,6 + 2,7)/(1,5) DMN, Rc = 0.6(MPI-1) + 0.37
Conclusion

In Conclusion, the Maastrichtian coal and shale of Gombe Formation, Gongola Basin show moderate-to-good source potential. The organic matter in the source rocks is mainly Type III from terrestrial land plant contribution though few samples fall under Type IV. The source rocks (coal and shale) have the potential to generate mainly gas and may probably generate oil if higher thermal maturity level is attained. Also, the coal facies were formed from organic matter derived from mainly terrestrial/terrigenous organic matter under oxidizing condition and deposited in the fluvial/deltaic environments, while the shaly facies were from mixed sources deposited under reducing environments.

Open Access This article is distributed under the terms of the Creative Commons Attribution 4.0 International License (http://creativecommons.org/licenses/by/4.0/), which permits unrestricted use, distribution, and reproduction in any medium, provided you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license, and indicate if changes were made.

Fig. 22 Photomicrograph of coal sample a MG1D, b MG1W and c MG2H. (Liptinite shows grayish colouration, vitrinite is dark coloured, and inertinite is white under reflected light. V: vitrinite, I: inertinite, L: liptinite)
Appendix

\[ m/z \ 191: \text{Tri- and Pentacyclic Terpanes for sample BH15B} \]
\[ m/z \ 217: \text{Sterane for sample BH15B} \]
\[ m/z \ 218: \beta \beta \text{ Sterane for sample BH15B} \]
\[ m/z \ 231: \text{Triaromatic steroid for sample BH15B} \]
\[ m/z \ 253: \text{Monoaromatic steroid for sample BH15B} \]
\[ m/z \ 245: \text{Triaromatic methylsteroid for sample BH15B} \]

References

Abubakar MB, Obaje NG, Luterbacher HP, Dike EFC, Ashraf AR (2006) A report on the occurrence of Albian-Cenomanian elater-bearing pollen in Nasara-1 well, upper Benue Trough, Nigeria. Biostratigraphic and palaeoclimatological implications. J Afr Earth Sci 45:347–354

Benkhelil J (1989) The origin and evolution of the cretaceous Benue Trough (Nigeria). J Afr Earth Sci 8:251–282. https://doi.org/10.1016/S0899-5362(89)80028-4

Clayton JL, Law EB (1993) Composition of crude oils generated from coals and coaly organic matter in shales. In: Rice, DD (ed) Hydrocarbons from coal, vol 38, American Association of Petroleum Geologist Studies in Geology, pp 185–201

Clayton J, King J (1987) Effects of weathering on biological marker and aromatic hydrocarbon composition of organic matter in phosphoria shale outcrop. Geochim Cosmochim Acta 51:2153–2157

Ficken KJ, Li B, Swain DL, Eglinton G (2000) An n-alkane proxy for the sedimentary input of submerged/ floating fresh water aquatic macrophytes. Org Geochem 31(7-8):745–749

Hughes WB, Holba AG, Dzhou LIP (1995) The ratios of dibenzothiophene to phenanthrene and pristane to phytane as indicators of depositional environment and lithology of petroleum source rocks. Geochim Cosmochim Acta 59:3581–3598

Hunt JH (1991) Generation of gas and oil from coal and other terrestrial organic matter. Org Geochem 17(6):673–680

Hunt JH (1996a) Petroleum geochemistry and geology, 2nd edn. Freeman, New York, p 743

Hunt JM (1996b) Petroleum geochemistry and geology, 2nd edn. W.H. Freeman and Company, New York

Inaba T, Suzuki N, Hirai A, Sekiguchi K, Watanabe T (2001) Source rock lithology prediction based on oil diacholesterol abundance in the siliceous-clastic Akita sedimentary basin, Japan. Org Geochem 32(7):877–890
Isaksen GH, Curry DJ, Yeakel JD, Jensen AI (1998) Controls on the oil and gas potential of humic coals. Org Geochem 29:23–44
Jimoh AY, Ojo OJ (2016) Rock Eval pyrolysis and organic petrographic analysis of the Maastrichtian coals and shales at Gombe, Gongola Basin, Northeastern Nigeria. Arab J Geosci. https://doi.org/10.1007/s12517-016-2467-x.9:443
Killops SD, Killops VJ (1993) An introduction to organic geochemistry. Longman Group Ltd, UK
Killops SD, Killops VJ (2005) Introduction to organic geochemistry, 2nd edn. Blackwell Publishing Limited, UK
Langford FF, Blanc-Valleron MM (1990) Interpreting Rock Eval pyrolysis data using graphs of pyrolyzable hydrocarbons versus total organic carbon. Am Assoc Pet Geol Bull 74:799–804
Lar UA, Bata TP, Dibal HU, Daspan RI, Isah LC, Fube AA, Bassi DA (2018) Field, petrographic and geochemical studies of basement, clastic and carbonate petroleum reservoirs in The Northern Benue Trough, Nigeria. Pet Technol Dev J 8(2):36–52
Lo H, Cardott B (1995) Detection of natural weathering of upper Mcalester coal and Woodford shale, Oklahoma, USA. Org Geochem 22:73–83
Miranda ACML, Loureiro MRB, Cardoso JN (1999) Aliphatic and Aromatic hydrocarbons in Candiota col samples: novel series of bicyclic compounds. Org Geochem 30:1027–1028
Moldowan JM, Seifert WK, Gallegos EJ (1986) Use of biological markers in petroleum exploration. In: Johns RB (ed) Methods in geochemistry and geophysics, vol 24. Elsevier, Amsterdam, pp 261–290
Snowdon LR (1991) Oil from Type III organic matter: resinite revisited. Org Geochem 17:743–747
Tissot BP, Welte DH (1984) Petroleum formation and occurrence. Springer, Berlin
Van Krevelen DW (1961) Coal: typology—chemistry—physics—constitution, 1st edn. Elsevier, Amsterdam, p 514
Wang P, Li M, Larter SR (1996) Extended hopanes beyond C40 in crude oils and source rocks extracts from Liaohe Basin, NE China. Org Geochem 24(5):547–551
Whiteman A (1982) Nigeria: its petroleum geology, resources and potential. London, Graham and Trotman, p 381
Wright JB (1968) South Atlantic continental drift and the Benue Trough. Tectono Phys 6:301–310

Publisher’s Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.