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

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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 C_{10}–C_{41} in the coals and C_9–C_{41} in the shales. The n-alkanes distributions in the coals maximize at C_{29} indicating terrestrial sourced organic matter, while the shales maximize at C_{16} and C_{29} indicating mixed sources. 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_{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 spectroscopy. 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.

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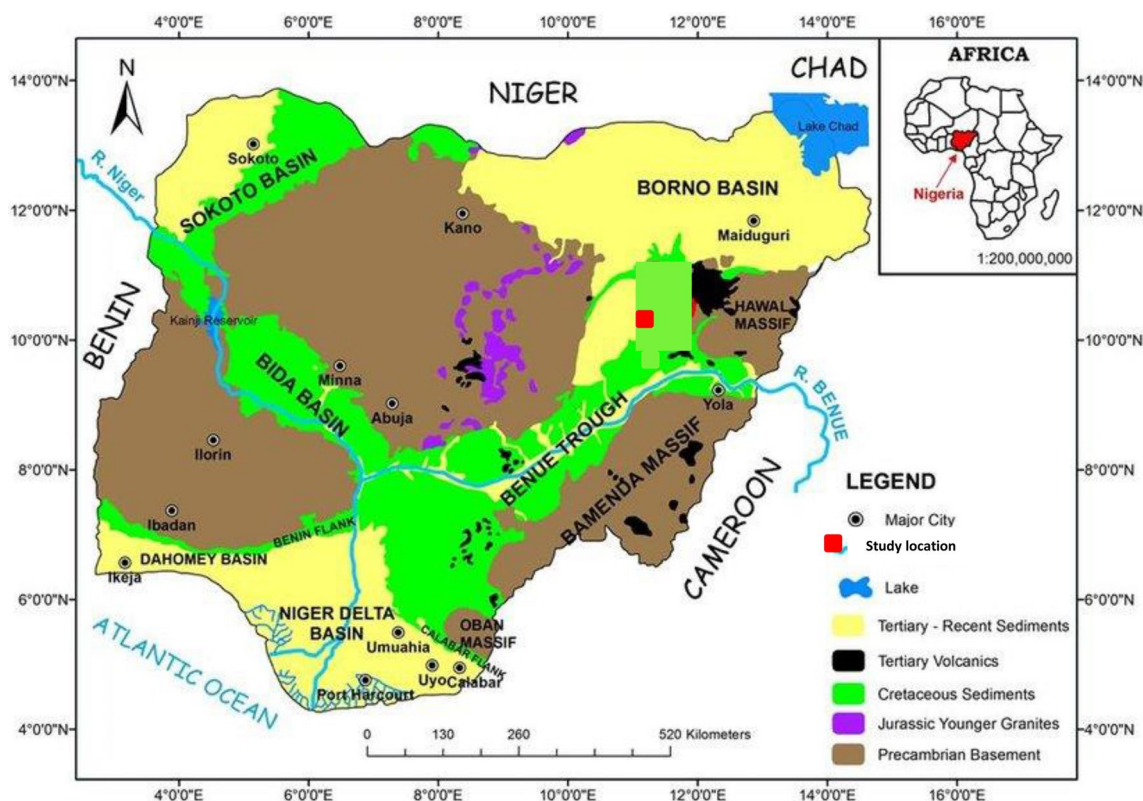
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Fig. 1 Geological map of Nigeria showing the Basement Complex, sedimentary basins and the study location (Northern Benue Trough). Modified after Lar et al. (2018)

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.
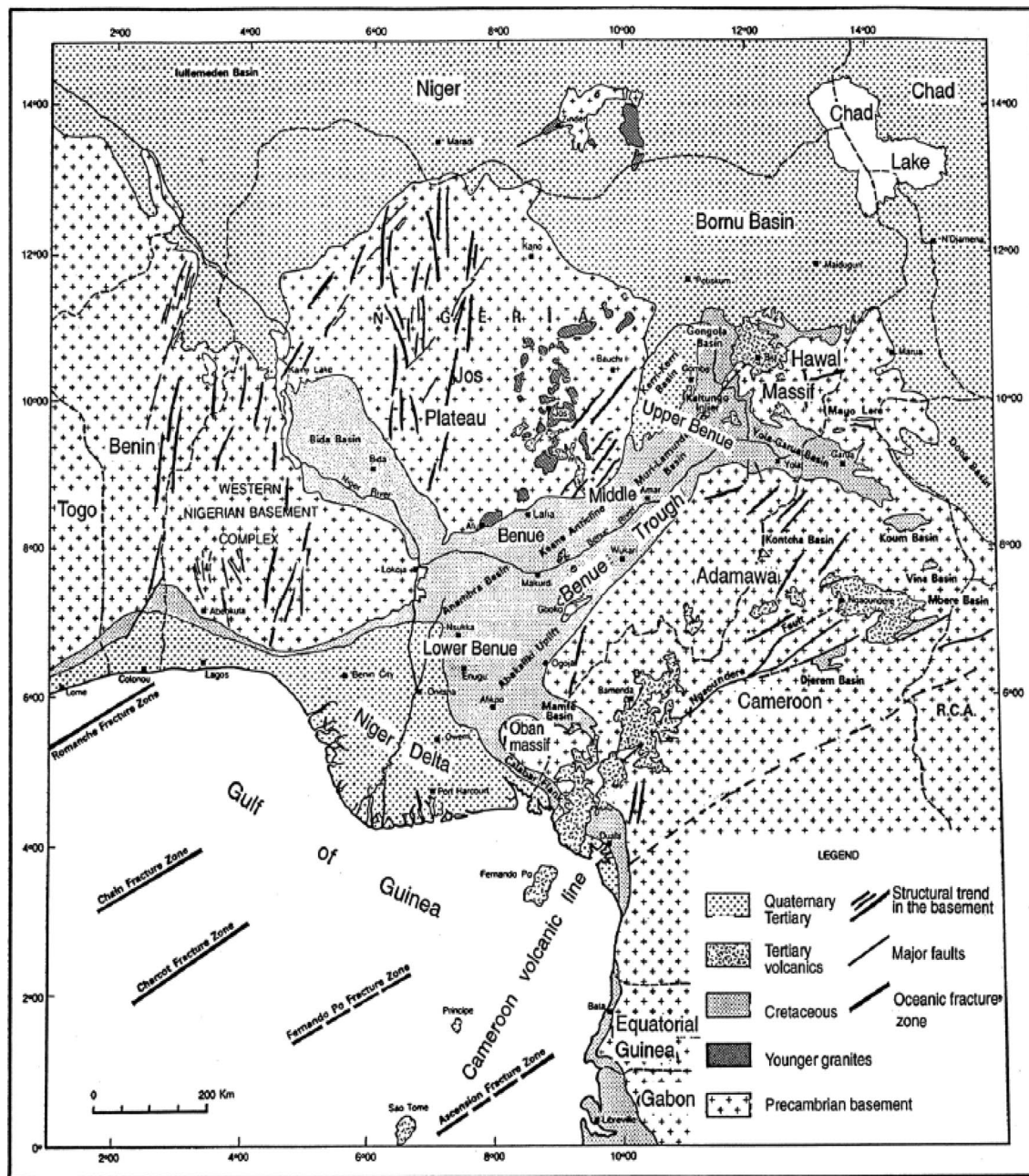
Fig. 2 Geological map of the Benue Trough. The traces of the equatorial fracture zones are indicated. After Odebode (2010)

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

Fig. 3 Stratigraphy of northern Benue Trough. Modified after Abubakar et al. (2006)

Age	Formation (Gongola Basin)	Lithology	Paleoenvironment
Paleogene	Kerri-Kerri	Sandstone Conglomerate	Continental (Fluvial/Lacustrine)
Maastrichtian	Gombe	Sandstone, Coal, Shale, Siltstone, Claystone	Fluvial/Deltaic- Open marine
Campanian			
Santonian			
Coniacian	Pindiga	Shale, Limestone	Marine (Estuarine)
Turonian			
Cenomanian	Yolde	Sandstone, Shale, Siltstone	Deltaic-Barrier island
Albian	Bima	Sandstone	Continental (Braided/Lacustrine/ Alluvial)
Precambrian	Basement Complex	Granite/Gneiss/ Migmatite/Schist	Igneous/ Metamorphic

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 ($S1$), hydrocarbon generated by thermal cracking of the kerogen ($S2$) and the maximum temperature of the kerogen decomposition (T_{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
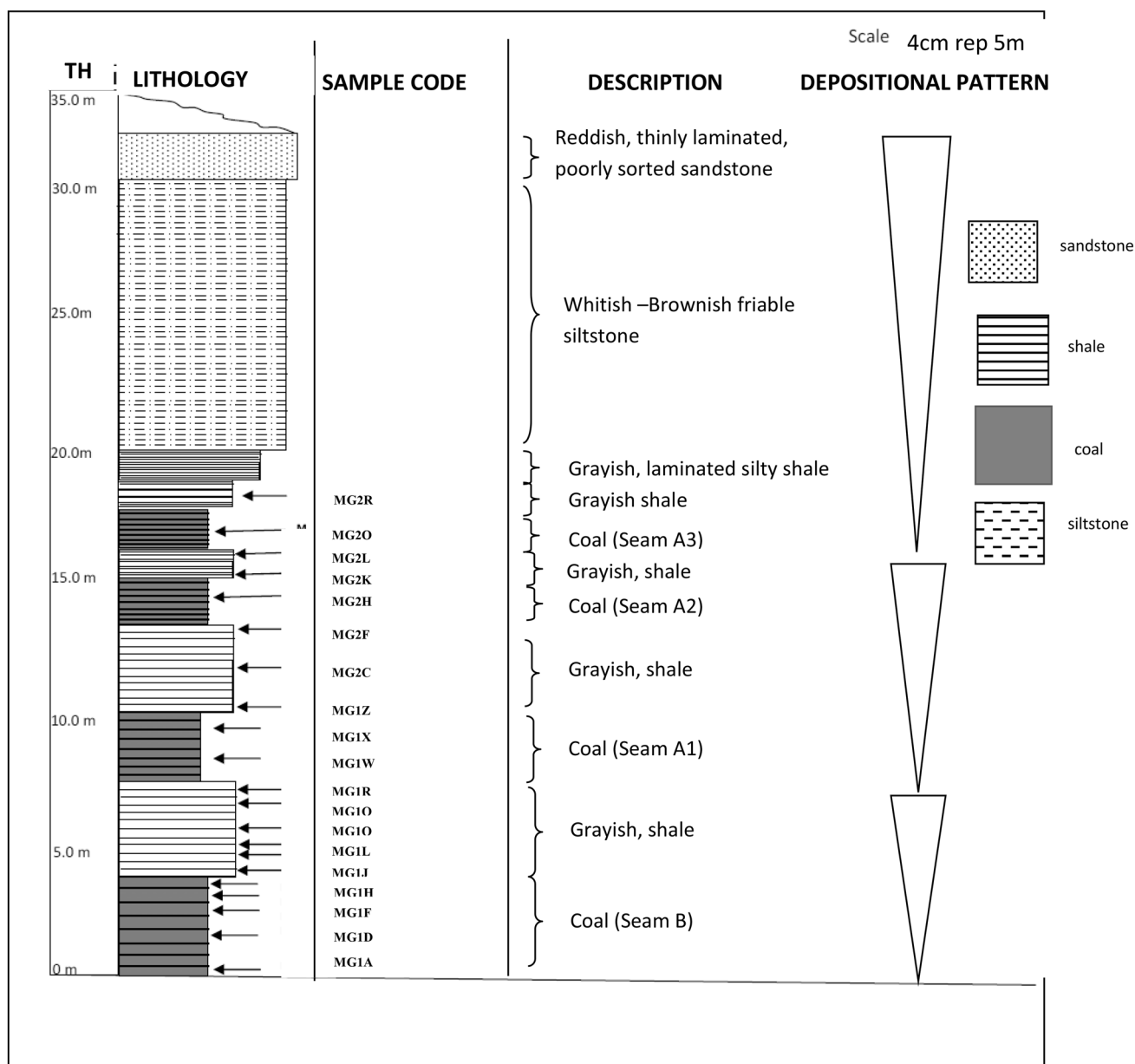
Fig. 4 Lithologic succession of Gombe Formation at the Maiganga mine (Phase I). The succession coarsens upward from coal, shale, siltstone to sandstone. The arrows indicate the selected and analysed samples. Note the coordinates: 10°02′39″N, 11°12′17″E

with *n*-hexane as the mobile phase. The extracts were first deasphaltened 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
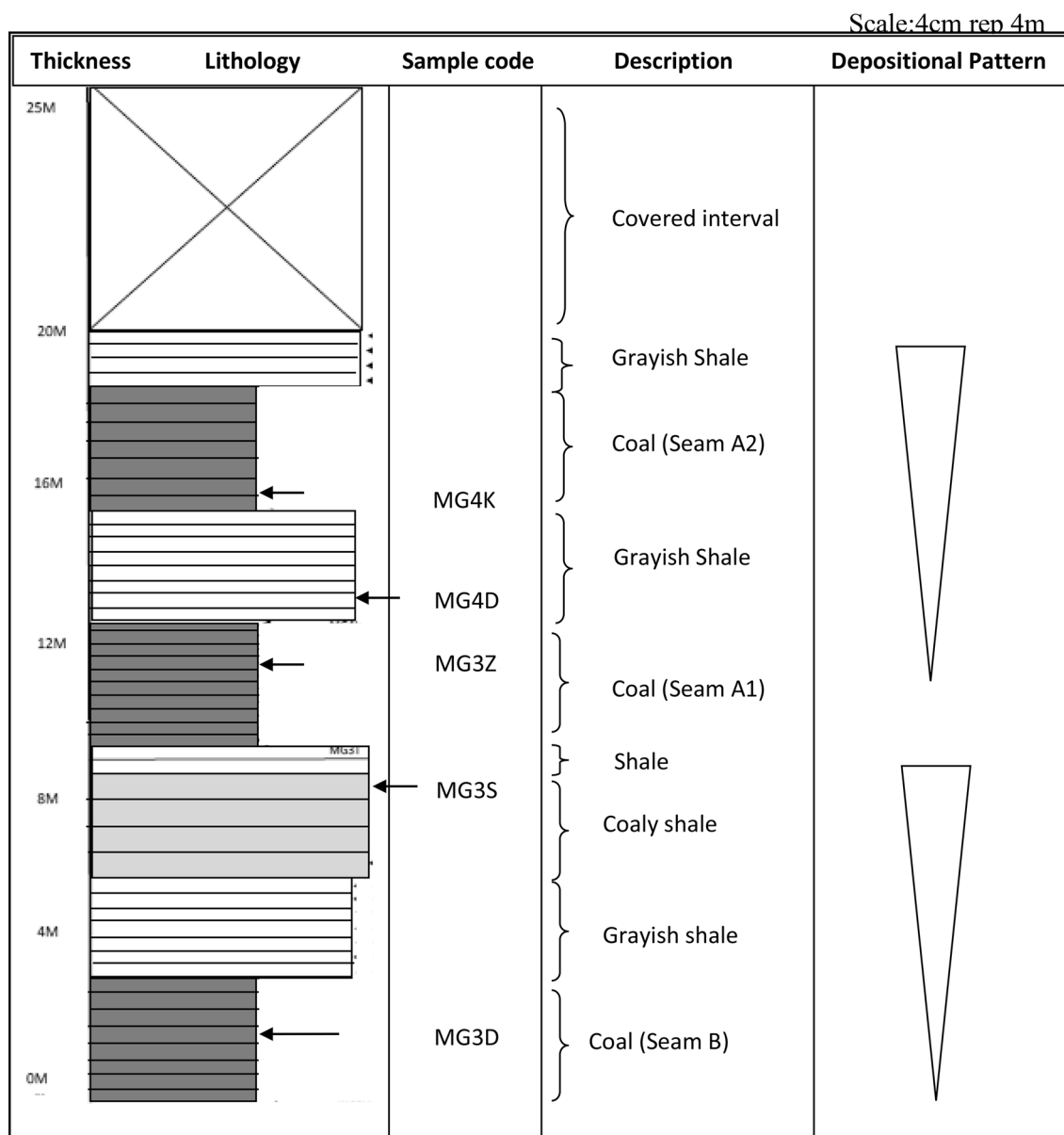
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Thickness	Lithology	Sample code	Description	Depositional Pattern
			Covered interval	
			Grayish Shale	
			Coal (Seam A2)	
		MG4K		
			Grayish Shale	
		MG4D		
		MG3Z	Coal (Seam A1)	
			Shale	
		MG3S	Coaly shale	
			Grayish shale	
		MG3D	Coal (Seam B)	

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)

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, cholane 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
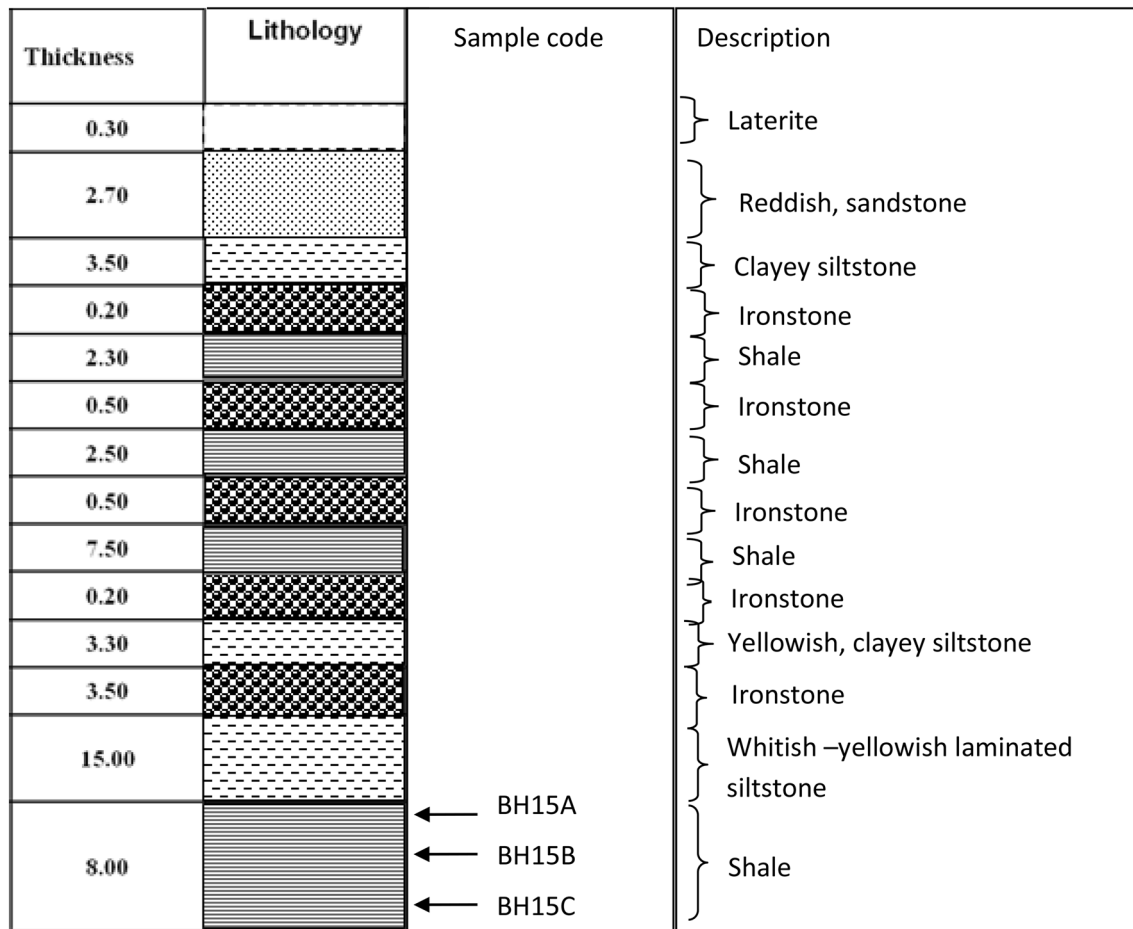
Fig. 6 Litholog of borehole sampled at Maiganga. GPS: $x = 734,251$, $y = 1,105,749$, $z = 458$. The log shows shale, siltstone and ironstone intercalation. Arrow indicates selected samples

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).

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	
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	2	6.98	

TOC: Total organic carbon (wt%) S1: hydrocarbons already present in the sample which are mainly stripped at temperatures about 300 °C. S2: Hydrocarbons generated through thermal cracking of kerogen at temperatures in the range of 300–650 °C. S3: CO_2 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), HI = S2*100/TOC. OI: (Oxygen index). An indicator of kerogen type measured in (mgCO₂/gTOC), OI: S3*100/TOC. PI: (Production index) (S1/(S1 + S2), indicative of free petroleum content

GP genetic potential. VR vitrinite reflectance

Fig. 7 $S2$ versus TOC plots for coal and shale samples of Maiganga coal mine, Gongola Basin. The plot indicates type III kerogen (gas prone) for the samples. The shale samples are organically lean. After Langford and Blanc-Valleron (1990)
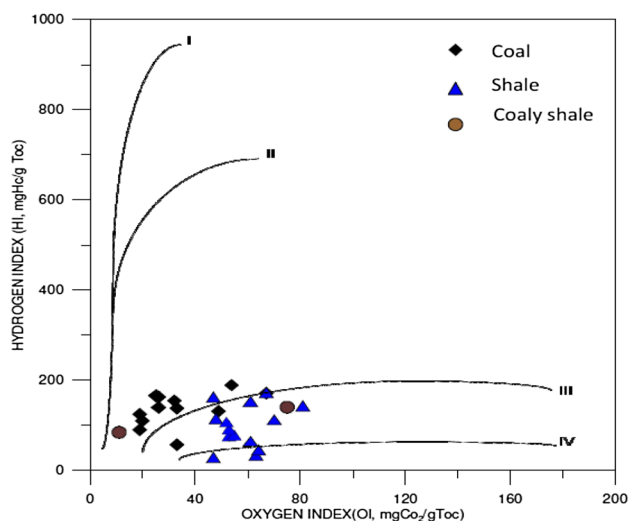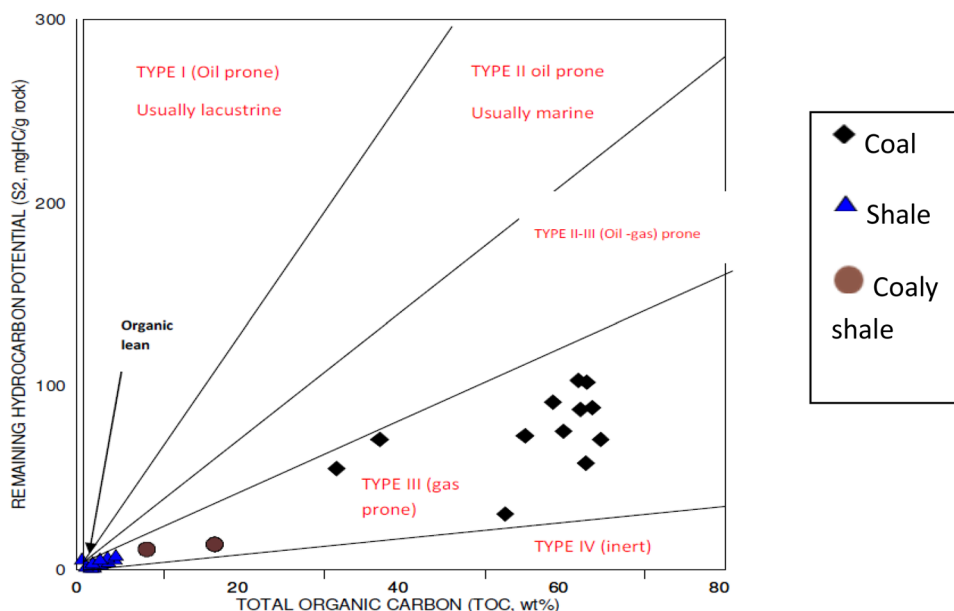

Fig. 8 Plot of HI versus OI for the classification of kerogen (1–1 V) of coal, coaly shale and shale samples from Maiganga mine, Gongola Basin. Most of the samples fall within type III kerogen (gas prone). After Van Krevelen (1961)

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_{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 $S2/S3$ 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 $S2$ 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 $S2$ 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

Fig. 9 Plot of hydrogen index (HI) against T_{max} of coal, coaly shale and shale samples from Maiganga mine, Gongola Basin. Most of the samples are immature and fall within type III kerogen (gas prone) typical of terrestrially sourced organic matter
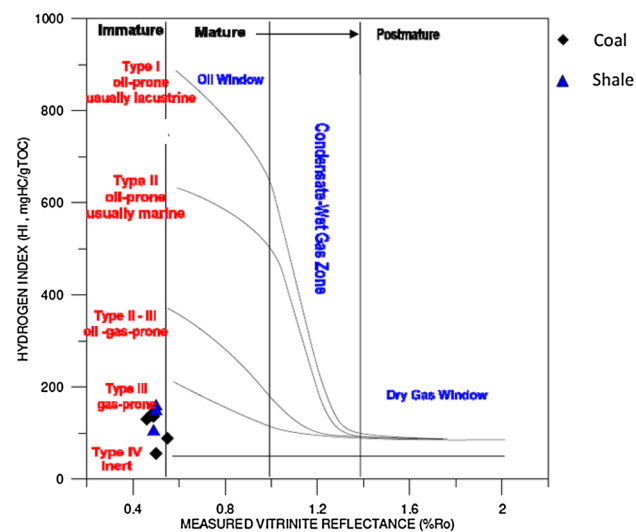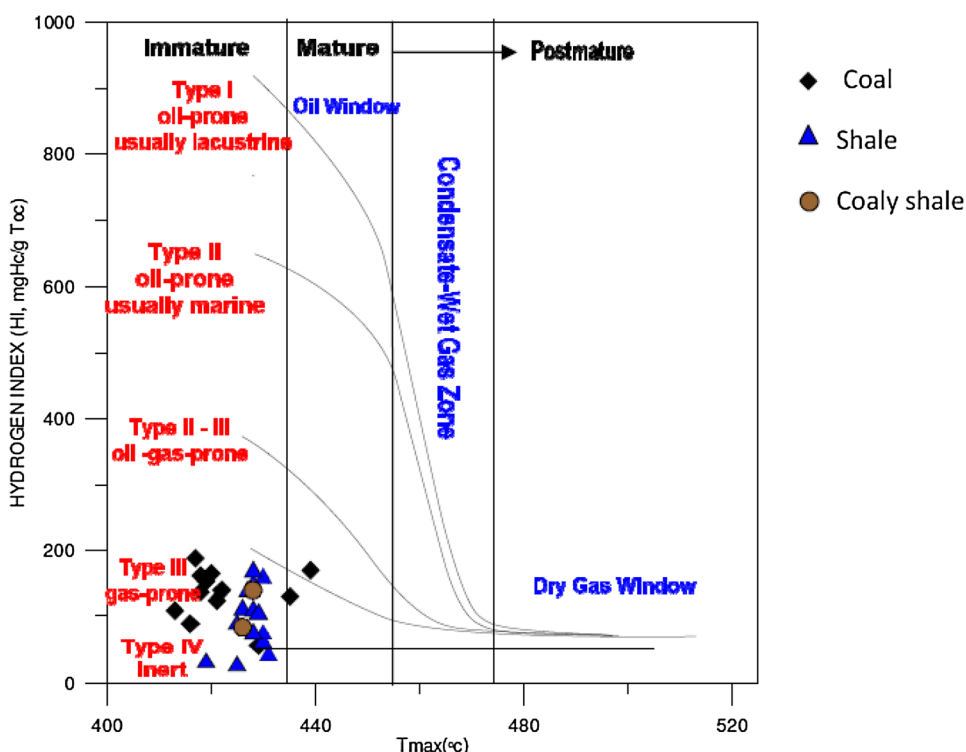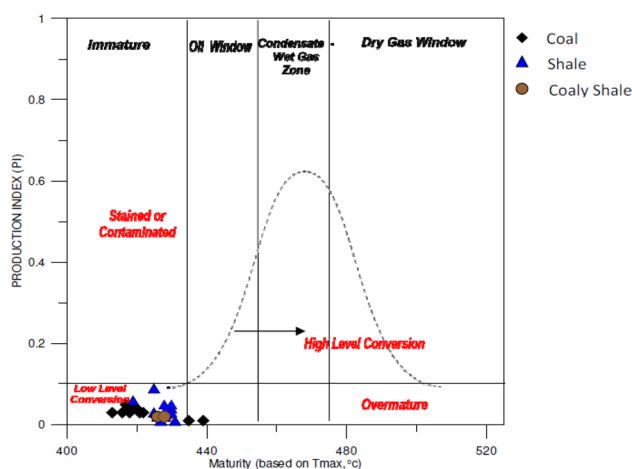

Fig. 10 Plot of hydrogen index (HI) against vitrinite reflectance of coal and shale samples from Maiganga mine, Gongola Basin. Most of the samples are immature and fall within type III kerogen (gas prone) typical of terrestrially sourced organic matter

Fig. 11 Plot of production index (PI) against T_{max} of coal, coaly shale and shale samples from Maiganga mine, Gongola Basin. All the samples are immature and have low level conversion of kerogen to oil

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
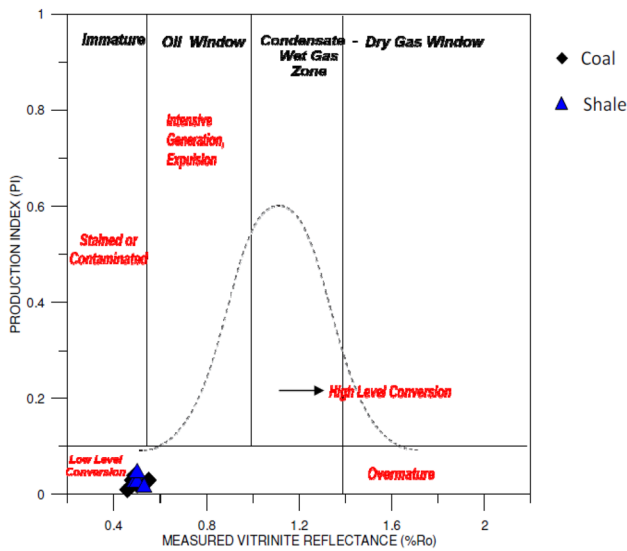
Fig. 12 Plot of production index (PI) against vitrinite reflectance of coal and shale samples from Maiganga, Gongola Basin. All the samples are immature and have low level conversion of kerogen to oil

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 (n-C_{17}) over phytane (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),

Table 2 n-Alkanes, isoprenoids parameters and percentage composition of the extracts from coal and shale of Maiganga, Gombe Formation

S/n	Sample No./code	Lithology/rank	Depth (ft)	Ext (gm)	Ext (ppm)	Pr/Ph	Pr/n-C_{17}	Ph/n-C_{18}	$n$$C_{17}$/$n$$C_{29}$	$n$$C_{18}$/$n$$C_{19}$	C_{range}	CPI	SAT%	ARO%	NSO%	ASPH%	Total Recov%
1	MG1G	Coal (SB)	105	42	6000	2.15	2.82	0.74	1.21	0.01	C_{11-41}	3.30	9.09	9.09	39.09	42.73	100
2	MG2H	Coal (SB)	62	50.6	7229	3.51	3.63	0.92	1.10	0.03	C_{10-41}	3.28	6.64	6.27	33.95	53.14	100
3	MG2O	Coal (SB)	59	63	9000	2.94	1.30	0.34	1.32	0.03	C_{11-41}	3.54	8.21	4.40	30.21	57.18	100
4	MG4K	Coal (SB)	14	891	15,872	2.55	0.71	0.26	0.96	0.04	C_{11-41}	3.76	14.3	2.92	32.75	50.00	100
5	MG3S	C. Shale	38	190	27,035	2.37	1.16	0.46	0.85	0.07	C_{11-35}	2.74	9.52	3.17	31.75	55.56	100
6	MG4D	Shale	23	19.9	2985	2.13	0.56	0.28	1.15	0.08	C_{11-41}	3.02	24.1	5.36	41.96	28.57	100
7	BH15B	Shale	157	60.9	7382	0.95	0.34	0.18	1.87	0.15	C_{9-39}	2.82	31.3	4.17	43.75	20.83	100

Key: Ext-Extract (gm, ppm) SB sub-bituminous, Pr pristane, Ph phytane, CPI carbon preference index, Sat saturate, Aro aromatic, NSO Resin, $ASPH$ asphaltene, $Recov$ recovery

Table 3 Selected Saturate biomarker source and depositional indices of coal and shale of Maiganga, Gombe Formation

S/n	Sample no/code	%C$_{27}$α ββS	%C$_{28}$α ββS	%C$_{29}$αββS	%C$_{27}$αααR	%C$_{28}$αααR	%C$_{29}$αααR	Ga/Ho	D/Ho	H35/H34	C$_{23}$/C$_{24}$ TT	St/Ho
1	MG1G	3.8	6.4	89.9	9.9	51.2	38.9	0.2	0.19	0.59	1.51	0.06
2	MG2H	3.4	4.4	92.2	22.0	38.0	40.0	0.25	0.10	0.64	1.46	0.10
3	MG2O	5.3	44.2	50.6	12.5	39.3	48.3	0.29	0.05	0.51	1.97	0.12
4	MG4K	–	–	–	–	–	–	0.23	0.2	–	–	–
5	MG3S	–	–	–	–	–	–	–	3.6	–	–	–
6	MG4D	–	–	–	–	–	–	0.12	0.67	–	1.72	–
7	BH15B	26.6	29.4	44.0	34.1	24.3	41.7	0.12	0.11	–	1.67	0.35

Key: *Ga* gammacerane, *Ho* hopane, *H* hopane, *TT* tricyclic terpane, *St* sterane

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$_{27}$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 dibenzothiophene 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$_{28}$ MAS in the shale sample BH15B which may suggest mixed sources. The presence of 1,2,5- and 1,2,7-trimethylnaphthalene (TMN) in the coal samples indicates both angiosperm and gymnosperms material contribution to the organic matter that formed the coals (Killops and Killops 2005).
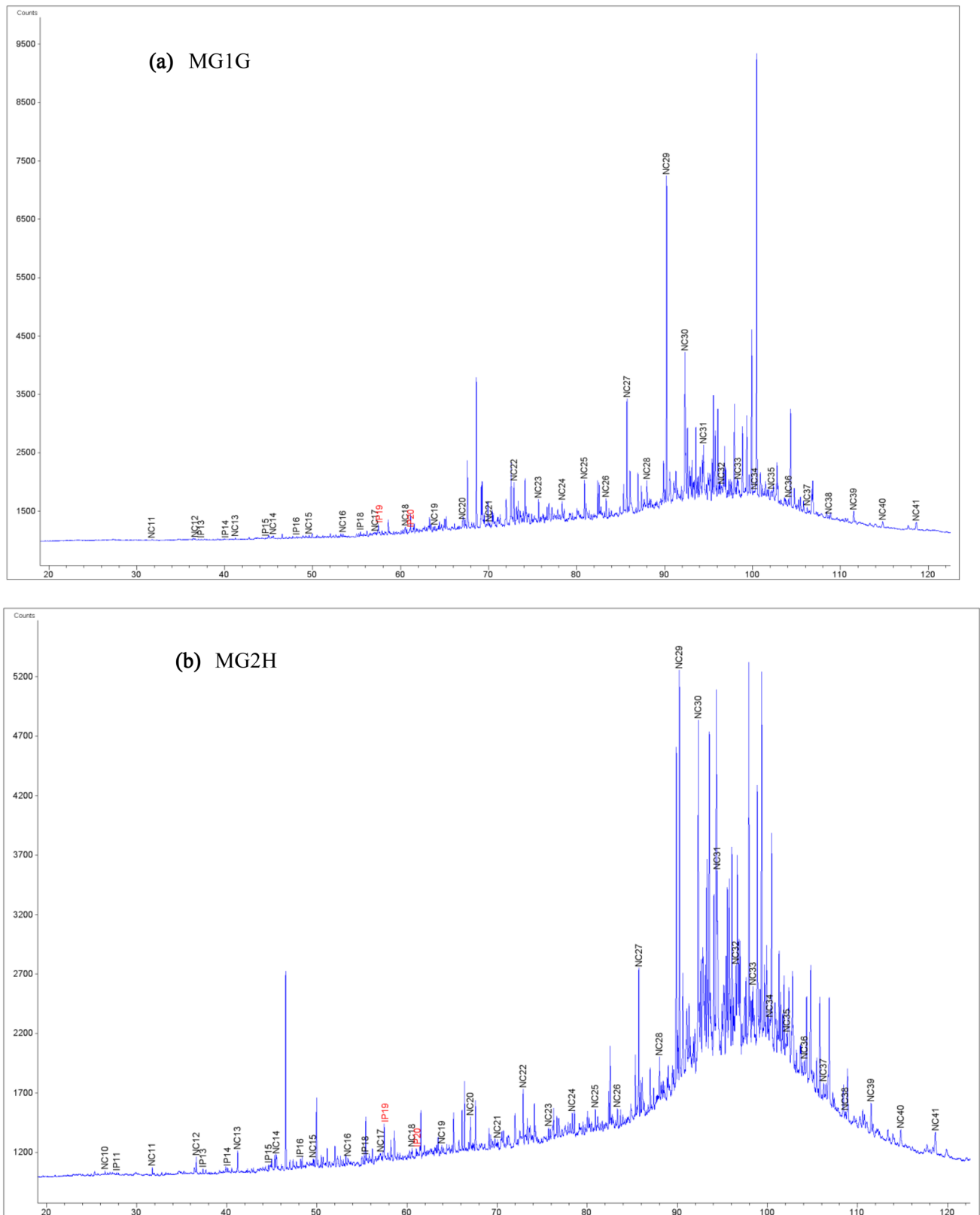
Fig. 13 Ion chromatograms of samples showing the distribution of *n*-alkanes. **a** MG1G, **b** MG2H, **c** MG2O, **d** MG4K, **e** MG3S, **f** MG4D, **g** BH15B
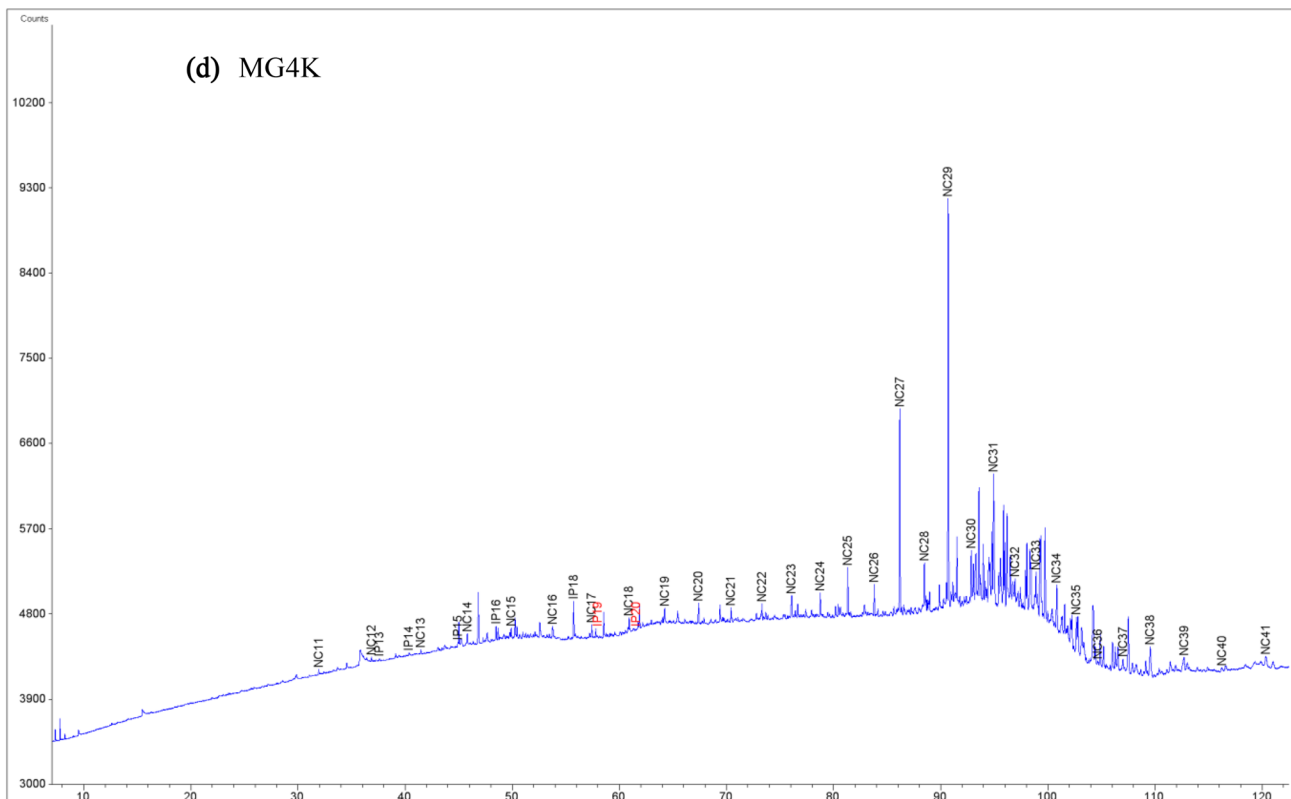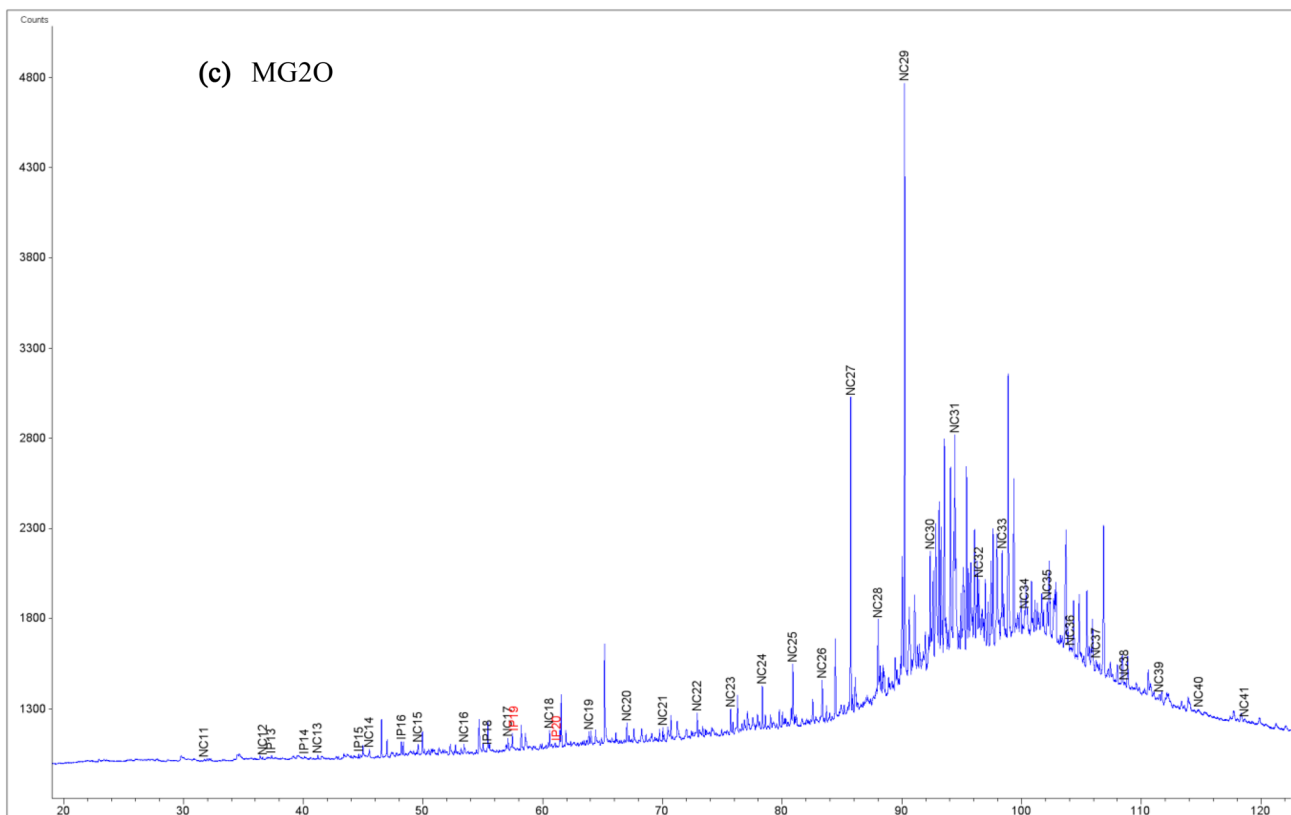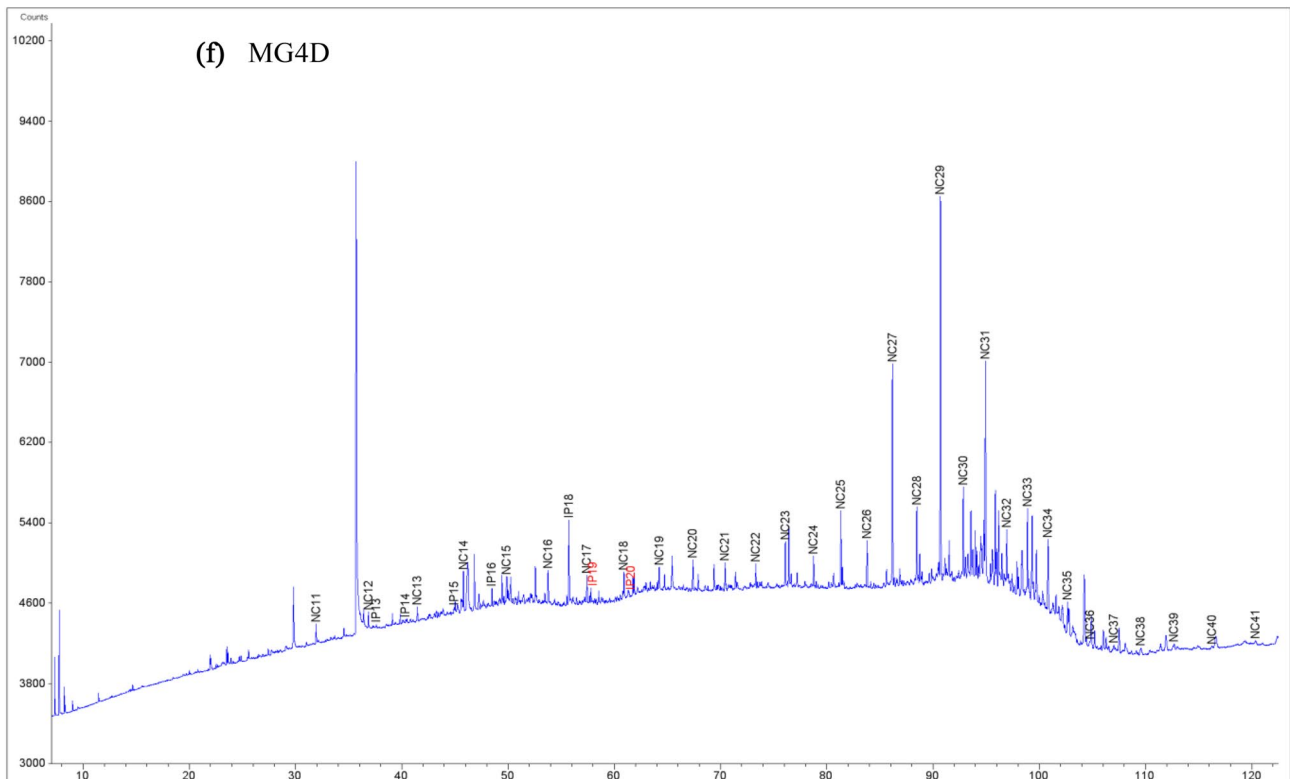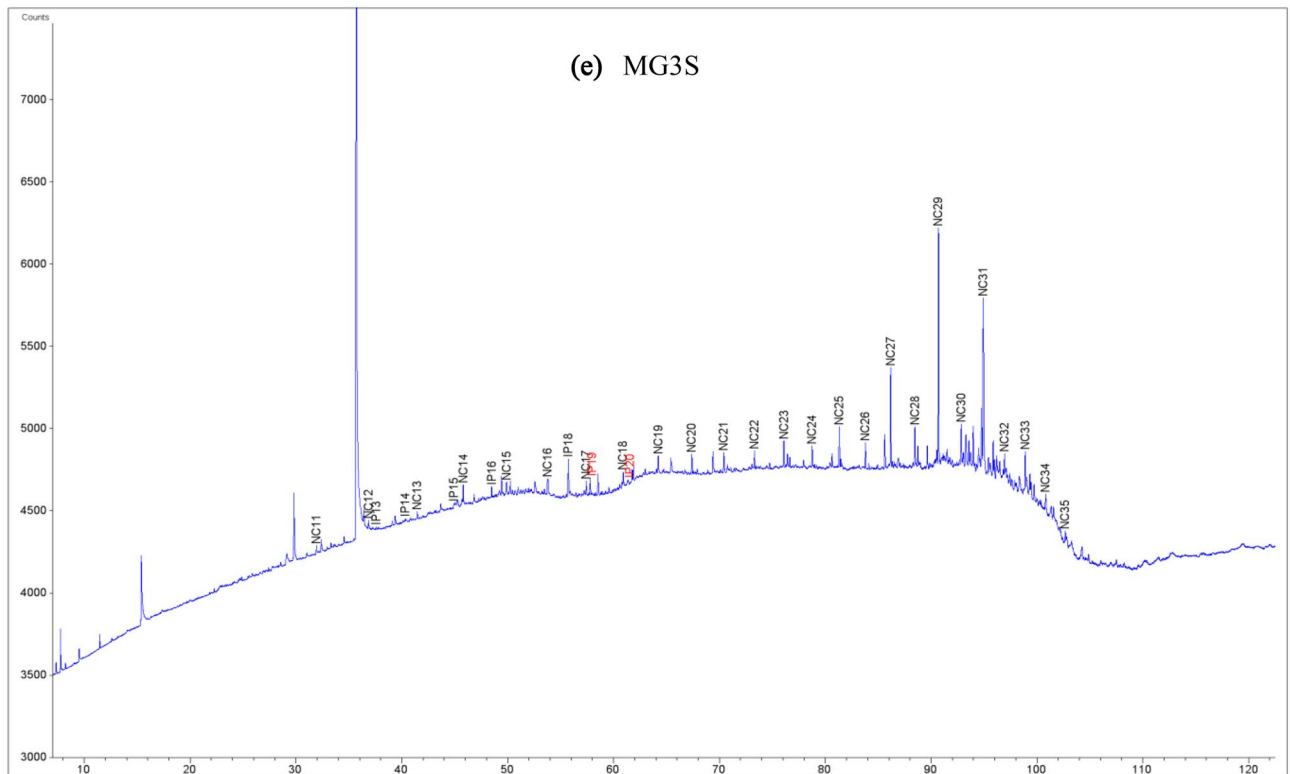
Fig. 13 (continued)

(e) MG3S

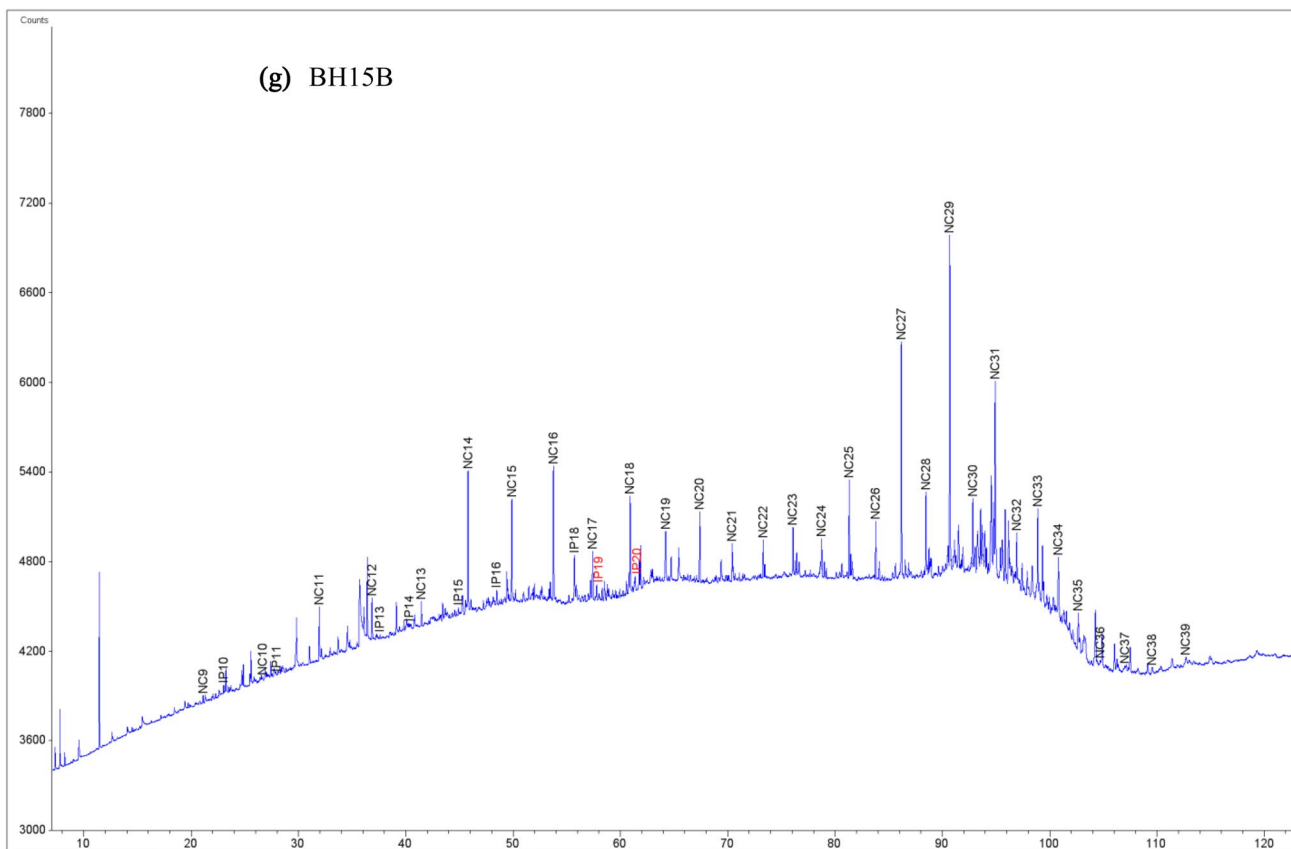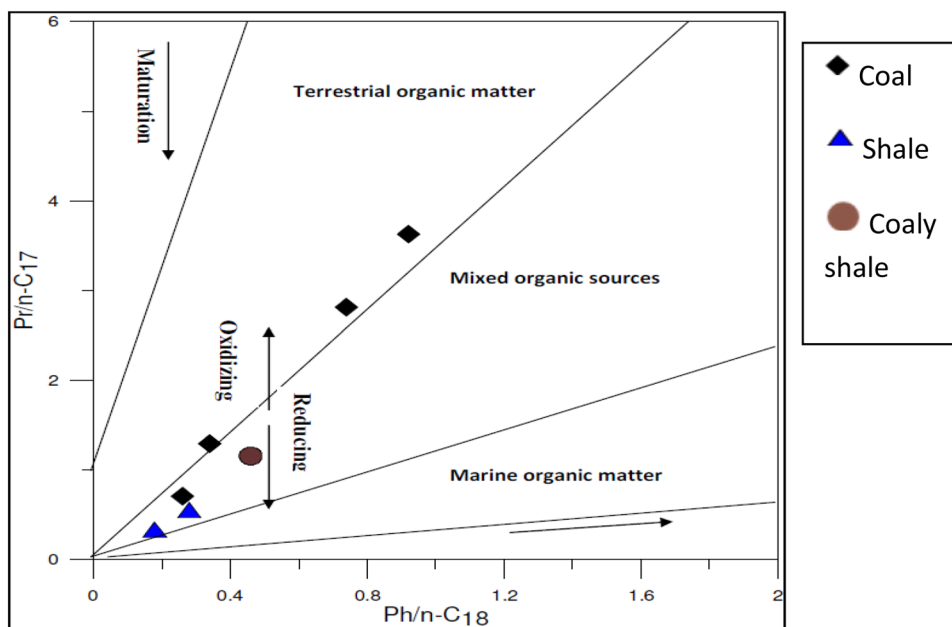(f) MG4D

Fig. 13 (continued)

Fig. 13 (continued)

Fig. 14 Plot of Pr/n-C$_{17}$ versus Ph/n-C$_{18}$ of coal and shale samples of Maiganga, Gombe formation. The coals fall within terrestrial sources in oxic depositional settings, shales plotted within the mixed sources. after Peters et al. (2005)
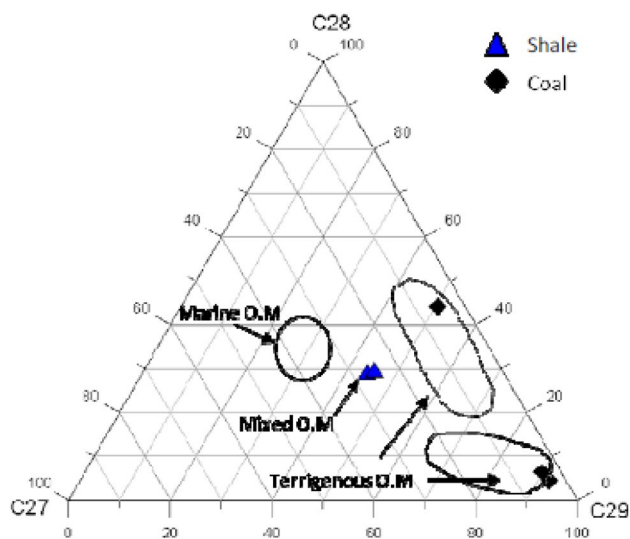
Fig. 15 Ternary diagram of the distribution of C_{27}, C_{28}, C_{29} steranes in Maiganga coal and shale, Gombe Formation. The coals are plotted in terrigenous portion due to predominance of C_{29}. After Moldowan et al. (1985)
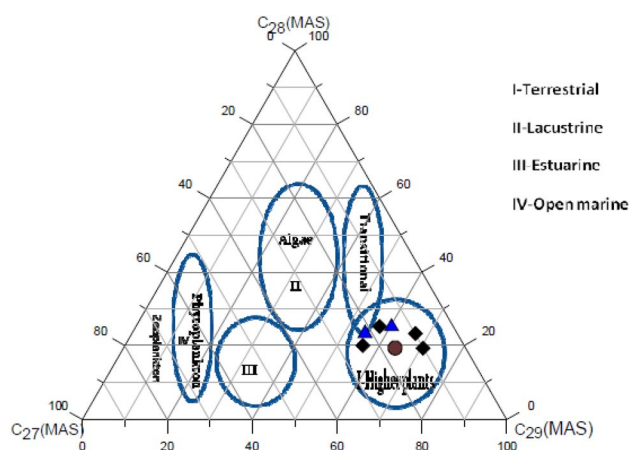

Fig. 16 Ternary diagram of the distribution of C_{27}, C_{28}, C_{29} monoaromatic steroid in Maiganga coal, shale and coaly shale, Gombe Formation. The coals are plotted in terrigenous portion due to predominance of C_{29} MAS. After Moldowan et al. (1985)

## Thermal maturity and hydrocarbon potential

The thermal maturity of all the samples is represented by T_{max} range of 413–439 °C. The T_{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_{max} less than 435 °C with exception of two coal samples having 435 °C and 439 °C. The spiked T_{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 level to marginal maturity and late diagenetic stage for oil generation (Killops and Killops 2005). The plot of HI versus T_{max} (Fig. 9), HI versus VR (Fig. 10), PI versus T_{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 it 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

Fig. 17 Organic matter source discrimination of Maiganga samples (After Ficken et al. 2000). The source of the organic matter in the samples was probably from land plant source
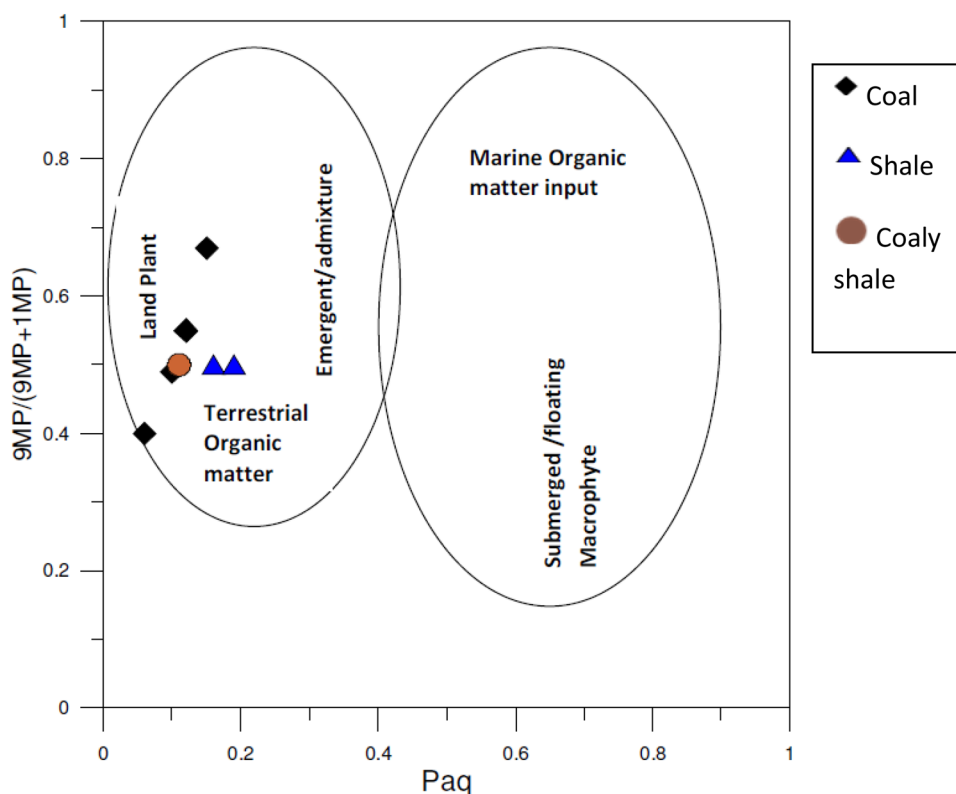
indicating low maturity status (Table 7) and hence have not reached onset of oil generation. The moretane/hopane and $T_s/T_s + T_m$ ratios in the samples range from 0.42–0.68 to 0.07–0.12, respectively, also indicate low maturity (Table 6).

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	MG3S	–	–	–	–	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, *9MP* 9-methylphenanthrene, *1MP* 1-methylphenanthrene, Paq $= nC_{23} + nC_{25}/nC_{23} + nC_{25} + nC_{29} + nC_{31}$—alkanes
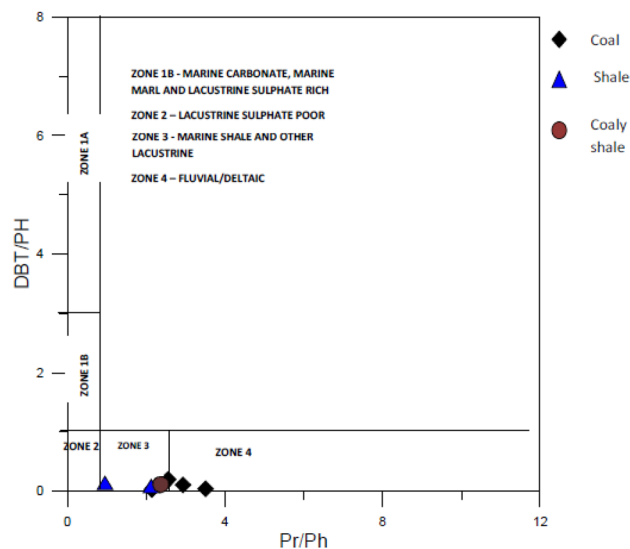

Fig. 18 Cross plot of dibenzothiophene/phenanthrene (DBT/PH) versus pristane/phytane (Pr/Ph) of coaly and shaly source rocks of Maiganga, Gombe Formation (Hughes et al. 1995). The coals, shale and coaly shale plotted in Zones 3 and 4 (Marine shale, lacustrine and fluvial/deltaic environment)
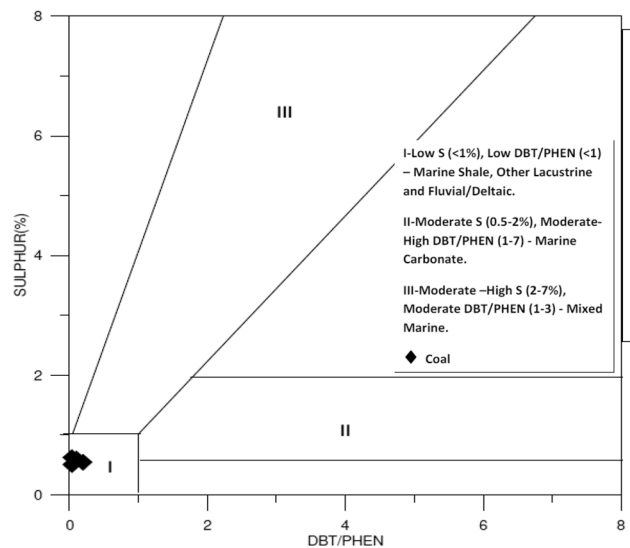

Fig. 19 Cross plot of total sulphur versus dibenzothiophene/phenanthrene (DBT/PHEN) for Maiganga coal (after Hughes et al. 1995). The coal samples are characterized by low sulphur, and low DBT/PHEN (< 1) indicates fluvio-deltaic deposition

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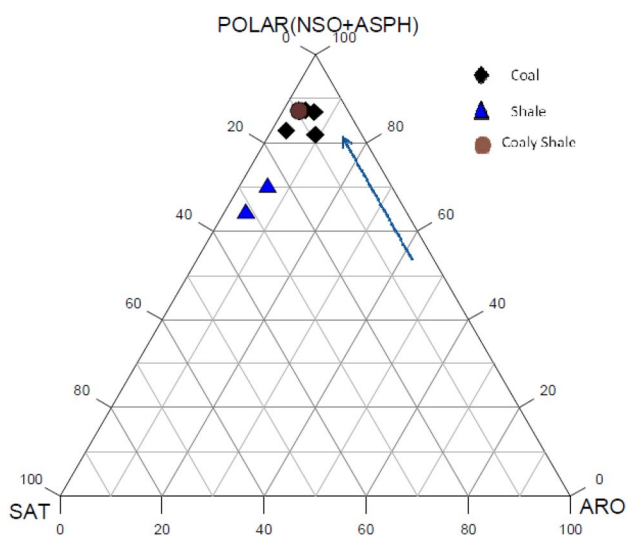
Fig. 20 Ternary diagram showing the proportion of saturated hydrocarbons, aromatic hydrocarbons and polar fraction (NSO + asphaltenes) in extracts of coal, shale and coaly shale source rocks of Maiganga, Gombe Formation. The samples have higher percentage of heterocompounds indicated by the arrow. After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

Table 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} $\alpha\alpha\alpha$)	0.1–0.28	0.55	0.80	IM–LM
2	(C_{29}) $\beta\beta S/(\beta\beta S+\alpha\alpha R)$	0.04–0.61	0.70	0.90	IM–LM
3	Diasterane/C_{27} $\alpha\alpha\alpha$ 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	$T_s/(T_s+T_m)$	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

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} $\alpha\alpha\alpha$)	$C_{29})\beta\beta S/(\beta\beta S+\alpha\alpha R)$	T_s/T_s+T_m	Mor/Hop	H32$S/(R+S)$	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_s/T_s+T_m = Trisnorhopane, Mor/Hop = moretane/hopane, TT/Hop = tricyclic terpane/hopane, H32$S/(R+S)$ = homohopanes, $S/(S+R)$(C29 $\alpha\alpha\alpha$), C29)$\beta\beta S/(\beta\beta S+\alpha\alpha R)$, SB (sub-bituminous)

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	–	0.33	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 = methyldibenzothiophene 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

humic coals dominated by huminite could generate oil, though liptinite macerals and are more oil prone than huminite because of its high paraffinicity in structure and its ability to generate long-chain aliphatic component (> C_{20}–C_{25}) (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 alginite 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.
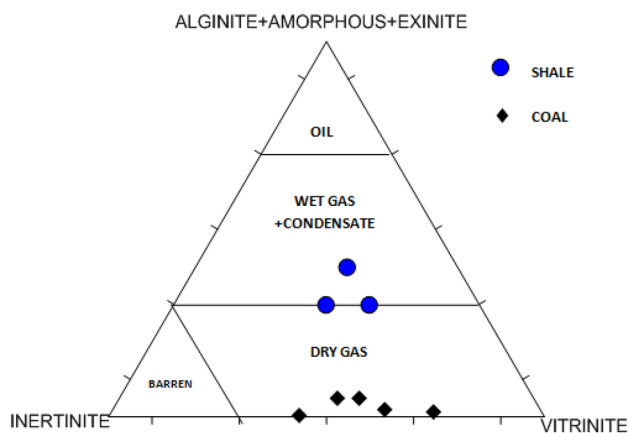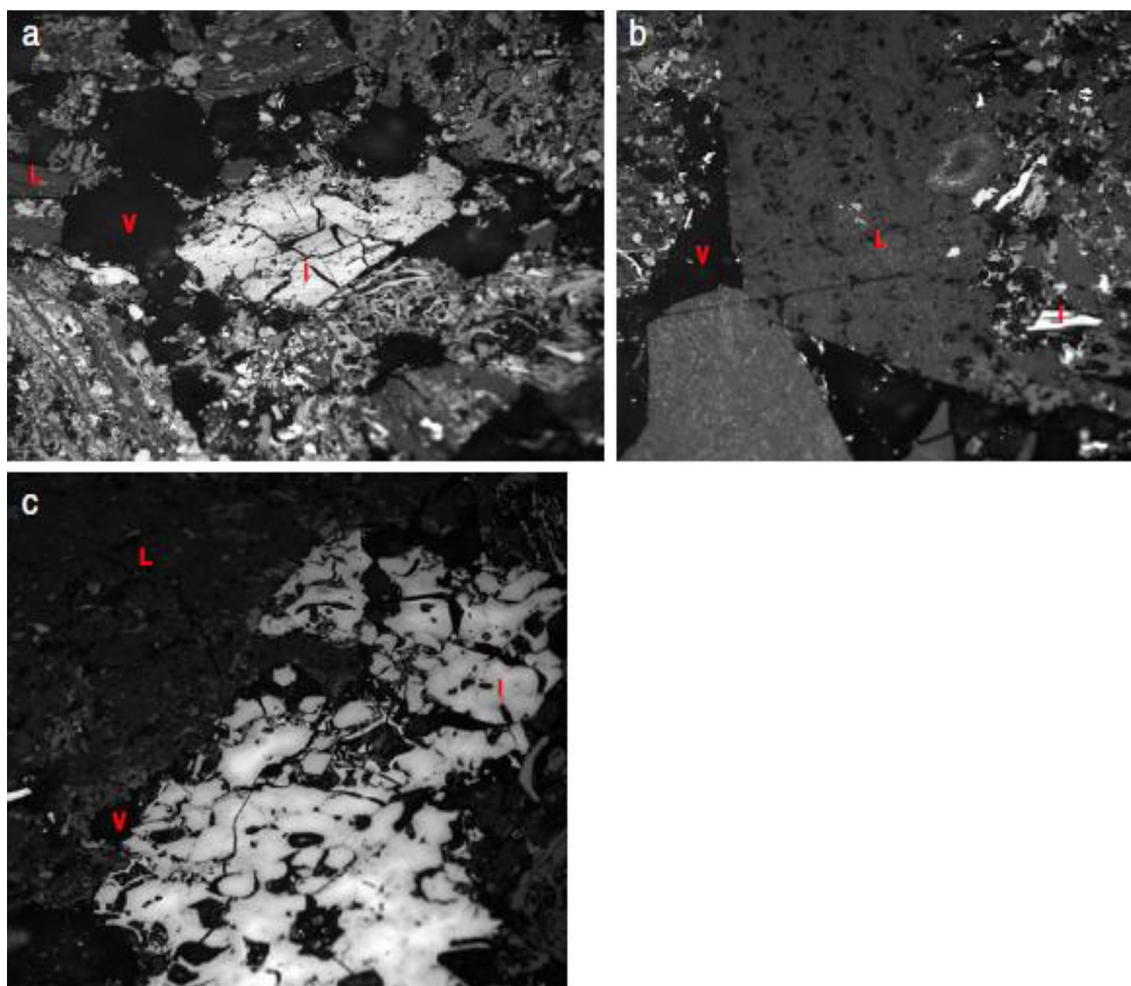

Fig. 21 Ternary plot indicating the proportion of the vitrinite, inertinite and liptinite and the corresponding hydrocarbon compared with the reference diagram of Tissot and Welte (1984). Plot indicates that the coal and shales are gas prone

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
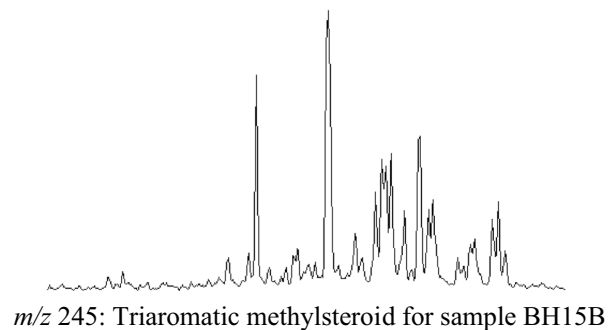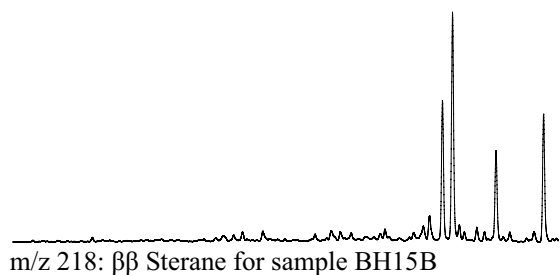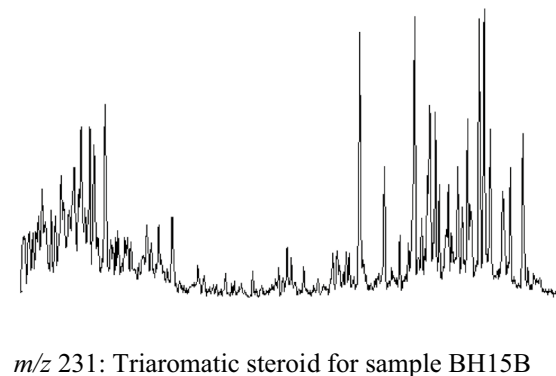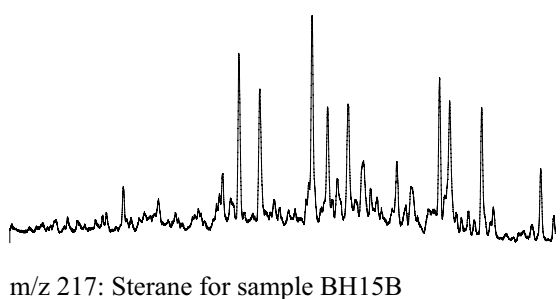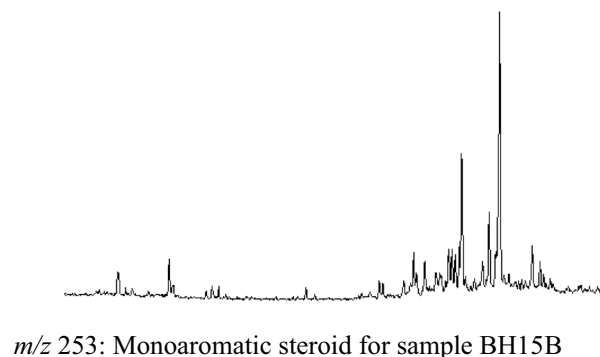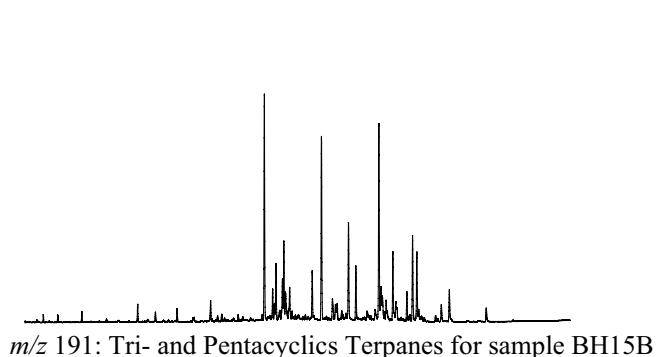
Conclusion

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.

Appendix

m/z 191: Tri- and Pentacyclics Terpanes for sample BH15B

m/z 253: Monoaromatic steroid for sample BH15B

m/z 217: Sterane for sample BH15B

m/z 231: Triaromatic steroid for sample BH15B

m/z 218: ββ Sterane for sample BH15B

m/z 245: Triaromatic methylsteroid for sample BH15B

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