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Geochemical and Organic Petrological Evaluation of Organic Matter from Tertiary Sediments of Baga Field in Chad Basin, Northeast Nigeria

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ABSTRACT: The Upper Cretaceous sediments in Bornu are considered an important regional source rock in the Chad Basin. This study therefore evaluated the geochemical and organic petrological evaluation of organic matter in tertiary sediments of Chad Basin, Northeast Nigeria by collecting 25 cutting samples from these organic-rich shale sediments from four wells drilled in Baga field in the Nigeria sector (Bornu) of the Chad Basin, in order to geochemically assess the type of organic matter, thermal maturity, and palaeoenvironmental conditions. Results reveal that Bornu sediments contain high organic matter more than 2.0 wt% TOC and have an excellent oil-generation potential. This is supported by high bitumen extractions and hydrocarbon yields with values 5827 and 3547 ppm, respectively. The investigated biomarkers indicated that the shale sediments contain a high abundance of C_{27} regular sterane concentrations, high C_{27}/C_{29} regular sterane ratios and relatively low value of the biomarker sterane/hopane ratio as well as the presence of tricyclic terpanes. A mainly suboxic to relatively anoxic preservation conditions is inferred from Pr/Ph ratios (1.03–2.53). This is support by normal alkane distributions, which are characterized by dominance of types I/II kerogen and low medium molecular weight n-alkane compounds, respectively. This is further supported by lower amounts of acyclic isoprenoids compared to n-alkanes (e.g., pristane/n-C_{17} and phytane/n-C_{18} ratios). This is also supported by a mixture of algal and amorphous organic matter that was deposited in a lacustrine environment identified from kerogen microscopy. Based on the analyzed biomarkers, triterpanes and terpanes thermal maturity indicates that the Bornu sediments organic matters have entered into early mature stage for oil generation. This is also supported by vitrinite reflectance values of 0.57–0.71 % Ro indicate that these organic matters have reached oil window maturity.

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Petroleum source rocks are the primary component of the petroleum system concept introduced by Magoon and Dow (1994). They constitute the precursors of petroleum which, under favourable conditions, may subsequently migrate to reservoirs and be sealed to form accumulation. Nigeria's current national petroleum reserves asset (proven), put at 32 billion bbl of oil and about 170 trillion standard ft^3 of gas (Nexant, 2003), derives solely from the Niger Delta onshore and offshore. Some exploration campaigns have been undertaken in the inland basins with the aim of expanding the national exploration and production base and thereby add to the proven reserves asset (Obaje *et al*., 2004). Some exploration campaigns have been undertaken in sedimentary basins of Northern Nigeria with the aim to expanding the national exploration and production base and to thereby add to the proven reserves asset (Obaje *et al*.,

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2006). Sedimentary basins of Northern Nigeria comprise the Middle and Upper Benue Trough, the southeastern sector (Bornu) of the Chad Basin, the Mid-Niger (Bida) Basin, and the Sokoto Basin. However, these inland basins have continued to frustrate the efforts of many explorers, principally because of the poor knowledge of their geology and the far distance from existing infrastructure (discovery must be large enough to warrant production investments), and for these reasons, many international companies have turned their focus away from frontier onshore to frontier deep-water and ultra-deep-water offshore (Obaje *et al*., 2006). This study therefore evaluated the geochemical and organic petrological evaluation of organic matter in tertiary sediments of Baga field in Chad Basin, Northeast Nigeria

MATERIALS AND METHODS

The sedimentary basins of Northern Nigeria are one part of a series of Cretaceous and later rift basins in Central and West Africa whose origin is related to the opening of the South Atlantic (Figs. 1&2). Commercial hydrocarbon accumulations have recently been discovered in Chad and Sudan within this rift trend. In SW Chad, exploitation of the Doba discovery (with an estimated reserve of about 1 billion barrels of oil) has caused the construction of a 1070 km-long pipeline through Cameroon to the Atlantic coast. In the Sudan, some "giant fields" (*Unit 1 & 2, Kaikang, Heglig*, etc.) have been discovered in the Muglad basin (Mohamed *et al*., 1999). The major source rocks and reservoirs are in the Aptian-Albian-Cenomanian continental deposits of the Abu Gabra and Bentiu formations, respectively, which are similar and correlatable to the well-developed Bima Sandstone in the Nigerian upper Benue trough. In Niger Republic, oil and gas shows have also been encountered in Mesozoic–Cenozoic sequences in the East Niger graben, which is structurally related to the Benue-Chad-Sudan-Libyan rift complexes (Zanguina *et al*., 1998). Within the sedimentary basins of Northern Nigeria, the Nigerian National Petroleum Corporation (NNPC) through its frontier exploration services arm (NAPIMS) has drilled some wells in the Nigerian sector of the Chad Basin and only gas shows were encountered. The first well in the Benue Trough region, *Kolmani-River-1*, drilled by Shell Nigeria Exploration and Production Company (SNEPCO) to a depth of about 3000 m in 1999 encountered some 33 billion standard cubic feet of gas and little oil (that has been the only well drilled by that company in that area to date). Two other wells, *Kuzari-1* and *Nasara-1*, drilled by Elf Petroleum Nigeria Limited (Total Fina Elf) in 1999 to a depth of 1666 m and Chevron Nigeria Limited (ChevronTexaco) in 2000 to a depth of about 1600 m, respectively, were reportedly dry (Obaje *et al*., 2006). With this development, it has become necessary to adequately evaluate and characterize the petroleum source rocks in this basin by using well established geochemical techniques. At the core of any petroleum system is a good-quality source rock (total organic carbon (TOC) > 0.5%, hydrogen index (HI) > 150 $mgHCg^{-1}TOC$, liptinite content > 15%, $T_{max} \geq 430^{o}C$, and R_o 0.5-1.2%, biomarker validation). However, other petroleum system elements must also include, apart from established source rocks, reservoir and seal lithologies, establishable trapping mechanisms, and favourable regional migration pathways (Obaje *et al*., 2004). The aims of this study were to evaluate a shale sample from the basin using modern techniques of petroleum geochemistry in order to: (i) assess in detail the quality of its organic matter; (ii) evaluate its thermal evolution, and (iii) highlight its potential as a source. The results of this study may stimulate further interest in petroleum exploration and exploitation in the Chad basin.

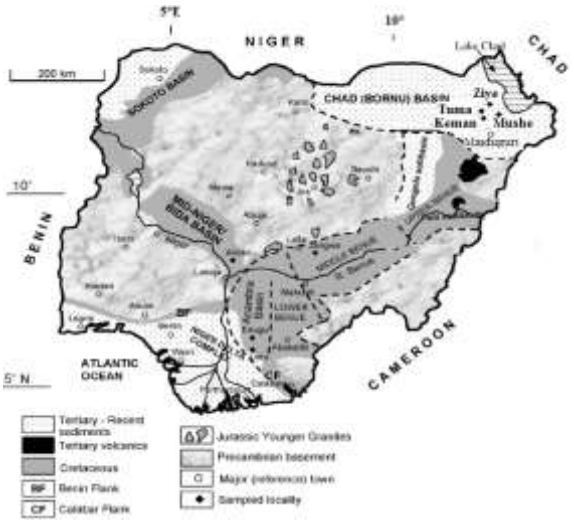

Fig. 1: Sketch geological map of Nigeria showing the inland basins and sample localities (Obaje *et al*., 2006).

A total of 25 cutting samples from four wells in the Bornu (Baga) field, Nigeria sector (sub-basin) of the Chad Basin were analyzed (Table 1). The samples were collected from organic-rich shale and claystone intervals within the Bornu sediments (Fig. 2). Geochemical and some organic petrological analyses include determination of total organic matter (TOC) and sulfur (TS) contents, bitumen extraction, gas chromatography – mass spectrometry (GC-MS), and vitrinite reflectance measurements. Elemental content was subsequently performed on approximately 100 mg pulverized whole sample using elemental analyzer instrument (Multi EA2000 model) to determine the TS and TOC contents

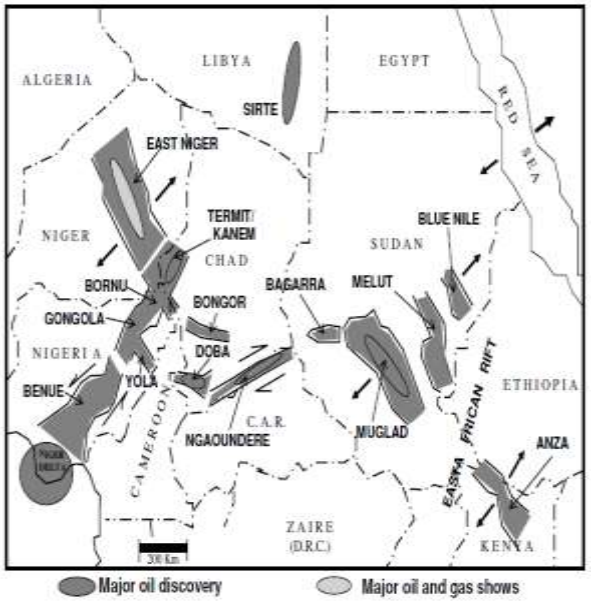

Fig. 2: Regional tectonic map of western and central African rifted basins showing the relationship of the Muglad, Doba and East Niger Basins to the Benue Trough/Gongola Basin. Locations of regional shear zones (marked with half-arrow) and major zones extension (complete arrow) are shown (Adapted from Schull, 1988).

Bitumen extractions were performed on the powdered samples using a Soxhlet apparatus for 72 h using an azeotropic mixture of dichloromethane and methanol (CH_3OH) (93:7v/v). The extracts were separated into saturated hydrocarbon, aromatic hydrocarbon, and NSO compounds by liquid column chromatography. A chromatographic column (30×0.72 cm) was packed silica gel of 60–120 mesh that was activated for 2 h at 120 °C and capped with a few centimeters of alumina. Only the saturated fractions were analyzed in this study. The saturated fractions were dissolved in petroleum ether and analyzed using GC-MS. The GC-MS analysis was performed on an Agilent 5975B inert MSD mass spectrometer with a gas chromatograph attached directly to the ion source (70 eV ionization voltage, 100 mA filament emissions current, 230 °C interface temperature). Samples for petrographic examinations were made using standard petrographic preparation techniques. Petrographic examinations were carried out under oil immersion in a plane polarized reflected light, using a Leica DM 6000M microscope and Leica CTR6000 photometry system equipped with fluorescence illuminators. The filter system consists of BP 340–380 excitation filters, a RKP 400 dichromatic mirror, and a LP425 suppression filter. Measurement of vitrinite reflectance was carried out using a microscope under reflected white light, with ×50 oil immersion objectives using immersion oil with a refractive index (ne) of 1.518 at 23 °C. A sapphire glass standard with 0.589 % reflectance value was used for calibration. Reflectance measurements were determined in the random mode (Rrand) on vitrinite maceral at a wavelength of 546 nm, and the values reported were arithmetic means of at least 25 measurements per sample. Kerogen typing analysis was carried out using the single scan method, where identification of kerogen was done using both normal reflected "white" light and ultraviolet (UV) light.

Regional Geologic Setting: The Benue Trough of Nigeria is a rift basin in central West Africa that extends NNE-SSW for about 800 km in length and 150 km in width. The trough contains up to 6000 m of Cretaceous–Tertiary sediments of which those pre-dating the mid-Santonian have been compressionally deformed, faulted, and uplifted in several places. Its southern limit is the northern boundary of the Niger Delta, while to the north it is bordered by the Chad Basin. The Trough can be divided into Lower, Middle and Upper portions (Fig. 1). It contains up to 6000 m of Cretaceous – Tertiary sedimentary rocks, of which those pre-dating the mid-Santonian have been folded, faulted and uplifted. The Upper Benue Trough can be subdivided into the east–west trending Yola Basin (or "Arm") and the north–south trending Gongola Basin (Fig. 1). Guiraud (1990) and Dike (2002) identified a third basin, the NE – SW trending Lau Basin or Main Arm. Reviews on the geology of the Benue Trough, and particularly the Upper Benue Trough, have been presented by Petters, 1982; Petters and Ekweozor, 1982; Benkhelil, 1982; Dike, 1993, 2002; Obaje, 1994; Zaborski *et al*., 1997; and Zaborski, 2000, 2003.

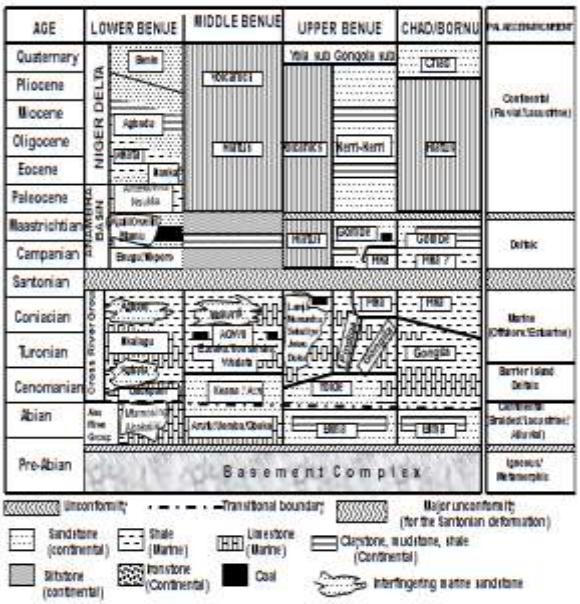

Fig. 3: Stratigraphic successions in the Benue Trough, the Nigerian sector of the Chad Basin and the relationship to the Niger Delta (Obaje *et al.*, 2006).

The stratigraphic succession in the Upper Benue Trough is illustrated in Fig. 3. The oldest sediments consist of continental deposits of the Late Jurassic to Albian Bima Formation which rest unconformably on Precambrian basement rocks. The Bima Formation is conformably overlain by the Cenomanian continental to marine Yolde Formation, which consists of sandstones and shales at the base, and sandstones, shales and calcareous sandstones above. The formation is overlain by contemporaneous marine successions of the Pindiga and the Gongila/Fika Formations in the Gongola Basin, and their lateral equivalents (Dukul, Jessu, Sekuliye, Numanha and Lamja Formations) in the Yola Basin. Zaborski *et al.* (1997) proposed that the Pindiga Formation consists of five members in the Gongola Basin. The youngest Cretaceous sedimentary rocks in the Upper Benue Trough are restricted to the Gongola Basin, and are represented by the lacustrine to deltaic Gombe Formation which unconformably overlies the pre-mid–Santonian sequences in some places. The continental claystones (sandstones and siltstones) and shales of the Paleogene Kerri – Kerri Formation mark the end of sedimentation in the Upper Benue Trough.

Details on the evolution and stratigraphic framework of the Chad Basin have been given in Avbovbo *et al.* (1986) and Olugbemiro *et al.* (1997). Details on the stratigraphic successions in the Benue Trough and the Chad Basin and as they relate to the Anambra Basin and the Niger Delta are depicted on Fig. 3.

RESULTS AND DISCUSSION

Maceral Properties: Under incident light, microscopy indicates that the organic matter of the Bornu sediments in the Chad Basin is dominated by liptinitic material of which fluorescing amorphous organic matter (AOM), intimately associated with mineral matter, constitutes the major component (Fig. 3a–c). The AOM appears well aggregated, light brown in color and irregular in shape under normal white light (Fig. 3a–c) and commonly shows yellow fluorescent under ultraviolet light excitation (Fig. 3b, c). Alginite with morphology similar to the extant Botryococcus alga is observed in all the samples (Fig. 3d–f). The kerogen composition implies a high proportion of type II kerogen (alginite) followed by smaller amounts of type I kerogen (AOM + liptinite). The common association of alginite with the fluorescing AOM indicates a potential for oil prone and was derived to a large extent from the degradation of algal material or other phytoplanktonic origin (Hakimi *et al.* 2012a, 2013; Makeen *et al*., 2013). In addition, the mean vitrinite reflectance (%Ro) for the studied Chad Basin samples that ranges between 0.57 and 0.71 % (Table 1) indicate that these sediments are thermally early mature to mature and have reached oil generation window. This thermal maturity rank is also supported by relatively low fluorescence of alginite and amorphous organic matter under UV light excitation (Fig. 3b–f).

Geochemical Evaluation: The ability of source rocks to generate hydrocarbons is determined by the kerogen's quantity of organic matter, expressed as total organic carbon (TOC) content. The TOC determination was carried out on 25 samples consisting of claystones and shales (Table 1). The analyzed Bornu sediments have high TOC values ranging from 1.20 to 7.20 wt%, ranking these samples as good to very good source rocks (Tissot and Welte, 1984).

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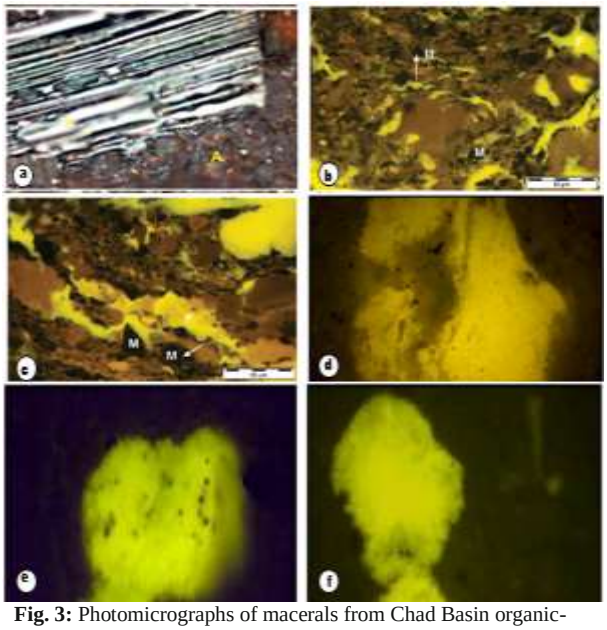

Fig. 3: Photomicrographs of macerals from Chad Basin organic-rich sediments in the Nigerian sector sub-basin; amorphous organic matter (a–c) and alginite (d–f) under reflected light white (a) and under UV light (b–f), field width=0.2 mm

In addition to the determine the organic richness and source rock potential from TOC content, the quantity of the extractable organic matter obtained from source rocks were examined to understand the gross composition. The concentrations of extractable organic matter (EOM) together with the relative proportions of saturated, aromatic fractions, and NSO compounds were calculated (Fig. 4). The saturated and aromatic fractions formed the crude-like (hydrocarbon) fraction; which the sum of these two fractions is known to be HCs. The EOM yields a range from 1638 to 5827 ppm (Table 1), and it is shown that the EOM contains a complex mixture of hydrocarbons and non- hydrocarbon components (NSO) as seen in Fig. 5. The saturated fractions and NSO components

contain the major fractions in the analyzed samples (Table 1).

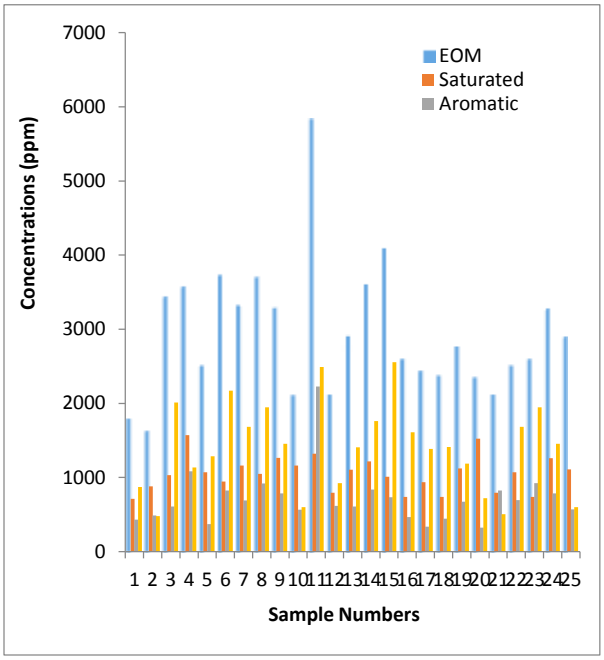

Fig. 4: Histogram of quantity of extractable organic matter (EOM), saturated and aromatic fractions and NSO compounds derived from Bornu sediments

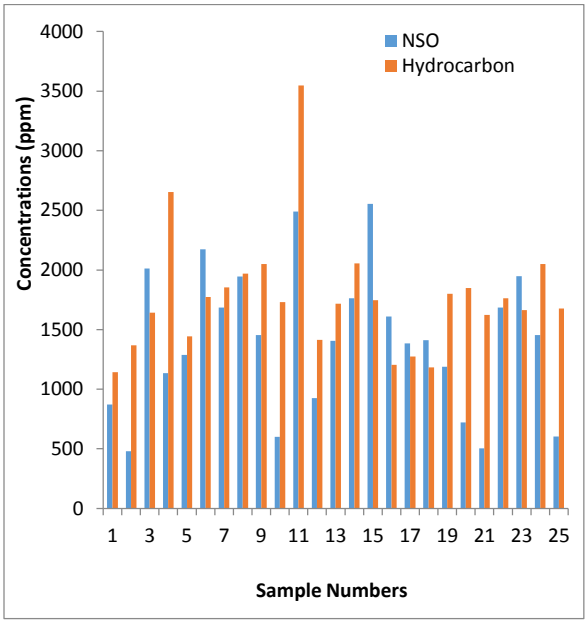

Fig. 5: Histogram of quantity of hydrocarbon compounds (saturated + aromatic) and non-hydrocarbon compounds (NSO) derived from EOM content.

The saturated fractions and NSO compounds are ranging from 22.64 to 64.57% and 20.69 to 73.70%, respectively, whereas aromatic fractions range from 13.77 to 38.88% (Table 1). Since the hydrocarbon portion of the bitumen extracted from sediment is the crude-like portion, it is used as an important parameter in the source-rock evaluation (Philippi, 1957; Baker, 1972). In this respect, most of the Bornu samples are likely the most prolific petroleum sources where abundant naphthenic oils might be expected to be generated (Fig. 6). This is suggested by high hydrocarbon fractions (42.70–81.59 %) in Fig. 5 and relatively high saturated hydrocarbon proportions (22.64 - 64.57%).

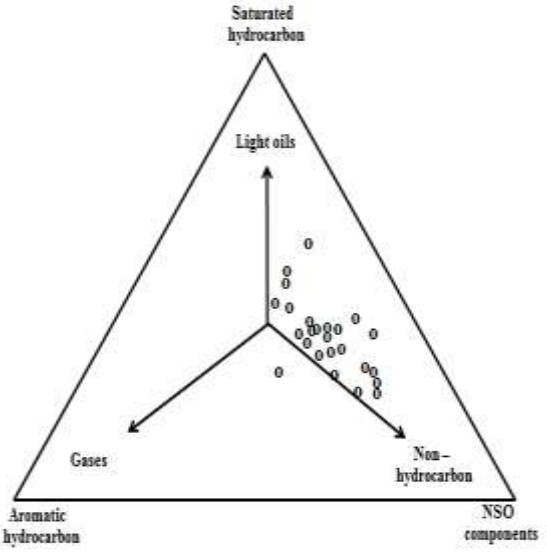

Fig. 6: Triangular diagram of saturated – aromatic–NSO compounds derived from EOM content.

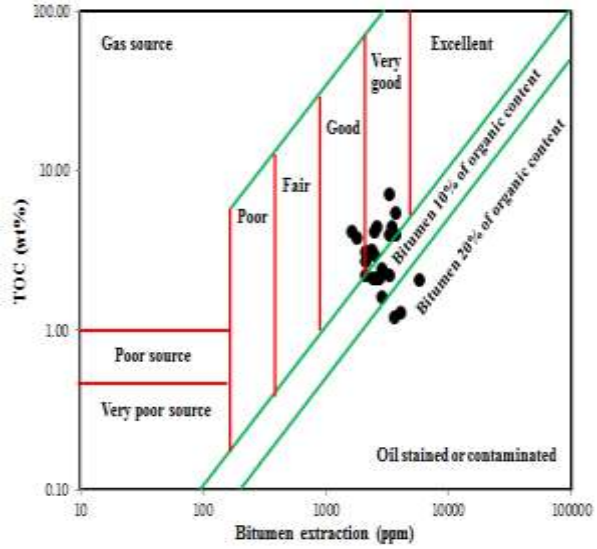

Fig. 7: Plot of TOC content vs. bitumen extractions, showing source potential rating and hydrocarbon source-rock richness for the studied Bornu samples

The hydrocarbon generative potential of a source rock can also be estimated from plot of TOC content versus EOM (Fig. 7) and plot of hydrocarbon yields vs. TOC (Fig. 8). These plots shows that the analyzed Bornu samples have very good source rock potential for oil-generation based on classification by Peters and Cassa (1994) as supported by high TOC content (>1.0 wt%).

Table 1: Bulk geochemical results of extractable organic matter (EOM) yields (ppm), relative proportions of saturated hydrocarbon fractions, aromatic hydrocarbon fractions, and NSO compounds of the EOM (in wt%) and measured Total organic carbon (TOC), sulfur content (TS), and vitrinite reflectance values (%Ro) of the analyzed Bornu samples

Wells	Sample N0.	Depth (m)	TOC (wt%)	TS (wt%)	Ro (%)	Bitumen extraction data: Bitumen extraction and chromatographic fractions (ppm of whole rocks)					Chromatographic fractions of bitumen Extraction (%)				Bitumen/ TOC
						EOM	Sat.	Aro.	NSO	HCs	Sat./ EO M	Aro./EOM	NSO/EOM	HCs	
ZIYE	ZY-02	1020	3.80	0.72	0.59	1805	711	433	871	1143	39.37	23.98	48.27	63.35	0.07
″	ZY-03	1050	4.20	0.73	0.61	1638	882	487	479	1369	53.86	29.74	29.23	83.60	0.07
″	ZY-04	1110	4.50	0.48	0.65	3446	1032	611	2013	1643	29.95	17.73	58.42	47.67	0.09
″	ZY-06	1140	4.00	0.48	0.65	3579	1572	1082	1134	2654	43.94	30.23	31.69	74.17	0.10
″	ZY-07	1170	4.20	0.44	0.62	2522	1070	373	1289	1444	42.43	14.80	51.10	57.23	0.08
″	ZY-09	1200	4.00	0.65	0.62	3734	946	826	2172	1772	25.34	22.12	58.16	47.46	0.11
Mushe	MS-11	1150	7.10	0.98	0.63	3330	1162	693	1685	1855	34.91	20.80	50.61	55.70	0.07
″	MS-13	1180	5.50	0.82	0.66	3706	1048	922	1946	1970	28.27	24.88	52.52	53.15	0.08
″	MS-14	1210	4.00	0.43	0.67	3295	1265	785	1455	2050	38.39	23.82	44.16	62.22	0.10
″	MS-15	1240	3.10	0.80	0.71	2120	1162	568	600	1730	54.82	26.77	28.32	81.59	0.09
″	MS-17	1280	2.07	0.53	0.58	5827	1319	2227	2491	3547	22.64	38.22	42.74	60.86	0.30
″	MS-18	1310	2.72	0.52	0.57	2128	796	617	925	1414	37.42	29.00	43.44	66.42	0.09
″	MS-20	1340	1.60	0.46	0.58	2912	1108	609	1406	1716	38.03	20.90	48.28	58.93	0.21
Tuma	TM-21	1550	1.22	0.64	0.62	3607	1218	837	1762	2055	33.77	23.21	48.85	56.97	0.27
″	TM-22	1665	1.30	0.76	0.63	4092	1012	735	2555	1748	24.73	17.97	62.43	42.70	0.22
″	TM-23	1720	4.50	1.08	0.61	2605	740	465	1611	1205	28.39	17.86	61.81	46.25	0.07
″	TM-25	1900	2.10	0.32	0.63	2449	937	337	1385	1275	38.27	13.77	56.54	52.04	0.13
″	TM-27	2110	3.21	0.66	0.66	2385	738	445	1412	1183	30.96	18.65	59.20	49.61	0.09
″	TM-28	2170	2.10	0.45	0.59	2778	1125	674	1189	1799	40.48	24.28	42.80	64.76	0.16
Keman	KM-30	2210	3.10	0.56	0.61	2360	1524	325	721	1849	64.57	13.77	30.56	78.34	0.10
″	KM-32	2270	2.20	0.44	0.59	2127	795	827	505	1622	37.38	38.88	23.74	76.26	0.12
″	KM-33	2350	3.00	0.46	0.58	2520	1069	694	1686	1763	42.41	27.54	66.89	69.95	0.08
″	KM-34	2360	2.13	0.65	0.63	2606	740	923	1947	1663	28.39	35.41	74.70	63.80	0.09
″	KM-36	2380	2.23	0.47	0.64	3285	1263	786	1455	2049	38.45	23.93	44.30	62.38	0.12
″	KM-37	2520	2.42	0.77	0.67	2910	1108	569	602	1677	38.07	19.55	20.69	57.62	0.10

Table 2: *n*-alkane and isoprenoids biomarker ratios calculated from GC (TIC), m/z 191 and m/z 217, mass fragmentograms of analyzed Bornu extracts.

Wells	Sample N0	Depth (m)	n-alkane and isoprenoids: Pr/Ph	Pr/C_{17}	Ph/C_{18}	CPI	Triterpanes and terpanes (m/z191): C_{32} 22S/ (22S+22R)	C_{29}/C_{30}	C_{31}R/ C_{30}H	MC$_{30}$/ HC$_{30}$	Ts/Tm	C_{29} 20S/ (20S+20R)	C_{29} ββ/ (ββ+αα)	C_{27}/ C_{29}	Regular sterane C_{27}	C_{28}	C_{29}	Diasteranes/ steranes (%)	Steranes /hopanes
ZIYE	ZY-02	1020	1.71	0.31	0.21	1.07	0.55	0.63	0.15	0.21	1.05	0.35	0.35	2.41	58.72	16.82	24.52	0.44	0.26
″	ZY-03	1050	1.56	0.84	0.61	1.14	0.59	0.52	0.17	0.18	1.18	0.41	0.37	2.00	55.42	16.72	27.92	0.83	0.62
″	ZY-04	1110	2.16	0.63	0.34	1.19	0.60	0.56	0.14	0.14	1.74	0.47	0.35	1.87	54.02	16.92	29.12	0.74	0.60
″	ZY-06	1140	1.47	0.86	0.70	1.09	0.57	0.64	0.16	0.18	1.10	0.36	0.32	1.90	53.82	17.52	28.72	0.40	0.37
″	ZY-07	1170	2.53	0.57	0.28	1.10	0.60	0.52	0.14	0.21	1.33	0.42	0.39	1.63	51.82	16.12	32.12	0.88	0.83
″	ZY-09	1200	1.53	0.52	0.37	1.15	0.52	0.60	0.20	0.19	0.81	0.35	0.41	4.45	66.02	19.12	14.92	0.35	0.25
Mushe	MS-11	1150	1.80	0.70	0.41	1.16	0.52	0.65	0.13	0.14	0.84	0.35	0.27	3.78	66.72	15.62	17.72	0.26	0.15
″	MS-13	1180	1.91	0.58	0.34	1.20	0.56	0.66	0.13	0.15	0.86	0.28	0.30	2.54	60.92	15.02	24.22	0.31	0.21
″	MS-14	1210	1.27	0.88	0.68	1.19	0.58	0.59	0.15	0.18	1.28	0.40	0.37	2.69	60.02	17.52	22.52	0.85	0.54
″	MS-15	1240	1.19	0.61	0.47	1.14	0.60	0.63	0.16	0.17	0.99	0.50	0.43	1.96	50.72	23.12	26.12	1.42	0.99
″	MS-17	1280	1.10	0.80	0.69	1.07	0.57	0.58	0.15	0.19	1.07	0.47	0.35	2.39	58.32	17.12	24.62	0.36	0.49
″	MS-18	1310	1.16	0.72	0.62	1.10	0.60	0.58	0.18	0.26	1.11	0.39	0.35	2.24	57.32	17.02	25.72	1.06	0.43
″	MS-20	1340	1.23	0.79	0.65	1.07	0.55	0.55	0.15	0.19	0.99	0.36	0.36	2.02	54.42	18.42	27.22	0.79	0.50
Tuma	TM-21	1550	1.05	0.48	0.45	1.03	0.54	0.74	0.22	0.14	1.52	0.55	0.42	1.64	50.32	18.62	31.12	0.59	1.01
″	TM-22	1665	1.58	0.60	0.37	1.02	0.51	0.50	0.15	0.20	0.73	0.35	0.32	2.15	52.32	23.22	24.52	0.60	0.34
″	TM-23	1720	1.68	0.62	0.44	1.20	0.51	0.51	0.15	0.20	0.74	0.44	0.29	0.96	42.52	12.22	45.32	0.61	0.39
″	TM-25	1900	1.78	0.56	0.35	1.12	0.50	0.33	0.18	0.24	1.64	0.27	0.36	2.15	52.72	22.62	24.72	0.90	0.35
″	TM-27	2110	1.20	0.65	0.52	1.18	0.55	0.50	0.20	0.15	1.44	0.49	0.43	1.25	43.62	20.92	35.52	0.94	0.70
″	TM-28	2170	1.03	0.62	0.52	1.07	0.58	0.64	0.17	0.14	1.43	0.54	0.55	1.73	48.32	23.42	27.02	0.90	0.97
Keman	KM-30	2210	1.60	0.55	0.36	1.07	0.61	0.49	0.18	0.17	1.48	0.54	0.47	1.84	49.22	23.82	28.32	0.87	0.83
″	KM-32	2270	1.54	0.53	0.38	1.16	0.53	0.61	0.21	0.21	0.82	0.36	0.43	4.46	66.03	19.13	14.82	0.36	0.26
″	KM-33	2350	1.82	0.71	0.42	1.17	0.54	0.66	0.14	0.15	0.85	0.36	0.28	3.79	66.73	15.63	17.62	0.27	0.16
″	KM-34	2360	1.92	0.59	0.35	1.21	0.57	0.67	0.14	0.16	0.87	0.29	0.32	2.53	60.93	15.03	24.23	0.32	0.22
″	KM-36	2380	1.37	0.89	0.69	1.21	0.59	0.60	0.16	0.19	1.29	0.42	0.38	2.71	60.03	17.53	22.54	0.86	0.56
″	KM-37	2520	1.21	0.62	0.48	1.15	0.62	0.64	0.17	0.18	1.10	0.53	0.44	1.97	50.73	23.13	26.14	1.43	0.96

n-Alkanes and isoprenoids: Whole extract gas chromatograms of the Bornu organic-rich sediments show that n-alkanes are the dominant components. The n-alkane distributions display a full suite of saturated hydrocarbons between C_{12}–C_{34} n -alkanes and isoprenoids pristane (Pr) and phytane (Ph) (Fig. 9) and shows a predominance of low to medium molecular weight compounds (n-C_{14}–n-C_{23}) with the presence of significant waxy alkanes (+n-C_{23}) thus gave moderate CPI values (Table 2).

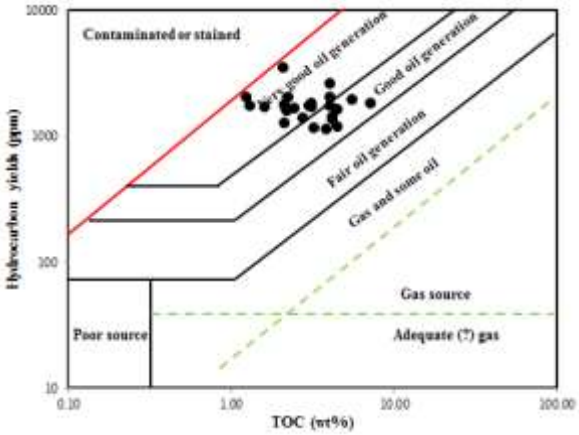

Fig. 8: Plot of hydrocarbon yields vs. TOC, showing source potential rating and hydrocarbon source-rock richness for the studied Bornu samples

These distributions are typical of lacustrine sediments receiving mixed algal with a minor amount of terrigenous organic matter input (e.g., Gülbay *et al*., 2012). Acyclic isoprenoids occur in a significant amount in all studied Bornu samples (Fig. 9), and diagnostic biomarker ratios are listed in Table 2. Pristane (Pr) and phytane (Ph) are usually the most important acyclic isoprenoids hydrocarbons in terms of concentration (Powell and McKirdy, 1973) and frequently occur in sediments and oils (Chandra *et al*., 1994). The pristane to phytane ratios of ancient sediments and oils reflect the palaeoenvironmental conditions of source rocks and are considered as potential indicators of the redox conditions during sedimentation and diagenesis (Didyk *et al*., 1978). Isoprenoids, in particular, pristane, occur in high relative concentrations, possessing pristane/phytane (Pr/Ph) ratios in the range of 1.03–2.53 suggest that the Bornu sediments were deposited under suboxic to relatively anoxic conditions (Peters and Moldowan, 1993; Hakimi *et al*., 2011, 2012b). Furthermore, lower amounts of acyclic isoprenoids compared to n-alkanes (Fig. 9), thus giving distinctively low pristane/n- C_{17} and phytane/n-C_{18} ratios in the range of 0.31–0.89 and 0.21–0.70, respectively, corresponding to mixed organic matter deposited under suboxic to relatively anoxic conditions (Fig. 10).

Biomarkers (terpane and sterane): Triterpane and sterane biomarkers were measured from m/z 191 and m/z 217 mass chromatograms, respectively (Figs. 11 and 12). Peaks identification of all these compounds, including the steranes and diasteranes (in m/z 217), are based on their retention times and comparison of mass spectra with those previously published (Philp, 1985; Hakimi et al., 2012a,b) (Table 3). The m/z 191 mass fragmentograms display high proportions of hopanes and tricyclic terpanes occur in significant abundance (Fig. 11). C_{30} hopane is found in high concentrations and C_{29} norhopane is lower with C_{29}/C_{30} ratios in the range of 0.33–0.74 (Table 2). The predominance of C_{30} hopane is frequently associated with clay-rich source rocks (Gürgey, 1999). This statement is consistent with the lithofacies of the analyzed Bornu extracts. The Ts [C_{27} 18α(H) 22,29,30-trisnorneohopane] generally predominates over Tm [C_{27} 17α(H)-22,29,30-trisnorhopane] (Fig. 11). The Bornu extracts possess a wide range of Ts/Tm ratio values and ranging from 0.73 to 1.74 (Table 2). Values of Tm [C_{27} 17α(H)-22,29,30-trisnorhopane] and Ts [C_{27} 18α(H)-22,29,30-trisnorneohopane] are well known to be influenced by maturation, type of organic matter and lithology (Moldowan *et al*., 1985). Homohopane distribution is represented by the dominance of C_{31} and the concentration decreases toward high numbered homohopanes (Fig.11). Typically, such a homohopane distribution commonly represents clastic facies (Waples and Machihara, 1991) or a clay-rich character (Obermajer *et al*., 1999) as is the case of the Bornu shale and claystone sediments. The biomarker maturity parameters, C_{32} 22S/(22S+20R) and moretane/hopane ratios are also calculated (Table 2). In addition, gammacerane was detected in the Bornu extract samples (Fig. 11).

Steranes and diasteranes were measured from m/z 217 mass chromatograms. The m/z 217 mass fragmentograms of all the analyzed samples display high abundances of C_{27}, C_{28}, and C_{29} steranes and low diasteranes (Fig. 12). A particularly high abundance of C_{27} sterane (relative to C_{28}–C_{29} steranes) is present in the analyzed Bornu samples (Fig. 12).Relative abundances of C_{27}, C_{28}, and C_{29} regular steranes are calculated, and the results are given in Table 2. The diasterane/sterane ratios of the analyzed samples were calculated as shown in Table 2. The high diasteranes to steranes ratio (0.26–1.43) correspond to the higher clay contents in the former (Gürgey, 1999) as suggested by predominance of C_{30} hopane in the m/z 191 mass fragmentograms (Fig. 11). The C_{27}/C_{29} sterane and sterane/hopane ratios and two different sterane thermal maturity parameters, C_{29} 20S/(20S+20R) and the C_{29} αββ (αββ+ααα), are also calculated and listed in Table 2.

Table 3: Peaks for alkane hydrocarbons in the gas chromatograms of saturated fractions in the m/z 191 and 217 mass fragmentograms compound abbreviation.

Peak number (m/z 191)		
Ts	18α(H),22,29,30-trisnorneohopane	Ts
Tm	17α(H),22,29,30-trisnorhopane	Tm
29	17α 21β(H)-nor-hopane	C_{29} hop
30	17α,21β(H)-hopane	Hopane
30 m	17 β,21α (H)-Moretane	C_{30}Mor
29 m	17β (H),2lα (H)-30-norhopane (normoretane)	Normoretane
31 S	17α,21β(H)-homohopane (22S)	C_{31}(22S)
31 R	17α,21β(H)-homohopane (22R)	C_{31}(22R)
32 S	17α,21β(H)-homohopane (22S)	C_{32}(22S)
32 R	17α,21β(H)-homohopane (22R)	C_{32}(22R)
33 S	17α,21β(H)-homohopane (22S)	C_{33}(22S)
33 R	17α,21β(H)-homohopane (22R)	C_{33}(22R)
34 S	17α,21β(H)-homohopane (22S)	C_{34}(22S)
34 R	17α,21β(H)-homohopane (22R)	C_{34}(22R)
35 S	17α,21β(H)-homohopane (22S)	C_{35}(22S)
35 R	17α,21β(H)-homohopane (22R)	C_{35}(22S
Peak number (m/z 217)		
a	13β,17α(H)-diasteranes 20S	Diasteranes
b	13β,17α(H)-diasteranes 20R	Diasteranes
c	13α,17β(H)-diasteranes 20S	Diasteranes
d	13α,17β(H)-diasteranes 20R	Diasteranes
e	5α,14α(H), 17α(H)-steranes 20S	ααα20S
f	5α,14β(H), 17β(H)-steranes 20R	αββ20R
g	5α,14β(H), 17β(H)-steranes 20S	αββ20S
h	5α,14α(H), 17α(H)-steranes 20R	ααα20R

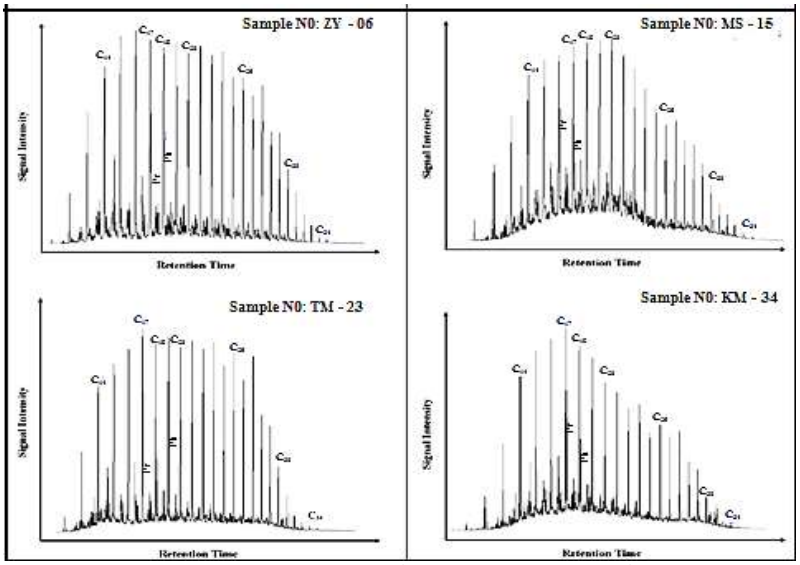

Fig. 9: Gas chromatograms–mass spectrometry (TIC) of saturated hydrocarbons of the analyzed Bornu extracts

Maturity Indicators: Several parameters have been suggested and used to evaluate the level of organic maturity such as mean vitrinite reflectance data (%Ro), pyrolysis Tmax, thermal alteration of spore-pollen (TAI), and biomarker thermal indicators. In this study, the thermal maturity was evaluated based primarily on biomarker distributions and supported with the mean vitrinite reflectance (%Ro) as VR is considered more satisfactory and widely accepted by many authors and exploration geologists as a technique for measuring the thermal maturity of source rocks (e.g., Douglas and Williams, 1981; Peters and Moldowan, 1993). In this study, a variety of biomarker maturity indicators have been used to evaluate the level of thermal maturity of the Bornu organic-rich sediments; these include pentacyclic triterpanes and regular sterane isomer ratios. The ratios of C_{32} homohopane 22S/(22S+22R), moretane/hopane, and C_{29} sterane 20S/(20S+20R) and ββ/(ββ+αα) can be used to evaluate the thermal maturity of the analyzed samples (Peters and Moldowan, 1993; Peters et al., 2005). A widely used biomarker maturity parameter is the [22S/(22S+22R)] homohopane ratio (Ensminger, 1977). The ratios of C_{32} 22S/ (22R+22S) are increase from 0 to about 0.6 at equilibrium (Seifert and Moldowan, 1986) during

maturation. Values in the range of 0.50–0.54 have barely entered oil generation, whereas ratios from 0.57 up to 0.62 indicate that the oil window has been reached. Most of Bornu extracted samples have C_{32} 22S/22S+22R values in the range of 0.50–0.62, suggesting that they have reached equilibrium and that the oil window has been reached. The 20S/(20S+20R) and ββ/(ββ+αα) C_{29} sterane ratios of the Bornu extracts are ranging from 0.27 to 0.55 (Table 2). These biomarker maturation ratios are indicating that the analyzed Bornu samples have entered early mature for oil generation window (Fig. 13). This is supported by moretane/hopane ratios consistent with low relative abundance of C_{30} moretane. Moretane converts to C_{30} hopane with increasing thermal maturity (Seifert and Moldowan, 1986), and thus, moretane decreases as thermal maturity increases. The ratio of moretane to their corresponding hopanes decreases with increasing thermal maturity, from about 0.8 in immature sediments to about 0.15–0.05 in mature source rocks and oils (Mackenzie *et al*., 1980; Seifert and Moldowan, 1986). The Bornu samples have moretane/hopane ratio in the range of 0.14–0.26, suggesting that samples are early mature. Overall, the biomarker thermal maturity parameters are indicating that all analyzed Bornu samples are at least early mature, and are likely to be approaching oil window maturity (Fig. 13) and therefore support the mean vitrinite reflectance (%Ro) of 62%Ro that from 0.57 to 0.71 %Ro (Table 1). In addition, the maturity of an organic matter can be expressed by its bitumen/TOC ratios, which is defined as the ratio of the amount of free hydrocarbons generated to the total amount of the organic matter due to maturity. The bitumen/TOC ratios are between 0.07 and 0.22, which correspond to early-peak oil window maturity as previously reported by Peters and Cassa (1994).

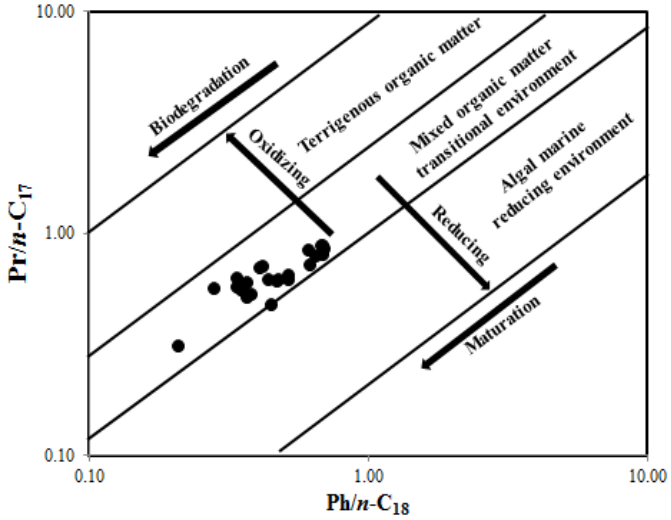

Fig. 10: Plot of Ph/n-C_{18} vs. Pr/ n-C_{17}, indicating depositional conditions of Bornu extracts (modified after Shanmugam, 1985).

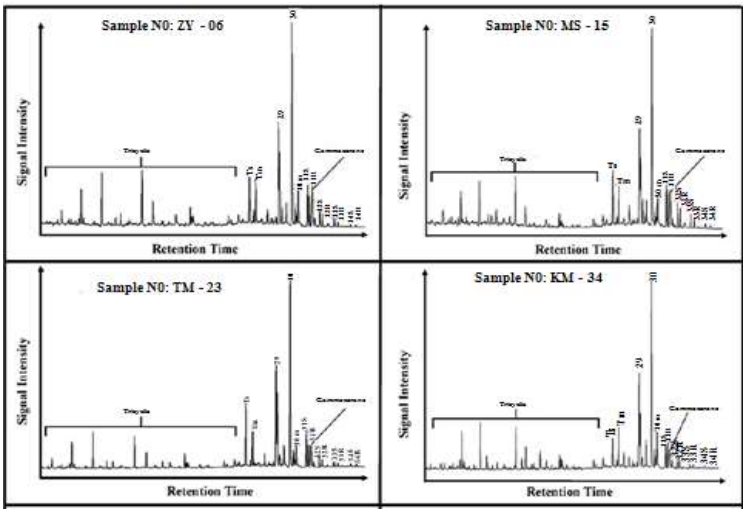

Fig. 11: The m/z 191 mass fragmentograms of saturated hydrocarbon fractions of the analyzed Bornu extracts.

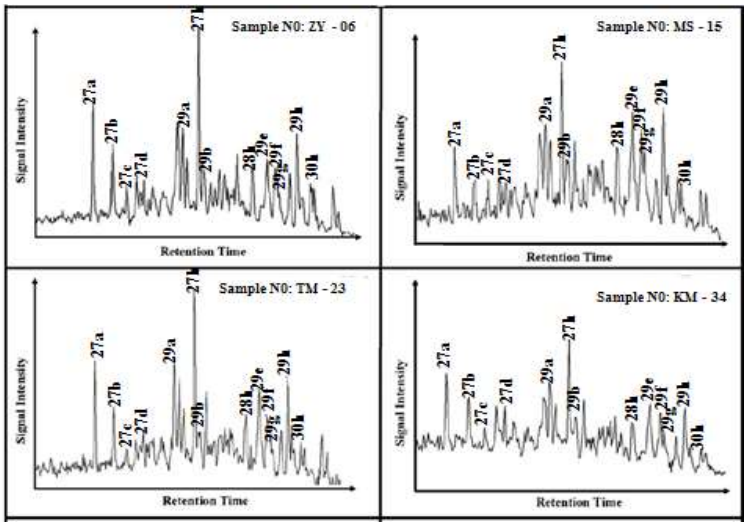

Fig. 12: The m/z 217 mass fragmentograms of saturated hydrocarbon fractions of the analyzed Bornu extracts.

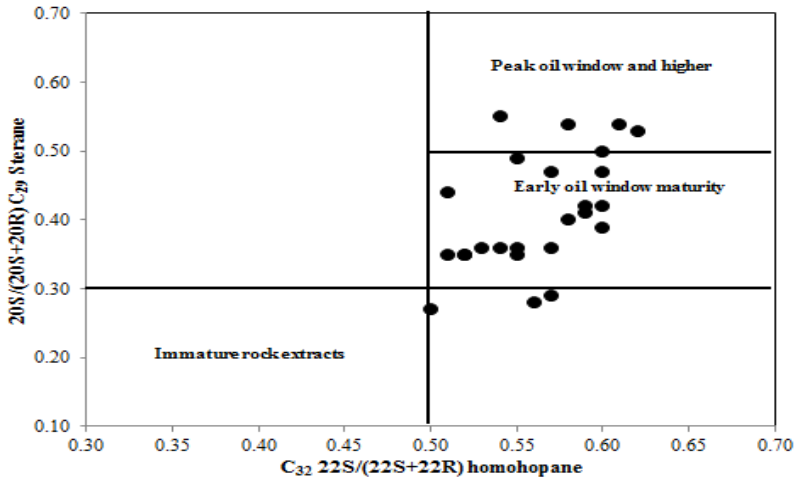

Fig. 13: A cross-plot of two biomarker parameters sensitive to thermal maturity of the Bornu extracts, which shows that most of the Bornu samples plot in the area of early oil window maturity (modified from Peters and Moldowan, 1993)

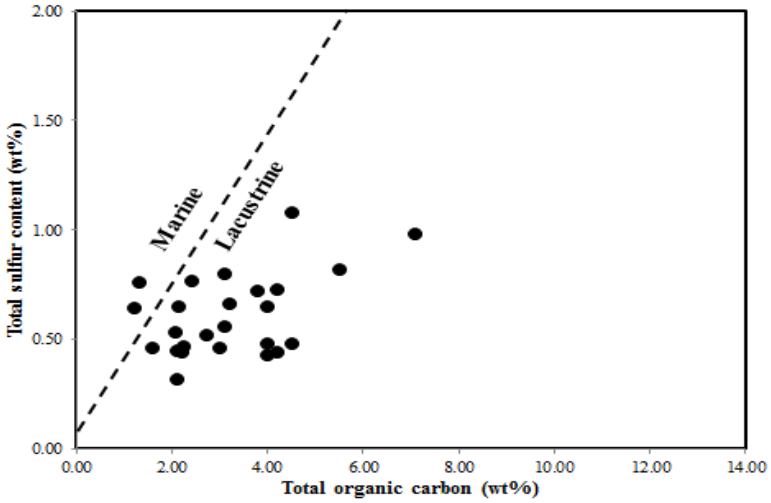

Fig. 14: Sulphur content plotted against TOC content, suggesting that the depositional environment of the analyzed Bornu sediments was dominantly freshwater lacustrine environment (modified after Berner and Raiswell, 1983)

Palaeoenvironmental conditions: The Lower Cretaceous Bornu sediments have been interpreted to have been deposited in a principally lacustrine environment under relatively low oxygen-deficient conditions (suboxic -anoxic). The results indicate that the Bornu sediments are characterized by relatively low TS contents (Table 1) and relatively high amounts of organic carbon with alginite and amorphous organic matter (Fig. 3). TS contents are generally within the range observed in a freshwater lacustrine (Fig. 14; after Berner and Raiswell, 1983), and the presence of alginite with morphology similar to the recent alga Botryococcus in several of the samples further implies a lacustrine origin. This interpretation is also consistent with biomarker distributions (Table 2). The n-alkane distributions are consistent with a typical of lacustrine setting receiving mixed algal and amorphous organic matter as indicated by kerogen microscopy (Fig. 3) and lower amounts of acyclic isoprenoids compared to n-alkanes (Fig. 11).

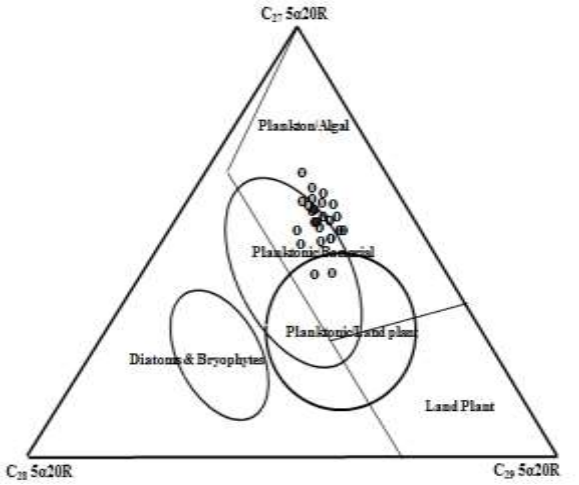

Fig. 15: Relationship between regular sterane compositions, organic matter input, and depositional environment for the analyzed Bornu extracts (modified after Huang and Meinschein, 1979).

This is suggested by a strong predominance of C_{27} regular steranes, consisting predominantly of plankton/algal with bacterial organic matter (Fig. 15; after Huang and Meinschein, 1979). These conclusions are also supported by high values of C_{27}/C_{29} regular sterane ratios (Fig. 16) and the presence of relatively high concentrations of tricyclic terpane in the m/z 191 mass fragmentograms (Fig. 11). The high tricyclic terpane concentrations detected in the Bornu sediments (Fig. 11) is consistent with being derived from the lacustrine algal organic matter (Simoneit *et al*., 1986; Aquino neto et al., 1989; Kruge et al., 1990; Waples and Machihara, 1991). The C_{31}-22R– hopane/C_{30}−-hopane ratio is also used to distinguish between marine and lacustrine environments.

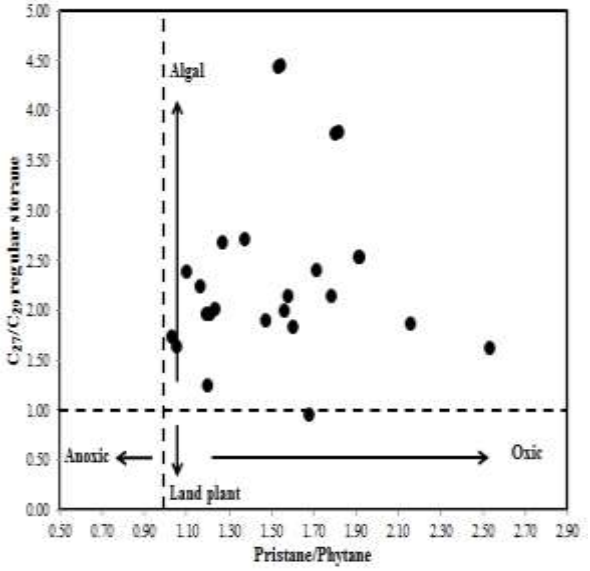

Fig. 16: A plot of pristane/phytane versus C_{27}/C_{29} regular steranes, indicating organic matter input and depositional conditions of the Bornu extracts.

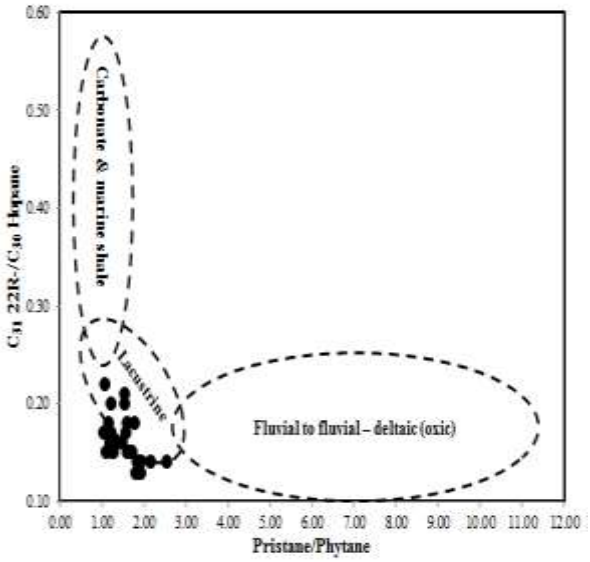

Fig. 17: Hopane and isoprenoid ratios of extracts which allow discrimination of the depositional environments of Bornu sediments in the Chad basin (modified after Peters *et al*., 2005).

This ratio is generally higher than 0.25 for marine environments, whereas it is lower than 0.25 for lacustrine settings (Peters et al., 2005). $C_{31}R/C_{30}$ hopane ratios of Bornu extract samples are in the range of 0.13 – 0.22, indicating a lacustrine environment (Peters *et al*., 2005). Furthermore, an aliphatic isoprenoids pristane and phytane (Pr/Ph) in combination with low hopane-biomarker ratios (C_{31}-22R-hopane/C_{30}-hopane) (Fig. 17) and low

sterane/hopane ratios (C_{29}-sterane/C_{30}-hopane) strongly argue for a lacustrine depositional environment (Peters *et al*., 2005) of the organic matter. In addition, the fine grain size of the inorganic matter (clay) and low-oxygen conditions (suboxic– anoxic) implies a low-energy depositional environment and favored the preservation of organic matter, as indicated by elevated TOC contents and the high proportion of fluorescing AOM.

With regard to thermal maturity, the Bornu organic-rich sediments sequence under current investigation is thermally early mature oil-window generation as indicated by 22S/22S+22R C_{32} homohopane and 20S/(20S +20R) and ββ/(ββ+αα) C_{29} sterane that have values in the range of 0.50–0.62 and 0.27 to 0.55, respectively. This is conclusion consistent with vitrinite reflectance values ranging from 0.57 to 0.71 % Ro, supporting that the oil window has been reached.

Conclusions: In conclusion, the geochemical analyses indicate that the Bornu sediments in the Nigerian sector (sub-basin) are organic-rich source rock with a very good oil-generative potential. This is supported by high total organic carbon and bitumen and hydrocarbon yields. The organic petrological analysis and biomarker distributions have been performed on the Bornu sediments in order to elucidate its depositional environment conditions and thermal maturity. However, the biomarker characteristics suggest that the source rock organic matter has high abundance of lacustrine-derived aquatic organic matter (algal and bacterial) that preserved under suboxic to relatively anoxic conditions. This is further supported by an *n*-alkane distribution, acyclic isoprenoid ratios, and terpane and sterane biomarkers. This biomarker characterization is in agreement with the microscopic investigation as evidenced by the significant occurrence of alginite and liptinitic amorphous kerogen. Periods of desiccation envisaged during deposition of the Bornu sediments is, according to the presence of gammacerane, also present.

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