



**GEOCHEMICAL INVESTIGATION OF ORGANIC MATTER FROM SOURCE ROCK OF CRETACEOUS DUKULSEDIMENTS OF THE UPPER BENUE TROUGH, NORTHEAST NIGERIA.**

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**Abstract**

Shale from Cretaceous strata of the Dukul Formation has been characterized by Rock-Eval pyrolysis and geochemical techniques. The objectives of this study is to investigate the quality of the organic matter, evaluate its thermal evolution, highlights its potential as a source rock and paleoenvironment of the organic matter. The total organic carbon (TOC) (0.58wt%) of the shale constitutes that of a poor to fairly source rock with gas-prone kerogen indicated by Rock-Eval  $S_2/S_3$  (0.60). The low oxygen index (OI) (0.98 mgCO<sub>2</sub>/g TOC) and the presence of biomicritic limestone suggest deposition under low energy environments. The plots of HI against Tmax and predominance of vitrinite rich macerals classified the organic matter as Type III kerogen. The poor concentration of OM is thought to account for its current hydrogen index (35.0 mgHC/gTOC). The predominance of Type III kerogen in the Dukul Formation suggests their potential to generate gas in the deeply buried sections. The Tmax values from the pyrolysis of the shales of the Dukul Formation ranges from 431 to 442°C whereas Ro of the shales ranges from 0.57 to 0.75%. These values correspond to maturity levels within the oil formation. Pr/Ph ratios range from 0.80 to 2.98 that indicate anoxic to oxic depositional environments. Steranes are dominantly of the C<sub>27</sub> forms. The predominance of type-III organic matter in this formation with dominantly marine depositional environments of C<sub>27</sub> steranes may be attributed to high oxic levels which have downgraded organic matter preservation in the marine system. The relatively high Ts/Tm and low moretane/hopane ratios validate maturity levels that have entered the main phase of oil generation.

**Keywords:** Benue Trough, Source rock, Kerogen type, Sterane, Maturity, Cretaceous.

**1.0 Introduction**

The Benue rift basin is a sediment-filled northeast trending structure in Nigeria (Cratchley and Jones, 1965; Burke et al., 1970). It is divided geographically into the

lower, middle and upper Benue regions (Fig. 1) and has been a subject of several publications and discussions (King, 1950; Grant, 1971; Burke and Whiteman, 1973; Olade, 1975; Odebode, 1988). Although the

associated basins are thought to have formed from extensional processes, recent studies by Benkhelil (1982; 1987; 1989) suggest the importance of sinistral wrenching as a dominant process for the structural readjustment and geometry of the different subbasins. Two subbasins, the NNE/SSW trending Gongola and the E/W trending Yola Basins, are delineated in the Upper Benue Trough.

The petroleum geology of the upper Benue rift basins (Gongola and Yola basins) has been of great interest to geologists working in the Benue Trough, in the past few years (Idowu and Ekweozor, 1993; Obaje et al., 1999). This study is part of on-going project

to understand the depositional environments and petroleum potentials of the region (Akande et al., 1998a, 1998b; Ojo and Akande, 1998; Ojo, 1999). In the present work, source rock samples from the borehole and outcrop sections of the Dukul Formation in the Yola Basin (Fig. 1) were investigated. The paleoenvironment of the Turonian Dukul Formation based on the Rock-Eval pyrolysis and biomarker analyses of outcrop sections, are investigated. The source rock potential and thermal maturity are evaluated on the basis of total organic carbon, Rock-Eval pyrolysis, biomarker and vitrinite reflectance measurements on 11 samples from shallow water borehole and outcrop sections.

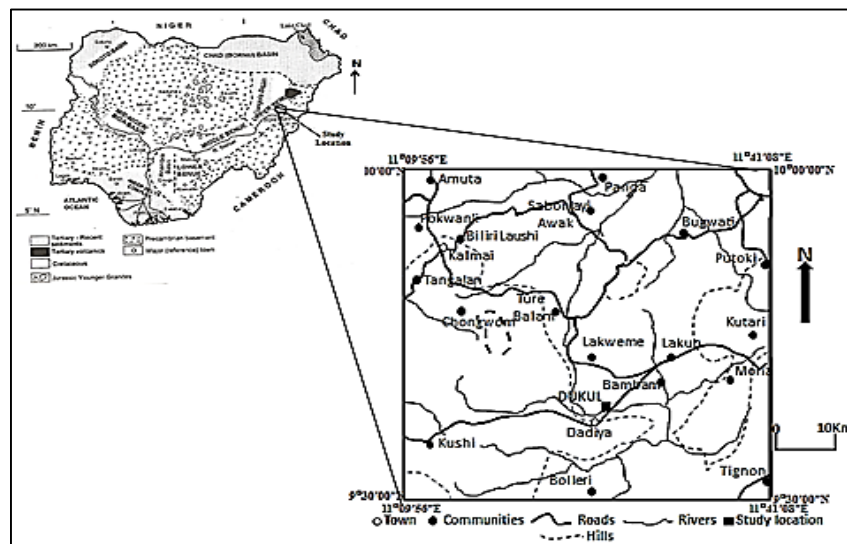


Fig. 1: Map showing the study location in the Upper Benue Trough (inset: Geological map of Nigeria showing the location of the study area; modified from Obaje et al., 2004).

## 2.0 Regional stratigraphic setting

Cretaceous successions in the Upper Benue Trough are flanked by the Precambrian-Late Paleozoic basement gneisses and granite which occur as inlier on occasion (e.g the

Kaltungo inlier). The Precambrian basement rocks are overlain by the Albian Bima Sandstone as the oldest Cretaceous sediment in the region. This is overlain by the transitional Yolde Formation (Cenomanian-

Turonian), and succeeded by the marine Turonian to Coniacian Pindiga Formation, Gongila Formation in the Gongola Basin and its lateral equivalents; the Dukul, Jessu and Numanha formations in the Yola Basin (Fig. 2). These successions are overlain by the

Campanian-Maastrichtian Gombe Sandstone in the Gongola Basin and the lateral Lamja Sandstone (lateral equivalents) in the Yola Basin. The Tertiary Kerri-Kerri Formation capped the succession west of Gombe in the Gongola Basin.

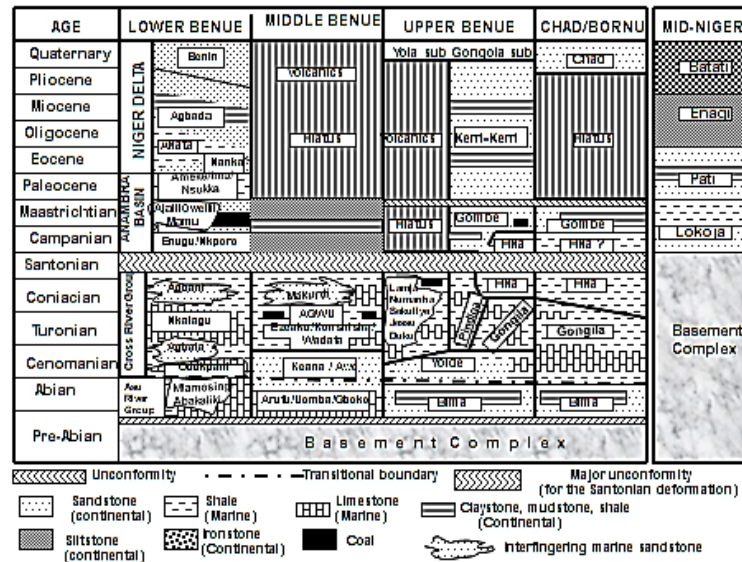


Fig. 2: Stratigraphic succession in the Benue trough, the Nigerian sector of the Chad Basin, the Mid-Niger Basin and relationship to the Niger delta (after Obaje et al., 2006).

The Dukul Formation was defined by Carter et al. (1963) as comprising a sequence of shale and thin limestone intercalations with a type locality at Dukul in the north-eastern part of Dadiya syncline. In this study, the formation was found to be composed of grey shales with thin limestone and siltstone beds. The thin limestone beds are evenly distributed in the studied section at Lakun which has a thickness of 30 m (Uzoegbu et al., 2013). The thin siltstone beds occur in the middle and towards the top of the Kutari and Lakun sections respectively. The entire sections at these two localities form part of the Dukul Formation. The overlying Jessu Formation is a marginal marine unit. The upper boundary of the Dukul Formation in

the Lakun and Kutari sections was not encountered. At Dukul the formation measures about 60 to 91 m (Carter et al. (1963) and 80 m (Zaborski, 1990). A good section of the unit is also exposed at Jessu. In all these sections, the lithofacies of the unit is composed of shales with thin interbedded limestone, which may measures a few centimeters to a maximum of 1m, and siltstones. The section of the unit described by Ojo and Akande (2000) from Dukul contains thicker beds of limestone when compared with the other sections from the area. They reported a basal limestone which measures about 2.2 m and an upper limestone bed intercalated between shales. The second limestone bed measures about 2.1 m. The

limestones are grain supported and rich in bivalves and gastropods. The section at Jessu consists mainly of shale, siltstone and limestone intercalations. The limestone have average thickness of about 0.5 m, they are grey and grain to mud supported. The shales have average thickness of 0.45 m (Ojo and Akande, 2000). The siltstone beds occur near the top and at the base of the section. The limestones are rich in macrofossils as demonstrated by the frequency occurrence of bivalve shells and shell fragments.

### **3.0 Materials and methods**

The Dukul Formation is situated on latitudes 9°30'00" N to 10°00'00"N and longitudes 11°09'56"E to 11°41'08"E. (Fig. 1). Eleven fresh outcrop sections of the Dukul Formation of limestones and shales located at Lakun, Kutari and Dukul. Three shale samples (ditch cutting) from a shallow borehole (GSN BH 1612) located at Numan and penetrating Dukul and Yolde Formations (Dukul samples) were selected and subjected to organic geochemical techniques. Care was taken to avoid weathered portions of the outcrop and to obtain material sufficient for various geochemical analyses. The samples were hard, thickly laminated but not fissile, with texture indicative of low permeability. This macro-structure suggests minimum risk of organic matter oxidation.

In the laboratory, the samples were reshaped using a rotating steel cutter to eliminate surface that could be affected by alteration. Chips were cut from the samples and dried in an oven at 105°C for 24 hours. A portion of the dried sample was pulverized in a rotating disc mill to yield about 50 g of sample for

analytical geochemistry. The total organic carbon (TOC) and inorganic carbon (TIC) contents were determined using Leco CS 200 carbon analyzer by combustion of 100 mg of sample up to 1600°C, with a thermal gradient of 160°C min<sup>-1</sup>; the resulting CO<sub>2</sub> was quantified by an Infrared detector. The sample with known TOC was analyzed using a Rock-Eval 6, yielding parameters commonly used in source rock characterization, flame ionization detection (FID) for hydrocarbons thermal conductivity detection (TCD) for CO<sub>2</sub>.

Nine shale samples were subjected to liquid chromatography (LC) and gas chromatography – mass spectrometry (GC-MS). Asphaltenes were precipitated from oils using excess n-hexane followed by filtration. Using column chromatography, the deasphalted oil (maltene) was fractionated to saturate, aromatic and polar (NSO) by eluting with n-hexane, benzene and methanol respectively. Then complementary steps such as gas chromatography (GC), gas chromatography-mass Spectrometry (GC-MS) and stable isotope analysis were performed.

The saturated as well as aromatic hydrocarbon fractions were analyzed on a gas chromatograph equipped with a 30-m DB-1 fused silica capillary column (i.d. 0.25- mm; 0.25- μm film thickness) and coupled to a Finnigan MAT GCQ ion trap mass spectrometer.

The oven temperature was programmed from 70 to 300 °C at a rate of 4 °C/min followed by an isothermal period of 15 min. Helium was used as the carrier gas. The sample was injected split-less with the injector

temperature at 275 °C. Data processed with a Finnigan data system. Relative percentages and absolute concentrations of different compound families in the saturated and aromatic hydrocarbon fractions were calculated using peak areas from the gas chromatograms in relation to those of internal standards (deuterated n-tetracosane and 1,1-binaphthyl, respectively).

#### 4.0 Results and discussion

##### 4.1 Organic matter concentration

The amount of organic carbon (TOC) is a measure of the quantity of organic matter (OM) in the source rocks (Tissot and Welte,

1984). As shown in Tables 1 and 2, the Total Organic Carbon (TOC%) for the Turonian shales in the Dukul Formation vary from 0.25 to 1.15%. The average TOC value 0.58wt% for the Dukul shales indicates a moderate organic matter concentration (Hunt, 1979; Tissot and Welte, 1984). In the Numan borehole, within depth interval of 70 to 180m, a vertical down-hole variation in TOC values of the shaly horizons which reflects changing paleoenvironments is observed (Ojo and Akande, 2002; Uzoegbu et al., 2013). An exceptionally high value of TOC (12.9%) was recorded at depth of 93m in this borehole.

Table 1: Rock-Eval pyrolysis data of samples from Dukul Formation.

Sample N0.	Loation	Formation	Lithology	TOC (wt.%)	S <sub>1</sub> mgHC/g	S <sub>2</sub> mgHC/g	S <sub>3</sub> mgCO <sub>2</sub> /g	S <sub>1</sub> +S <sub>2</sub>	S <sub>2</sub> /S <sub>3</sub>	Tmax (°C)	HI (mgHC/gTOC)	OI (mgCO <sub>2</sub> /gTOC)
DUK-1	Lakun	Dukul	Shale	0.64	0.02	0.10	0.36	0.12	0.28	438.00	20.00	0.43
DUK-2	"	"	"	1.15	0.03	0.96	0.60	0.99	1.60	434.00	64.00	1.39
DUK-3	"	"	"	0.48	0.01	0.08	0.39	0.09	0.21	439.00	27.00	0.43
DUK-4	"	"	"	0.77	0.01	0.14	0.20	0.15	0.70	431.00	55.00	0.91
DUK-5	Kutari	"	"	0.42	0.02	0.12	0.17	0.14	0.71	440.00	17.00	1.68
DUK-6	"	"	"	0.58	0.02	0.16	0.83	0.18	0.19	435.00	23.00	0.33
DUK-7	"	"	"	0.55	0.03	0.08	0.18	0.11	0.44	438.00	51.00	0.81
DUK-8	"	"	"	0.71	0.01	0.15	0.26	0.16	0.58	435.00	39.00	0.81
DUK-9	Dukul	"	"	0.25	0.15	0.06	0.31	0.06	0.19	430.00	24.00	0.77
DUK-10	"	"	"	0.53	0.33	0.22	0.19	0.55	1.16	431.00	41.00	2.18
DUK-11	"	"	"	0.33	0.21	0.08	0.23	0.08	0.35	442.00	24.00	1.05

Table 2: Calculated data using Rock-Eval pyrolysis parameters of samples from Dukul Formation.

Sample N0.	Loation	Formation	Lithology	S <sub>1</sub> /TOC	HI/OI	TS	TOC/TS	PI= S <sub>1</sub> /(S <sub>1</sub> +S <sub>2</sub> )	Ro (%)	HGP
DUK-1	Lakun	Dukul	Shale	0.01	3.21	1.04	1.26	1.10	0.63	2.05
DUK-2	"	"	"	0.01	35.00	1.59	1.25	1.51	0.58	4.49
DUK-3	"	"	"	0.00	18.21	2.19	0.95	1.08	0.62	1.34
DUK-4	"	"	"	0.00	5.93	1.35	2.18	1.14	0.57	2.70
DUK-5	Kutari	"	"	0.03	1.33	0.90	1.68	1.12	0.62	1.60
DUK-6	"	"	"	0.02	4.43	1.10	2.68	1.16	0.71	2.15
DUK-7	"	"	"	0.03	1.56	0.88	1.52	1.08	0.62	1.71
DUK-8	"	"	"	0.03	3.00	1.23	1.12	1.15	0.69	2.63
DUK-9	Dukul	"	"	0.03	4.14	1.30	1.18	1.05	0.73	0.95
DUK-10	"	"	"	0.03	4.29	1.93	1.24	1.22	0.65	2.07
DUK-11	"	"	"	0.03	4.18	1.67	1.21	1.17	0.75	1.16

nd = Not determine

This interval, interpreted as swamp facies (Akande, 1998a; Ojo, 1999) is probably rich in organic matter due to its proximity to organic sources (Bustin, 1988; Bustin and Chonchawalit, 1997). The source rocks of the Dukul sediments in the section have lower organic matter concentration (0.25 to 1.15%) averaging 0.58% in all the samples. The TOC values of the source rock facies of the Dukul Formation is in the average of 0.58%. Higher TOC values in the Dukul Formation were obtained in the outcrop sections at Dukul, Lakun and Kutari areas where the average TOC is 0.76%. The lateral variation in organic content probably reflects localized changes in organic productivity and preservation. The HI values for the Dukul Formation range from 17 to 64mgHC/g TOC.

#### 4.2 Types and Quality of Organic Matter

The quality of organic matter (OM) in the source rock facies of the Dukul Formation was done by using Rock-Eval generated data (HI and Tmax).

The shale samples of the Dukul Formation plot mainly along the gas prone kerogen evolutionary pathway as indicated by the plot of HI against Tmax (Fig. 3). This confirms that a substantial proportion of the organic matter is of terrestrial origin with gas potential despite their marine environment of deposition. This view is further supported by the ratios of A-factor and C-factor (1.3), which shows predominance of gaseous prone type III organic matter in the shales of the Dukul Formation (Uzoegbu and Ugwueze, 2021). They contain relatively high carbonyl/carboxyl groups and moderate aliphatic groups (Ojo and Akande, 2002).

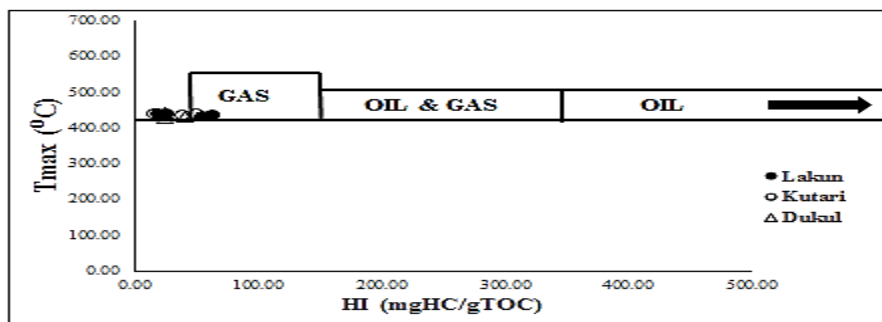


Fig. 3: Classification of kerogens of the Dukul Formation on the HI – Tmax.

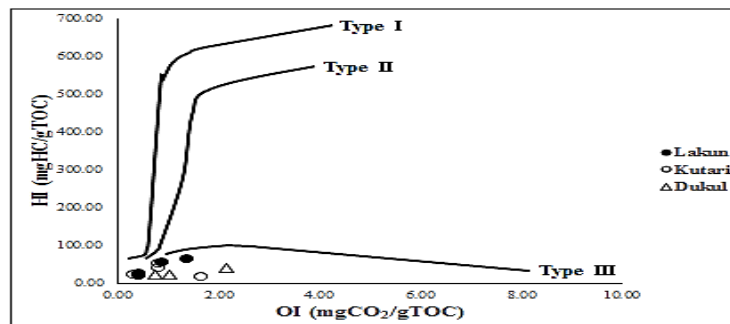


Fig. 4: Composite HI – OI classification of kerogen types of source rocks in the Dukul Formation.

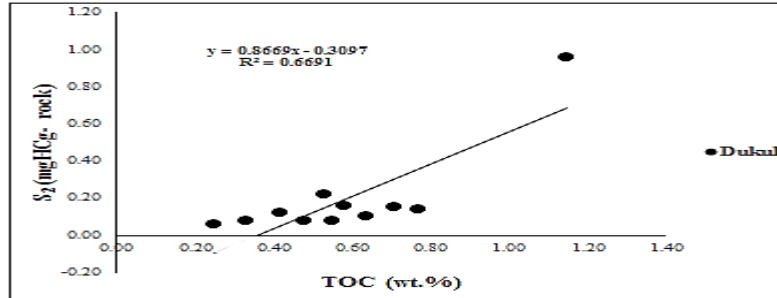


Fig. 5: Classification of kerogens of the Dukul Formation on the S<sub>2</sub> – TOC.

This is further supported based on the Mukhopadhyay and Hatcher (1993) classification of kerogens relative to HI and OI, all of the samples plots within Type III field, where desmocollinite (collodetrinite) is dominance macerals as a good potential for the generation of gas (Fig. 4). The gas-prone nature of this rock rules out Type II kerogen, which usually shows S<sub>2</sub>/S<sub>3</sub> greater than 5, while the maturity from vitrinite reflectance as well as Tmax suggest that the current HI results from thermal evolution of a Type III kerogen, with initial HI between 600 mg HC/g-1TOC and 850 mg HC/g-1TOC (Lafargue et al., 1998).

Low TOC contents (as low as 0.58 wt %) and HI between 17.00 and 64.00 mg HC/g TOC characterize the shale beds of the Dukul Formation. The regression equation based on the S<sub>2</sub> vs. TOC diagram gave an average HI value of 87.00 mg HC/g TOC for the Dukul shales (Fig. 5). A plot of S<sub>2</sub> vs. TOC and determining the regression equation has been used by Langford and Blanc-Valleron (1990) as the best method for determining the true average HI and measuring the adsorption of hydrocarbons by rock matrix.

Peters (1986) has suggested that at a thermal maturity equivalent to vitrinite reflectance of 0.6% (Tmax 435°C), rocks with HI above 300 mg HC/g TOC produce oil, those with HI

between 300 and 150 produce oil and gas, those with HI less than 50 are inert. The TOC is a primary parameter in source rock appraisal, with a threshold of 0.50-1.00 wt% at the immature stage for potential source rocks (Tissot and Welte, 1984; Bordenave et al., 1993; Hunt, 1996). The value of 0.58 wt% of the shale studied falls within this threshold. High TOC of 4.45 wt% was obtained in Mamfe basin and this value exceeds the threshold for oil generation (Eseme et al., 2006). However, high TOC is not a sufficient condition for oil generation. Coals usually have high TOCs that exceed 50 wt% but do not generate oil except when rich in liptinite, indicating the relevance of maceral composition. In contrast, deltaic sediments may have TOCs below 1 wt% but generate commercial accumulations of petroleum due to deposition of large volumes of sediments, as seen in the Dukul. High TOC content in shales indicates favorable conditions for preservation of organic matter produced during deposition. This may related to the redox condition, with high oxygen favoring organic matter oxidation, but also amount of organic matter produced.

The van Krevelen diagram is generated by plotting the Hydrogen Index (HI) against maximum temperature (Tmax) as shown in Fig. 6. The corresponding HI-Tmax diagram

based on the values given by Peters (1986) indicates some potential between oil and gas with gas dominating (Fig. 3 and 5). Plots of HI versus Tmax (the maximum temperature of pyrolysis) (Fig. 6) and Tmax versus HI (Fig. 3), also shows that the organic matter in the samples is mainly type III with oil, oil/gas and gas prone. The above results are in

agreement with the data obtained by earlier workers (Akaegbobi and Schmitt, 1998; Akande et al., 2007; Ogala, 2011; Shanmugam, 1985; Uzoegbu and Ikwuagwu, 2016b)). Majority of the samples falls into fields that have hydrocarbon generative potential (Figs. 3 and 5).

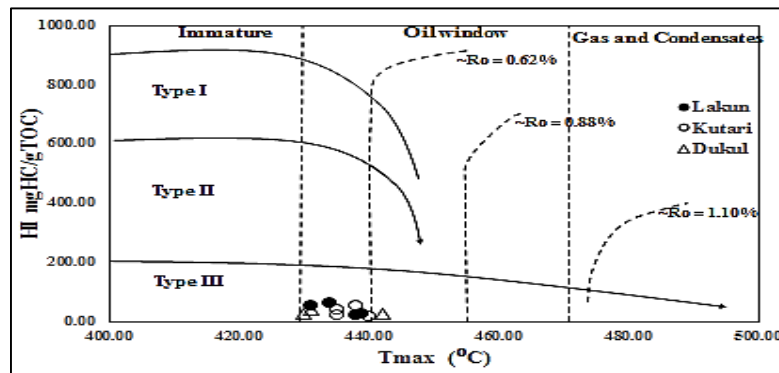


Fig. 6: A plot of HI versus Tmax indicating type of organic matter and maturity level.

A Plot of the SOM (extract yield) against TOC as proposed by Landis and Connan (1980) in Jovancicevic et al. (2002) for the shale samples indicates migration of oil has taken place in sample number Dukul, Kutari and Lakun respectively (Fig. 7). This

diagram does not recognize the oil source rock potential of coals and coaly samples and can therefore not be used to evaluate such samples.

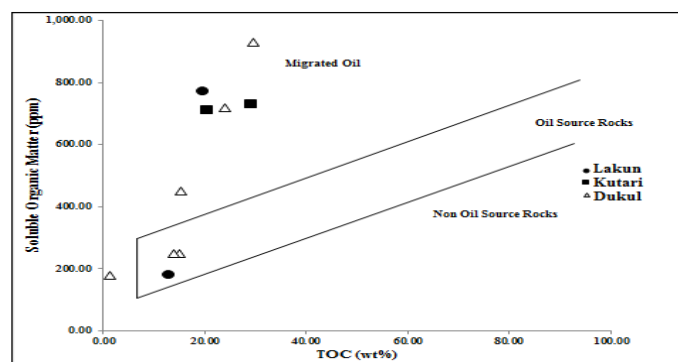


Fig. 7: A diagram showing the characterization of organic matter: SOM. vs TOC (based on Landis and Connan in Jovancicevic et al., 2002) of samples from Dukul Formation indicating migrated oil in the area.



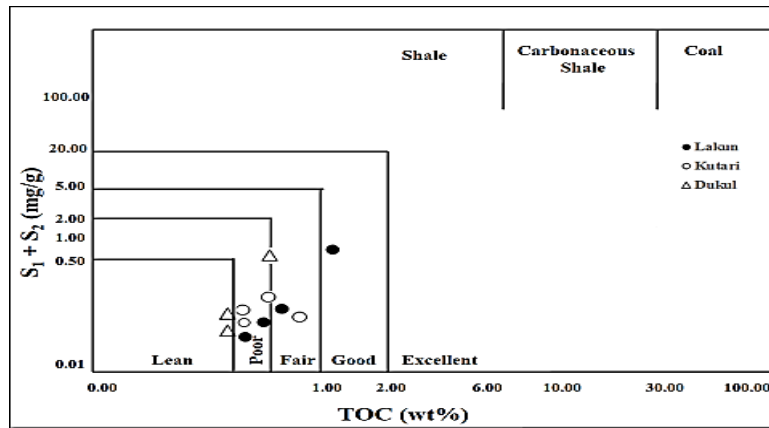


Fig. 8: A diagram indicating the quality of kerogen type in the shale samples:  $S_1 + S_2$  vs. TOC.

This is supported by the diagram of  $S_1 + S_2$  vs TOC (Fig. 8) characterizing the shale samples from the study area as good source rocks with TOC and  $S_1 + S_2$  above 0.1 wt% and 0.55 mg/g respectively. This is also supported by the report of Beka et al. (2007) from their investigations on shaly facies of gas prone sequences in the Dukul Formation based on the values of TOC (1.3-3.0 wt.%) and soluble organic matter (SOM) (180-930ppm) which are indicative of good to excellent source potential (Fig. 8).

Udofia and Akaegbobi (2007) also investigated the Cretaceous sediments around Yola arm of the Dukul Formation which revealed the exceeding minimum threshold TOC value (0.65-1.82 wt %) for shale samples.

According to Baskin (1997) classification, source rock with (HI) between 100-200 mg HC/g TOC are product of the organic matter type III. Peters and Cassa (1994) classified samples with (HI) less than 50 mg HC/g TOC as organic matter type IV. The relatively low hydrogen index (HI) values of the studied samples which range from 17.40 mg HC/g

TOC to 64.00 mg HC/g TOC (Table 1) suggest that the source rocks have potential for gas. The low (HI) values reported in this study is comparable with low trend of HI reported in the Cretaceous source rock facies (except Coal samples) in the Nigerian sedimentary basins (Obaje et al, 2004; Ehinola et al, 2005, Ogala, 2011, Uzoegbu, 2018; Uzoegbu and Ikwuagwu, 2016a,b; Uzoegbu and Amoke, 2017).

The production index ( $PI = S_1 / (S_1 + S_2)$ ) values > 0.1 (Table 2) observed on samples indicate possible impregnation by migrated bitumen or contamination by mud additives as seen in Figure 9 (Clementz, 1979; Durand and Oudin, 1979; Uzoegbu and Ikwuagwu, 2016b). Other samples with PI-values within 0.1 correspond to the expected results.

The study samples are mixed with recycled terrestrial organic worked organic matter (OM) pointing to type III (Fig. 4). These recycled organic matter (OM) might have been transported by fluvial processes (Bird et al., 1995; Obaje et al., 2004) as found in prodelta shales of a delta system.

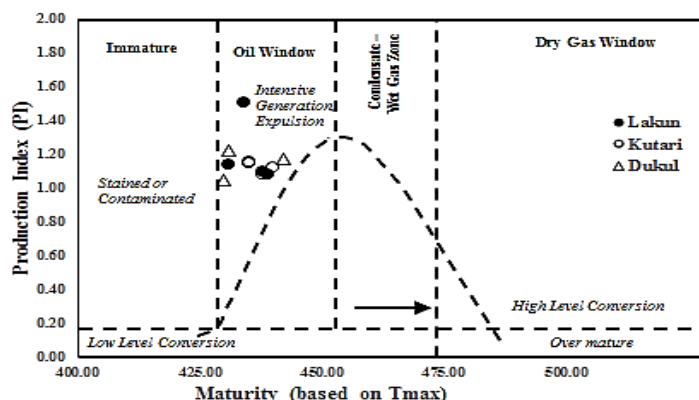


Fig. 9: Plot of production index (PI) versus Tmax showing kerogen conversion and maturity of the analyzed sediments in Dukul Formation (adapted from Hakimi et al. 2011).

### 4.3 Geochemical characteristics

The concentrations of extractable organic matter (EOM) together with the relative proportions of saturated, aromatic fractions, and NSO compounds have been calculated. The saturated and aromatic fractions together make up the petroleum-like hydrocarbon fraction; thus, the sum of these two fractions is referred to as HCs. The EOM yields a range from 1533.2 to 5722.2 ppm (Table 3), and it is noted that the EOM contains a complex mixture of hydrocarbons and non-hydrocarbon components (NSO).

The saturated fractions and NSO components are representing major fractions of the analyzed samples (Table 3). The saturated fractions and NSO compounds are ranging from 21.2 to 62.9 % and 24.4 to 61.4 %, respectively, whereas aromatic fractions range from 9.8 to 37.1 % (Table 3).

Since the hydrocarbon portion of the bitumen extracted from sediment is the petroleum-like portion, it is used as an important parameter in the source-rock evaluation (Philippi 1957; Baker 1972). In this respect, most of the Dukul samples are likely the most prolific

petroleum sources where abundant naphthenic oils might be expected to be generated (Fig. 10).

This is suggested by high hydrocarbon fractions (38.6–75.6 %; Table 3) and relatively high saturated hydrocarbon proportions (21.2–62.9 %). The hydrocarbon generative potential of a source rock can also be estimated from plots of TOC content versus extractable organic matter (EOM) and hydrocarbon yields (Fig. 10).

Pristane/phytane ratios range from 1.27 to 2.51 that indicate anoxic to oxic depositional environments. Steranes are dominantly of the C<sub>27</sub> forms. The predominance of type-III organic matter in this basin with dominantly marine depositional environments (as confirmed by the high contents of C<sub>27</sub> steranes) may be attributed to high oxic levels (high Pr/Ph ratios) which have downgraded organic matter preservation in the marine system. The relatively high Ts/Tm and low moretane/hopane ratios validate maturity levels that have entered the main phase of oil generation.

Table 3: Bulk geochemical results of extractable organic matter (EOM) yields (ppm) of the analyzed Dukul Formation samples.

Sample No.	Location	Formation	Lithology	Bitumen extraction data										
				Bitumen extraction and chromatographic fraction (ppm of whole rocks)					Chromatographic fraction of bitumen extraction (EOM wt%)					Bitumen/TOC (mg/g)
				EOM	Sat.	Aro.	NSO	HCs	Sat./EOM	Aro./EOM	NSO/EOM	HCs		
DUK-1	Lakun	Dukul	Shale	1699.60	605.60	327.80	766.20	933.40	35.60	19.29	45.08	54.90	0.05	
DUK-2	"	"	"	1533.20	777.20	382.20	373.80	1159.50	50.70	24.93	24.38	75.60	0.05	
DUK-3	"	"	"	3341.10	927.00	505.90	1908.20	1432.90	27.90	15.14	57.11	43.00	0.07	
DUK-4	Kutari	"	"	3473.50	1467.40	976.90	1029.20	2444.30	27.90	28.12	29.63	70.40	0.08	
DUK-5	"	"	"	2417.40	965.00	268.20	1183.90	1233.50	39.92	11.09	48.97	51.10	0.09	
DUK-6	"	"	"	3629.10	841.20	721.00	2066.90	1562.20	23.18	19.87	56.95	43.10	0.05	
DUK-7	Dukul	"	"	3225.10	1057.40	587.50	1580.20	1644.90	32.79	18.22	49.00	51.10	0.06	
DUK-8	"	"	"	3601.10	942.70	817.00	1841.40	1759.70	26.18	22.69	51.13	48.90	0.08	
DUK-9	"	"	"	3189.60	1159.90	679.90	1349.80	1839.80	36.37	21.32	42.32	57.70	0.07	

EOM extractable organic matter (bitumen extraction), Sat. Saturated hydrocarbons, Aro aromatic hydrocarbons, NSO nitrogen, sulfur, and oxygen components, HCs hydrocarbon fractions (saturated+aromatic).

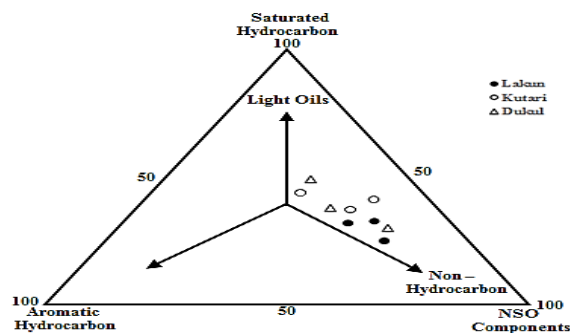


Fig. 10: Triangular diagram of saturate–aromatic–NSO compounds derived from EOM content.

#### *n*-Alkanes and isoprenoids

Whole extract gas chromatograms of the Dukul organic rich sediments show that *n*-alkanes are the dominant components. The *n*-alkane distributions display a full suite of saturated hydrocarbons between C<sub>12</sub>–C<sub>34</sub> *n*-alkanes and isoprenoids pristane (Pr) and phytane (Ph) (Fig. 11) and shows a predominance of low to medium molecular weight compounds (*n*-C<sub>14</sub>–*n*-C<sub>23</sub>) with the presence of significant waxy alkanes (+*n*-C<sub>23</sub>) thus gave moderate CPI values (Table 4). These distributions are typical of marine

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 Dukul samples (Fig. 12), and diagnostic biomarker ratios are listed in Table 4. 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).

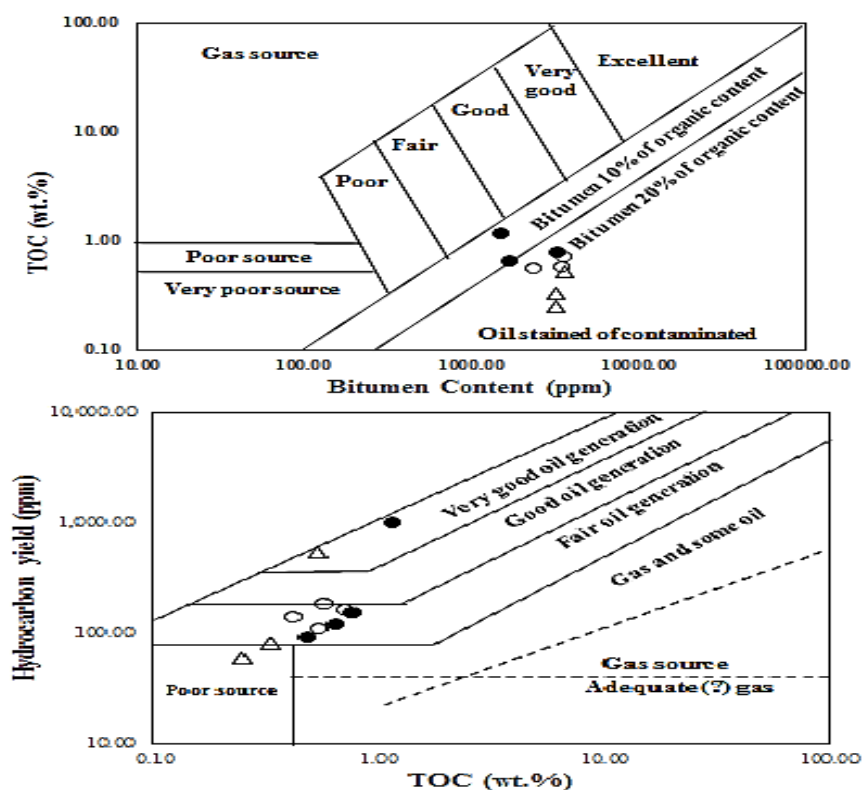


Fig. 11: Plots of TOC content versus bitumen extractions and hydrocarbon yields, showing source potential rating and hydrocarbon source-rock richness for the studied Dukul samples.

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 1.01–2.51 suggest that the Dukul 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. 12), thus giving distinctively low pristane/n- $C_{17}$  and phytane/n- $C_{18}$  ratios in the range of 0.03–

1.27 and 0.24–1.81, respectively, corresponding to mixed organic matter deposited under suboxic to relatively anoxic conditions (Fig. 13).

#### *Biomarkers (terpane and sterane)*

Triterpane and sterane biomarkers were measured from m/z 191 and m/z 217 mass chromatograms, respectively (Fig12). 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) (see Table 6).

The m/z 191 mass fragmentograms display high proportions of hopanes and tricyclic

terpanes occur in significant abundance (Fig. 12). C<sub>30</sub> hopane is found in high concentrations and C<sub>29</sub> norhopane is lower

with C<sub>29</sub>/C<sub>30</sub> ratios in the range of 0.31–0.72 (Table 4).

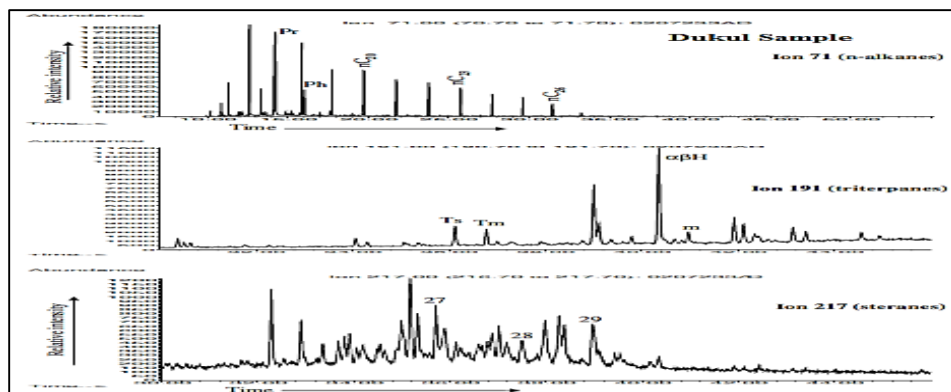


Fig. 12. Mass chromatograms of ions 71 (n-alkanes), 191(hopanes) and 217 (steranes) of shale samples from the Dukul Formation.

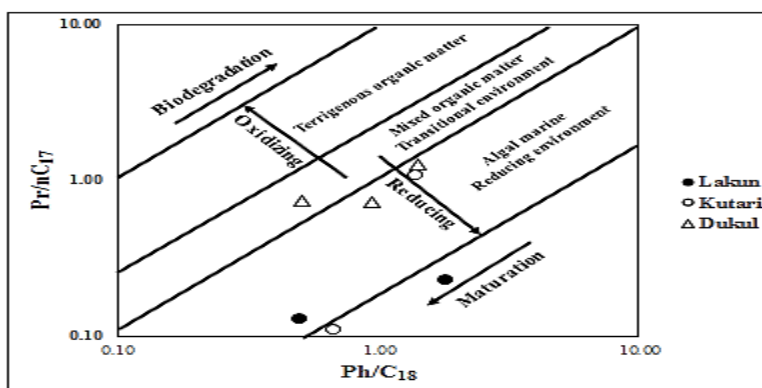


Fig. 13: Phytane to n-C<sub>18</sub> alkane(Ph/n-C<sub>18</sub>) versus pristane to n-C<sub>17</sub> alkane (Pr/ n-C<sub>17</sub>), showing depositional conditions of Dukul extracts (modified after Shanmugam 1985).

The predominance of C<sub>30</sub>hopane is frequently associated with clay-rich source rocks (Gürgey 1999). This statement is consistent with the lithofacies of the analyzed Dukul extracts. The Ts [C<sub>27</sub>18α(H) 22,29,30-trisnorneohopane] generally predominates over Tm [C<sub>27</sub> 17α(H)-22,29,30-trisnorhopane] (Fig. 12).

The Dukul extracts possess a wide range of Ts/Tm ratio values and ranging from 0.71 to 1.72 (Table 4). Values of C<sub>27</sub>17α(H)-22,29,30-trisnorhopane (Tm)and C<sub>27</sub> 18α(H)-

22,29,30-trisnorneohopane (Ts) are well known to be influenced by maturation, type of organic matterand lithology (Moldowan et al., 1985).

Homohopane distribution is represented by the dominance of C<sub>31</sub>and the concentration decreases toward high numbered homohopanes (Fig. 13). 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

Dukul shale and claystone sediments. The biomarker maturity parameters,  $C_{32}22S/(22S+20R)$  and moretane/hopane ratios are also calculated (Table 5). In addition, gammacerane was detected in the Dukul extract samples (Fig. 12). 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  $C_{29}$  sterane (relative to  $C_{27}$ – $C_{28}$ steranes) is present in the analyzed Dukul samples (Fig. 12). Relative abundances of  $C_{27}$ ,  $C_{28}$ , and  $C_{29}$  regular steranes are calculated, and the results are given in Table 5. The diasterane/sterane

ratios of the analyzed samples were calculated as shown in Table 2. The high diasteranes to steranes ratio (0.24–0.86) 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. 12). The  $C_{27}/C_{29}$  sterane and sterane/hopane ratios and two different sterane thermal maturity parameters,  $C_{29} 20S/(20S+20R)$  and the  $C_{29} \alpha\beta\beta$  ( $\alpha\beta\beta+\alpha\alpha\alpha$ ), are also calculated and listed in Table 5.

Table 4: n-alkane and isoprenoids biomarker ratios calculated from GC (TIC) and m/z 191 mass fragmentograms of analyzed Dukul Formation extracts.

Sample No.	Loation	Formation	Lithology	n-alkane and isoprenoids					Triterpanes and terpanes (m/z191)				
				Pr/Ph	Pr/C <sub>17</sub>	Ph/C <sub>18</sub>	CPI	OEP	C <sub>32</sub> 22S (22S+2)	C <sub>29</sub> /C <sub>30</sub>	C <sub>31</sub> R/C <sub>30</sub> H	MC <sub>30</sub> /HC <sub>30</sub>	Ts/Tm
DUK-1	Lakun	Dukul	Shale	1.02	0.23	1.81	1.99	1.86	0.53	0.61	0.13	0.19	1.03
DUK-2	"	"	"	0.98	0.13	0.50	1.87	1.89	0.57	0.50	0.15	0.16	1.16
DUK-3	"	"	"	1.12	0.03	0.30	1.91	1.93	0.58	0.54	0.12	0.12	1.72
DUK-4	Kutari	"	"	1.45	0.06	0.24	1.85	1.80	0.55	0.62	0.14	0.16	1.08
DUK-5	"	"	"	0.92	0.11	0.68	1.96	1.88	0.58	0.50	0.12	0.19	1.31
DUK-6	"	"	"	1.25	1.08	1.40	3.00	3.08	0.50	0.58	0.18	0.17	0.79
DUK-7	Dukul	"	"	0.94	0.72	0.94	5.82	5.16	0.50	0.63	0.11	0.12	0.82
DUK-8	"	"	"	1.32	1.27	1.42	8.72	7.06	0.54	0.64	0.11	0.13	0.84
DUK-9	"	"	"	1.20	0.74	0.51	7.20	6.28	0.56	0.57	0.13	0.16	1.26

Pr pristane; Ph phytane; CPI carbon preference index  $(2[C_{23}+C_{25}+C_{27}+C_{29}]/[C_{22}+2\{C_{24}+C_{26}+C_{28}\}+C_{30}])$ ; Ts [ $C_{27}$  18 $\alpha$ (H)-22,29,30-trisnorneohopane]; Tm [ $C_{27}$  17 $\alpha$ (H)-22,29,30-trisnorhopane];  $C_{29}/C_{30}$   $C_{29}$  norhopane/ $C_{30}$  hopane;  $MC_{30}/HC_{30}$   $C_{30}$  moretane/ $C_{30}$  hopane; H Index  $(C_{35}/(C_{31}-C_{35}))$  homohopane; Ts/Tm Ts/(Ts+Tm); diasterane/sterane ratio  $C_{29}$  diasteranes/ $C_{29}$  regular steranes.

Table 5: n-alkane and isoprenoids biomarker ratios calculated from GC (TIC), m/z 217 mass fragmentograms of analyzed Dukul Formation extracts.

Sample No.	Location	Formation	Lithology	m/αβH	Steranes and diasteranes (m/z217)						Steranes/	
					C <sub>29</sub> 20S+20R	C <sub>29</sub> αββ	C <sub>27</sub> /C <sub>29</sub>	Regular steranes (%)			Diasteranes	hopanes
					20S+20R (ββ+αα)	C <sub>27</sub>	C <sub>28</sub>	C <sub>29</sub>				
DUK-1	Lakun	Dukul	Shale	0.12	0.54	0.54	1.10	36.90	21.40	41.70	0.42	0.24
DUK-2	"	"	"	0.10	0.55	0.54	0.20	35.90	22.80	41.30	0.81	0.60
DUK-3	"	"	"	0.14	0.55	0.53	0.30	35.50	22.00	42.50	0.72	0.58
DUK-4	Kutari	"	"	0.14	0.52	0.53	0.80	36.20	21.40	42.40	0.38	0.35
DUK-5	"	"	"	0.18	0.51	0.54	0.20	36.20	22.10	41.60	0.86	0.81
DUK-6	"	"	"	0.21	0.52	0.54	1.10	35.10	22.10	42.80	0.33	0.23
DUK-7	Dukul	"	"	0.27	0.53	0.56	1.00	36.00	22.00	42.00	0.24	0.13
DUK-8	"	"	"	0.12	0.54	0.56	1.10	35.60	20.40	44.00	0.29	0.19
DUK-9	"	"	"	0.36	0.55	0.57	1.90	35.90	21.60	42.50	0.83	0.52

## (I) Peak number

Ts	18α(H),22,29,30-trisnorneohopane	Ts
Tm	17α(H),22,29,30-trisnorhopane	Tm
29	17α,21β(H)-nor-hopane	C <sub>29</sub> hop
30	17α,21β(H)-hopane	Hopane
30M	17β,21α(H)-Moretane	C <sub>30</sub> Mor
29M	17β(H),21α(H)-30-norhopane (normoretane)	Normoretane
31S	17α,21β(H)-homohopane (22S)	C <sub>31</sub> (22S)
31R	17α,21β(H)-homohopane (22R)	C <sub>31</sub> (22R)
32S	17α,21β(H)-homohopane (22S)	C <sub>32</sub> (22S)
32R	17α,21β(H)-homohopane (22R)	C <sub>32</sub> (22R)
33S	17α,21β(H)-homohopane (22S)	C <sub>33</sub> (22S)
33R	17α,21β(H)-homohopane (22R)	C <sub>33</sub> (22R)
34S	17α,21β(H)-homohopane (22S)	C <sub>34</sub> (22S)
34R	17α,21β(H)-homohopane (22R)	C <sub>34</sub> (22R)
35S	17α,21β(H)-homohopane (22S)	C <sub>35</sub> (22S)
35R	17α,21β(H)-homohopane (22R)	C <sub>35</sub> (22R)

## (II) Peak number

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. 8. The C<sub>27</sub>/C<sub>29</sub> sterane and sterane/hopane ratios and two different sterane thermal maturity parameters, C<sub>29</sub> 20S/(20S+20R) and the C<sub>29</sub> αββ (αββ+ααα), are also calculated and listed in Table 4.

#### 4.4 Thermal Maturity

Thermal maturity provides an indication of the maximum paleotemperature reached by a source rock. The T<sub>max</sub> values range from 431 to 439 °C, 435 to 440 °C and 431 to 442 °C in Lakun, Kutari and Dukul respectively (Table 1).

Shale samples of the Dukul Formation in the study area have T<sub>max</sub> values in the range of 431 to 442 °C and vitrinite reflectance values 0.57 to 0.75 Ro% (Table 2). These values correspond to maturity levels within the oil formation zone (Espitalié et al., 1984;

Ramanampisoa and Radke, 1992; Plumer, 1994; Gries et al., 1997).

Average T<sub>max</sub> and vitrinite reflectance of the Dukul Formation indicate a fairly increasing thermal evolution from the youngest Jessu Formation to oldest Yolde Formation.

Table 6: Peak assignments for alkane hydrocarbons in the gas chromatograms of saturated fractions in the m/z 191 (I) and 217 (II) mass fragmentograms compound abbreviation.

The thermal maturity levels attained by the source rock facies in the Yolde basin compared to the immaturity status of their stratigraphic equivalents (Pindiga and Gongila Formations) in Gongola Basin (Alkande et al., 1998a; Ojo, 1999) suggest possible influence of additional reheating by Tertiary Volcanics in the Yola Basin.

The production index (PI) is used to assess the generation status of source rocks but is often useful when homogeneous source rocks of different rank are compared, in which case it is characterized as the transformation ratio (Bordenave et al., 1993). The value of 1.16 of this shale is consistent with its vitrinite reflectance of 0.65%Ro. This maturity is also consistent with the fairly well fluorescing organic matter as well as Rock Eval T<sub>max</sub> of 436°C, reaching the 431-442°C for high sulphur mature source rocks containing Type III (Bordenave et al., 1993; Hunt, 1996). The PI is not affected by expulsion (Rullkötter et al., 1988) and this will not limit its use as an indicator of the organic matter transformation because generation may start for rocks with Type II at 0.55%Ro (Leythaeuser et al., 1980). Rullkötter et al. (1988) used a mass balance scheme to show that, at 0.68% Ro,

the transformation ratio in the Posidonia shale from northern Germany had reached 30%.

Wenger and Baker (1987) suggested that a PI from 1.08 to 1.96 is characteristic of source rocks in the oil window. The mean value of 1.2 for the shale samples is consistent with its Ro of 0.56 (Jovancicevic et al., 2002). This maturity is also consistent with the lowering of fluorescing organic matter, as well as Rock-Eval T<sub>max</sub> of 436°C, lower than the 430°C and 431 °C above 435°C for high sulphur mature source containing type III kerogen (Wenger and Baker, 1987; Landis and Connan, 1980; Uzoegbu and Ikwuagwu, 2016a, b; Uzoegbu and Amoke, 2017).

Several authors have attempted to relate the thermostability (T<sub>max</sub>) of OM to carbon structures (Oberlin et al., 1980; Monthious et al., 1982; Landis et al., 1984), their terrestrial or marine origin (Walker et al., 1983; Leckie et al., 1988) and the initial hydrogen content as a function of reaction kinetics (Fang and Jianyu, 1992). This has led to the proposal that the thermostability of carbon structure increases with the increase in oxygen content due to cross linkages such that the oxygenated functions can form between stacks of adjacent molecules (Rouxhet et al., 1979; Furimsky et al., 1983).

Walker et al. (1983) suggested marine derived amorphous-rich kerogens mature at lower temperatures than land plant (Type III) OM. Other investigators have demonstrated that terrestrial vitrinitic OM matures more rapidly than liptinic dominated organic matter (Tissot and Welte, 1984; Price and Barker, 1985; Wenger and Barker, 1987; Ikwuagwu and Uzoegbu, 2018) or that T<sub>max</sub>



values for marine sediments increase more slowly with increasing maturation than for non-marine sediments (Leckie et al., 1988). Tmax vs Oxygen index are plotted (Fig. 14) as proposed by Landis et al. (1984) who concluded that the initial hydrogen contents of vitrinite macerals under different redox conditions are different and hence have different reaction kinetics, because there is a strong deviation from the normal trend expected of Type III OM on their OI vs. Tmax diagram. This led to their conclusion that the anomalies recorded are not a result of the thermal conditions. Applying these principles to the area of study, samples are plotted on the OI vs. Tmax diagram of Landis et al. (1984) to test if samples followed the typical trend for Type III OM, and hence to determine if the Tmax anomalies have any relationship to thermal conditions. The trends in the study shale samples show essentially a decrease in OI as Tmax increases indicating the maturity level (Fig. 14). This reflects the

samples under study contain essentially Type III kerogen since they follow Landis et al. (1984) typical trend for Type III OM and immature to marginally mature stage.

Also in agreement with the report of Fang and Jianyu (1992), thermal conditions are partially responsible for the suppression of Tmax in the samples. According to Fang and Jianyu (1992), if Ro variations are caused by thermal conditions, Tmax and Ro values would have to increase with decreasing OI values as obtained in this study. In the diagram Tmax vs. HI (Fig. 6) indicates that most of the OM falls into oil and gas portion which implies that the OM contains oil and gas potential. Thermal maturity was confirmed by plotting the profiles of Tmax vs TOC (Fig. 15) showing that shale samples from the study area attain to “oil window” (430°C).

This is supported by the plots of HI versus pyrolysis Tmax and production index (PI) versus pyrolysis Tmax (Fig. 9).

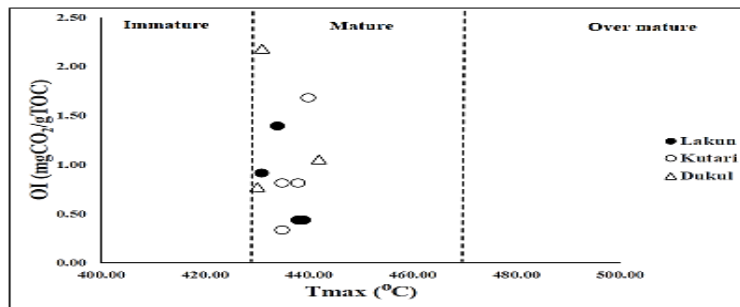


Fig. 14: A diagram of OI versus Tmax describing the anoxic and thermal of maturity organic matter in the shale samples within the diagenesis to catagenesis stages.

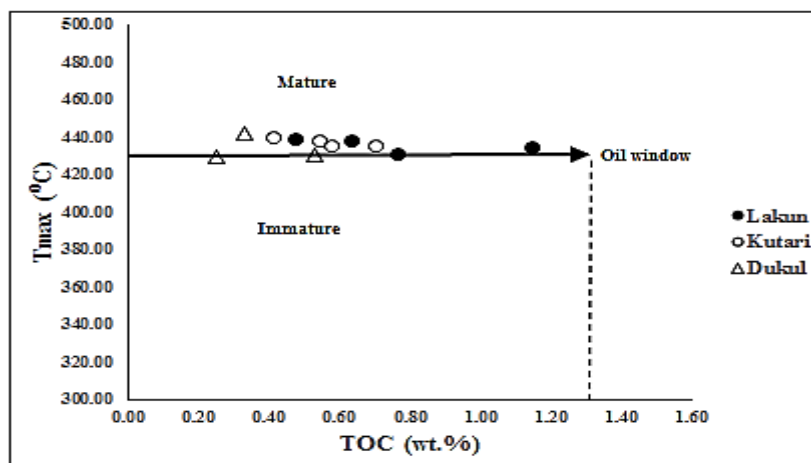


Fig. 15: To determine the maturity of the organic matter from shale samples using Tmax vs. TOC.

A variety of biomarker maturity indicators have been used to evaluate the level of thermal maturity of the Dukul organic-rich sediments; these include pentacyclic triterpanes and regular sterane isomer ratios. The ratios of C<sub>32</sub>homohopane 22S/(22S+22R), moretane/hopane, and C<sub>29</sub>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<sub>32</sub> 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 Dukulextracted samples have C<sub>32</sub>22S/22S+22R values in the range of 0.48–0.59, suggesting that they have reached equilibrium and that the oil window has been reached. The 20S/(20S+20R) and ββ/(ββ+αα)

C<sub>29</sub>sterane ratios of the Dukulextracts are ranging from 0.25 to 0.53 (Table 4). These biomarker maturation ratios are indicating that the analyzed Dukul samples have entered early mature for oil generation window (Fig. 16).

This is supported by moretane/hopane ratios consistent with low relative abundance of C<sub>30</sub> moretane. Moretane converts to C<sub>30</sub> 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 0.80 in immature sediments to about 0.15–0.05 in mature source rocks and oils (Mackenzie et al., 1980; Seifert and Moldowan, 1986). The Dukul samples have moretane/hopane (m/αβH) ratio in the range of 0.12–0.36, suggesting that samples are early mature. Overall, the biomarker thermal maturity parameters are indicating that all analyzed Dukul samples are at least early mature, and are likely to be approaching oil window maturity (Fig. 16) and therefore support the mean vitrinite

reflectance (%Ro) that from 0.57 to 0.75 %Ro (Table 2). 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 0.05 and 0.25, which correspond to early-peak oil window maturity as previously reported by Peters and Cassa (1994).

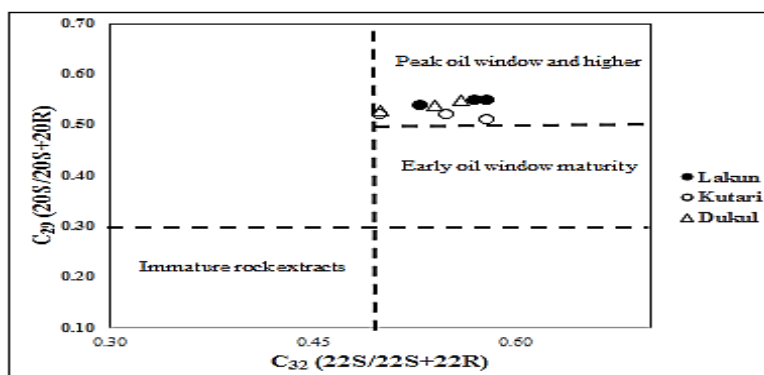


Fig. 16: A cross-plot of two biomarker parameters sensitive to thermal maturity of the Dukul extracts, which shows that most of the Dukul samples plot in the area of early oil window to peak oil window and maturity (modified from Peters and Moldowan 1993).

### Depositional Environment

In well-oxygenated formations, such as the Dukul Formation organic matter is reworked and aerobically oxidized to such an extent that it becomes unstable for anaerobic microorganisms. In this case, sulphate reduction and subsequent methane generation are suppressed. The values of HI/OI range from 1.33 to 35.00 (Table 2) indicating the land derived organic matter. Samples with highly oxygenated OM are distinguishable by their very low HI/OI ratio despite a terrestrial origin of the OM (Galimov, 2004; Uzoegbu, 2010). The majority of the terrestrial material is probably transported by fluvial processes (Bird et al., 1995; Obaje et al., 2004) contributing to the terrestrial source of OM of the studied samples.

The shale samples which is of kerogen type III with average OI of 0.98 mgCO<sub>2</sub>/gTOC and total sulphur (TS) of 1.38 wt.% describe depositional environment of the shale samples as shallow marine anoxic condition (Uzoegbu, 2010; Uzoegbu and Ikwuagwu, 2016b; Uzoegbu et al., 2013). The shale samples have elevated sulphur (S) contents (Table 2). The overall tendency towards lower sulphur (S) with increasing TOC, as well as the TOC/TS ratios for shale (0.95 to 2.68 wt.%; av. TOC/TS = 1.48) samples, argues for a marine environment during deposition as observed from the sulphur content (Berner, 1984; Ratnayake and Sampei, 2015a; Uzoegbu, 2010; Uzoegbu and Ikwuagwu, 2016b).

The Cretaceous Dukul sediments have been interpreted to have been deposited in a principally marine environment under

relatively low oxygen-deficient conditions (suboxic–anoxic). The results indicate that the Dukul sediments are characterized by relatively low TS contents (Table 2) and relatively high amounts of organic carbon with alginite and terrigenous organic matter (Fig. 17). TS contents are generally within the range observed in a freshwater deltaic

(Fig. 17; after Berner and Raiswell, 1983), and the presence of alginite with morphology similar to the recent alga sporinite and resinite in several of the samples further implies a terrestrial origin. This interpretation is also consistent with biomarker distributions (Table 4).

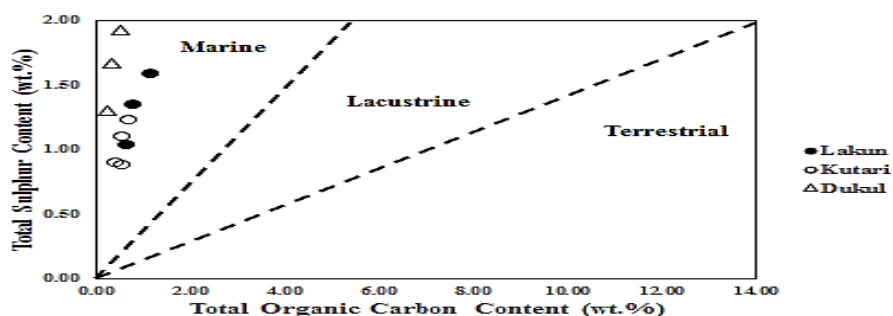


Fig. 17: Sulphur content plotted against TOC content, suggesting that the depositional environment of the analyzed Dukul sediments was dominantly freshwater lacustrine environment (modified after Berner and Raiswell 1983).

The n-alkane distributions are consistent with a typical of deltaic setting receiving mixed algal and terrestrial organic matter as indicated by kerogen microscopy (Fig. 3) and lower amounts of acyclic isoprenoids compared to n-alkanes (Fig. 12). This is suggested by a strong predominance of

C<sub>29</sub> regular steranes, consisting predominantly of plankton/algal with land plant organic matter (Fig. 18; after Huang and Meinschein, 1979).

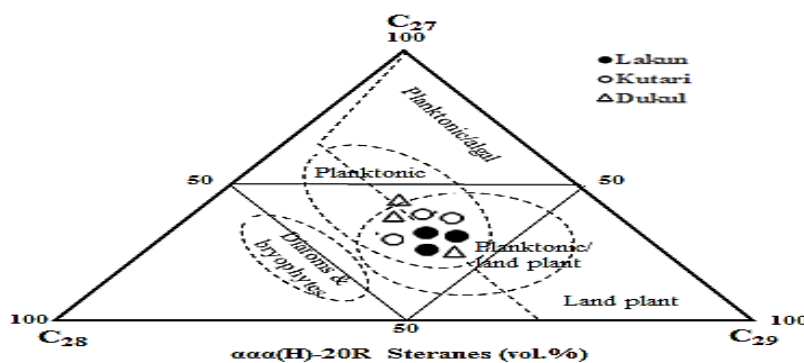


Fig. 18: Relationship between regular sterane compositions, organic matter input, and depositional environment for the analyzed Dukulextracts (modified after Huang and Meinschein, 1979).

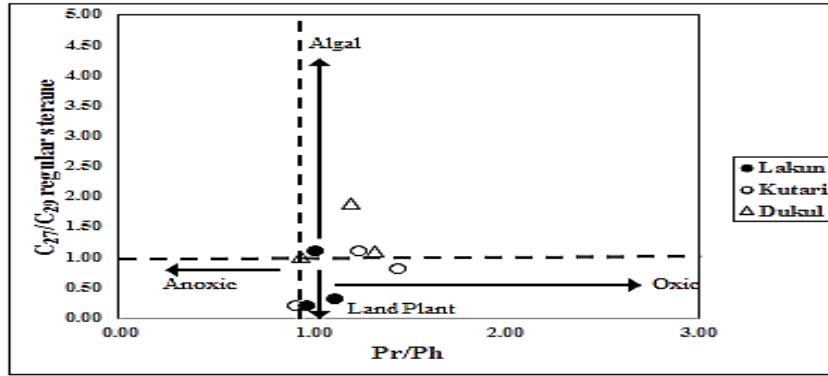


Fig. 19: A plot of pristane/ phytane versus  $C_{27}/C_{28}$  regular steranes, indicating organic matter input and depositional conditions of the Dukul extracts.

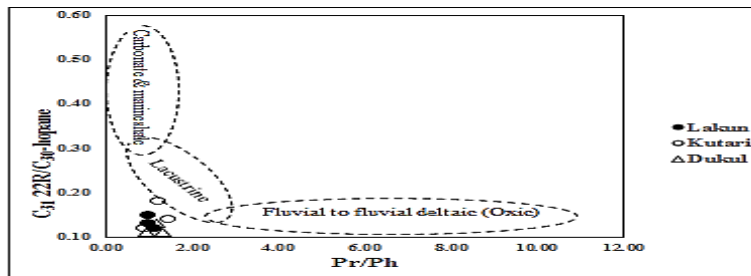


Fig. 20: Hopane and isoprenoid ratios of extracts which allow discrimination of the depositional environments of Dukul sediments (modified after Peters et al., 2005).

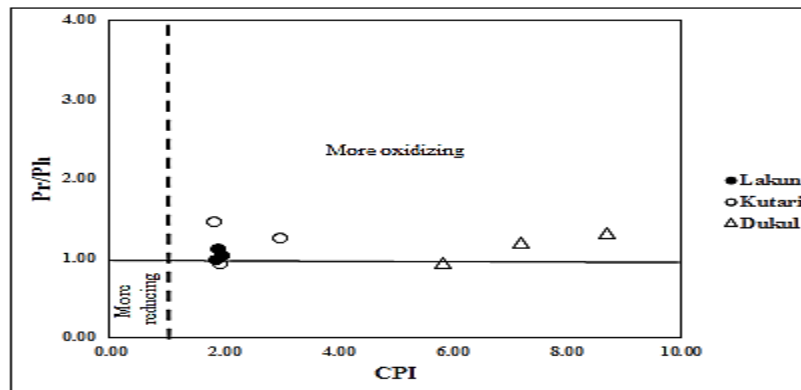


Fig. 21. Cross-plot of pristane/phytane ratios versus CPI indicating the depositional environment conditions of the studied samples (Adapted from Akinlua et al. 2007; El Diasty and Moldowan, 2012).

These conclusions are also supported by low values of  $C_{27}/C_{29}$  regular sterane ratios (Fig. 19) and the presence of relatively low concentrations of tricyclic terpane in the m/z 191 mass fragmentograms (Fig. 12). The low tricyclic terpane concentrations detected in the Dukul sediments (Fig. 20) is consistent

with being derived from the terrestrial 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. 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 Dukul extract samples are in the range of 0.11–0.20, 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. 20) and low sterane/hopane ratios ( $C_{29}$ -sterane/ $C_{30}$ -hopane) strongly argue for a marine depositional environment (Peters et al. 2005) of the organic matter. This is also supported by the plot of Pr/Ph against CPI (Fig. 21). In addition, the fine grain size of the inorganic matter (clay) and low-oxygen conditions (suboxic–anoxic) implies a low-energy depositional environment and favoured the preservation of organic matter, as indicated by elevated TOC contents and the high proportion of fluorescing sporinite and resinite organic matter.

### Conclusion

This study has shown that the sediments of the Cretaceous Dukul Formation in the Upper Benue Trough, NE Nigeria has an average total organic carbon (TOC) contents of up to 0.58 wt.%. The hydrogen index (HI) and generative potential (GP) of the shale samples in this study are fairly above the minimum values required for a potential source rock, suggesting that the sediments have gas generating potential. The organic matter is predominantly gas prone (mostly Type III). The level of thermal maturation derived from the Rock-Eval data show that the shale sediments are immature

to marginally mature and partly within the oil window. The values of OI (0.33–2.18 mgCO<sub>2</sub>/gTOC), TS (1.38) and TOC/TS (1.48) suggest deposition in a shallow marine environment.

The geochemical analyses indicate that the Dukul sediments 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 biomarker distributions have been performed on the Dukul 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 terrestrial-derived organic matter (algal and land plant) that preserved under suboxic to relatively anoxic conditions. This is further supported by 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 kerogen. Periods of desiccation envisaged during deposition of the Dukul sediments is, according to the presence of gammacerane.

With regard to thermal maturity, the Dukul organic rich sediments sequence under current investigation is thermally early mature oil-window generation as indicated by 22S/22S+22R  $C_{32}$  homohopane, 20S/(20S+20R) and  $\beta\beta/(\beta\beta+\alpha\alpha)$   $C_{29}$  sterane that have values in the range of 0.50–0.58, 0.51–0.55 and 0.53–0.57, respectively. This conclusion is consistent with vitrinite reflectance values

ranging from 0.57 to 0.75 % Ro, supporting that the oil window has been reached.

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